



NanoSPD8

8th International Conference on Nanostructured Materials by Severe Plastic Deformation

26 February - 3 March 2023



Organised by :

Department of Materials Engineering Indian Institute of Science, Bangalore - 560 012

Materials Engineering @ 75 : Celebrating 75 Years of Excellence in Research and Teaching

26TH FEBRUARY 2023 (SUNDAY)

Welcome and Registration – 4.30 to 6.30 pm

J N Tata Auditorium

27TH FEBRUARY 2023 (MONDAY)

		Inauguration: 09:15-)9:45 am (Main Hall, J N Tata Auditorium	1)		
About conf.	09:45-10:15	Expert Talk on Proceedings of NanoSPD by R	Expert Talk on Proceedings of NanoSPD by Ruslan Z Valiev			
Talk 1	am					
		Chair: Leo A. I. Kestens				
Plenary 1	10:15-10:50	Valery I Levitas				
(Main Hall)	am	Recent in situ experimental and theoretical adv	ances in severe plastic deformations, strain-induced	phase transformations, and nanostructure evolution		
		under high pressure				
Plenary 2	10:50-11:25	Boris Straumal				
(Main Hall)	am	Phase transitions driven by the high-pressure to	rsion in the binary Ti alloys H22			
		С	offee Break: 11.25-11.50 am			
		Main Hall	Hall - A	Hall - B		
		Chair: Werner Skrotzki	Chair: Subodh Kumar	Chair: R. S. Mishra		
Invited	11:50-12:20	Laszlo S Toth	H-G Brokmeier	David P Field		
(1-3)	pm	The basic mechanics of severe plastic	In-situ grain refinement down to nano scale by co-	Development and analysis of heterogeneous SPD		
		deformation processes	deformation of metals such as Cu-Nb extrusion	microstructures		
Invited	12:20-12:50	Nilesh P Gurao	Apu Sarkar	Stuart Wright		
(4-6)	pm	Effect of initial texture on high pressure	Severe plastic deformation of Nb-1Zr alloy: Effect	Spherical indexing of electron back-scattered		
		torsion induced omega phase transformation	of annealing on microstructure and mechanical	diffraction patterns in highly deformed materials		
		in commercially pure titanium	properties			
			Lunch: 12.50 – 02.00 pm			
		Chair: Kamanio Chattopadhyay				
Plenary 3	02:00-02:35	Andrea Bachmaier				
(Main Hall)	рт	Magnetic materials by severe plastic deformatio	n			
Plenary 4	02:35-03:10	Rimma Lapovok				
(Main Hall)	pm	Design of SPD - Made Hybrid Materials for En	ergy Applications			
		Main Hall	Hall - A	Hall - B		
		Chair: H.S. Kim	Chair: Vikram Jayaram	Chair: Ashok M Raichur		
Invited	03:10-03:40	Yuntian Zhu	James Mann	Kaveh Edalati		
(7-9)	pm	Plastic deformation processing of	Single step production of large scale foil, sheet	From functional nanomaterials to the origin of life by		
		heterostructured materials: An overview	and wire by shear based deformation processing	high pressure torsion		

Invited	03:40-04:10	Ruslan Valiev	K Trumble	Kaushik Chatterjee
(10-12)	pm	Designing nanoSPD materials for their	The cutting of gummy metals	Surface severe plastic deformation to enhance the
	-	multifunctional properties		biomedical performance of orthopaedic biomaterials
			Coffee break: 4.10 – 4.30 pm	
Invited	04:30-05:00	R Manna	Rajeev Kapoor	T S Sampath Kumar
(13-15)	pm	Microstructure and mechanical properties of	Achieving high strength ultra-fine grained	Nanostructured metallic implants with enhanced bio-
		ultrafine-grained materials	austenitic stainless steels by the process of	functionalization
			reversion of strain induced martensite	
Oral	05:00-05:20	Nagamani Jaya Balila	Shabnam Taheriniya	N Ramesh Babu
	pm	Deformation and fracture micro-mechanisms	Phase transition overview in the nanocomposite	Biomimetic PEO coatings for implants made of
		in HPT processed maraging steels	high entropy alloy produced by high pressure	nanostructured titanium alloys
			torsion	
Oral	05:20-05:40	M Ravi Shankar	Tatsuya Sugihara	Sabavath Janakiram
	pm	Confinement of low dimensionality unlocks	Direct observation of large-strain deformation at	An in-situ TEM study on restoration mechanisms during
		severe plastic deformability in Mg alloys	contact interface in wedge indentation of	low-temperature annealing of 80% cold rolled ferrite-
			aluminium	pearlite AHSS steels
		$28^{\mathrm{TH}}\mathrm{F}$	EBRUARY 2023 (TUESDAY)	
		Chair: Atul Chokshi		
Plenary 5	09:15-09:50	Chair: Atul Chokshi Jae-il Jang		
Plenary 5 (Main Hall)	09:15-09:50 am	Chair: Atul Chokshi Jae-il Jang Nanoindentation and novel structural materials	development	
Plenary 5 (Main Hall) Plenary 6	09:15-09:50 am 09:50-10:25	Chair: Atul Chokshi Jae-il Jang Nanoindentation and novel structural materials Werner Skrotzki	e development	
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Oral	12:00-12:20	Niraj Chawake	Anish Karmakar	Abhishek Tripathi
	pm	HPT studies on FCC medium entropy alloys and	Influence of equal channel angular rolling (ECAR)	Texture and Microstructure developments during
		understanding the role of GSFE on their	on the gradual microstructural evolution in Al-	multipass friction stir processing of Magnesium Alloy
		deformation behavior	4.5Mg-0.14Si alloy	AZ31
Oral	12:20-12:40	Surya Nilamegam Kumaran	Raj Bahadur Singh	Vasanth Chakravarthy Shunmugasamy
	pm	Texture prediction of severely deformed high-	Deformation of Inconel 800 through equal channel	Preparation of ultra-thin walled magnesium AZ31 alloy
		entropy alloy by crystal plasticity simulations	angular pressing	tubes using friction stir extrusion
Oral	12:40-1:00	Piotr Bazarnik	Dmitrii Panov	Harpreet Singh Arora
	pm	Effects of aluminum purity and addition of	Effect of swaging and following annealing on	Nano-tubular architecture through severe physical
		carbon nanotubes on the hardness and thermal	gradient structure formation in austenitic stainless	deformation for high performance supercapacitor
		stability of CNT reinforced aluminum	steels with various stacking fault energy	
		nanocomposites processed by the high-pressure		
		torsion technique		
			Lunch: 1.00-2.00 pm	
DI 7	2 00 2 25	Chair: Gerhard Wilde		
Plenary 7	2:00-2:35	Leo Kestens		1 · · · · · · ·
(Main Hall)	pm	Texture control in SPD processed alloys by (dy	namic) high-pressure torsion and accumulative roll t	oonaing
Plenary 8	2:35-3:10	Julia Ivanisenko		
(Main Hall)	pm	Scaling-up of Severe Plastic Deformation –a S	tory of Success without a Happy-end	
		Poster	Session and High Tea: 3.10-5.30 pm	
		BANQUET A	AT GOKULAM GRAND: 07.00 – 9:30 pm	
		1 ST MARCH 2023	(WEDNESDAY): EXCURSION-MYSORE	E
		2 ND	MARCH 2023 (THURSDAY)	
	1	Chair: Julia Ivanisenko		
Plenary 9	09:15-09:50	Atul H Chokshi		
(Main Hall)	am	The Zen of Grain Boundaries		
Plenary 10	09:50-10:25	Sergiy Divinski		
(Main Hall)	am	'Non-equilibrium' grain boundaries in additiv	ely manufactured alloys: CoCrFeMnNi high-entropy	alloy as a case study
		Main Hall	Hall - A	Hall - B
		Chair: Kaveh Edalati	Chair: Laszlo S. Toth	Chair: S. Chandrasekhar
		Co-Chair: Chandan Srivastava	Co-Chair: S. Sankaran	Co-Chair: Prosenjit Das
Invited	10:25-10:55	Gerhard Wilde	Pradipta Ghosh	Vivek Pancholi
(22-24)	am	Impact of structural transformations and	Origin of anistropic mechanical behaviour of high-	Microstructural characterization of friction-stir
		defect interactions at grain boundaries in nanocrystalline metallic materials	pressure torsion deformed metals and alloys	processed green Al powder compact

Oral	10:55-11:15	Roman Karelin	Soumita Mondal	Ajay Kumar		
	am	Low-temperature processing of NiTi shape	Investigation on the effect of high pressure torsion	Friction-stir processing of squeeze cast A356 with		
		memory alloy by ECAP in shells	on the Precipitates in Sc modified Al –Li alloy	surface compacted graphene nanoplatelets (GNPs) for		
				the synthesis of metal matrix composites		
	Coffee Break: 11.15-11.30 am					
Invited	11:30-12:00	Somjeet Biswas	R Jayaganthan	K S Suresh		
(25-27)	pm	Twin induced compressive strain hardening	Mechanical properties and microstructural	Origin of grain size stability in friction stir processed		
		behaviour in titanium	evolution of Zr-4 alloy processed though rotary swaging	Al powder compact		
Oral	12:00-12:20	Simon Pillmeier	G Bharat Reddy	Arockia Kumar Raju		
	pm	Fatigue crack growth behavior of a nanocrystalline deformed Ti Nb alloy	Producing hierarchical nano-twins in Zr alloys through multiaxial cryo forging for strength ductility enhancement	Friction stir processing of Zn-Mg biodegradable alloys		
Oral	12:20-12:40	Kausik Chattopadhyay	Swati Mahato	Máté Szűcs		
	pm	Corrosion and low cycle fatigue behaviour of	Development of metastable dual-phase ternary	Metal bonding by Friction-Assisted Lateral Extrusion		
		ultrasonic shot peened Ti 13Nb 13Zr alloy	medium entropy alloy using cryorolling technique	Process (FALEP) at room temperature		
Oral	12:40-1:00	Sandeep Sahu	Krishan Kumar Pandey	Shivram Thapliyal		
	pm	Microstructure and corrosion behaviour of	In situ experimental studies of plastic strain induced	Underwater friction stir welding route for developing		
		additive manufactured AlSi10Mg alloy	$\alpha \rightarrow \omega$ phase transitions in ultra-pure Zr and	hybrid joints of aluminum and polymer of dissimilar		
		processed by HPT	Zr2.5Nb alloy	thickness		
			Lunch: 1.00-2.00 pm			
			Online Talks (Main Hall)			
		Chair: Praveen Kumar				
Online Talk	02:00-02:30	Nobuhiro Tsuji				
	pm	Nucleation of New Deformation Modes in Fully	Annealed Nanostructured Metals			
Online Talk	02:30-03:00	Zenji Horita				
	pm	In Situ Synchrotron X-ray Analysis for Allotrop	ic Transformation of ZnO processed by SPD			
Online Talk	03:00-03:20	Borisovich Naimark				
	pm	On thermodynamics of nanostructured state and	l mechanical properties of NanoSPD materials in wide	range of load intensity		
Online Talk	03:20-03:40	Nariman Enikeev	1 · 1 1 · 1 ·			
	pm	Under water friction stir welding route for deve	loping hybrid joints of Al and polymer of dissimilar thi	ckness		
			Coffee Break: 3.40-4.00 pm			
		Presentation by Sponsors (Main Ha	nll): 4:00 – 5:20 PM Chair: Surendra	K. Makineni		
			High Tea: 5:20 – 6:00 PM			
	CULTURAL PROGRAM (Main Hall): 6:00-7:00 pm					

	3 RD MARCH 2023 (FRIDAY)				
		Main Hall Chair: M.J.N.V. Prasad Co-Chair: Ankur Chauban	Hall – A Chair: Sergiy V. Divinski Co-chair: B S S Daniel	Hall – B Chair: Borris Stramual Cochair: Nilesh P. Gurao	
Invited (31-33)	09:15-09:45 am	Uday Chakkingal Improvement in formability of Ti and Mg alloys by equal channel angular pressing	Rama Krishna Sabat Ductility enhancement in Mg-0.2%Ce alloys	Megumi Kawasaki In-situ heating neutron diffraction analysis of structural relaxation in additive-manufactured 316L stainless steel	
Invited (33-36)	09:45-10:15 am	Shashank Shekhar Redesigning constrained groove pressing technique to overcome strength-ductility limitations	B S S Daniel Dynamic working and mechanical property of homogenised AA7068	Venkateswarlu K The Influence of SPD process on 3-D printed Al-10 Si- 0.5 Mg alloy	
Invited (37)	10:15-10:45 am	S K Panigrahi Investigation into microforming capabilities of engineered UFG Al and Mg alloys	Oral (10:15-10:35) Debdas Roy Improvement in mechanical and electrical properties of Cu-graphene nanocomposites prepared by high pressure sintering	Oral (10:15-10:35) Dan Sathiaraj Surface severe plastic deformation of wire arc additive manufactured pure copper	
Oral	10:45-11:05 am	Devinder Yadav Friction surfacing: A new way to repair surface cracks	Bheemreddy Prathyusha A novel manufacturing method to develop ultra-fine grained Al/Cu bimetals	Prakash Chandra Gautam Effect of ECAP routes on microstructure, texture, and mechanical properties evolution in pure magnesium	
			Coffee Break: 11.05-11.20 am		
Invited (38-40)	11:20-11:50 am	V Subramanya Sarma Effect of strain rate on the retained austenite stability in a Medium-Mn steel	S Sankaran Thermal stability of microstructure and texture in UFG Al-Mg-Sc-Zr alloys processed through severe cold rolling and annealing	M J N V Prasad Development of gradient metallic materials and coatings by electrodeposition: A strategic approach to improve the resistance to mechanical degradation	
Oral	11:50-12:10 pm	Prakash G Ranaware Investigation of effect of severe plastic deformation on laser transformation hardening of low carbon low alloy steel	Esakkiraja Neelamegan Interdiffusion behavior in a severely deformed Cu Ni diffusion couple	Yagnesh Shadangi Surface nanostructuring and thermal stability of IN718 superalloy treated by ultra-shot peening	
Oral	12:10-12:30 pm	Saurav Sunil Mechanical behaviour of ultrafine grained SS304L produced by reversion of strain induced martensite formed by severe plastic deformation	Shavi Agrawal Evolution of microstructure and mechanical properties of 2099 aluminium alloy deformed by high-pressure torsion	Balkrishna C Rao Ultra-fine-grained foils from extrusion machining of a nickel-based super alloy	
Oral	12:30-12:50 pm	B Aashranth Severe plastic deformation of a lath martensitic steel: exploring the microstructural origins of exceptional strengthening	Girish Bojjawar Influence of Co content on the simultaneous enhancement of strength and ductility in severely drawn textured Ni-Co microwires	Manoel Kasalo Mechanical and tribological performance of severe plastically deformed Ni base super alloy composites	
Oral	12:50-01:10 pm	Chavan Akash Naik	Malgorzata Lewandowska	Abheepsit Raturi	

		Structure-property correlations at a micro- meter length scale of hot rolled and cold rolled dual phase steels	Ductility and formability of ultrafine-grained aluminium plates	Microstructure and microtexture development of non- equiatomic Mo-Nb-Ta-V-W refractory HEA during HPT at 473K	
	-	-	Lunch: 1.10-2.00 pm		
	Time	Main Hall Chair: Uday Chakkingal Co-Chair: Abbik N. Chowdhury	Hall – A Chair: Megumi Kawasaki Co-Chair: Sachin Rondiya	Hall – B Chair: Reema Lapovok Co-Chair: Bhagwati Prasad	
Invited	02:00-02:30	Koteswararao V Rajulapati	Koushik Viswanathan	Satish V Kailas	
(41-43)	pm	Mechanical response of multi-phase nanostructured high-entropy alloys prepared by different severe plastic deformation methods	A robust in-situ technique for evaluating sub- surface plastic strains	Polymer derived ceramic metal matrix composites; High strength, High Ductility, High temperature grain stability	
Oral	02:30-02:50 pm	Narayan K Sundaram <i>Modelling and simulation of large strain</i> <i>complex plastic flows in polycrystalline</i> <i>aggregates</i>	Saeid Akrami CO ₂ photo-reduction using high-pressureTiO2-II synthesized by high-pressure torsion (HPT)	Lukas Weissitsch Bulk rare earth free permanent magnets by severe plastic deformation and advanced annealing procedures	
Oral	02:50-03:10 pm	Anup Basak Grain boundary- induced martensitic nanostructures in bicrystals and tricrystals - Large strains-based phase-field study	Parisa Edalati Using the high-presure torsion (HPT) to fabricate a high-entropy oxynitride for photocatalytic H2evolution	Franziska Staab Hard magnetic SmCo5-Cu nanocomposites produced by HPT	
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Oral	04:05-04:25 pm	Fedor Vodolazskiy Structure refinement and property evolution of the Ti-3AL-2.5V alloy during tube producing by the TREX technology	Sanika Paranjape Simultaneous improvement in Strength and Ductility in deformed pure magnesium	Sunil Kumar Recent advancements in constrained Groove pressing process	
Oral	04:25-04:45 pm	Elias C. Aifantis (Pre-recorded Talk) Revisiting the classical laws for nanomechanics	Timur B. Minasov (Pre-recorded Talk) In vivo osseointegration of nanotitanium implants	A Krishnaiah Investigations on performance of EDM by modified copper electrodes with relief angle and corner radius	
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Recent in situ experimental and theoretical advances in severe plastic deformations, strain-induced phase transformations, and nanostructure evolution under high pressure

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ABSTRACT

The main problem in studying plasticity, plastic strain-induced phase transformations (PTs), and structural changes is that they depend on five components of the plastic strain tensor and its entire path [1,2], making a huge number of combinations of independent parameters. New in situ experimental results are obtained under compression of materials in a diamond anvil cell (DAC) and torsion in rotational DAC (RDAC). Materials under study include carbon [3], Zr [4,5], BN, Si, and olivine. Drastic reduction (by one to two orders of magnitude [3-5]) of the PT pressure compared with hydrostatic loading and the appearance of new phases are demonstrated. Thus [3], graphite in RDAC was transformed to nanocrystalline hexagonal and cubic diamond at 0.4 and 0.7 GPa, respectively, which are 50 and 100 times lower than the PT pressures under hydrostatic compression and well below the phase equilibrium pressure of 2.45 GPa. This could be a precursor of new technology of plastic strain (defect) induced diamond synthesis and explains the appearance of microdiamonds in the severely sheared regions of the Earth's crust at low pressure and temperature. The rough diamond anvils (rough-DA) are introduced [5] to reach maximum friction equal to the yield strength in shear, which allows the determination of pressure-dependent yield strength and reproduces conditions under high-pressure torsion with metallic/ceramic dies. The rough-DA were used for compression of severely pre-deformed Zr. It is found in situ that after severe straining, crystallite size and dislocation density of α and ω -Zr are getting pressure-, strain- and strain-path-independent, reach steady values before and after PT, and depend solely on the volume fraction of ω -Zr during PT. Immediately after completing PT, ω -Zr behaves like perfectly plastic, isotropic, and strain-path-independent. The minimum pressure for plastic strain-induced α - ω PT is also independent of plastic strain- and strain-path [4,5]. It was reduced from 1.36 GPa in smooth DAC to 0.67 GPa with rough DAC. This is the record low PT pressure for Zr, much lower than 6.0 GPa under hydrostatic conditions, and phase equilibrium pressure, 3.4 GPa. The kinetics of strain-induced PT, in addition to plastic strain, unexpectedly depends on time. This opens an unexplored field of the simultaneous strain- and stress-induced PTs under pressure. The obtained results create new opportunities in material design, synthesis, and processing of nanostructured materials by severe plastic deformations at low pressure. The results are interpreted in terms of four scale theories for plastic strain-induced PTs [1] and challenge some aspects of the theory. Molecular dynamic [6,7] and first-principle [8] simulations were used to determine lattice instability conditions under all six components of the stress tensor. At the nanoscale and microscale, nucleation at various evolving dislocation configurations was studied utilizing developed nanoscale [9] and scale-free [10] phase-field approaches. The possibility of reducing PT pressure by more than an order of magnitude due to stress concentration at the shear-generated dislocation pileup is proven. At the microscale, a strain-controlled kinetic equation was derived and utilized in the large-strain macroscopic theory for coupled PTs and plasticity. At the macroscale, the behavior of the sample in DAC/RDAC is studied using the finite element approach [11]. Various experimental effects are reproduced. The theory also resolves the main puzzles of the PT-based mechanism of the deep-focus earthquake [12].

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Phase transitions driven by the high pressure torsion in the binary Ti alloys

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ABSTRACT

The high pressure torsion (HPT) of various binary Ti alloys with β -stabilizers (Fe, Co, Ni, Mo, Nb, Ta) was studied. Before HPT, the samples were annealed and contained (i) pure β -phase, (ii) α + β mixture with different portion of phases, (iii) α' or α'' martensites, (iv) the mixture of α -Ti and respective intermetallic phase. The microstructure of Ti alloys before and after HPT was studied by scanning and transmission electron microscopy (also high resolution one), X-rays diffraction (including the high-temperature in situ one), differential scanning calorimetry, atomic probe tomography, synchrotron irradaiation.

During HPT the (usual) strong grain refinement took place. Also, it was observed that HPT can lead to various phase transitions in the Ti alloys. In particular, the metastable high-pressure ω -phase and α' or α'' martensites can form. The composition of phases (similar to their grain size) reached the steady-state value after about 1.5 plunger revolutions. In some cases, the equifinality of the HPT-driven phase transitions was observed. Equifinality means that the composition and portion of phases after HPT do not depend on the composition and portion of phases before HPT.

The HPT-driven phase transitions can be martensitic (i.e. without or almost without mass transfer) of diffusional (i.e. with mass transfer). In case of martensitic β -to- ω or α -to- ω phase transitions, the certain orientation relations between β - and ω or α and ω were observed. The thermal stability of the ω -phase obtained by HPT has been studied by the in-situ X-rays diffraction at high temperatures. The ω -phase in the HPT-treated Ti alloys with β -stabilizers can remain in the samples up to 500-600°C. It is much higher than in pure titanium (~150°C).

Thus, the HPT-driven phase transitions open the new way for tailoring of grain size and phase composition of Ti-based alloys. In turn, it gives the new instrument in hands of engineers to improvement of the technologically important properties of Ti-based alloys. It is especially important for the medical application like the teeth of bone prosthesis.

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The basic mechanics of severe plastic deformation processes

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ABSTRACT

Severe plastic deformation (SPD) processes have been extensively researched since about 1990. Most contributions to the evolution of SPD have originated from materials scientists. However, achieving the full potential of SPD requires a good understanding of the mechanics of the applied processes. In this presentation both the macro- and micro-mechanics underlying the major SPD techniques will be examined. Special attention will be given to the definition of equivalent strains, the role of friction, the hydrostatic stress, and the mechanical aspects of the grain fragmentation processes, which is one of the main benefits of using SPD processes.

In-situ grain refinement down to nano scale by co-deformation of metals such as Cu-Nb extrusion

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ABSTRACT

Co-deformation of metals leads to very special properties, which have long been the focus of intensive study. One of the main reasons is the mutual influence during deformation. Co-deformation of materials can be divided into several groups.

First, there is the group of same crystal structures such as Al-Pb or Al-Cu composites with fcc structures. Here the individual materials differ mainly in the stacking fault energy and not in the slip systems. The deformation resistances are comparatively low, so that very high degrees of deformation are required to achieve grain refinement down to the nano range.

A second group are fcc-bcc systems such as Cu-Nb or Cu-Fe. It is well known that bcc and bcc materials have different deformation mechanisms, which can lead to significantly different deformation resistances. Two phenomena have been identified here. In the Cu-Nb system, where there are no intermetallic compounds, a high mutual influence of both components occurs, resulting in high grain boundary stresses. With the methods used in the early 2000s, the grain refinement effect could only be guessed at, which could be proven using more recent EBSD technology. In the Cu-Fe system, another effect has been found, based on the fact that the deformation resistances of Fe and Cu do not vary very much. It was known that Cu-Fe composites form a curling microstructure. The texture evolution suggests that during increasing deformation (increasing von Mises stress), the ratio of deformation resistances Cu/Fe reverses and this several times. Sometimes Fe has the higher deformation resistance, sometimes it is Cu. The resulting high grain boundary stresses have a great influence on the microstructure. This is of course also dependent on the mixing ratio. A third group are composites between hexagonal and cubic metals. These can include classic powder-metallurgically produced composites such as Mg-Al, as well as reactive in-situ two-phase materials such as Ti-Ti. In these systems with hexagonal materials, forming usually takes place at high temperatures. Recrystallization has a negative effect on grain

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Development and Analysis of Heterogeneous SPD Microstructures

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ABSTRACT

Severe plastic deformation techniques have been used to develop structures with fine grain sizes in an attempt to improve the resultant mechanical properties. This has subsequently led to techniques that can produce structures with gradients in grain sizes and crystallographic textures that result in improved properties, sometimes both strength and ductility are improved simultaneously. The processes employed typically involve complex flow patterns and sometimes recrystallization in either dynamic or static conditions. This presentation focuses on microstructures observed from specimens produced by various gradient producing techniques including severe shot peening, high pressure torsion, and shear assisted pipe extrusion (ShAPE forming). The microstructure evolution during these processes is complex and results in structural gradients during the forming process before the final structure is reached. Textural analysis by electron backscatter diffraction (EBSD) along with crystal plasticity modeling can be used to back out the material flow during these processes by identifying the major shear planes and directions as indicated from the observed textures. Measurements using EBSD are used to demonstrate the rich structures that develop during these processes.

Acknowledgments

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Effect of initial texture on high pressure torsion induced omega phase transformation in commercially pure titanium

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ABSTRACT

The role of initial texture on grain refinement induced omega phase transformation during high pressure torsion of commercially pure hcp titanium was studied. To this end discs with initial basal and prismatic-pyramidal texture along the normal direction machined from a rolled and annealed block of commercially pure titanium were subjected to high pressure torsion at a pressure of 5 GPa at room temperature to five turns. Detailed microstructural characterization performed using micro-focus synchrotron diffraction, electron backscatter diffraction and orientation imaging microscopy in transmission electron microscope indicated initiation of hcp alpha to hexagonal omega phase transformation at a lower strain in prismatic-pyramidal sample while the basal orientation showed a higher fraction of omega phase after 5 turns. The micro-mechanisms of grain refinement induced phase transformation for the two initial textures will be elaborated for different transformation pathways. The correlation of deformation microstructure and micro-hardness evolution during high pressure torsion in the context of grain refinement and dislocation density of the two phases along with the evolution of micro-hardness will be discussed.

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Severe Plastic Deformation of Nb-1Zr alloy: Effect of annealing on microstructure and mechanical properties

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ABSTRACT

Nb-1wt.%Zr was subjected to severe plastic deformation at room temperature using multi-axial forging (MAF) in a closed channel die up to different effective strains. Microstructure and mechanical properties of the deformed samples were investigated using EBSD, X-ray line profile analysis and compression testing. Dislocation density in the material increased up to a certain effective strain and then showed decreasing trend. Specimens prepared form a sample deformed up to an effective strain of 4.6 were subsequently annealed for 1 h at 450, 550, 600 and 700 °C to study the thermal stability of the microstructure after severe deformation. EBSD measurements were used to characterize the microstructure in terms of grain size, local misorientations within grains and the fraction of recrystallization after annealing using the criteria of grain orientation. Subsequent annealing resulted in regions of recrystallized grains and an overall reduction in local misorientations. Annealing at 700 °C showed a bimodal grain size distribution with large recrystallized grains surrounded by deformed regions. The tensile strength reduced with increasing annealing temperature. The as-MAF and the 450, 550 and 600 °C annealed conditions showed low ductility (16%), whereas the 700 °C annealing condition showed a moderate ductility of 28% as compared to the coarse grained condition which showed a 44% ductility. It was estimated that the contribution of strength increase after MAF was more due to increase in dislocation density as compared to the reduction in grain size.

Spherical Indexing of Electron Backscatter Diffraction Patterns in Highly Deformed Materials

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ABSTRACT

Traditionally, electron backscatter diffraction (EBSD) patterns have been indexed using a combination of the Hough Transform for detecting the bands in the patterns coupled with various indexing schemes to identify the crystal planes associated with the detected bands primarily based on the angles between the bands in comparison with the interplanar angles. However, this approach can struggle with patterns of poor contrast as are often the case in highly deformed materials due to both spatial resolution limitations as well as local perturbations of the crystal lattice due to a high density of dislocations. Dictionary indexing (Chen et al, 2015) has shown promise for improved indexing of low-quality patterns. Further developments of the dictionary indexing include (1) cross-correlating a back-projected experimental pattern onto a sphere using a spherical harmonic transform (Hielscher, Bartel and Britton, 2019; Lenthe, Singh and De Graef, 2019) and (2) local refinement of crystal orientation using an iterative targeted dictionary indexing approach (Tanaka and Wilkinson, 2019; Pang, Larsen and Schuh, 2020). These techniques have been applied to several severely deformed materials to evaluate the effectiveness of these approaches. Dramatic improvements have been achieved in several cases. An example is shown in figure 1 for highly deformed duplex steel wire. The advantages and disadvantages of these approaches is presented both in terms of indexing performance as well as computational resources.

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Figure 1. Indexing results for heavily deformed duplex steel wire

Magnetic materials by severe plastic deformation

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ABSTRACT

Severe plastic deformation (SPD) is a well-known method of nanocrystalline and ultrafine grained material synthesis. One key feature of SPD is the constancy of sample's volume and shape during the deformation process. Thus, it enables the production of bulk samples. The use of SPD further allows the generation of innovative metastable nanomaterials with compositions and phase constituents beyond the equilibrium phase diagram and with fewer thermodynamic restrictions. Additionall, nanocomposites can be manufactured by SPD.

SPD processing of magnetic materials and its influence on the magnetic properties have attracted increasing research interest in the last years. In this talk, the use of high-pressure torison (HPT) deformation to synthesize different nanostructured soft and hard magnets is in focus and our recent experimental results are discussed.

In HPT deformed binary and ternary immiscible systems, soft magnetic properties are obtained. In Cu-Co and Cu-Fe-Co alloys, for example, the coercivity can be tuned by varying the chemical composition. Soft magnetic properties are also achieved by HPT induced amorphization of Co-Zr and subsequent annealing. From an application viewpoint, the most important magnetic properties are magnetoresistivity and magnetostriction. Thus, the magnetoresistive and magnetostrictive properties in the immiscible material systems are investigated as well. Additonally, the use of the HPT deformation process to synthesize magnetic nanocomposites and exchange biased permanent magnets is presented.

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Design of SPD - Made Hybrid Materials for Energy Applications

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ABSTRACT

In the last decade, there was extensive interest in hybrid materials made by Severe Plastic Deformation (SPD) techniques. It was demonstrated that the properties obtained by severe plastic shear co-deformation of metal-metal composites results in superior properties of hybrid material compared to those obtained by conventional processing routes.

Significant enhancement of the properties of these hybrid materials is attributed to the formation of measurable interfaces. The intensive shear deformation with imposed hydrostatic pressure causes deformation-induced atom mixing and intensified diffusion, which results in the formation of measurable interface zone with properties different from the individual constituents' properties. The simultaneous grain refinement during co-deformation and localised shear strain adds to design tools of hybrid material for specific applications.

In this presentation, our work over the last eight years on design and optimization of new types of hybrid materials, made be SPD, with improved strength and electrical conductivity is reviewed [1-8]. A thrust of our research was primarily directed at the practical applications, to invent the nanostructured high-strength electrical conductor cables for efficient energy transmission.

Especial attention was given to the (i) choice of constituent materials for hybrid conductor, (ii) effect of SPD method on the optimal balance of properties, (iii) role of measurable interface zone formation, and (iv) volume fraction, geometry and shape of constituents defining the inner architecture of a hybrid material. Material constituents used in fabricating of hybrid conductors included: Al-Fe alloys, Al-RE alloys, Al-Steel, and Al-Cu couples. Different SPD techniques such as equal channel angular pressing (ECAP), symmetric and asymmetric accumulative roll bonding (ARB, AARB), twist extrusion (TE), high pressure torsion extrusion (HPTE) and reciprocal extrusion (RE). The examples of inner architecture, illustrated in Figure 1 and many others will be presented in the talk.



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Plastic Deformation Processing of Heterostructured Materials: An Overview Yuntian Zhu^{*}

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ABSTRACT

Heterostructured materials consist of heterogeneous zones with dramatic (>100%) variations in mechanical and/or physical properties. The interaction in these hetero-zones produces a synergistic effect where the integrated property exceeds the prediction by the rule-of-mixtures. Heterostructures are found to have the capability to produce unprecedented strength and ductility that are considered impossible from our textbook knowledge and materials history. Importantly, HS materials can be produced by plastic deformation techniques plus thermal treatments using current industrial facilities at large scale and low cost. Heterostructured materials can be regarded as a novel direction of the severe plastic deformation with both new science and potential applications. In this talk I'll present an overview on the processing of various heterostructures and their effectiveness in improving mechanical properties.

Single-Step Production of Large-Scale Foil, Sheet, and Wire by Shear-Based Deformation Processing

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ABSTRACT

Virtually all commercial metal foil, sheet, and flat-wire (strip) used in the transportation, aerospace, electronics, and power generation and distribution sectors, are produced by multi-stage deformation processing (rolling or drawing), wherein large thickness reductions (plastic strains) require multiple stages of incremental deformation. While these traditional, entrenched manufacturing processes are capable of large-volume production, they are multi-step, energy intensive, and require large-scale, inflexible infrastructure. This poses a variety of constraints to product design, performance, and efficiency – especially in materials for structural and electrical power applications. Most of the reduction (strain) in commercial rolling is accomplished in hot-working steps, where large cast ingots are preheated to high temperatures – an energy-intensive step – to enhance workability. Subsequent cold-rolling steps are required to precisely control size and surface quality. Intermediate annealing treatments are also often necessary to prevent cracking and enhance workability. Indeed, in many cases, the gauge produced is limited to specific thickness ranges to avoid cracking or edge damage. The currently available thin-gauge formats, which can also be extremely expensive to produce (cost typically increasing exponentially with decreasing gauge thickness), and the limited thickness range available, both pose challenges for effecting weight and cost reductions in applications.

We describe large-strain (shear-based) deformation processing, structured around chip-formation by machining, that can produce metal sheet, foil and wire in a single deformation step from ingot. Processing to 100-mm width, and down to $50-\mu$ m thickness, is demonstrated from a variety of metals and alloys, including copper, aluminum, Fe-4Si electrical steels, magnesium AZ31B, stainless steels and titanium. **Figure 1** shows examples of sheets and strips produced by the shear-based process. The controllable shear deformation enables crystallographic shear textures in the strip that are favorable for formability. We compare and contrast our single-step deformation processing with the conventional multistage deformation processing in terms of various strip product attributes – plastic strain range, controllability of the (large-strain) deformation independent of strip thickness, surface finish and edge quality, formability, microstructure/texture, process specific energy – to illustrate the process capability and limitations. Implications for commercial production of sheet, foil and flat wire, including infrastructure requirements and point-of-use production capabilities, are discussed.



Figure 1 Copper sheet (100 mm x 0.2 mm x 50 mm) and aluminum alloy strip (50 mm x 0.150 mm x 1000 mm) produced by single-step shear-based deformation processing.

From Functional Nanomaterials to the Origin of Life by High-Pressure Torsion

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ABSTRACT

Nanomaterials, including high-entropy alloys and high-entropy ceramics, are of current interest for various functional applications. In recent years, severe plastic deformation (SPD) methods such as high-pressure torsion (HPT) have been used not only for the nanostructuring of materials but also for designing and developing new nanomaterials with enhanced functional properties. In this talk, some of the recent studies on developing functional nanomaterials including high-entropy alloys and ceramics by HPT are reviewed. The talk also explains how the capability of HPT in synthesizing new materials can shed new light on the origin of life in early Earth conditions.

Designing nanoSPD materials for their multifunctional properties

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ABSTRACT

Multiple recent studies have proved severe plastic deformation (SPD) techniques are a very reliable approach to produce nanostructured metals with significantly improved mechanical and functional properties [1,2].

SPD processing, in contrast to conventional large plastic deformation by extrusion or rolling, is a non-monotonic shear deformation process, where heavy straining is implemented under high applied pressures [3]. To date, quite a few SPD techniques have been developed, but the most popular include high pressure torsion (HPT) and equal channel angular pressing (ECAP). By applying these or other SPD techniques, various ultrafine grained (UFG) metallic materials with grain sizes in the nanometer range can be efficiently produced. Moreover, since SPD processing usually leads to unusual phase transformations related to dissolution and/or precipitation of secondary phases, formation of amorphous states, and nanocrystallization, the nanostructured elements such as nanoclusters, segregations of alloying elements at grain boundaries, nanotwins, etc., are also formed in the structure of materials [4]. In the present report we consider the scientific aspects of this problem and demonstrate the results of critical experimental studies.

At the same time the report also focuses on the unusual properties of SPD-produced nanomaterials that demonstrate the unique combination of mechanical and physical and chemical properties, such as superstrength and ductility, combination with high electrical conductivity, magnetic properties, etc. The nature of such multifunctional properties based on microstructural features of nanoSPD materials and their grain boundaries are considered as well.

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The Cutting of "Gummy" Metals

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ABSTRACT

It is well known that highly strain-hardening metals such as commercially pure aluminum, and copper; stainless steels, some nickel-based alloys, and refractory metals such as tantalum and niobium, are difficult to cut - characterized by large forces, thick chips and poor machined surface quality - despite being relatively soft. This difficulty in cutting - a large-strain deformation process – has earned them the moniker "gummy." Using high-speed, in situ imaging, we show that the fundamental cause of the problem is the tendency of these metals to deform via an unsteady, and highly redundant, mode of plastic deformation termed sinuous flow. This flow mode is characterized by large-amplitude folding and significant thickening (and non-homogeneous straining) of material during chip formation, Fig. 1(a). We demonstrate that the "gummy-metals" cutting challenge can be overcome by suppressing the sinuous flow mode using a unique chemical effect in large-strain (surface) plastic deformation of metals. This chemical effect involves use of benign organic media (e.g., inks) as 100 to 200 nm thick films, that are adsorbed onto the initial workpiece surface remote from the tool-chip contact. By locally embrittling the material in the deformation zone, the media effect a ductile-to-brittle transition (DBT) in the chip formation process: the energy-intensive sinuous flow is now replaced by a fracture-controlled segmentation (flow) mode with 50 to 80% reduction in cutting forces (Fig. 1), and nearly an order of magnitude improvement in surface finish. Lastly, we show, using a molecular self-assembly technique, that even chemisorption of an organic monolayer (one-molecule thick) on the workpiece surface is sufficient to cause the local DBT. Implications of the observations for cutting and comminution of gummy metals are discussed.



Figure 1 Transition from sinuous flow (a) to segmented flow (b) in cutting of aluminum due to chemical effects on surface plasticity enabled by nanoscale organic films. The graph shows the remarkable reduction in specific force/energy with different formats of surface media; ink, alkoxide, and self-assembled monolayers (SAMs).

Surface Severe Plastic Deformation to Enhance the Biomedical Performance of Orthopedic Biomaterials

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ABSTRACT

Metals and alloys constitute a popular class of materials used to prepare biomedical implants for both short-term and long-term use, primarily for orthopedic and dental applications. In addition to their bulk composition and properties, the performance metrics of these implanted devices are determined by their surface characteristics. Among the many surface engineering techniques that have been developed, surface severe plastic deformation (S²PD) techniques constitute a popular category of techniques to enhance the performance of metallic biomaterials. Among the different S²PD processes, our group has extensively explored the use of surface mechanical attrition treatment (SMAT) to modify the surfaces of a wide variety of alloys, including 316 L stainless steel, commercially pure Titanium, and the new generation of low modulus beta-Ti alloys. We have observed that SMAT tends to generate a graded microstructure with a nanocrystalline surface. A consequence of SMAT is the change in performance of the biomaterial, such as fatigue life, wear behavior, corrosion resistance, and biocompatibility through various mechanisms, such as surface hardening and alterations to the surface oxide layer. In this talk, I will describe our findings describing the microstructural changes induced by SMAT and its effect on performance for a variety of biomedical alloys.

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Microstructure and mechanical properties of ultrafine-grained materials

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ABSTRACT

Severe plastic deformation can be utilized to produce submicron sized grains or ultrafine-grained metals and alloys. Equal channel angular pressing (ECAP) is utilized to process aluminum alloys and steels. The degree of grain refinement in a material increases with increasing melting point, amount of alloying elements, imposed strain and decreasing stacking fault energy. The degree of grain size reduction is highest in the initial few passes, thereafter it decreases with strain. The refinement process at low to high strain level involves elongation of the existing grains by shear deformation, their subdivision into bands and subgrain formation within bands, intersection of the bands during subsequent passes and finally progressive conversion of the subgrains to grains. ECAP can accelerate precipitation of supersaturated alloy or dissolution of precipitates in aged/annealed alloy, respectively. The presence of second phase enhances refinement processes.

The yield and tensile strengths increase rapidly at first few passes and then reach a saturation level after a critical amount of equivalent strain. Improvement in mechanical strength at initial passes of ECAP is due to work hardening, formation of subgrain or dislocation cell formation and refinement in grain size. However, strengthening after critical amount of equivalent strain is mainly due to grain refinement. Yield strength of aluminum, single-phase aluminum alloys containing Mg, Zn or Ag, interstitial-free steels, low carbon steel and high alloy stainless steels, can be enhanced to 3.0 to 4.5 times by ECAP. Strengthening affect is highest for Mg and lowest for Ag due to their differential solid solution strengthening and refinement. There is some difference in yield strength and tensile strength at initial few passes. However, such a difference ceases to exist with increasing number of passes when grain size reaches ultra fine level. Consequently no work hardening takes place.

Ductility is partially regained after sufficient number of ECAP passes when microstructure becomes nearly equiaxed and ultrafine in size with low dislocation density. Ductility is regained significantly in low carbon steel (LCS) by modifying the microstructure to bimodal grain size distribution in which appreciable amount of grains is of micron sized. Bimodal structure maintains strength by resistance to deformation of UFG grains but micron sized grains adds ductility for the material by changing the nature of failure from brittle to ductile one. UFG materials can be further deformed by cold rolling or cryorolling. Flash annealing of cold rolled LCS above secondary recrystallization temperature produces bimodal grain size distribution of micron sized grains in UFG matrix. Bimodal grain size distribution is also obtained by electopulsing of UFG material and ductility is regained apreciably.

Achieving high strength ultra-fine grained austenitic stainless steels by the process of reversion of strain induced martensite

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ABSTRACT

Metastable austenitic stainless steels, when deformed, result in the formation of strain induced martensite, with the fraction of martensite increasing with strain to a saturation value. Heating beyond a temperature reverts the martensite back to austenite, and depending on the temperature and time of thermal treatment, an ultra-fine grained microstructure can be obtained. Presented here is one such study on AISI 304L where a plate was rolled at room temperature to a reduction of 93%. This produced about 70 % strain induced martensite with the remaining being untransformed austenite in the deformed state. Microstructure of the rolled of SS304L showed subgrain formation with high dislocation density, a median boundary spacing was 700 nm, hardness of 5.5 GPa and strength of 1.7 GPa. Different thermal treatments ranging from 650 to 800 °C were carried out at different heating times from minutes to hours, so as to obtain an optimum condition for reversion with a resultant ultrafine grained microstructure and high strength. An optimum condition of 700 °C for 30 min gave a median austenite grain size of 0.8 µm with strength of above 1 GPa and about 40% ductility. Similar studies of reversion of strain induced martensite to ultra-fine grained austenite were also carried out for AISI 321 and a 14wt.%Cr-8wt.%Mn-1wt.%Ni stainless steel. The microstructure and properties after reversion of these steels are compared. Apart from reversion studies, a warm thermal treatment at 400 °C for 3 to 5 days was also carried out that resulted in nano-sized precipitates in the strain induced martensite. This resulted in the increase of the strength of this steel to beyond 2 GPa.

Nanostructured Metallic Implants with Enhanced Biofunctionalization

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ABSTRACT

Titanium and its alloys are widely tried for load bearing medical implants due to its good biocompatibility, high corrosion resistance and high strength to weight ratio. However, the bioactivity of titanium, i.e., ability to form a hydroxyapatite (HA) layer, similar to the mineral phase of the bone on its surface in biological environment is still poor. On the other hand, magnesium and its alloys have the favorable properties for degradable materials targeted for temporary implants in cardiac and orthopedic applications. But, controlling the degradation rate is still the crucial issue in developing magnesium based implants. As cells live in a nano-featured environment of a complex mixture of pores and fibers of extracellular matrix, there is a great interest in the formation of submicron to nanosized grain materials to enhance its biofunctionalization. Severe plastic deformation (SPD) processes have the potential for achieving considerable grain refinement, typically to the submicrometer or nanometer level. The application SPD processes to develop fine grain titanium and magnesium based materials for implant applications seems to be a promising strategy in developing new generation metallic biomaterials. For titanium, increased mechanical properties and bioactivity are the benefits achieved with nanostructuring. However for magnesium, controlled degradation due to higher biomineralization with enhanced tissue response are the advantageous of grain refinement. A comprehensive summary of the progress achieved using SPD processes in developing ultra fine grain structured titanium and magnesium for implant applications will be presented [1]. Role of submicron to nano grain size on enhancing biomaterial properties will also discussed [2,3] Overall, the feasibility of commercial production of clinically applicable biofunctional metallic implants will be highlighted.

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Deformation and fracture micro-mechanisms in HPT processed maraging steels

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Maraging steels are a class of Fe-Ni steels with a bcc martensitic matrix which are precipitation strengthened by aging. Planar slip is known to operate in conventionally hot-rolled maraging steels, and they derive their peak strength through a two different types of coherent and incoherent precipitates. Upon overaging, reverted austenite nucleates due to dissolution of Ni rich precipitates locally, which leads to loss in strength. In the present study, maraging steels subjected to high-pressure-torsion (HPT) processing in the solutionised state are further aged to different conditions. The severe plastic deformation processing leads to a high density of dislocations and nanostructuring of the original lath microstructure of the martensite matrix, resulting in acceleration of aging kinetics. Aging of such steels leads to changes in Fe-Mo precipitate morphology from spherical to plate like. The impact of such nanograins and nanoscale precipitates on the deformation and fracture micro-mechanisms, determined through in-situ characterization techniques along with transmission electron microscopy and atom probe tomography analysis, is presented. Digital image correlation has been used to quantify the full field strain maps in regions of severe strain localization as well as to determine the fracture toughness through critical crack tip opening displacements. It is seen that the phenomenon of planar slip leads to strain softening under uniaxial deformation and to crack branching under a triaxial stress state in hot rolled maraging steels. On the other hand, nanostructuring after HPT processing creates a large number of high angle grain boundaries as dislocation barriers, leading to strain hardening under uniaxial tension and nearly straight crack path with catastrophic fracture under triaxial stress state. Upon overaging, both conditions show strain induced transformation of reverted austenite to martensite at the crack tip. This leads to a higher fracture toughness even while achieving high strengths in the overaged conditions of the nanocrystalline HPT overaged samples.

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Phase transition overview in the nanocomposite high entropy alloy produced by high pressure torsion

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ABSTRACT

Severe plastic deformation (SPD) in the form of high-pressure torsion (HPT) can expose the material to non-equilibrium conditions which make the emergence of nonconventional phases possible. The nanocomposite high entropy alloy (HEA) was produced by the forced mechanical mixing of two separate HEA disks using the HPT technique. Single phase fcc CoCrFeMnNi in the equiatomic composition and single phase equiatomic HfNbTaTiZr with a bcc structure were cut in the shape of disks with 8 mm diameter. The disks were polished down to a final thickness of 0.5 mm and had a mirror like surface prior to deformation. The intermixing of two initial HEAs starts in a lamellar fashion as the stacked disks are exposed to a constant pressure of 9 GPa while the top anvil rotated with a speed of 1 rpm. According to the preliminary microstructure analysis with the scanning electron microscope and energy dispersive spectroscopy (EDS) increasing the number of revolutions to 5, 10 and 15 results in the development of a rather complex microstructure with numerous vortices which is most prominent at the edge of the disk with 15 revolutions, but the chemical interfaces appear to have retained their sharpness. In-depth analyses of the chemical and structural aspects using the scanning transmission electron microscopy (S/TEM) and nanobeam diffraction mapping (NBD) reveals that in addition to the initial fcc and bcc phases, a new amorphous phase has emerged which corresponds to a region with 10 elements as verified by the STEM EDS and the atom probe tomography compositional line scans (1). STEM EDS and NBD have been applied for a more in-depth study of the thermally induced phase transformation in the nanocomposite HEA. The results show a combination of phase separation and nanoprecipitation to have taken place after annealing the sample in bulk for 20 minutes at 680°C, but the phase with 10 elements has retained its amorphous structure. Vibrating sample magnetometer shows that an SPD induced magnetic phase transformation has occurred in the as-deformed nanocomposite HEA, which according to the STEM differential phase contrast corresponds to the nanocrystalline CoCrFeMnNi phase. Our extensive study, focused on different types of phase transformation in the nanocomposite HEA, shows that exposure to SPD has resulted in the development of several new phases which highlights the diversity of possibilities associated with this technique.

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Biomimetic PEO coatings for implants made of nanostructured titanium alloys

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ABSTRACT

Current developments of permanent implants for orthopedic surgeries require transition to 4th generation of devices that feature smart surfaces interacting with the bone cells. The biomimetic concept suggests application of the devices that imitate living systems at all levels: mechanical, physical, chemical and biological. In terms of permanent implants that are produced from titanium alloys, this requires lowering the elastic modulus to the level of a bone, development of a surface layer having physical morphology, chemical and phase composition similar to the bone, and providing cell signaling via bio-chemical mechanisms. In this research, the first step was achieved by development of nanostructured titanium alloys by equal channel angular pressing (ECAP) and high pressure torsion (HPT). Titanium Grade 4 was used as a reference, and its nanostructured version nano-Ti was obtained by ECAP [1] for development of sample dental implants. Also, a novel shape memory effect alloy Ti-18Zr-15Nb (at. %) which elastic modulus and superelastic mechanical behavior are closer to that of a bone compared to medical Grade 5 Ti was used for the sample spinal implants. Its ultra-fine grained version was obtained also by ECAP and HPT [2].

The second and third steps towards the biomimetic surface were achieved in this research by plasma elelctrolytic oxidation (PEO) which proved itself as a reliable tool for the Ti implant surface functionalization [3]. The PEO technology helps to produce porous layers that physically imitate the porous bone structure due to the action of microdischarges as the key feature of the process mechanism. Moreover, by variation of the PEO electrolyte, the chemical composition of the coating can be developed close to that of a human bone that includes calcium phosphates, and, especially, hydroxyapatite. In this study, the PEO process was optimized to produce coatings on the novel Ti-18Zr-15Nb alloy, with coarse grain and ultrafine-grain structures [4]. As a result, the sample dental implants were produced and coated with the PEO technique. The fourth step was achieved by filling the PEO coating pores with functional molecules. Oligopeptides were used to modify the PEO coating on titanium; the RGD tripeptide sequence (arginine-glycine-aspartic acid) was anchored to the surface with a bisphosphonate linker to improve the implant biocompatibility [3]. Hyaluronic acid bisphosphonates as antifouling antimicrobial coatings for PEO-modified titanium implants were also developed [5]. The influence of the structure of the Ti alloy substrate was revealed, so that the best performance in terms of the osseointegration and antimicrobial action belongs to the PEO-organic biomimetic coatings on nanostructured and ultrafine-grained titanium alloys.

As a result, this approach forms the biomimetic surface that increases all the aspects of the implant biocompatibility, and opens the possibility to produce 4th generation of implants. Furthermore, dental implants Synthes Pro made of nanostructured titanium and having the PEO coating are available commercially in Russia from Dental Synthesis company with prospectives for export to India.

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Confinement of Low Dimensionality Unlocks Severe Plastic Deformability in Mg Alloys

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ABSTRACT

Mg with its hexagonal close-packed structure is characterized by uniaxial symmetry and a highly anisotropic critical resolved shear stress (CRSS) among the various available dislocation systems. Here, slip on the basal plane with its low CRSS dominates over non-basal modes (with large CRSS). Typically, accommodation of multiaxial plastic strains requires the activation of multiple dislocation slip systems. Inducing rampant non-basal slip (prismatic, pyramidal systems) is critical for large strain plasticity. The high CRSS for these additional modes however, present a challenge in Mg alloys under ambient conditions. This contributes to the poor formability of Mg alloys and their inability to sustain large strain deformation.

We show that Mg alloys are capable of accommodating severe plastic strains larger than 2 in a simple shear comfiguration without undergoing shear localization or concomitant fracture even at room temperatures. This emerges when a single characteristic dimension of the deformation is refined to the ~100nm scale. This can enable large strain deformability even when the other two dimensions are macroscopic (at least orders-of-magnitude larger). This is demonstrated in a plane strain machining configuration that mimics essential features of asymmetric wedge indentation. Using this configuration as a test of material response, the effect of refinement of a single characteristic length (i.e., depth of cut) is systematically varied from the nano to the micrometer-scales. At the finer nanometer scales, the deformation is found to be homogeneous, where the large strain deformation leads to rampant multiplication and concomitant refinement of the microstructure. Mg alloys undergo homogeneous severe plastic deformation when one length-scale of the deformation zone is refined. Nanocrystalline chips emerge with nanometer-scale depths of cut. Also, this behavior is agnostic to the crystal orientation, where polycrystalline systems sustain compatible deformation even across grain boundaries.

Our scoping calculations suggest that suppression of twinning during deformation in these configurations removes an important precursor to the onset of strain localization and shear banding. This is traced to the large strain gradients that emerge ahead of the deformation zone during plane-strain deformation with refined depths of cut. This can simultaneously reduce the mismatch in the CRSS that is necessary for triggering activity on basal and non-basal slip systems. Hence, offering a pathway for multi-axial deformation, which results in the accommodation of severe shear deformation when the depth of cut set to ~100nm in plane-strain machining. Larger depths of cut however, lead to the expected shear localization and fracture.

A broader motif for magnifying large strain deformability in Mg alloys comes into focus. Modulating strain gradients along the principal shear direction using microstructure that are refined along a single dimension may be sufficient for ductilizing Mg. Opportunities emerge for designing unusual microstructural topologies for improving the formability of Mg alloys. The insensitivity of this mechanism to the underlying microstructure points to opportunities with (nano) laminated structure involving heterogeneous and multimaterial interfaces, where deformability to large strains becomes feasible.

Acknowledgments

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Direct Observation of Large-Strain Deformation at Contact Interface in Wedge Indentation of Aluminum

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Die-material contact interfaces in manufacturing processes are quite different from conventional engineering sliding contacts. For example, the work material adjoining the interface undergoes severe plastic deformation; the real area of contact is equal to the apparent area; and the contact region is freshly generated with intimate contact and large adhesion between the die and work material. The severe plastic straining in this interface region is the primary origin of frictional dissipation in metalworking processes. Understanding the contact deformation phenomena is critically important for workpiece surface quality and for modeling of friction in metalworking. In this study, we examine large-strain plastic flow in the vicinity of an indenter-material interface in wedge indentation of aluminum (model system) using direct *in situ* high-speed imaging coupled with particle image velocimetry (PIV) analysis. The displacements and large-strain plastic deformation fields in the indentation zone are obtained at high-resolution for different indenter angles, and used to demarcate essential features of the material flow phenomena (**Fig. 1 (a)**) in the "boundary layer" adjoining the contact, and in the far-field. Furthermore, it is shown that the surface layer adjoining the indentation expands considerably due to the intense straining, by as much 20 to 40 times its initial length. This expansion is characterized at the micrometer scale using markers (**Fig. 1 (b**)). Based on the observations, we analyze the quantitative relationship between the surface expansion, "boundary layer" deformation, and interface friction and adhesion. Implications for friction in metal forming and cutting processes are briefly discussed.

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Figure 1: large-strain deformation field characteristics in wedge-indentation of aluminum (a) Distribution of vertical velocity component, showing the fundamental characteristics of cutting mode (30 deg: left) and radial compression mode (120 deg: right) of deformation. (b) Distribution of surface expansion ratio is obtained by tracking virtual makers using PIV analysis.

An in-situ TEM study on restoration mechanisms during low-temperature annealing of 80% cold rolled ferrite-pearlite AHSS steels

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ABSTRACT

To design and develop advanced high-strength steels (AHSS) with desired properties, it is essential to understand the relationship between changes in steel microstructure caused by thermomechanical processing and the corresponding changes in their properties. AHSS consists of ferrite-pearlite microstructure after cold rolling, recovery, and and recrystallization later transformed multiphase microstructures stages to two or such as ferrite/bainite/martensite/retained austenite after intercritical annealing. This study investigates restoration micromechanisms in individual phases/features during low-temperature (up to 500 °C) annealing of 80% cold rolled steel sheets. Internal structural changes in individual phase features have a critical impact on mechanical performance for various heavily cold-rolled materials. By a combination of post-annealing and *in-situ* annealing transmission electron microscopy observations, nanoindentation and EBSD we investigated the structural evolution. During annealing, the cementite lamellae morphology in pearlite remains stable up to temperature lower than 450 °C. Meanwhile, in the pearlitic ferrite and bulk ferrite dislocation rearrangements take place at (150 °C to 225 °C) followed by stabilization of dislocation structure as confirmed by TEM. Interestingly, hardness increases during the above microstructural changes. Nanoindentation mapping confirms hardness initially increases in the pearlite region followed by ferrite. The underlying restoration rate-controlling mechanisms are modeled and characterized.

Nanoindentation and Novel Structural Materials Development

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ABSTRACT

Over the past 3 decades since it was first introduced in 1980s, nanoindentation technique has been widely used to estimate the various mechanical properties of the small volume in a material at much smaller loads and size scales than conventional (micro-)hardness tests. Now, the technique is being considered not only as a characterizing tool but as a promising technology for better understanding of the mechanisms of small-scale mechanical/physical behavior from materials science viewpoints. In this talk, based on the research of my group and colleagues, I would like to introduce which properties can be "additionally" estimated by nanoindentation and then to explain how this becoming-somewhat-old technique (whose procedure and equipment were already standardized and commercialized) can be "still" very valuable tool for developing novel structural materials.

Mechanisms of Nanoplasticity

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ABSTRACT

The evolution of microstructure and texture of a face-centered cubic (fcc) nanocrystalline (nc) Pd-10at.%Au conventional alloy (CA) and coarse-grained (cg) CrMnFeCoNi high-entropy alloy (HEA) subjected to severe plastic deformation by high pressure torsion (HPT) at room temperature is investigated by X-ray line profile analysis and by X-ray microdiffraction. In addition, the microhardness is measured. During HPT the microstructure changes towards a steady state with regard to crystallite size, dislocation density, density of stacking faults and twins, and strength. In the case of the nc CA, grain coarsening takes place, while for the cg HEA it is grain refinement. For the nc CA the steady state is reached at a shear strain of about one, for the HEA it is about 25. Starting with a random texture in both alloys shear components typical for fcc metals develop. In the CA the resolved strength with decreasing crystallite size goes over a maximum (1/15 theoretical shear strength) at about 20 nm. The correlated changes in microstructure, texture and strength highly suggest the transition from dislocation slip to grain boundary sliding dominated deformation mechanism. In the HEA the maximum resolved strength for a crystallite size of 24 nm is 1/20 of the theoretical shear strength. The results are discussed in terms of different deformation mechanisms, including dislocation generation and propagation, twinning and grain boundary sliding.

Cold Sintering of High Entropy Alloy Powders Using High-Pressure Torsion

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ABSTRACT

Alloys operating at extremely low temperatures, such as those used for outer-space applications or cryogenic cargo valves and pumps, must possess special physical and mechanical properties, such as high elasticity, high strength, and high durability to ensure safety during the service. As an innovative approach to alloy design, high entropy alloys offer a wide range of opportunities to meet industrial demands of superior properties desirable for application in extreme environments. The MPEAs have been of interest to scientists and engineers due to their remarkable mechanical responses at cryogenic temperatures, nominating them as potential candidates for cryogenic applications.

Besides the inherent characteristics of the alloys, the manufacturing process plays a vital role in achieving flawless structures leading to the desired mechanical performance. Powder metallurgy (PM), a versatile and profitable manufacturing technique, has widely been used to synthesize novel high entropy alloys. However, PM-processed parts usually lack ductility under tensile stress than their counterparts fabricated with casting-based techniques. The pores, contamination of powders during synthesis, oxidation, and undesired phase formations during high-temperature sintering deteriorate the tensile ductility of PM-processed parts. It is essential to control these factors to achieve proper tensile properties during manufacturing. A new PM-based manufacturing approach, cold-sintering process using high-pressure torsion (HPT) followed by annealing, has been recently applied on high-entropy alloy. This method fabricated approximately fully dense structures with outstanding tensile properties than those reported for high entropy alloys through conventional PM methods. After that, this approach was employed to fabricate high entropy alloy-matrix composites, which led to heterogonous structures with excellent mechanical properties. In the present research, we fabricated CoCrFeNi medium-entropy alloy (MEA) samples by cold-consolidating gas-atomized powders using HPT followed by annealing. Microstructural evolutions and mechanical responses were systematically investigated to evaluate the feasibility of using the CoCrFeNi MEA fabricated by the proposed method in cryogenic applications.

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Atomistic simulations of idealized equal channel angular pressing process

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ABSTRACT

Atomistic simulations have now become the cornerstone of computational material science. Such simulation methods allow for tracking the trajectory of individual atoms. They are regularly used to study the mechanical behavior of materials, particularly to illuminate mechanisms of deformation at the nanoscale. Additionally, software codes for performing such simulations scale very well on modern high performance computing resources making ultra large scale simulations a simple case of using more computing nodes. However, they have rarely been used to study grain refinement during severe plastic deformation processes. Two reasons can be directly attributed to this end: a) the development of nanostructures necessitates the usage of large simulation boxes, which requires ultra large scale simulations and hence large computing resources, and b) the simulation times of 10 ns or greater required to impose strains of 200% or larger results in data storage of typically 50 TB per simulation. Analysis of such simulations would hence require increased automation, particularly to identify the refined grains and to understand the mechanisms of deformation.

In this work, we present our very first results on atomistic simulations of severe plastic deformation of single crystals. We model a single pass of the equal channel angular pressing (ECAP) process. The deformation is idealized as simple shear. A large simulation box containing a single crystal with approximately 100 Mio atoms is subjected to a shear of 200%. To assess the formation of orientations and grain refinement during the deformation, we use a newly developed algorithm called OrISODATA [1] that allows us to specify thresholds for both misorientation as well as the grain orientation spread. Additionally, the inhomogeneity of deformation is evaluated using unsupervised statistical machine learning techniques [2]. The results are discussed in terms of the mechanisms and their implications for higher scale modelling.

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Friction Stir Processing of Thermally Stable Nanostructured Immiscible Alloys and Metastable High Entropy Alloys

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ABSTRACT

Friction stir processing (FSP) is a high temperature severe plastic deformation (HTSPD) method that can be used to produce nanostructured immiscible alloys and metastable high entropy alloys (HEAs). In immiscible alloys, the forced mechanical mixing in the intense shear layer produces nanostructured features that are thermally stable. In this overview, the progress with Cu-based binary and ternary immiscible alloys have been reviewed. Remarkably, the Cu-Ag-Nb immiscible system displayed exceptional thermal stability up to 500°C. Initial results show that the approach is amenable to manipulation of matrix and the Cu-Al-Nb system shows low stacking fault energy matrix with thermally stable nanostructures. Implications of these on the high temperature performance will be discussed. Additionally, results on metastable HEAs show ability to create highly interfaced materials. Solid state processing of such nanostructured alloys using FSP as HTSPD method opens a new domain not easily accessible by other processing techniques.

Solid solution effects on structure evolution and mechanical properties of nanostructured binary (Cu-X) and high entropy alloys (Cantor-Ni) after high pressure torsion

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ABSTRACT

Two different alloy series (Cu-X, Ni-Cantor) have been selected to investigate the effects of solutes on the saturation grain size, the thermal stability and mechanical properties after high pressure torsion. The results of the Cu-X series indicate that the saturation grain size does not correlate with the stacking fault energy but show good agreement with solid solution hardening according to the Labusch model. Moreover, this correlation does not only hold for binaries, but also for chemically complex high entropy alloys (Ni-X) in the form of $(CrMnFeCo)_xNi_{1-x}$, where the Varvenne model is used to describe solid solution hardening. The alloy series exhibit a grain size in the range of 50 – 425 nm after high pressure torsion and the solutes increase the strength as well as the thermal stability of the alloys after annealing. The nanostructured alloys exhibit an enhanced strain rate sensitivity exponent, as determined from nanoindentation strain rate jump and constant contact pressure creep testing, whereas an enhanced rate sensitivity is found at low strain rates. The relatively lower rate sensitivity of the alloys as well as their higher thermal stability indicate, that defect storage and annihilation is strongly influenced by a complex interaction of solutes, dislocations and grain boundaries.

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Effect of process parameters during ECAP on microstructure and mechanical properties improvement in cast Al-Zn-Mg alloys

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7xxx series Al-Zn-Mg alloys are used in aerospace, traffic, rail and civil infrastructure [1,2]. These alloys have secondary phases, like Al₂Mg₃Zn₃ (T phase) and MgZn₂ (η phase) [1,2]. In cast materials, these phases are forming at the dendrite boundaries, hence limiting their second phase strengthening effect and ductility. Also, both Mg and Zn have limited strengthening ability [3]. Though these alloys have good dimensional stability and machinability, their constraint is poor strength and ductility, especially in the cast condition. These could be considerably increased by following SPD techniques. The properties of 7xxx series alloy could be considerably increased using SPD techniques, mainly using ECAP. This paper presents effect of temperature during ECAP of Al-Zn-Mg alloys, with different Zn wt percentages. Zn percentage was varied as 5, 10 and 15 wt%. Magnesium content was fixed at 2 wt%. Route Bc was adopted for ECAP process. The internal angle between two channels (Φ) was 120°. All the alloys were attempted to process at minimum possible temperature. Microstructural characterization was carried out at each stage of processing. Mechanical properties and facture studies were carried out on the samples.

The test results indicate that the alloy with 5 wt% Zn could be ECAP processed at 150 °C for 4 passes whereas the alloys with 10 and 15 wt% Zn could be processed only at 200 °C for 4 passes. Significant grain refinement was observed in all alloys. Significant increase in the dislocation density was observed and the precipitate morphology was altered after ECAP processing. Precipitates were GP zones and η' initially and after multiple ECAP passes, the precipitates were η' and stable η . The grain refinement, increase in dislocation density and formation of precipitates contributed for increase in the strength of the processed alloys. The alloy with 5 wt%Zn, had it's strength increased to 280 MPa from initial 122 MPa. Corresponding values for the alloys with 10 wt%Zn and 15 wt% Zn were 355 MPa and 405 MPa from initial 140 MPa and 160 MPa, respectively, after 4 ECAP passes at lowest possible temperature. Fracture mode was also changed from initial brittle to ductile mode, after ECAP processing. Similarly, ductility also increased considerably due to ECAP.

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Friction stir processing: A tool to develop functional materials

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ABSTRACT

Friction stir processing (FSP) is a method to process the surface of materials. The surface undergoes intense plastic deformation during the process that leads to severe microstructure refinement. In the case of two-phase materials, the dispersed phase (otherwise second phase or precipitates) also undergoes refinement. The dispersed phase's shape, size and distribution can significantly be altered using FSP. More than a decade of research established this process as a reliable route to refine the microstructure that leads to improvement in mechanical properties and wear resistance of non-ferrous alloys based on aluminium, magnesium and copper. However, the usage of FSP to produce functional materials paid less attention. Therefore, in our research work, we explored the ability of FSP to develop functional materials i.e. high-strength high-electrical conductivity copper alloys and high-damping aluminium alloys/ composites. Selected copper alloys (Cu-Cr, Cu-Zr & Cu-Cr-Zr) were friction stir processed and their mechanical, wear and electrical conductivity properties were studied. It was observed that the hardness of friction-stir processed copper alloys was 20-56% [1] higher than their initial state. At the same time, the decrease in electrical conductivity was only about 3 %IACS after processing. The room temperature damping of commercial-pure aluminium and copper improved by 1.5-2.3 times after FSP [2-3]. Whereas the damping of AA6061 at 250 °C, as determined through a dynamic mechanical analyser, was observed to increase by 350% after FSP. The influence of FSP on the microstructure thus on damping capacity will be presented in detail.

Acknowledgements

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Superplastic behaviour of high-pressure torsion processed high entropy alloys

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ABSTRACT

High entropy alloys (HEAs) are emerging novel alloys with a unique alloy design concept based on many principal elements. This unique alloy design concept provides a huge compositional range to develop various new alloys for various structural applications. Besides, these alloys possess superior properties such as a high strength-ductility combination, excellent fracture toughness, and good wear resistance compared to conventional alloys. The superplastic behaviour of HEAs as forming ability is also essential for possible industrial applications of these emerging alloys. Materials with refined grains (< 1 μ m) are prerequisites to achieving superplastic elongation of greater than 400%. High-pressure torsion (HPT) is a well-known severe plastic deformation technique in achieving exceptional grain refinement, and HEAs with very fine grains are achieved by HPT and subjected to superplasticity experiments.

In this presentation, an overview of the microstructural effects on superplastic behaviour at different strain rates, superplasticity mechanism, and cavity formation will be discussed based on the results obtained from HPT processed Al₉(CoCrFeMnNi)₉₁ (at%), Al₉(CoCrNi)₉₁ (at%), Fe₅₅Co₁₈Cr_{12.5}Mo_{7.5}Ni₇ (at%), and Fe₃₅Ni₂₅Cr₁₅Co₁₀V₁₀Mn₅ (at%) HEAs.

Superposition of strengthening mechanisms: A statistical approach based on circle rolling

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ABSTRACT

High-performance engineering alloys are usually comprised of multiple obstacles such as solutes, precipitates, dispersoids, forest dislocations etc. to deliver optimum mechanical properties. The interaction of mobile dislocations with these obstacles on the glide plane controls the overall mechanical properties such as yield strength, work hardening etc. To ensure continuing progress in the field of developing high performance alloys, it is essential to understand the individual strengthening contribution from these obstacles as well as their superposition. In this regard, classical circle rolling method based on the idea of Foreman and Makin was used to simulate the motion of a mobile dislocation through an array of randomly and non-randomly distributed obstacles and determine the critical resolved shear stress. Systematic simulations were performed for unary, binary and ternary set of obstacles with varying breaking angles (0-180°) to determine the superposition exponent. Lastly, contour maps for the superposition exponent are developed for both random and non-random distribution to aid accurate prediction of strength for different obstacle populations.

Microstructural response of FSPed Al-7.3Zn-2.2Mg-2Cu (Al7068) alloy Nitish Raja^{1*}

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ABSTRACT

For microstructural uniformity, a large-scale atomic movement is essential, which can be achieved by the introduction of plastic strain through various severe plastic deformation techniques (friction stirring processing, torsion, equal channel angular pressing (ECAP), and equal channel angular rolling (ECAR). SPD is an essential and globally practised method to obtain an upgraded mechanical property. FSP is a majorily studied topic, but the plastic response of a very high strength Al7068 alloy is less explored. The ultrasonically mixed Al7068 alloy has been processed at an r.p.m of 1025 with a traverse speed of 10mm/min. Also, the processing condition did not show any defect. The introduction of a very large plastic strain has yielded in a server grain refinement from 190 μ m to 10 μ m and mechanical characteristics from 230 MPa to 695 MPa. The improvement in mechanical properties is mainly due to the distribution of nano-sized precipitates. The EBSD analysis confirmed the occurrence of continuous dynamic recrystallization (C-DRX) during plastic straining. Also, the grain orientation spread map indicated a fair extent of recrystallization (\geq 80%).

Keywords: severe plastic deformation, friction stirring processing, Al7068

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HPT studies on FCC medium entropy alloys and understanding the role of GSFE on their deformation behavior

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ABSTRACT

This study investigates equiatomic Ni-based FCC alloys with low to medium entropy (Ni-Co, Ni-Co-Fe, and Ni-Co-Cr-Fe systems). X-ray diffraction studies confirm that all alloys have a single-phase FCC structure at room temperature and a lattice expansion from binary to quaternary system. Alloy discs (8 mm in diameter) were plastically deformed by 180° rotation of high-pressure torsion (HPT) at 7 GPa in a quasi-constrained condition. The alloys were subsequently subjected to recrystallization annealing (700 °C). The deformation response of alloys and the correlation between their microstructure and micro-mechanical behavior were studied. All alloys showed extensive grain size refinement after HPT deformation. They also exhibited heterogeneity in the microstructure and the hardness values. The microstructure of a disc was refined more toward its periphery as the strain in the HPT deformation process increases radially. Also, the hardness increase was non-monotonic as a function of radial distance. However, the degree of heterogeneity varies from alloy to alloy. After 6 minutes of annealing, the alloys exhibited mostly recrystallized structures. A comparative observation revealed that the low-angle grain boundaries in the deformed state had transformed into high-angle grain boundaries in the recrystallized state. The formation of deformation bands was observed in NiCoFe, showing an abrupt transition in grain size from edge to center of the disc. Additionally, to understand the deformation behavior of these alloys, the atomistic modeling was done using the supercell approach to compute the generalized stacking fault energy (GSFE) of alloys using the Vienna ab initio simulation package. These computational studies show that equiatomic NiCoCrFe has the lowest SFE alloys and a higher work hardening rate. In contrast, equiatomic NiCoFe has the highest SFE and the lowest work hardening.

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Influence of equal channel angular rolling (ECAR) on the gradual microstructural evolution in Al-4.5Mg-0.14Si alloy

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Abstract: It is well established that severe plastic deformation (SPD) has been utilised for many engineering materials to reduce the grain size below 1 micron with a noticeable improvement in mechanical properties. Even though equal channel angular pressing (ECAP), high pressure torsion (HPT), RCS have been widely studied, the present investigation was aimed to use the equal channel angular rolling (ECAR) on Al-4.5Mg-0.14Si alloy, a less explored one. The Al-alloy has been processed for 5 passes and no major reduction was observed in the thickness and width of the specimen. As expected, ECAR has influenced grain size reduction from 12 micron to 7 micron considering a threshold grain boundary misorientation value of > 15°. Parallelly the strength was improved from 240 to 360 MPa. Moreover, the EBSD analysis has been carried out on all processed samples. The results clearly demonstrate the conventional dynamic recrystallization (Conv-DRX) of the sample after 4 passes by imposing the critical strain of. 0.6. A randomization of texture of the freshly nucleated DRX grains has been observed. The role of precipitates (dominantly Mg₂Si) has also been evaluated in the microstructural transition during the ECAR processing.

Keywords: Severe Plastic Deformation, Equal channel angular rolling, Grain refinement

Texture and Microstructure Developments during Multi pass Friction Stir Processing of Magnesium Alloy AZ31

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ABSTRACT

Commercial magnesium alloy AZ31 was processed through multi-pass (1 to 4 passes) friction stir processing (FSP). Electron backscattered diffraction was used to monitor microstructure developments, while X-ray pole figures were examined to index developments in asymmetry in crystallographic texture. Multiple passes had insignificant effects on grain refinement, in-grain misorientation developments and developments in crystallographic texture. These had relatively minor modifications. Though a strong asymmetry in X-ray pole figures was noted, such asymmetry also got carried through the FSP passes. A two step numerical simulations were performed to understand the microstructural and texture developments in the processed material. At macro scale, thermo-mechanical finite element simulations (FEM) were performed to obtain the local temperature and strain (both the magnitude and the modes) distributions. At mesoscale the strain modes from FEM were fed into viscoplastic self consistent model to simulate the texture and gain insights into the origin of experimentally observed texture asymmetry.

Texture prediction of severely deformed high-entropy alloy by crystal plasticity simulations

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ABSTRACT

An induction melted non-equiatomic Al₆(CoFeMnNi) (at. %) high entropy alloy was severely deformed by highpressure compressive shearing. Microstructure and texture evolution at three intervals of strain were studied by EBSD, TKD, TEM, and XRD. The high deformation led to a significant increase in the strength, up to ≈ 1.073 GPa. X-ray diffraction macro-texture analysis revealed a shear texture with the dominance of the B/\overline{B} {112}<110>-type component whose intensity varied with strain. A schematic procedure was determined to reproduce experimental texture using various Taylor-type polycrystal plasticity simulations including grain fragmentation, deformation twinning, and grain boundary shearing. The successful procedure was able to reproduce the texture with a correlation value of 91%.



Correlation: 91%

Deformation of Inconal 800 through Equal Channel Angular Pressing

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ABSTRACT

In this study, Inconal 800 rods of 15 mm diameter and 800 mm length were deformed through equal channel angular pressing (ECAP) at room temperature with lubrication. The ECAP die has an inner intersection angle 120° and outer arc angle 60°. Hydraulic press of 100 tonn capacity was used to press the sample during ECAP process. Each sample was deformed for 1 pass of ECAP which imposed an equivalent strain 0.6. As-received and ECAPed samples were characterized for microstructural analysis using optical microscopy and scanning electron microscopy. Mechanical properties were evaluated using tensile testing and hardness testing. Tensile tests were also performed at elevated (600°C) temperature. Results show that coarse grains are elongated in the extrusion direction. Submicron sized bands were formed within the coarse grains, which were the results of severe plastic deformation during ECAP process. Yield strength of the as received material was 217 MPa and 152 MPa at room temperature and 600°C, respectively. The yield strength of ECAPed material is 799 MPa and 502 MPa at room temperature and 600°C respectively. At room temperature, hardness was also increased from 145 VHN to 298 VHN. The increase in strength and hardness is attributed to the increase in grain boundary length and an increase in the dislocation density during ECAP process. High temperature strength of the Inconal 800 after ECAP process was more than 3-times of undeformed material. Fractographs show the ductile fracture in as-received materials and ductile-brittle fracture in ECAPed materials ir-respective of the testing temperature.

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Preparation of Ultra-thin walled Magnesium AZ31 Alloy Tubes Using Friction Stir Extrusion

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Abstract

Magnesium alloys are increasingly being considered for structural systems across different industrial sectors, including precision components of biomedical devices owing to their high specific strength and stiffness, biodegradability. For example, tubular devices such as coronary stents manufacture require defect-free, high-quality tubes with thin walls (100 μ m – 1 mm) as a precursor. Here, we have produced fully consolidated, structurally sound ultra-thin walled (~ 400 μ m) AZ31 Mg alloy tubes by friction stir back extrusion (FSE) — a relatively new severe plastic deformation process used typically to manufacture thick metallic tubes and rods. The tube cross-sectional microstructure was layered and consisted of a severely deformed stir zone with refined grains near the inner edge, a back-extruded zone with small grains near the outer edge, and a thermomechanically affected zone (TMAZ) with coarse grains develop within the central region. On the other hand, the inner tube surface microstructure had an average grain size of 4.1 \pm 1.9 μ m and a strong basal texture. In comparison, the outer tube surface microstructure was coarse, with an average grain size of $13.3 \pm 6.4 \,\mu\text{m}$ with no preferred orientation. The microhardness variation along the tube wall thickness indicated the operation of multiple deformation paths during FSE. Upon exposure to Hank's balanced salt solution at 37°C, microgalvanic coupling resulting from the gradient through-thethickness grain size and texture differences between the tube inner and outer surfaces, and residual strain arising from the FSE process, promoted a localized attack that preferentially initiated on the outer tube surface and progressed inwards. Static recovery induced by a short duration heat treatment at low temperature did not alter the microstructure but promoted a more general corrosion attack with a significantly reduced corrosion rate. Our results establish that FSE is a viable single-step process to manufacture ultra-thin Mg alloy tubes suitable for degradable precision tubular applications.

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Effects of aluminium purity and addition of carbon nanotubes on the hardness and thermal stability of CNT reinforced aluminum nanocomposites processed by the high-pressure torsion technique

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ABSTRACT

In this study, an Al-based composite containing 0.5wt.% of carbon nanotubes (CNTs) was fabricated using a process of severe plastic deformation through high pressure torsion (HPT). Two aluminium alloys with a wide range of purity levels such as 99.5% (A1050), 99.99% (4NAl) were used. The uniform dispersion of CNTs in the Al matrix has been identified as being critical to the pursuit of enhanced properties. That is why HPT synthesis was carried out on a stack of two aluminium disks between which carbon nanotubes were scattered and the total number of turns was 100. Transmission electron microscopy observations showed a strong microstructure grain refinement to 200-300 nm and this was much smaller than the grain size in samples without CNTs. High resolution electron microscopy revealed that CNTs were present at grain boundaries. A significant increase of hardness (120 Hv) when compared to initial state was observed. It was found that the addition of CNTs to aluminium increased its thermal stability from 150°C to 250-300°C in comparison to pure alloys after HPT processing.

Acknowledgments

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Effect of Swaging and Following Annealing on Gradient Structure Formation in Austenitic Stainless Steels with Various Stacking Fault Energy

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ABSTRACT

Gradient structure formation is considered as a promising pathway to overcome obvious strength-ductility trade-off of austenitic stainless steels. Therefore, simultaneous development of the yield strength and ductility can be achieved. To produce gradient structure, cold swaging is applied. Following austenite-based alloys are studied to receive the effect of stacking fault energy (SFE) on gradient structure formation: a medium entropy alloy Fe-30Mn-10Co-10Cr-0.5C (Alloy 1) with SFE = 13 mJ/m² where twinning and strain-induced $\gamma \rightarrow \epsilon$ martensitic transformation are the main deformation mechanisms; a metastable austenitic stainless 321-type steel (Alloy 2) with SFE = $18-20 \text{ mJ/m}^2$ where twinning and strain-induced $\gamma \rightarrow \alpha$ ' martensitic transformation are expected during plastic strain; a austenitic stainless 316-type steel (Alloy 3) with SFE = 39 mJ/m² where twinning is dominant deformation mechanism. Fine element simulation (FES) shows that moderate tensile stresses are produced during swaging, while considerable compression stresses are predicted in the surface layeres. Thereby, non-uniform stress condition is realized in the bar during processing. In Alloy 1, strong axial one component (111//bar axies (BA)) texture gradient and gradient of twin density are performed due to development mechanical twinning. However, cold rotary swaging of Alloy 2 produces pronounce axial two component (111//BA and 001//BA) texture gradient of austenite vie intensive twinning and dislocation slip, respectively, and one component (101//BA) texture gradient of martensite due to development of strain-induced $\gamma \rightarrow \alpha$ ' martensitic transformation. Furthemore, gradient of phase composition and twin density are also obtained. In case of Alloy 3, only axial two component (111//BA and 001//BA) texture gradient of austenite and gradient of twin density are found. Interestingly, the most pronounced gradient is attained in Alloy 2. Hence, to entensify the obtained structure gradient, Alloy 2 is subjected to subsequent low temperature annealing that results in presipitation of nanocarbides within α '-martensite grains and, thereby, additional gradual hardening of the bar according to gradient of phase composition. After cold swaging and following annealing, Alloy 2 possesses extraordinary combination of high strength (UTS>1500 MPa) and excelant toughness (>1.4 MJ/m^2). Thus, the applied approach is a perspective method for the enhancement of mechanical properties of metastable austenitic stainless steels.

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Nano-tubular Architecture through Severe Physical Deformation for High Performance Supercapacitor

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ABSTRACT

The current times are witnessing huge technological advancements such as digital transformation including Internet of things (IoT), e-mobility, and green hydrogen mission (GHM), to name a few. The devices/components in these systems such as sensors/electronic circuitry in IoT, a prime-mover in e-mobility and an electrolyzer in GHM needs a seamless, uninterrupted power for proper functioning. Supercapacitors along with batteries play a crucial role to power these components. Benefitted by the short-diffusion length, nano-materials have played a key role in this direction. However, the inadequate ion-accessible surface area of existing nano-porous/nano-textured electrodes and long synthesis time of the current technology impairs the further developments in the field. In addition, the tedious precursor selection, interface resistances by the binding layer, and compositional dependence on the evolution of nano-scale features are additional challenges being faced by existing supercapacitor technology. Breaking the barriers of sub-homologous nano-moulding in crystalline materials, herein, we introduce a disruptive technology to synthesize hierarchical nano-tubes in transition metal oxide with large ion-accessible surface area and outstanding electrochemical energy storage. The proposed technique utilizes short burst of localized physical deformation to facilitate the material flow into the nano-moulds at temperatures ~ 0.3 times the alloy melting point. The technique provides high flexibility in controlling the morphology of nano-structures, resulting in solid nano-pillars at shorter deformation time (5 minutes) and hollow nano-tube morphology at larger time duration (10 minutes). The hierarchical architecture of electrochemically dealloyed nano-tubes showcased excellent specific capacitance of 1000 F/cm3 at 5.5 A/cm3 current density. A symmetric supercapacitor device synthesized using 50 µm thick electrodes of electrochemically dealloyed nano-tubes delivered an outstanding energy density between ~ 90 Wh/L to ~ 40 Wh/L at power density of ~ 0.5 kW/L and 5 kW/L, respectively along with excellent cyclic stability of 94% after 10,000 cycles. The integration of multiple devices was successfully demonstrated to power high-rated LEDs, establishing its technological readiness. The facile and highly efficient top-down strategy for developing nano-tubular structures showcased here is the demonstration of a template and not unique to the particular composition presented in this work and application domain. The technique presented here opens new research directions and is poised to impact materials development for various applications including hydrogen generation, bio-sensing, electro-catalysis, besides traditional structural components

Texture control in SPD processed alloys by (dynamic) High-Pressure Torsion and Accumulative Roll Bonding

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ABSTRACT

Plastic deformation modifies the crystallographic texture of a polycrystalline metal alloy. Conventional processes are often fine-tuned in order to form a specific texture so as to control the anisotropy of certain mechanical properties, such as e.g. is the case for deep drawing steel sheet with improved Lankford R-value. Both the strain mode and the strain amplitude are of crucial importance for the formation of the deformation texture. According to conventional crystal plasticity theories plane strain compression during sheet rolling will eventually produce a stable single component texture, whereas torsion produces a shear texture that characteristically shifts between a number of stable fiber-components. In reality, however, it is observed that when true strain values exceed a critical value (that is dependent of strain mode and strain rate) the texture tends to randomize both during Accumulative Roll Bonding (ARB) and during (dynamic-) High Pressure Torsion (d-HPT). Texture randomization is associated with grain fragmentation and therefore of crucial importance for the formation of the ultra-fine microstructure, which is the primary goal of the SPD process. The crystallographic texture, similar to other microstructural state variables, evolves to a dynamically stable state beyond a threshold value of the plastic strain amplitude. The nature of this meta-stable will be critically assessed

Scaling-up of Severe Plastic Deformation – a Story of Success without a Happy-end

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ABSTRACT

Severe plastic deformation (SPD) was proposed as a tool to refine the microstructure in metals and alloys down to micrometer- and even nanometer scale almost forty years ago. High pressure torsion (HPT) is the most effective SPD method in respect of the microstructure refinement and processing costs. Furthermore, application of HPT frequently leads to unusual phase transformations, which opens further opportunities for materials design. Despite attractive mechanical and physical properties demonstrated by HPT-processed materials, this technique cannot be used in industry due to a limited size of processed samples. To overcome this problem, a plenty of SPD methods, allowing processing of large-scale billets, was developed. The most prominent examples are Equal Channel Angular Pressing combined with Conform[™] process, Accumulated Roll Bonding, Multidirectional Forging and some others. However, SPD has not found any application in industry, except a few cases, which are rather exceptions that confirm the rule. In this presentation we'll try to analyze the reasons why industrial players remain reluctant to use SPD in technological processes. For this purpose we'll discuss the advantages and drawbacks of the High Pressure Torsion Extrusion method of SPD [1]. This method allows to realize the stress-strain conditions, very similar to these at conventional HPT, in a billet sample and achieve large strains in a single processing pass.

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The Zen of Grain Boundaries

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ABSTRACT

Grain boundaries are ubiquitous and prominent microstructural features in ultrafine grained and nanocrystalline materials processed by severe plastic deformation and other techniques. Using a few specific examples, this presentation examines the role of grain boundaries in such materials in terms of their contradictory behaviors. Thus, grain boundaries lead to strengthening in conventional materials with grain sizes of $> 1 \mu m$ whereas they may lead to weakening in materials with grain sizes of < 10 nm. Similarly, grain boundaries contribute significantly to superplastic deformation by grain boundary sliding, but this also enables fracture by the nucleation, growth and interlinkage cavities.

'Non-equilibrium' grain boundaries in additively manufactured alloys: CoCrFeMnNi high-entropy alloy as a case study

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ABSTRACT

The existence of 'non-equilibrium' or 'deformation-modified' state of grain boundaries in metals and alloys after a severe plastic deformation is well known for decades now [1]. Additive manufacturing via laser powder bed fusion is an attractive route to produce functional materials with complex shapes. Due to the severe processing conditions which include rapid melting and solidification, various crystallographic defects are known to be introduced in a material. In this report, we will present unambiguous proofs of the existence of non-equilibrium grain boundaries in a 3D-printed multi-component alloy. In particular, grain boundary diffusion in an additively manufactured equiatomic CoCrFeMnNi high-entropy alloy is systematically measured. In the as-printed state, general (random) grain boundaries are found to be characterized by orders-of-magnitude enhanced diffusivities and by a nonequilibrium segregation of (dominantly) Mn atoms [2]. These features are explained in terms of the non-equilibrium state of grain boundaries after the rapid solidification. The grain boundary diffusion rates are found to be almost independent on the scanning/building strategy used for the specimen's manufacturing, despite pronounced microstructure differences. Grain boundary migration during diffusion annealing turned out to preserve the non-equilibrium state of the interfaces due to continuous consumption of the processing-induced defects by moving boundaries. Whereas the kinetically 'non-equilibrium' state of the interfaces relax after annealing at a moderate temperature of 773 K, the non-equilibrium segregation survives, being further accompanied by a nano-scale phase decomposition at the grain boundaries.

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Impact of structural transformations and defect interactions at grain boundaries in nanocrystalline metallic materials

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ABSTRACT

The development of high-strength metals has driven the endeavor of pushing the limit of grain size reduction according to the Hall-Petch law. However, a continued grain refinement is technologically particularly challenging and incorporates issues concerning microstructural stability and raises also the problem of a possible inverse Hall-Petch effect. However, utilizing structural transformations at – or of grain boundaries as well as more complex interactions of dislocations at / with grain boundaries can provide additional options and new pathways for optimizing the mechanical properties and / or the stability of ultrafine grained - or nanocrystalline materials. Importantly, a set of examples now exist that indicate the applicability of such strategies also for commercial, complex and / or multi-component alloys.

Advanced approaches might be based on different strategies; one example is given by strengthening nanocrystalline materials via dislocation exhaustion through a localized structural transformation at grain boundaries that decreases the density of residual lattice dislocations considerably. Thus, the plasticity in the dislocation-exhausted nanomaterial becomes dominated by the activation of grain boundary-dislocation sources, leading to an ultra-hardening effect [1].

Another approach is based on dislocation-twin boundary interactions. Twins are generally regarded as obstacles to dislocations in face-centered cubic metals and can modify individual dislocations by locking them in twin boundaries or obliging them to dissociate. Yet, as recently shown, dislocations can also be transported by twin lamella via periodic twinning and detwinning at the atomic scale. Our results reveal a novel evolution route of dislocations by a dislocation-twin interaction where the twins act as transport vehicles rather than as obstacles [2].

Further options are based on transformations of the grain boundary core structure itself, which has been shown to affect precipitation behavior and, most likely, the local thermo-mechanical coupling during severe straining. The coupled analyses by tracer diffusion and high-resolution transmission electron microscopy indicate that a metastable grain boundary transition occurred ina deformed Ni-base multicomponent alloy, which is explained by a concomitant relaxation of transformation-induced elastic strains occurring on a longer time scale in comparison to those for grain boundary diffusion. These analyses provide solid evidence towards the existence of grain boundary phase transitions, even in complex, multi-component alloys [3].

In this contribution, recent examples for advanced strategies that utilize grain boundary transformations or grain boundaries as a vehicle for transitions or defect interactions are highlighted.

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Origin of Anisotropic mechanical behaviour of High Pressure Torsion deformed metals and alloys

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ABSTRACT

High pressure torsion (HPT) is one of the severe plastic techniques which have been used successfully for achieving homogeneous ultrafine grained and nanocrystalline microstructure. The high strength of HPT deformed materials has been well investigated and generally attributed to high dislocation density, refined grain size and solid solution strengthening from partial to complete dissolution of precipitates [1-3]. Anisotropy in mechanical properties is another characteristic of SPD processed materials which is not that well investigated [3-5]. It has been observed that high strength is not always accompanied by uniform deformation.

In this presentation some of the important aspects of anisotropic mechanical properties in single phase and multiphase alloys will be discussed in relation to their microstructure, crystallographic texture and composition

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Microstructural characterisation of friction stir processed green Al powder compact

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ABSTRACT

The samples were prepared by friction stir processing of cold compacted green Al powder compact. Friction stir processing was carried out at different rotation speeds of 720 to 2260 rpm. The cold compacts were quite fragile since the powder particles did not have a metallurgical bond. Friction stir processing accomplished two objectives, first to create bonds between particles and second to refine the microstructure. Also, the density after friction stir processing increased to 2.7 g/cm³ from 2.3 g/cm³. Extensive grain boundary characterization was carried out after friction stir processing. Samples processed at lower rpm showed a typical bimodal misorientation distribution. It was observed that the length of the very low angle grain boundaries ($\theta \le 5^\circ$) was almost unaffected by the processing parameters. At higher rotational speed, the length of the high-angle grain boundaries ($\theta \ge 15^{\circ}$) increased at an expense of low-angle grain boundaries ($5^{\circ} \le \theta \le 15^{\circ}$). Thus, random misorientation distribution was achieved when the rotational speed was above 1525 rpm. In the cold compact, the particle boundaries had grain boundaries with misorientation above 40°. These grain boundaries existed even after friction stir processing. Interestingly some grain boundaries were not identified by the OIM software during analysis though they were clearly visible in the image quality (IQ) map. These grain boundaries were characterized via transmission Kikuchi diffraction (TKD). The step size of the scan varied from 50 nm to 200 nm. The scans were cleaned via the neighbour orientation correlation method. At 200 nm step size, a significant fraction of the grain boundaries remained unidentified. However, the presence of very fine grain/subgrains in the unidentified grain boundaries was revealed at 50 nm step size.

Low-Temperature Processing Of NiTi Shape Memory Alloy By E.C.A.P. In Shells R. Karelin

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ABSTRACT

The severe plastic deformation (SPD) allows obtaining an ultrafine-grained structure and considerable improving mechanical and functional properties of NiTi shape memory alloys (SMA). Previous studies showed that formation of completely nanocrystalline structure (NCS) in bulk samples using SPD by traditional ECAP was unsuccessful. For further grain refinement, the possibility of Ti-50.0 at.% Ni SMA processing by ECAP in shells at lower temperatures was studied in the present work.

ECAP was performed in steel and iron shells in a temperature range from room temperature to 350 °C. NiTi samples with a diameter of 6 and 12 mm were successfully pressed in iron shells with a diameter of 20 mm for one pass at 200 °C. Lowering of deformation temperature both to 100 °C and to room temperature leads to the distraction of TiNi core during the first ECAP pass. Steel was defined as a unsutubale material for the shell production due to the premature destruction during the first ECAP pass at temperatures below 300 °C.

The obtained structure, kinetics of martensitic transformations, mechanical and functional properties of NiTi samples after ECAP were studied using X-ray diffractometry, transmission electron microscopy, differential scanning calorimetry, tensile, hardness and bending tests.

ECAP in shell at 200 °C with a channel intersection angle of 120° for NiTi sample with diameters of 12 and 6 mm leads to the formation of the elongated band martensite structure, with the size of structural bands about 100 nm. ECAP at the temperature of 200 °C for 1 pass leads to the significant increase in the value of mechanical properties in comparison with reference treatment (annealing at 750°C for 30 min with subsequent cooling in water). The highest mechanical (yield stress $\sigma_y = 1,045$ MPa, ultimate tensile strength $\sigma_B = 1,200$ MPa) and functional (maximum value of completely recoverable strain of 7.4%) characteristics was obtained after ECAP with $\phi = 120^\circ$ of TiNi sample with the diameter of 12 mm and the addition of post-deformation annealing (PDA) at 400 °C for 1 h.

Based on the obtained results it can be concluded that processing of NiTi samples by ECAP in shells is a promising method allowing to considerably refine the obtained structure and improve mechanical and functional characteristics. For the deveolment of technologicall possibilities of ECAP in shells the number of passes should be increased from one to several in order to obtain a more equiaxed NCS and additionally improve the functional properties.

Acknowledgments (if any)

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Investigation on the Effect of High Pressure Torsion on the Precipitates in Sc modified Al – Li alloy

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ABSTRACT

AA2195 Al – Li alloy was modified with Sc addition and subjected to severe plastic deformation (SPD) by high pressure torsion (HPT) processing technique. The three compositions of the Al – Li alloy: 0 wt.% Sc (NoSc), 0.025 wt.% Sc (LoSc) and 0.25 wt.% Sc (HiSc), were subjected to HPT processing at room temperature under 6 GPa hydrostatic pressure upto 5 rotations. The microstructure, microhardness, and textures were studied using transmission electron microscopy, Vicker's microhardness indentation, and X-ray diffraction techniques, respectively. Atom probe tomography (APT) was performed on the LoSc modified alloy at different strain levels as it exhibited the best microstructure and hardness. APT studies correlated with TEM investigations were utilised to understand the effect of severe plastic strain on the precipitate composition, size and distribution. The studies revealed increased solute segregation at the SPD induced non-equilibrium grain boundaries during HPT processing. The precipitates present prior to HPT processing. The results indicated an enhancement in the diffusion activity of solute atoms in the Al matrix during HPT processing. The possible factors contributing to enhanced atomic mobilities of the solute atoms were assessed, namely, thermally activated diffusion, diffusion aided by SPD induced non-equilibrium grain boundaries.

Friction stir processing of squeeze cast A356 with surface compacted graphene nanoplatelets (GNPs) for the synthesis of metal matrix composites

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ABSTRACT

Friction stir processing (FSP) was applied to graphene nanoplatelets (GNPs) physically compacted on the surface of squeeze cast A356 alloy to incorporate GNPs within the matrix and to improve its mechanical properties. Squeeze casting resulted in finer size silicon and intermetallic compounds in cast microstructure, and subsequently FSP further refined the microstructure of squeeze cast A356 alloy, and GNP reinforced A356 alloy. The finer Si particles, intermetallics and graphene dispersed in the matrix increased the yield and ultimate tensile strength of FSP squeeze cast A356 alloy compared to the results reported in prior literature for FSP A356 alloy. Eutectic Si needles have been converted to fine spherical particles during FSP and were uniformly distributed within the nugget zone. The crystallite size of GNPs which were physically adhered to the surface of squeeze cast alloy prior to FSP decreased after FSP as a result of deformation. Thus, a combination of squeeze casting, and friction stir processing and incorporation of GNPs reinforcement in the A356 matrix is a promising route to further improve its mechanical properties.

Twin-induced compressive strain hardening behaviour in Titanium

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ABSTRACT

This work investigates the twin-induced compressive strain hardening behaviour in commercially pure Titanium (Ti). The progressive evolution of the microstructure and texture was studied by electron backscattered diffraction (EBSD) by interrupting the quasistatic uniaxial compression tests. The microstructural features indicated the presence of $\{10\bar{1}2\}\langle\bar{1}011\rangle$ extension twins (ET), $\{11\bar{2}2\}\langle11\bar{2}\bar{3}\rangle$ contraction twins (CT) and their interactions [1, 2]. It was observed that at low strains, geometrically hard ET originate in texturally soft grains. ET- ET interaction occurs forming ~56.8°-(10\bar{1}0) angle-axis pair boundaries. With increasing strain, the ET lamellar structures broaden to consume the entire parent grains. CT develop on texturally hard ET domains at intermediate strains and were geometrically softer. CT- CT, ET-CT type interaction and CT-ET double twins with ~77°-(10\bar{1}0), ~87°-(4\bar{2}\bar{2}1) and ~44.5°-(5\bar{1}\bar{4}0) angle-axes pair boundaries evolve.

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Mechanical properties and Microstructural evolution of Zr-4 Alloy processed through Rotary Swaging

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ABSTRACT

Zr-4 alloy are used as a cladding material in nuclear reactors due to its high neutron absorption cross section, excellent tensile properties, fracture strength, and corrosion resistance. A rotary swaging is a process that reduces the cross-sectional area of solid rods by repeated radial blows with one or more pair of opposed dies. An excellent combination of ductility and strength of Zr-4 alloy was achieved through low-strain-swaging followed by annealing treatment in the present work. Improvements in mechanical properties, especially ductility enhancement, were substantiated with the detailed and targeted microstructural analysis with EBSD and TEM. The grain size, misorientation distribution and texture of the annealed Zr-4 alloy is characterized by using EBSD. The dislocation mobility, precipitates, and formation of annealing twins were characterized by TEM. The mechanism of texture evolution, i.e., stable pyramidal (especially in Zr-4 alloy), is explored to establish an excellent correlation between ductility, strength, and evolved fine-grained microstructure.

Keywords: Swaging; Zr-4; Annealing; EBSD; TEM
Origin of grain size stability in Friction Stir Processed Al powder compact

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ABSTRACT

Ultra-fine grained and nanostructured materials have received great attention owing to the enhancement in the mechanical properties. However, the log-term stability of these microstructures, particularly in materials having low melting point is not addressed adequately. In this regard to sustain the grain size of ultra-fine grain Al, a modified approach is utilized using Friction Stir Processing (FSP). Aluminum powder compacts produced using cold iso-static pressing were subjected to FSP [1]. To compare the microstructure and mechanical properties, at similar processing conditions, as-cast Al was also subjected to FSP. All the processed samples were given a heat treatment at 450 °C for 2 hrs. Microstructure analysis through electron back-scattered diffraction (EBSD) and transmission electron microscopy, mechanical property characterization through quasi-static tensile tests were conducted. The grain size distribution of FSPed powder compacts remained almost similar after the annealing, with an average grain size of 10 µm. As a result, the yield strength and ultimate tensile strength remained almost similar and were more than 100 % compared to FSPed as-cast counterparts. Energy filtered TEM chacracterization of the FSPed pwder compacts indicate presence of oxygen clusters. In-situ heating experiments confirmed the pinning of grain boundaries by these oxygen clusters. Thin oxide layers from the surface of the powder particle have shown to be effective to restrict the grain growth in Al at 450 °C.

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Fatigue Crack Growth Behavior of a nanocrystalline HPT-deformed Ti-Nb alloy

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ABSTRACT

Titanium alloys exhibiting a low Young's modulus close to that of human bone and containing only non-toxic alloying elements are promising materials for hard tissue implants, such as Ti-45wt.% Nb. Common drawbacks with these alloys are their relatively low hardness, strength and fatigue strength. This can be overcome with methods of severe plastic deformation (SPD), for example high pressure torsion (HPT). Due to the dynamic loading that implants are facing during service-life, the fatigue crack growth (FCG) behavior is of great interest. Previous studies already showed promising results regarding the fracture toughness [1,2] of this Ti-45Nb alloy. Therefore, the FCG behavior of this alloy was investigated in various material states and orientations. After HPT deformation the microstructure exhibits a typical HPT-microstructure with elongated grains along the shear plane and a grain size in the nanocrystalline regime with grains of about 40 nm (short grain axis). Thereby the hardness increased by about 60%. Unlike the majority of studies on other SPD-processed materials, this alloy shows only a relatively weak anisotropic FCG behavior. This result was mainly attributed to the mostly transcrystalline fracture mode, which is somewhat unusual in nanocrystalline bcc metals [3]. Additional cold rolling was performed to increase the aspect-ratio of the grains and to enhance the anisotropy. Surprisingly this was not evident, which is due to the occurrence of shear bands dividing the grains into shorter grains again. After a heat treatment of 10 h at 300°C the hardness was further raised by about 10% and a weak cyclic R-curve behavior was found, resulting in a difference of short and long crack threshold values. An overall trend of decreasing threshold values towards higher load ratios was found, which is caused by a diminishing contribution of plasticity induced crack closure at higher load ratios. FCG experiments performed under vacuum conditions showed reduced crack growth rates in the near-threshold regime by about one order of magnitude and a somewhat increased threshold compared to lab air. This indicates a strong environmental influence on the FCG behavior.

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Producing hierarchichal nano-twins in Zr alloys through multiaxial cryo-forging for strength-ductility enchancement

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ABSTRACT

Severe plastic deformation technique has been largely employed to produce materials with refined and gradient microstructures leading to improvement in several mechanical properties. A recent work on cryo-multiaxially forged Ti below a total strain of 50% was shown to refine the microstructure by the formation of nano-hierarchical twins. Thus refined microstructure was shown to possess superior strength and ductility than its coarse-grained counterpart. In this work, we utilized the cryo-forging method to produce nano-hierarchical twins in Zr and Zircaloy-4 with the primary aim of optimizing the strain path for maximizing the strength-ductility enchanement. The strain path was optimized based on the initial texture by exploiting the profusivity of twinning. After each pass of deformation $(0.05 < \varepsilon < 0.10)$, the material was rotated by 90° and pressed so as to produce twins within the exisiting twins and minimize the activity of pyramidal < c+a> slip. The total strain was restricted to below 50%. The microstructural and mechanical characterization of thus cryo-forged Zr and Zircaloy-4 will be presented.

Multiaxial cryo-forging of Zr

Before deformation

After deformation



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Friction stir processing of Zn-Mg biodegradable alloys

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ABSTRACT

Magnesium, iron and zinc-based alloys are potential metallic biodegradable alloys for bioimplant applications. The issues of hydrogen evolution along magnesium's surface and the release of bulky corroded products of iron alloys during its usage necessitated to look for an alternate alloy system for biodegradable implant applications. Zn-Mg is one such alternate alloy that is widely explored by several researchers. The typical cast microstructure of Zn-Mg alloy contains α -Zn and Mg2Zn11. The cast alloy exhibits poor ductility as the intermetallic Mg2Zn11 phase preferentially forms along the grain boundaries of α-Zn. Therefore, the cast alloys were subsequently processed through hot rolling, hot extrusion and rarely through severe plastic deformation to enhance the mechanical properties. In the current study, for the first time, friction stir processing (FSP) has been used to refine the microstructure of Zn-Mg alloys with an intention to enhance the mechanical properties. Indeed, it is a challenge to friction stir process the Zn alloys as they have poor ductility (< 10%) in as-cast condition; whereas 13% ductility is required for successful processing. By choosing tool rotational speed that can impart significant heat input to the processed regime, this study demonstrated successful FSP of Zn and Zn-(0.5, 1.0 & 2.0) wt.% Mg alloys. The pure and Zn-Mg alloys are FSPed using constant tool traverse speed of 20 mm/ min and variable rotational speeds of 560, 710 and 900 rpm. Hardness of as-cast Zn was 32 VHN whereas addition of 2% Mg increased it to 105 VHN. Hardness of zinc increased to 39 VHN after FSP using 560 rpm; at the same time, increase of rotational speed retained the hardness around 40 VHN. On the other hand, hardness of Zn-Mg alloys significantly improved after FSP. Interestingly, hardness of alloys increased with the rotational speed. Usually, increase of rotational speed increases heat input in to the processed regime thus assists the development of coarse microstructure thus degradation in mechanical properties. An opposite trend has been observed in the current study. The possible reasons for this notable observation will be discussed.

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Corrosion and low cycle fatigue behaviour of ultrasonic shot peened Ti-13Nb-13Zr alloy

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ABSTRACT

Severe plastic deformation via ultrasonic shot peening (USSP) is a relatively new technology through which nanostructure can be developed at the surface of the metallic material and subsequently enhance the corrosion and fatigue resistance of the structural metals and alloys [1-5]. In the present investigation USSP technique was used to modify the near surface microstructure of Ti–13Nb-13Zr alloy. USSP was performed for 120 to 360 seconds of duration in order to obtain nanostructure and compressive residual stress at the surface. Microstructural changes were examined using XRD, SEM and TEM. Detailed transmission electron microscopy (TEM) investigation revealed nanocrystallization up to ~12 nm. The average surface roughness was found to increase with increase in USSP duration. Microhardness was observed to be highest in the surface region, and gradually decrease towards the substrate. Microhardness and also the depth of modification increased with duration of USSP treatment. No phase transformation was observed due to USSP treatment, as confirmed through XRD.

The effect of USSP on corrosion behaviour of the Ti-13Nb-13Zr alloy was also studied using Ringer's solution. Electrochemical impedance spectroscopy, potentiodynamic polarization tests and static immersion tests were performed. The electrochemical study revealed reduction in corrosion of the USSP treated samples. The grain refinement and surface roughness were the two opposing factors controlling the corrosion behaviour. Besides refined grain size and the presence of induced surface compressive residual stresses, the appropriate duration of USSP treatment produced optimal corrosion resistance that was found maximum in the 30 s of USSP treatment. However, the corrosion resistance decreased with an increase in the USSP duration from 30 s. Composition of passive layer was analysed using XPS technique. The improvement in the corrosion resistance from the USSP was mainly attributed to grain refinement and the associated compressive residual stresses in the surface region.

LCF tests were first conducted for $\pm 0.70\%$ total strain amplitude for the samples USSP treated for 120, 240 and 360 s of duration to find out the best fatigue life with respect to the peening duration. LCF life was increased considerably for all the specimens subjected to USSP treatment of 120, 240 and 360 s but the optimum enhancement was seen in the specimen USSP treatment of 4 mins, in which fatigue life was enhanced nearly three times. Low cycle fatigue tests were then conducted for Un-USSP, USSP (240 s), and USSP (240 s)-SR condition under fully reversible (R = -1) axial loading, with triangular waveform, at total strain amplitudes of $\pm 0.55\%$, $\pm 0.60\%$, $\pm 0.70\%$, $\pm 0.80\%$, and $\pm 0.90\%$ and at a fixed strain rate of 0.005 s-1. The microstructure and fracture morphology of the fatigue tested specimens were analyzed using scanning electron microscope. Fatigue crack initiation in the un-treated and USSP-SR samples was found from the surface whereas it was from subsurface in the USSP treated samples. It can be concluded that the corrosion and fatigue resistance of Ti-13Nb-13Zr are enhanced substaintially thorugh USSP using optimized process parameters.

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Development of metastable dual-phase ternary medium entropy alloy using cryorolling technique

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ABSTRACT

Medium entropy alloys (MEAs) are new emerging materials that overcome the strength-ductility trade-off. MEAs are subsets of high entropy alloys (HEAs) having configurational entropy in the range of 1R to 1.5R. Metastability engineering in high entropy alloy (HEA) is a well-reported technique in circumventing the strength-ductility trade-off by the combination of interface hardening and solid solution hardening¹. An attempt has been made to develop a low stacking fault energy (SFE) novel medium entropy alloy having dual phases by cryorolling technique to impart transformation-induced plasticity (TRIP) along with twinning-induced plasticity (TWIP)². Equiatomic MEA FeCrNi subset of cantor alloy proven to be displaying better mechanical properties than its other subsets. The yield strength of 700MPa, UTS of 1.03GPa and 54% ductility³. Furthermore, the composition of FeCrNi is varied along with minor addition of Si to lower the SFE of the system⁴. Deformation at the cryotemperature range significantly inhibits the dynamic recovery and recrystallization, thus preserving the defects generated by deformation and leading to the formation of twins, shear bands, and deformation bands⁵. Consequently, suppression of dynamic recovery along with the combination of transformation-induced plasticity (TRIP) and twinning-induced plasticity (TWIP) imparts high strength which can be further short-term annealed to attain good ductility. The work aims to develop dual-phase MEA providing a key to tune the mechanical properties using phase fraction. The novelty of the phase transformation of single-phase to metastable dual-phase after cryorolling provides a reference to future research in the field of TRIP + TWIP dual-phase MEAs.

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Metal bonding by Friction-Assisted Lateral Extrusion Process (FALEP) at room temperature

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ABSTRACT

The Friction-Assisted Lateral Extrusion (FALEP) is an SPD process [1,2] which can apply extremely large shear strain to a bulk or powder metal, in a single step, to obtain ultra-fine-grained sheet metal. In the present work, FALEP is applied for metal bonding of different layers of aluminum alloys at room temperature (examples shown in Fig. 1). Because of the excellent bonding capacity, FALEP can be used as a semi-continuous process producing sheet with unlimited length. The good bonding capacity is due to the huge increase by shear strain between the initial contact surfaces during a single step (in the order of 10 times). Therefore, it is expected that FALEP - when used in several cycles - can become a strong competitor to the well-known ARB (accumulative roll bonding) process, where the increase in the surface contact in one cycle is only a factor of 2. The cyclic FALEP process can be called 'ASB': accumulative shear bonding.

Keywords: FALEP, SPD, metal bonding, accumulative roll bonding, accumulative shear bonding



Fig. 1. Metal bonding by FALEP (a): Al5052-Al1050, (b): Al2024-Al1050 (SEM image).

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Microstructure and corrosion behaviour of additive manufactured AlSi10Mg alloy processed by high-pressure torsion

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ABSTRACT

Additive manufacturing (AM) has become an increasingly popular method that challenges traditional manufacturing techniques. However, additive manufactured components often lack mechanical and functional properties due to high porosity and higher grain size. In this work, we have explored the combination of additive manufacturing and severe plastic deformation (SPD) on the microstructure and corrosion behaviour of the AlSi10Mg alloy. First, the alloy was fabricated using laser powder bed fusion (LPBF) AM technique. Subsequently, it was processed using high-pressure torsion (HPT) at a constant pressure of 6 GPa for varied torsional rotations between a quarter and ten turns. The study of microstructure showed that the cellular structures and melt pool size obtained after LPBF become increasingly refined, accompanied by significant porosity reduction with increased HPT turns. X-ray diffraction (XRD) analysis demonstrated increased dislocation densities with increased HPT turns. It results in a significant increase in microhardness, nanohardness and Young's modulus. The change in corrosion behaviour was studied through electrochemical test in 3.5% NaCl solution, having 3.62 mg/L dissolved oxygen and pH 7 at room temperature using open circuit potential (OCP), potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS).

In situ experimental studies of plastic strain induced $\alpha \rightarrow \omega$ phase transitions in ultra-pure Zr and Zr2.5Nb alloy

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ABSTRACT

Unlike pressure- and stress-induced phase transformations (PTs), which initiate by nucleation at pre-existing defects (e.g., dislocations) below the yield, strain-induced PTs occur by nucleation at new defects generated during plastic flow [1-3]. Here, we report the first detailed in-situ quantitative study of strain-induced PTs in ultrapure Zr [4] and Zr2.5Nb alloy. The samples were initially pre-deformed plastically at ambient conditions to achieve saturated hardness, subsequently subjected to severe plastic deformation employing rotational diamond anvil cell (RDAC). In situ X-ray diffraction (XRD) and X-ray absorption scans were performed across anvil culet of RDAC at different load-shear pathways at the 16-ID-B beamlines at Advanced Photon Source, USA and Extreme conditions X-ray diffraction beamline (BL-11) at Indus-2 Synchrotron source, India. Phase fraction of α and ω phases and pressure distribution in each phase across sample in RDAC was determined using XRD analysis of diffraction pattern at each scanning position. X-ray absorption measurement along the radius was used to determine the sample thickness (h) profile. An analytical kinetic equation for volume fraction of the high-pressure phase was determined using experimental parameters at the sample center, defining the accumulated plastic strain q as $\ln(h_o/h)$ with initial thickness h_o . Our measurements show significant reduction in transition pressure in both ultra pure Zr and Zr4.5Nb alloy under compression and shear in RDAC as compared to that under hydrostatic compression. The kinetics of $\alpha \rightarrow \omega$ phase transition in both the samples is found to be independent of the magnitude of preliminary plastic strain (above the value causing saturated strain hardening), pressure-plastic strain path, and compression-shear path. The last result demonstrates that the strain-induced PTs under compression in DAC and torsion in RDAC do not differ fundamentally. Kinetic parameter for Zr4.5Nb alloy is found to be lower than that for ultra pure Zr.

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Underwater friction stir welding route for developing hybrid joints of aluminum and polymer of dissimilar thickness

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Abstract: - The hybrid joints of Aluminum 6061-T6 alloy (AA) and polypropylene (PP) were developed using the underwater friction stir welding route. The joint quality depends on mechanical interlocking and adhesive bonding between the base materials. Increasing the rotational speed and welding speed improves the material flow and better mechanical interlocking, and improves the joint quality. Process parameters having a rotational speed of 900 rpm, welding speed of 80 mm/min with the tool tilt angle of 2^{0} , and plunge depth of 0.2 mm exhibited sound weld owing to mechanical interlocking. The weld showed a shear weld strength of 1.43 MPa with a maximum hardness value of 93.2±0.2 HV at the weld center.

Keywords: Underwater friction stir welding; hybrid welding; Aluminum; Polymer

Nucleation of New Deformation Modes in Fully Annealed Nanostructured Metals

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ABSTRACT

Advanced structural materials are required to show both high strength and large ductility/toughness, but we have not yet acquired the guiding principle for that. The bulk nanostructured metals are polycrystalline metallic materials having bulky dimensions and average grain sizes smaller than 1 µm. Bulk nanostructured metals show very high strength compared with that of the coarse-grained counterparts, but usually exhibit limited tensile ductility, especially small uniform elongation below a few %, due to the early plastic instability. On the other hand, we have recently found that particular bulk nanostructured metals can manage high strength and large tensile ductility. In such bulk nanostructured alloys with fully annealed structures, unusual deformation modes different from normal dislocation slips were unexpectedly activated. Unusual $\langle c+a \rangle$ dislocations, deformation twins with nano-scale thickness, and deformation induced martensite nucleated from grain boundaries in the bulk nanostructured Mg alloy [1,2], high-Mn austenitic steel [3-5], and Ni-C metastable austenitic steel [6,7], respectively. Those unexpected deformation modes enhanced strain-hardening of the materials, leading to high strength and large tensile ductility. It was considered that the nucleation of such unusual deformation modes was attributed to the scarcity of dislocations and dislocation sources in each recrystallized ultrafine grain, which also induced discontinuous yielding with clear yield drop universally recognized in bulk nanostructured metals having recrystallized structures. Based on the findings in bulk nanostructured metals, we proposed a strategy for overcoming the strength-ductility trade-off in structural metallic materials [7-9]. Sequential nucleation of different deformation modes would regenerate strain-hardening ability of the material, leading to high strength and large tensile ductility. The strategy could be a guiding principle for realizing advanced structural materials that manage both high strength and large tensile ductility.

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In Situ Synchrotron X-ray Analysis for Allotropic Transformation of ZnO processed by SPD

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ABSTRACT

Zinc oxide (ZnO) with rocksalt (RS) crystal structure is attractive because of the bandgap which lies in the range of visible light absorption (1.8-2.2 eV). However, the RS structure is not stable at ambient pressure and temperature [1]. Nevertheless, application of high-pressure torsion (HPT) has shown that the RS structure can exist even at ambient pressure and temperature [2]. This study thus investigates the stability of RS produced by the HPT processing in terms of pressure, temperature and imposed strain. *In situ* X-ray diffraction (XRD) analysis is conducted using a high-pressure sliding (HPS) facility at BL04B1 of SPring-8 [3]. It is shown that RS is present after HPT processing under 6 GPa for N=3 turns but not for N=0 (only loading). The RS is not formed under 2 GPa even after N=3. The *in situ* XRD analysis reveals that the RS structure disappears as the temperature increases to more than ~150 °C at ambient pressure. However, under application of ~6 GPa, the fraction of the RS structure increases with temperature and reaches almost 100% at ~150 °C. The RS structure remains 100% after cooling to room temperature under the high pressure and even after unloading. It is shown that it is possible to produce 100% of the RS structure at the ambient condition by the assistance of plastic strain.

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On thermodynamics of nanostructured state and mechanical properties of NanoSPD materials in wide range of load intensity

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ABSTRACT

Thermodynamics of polycrystalline state was developed to link the collective behaviour of (intra- and intergrain) mesodefects with qualitative changes in mechanical responses under grain refining [1]. It was shown that mesoscopic phase transition in polycrystalline (quasi-brittle, ductile, fine-grain) states [2] can be associated with special type of critical phenomena – the structural-scaling transition [3] with drastic changes in the behaviour of mesodefect ensemble and generation of collective modes of defects influencing on metastable relaxation properties (plasticity) and damage localization. The transition to fine-grain (nanostructural) states is the consequence of degeneration of collective orientation mode of defects and the conventional intra-grain crystal plasticity mechanism is replaced by the intergrain sliding. The degeneration of collective orientation modes has the similarity with the first-kind phase transition, leads to the charp changes in the mechanism of momentum transfer and the criticality that is well-known as the Hall-Petch law violation in the term of the yield flow sudden break [4]. Out-of-equilibrium nature both crystal (intra-coarse grain) conventional plasticity and inter-fine grain plasticity has typical for critical phenomena different power (scaling) laws for observable parameter (the yield stresses). The criticality effect for this transition at some characteristic grain size was supported by the study of the difference in the energy absorbtion for the cyclic and dynamic loaded coarse- and fine grained alloys combined with "in-situ" infra-red imaging and estimation of the energy dissipation part. The scenario of damage-failure transition reveals also qualitative different scenario for conventional and fine grain materials. Degeneration of collective orientation modes of defects for fine-grain materials leads to the expansion of the process zone at the crack tip area in both transverse and longitudinal directions increasing the stored energy capacity preceding the daughter crack origin to provide the main crack advance. This tendency can be considered as the way to improve the mechanical properties of NanoSPD materials to increase the fatigue limit and the fracture toughness under dynamic loading.

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Improvement in Formability of Ti and Mg alloys by Equal Channel Angular Pressing

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ABSTRACT

The process of equal channel angular pressing (ECAP) has been widely investigated over the last two decades. Most of the research has focused on aluminium alloys with an emphasis on improving the mechanical properties. In this talk, research on ECAP where the objective has been to improve formability and workability of relatively brittle hcp metals like titanium and magnesium alloys has been summarized. ECAP of Mg alloy AZ31B was carried out for three passes through routes Bc and C using a die with die angle of 120°. The first pass was carried out at 250°C and subsequent passes were carried out at 150°C. Similarly ECAP of commercial purity titanium was carried out at 400°C. Workability was determined using upsetting tests on cylindrical specimens in the annealed and ECAPed condition at 150°C for the magnesium alloy and room temperature for CP Ti. It is observed that, for some passes, there is no decrease in the workability or even an improvement in workability even though ECAP increases the strength considerably. This suggests that fine grained microstructures produced by ECAP permits easier cold forming (for CP Ti) or warm forming (for AZ31B).

Ductility enhancement in Mg-0.2%Ce alloys

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Abstract

Ductility of Mg alloys can be enhanced by alloying, controlling the grain size and randomizing the texture. In this study, Mg-0.2%Ce alloys were processed using rolling, multi-axial forging (MAF) and equal channel angular pressing (ECAP) to fabricate three different textured samples from the same alloy. The samples were further annealed to produce similar grain size without altering texture. Rolled sample had a strong basal {0001} texture, the MAF sample developed a weak $\{01\overline{12}\}$ $[\overline{2}3\overline{1}2]$ texture component in addition to the basal texture and the ECAP sample exhibited a strong non-basal $\{\overline{1}2\overline{1}3\}$ $[\overline{2}11\overline{1}]$ texture component. The tensile properties, texture evolution and relative slip/twin activities in the samples were investigated experimentally and numerically. The tensile yield strength, ultimate strength and uniform elongation of the rolled, MAF and ECAP samples were 110MPa, 250MPa, 17%; 60MPa, 200MPa, 30% and 55MPa, 250MPa and 40%, respectively. The non-basal texture components in ECAP and MAF samples favoured the formation of extension twins and pyramidal $\langle c + a \rangle$ slip during tensile loading. Full field crystal plasticity finite element modelling (CPFEM) using the initial texture of the materials as input provided insights into the activation of different deformation modes and observed differences in hardening mechanisms as well as strain localization and premature failure of the rolled samples. CPFEM analysis confirms that Ce addition reduces the relative values of the critical resolved shear stress (CRSS) for the slip and twinning systems which, in turn, allows for texture modification during material fabrication. These, combined with the ability to control grain size in Mg–Ce alloy with excess Ce, provide options for ductility enhancement in Mg alloys.

Keywords: Mg-0.2%Ce, Ex-situ EBSD, CPFEM, Deformation twinning

In-situ heating neutron diffraction analysis of structural relaxation in additive-manufactured 316L stainless steel

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ABSTRACT

Structural evolution through nanostructuring and relaxation upon heating are examined by X-ray diffraction and in-situ heating neutron diffraction analyses, respectively, on an additive-manufactured (AM) 316L stainless steel. Significant structural changes occur in a very early stage of nanostructuring by high-pressure torsion leading to severe lattice distortion by the excess of dislocations and defects. The sequential information on the structural relaxation during in-situ heating neutron diffraction analysis provides the texture development, linear thermal lattice expansion, and stress relaxation of the nanocrystalline steel with increasing temperature up to 1300K, as shown in Fig. 1 [1]. COmpremented by the hardness measurements after heating, the results of the structural evolution are interpreted to describe microstructural recovery, recrystallization and grain growth behavior and the thermal stability of the nanocrystalline stainless steel. This study demonstrates considerable potential for utilizing severe plastic deformation for exploring post- and hybrid-additive manufacturing treatments to enhance physical and mechanical properties of the AM metals and alloys.



Fig.1 Contour plots showing variation in neutron diffraction patterns with time and temperature for the AM 316L SS after HPT for 15 turns [1].

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Re-desigining Constrained Groove Pressing Technique to overcome Strength-Ductility Limitations

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ABSTRACT

Constrained Groove Pressing (CGP) is known to be a versatile severe plastic deformation technique. It is also easy to scale this technique by applying the die design to constrained groove rolling. However, there are some inherent drawbacks in this technique that needs to be overcome before it can be commercialized. The standard 45° die used for CGP is known to lead to inhomogeneity in microstructure, and in turn, anisotropy and non-uniformity in mechanical properties. Our studies have shown that this inhomogeneity in microstructure can be related to stress concentration at the edge of the die tooth, resulting in the drastically reduced ductility and anisotropy of the severely deformed samples. This study led us to the understanding that the strength-ductility limitations can be overcome by redesigning the die profile. A new die with corrugated profile was designed with optimized geometry to obtain uniform strain distribution and homogeneous microstructure (Patent Application No. 202211064135). CGP of copper alloy processed using the newly designed die showed outstanding results with improvement in toughness, homogeneity, and isotropy.

The microstructural inhomogeneity, inherent in CGP, also leads to defect generation which leads to pre-mature failure during tensile tests. This led us to hypothesize that if an appropriate post-process heat treatment can be designed which results in controlled recrystallization, then the crack-propagation at the sites of microstructural inhomogeneity can be subdued and the toughness of the material can be enhanced. Our results show that using this approach, we can achieve toughness better than that of the starting material.

Dynamic working and mechanical property of homogenised AA7068

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Abstract

Plastic working of homogenized Al7068 alloy in the thermomechanical domain of 250-450°C and 0.001-1 s⁻¹ is analysed to obtain a dendritic-free microstructure and improved mechanical property after single step uniaxial forging. The rheological plot indicated an increase in the true stress values with a decrease in temperature and processing time. Other than thermomechanical variables, the true stress value also depend on the size and morphology of the precipitate phases. The constitutive calculation at peak stress estimated an average hot deformation activation energy of 228 kJ mol⁻¹. 3-dimension workability map indicated an increase in the power dissipation efficiency (PDE) with strain. However, the peak PDE of 54% is observed near 347-359°C and 0.001 s⁻¹. Microstructural evolution through SEM-EBSD revealed dominance of strained grains at all deformation conditions. However, strain-free grains are located in the grain boundary region, forming a necklace type of pattern. The microstructural analysis through EBSD and TEM indicated an increase in the dynamic softening with an increase in deformation temperature and process time, where the highest extent of recrystallization is seen at 450°C-0.001 s⁻¹. The microstructural analysis confirmed the occurrence of DRV and DRX during deformation at low and high temperatures, respectively. DRX is carried mainly by CDRX mechanism. The mechanical property analysis in the optimum working region is analysed through open forging at 350°C-1 s⁻¹, 450°C-1 s⁻¹, 350°C- 0.001 s^{-1} , 450°C - 0.001 s^{-1} , and base alloy. All the specimens have shown an increase in strength compared to the as-received alloy. But the specimen forged at 450°C-0.001 s⁻¹ has shown the tensile strength of 340MPa which is double the tensile strength of as-received alloy.

The Influence of SPD process on 3-D printed Al-10 Si-0.5 Mg alloy

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ABSTRACT

The paper deals with two aspects. The first one is making an Al-10Si-0.5Mg alloy through 3-D printing i.e. additive manufacturing (AM) method. The second method is to process this allov using repetitive corrugation and straightening (RCS). By employing these processes, it was expected to reduce the grain size below one micron and achieve improved mechanical properties. The purpose of carrying out two processes was to understand the grain size reduction in the 3D process and how far the grain size can be reduced after RCS, one of the SPD processes. From the SPD routes, it was very clear that the strength and hardness of the material increase substantially with plastic deformation and mostly without any change in the specimen dimensions. The 3D printed Al-10Si-0.5Mg product has been made by direct metal laser sintering where melting and fusing of the metallic powder was carried out. In addition to this, ageing treatment was also carried out to understand the microstructural changes. As expected, the age-hardened sample showed significantly improved strength of 396 MPa and ductility 14.537 and hardness increased approximately from 80 HV to 91 HV. After agehardening, the sample was processed for RCS. All the microstructural and mechanical properties of the as-cast and 3D printed and SPD processed Al-10Si-0.5Mg alloy were discussed in detail.

Keywords: Severe Plastic Deformation, Repetitive corrugation and straightening, Additive Manufacturing, Direct Metal Laser Sintering.

Investigation into microforming capabilities of engineered ultra-fine grained aluminium and magnesium alloys

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ABSTRACT

Microforming has emerged as an effective micro-manufacturing technique to address the current rising demand of micro-components in the fields of consumer electronics, avionics and biomedical equipment. The production rate and yield of micro-components via microforming is potentially much higher than other established micro-manufacturing processes. However, effective material deformation in microscale remains a big challenge, which limits the wide industrial practice of microforming. The difficulty arises when the specimen size become equivalent to the grain size of the material. This causes fewer numbers of grains to be present in the deformation zone and thereby leads to deviation from the expected trend in the deformation and increased scatter. This phenomenon is termed as 'size effect'.

The proposed work established the stratergies to overcome few microforming based challenges. In order to neutralize the size effect, present work proposes to substitute the conventional coarse-grained microstructure with specially engineered ultra-fine grained (UFG) microstructures in both Al and Mg alloys. Severe plastic deformation routes were used to develop engineered UFG microstructures in Al and Mg alloys. The microforming capability of the UFG microstructures has been assessed by comparing the limiting drawing ratio, flow curve and forming limit diagram of the UFG material with the conventional coarse-grained material. The feasibility study of a newer superplastic microforming technology was also explored on engineered UFG microstructures. Investigation reveals significantly improved microformability in case of UFG materials of both Al and Mg alloys with less processing scatter. Micro-mechanics investigation and structure-property correlation have been carried out to substantiate the improved microforming capabilities of the UFG materials.

Improvement in mechanical and electrical properties of Cu-Graphene nanocomposites prepared by high-pressure sintering

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Abstract

Graphene reinforced Cu-based nanocomposite (Cu-Gr), synthesized using PM-route followed by high-pressure forming (up to ~8 GPa at 300°C), achieved 96% of relative density, improved electrical conductivity up to 84% IACS, along with a significant increase in hardness, and Young's modulus up to ~94 GPa. The structure-property correlation and evolution of defects densities were investigated using electron microscopy and nanoindentation approach, and then backed up computationally. Further Molecular dynamics (MD) over the nanoindentation reveals that during loading, load transfer occurs from Cu matrix to the graphene reinforcement and graphene starts distortion, which further strengthens the theory. Grain growth and grain boundary sliding (GBS) are dominant deformation mechanism involves grain boundary movement through dislocation motion, stacking faults (SFs) and TBs formation, along with matrix failure through grain growth and GBS with an increase in loading, and subsequently bending (bow shape) processes.

Keywords: Cu-Graphene nanocomposites, High pressure sintering, Electron Microscopy, and Defects

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Surface Severe Plastic Deformation of Wire Arc Additive Manufactured Pure Copper

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ABSTRACT

Pure copper is mainly used for high heat exchangers, electronic components, and mechanical components such as springs, bearings, etc., owing to its good electrical and thermal conductivity. Nanostructure formation on surface layers of bulk materials has significant interest due to having extraordinary structural and physical properties. In the present study, the Surface mechanical attrition treatment method (SMAT) was applied to pure copper fabricated by Wire arc additive manufacturing to obtain the nanostructured grains in the surface layer. The WAAM samples were processed by SMAT at ambient temperature for 5, 10, and 20 minutes. X-ray analysis of SMAT processed samples showed peak broadening and increased FWHM, revealing refined grain sizes below 50 nm on the surface layer. Besides, the increasing treatment time above 15 minutes led to a decrease in the dislocation densities and deformation twins. It was observed due to the beginning of the dynamic recovery in the 20 min samples. Hence, the optimum time of the SMAT process was found to be 10-15 min based on the gradient structure layer's volume fraction, which gives the good mechanical strength of copper samples.

Acknowledgments

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Friction Surfacing: A new way to repair surface cracks

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ABSTRACT

Engineering materials experience a complex state of stress, strain and corrosive environment during their service. Most of the time the damage occurs on the surface and this significantly lowers the life of the entire component. For example, fatigue failure usually starts with surface cracks. Once the crack is detected, it must be addressed immediately before it propagates. There are limited ways through which the surface cracks can be repaired to put the damaged component to service once again.

In the present work, we demonstrated that friction surfacing can be used a tool to repair surface cracks on engineering components. The repaired component can be put to service once again with enhanced service life. Crack repair was performed by depositing coating of Inconel 718 on a self-mating substrate. The artificial crack, made on Inconel 718 surface, was completely filled and the coating exhibited sound bonding with the substrate. Process parameter were optimized in accordance with crack dimensions for complete filling of the crack. Microstructure characterization showed equiaxed fine grains with an average grain size of 1-3 μ m and significant amount (>78%) of high angle grain boundaries in the coating microstructure. The microstructure development in the coating is attributed to occurrence of dynamic recrystallization due to the combination of high strain rate and high temperature during friction surfacing. The hardness of the coating was higher than the base substrate material. Three-point bending test showed no delamination up to a deflection angle of 108 degrees confirming strong adhesion between the coating and the substrate. The method opens up a new way to repair surface cracks and it promotes re-use, over replacement of components.



A novel manufacturing method to develop ultra-fine grained Al/Cu bimetals.

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ABSTRACT

Owing to their hybrid properties, bimetals find a huge application in the field of various industries including building, transportation, consumer products, electronic products, industrial machinery, etc. Al/Cu bimetals are in high demand in electrical components like cables, connectors, busbars, and washers. Al/Cu bimetals provide a significant weight reduction to the component without compromising the electrical and thermal performance with enhanced corrosion properties. The various manufacturing routes to develop Al/Cu bimetals include explosive welding, diffusion bonding, and roll bonding process. The Al/Cu bimetals developed by the aforementioned routes often show an inherent tendency of intermetallic formation at the interfacial zone and lead to diminishing the properties of bimetallic sheets like poor interfacial bond strength and poor static strength. Bulk ultra-fine grained (UFG) materials often show superior properties than coarse-grained counterparts in terms of strength, superplasticity, wear resistance, etc. Al/Cu bimetallic sheets with ultra-fine grained microstructures will be the right candidates for industrial applications.

The present work aims to develop UFG Al/Cu bimetallic sheets via a novel hybrid manufacturing route. The hybrid novel manufacturing route is a combination of solution treatment followed by cryorolling (step 1), optimized surface preparation(step 2), and roll bonding (step 3). During step 1, cryorolling engineers an ultra-fine grained microstructure along with heavily stored energy in Al and Cu leads to (i) improve the static strength of both metals and (ii) promote the partial diffusion kinetics upon roll bonding process. The engineered Al and Cu sheets are sectioned to the required dimensions and subjected to an optimized surface preparation (step 2) including drilling, degreasing, scratch brushing with a distinct wire brush, stacking, and riveting before the roll bonding process. In the last step, UFG Al and Cu samples are subjected to a roll bonding process with an optimum thickness reduction at room temperature. The developed UFG Al/Cu bimetallic sheets showed an exceptional bond strength of ~ 17 to 19 N/mm and excellent tensile strength of ~323 MPa. These properties obtained in the present work are significantly higher than the Al/Cu bimetals developed by the existing manufacturing routes. The scientific know-how and governing mechanisms responsible for obtaining such extraordinary properties from the ultra-fine grained engineered Al/Cu bimetals will be presented in detail.



Fig.: Illustrates the (a) bond strength vs peel extension and (b) engg strain vs engg stress, plots of Al/Cu bimetals.

Keywords: ultra-fine grained material, roll bonding, asymmetrical rolling, heat treatment, bimetallic sheets.

Effect of ECAP routes on microstructure, texture, and mechanical properties evolution in pure magnesium

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ABSTRACT

Mg and its alloys have a potential for automobile, aerospace, electronics, and bio-implant structural applications primarily due to weight-saving properties. However, the major drawback is poor strength and formability. Equal channel angular pressing (ECAP) is a widely known severe plastic deformation (SPD) technique to obtain ultrafine grains to improve such properties. Notably, microstructure, and texture are strongly influenced by ECAP routes due to their different strain paths. In this work, pure magnesium was deformed by ECAP via routes A, B_C, and C up to 8th pass to investigate their effect on the microstructure, texture, and mechanical properties. The microstructure and texture were investigated using optical microscopy, and electron-backscattered diffraction (EBSD) in scanning electron microscopy (SEM) to understand the deformation behavior. Micro-Vickers hardness, and uniaxial compression tests were performed to investigate the mechanical properties in correlation with microstructure and texture.

Keywords: Equal channel angular pressing (ECAP); Magnesium; Microstructure; Texture; Mechanical properties

Effect of strain rate on the retained austenite stability in a Medium-Mn steel

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ABSTRACT

Medium Mn steels are one of the most promising candidates among the third-generation AHSS for automotive applications due to the enhanced strength and ductility exhibited by them. The superior mechanical properties can be attributed to their extended strain-hardening behavior which is dependent on the stability of the retained austenite present in the microstructure. The current work involves the study of the effect of a high strain rate on a medium Mn steel composition designed and developed using the CALPHAD approach to optimize critical parameters such as austenite stability and stacking fault energy [1]. The objective of this study is to understand the effect of different strain rates (10^{-3} , 1, and 10^3 s⁻¹) on the stress-strain response and the austenite to strain-induced α' -martensite phase transformation under uniaxial compression. The results show an enhanced stability of the retained austenite and consequently a lower strain hardening behavior with an increase in the strain rate. This stability observed in the retained austenite can be attributed to the adiabatic heating which becomes predominant at higher strain rates and the retardation in the strain-induced transformation of the retained austenite as a function of strain rate [2]. The study aims to deconvolute and understand both possible reasons and highlight their contributions to the overall stability of the retained austenite.

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Thermal stability of microstructure and Texture in Ultra-Fine Grained (UFG) Al-Mg-Sc-Zr alloy processed through severe cold rolling and annealing

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Abstract

The present investigation is aimed at exploring the microstructural stability of ultra-fine grain (UFG) non heat treatable Al-alloy AA5024 (Al-Mg-Sc-Zr), containing thermally stable and coherent precipitate $Al_3(Sc_x, Zr_{1-x})$. In this investigation, alloy has been purposefully given a severe rolling reduction of 90%, 95% and 98% in unidirectional (UDR) and cross-directional (CR), and subsequently annealed at 350°C starting from 1h to 100h, in order to assess the role of defect populated microstructure and crystallographic texture on stability of this alloy. It has been observed that microstructure remains stable with minimal recrystallisation till 10 hours in nearly all cases. An interesting observation has been made that recovery is prevalent, that too with very slow kinetics, that has been attributed to the stability of the precipitates till end. Crystallographic texture is Cu type (a typical of high SFE alloy) that remains identical for annealed and deformed state for both UDR and CR sheets. However, a decrease in strength of orientation distribution function (ODF) has been observed at 98% of UDR whereas this drop comes early at 95% in CR case. Variation in hardness also exhibited nearly similar trend for UDR and CR samples as well as for annealed sheets. These findings indicate the possibility for intermittent dynamic recovery just before these reduction levels in addition to slight increase in fraction of recrystallised grains. A comparison of texture obtained from VPSC simulation with the experimental texture clearly showed the contribution of recrystallisation in overall texture that caused a drop in texture intensity.

Development of Gradient Metallic Materials and Coatings by Electrodeposition: A Strategic Approach to Improve the Resistance to Mechanical Degradation

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Addition of alloying elements to nanocrystalline (NC) metals has been studied continually in order to enhance its strength and thermal stability. However, the utilization of these NC alloys into their potential applications is delayed due to their limited toughness. A heterogenous gradient comprising of varying microstructure and composition can be a strategic approach in achieving synergy between some competing properties. Alloys with a gradient in both microstructure and composition can be produced using electrodeposition, as the method offers flexibility to tailor the structure and the properties by controlling the deposition parameters. In the present work, microstructurally and compositionally graded Ni-Co alloys are synthesized through electrodeposition route with and without grain refiner. The synthesized Ni-Co alloys are tested for their thermal stability, hardness, and tensile behavior. The microstructures of the as-deposited, heat treated and their post-deformed specimens are examined in-detail using electron microscopy. The microstructure of the heattreated gradient samples showed lesser grain growth as compared to the homogeneous samples indicating a better thermal stability. The sharp peak in the DSC curve corresponding to the grain growth in the homogenous alloy manifested as a diffused peak in the graded alloy. The gradient Ni-Co exhibits improved ductility along with a gradual change in hardness when compared to the homogeneous alloy, in both as-deposited and heat-treated conditions. The fractography shows the variation in dimple morphology in the graded specimen indicating non-uniform deformation, which could be associated to the increase in ductility. Various multiscale strengthening and plastic deformation mechanisms occurring due to the varying composition and microstructure across the thickness of the graded alloy could be attributed to the enhanced strength-ductility trade-off in the gradient alloy.

Keywords: Ni-Co alloy; Gradient microstructure; Electrodeposition; Thermal Stability; Deformation behavior.

Investigation of Effect of Severe Plastic Deformation on Laser Transformation Hardening of Low Carbon Low Alloy Steel Prakash G. Ranaware^{1,*}

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ABSTRACT

Laser transformation hardening (LTH) is the efficient process used for synthesizing a hard, wear resistant surface layer on steel components through the ultrafast heating action of a high energy scanned laser beam. Initial microstructure of steel plays very significant role on nucleation and growth kinetics of austenite formation during ultrafast heating of laser treatment. Severe plastic deformation can produce nanocrystalline microstructure in steel which can serve as an interesting microstructure as initial microstructure for LTH. In the present work, severe plastic deformation of low carbon low alloy steel specimens containing tempered martensitic structure was performed to produce nanocrystalline structure. These specimens then subsequently surface hardened by Laser transformation hardening process. Specimens of AISI8620 steel were used for investigation. Tempering was done at three different temperatures (namely 205°C, 425°C, and 650°C) before severe plastic deformation. SPD was performed using shot-peening method. Effect of these initial microstructures on LTH was investigated using optical microscopy, electron microscopy, microhardness traverse and XRD. It was observed that nanocrystalline structure containing more martensitic volume fraction and lesser carbides before SPD produced finer martensitic structure with shallow hardened case depths after LTH. On the other hand microstructure containing more volume fraction of ferrite and carbides produced coarser martensite with deeper hardened case depths.

Key words: Severe plastic deformation, Shot-peening, Laser transformation hardening, AISI 8620, Tempered structure, Ultrafast heating

Interdiffusion behavior in a severely deformed Cu-Ni diffusion couple

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ABSTRACT

For coarse-grained polycrystalline alloys, interdiffusion of the Cu-Ni system is well investigated, and the composition dependent interdiffusion coefficients decrease with an increase in the Ni concentration [1]. In the present study, the influence of severe plastic deformation of the end-members on the interdiffusion behavior is explored. Severe plastic deformation of pure Cu and Ni is carried out via high-pressure torsion (HPT) to obtain ultra-fine grain sizes. The interdiffusion behavior for undeformed (coarse-grained) and deformed (ultra-fine-grained) materials is compared. Hardness and reduced modulus are measured by nanoindentation using a Berkovich tip. The composition dependency on these properties is examined.

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Surface-nanostructuring and thermal stability of IN718 Superalloy treated by ultrasonic shot peenning

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ABSTRACT

The hot corrosion, oxidation, and low cycle fatigue (LCF) are prevalent damage processes of aero engine components. Therefore, it is of utmost importance to investigate the resistance of futuristic alloy in simulated service environment and to develop new technologies to prevent their degradation and premature failure. Like oxidation, fatigue is a surface sensitive phenomenon since fatigue cracks initiate from the surface. Therefore, improvement in oxidation, hot corrosion and fatigue resistance of such components through surface modification is quite relevant. There are several methods of surface modification. However, ultrasonic shot peening is relatively new and efficient process compared to other surface modification techniques to produce nanostructure and compressive residual stress, which in turn may improve fatigue resistance in harsh environment [1, 2]. Therefore, in the present investigation efforts were made to investigate the structure, microstructure, microhardness, residual stress and thermal stability of peak-aged IN718 subjected to USSP treatment.

The present investigation deals with the important issue of the thermal stability of surface nanostructured IN718 peak-aged superalloys by USSP. These samples were USSPed for a duration of 1, 3 and 5 min followed by heat treatement up to a temperature of 650 °C. The structure, microstructure and mechanical properties of the USSPed, and USSPed and heat treated samples were ascertained through X-ray diffraction (XRD), transmission electron micscopy (TEM), SEM-EDS and EBSD analysis, residual stress analysis and microindentation respectively. It was observed that with increasing the duration of USSP from 1 min to 5 min, the depth of the surface modification increases from ~58 μ m to 145 μ m. The nanostructured layer in USSPed was found to be thermally stable up to 650 °C as no significant reduction in the deformed layer. The microhardness of the USSP treated at 250°C, 350°C and 450°C samples were higher than the peak-aged IN718 even up to a depth of 150 μ m. Further, the diffraction constrast imaging of USSPed and heat-treated samples confirmed the nanocrystalline nature of the IN718 alloy up to 650 °C. The feeble spots corresponding to the superlattice reflection confirmed the presence of the precipitates along with the matrix even at higher temperatures.

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Mechanical Behaviour of Ultrafine-grained SS304L Produced by Reversion of Strain Induced Martensite formed by severe plastic deformation.

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ABSTRACT

Ultra-fine grained SS304L was produced using the technique of reverting strain induced martensite. In this method, severe plastic deformation was given to the specimen to form martensite and reversion treatment to form an austenite phase with ultra-fine grains. The commercially available plate of SS304L of composition 0.03C, 1.065Mn, 0.54Si, 18.27Cr, 8Ni, 0.31Cu, and 0.320Mo was rolled up to an effective strain of ~2.0. This was done by dipping the plate in liquid nitrogen, removing and rolling it to a few passes; this process was repeated till the final effective strain of 2 was achieved. Subsequently, the rolled plate was thermally treated at 700°C for 30 mins. The median grain size obtained after the thermal treatment was 800 nm. The flow behaviour of this ultra-fine grained (ufg) SS304L was studied in compression at the strain rate of 10⁻³/s and was compared with that of the coarse grained SS304L. The evolution of strain induced martensite with strain in ufg-SS304L was determined using Ferrite-scope. The fracture properties and fatigue crack growth rate (FCGR) of this ultra-fine grained SS304L was found to be 900 kJ/m² and 200kJ/m², respectively. FCGR of ufg-SS304L(da/dN) was higher and threshold stress intensity range(ΔK_{th}) associated with da/dN was lower as compared to coarse grained SS304L. The electron back scatter diffraction of the crack front revealed crack propagation through strain induced martensite for both ufg and coarse grained SS304L.

Evolution of microstructure and mechanical properties of 2099 aluminium alloy deformed by high-pressure torsion (HPT)

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Experiments were conducted to see the microstructure and texture evolution in the 2099 aluminium alloy deformed by high-pressure torsion (HPT). Disk shape samples of 0.25, 2 and 5 rotations were prepared using HPT, which are 8 mm in diameter and 0.8 mm in diameter. Quantitative and qualitative microstructural studies were carried out using high-resolution electron backscattered diffraction (EBSD), convolution multiple whole profile (CMWP) analysis and transmission electron microscopy (TEM). Bulk texture measurements reveal that different shear texture components are present at different strain levels. EBSD analysis exhibits the continuous dynamic recrystallization behaviour of the HPT specimens owing to the high stacking fault energy of the material. Dislocation density increases with increasing the strain value but gets saturated after an approximate strain value of 27. Hardness values show a similar trend. Crystallite size shows the opposite trend, i.e. decreases with an increase in strain values and acquires saturation after a certain strain level. TEM micrographs reveal the precipitation evolution and dissolution at different strain levels.

Keywords: - High pressure torsion; 2099 Aluminium alloy; EBSD; CMWP; TEM

Ultra fine grained foils from extrusion machining of a nickel-based super alloy

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ABSTRACT

Large strain extrusion machining (LSEM) is a hybrid manufacturing operation combining machining and extrusion to afford effective control of both microstructure and geometry. This effort presents the *extrusion machining* of a nickel-based super alloy, Inconel 718, for obtaining foils with an *ultra fine grained* microstructure. This has been accomplished with a combination of numerical computations and experimental work. In particular, a new constitutive law developed from the *Zerilli Armstrong* formulation has been used along with real time machining data to model deformation in metal cutting operations. The ZA law simulates both continuous- and localized-shear with corresponding parameter values in a single ZA equation. The ZA law, which captures plastic flow at large strains, is used with finite element models of two-dimensional plane-strain orthogonal machining to simulate continuous- and localized-shear. Other than successfully predicting important parameters in machining of Inconel 718, finite element predictions were also used for setting up an *extrusion machining* setup to generate UFG foils of Inconel 718, see Figure 1.



Figure 1. Use of (a) finite element predictions (b) for setting a extrusion-machining operation to generate UFG foils of Inconel718



Severe plastic deformation of a lath martensitic steel: exploring the microstructural origins of exceptional strengthening

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ABSTRACT

Severe plastic deformation (SPD) is known to strengthen metallic materials through drastic refinement in the grain size. Concomitant sources of strengthening are increase in defect density, alignment of defects and increase of effective stacking fault energy. An attempt is made to test the limits of this paradigm on a material that is already strengthened by these same sources, where further deformation is expected to yield diminishing returns.

The material selected is a lath martensitic steel- an alloy with a dense network of martensitic boundaries and a high dislocation density, especially in the untempered state. Further, the material has a very high stacking fault energy due to its body-centred crystal structure. This already-strengthened alloy is imparted SPD through high pressure torsion (HPT).

It is demonstrated that HPT leads to exceptional strengthening, despite several strengthening sources already built-in to the alloy. This strengthening is sensitive to the starting microstructure, and appears to have a multiplying effect within the tested range. The applied hydrostatic pressure appears to play a significant role in achieving high strain, primarily through suppression of macroscopic defects. This is highlighted by contrasting the outcomes of HPT with those of uniaxial deformation. An attempt is made to delineate the effect of starting microstructure are discussed, with emphasis on the uncertainty associated with interpretation. It is concluded that the limits of strengthening as well as microstructural refinement arising from SPD may require re-evaluation, towards which some strategies are proposed.

Influence of Co content on the simultaneous enhancement of strength and ductility in severely drawn textured Ni-Co microwires

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ABSTRACT

Strength and ductility are mutually exclusive properties that are frequently required for structural applications. Nanocrystalline (NC) (grain size d <100 nm) and ultrafine grain materials (UFG) (d 100 to 1000 nm) have excellent strength but low ductility at room temperature, which limits their application. These are typically manufactured by electrodeposition and severe plastic deformation (SPD) techniques. Unlike HPT and ECAP techniques, wire drawing can produce bulk UFG and NC grains with a strong fiber texture. The present study investigated the influence of stacking fault energy on strength and ductility in Ni-Co microwires by varying Co addition ranging from 30 to a maximum of 60 wt.%. Ni-Co microwires were cold drawn to a maximum strain of 5.88 and were tested in tension. Both drawing strain and Co content increased strength and ductility simultaneously. The enhanced strength is a consequence of the finer grain size with an increase in Co, and the larger ductility is related to a combination of greater strain hardening and a higher strain rate sensitivity with an increase in Co. The textured drawn Ni-Co wires exhibited higher strengths than those obtained by severe plastic deformation with comparable grain sizes.
Mechanical and tribological performance of severe plastically deformed Ni-base superalloy composites

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ABSTRACT

The operating lifespan of mechanically stressed components can be extended by reducing wear and friction, which is classically realized by liquid lubricants. However, their application is limited to low operating temperatures, as they degrade at higher temperatures. Self-lubricated metal matrix composites (MMCs) seem promising for high-temperature applications [1,2]. In MMCs, solid lubricants, exhibiting significantly higher decomposition temperatures, are dispersed in a metal matrix. In this work, different quantities of graphite were integrated into Ni-base superalloys (Inconel 718 and Inconel 625) by using high-pressure torsion deformation. The idea is, on the one hand, to reduce wear by increasing hardness through grain refinement and, on the other hand, to reduce friction by reincorporating a proper amount of graphite without compromising mechanical properties. The aim is to find the appropriate process parameters and amount of solid lubricant to achieve the best properties.

Scanning electron microscopy reveals a smaller agglomerate size and a better distribution of the solid lubricant phase when the powders were colloidally mixed prior to HPT deformation. The self-lubricity of the composites was studied by using a ball-on-flat tribometer. Mechanical properties were assessed by micro tensile tests and microhardness measurements, observing increased values in the latter.

Acknowledgments

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Structure-property correlations at a micrometer length scale of hot rolled and cold rolled dual phase steels.

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Advanced high-strength steels (AHSS) have wide variety of applications in various parts of car body due to their combination of good strength and ductility. In recent years significant efforts have been made to further enhance the mechanical properties of these steels by altering the thermomechanical processing (TMP). While a dual phase ferrite-martensite microstructure obtained after inter-critical annealing is the typical end result, several prior processing steps including hot rolling, coiling, cold rolling, etc have significant effect on the final microstructure. In this regard, we generate different strengthening phase microstructures with ferrite matrix along with pearlite, bainite and martensite for a DP600 grade sheet steel. The different microstructures are obtained by coiling at different temperatures subsequent to hot rolling. The samples were also subjected to different cold rolling reductions after coiling. The microstructure and mechanical properties of these specimens were characterized by various tools such as optical, SEM, EBSD, micro-Vickers hardness and high-speed nanoindentation mapping. An assessment of the microstructure-property correlation at the micrometer length scale will be presented along with quantification of the local hardness of the matrix and strengthening phase during the various stages of processing. A discussion on the effect of the strengthening phase on the microstructure and hardness evolution will also be presented.

Ductility and formability of ultrafine-grained aluminium plates

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ABSTRACT

Ultrafine grained (UFG) materials feature very high strength, which is however at the expense of ductility. On the other hand, low ductility in tensile test does not necessarily means poor formability under more complex stress states. The aim of this communication is to discuses ductility and formability of ultrafine-grained aluminium alloy plates processed by two SPD techniques, i.e. Incremental Equal Channel Angular Pressing (I-ECAP) and a hybrid process combining multi-turn Equal Channel Angular Pressing (mt-ECAP) and upset forging. The materials investigated here were 3XXX and 5XXX series aluminium alloys, i.e. AA3003 and AA5754. Using both techniques, it was possible to acquire plates of an average grain size of around 0.4 µm and a fraction of high-angle grain boundaries exceeding 60%. It was assumed that the improvement of ductility and formability is possible via a proper selection of temperature and strain rate. Thus, the samples cut of the UFG plates were tested in tension at various temperatures and strain rates. In addition, cupping tests were performed and formability expressed as a cup hight value. The results show that the tensile elongation as well as formability can be increased two-fold at elevated temperature without excessive grain growth. The improvement of ductility was attributed to reduced apparent activation volume due to grain refinement, which translated into enhanced strain rate sensitivity. It was also revealed that the formability of UFG plates can even exceed those of coarse grained counterparts.

Microstructure and Microtexture development of Nonequiatomic MoNbTaVW Refractory High Entropy Alloy during High-Pressure Torsion at 473 K

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ABSTRACT

Refractory high entropy alloys (RHEAs) have excellent potential for industrial application in aerospace, nuclear industry, anode for X-ray production, and hydrogen storage material. Among the various families of RHEA, the MoNbTaVW-based single-phase alloy demonstrated superior high-temperature properties over state-of-art single crystal Co, Ni, and Ti-based superalloys under extreme environmental conditions (above 1200 °C) [1]. Furthermore, the ultrafine grain (UFGs) microstructure of MoNbTaVW RHEA exhibits enhanced thermal stability at high temperatures (1100 °C) over long-duration (~3 days) [2]. However, due to its low ductility and higher crystal anisotropy, the UFG microstructure cannot be generated by conventional processing methods. High-pressure torsion (HPT), a technique that combines quasi-hydrostatic compression and torsion straining, can be an efficient technique for achieving an UFG microstructure as it distributes axial stresses more homogenously without causing material instability. Therefore, the present work investigates the evolution of microstructure and microtexture in the non-equiatomic MoNbTaVW RHEA as a function of strain (ε) during HPT deformation at 473 K. The high torsion strain and hydrostatic pressure assisted by thermal stimulation stretched and elongated the grains uniformly along the shear direction, leading to an increase in dislocation density, boundary serration, and grain fragmentation by subgrain formation. Later, at higher strain, the pinching off or perforations in elongated grain boundaries and progressive lattice rotation of subgrain boundaries result in a recrystallized UFG microstructure of 120 ± 70 nm. Studying the strain-induced dynamic transformation of crystallographic textures with regard to shear direction showed the dominance of simple shear components. A strong shear texture dominated by the $D_1(11\overline{2})[111]$ component was developed with some other weak components $E_1(0 \ 1 \ \overline{1})[1 \ 1 \ 1]$, $D_2(\overline{1} \ \overline{1} \ 2)[1 \ 1 \ 1]$, and $J_1(0 \ \overline{1} \ 1)[\overline{2} \ 1 \ 1]$ during deformation at warm temperature (473 K). Thus, the UFG microstructure observed after HPT deformation is believed to be the synergetic effect of continuous dynamic recrystallization (CDRX) and geometric dynamic recrystallization (GDRX) in the BCC phase of RHEA.

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Mechanical response of multi-phase nanostructured high-entropy alloys prepared by different severe plastic deformation methods

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ABSTRACT

High-entropy alloys (HEAs)/complex concentrated alloys (CCAs) have exhibited unique mechanical properties that were not realized in dilute material systems. Often these interesting properties are attributed to the atomic level mixing of different elements in very high concnetrations which were part of a given material system. It is a well documented fact that refinement of microstructural features (especially grain size) would add another dimension to the materials development with enhanced strength. To understand the influence of nanocrystalline or ultra fined grain diameters in the concentrated material systems, we have developed various alloys using mechanical alloying. Thus alloyed powders were made into bulk samples using either spark plasma sintering or high pressure torsion. Structural features were investigated using X-ray diffraction (XRD) and electron microscopic methods including high resolution transmission electron microscopy. Mechanical properties were evaluated using different indentation techniques such as Vickers microindentation and depth-sensing nanoindentation. Based on these methods, the reasons for the strengthening and origins of strain gradient plasticity in these novel alloys are deduced. This talk will address the structure-property correlations in several CCAs including the once that are based on refractory elements developed in our laboratory.

A robust *in situ* technique for evaluating sub-surface plastic strains

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ABSTRACT

Mathematical models for studying severe plastic deformation (SPD) processes are often plagued by unphysical simplifications to make them amenable to analysis. A prominent alternative, yet in many ways complementary, route is the use of direct kinematic measurements. In this work, we present one such method that employs direct in situ imaging and associated analysis, specifically to determine large strain deformation fields, typical of SPD, in the vicinity of interfaces and dynamically forming surfaces. The method proposed uses a novel digital image correlation scheme (DIC) that is tailored to work with interfaces, using little to no post processing, resulting in minimal data loss. In this work, we describe this technique, and benchmark its performance with classical DIC methods that employ uniform grids, potentially with adaptive refinement. Results of inverting known displacement fields are first presented, followed by application to public-domain heterogeneous deformation datatsets (termed DIC Challenge 2.0). We then use this scheme to analyze a model SPD process represented by a tool interacting with a workpiece. The scheme can accurately estimate instantaneous strain and, given its accuracy near interfaces, also evaluate residual surface and sub-surface strains. We believe that the method will be of significant interest to the SPD community for directly evaluating strain heterogeneities during deformation.

Polymer Derived Ceramic Metal Matrix Composites; High-Strength, High-Ductility, High Temperature Grain Stability

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ABSTRACT

The quest for materials with high strength to weight ratio without loss in ductility has been going on for a long time. In general, for example in aluminum, when the strength is increased by severe plastic deformation or making of composites, both the ductility and thermal stability drop. Alloying makes it possible to get higher strength without loss in ductility. However, the elements that are added to get high strength without loss in ductility add to the cost significantly and make recycling almost impossible (there are more than 150 different kinds of alloys of aluminum). A new class of materials called the Polymer Derived Ceramic Metal Matrix Composite (PDCMMC) is shown to hold promise to address these issues. In this material a polymer (a cross-linked polymethylhydrosiloxane - PMHS), that converts to a ceramic on heating, is distributed using plastic deformation of the aluminum matrix. The plastic deformation of the aluminum matrix (with controlled amount of the polymer) is carried out using Friction Stir Processing (FSP), Friction Assisted Lateral Extrusion Process (FALEP) or a new process called High Pressure Compressive Reciprocating Shear (HPCRS). During the plastic deformation of the aluminum, the polymer which is brittle, is fragmented into fine particles with particle sizes as low as 1 nm. Particle sizes of several 100 nm are also seen. The aluminum mixed with these polymer particles is then heated to temperatures above 300 ^oC for up to 10 hours. The polymer at these temperatures and times convert to fine SiOC amorphous ceramics. The presence of the small nm sized particles lead to dislocation bowing and looping that results in high ductility and the larger ceramic particles results in diclocation pileup that lead to higher strength. Thus by controlling the size and distribution of the PDC particles one could control the strength and ductility and get materials with high strength and ductility. Heat treatment of the PDCMMC at temperatures up to 550 °C shows remarkable grain stability with virtually no growth in the grain size, which is around 1 micron. This grain boundary stability is the result of grain boundary pinning by the nanometer sized ceramic particles. Thus control of strength and ductility (and high temperature grain stability) with one composition of the material is possible. The single composition (or limited compositions) make the usage of material amenable to recycling.

Acknowledgments

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Modeling and Simulation of Large-Strain Complex Plastic Flows in Polycrystalline Aggregates

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ABSTRACT

Metal forming, manufacturing, and surface generation processes like repeated sliding, free-machining, and large-strain extrusion machining often involve very large plastic strains and high strain-rates. In particular, simulating metal processing operations involving soft, ductile metals like CP aluminum and annealed OFHC copper can be very challenging at the 100 µm-few mm length-scale due to the occurrence of complex, unsteady, and highly dissipative plastic flows, and the need to model microstructure, high interfacial friction, and potential ductile failure.

In this work, we examine extreme plastic deformation and complex flows in the metal sliding and chip formation regimes using a simple but effective phenomenological "pseudograin" model of polycrystalline aggregate plasticity.

In the sliding regime, this model effectively captures surface bump and fold-formation and severe surface damage in a single sliding pass. We also introduce a general, adaptive remeshing and mesh-to-mesh transfer scheme to simulate the processing of such soft polycrystalline aggregates using finite element analysis (FEA), with no restrictions on the friction at the tool-workpiece interface, grain-size or tool / die geometry. In the chip formation regime, the simulations successfully capture many experimentally observed aspects of 'gummy' metals, including "sinuous" flow, surface-fold and self-contact formation, thick and mushroom-like chips at low rake-angle, and thin chips with shear-plane--like deformation at high rake-angle. The method permits accurate analysis of the cut surface, capturing the high strains, strain gradients, and deformed grain shapes in the wake of the tool which is not possible with traditional single-mesh Lagrangian FE. The presence of microstructure necessitates careful consideration of issues that do not arise in homogeneous remeshing simulations, including the need to model grain-splitting, grain-tracking, and mechanisms of material flow near the tool tip. The generality of the remeshing approach makes it suitable to accurately model and simulate a wide range of metal processing or tribological operations involving severe plastic deformation.

Lastly, we also report ongoing efforts to implement more "physics-based" models of sub-continuum plasticity, using crystal-plasticity finite elements (CPFE) and the technical challenges involved in going beyond phenomenological polycrystalline aggregate plasticity.



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CO₂ photoreduction using high-pressure TiO₂-II synthesized by high-pressure torsion (HPT)

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ABSTRACT

Global warming caused by CO_2 emission has been a critical concern of the world in recent years. Photocatalytic reduction of CO_2 to CO and other useful components such as CH_4 is the cleanest way to solve this problem. In this regard, TiO_2 is the most utilized photocatalyst for CO_2 conversion. Anatase and rutile are conventional phases of this material which have been improved by various strategies to increase the photocatalytic activity for CO_2 reduction. In this study, high-pressure torsion (HPT) was used to produce the high-pressure phase of TiO_2 as TiO_2 -II (clumbite) which was utilized for photocatalytic CO_2 conversion for the first time. The high-pressure TiO_2 -II phase showed higher photocurrent density and CO_2 conversion rate compared to the anatase. Annealing treatment after the HPT led to improve photocurrent density and photocatalytic activity for CO_2 conversion compared to both anatase and sample treated by HPT. Enhanced photocatalytic efficiency is attributed to the annihilation of oxygen vacancies formed in bulk by the HPT process. The samples including the TiO_2 -II phase showed low electron-hole recombination rate, low bandgap, and modified band positions, as the reasons for their enhanced photocatalytic activity for CO_2 conversion to CO.



Fig. 1 Improved photocurrent generation and photocatalytic CO_2 conversion to CO after TiO_2 -II phase formation using HPT. (a) photocurrent density and (b) CO generation rate versus time for initial anatase powder and samples after HPT and after annealing.

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Bulk Rare Earth Free Permanent Magnets by Severe Plastic Deformation and Advanced Annealing Procedures

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ABSTRACT

The rising public awareness in green technologies, inter alia, the increasing demand for wind power plants and general electric mobility, forces an annual production increase of permanent magnets (PM). Such magnets are essential components for generators and motors and mainly define their efficiency. However, they rely on high-performance PMs, which usually contain a significant amount of cobalt and rare-earth elements (REE), both already recognized as critical materials by international institutes. Supply shortages are a critical bottleneck for modern economic and social development and enormous efforts are undertaken to design magnetic materials. The general objective in material engeneering points towards economical competing REE-free PM material systems.

In the present study, we demonstrate the successful processing of the REE-free intermetallic α -MnBi phase, which is a promising alternative PM material system. As a direct processing by melting metallurgy techniques fails, this phase is difficult to obtain, but exhibits exceptional hard magnetic properties. Our sample manufacturing is realized by high-pressure torsion (HPT). HPT-deformation of equiatomic Mn and Bi powder blends lead to disc shaped samples exhibiting outer dimensions of several mm to cm, whereas the microstructure simultaneously features particle and grain sizes in the range between several micrometres to tens of nanometres, respectively. Subsequently a magnetic field assisted thermal treatment in vacuum allows the formation of the hard magnetic α -MnBi phase.

We further discuss the influence of HPT deformation on the evolving anisotropic α -MnBi phase formation and an resulting anisotropic magnetic behavior being beneficial for applications. Obtained microstructures are characterized by means of scanning electron microscopy, SQUID magnetometry and X-ray scattering techniques. In particular, in-situ annealing treatments in combination with synchrotron X-ray diffraction are performed. To study the origins of the α -MnBi phase formation as a function of applied shear strain the experiments are performed with and without the impact of an additional external applied magnetic field on the annealing procedure. Herein, the successful formation of the hard magnetic REE-free α -MnBi phase will be presented, while a positive influence of HPT-deformation and magnetic field annealing on the magnetic properties is described.

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Grain boundary-induced martensitic nanostructures in bicrystals and tricrystals - Large strains-based phase-field study

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The heterogeneities, including grain boundaries (GBs) and triple junctions (TJs) in materially uniform polycrystalline solids, play a central role in nucleation and nanostructure evolution during the martensitic transformations (MTs) [1-3]. GBs and TJs can significantly increase the martensitic start temperatures in a polycrystal compared to its single-crystal counterpart [4]. Complex nanostructures form about the GBs and TJs, which strongly depend on the energies of the GBs and TJs. In this work, we have developed a large strains-based thermodynamically consistent multiphase phase-field approach for studying MTs in a bicrystal induced by a planar symmetric and isotropic grain boundary (GB) considering two martensitic variants [4]. The model considers two order parameters (OPs) to describe the A<->M transformations and variant<->variant transformations, and N-1 independent OPs to describe N grains of the sample [5-7]. The free energy of the system accounts for the barrier energy between the phases and variants, thermal energy, gradient energies accounting for the energy of the phase boundaries, strain energy, and variable energy for the GB, considering the change in GB energy due to MTs. The system of coupled mechanics and Ginzburg-Landau equations are derived using the balance of linear and angular moment and the first and second laws of thermodynamics. The governing equations have been solved using FEM, and the evolution of microstructures shown for temperature variation. The effects of GB width and TJ energy on nanostructures are studied. The results are qualitatively in good agreement with the experimental and atomistic results.

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Using the high-presure torsion (HPT) to fabricate a high-entropy oxynitride for photocatalytic H₂ evolution

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ABSTRACT

Exessive CO_2 emision from burning the fossiel fuels has forced the human to find an alternative without CO_2 emision. H₂ is a clean fuel which can be used instead of fossil fuels and photocatalysis is the cleanest way to synthesize this clean fuel under sunlight. Metal oxynitrides are low bandgap photocatalysts which have shown promising potential for hydrogen production among all reported photocatalysts, but the suffer from low stability. High-entropy ceramics are new materials contain five or more than five elemnts with high stability due to their low Gibbs free energy. By considering the content of oxynitrides and high-entropy ceramics, a narrow bandgap high-entropy oxynitride (HEON) was synthesized and examined for photocatalytic hydrogen evolution. The xynitride (TiZrHfNbTaO₆N₃) with two FCC and monoclinic phases was synthesized by high-presure torsion and high-temperature oxidation in air and nitriding in ammonia. This material demonstrated a higher light absorbance compared with related binary and high-entropy oxides and could produce photocurrent and hydrogen under UV irradiation with high chemical stability.



Fig. 1 Enhanced light absorbance and photocatalytic H_2 production of TiZrHfNbTaO₆N₃. (a) Uv-vis spectrum of HEON compared to relevant high-entropy and binary oxides and (b) photocatalytic H_2 production amount versus time for TiZrHfNbTaO₆N₃.

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Hard magnetic SmCo₅-Cu nanocomposites produced by high-pressure torsion

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ABSTRACT

High-pressure torsion (HPT) as a severe plastic deformation (SPD) process enables the production of nanocomposite materials. When applied to powder blends of permanent magnetic phases like $SmCo_5$ and binder metallic phases such as Cu, it opens up a new route for the production of novel magnetic materials. HPT of powder blends thus enables a nearly free selection of the magnetic phase and the grain boundary phase, beyond the limitation of conventional powder metallurgical sintering routes.

In this work, HPT is applied for the first time on a powder blend consisting of SmCo₅ as hard magnetic phase and copper as grain boundary phase. The ductile copper powder serves as a binder phase, carrying the plastic deformation and furthermore it decouples the hard magnetic SmCo₅ grains due to its diamagnetic behaviour. For the production of nanocomposites, different mixing ratios between the two powders and a variation of the process parameters was investigated. Therefore, a two-step HPT process was applied. First the powder is precompressed with a pressure of 4.5 GPa and in the second step the actual deformation is done. After a process optimization, a consolidated disc with a nanosized microstructure and excellent magnetic properties with a coercivity of 1.4 T could be obtained in an as-HPT sample. After 20 HPT turns, a two phase microstructure, with strong refinement in particle size of SmCo₅ down to the single domain size was achieved. The plastic deformation results therefore in a grain refinement while at the same time the individual particles are well surround by Cu leading to magnetic decoupling of the SmCo₅ grains. Beside the magnetic properties also a texture analysis by XRD was done showing a deformation induced evolution of texture.

The results emphasize the wide freedom of microstructural design for magnetic materials using HPT of powder blends, which allows to tailor intrinsic properties via the material selection as well as extrinsic properties via the processing parameters.

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High-pressure columbite as an active photocatalyst: Ab initio calculations and experiments by high-pressure torsion

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ABSTRACT

Fossil fuels are one of the major sources of CO_2 emissions worldwide. Therefore, hydrogen has been demonstrated to be a clean alternative fuel. The clean production of hydrogen can be performed by photocatalysis and among all photocatalysts, TiO₂ is one of the most promising. However, the activity of TiO₂ is still limited for practical applications due to its wide bandgap. Columbite is a TiO₂ high-pressure phase that has shown good photocatalytic activity after being stabilized by high-pressure torsion (HPT)^{1,2}. The current research aimed to clarify the mechanisms of high photocatalytic activity of columbite utilizing ab initio calculations in the flavor of density functional theory (DFT) and experiments. Three possible pathways of high photocatalytic activity were theoretically investigated: (1) narrow electronic bandgap of high-pressure phase which was proved not to be correct, (2) decreasing optical bandgap by oxygen vacancies which was proved to be significant for columbite, and (3) low surface activation energy for water splitting on columbite, which was confirmed to be valid. Additionally, experiments were conducted using the HPT method to stabilize the columbite phase and validate the results of the calculations. It was shown that the activity of columbite is higher than anatase by a factor of four. In conclusion, columbite is not a low bandgap material, but there is a significant influence of oxygen vacancies in its bandgap narrowing and high surface photocatalytic activity.



Fig. 1. Energy vs reaction coordinate for water splitting on the columbite (101) surface.

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Enhanced hydrogen storage in Mg catalysed by CuNiCoFe_{0.9}Pd_{0.1} multi-component alloy

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ABSTRACT

Magnesium has been sought after material for hydrogen storage due to its high storage capacity (7.6 wt.%) and low cost combined with easy availability. However, sluggish kinetics and high absorption temperature (>300°C) make it inherently difficult to use in commercial applications such as automobile, which require hydrogen absorption/desorption near ambient temperature and pressure. In this work, multi-component transition metal alloy with the composition CuNiCoFe_{0.9}Pd_{0.1} has been synthesized by ball milling process and subsequently added as a catalyst to magnesium for hydrogenation. The selection of multi-component transition metal alloy CuNiCoFe_{0.9}Pd_{0.1} as a catalyst presents unique properties with each element possesses an effective capacity in bringing down the diffusion as well as dissociation energy barrier for formation of magnesium hydride. Hydrogen absorption capacity has been determined using custom built Sievert's apparatus. The hybrid has been characterized for hydrogen desorption temperature using Thermo-Gravimetric Analysis. The phase formation and composition analysis has been carried out using X-Ray Diffraction and Scanning Electron Microscopy, respectively. Synergistic effect of multiple transition metals in an alloy result in reduction in hydrogen desorption temperature, without compromising on the hydrogen storage capacity of the hybrid. Nanostructured Mg-based hybrids provide an economic and industrially scalable route for the commercial implementation of hydrogen-fuel cell combined system.

Keywords: Hydrogen Storage, Magnesium Hydride, Ball-milling, reduced graphene oxide, Multi-component alloy

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Influence of severe plastic deformation on the magnetic properties of SmCo permanent magnets

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ABSTRACT

The increasing global energy consumption over the last decades and the recent tendency to raise the amount of renewable energies is associated with a rising demand for high performance permanent magnets (PMs). State of the art PMs, like NdFeB, are based on combinations of rare earth and transition metals. A promising candidate for applications under tough working conditions are SmCo based PMs due to their high maximum operating temperature and enhanced corrosion resistance.

Conventionall PMs are produced using powder metallurgical techniques including powder refinement via milling, compaction of powder blends with simultaneous magnetic field alignment as well as subsequent sintering and annealing processes. To prevent oxidation, the sintering and annealing processes are performed in protective atmospheres ^[1].

In this study a novel approach to process SmCo based PMs is presented by using high pressure torsion (HPT), a method of severe plastic deformation. Compaction of the powder blends, texturing and grain refinement can be obtained simultaneously by HPT deformation. To prevent oxidation, SmCo powders are compacted in Ar atmosphere and subsequently HPT deformed.. Microstructures down to the nanoscale range are formed, depending on the respective deformation parameters (e.g. deformation temperature, applied strain). The formed microstructures are characterized using scanning electron microscopy (SEM), X-ray scattering techniques as well as SQUID magnetometry to measure the magnetic properties, which are correlated to the emerging microstructures. The results indicate a strong correlation between magnetic properties and the applied deformation parameters. As it is exemplary illustrated in Fig. 1(a) - (d), the microstructural refinement, and therewith the magnetic properties (Fig. 1(e)), are strongly affected by the chosen deformation temperature. Herein, we present the deformation behavior of intermetallic SmCo-phases (e.g. SmCo₅, Sm₂Co₁₇) and studied the influence of subsequent annealing treatments on the magnetic performance of the obtained PMs.



Fig. 1: SEM images of the formed microstructures of Sm_2Co_{17} after (a) powder compaction as well as 20 revolutions at (b) room temperature, (c) 250°C and (d) 400°C. (e) The results of the corresponding SQUID measurements indicate a strong influence of the formed microstructure on the magnetic properties.

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Study of the Deformation Behavior, Structure Formation, and Properties of Titanium Nickelide Subjected to Severe Plastic Torsion Deformation

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ABSTRACT

NiTi-based shape memory alloys are attractive functional materials that are widely used in different fields of engineering and medicine for production of various shape-memory devices. The severe plastic deformation (SPD) is one of the most effective methods for the formation of an ultrafine-graned structure (UFG) in NiTi SMA which is characterized by the best combination of functional properties. Development of SPD methods is concluded in a searching for thermomechanical procedures which allow producing bulk samples with a nanocrystalline structure.

The goal of this work is to evaluate the possibility of applying the torsional deformation to bulk NiTi samples at low temperatures (in the range of dynamic polygonization) to accumulate high strains and refine the structure.

In temperature range from 300 to 600 °C torsional deformation was applied for the first time. Torsional deformation at 350-600 °C results in accumulation of high strains (e > 4) without fracture. The deformation at 300 °C leads to a brittle fracture at the true strain of e = 0.2. Flow curves obtained by torsion at temperatures above 350 °C are characterized by the steady state appearance and fracture at much higher strains (e > 4).

Transmission and scanning electron microscopy, X-ray diffractometry, differential scanning calorimetry, light microscopy; mechanical and special (functional) thermomechanical tests were used to study the structure and properties of the NiTi samples.

Severe torsional deformation at 350-600 °C leads to the accumulation of crystal lattice defects and increase in mechanical and functional properties as compared to the reference treatment. The phase composition at room temperature after deformation by torsion does not change qualitatively and consists of B19'martensite. The reverse martensitic transformation temperatures A_s and A_f after torsional deformation at 350-600 °C remain stable and do not reduce by more than 20 °C.

Acknowledgments

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Control of Mechanical Properties in Commercially Pure Aluminum by a newly developed Severe Shear Plastic Deformation Technique

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Abstract

Commercially Pure (CP) Aluminum sheets were subjected to 75% reduction in thickness through a newly developed multi-pass Severe Shear Plastic Deformation technique. The processing was carried out at different strain rates varying from 0.2 s^{-1} to 30 s^{-1} . A three-fold increase in strength was obtained when the sample was processed at a low strain rate i.e., 0.2 s^{-1} . With the increase of strain rate, for different samples, an increase in ductility (from 20% to 33%) has been found with a gradual drop in strength (from 180MPa to 100MPa). Microstructural investigations show distinct "deformation bands" with a strong texture that is attributed to enormous grain refinement with the change of strain path. The samples subjected to lower strain rates displayed sub-micron size grains i.e., less than 500nm. The stress-strain response of the material can be controlled by the processing parameters and can be produced in a single step.

Keywords: Severe plastic deformation; mechanical properties, deformation bands; strain rate; commercially pure aluminum.

Induction of magnetic saturation in the non-magnetic austenitic stainless steels by trapezoidal notch wavy rolling SPD

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ABSTRACT

Steels are known as a family of universally stronger materials for structural, mechanical, and functional applications. However, the drawback of rusting (corrosion) has attracted many innovations to introduce and promote new grades and applications of steel. Among them, austenitic stability at room temperature is observed as renowned metallurgical evolution in stainless steels (non-corrosive). Thereby, the high corrosion resistance austenitic stainless steels have come to the frontline for a range of mechanical and civil applications with their high degree of castability, weldability, and formability. To increase their scope further in the field of magnetic core materials by replacement of electrical steel (ferritic, hard-magnetic, corrosive), the type 304 austenitic stainless steel, even non-magnetic at room temperature, was uniquely processed to induce soft magnetic nature in flat products that could be suitable for a wide range of electrical and electronic devices. For this investigation, thin (<1mm) sheets of type 304 stainless steel were fabricated so as to possess nano-wide deformation bands across the individual austenite grains by trapezoidal notch wavy rolling (TNWR) severe plastic deformation process.

The final grain refinement occurred by grain fragmentation at intersecting nano/micro-shear bands. As a result of severe plastic deformation and the associated grain refinement, the saturation magnetization was found to be quite high (>0.5T) as compared to that obtained upon the conventional cold rolling of 50% reduction in thickness (<0.5T). Other engineering magnetic properties of TNWR sheets like coercive force, remanent magnetization, permeability, and BHmax (hysteresis losses) were also studied and compared with the results of 50% cold rolled sheets.

Furthermore, the TNWR process at room temperature caused strengthening by strain hardening without changing the shape and size (reducing thickness) of the original sheet. Such an effect of severe plastic deformation was explained by microstructural and crystallographic changes obtained by optical/atomic force microscope (AFM) and XRD techniques.

Keywords: Austenitic stainless steel; magnetic properties; wavy rolling; severe plastic deformation; grain refinement

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Structure refinement and property evolution of the Ti-3Al-2.5V alloy during tube producing by the TREX technology

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ABSTRACT

Ti-3Al-2.5V is intermediate in strength between unalloyed titanium and Ti-6Al-4V. The alloy exhibits good corrosion resistance and is used extensively for producing seamless cold-worked tubes operating in hydraulic and fuel systems of aircraft and rockets.

The evolution of the structure and properties of the Ti-3Al-2.5V alloy during tube producing by the TREX technology using the methods of optical microscopy, REM, durometry, mechanical compression and tension tests was studied. The correlation between the structure and hardness over the cross section of the hot-deformed tube billet was established. It was shown that the structure over the billet section is comparatively homogeneous, the average β -grain size varies from about 600 microns in the center to 300 microns closer to the edge of the billet, and the hardness respectively increases from center to edge.

The evolution of the structure and its correlation with hardness, mechanical properties of hot-extruded tube was studied. It was shown that temperature of the tubes during hot extrusion did not undergo to a β -phase field. Also the hot extrusion at the recommended temperature range provided the complex of mechanical properties required for the subsequent cold rolling used at the final stages of TREX tube production.

The annealing after hot-extrusion led to recrystallization of α -phase and formation of more equilibrium $\alpha+\beta$ -state. These reduced the Vickers hardness deviation across the cross section of the hot-extruded tube and obtained a complex of mechanical properties with high ductility and medium strength.

The influence of the degree of cold deformation on the formation and changes of the mechanical properties of a hot-deformed alloy was evaluated. The optimal degrees of deformation for cold rolling were determined. It was shown that intermediate/final vacuum annealing activated the process of recrystallization of the α -phase. This helped to obtain the structure of final tube with 10 µm average grain size.

The effect of cold deformation and vacuum annealing on the formation of texture and properties of tubes of Ti-3Al-2.5V alloy was established. It is shown that cold rolling led to the formation of a circumferential texture {0001}TD<10-10>RD, which changed to {0001}TD<11-20>RD (TD – tangential direction, RD – rolling direction) during vacuum annealing. The final tube size 38x5 mm, the mechanical properties – TS \approx 690-700 MPa, YS \approx 550-570 MPa, $\delta \approx 23$ -29 %.

Acknowledgments

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Simultaneous improvement in Strength and Ductility in deformed pure magnesium

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ABSTRACT

Magnesium being the lightest structural metal (density 1.73g/cc), is an attractive option for weight saving applications. However, due to low strength and limited formability, its usage is limited. Hence to enhance the properties, severe plastic deformation of magnesium was carried out to modify the texture and grain size. The deformed pure magnesium samples were tested in tension at room temperature. Simultaneous improvement in strength and ductility was observed. As compared to the as-received sample, yield strength and ductility increased by 200% and 37% respectively. On examining the fracture surfaces after the tensile test, a change in the mode of fracture was observed. The mode changed from brittle cleavage fracture (as-received) to mixed (dimples + cleavage) fracture for the deformed sample. The increased toughness and observation of ductile features in the fractographs, indicates enhanced dislocation activity possibly due to activation of non-basal slip systems. The obtained results will be correlated to the grain size and texture through EBSD and XRD analysis.

Recent Advancements in Constrained Groove Pressing Process

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ABSTRACT

Among various severe plastic deformation (SPD) techniques, constrained groove pressing (CGP) has arisen as a famous technique for fabricating ultrafine-grained sheet metals and alloys in recent times. The severe strain is applied to improve the mechanical performance and properties. However, inhomogeneous grain refinement, lower formability, surface unevenness, and microcracks are the common limitations of conventional CGP-processed samples. Therefore, the latest progress to overcome such limitations and comparison of process efficacy with the conventional process will be highlighted through various advanced CGP processing techniques, such as modification of die design, annealing of the processed sample, cross CGP, ring CGP, hybrid SPD, covered sheet casing CGP, and rubber pad CGP. All techniques are emerging to upscale the procedure and improve grain refinement through various approaches, producing superior mechanical properties. The historic research of conventional CGP and its effect on grain refinement and mechanical properties will be initially briefed and then recent progress through different advanced techniques will be highlighted, especially in alloys.

Revisiting the Classical Laws for Nanomechanics

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ABSTRACT

A unifying framework is presented for revisiting the classical laws of mechanics: Newton, Hooke, and Fick, along with the classical interatomic potentials: van der Waals, London, and Lennard-Jones. The implications to the field of nanomechanics, nanomaterials and next-generation battery systems are outlined. Specific examples in pattern forming instabilities and size effects are discussed.

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In vivo osseointegration of nanotitanium implants

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ABSTRACT

High-strength commercially pure nanostructured Ti Grade 4 (nanotitanium) is very promising for manufacturing medical implants with improved design [1]. In this work the osseointegration properties of nanostructured (NS) and initial coarse-grained (CG) Ti Grade 4 with different surface treatments were studied: 1 - NS after mechanical polishing; 2 - NS after mechanical polishing and subsequent chemical etching; 3 - CG after mechanical polishing; 4 - CG after polishing and etching. Chemical etching of the samples was performed in ammonia piranha solution with an exposure time of 2 hours. Roughness parameters were measured for each sample type using an NT-MDT Integra Prima scanning probe microscope.

Biological tests for osseointegration properties were performed on 42 Wistar rats. The implants 10 mm in length and 1 mm in diameter were introduced into the distal third of the femur in the marrowy canal for 8 weeks. Morphological analysis of the tissue structures on the bone-implant boundary line was performed using a scanning electron microscope. The formation of bone tissue was revealed to be most rapid in the test subjects where the second group of implants was examined (Fig. 1). In this group, there was observed a newly formed trabecular bone tissue on the boundary line of the implant and bone in the volume of over 71.2 ± 12.4 SD, which was significantly higher than the volume in the group 1, where its indicator was less than $62.4\% \pm 8.3$ SD at the p < 0.05 significance level. The volume of trabecular bone tissue in the observations of groups 3 and 4 was also significantly lower compared to group 2 with mean values of 45.8 ± 6.5 SD and significance level p < 0.01.

The studies have shown that the nanostructuring and mode of surface treatment of Ti Grade 4 considerably influence the processes of osseointegration of implants on the boundary line of the implant and bone, which has important clinical significance.

Figure 1 - Influence of implant type on the tissue volume

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Investigations on Performance of EDM by Modified Copper Electrodes with Relief Angle and Corner Radius

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ABSTRACT

Electrical Discharge Machining is a process used to remove or cut material through the action of spark discharge between the electrode and work piece. The objective of this paper is to study and analyze the effect of relief angles and corner radius of electrode on the performance of EDM process. A conventional cylindrical electrodes, Electrode with relief angle 10° and land 1.0 mm, Electrode with relief angle 10° and land 1.5 mm, electrode with relief angle 10°, land 1.0 mm and corner radius 1.0 mm and Electrode with relief angle 10°, land 1.5 mm and corner radius 1.0 mm are used to perform on two types of work pieces (SS316 and EN31) to make through holes of dia 6 mm and depth of 6 mm. A total of fifteen holes are machined with three sets of input parameters and five types of electrodes on each work piece. The output parameters viz. machining time, radial over cut (ROC), material removal rate (MRR), tool wear rate (TWR) and surface roughness (SR) are measured for each electrode and for each work piece.

From the results, the majority output parameters have arrived with best values while performing with electrode type with relief angle, land and corner radius (D & E). Hence, it is concluded that, the performance of electrode type D & E is the best. The common features of relief angle, land and corner radius of these two designs are influenced the process for better performance.

Keywords: Electrical discharge machining, Copper electrode, SS316, EN31, Corner radius, Relief angle.

Effect of High-Pressure Torsion on Mechanical and Corrosion Properties of a Biomedical Mg-6Zn-0.2Ce Alloy

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ABSTRACT

Magnesium (Mg) has enormous potential for developing bioresorbable implants, especially implants to repair bone tissues. However, low strength and high corrosion rate limit its clinical application. The addition of zinc (Zn) increases corrosion resistance, and cerium (Ce) can impart anti-cancer, anti-inflammatory, and anti-bacterial activities to the material [1,2]. Here, we investigate the effect of high-pressure torsion (HPT), a severe plastic deformation (SPD) process, on the mechanical properties and corrosion of Mg-6Zn-0.2Ce alloy. This alloy was processed by quasi-constrained HPT under the applied pressure of 6 GPa at room temperature for 1, 2, and 5 turns. The hardness value at the center increases with the number of HPT turns. However, the hardness value at the edge gets saturated after one turn. There was a significant increase in yield strength and ultimate tensile strength, along with a reduction in percentage elongation to failure with HPT turns. A potentiodynamic polarization test was performed in a stimulated bodily fluid (SBF) solution. The two turn HPT samples exhibited the highest corrosion resistance. The increment in corrosion resistance can be attributed to the combined effect of the formation of basal texture, which has low surface energy, and the increase in the relative fraction of stable magnesium oxide to magnesium hydroxide [3,4]. Overall, HPT for two turns exhibits increased strength and decreased corrosion rate of the Mg-alloy, improving its suitability for bioresorbable implants.

Keywords: High-pressure torsion, Corrosion, Magnesium alloy, Potentiodynamic polarization, Bioresorbable.

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Effect of high pressure torsion on the microstructure, mechanical, corrosion and biological properties of β Ti-34Nb-3Zr-2Ta alloy for orthopedic implant application

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ABSTRACT

Ti-6Al-4V ($\alpha\beta$) alloy has been the most widely used material as a femoral stem in total hip replacement. However, recently this alloy has been under severe scrutiny due to the toxicity of Al and V. In addition, though the elastic modulus (~110 GPa) is less than CoCrMo (~230 GPa) and SS316L (~250 GPa), it is still three times higher than cortical bone (4-30 GPa) which leads to stress shielding effect. To tackle the above issues, β phase Ti alloys have been introduced and considered to be the potential candidate to replace a Ti-6Al-4V alloy due to their lower elastic modulus and non-toxic nature of the β stabilizing elements (Nb, Zr, Mo, Ta). Among the β phase Ti alloys, Ti-Nb-Zr-Ta (TNZT) alloy has shown promising results in biocompatibility and also possesses lower elastic modulus (50-60 GPa). So far, TNZT alloys have been processed through conventional methods such as as-casting, rolling, forging, and subsequent aging treatment to improve their strength. However, the strength of TNZT processed through the conventional route is slightly less than that of Ti-6Al-4V alloy. Further, age hardening also increases elastic modulus, which is undesirable. Severe plastic deformation is one of the effective ways to enhance strength without increasing the elastic modulus. Also, it is worth mentioning that reports on the effect of severe plastic deformation (SPD) on the structure-property relationship of TNZT alloy are scarce. Notably, the studies on corrosion resistance and biocompatibility of SPD processed β phase Ti alloys are very limited. So in the present work, we aim to investigate the effect of high pressure torsion (HPT) on the microstructure, mechanical, corrosion and biocompatibility of β Ti-34Nb-3Zr-2Ta alloy. The Ti-34Nb-3Zr-2Ta alloy was cast by vacuum arc melting using a tungsten electrode arc furnace. Then the as-cast alloy was subjected to solution treatment (ST) at 1000°C for 2 hours and followed by air quenching. The ST sample was then cut into coin-shaped dimension with 10 mm dia and 0.8 mm thickness and followed by an HPT process in which the lower anvil rotation and applied pressure was fixed at 1 rpm and 6 GPa, respectively, and the rotation number (turns T) was varied from 2T, 4T, and 6T. After HPT, the microstructure of each sample was initially analyzed by optical microscope (OM), scanning electron microscope (SEM) and transmission electron microscope (TEM). The crystallographic orientation and bulk texture analysis of the HPT sample were performed using electron backscattered diffraction (EBSD) and XRD, respectively. The mechanical properties, such as microhardness and tensile tests, were executed. The corrosion resistance of the HPT specimens were evaluated by performing open circuit potential, electrochemical impedance spectroscopy and potentiodynamic polarization using a potentiostat. Finally, the biocompatibility test, such as cytotoxicity, cell attachment and proliferation, were studied.

Phase stability of Al_xCoCrNi medium entropy alloys processed by high pressure torsion

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Abstract

Medium Entropy Alloys (MEAs) contain three or four elements in equiatomic or near equiatomic ratio such that the configurational entropy lies between 1.0R and 1.5R (R is universal gas constant). The multi-element matrix of MEAs lends them exciting properties and intriguing fundamental behaviour. CoCrNi MEA has shown excellent mechanical properties both in coarse as well as nanocrystalline state. The addition of Al is done to enhance the oxidation resistance by formation of passive Al₂O₃, which is further promoted by presence of Cr. In the present work, $Al_xCoCrNi$ (x= 0, 0.1 and 0.2) MEAs in coarse-grained form are processed by arc melting followed by homogenization at 1100 °C for 24 h. A single-phase FCC structure is observed for all the three alloys in coarse grained as well as nanocrystalline state. Nanocrystalline Al_xCoCrNi alloys are prepared by high pressure torsion (HPT) technique using 6GPa pressure and 10 turns. Thermal stability of bulk and nanocrystalline MEAs are examined through DSC/TGA measurements and isothermal heat treatments. The disc-shaped samples of as-homogenized and HPT alloys are subjected to annealing at temperatures 700, 800, 900 and 1000 C for 100 h to assess the microstructural evolution. X-ray diffraction (XRD), electron microscopy and electron back scattered diffraction (EBSD) are the major tools used for the characterization of as-processed and heat-treated alloy samples. Vicker's hardness measurements have also been performed on as-homogenized and HPT AlxCoCrNi MEAs to determine the influence of composition and grain size.

X-ray line profile analysis of nanostructured Cu-Al and Cu-Al-Co alloys processed through high-pressure torsion

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ABSTRACT

Cu-10%Al and Cu-10%Al-5%Co alloys were prepared using a vacuum arc melting furnace. Both the alloys were isothermally annealed at 1000 °C for 4 hours for homogenisation and solutionizing treatment. Microstructural analysis of Cu-10%Al has shown a single-phase FCC matrix, whereas Cu-10%Al-5%Co showed nanocrystalline precipitates of Co in the FCC Cu matrix. The diffraction pattern of precipitated region confirms that the Co precipitate is also FCC in nature. Further, ageing treatments were given to the Cu-10%Al-5%Co alloy at 500 °C for 7 hours. It resulted in the formation of fine nanocrystalline precipitates rich in Co and Al. The diffraction pattern of those precipitates showed superlattice spots and it confirms the formation of L1₂ Co₃Al precipitate.



Fig. 1: Microstructure and diffraction pattern of solution treated (a) Cu-10%Al, (b) Cu-10%Al-5%Co, (c) solutionized and aged Cu-10%Al-5%Co showing superlattice reflections.

All these materials were cut in the shape of 10 mm diameter discs and subjected to high pressure torsion under a load of 6 GPa for 1, 2, and 5 rotations, respectively. The effect of strain and both types of precipitates on the microstructural evolution such as crystallite size and dislocation density accumulation were investigated. To do so, highresolution X-ray diffraction patterns were recorded in each case on the centre (C), between centre and periphery (CP), and on the periphery (P) of the sample. Cu-10%Al showed a continuous reduction in the crystallite size and increase in dislocation density with an increase in torsional strain, till 2 rotations and a similar trend on moving from centre towards the periphery of the sample. After 5 rotations, it showed saturation and a slight increase in the crystallite size, which might be because of the coalescence of nanocrystalline Cu grain at very high strain level. Both cases of Cu-10%Al-5%Co (solutionized and solutionized + aged) samples showed early nano-crystallization compared to Cu-10%Al in terms of strain level. The saturated crystallite size was also found to be 30% lesser than Cu-Al alloy. Apart from that, no increase in crystallite size could be seen at very high strain level. It confirms the absence of grain coalescence mechanism due to the presence of either type of precipitate.

Microstructure, mechanical and surface characterization of SiO₂ and Al₂O₃ reinforced Al 6061 surface nanocomposites using friction stir processing

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ABSTRACT

The use of lightweight materials is preferred in the majority of industries including those that deal with aeronautics, automobiles, shipbuilding, etc. In order to achieve high strength for light weight materials different techniques are evolved, one being severe plastic deformation. Friction Stir Processing (FSP) is one of the widely used plastic deformation technique employed for development of surface composites. The present study aims to investigate the effects of FSP parameters on microstructure, mechanical properties and surface roughness values of SiO₂ and Al₂O₃ reinforced Al 6061 surface composites. In this work, SiO₂ and Al₂O₃ were used as a reinforcement particles (RPs) and Al 6061 was used as a base material. A groove was made to incorporate the RPs in the specimen of base material, Fig. 1. By altering the process parameters (rotational and traverse speed) and number of FSP passes, FSP was performed. A comparative study was done to analyze the microstructure, mechanical and surface characterization of the specimens. The results showed that the dispersion of RPs into the base metal resulted into the improvement of mechanical properties and well bonded interfaces were formed.

In SiO₂ reinforced Al 6061 nanocomposites, the ultimate tensile strength is higher in samples fabricated at 2000 rpm as compared to 1600 rpm. In each samples, the strength is higher in single pass FSP as compared to double pass. The lowest surface roughness value (Ra = $2.741 \mu m$) was found in sample fabricated at 1600rpm, 20mm/min, single pass, which is 61.3 % lower than sample fabricated at 2000 rpm, 20mm/min, single pass. In Al₂O₃ reinforced Al 6061 nanocomposites, sample fabricated with operational parameters of 2000 rpm, 30 mm/min, and double pass FSP, tensile strength is 1.24 times higher than the sample fabricated at 2000 rpm, 20 mm/min, and double pass. The low surface roughness value (Ra = $3.15 \mu m$) is found in sample fabricated with single pass as compared to double pass (Ra = $4.407 \mu m$). SiO₂ reinforced specimens exhibited better surface finish as compared to Al₂O₃ reinforced specimen. Also, fractography analysis is performed to understand the different failure modes of specimen during tensile testing using FESEM. Chemical analysis of composites is carried using EDS. The present work reports the effect of RPs for manufacturing the nanocomposites using FSP.



Fig.1. Schematic view of FSP: (a) Experimental set-up of FSP, (b) Groove machining (width=2.5mm, depth=2 mm), (c) RPs inserted into groove, (d) Groove closing using pin-less tool, (e) FSP operation using tool with pin.

A Comparative Study of Concurrent Microstructure Evolution and Mechanical Properties in Friction Stir Processing and High Pressure Torsion of Al-Si Alloys

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ABSTRACT

A comparative study of friction stir processing (FSP) and high pressure torsion (HPT) processing was conducted to evaluate microstructure evolution and its effect on mechanical properties in Al-Si binary alloys of varying silicon contents, representing from hypoeutectic to hypereutectic compositions including the eutectic composition, as a function of process variables. Microstructure examination in both the processes, as compared to the as-cast condition, exhibited redistribution of α -Al, Al-Si eutectic and primary Si phases towards a homogeneous structure along with overall grain refinement. However, the nature of progress in microstructure evolution in these two cases was noted to be substantially different, which varied as a function of process parameters and alloy compositions. The variation in microstructure and its effect on hardness were measured from the center to the end point of contact between the tool and specimen. The diametrical increment in hardness is explained by grain refinement and dispersion of Si particles. Also, found is a variation in elongation with a positive contribution from FSP but not so promising in the case of HPT. The variation in mechanical properties is explained quantitatively with the local variation in microstructure, wherein the phase proportion, grain size, particle size and morphology play important roles.

The observed results on microstructure evolution and mechanical properties in FSP and HPT were used to develop physical models that distinguish the nature and kinetics of strengthening and deformability of the samples subjected to these processes.

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Phase transformation microstructure evolution texture characteristics and mechanical properties of multi-pass friction stir processed AA7075T7352

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Abstract

Microstructure, mechanical properties, and texture characteristics of the multi-pass (upto-3pass) friction stir processed 7075 aluminum alloy are systematically studied in the current work. Phases are analyzed with the help of x-ray diffraction (XRD), and transmission electron microscope (TEM). XRD analysis display dissolution of the GPzone, and formation of the new inter-metallic (Al₅₀Mg₄₈Cu₂) phase after 1-pass FSP, whereas GPzone and n' phases are also dissolved after 2pass FSP. On the other hand, re-appearance of the GPzone, and η' precipitates including the intermetallic of Al₂Cu (θ) are noticed after 3-pass FSP. Such an observations are got corroborated to the enhanced mechanical performance of alloy. Bulk texture characteristics (ODF) display evolution of increased intensity of Goss {011} <100> re-crystallization texture component after subsequent passes, which creates large twist angle grain boundary at the adjacent grain. Residual stresses are measured using Panalytical Empyrean MRD system (HR-XRD) that display compressive stress of -78±3 MPa, in the as-received state, -163±5 MPa, in 1-pass FSP, which further reduced to -175±8 MPa, and 203±7 MPa after 2-pass, and 3-pass FSP respectively. Surface behavior is studied with the help of scanning probe microscope(SPM), which shows high surfaceroughness (av.~12.5nm) value in the as-received state, that reduced to 7.5 nm, and 1.8nm after 1pass, and 2-pass FSP respectively, thereafter further increases upto 7.8 nm, after 3-pass FSP. Such a compressive residual stress and improved surface performance are also pertaining to the enhanced mechanical and stress corrosion cracking performance of processed alloy. Micro texture characteristics are studied with the help of electron back-scattered diffraction (EBSD), which display 10 fold decrease of grain size, from $54\pm3.0 \,\mu\text{m}$, in the as-received state to $5.0\pm1.0 \,\mu\text{m}$, 3.0±0.8 µm, and 1.5±0.5 µm, after 1-pass, 2-pass, and 3-pass FSP respectively in the nugget zone of the processed specimens. High kernel average mis-orientation (KAM) value after increasing the FSP-pass display high strain localization within the region. TEM micrograph display GPzone, n' and η precipitates randomly distributed throughout the α -Al matrix, in the as-received state. Recovered microstructure, dynamic re-crystallization, dissolution of the GPzones are noticed after 1pass, whereas n' phase, including GPzones are dissolved after 2-pass. On the other hand, dynamic re-precipitation of small GPzones, and n' are noticed after 3-pass FSP. Such a precipitate formation and structural evolution are attributed to separated-nucleation, and growth mechanisms.

Keywords: Aluminum alloy, Dynamic-recrystallization, Separated nucleation, Micro-texture, Frictions stir processing, Electron back-scattered diffraction.

Effect of friction stir processing on microstructural and damping properties of Al6061/BN surface composites

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ABSTRACT

Friction stir processing (FSP) parameters are the most important factor in producing surface composites. In the present study, FSP was employed to produce surface composites on Al6061T6 alloy by incorporating 15% volume fraction of Boron Nitride (BN) particles. The surface composites were produced at 40, 80, 120, and 160 mm/min traverse speeds. and constant rotational speed of 1200rpm. Optical microscopy (OM) and Scanning electron microscopy (SEM) was used to find the distinct zones in the stir zone and the distribution of BN particles. The results showed that at low traverse speed causes the uniform distribution of BN particles. Energy dispersive spectroscopy (EDS) is used to confirm the presence of BN particles in the samples. The micro-hardness (Hv) is also measured. The hardness of the samples increased from 69 to 90.2Hv, and the minimum grain size was 5.1µm. The damping capacity of Al composites was analysed by a dynamic mechanical analyser (DMA) from room temperature to 300°C. The FSPed composites showed better damping capacity than base metal and damping peaks are observed for FSPed composites, which are attributed to the grain boundary sliding. The highest damping capacity was observed at high traverse speed (120 mm/min.) mainly because of its fine grain structure.

Keywords: friction stir processing, Al composites, damping properties, dynamic mechanical analysis, grain refinement.

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Microstrucural characterization of friction stir welded Al-Zn-Mg cast alloy for Automotive Applications

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ABSTRACT

Nowadays, there is a global need to develop next-generation lightweight materials for automotive components. Al-Zn-Mg cast alloys can be an excellent candidate to fulfill this demand. Utilization of an effective joining technique will increase its scope in automobiles. In this work, friction stir welding (FSW), which is an established solid-state joining technique for Al alloys, was used, and the microstructure evolution was thoroughly studied. FSW parameter optimization was carried out through detailed microstructural characterization and mechanical property evaluation of plates welded with different parameters (tool rotation and traverse speed). The microstructure of this new generation Al-Zn-Mg cast alloy was modified significantly during friction stir welding. The intermetallic phases present along the grain boundaries in the as-cast Al-Zn-Mg alloy are refined to the nano-scale and distributed uniformly in the welded region after friction stir welding. The grain size of the alloy was also refined significantly in the stir zone. A continuous type dynamic recrystallization (CDRX) process seems to have occurred in the microstructural evolution during FSW. The material is subjected to severe plastic deformation at elevated temperature during FSW, and it leads to dislocation generation, accumulation, and arrangement into low-energy configuration during the subsequent stages of dynamic recrystallization. These factors lead to a significant strengthening of the weld zone. The temperature profile across various locations was also measured during the FSW process to gain more insight into the thermomechanical process and the consequent microstructure development. Microstructural evolution along different weld zones during FSW for selected FSW parameters was studied through electron back-scattered diffraction (EBSD) and transmission electron microscopy (TEM). Nano-scale intermetallics were characterized thoroughly through scanning transmission electron microscope (STEM) analysis in different modes. Some preliminary studies on the effect of FSW on the precipitation behavior of the alloy were also carried out.

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Work hardening and wear behavior of Friction stir processed Ni-Al bronze (NAB) alloy Shivraman Thapliyal¹, Dheerendra Kumar Dwivedi^{2*}

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Abstract

The microstructure of surface and near surface layer of functional surfaces affects the mechanical properties and wear-behavior. Attempts have been made in this work to modify the microstructure of surface and near surface layers using solid state surface modification approach namely friction stir processing (FSP) in order to improve the sliding wear resistance. In this work, effect of number of passes during friction stir process on microstructure, mechanical properties and sliding wear behavior Ni-Al bronze has been reported. Near surface microstructural modification of as-cast Ni-Al bronze alloy was carried out for by multi-pass friction stir processing. The 13 mm thick commercial UNS C95500 NAB alloy sand-cast plates were used in this study. The FSP was performed under a tool rotation rate of 1216RPM and a traverse speed of 26 mm min⁻¹ with a tool tilt angle of 2° using a flat shoulder threaded pin type tool. The sequence of the multi-pass for processing was one, two and four with 100 percent overlap. It was found that increase in the number of passes refines the grain structure of the ascast Ni-Al bronze. The minimum average grain size of 9.30 µm in stir zone (SZ) was observed for the four passes surface with a maximum hardness value of 344±5 HV in the stir zone. The adhesive type sliding wear behavior of the processed surface was assessed using pin-on-disc tribometer. The wear mechanism was established by SEM analysis of the worn surfaces and sub surfaces.

Keywords: Friction stir processing, Sliding wear, Delamination; Wear debris

Ultra-fine-grained medium Mn low Ni austenitic stainless steel by thermomechanical processing routes

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ABSTRACT

Coarse-grained austenitic stainless steels are limited in their use in structural applications due to their relatively low yield strength. One promising way to improve their strength is by refining the grain size by the technique of reversion of strain-induced martensite to austenite. In the present work, an ultrafine-grained structure is obtained in a 14wt.%Cr-8wt.%Mn-1wt.%Ni-0.08wt.%C metastable austenitic stainless steel through the process of reversion of strain-induced martensite. This steel was annealed at 1050 °C for 1 hour and rolled at room temperature up to an effective strain of 0.6. The fraction of martensite obtained was around 60%. The grain refinement was achieved via repetitive rolling and austenite reversion treatment, wherein the strain-induced martensite formed due to deformation was reverted to ultra-fine grains (UFG) of austenite upon heating. The microstructure obtained through different reversion temperatures and times will be presented and compared. Apart from the high-temperature reversion treatment, a thermal treatment at warm temperatures results in nano-sized precipitates in the martensite phase, thereby increasing the strength of the steel to beyond 2 GPa. A combination of rolling-aging-reversion treatment, to obtain the nano-sized precipitates as well as reverted UFG of austenite will be presented. The fraction of martensite is quantified using ferritoscope, the microstructure is characterized using electron back-scatter diffraction, and mechanical properties through hardness and tensile tests.
Role of severe plastic deformation on the electrochemical behavior of austenitic stainless steel

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ABSTRACT

Austenitic stainless steels, known for excellent formability and corrosion resistance, suffer from poor wear and localized corrosion resistance. Need of enhancing the localized corrosion resistance (i.e., pitting resistance) and wear resistance of stainless steels made researchers to develop several surface deformation techniques and thermochemical surface treatments.

During severe plastic deformation (SPD) of austenitic stainless steels, reduction in grain-size, enhanced defect concentration, and strain induced martensite development occurs. Although each of these microstructural modifications are expected to alter the corrosion resistance, the individual and combined roles of these on corrosion characteristics are not yet fully understood. Against this background we have employed surface deformation techniques on stainless steels in such a way that we produce only high density of dislocations in one case and in another case, strain induced martensite is produced together with dislocations generation. After producing such microstructures on surfaces of 316 stainless steel, their electrochemical characteristics are investigated. Dislocations together with martensite development resulted in enhanced corrosion resistance while no such clear trend was observed for the case of only martensite development without deforming the remaining austenite. While altering the surface energy, dislocations also alter the kinetic mechanisms due to enhanced diffusivity especially of the interstitial elements which promote or demote the equilibrium chemical segregation of elements to surfaces during electrochemical reaction. Additionally, the thermodynamic stabilities of martensite and austenite, and the volumetric mismatch between oxide film on martensite and austenite phase are utilized to explain the observed electrochemical characteristics.

After studying the role of deformation-induced martensite and defects in austenite on electrochemical characteristics, the role of alloying the surface with N on the electrochemical characteristics of steels is investigated. The thermodynamic stability of N-alloyed austenite and martensite seems to have a decisive role on the corrosion characteristics of N-alloyed stainless steels.

Keywords: austenitic stainless steel; martensite; localized corrosion, severe plastic deformation; ultrasonic shot peening; cryo-rolling; dislocations; N-alloying

Microstructure, Texture, and mechanical property correlation in cold rolled DP-steel during inter-critical annealing

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ABSTRACT

Dual-phase (DP) steels are a potential candidate for automobiles owing to their high tensile strength. In this work, cold rolled DP steel sheets were subjected to inter and below-critical annealing treatment for different temperatures and time. The microstructure and texture evolution were studied using the electron back-scattered diffraction (EBSD) technique in a scanning electron microscope (SEM). X-ray line profile analyses were done to calculate dislocation density and crystallite size. Mechanical properties and strain-hardening behavior were obtained by tensile testing as per ASTM standards. The ferrite grain size, martensite island size and volume fraction, and fractography features were also obtained. The microstructure features and crystallographic texture were correlated with the mechanical behavior.

Keywords: Dual-phase steel, Thermomechanical processing, Mechanical characterization, strain hardening behavior,

Fractography

Effect of strain rate on the staircase-type strain hardening behavior in an ultra-fine-grained medium-Mn steel

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ABSTRACT

Due to their enhanced strength and ductility, Medium Mn steels are one of the most promising candidates among the third-generation AHSS for automotive applications. Their superior mechanical properties can be attributed to the presence of an extended strain-hardening regime. In the current work, we have designed and developed an ultra-fine-grained medium-Mn steel using the CALPHAD approach to optimize critical thermomechanical processing parameters to control austenite stability and stacking fault energy [1]. The developed steel shows excellent uniaxial tensile properties with UTS of ~1350 MPa and uniform elongation of ~26%. One of the interesting observations is the unique staircase-like feature and fine serrations exhibited throughout the plastic regime of the stress-strain curve. The staircase-like feature consists of a plateau with negligible strain hardening followed by an abrupt rise in the flow stress. Furthermore, these features become even more prominent at lower strain rates. The quantitative characterization of the microstructure evolution during the tensile deformation is crucial to understanding the strain-hardening behavior [2]. Hence, we have performed an in-situ tensile study coupled with high-energy synchrotron X-Ray diffraction to gain better insight into the microstructure, defect density, and phase evolution during the uniaxial deformation and the overall effect of strain rate on the same. Preliminary analysis indicates a direct correlation between the austenite/martensite volume fraction and the strain hardening behavior, with the staircase-like feature marked by a dip in the austenite fraction, indicating a major role of the TRIP effect.

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Nanostructure control for improving tensile properties of cost-effective AlCrFe₂Ni₂ high entropy alloy

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ABSTRACT

Eutectic high entropy alloys (EHEAs) with the lamellar arrangement of FCC and BCC phases have recently attracted considerable attention. EHEAs are shown to be amenable to microstructural tailoring for attaining superior mechanical properties. EHEAs such as AlCoCrFeNi_{2.1} shows excellent mechanical properties subjected to severe deformation by different thermo-mechanical processing (TMP) treatments. Unfortunately, costly alloying elements such as Co are prohibitive for engineering applications of these EHEAs. Therefore, designing cost-effective compositions is a pressing need for further developments of EHEAs for structural applications.

Towards this direction, A cobalt-free cost-effective AlCrFe₂Ni₂ HEA was investigated in the present work. The as-cast HEA revealed lamellar regions with FCC and B2 phases coexisting with fine regions showing the spinodal transformation. The HEA was subsequently processed by different TMP routes, namely severe cold-rolling (90% reduction in thickness) and moderate cryo-rolling, followed by annealing treatments for tailoring microstructure and mechanical properties. Cold-rolling (CR) and annealing (CR(90%)+800°C/1h) resulted in a microduplex structure with a markedly improved yield strength (YS) and appreciable ductility. Remarkably, moderate (65% reduction in thickness) cryo-rolling (CryoR) and annealing (CryoR(65%)+900°C/1h) resulted in a heterogeneous microstructure with superior strength-ductility synergy (Fig.1). The present results indicate the exciting possibility of developing cost-effective HEAs by tuning the underlying nanostructure. The properties of the present HEA could be further compared with different cobalt-free HEAs to establish the superiority of the present HEA for advanced structural applications.



Fig. 1: Key summary of the mechanical properties and associated microstructure.

Microstructure assisted strengthening of NiCoCr-based medium entropy alloys with the addition of Al and Ti

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ABSTRACT

Recent studies have shown that Medium Entropy Alloys(MEAs) exhibit various excellent properties such as high fracture toughness, good creep resistance, good corrosion resistance and a good tradeoff between strength and ductility at elevated temperatures[1]. Various mechanisms such as solid solution strengthening, grain boundary strengthening, dislocation and precipitation strengthening mechanisms are implemented to get the desired mechanical properties. However, the overall strengthening of an alloy through the cumulative effect of individual strengthening mechanisms is still a challenge to understand. In this work, we will evaluate the contribution of individual strengthening mechanisms towards overall strengthening using different thermomechanical processing conditions. The minor addition of Al and Ti into NiCoCr MEAs forms L12 precipitates which have a composition of (Ni, Co)₃(Al, Ti)[2]. The three compositions ((NiCoCr)₉₆Al₄, $(NiCoCr)_{96}Al_2Ti_2$, $(NiCoCr)_{96}Ti_4)$ have been selected to study the effect of composition on the solid solution and precipitation strengthening. In addition to that, severe plastic deformation modes such as Rolling and High-Pressure Torsion(HPT) are used to investigate the effect of deformation microstructure on overall strengthening. Different isothermal and/or isochronal annealing heat treatments of as-cast, rolled and HPT-deformed alloys are performed to estimate the contribution of different strengthening mechanisms. X-ray diffraction, Scanning electron microscopy (SEM) and Transmission electron microscopy(TEM) are used to characterize the microstructure. Micro-Vickers hardness technique is used to characterize the hardness evolution of the thermomechanical processed alloys. An attempt is made to understand the correlation between microstructure and mechanical properties of precipitate-strengthened MEAs.

Keywords: Medium Entropy Alloys, L1₂ precipitates, Transmission Electron Microscopy, Rolling, Annealing

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Structure-property correlations in fine grained medium entropy alloys prepared by mechanical alloying and spark plasma sintering

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ABSTRACT

Concentrated refractory alloys are envisaged as the potential replacement for the current high temperature materials with enhanced performance. After the development of MoNbTaW refractory high-entropy alloy (RHEA), there has been a tremendous interest in these alloy systems among the scientific community. These alloys are manufactured mainly using the melting and casting route, which has a significant drawback of inhomogeneity in the microstructure and larger grain size (micron regime). An alternative methodology to develop these alloys to achieve homogenous microstructure and finer grain size is mechanical alloying, a severe plastic deformation (SPD) technique. Here in this work, two concentrated nanocrystalline ternary refractory alloys (MoNbTi and MoTaTi) are developed using a mechanical alloying route where a single BCC phase is achieved with a finer crystallite size of around 3 nm. Further, to restrict grain growth during consolidation, spark plasma sintering (SPS) is used. After SPS, a two-phase (BCC+FCC) structure is evolved with retention of finer grain size. These alloys were characterized using X-ray diffraction (XRD) and Field emission scanning electron microscopy (FESEM) techniques to correlate the phase evolution. Mechanical properties were determined using Vickers microindentation. This talk will address the phase formation, phase stability during high strain rate deformation during milling as well as subsequent exposure to high temperature during SPS. In the end, the strengthening characteristics and indentation size effect observed in these new alloys will be discussed.

Hot deformation behavior of CoCrFeMnNi High Entropy Alloy: Physical Simulation and Processing Map

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ABSTRACT

High entropy alloys (HEAs) are composed of at least five major elements, each having an equiatomic or near an equiatomic concentration of 5 to 35 atomic percent. The workability of materials that undergo hot deformation is usually reduced significantly due to sub-optimal strain rates and temperature conditions. This is commonly ascribed to the presence of some instabilities, such as the occurrence of cracks. Even though a broad spectrum of defects can affect hot workability during dynamic processing, the optimization of processing deformation conditions over a specific range of strain rates and temperatures could enhance the system's overall efficiency through safe deformation mechanisms, such as dynamic recovery (DRV) and dynamic recrystallization (DRX). Therefore, this work aims to optimize the workability of thermo-mechanically treated CoCrFeMnNi HEA by physical simulation using Gleeble 3800 thermo-mechanical simulator. The hot deformation associated with isothermal compression will be performed in the temperature range of 700-1000°C and the strain rate range of $10^{-3} - 10^1 \text{ s}^{-1}$. The microstructural evolution will be carried out using optical microscopy, electron microscopy, and electron back-scattered diffraction (EBSD) studies. Finally, the hot workability of CoCrFeMnNi HEA will be optimized to obtain a processing map based on the dynamic material model (DMM).

Acknowledgments

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The origins of ultrafine grain refinement during hot deformation of body-centered cubic high-entropy alloys without severe plastic deformation

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Manufacturers of gas turbine engines have been continuously seeking to improve the operating temperature of engines to improve their performance and efficiency. Currently using Nickle-based superalloys are working at temperatures close to their physical limits and cannot withstand any further increase in the operating temperatures. Refractory high entropy alloys (HEAs) are a new class of alloys having at least five principal elements in equiatomic or near-equiatomic concentrations. Refractory HEAs have been reported to exhibit excellent mechanical properties beyond the melting temperatures of Ni-based superalloys. Therefore, refractory HEAs are being considered as possible candidate alloys to replace superalloys.

In this study, hot-deformation behaviors of an equiatomic HfNbTaTiZr refractory high entropy alloy having single phase body-centered cubic (BCC) crystal structure were investigated. Uniaxial compression was carried out at different temperatures ranging from 1000°C to 1200°C and various strain rates from 10⁻⁴ s⁻¹ to 10⁻² s⁻¹. Stress-strain curves indicated distinctive sharp drops at yielding followed by a continuous decrease of flow stress. The sharp drop of the stress was explained from a viewpoint of dislocation locking possibly by any element atom(s) and unlocking. Flow stress analysis was carried out by using Arrhenius type power law relationship. The flow stress analysis indicated high strain rate sensitivity (m) of the flow stress ($m \sim 0.35$). Microstructural observations indicated inhomogeneous microstructures that were primarily composed of coarse unrecrystallized regions surrounded by fine dynamically recrystallized (DRX) grains with necklace morphologies. The high *m* value suggested that the fine DRX grains in the necklace region might deform by grain boundary sliding (GBS) mechanism. The relevance of high *m* value in the inhomogeneous microstructures and related deformation mechanisms in the DRX necklace regions were systematically investigated. Adding to that, the origins of ultrafine grain refinement were investigated using dislocation pile-up model and the related details are discussed.

Finite element assisted self-consistent simulations to capture barreling and texture heterogeneity during hot compression and its subsequent effect on rolling and recrystallization texture

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ABSTRACT

Finite element (FE) simulations were performed to capture the influence of friction and temperature gradient on barreling and the local strain distribution across the cross-section of the hot compressed specimen. The FE predicted deformation histories for different regions of the hot compressed sample were exported to the viscoplastic self-consistent (VPSC) model to perform texture simulations in the variable velocity gradient mode and to capture the texture heterogeneity. Differences in texture intensity, as well as characteristics, were observed depending upon the distance from the central region of the hot compressed sample. The implications of texture inhomogeneity in the hot compressed sample during subsequent steps of the thermomechanical processing cycle (cold rolling and recrystallization annealing) was analyzed by performing additional VPSC simulations in plane strain deformation mode and recrystallization simulations considering the probability of nucleation of new grains and their corresponding growth probability.

Acknowledgments

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Modelling the effect of pre-deformation on precipitation kinetics of AA2195 alloy

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ABSTRACT

Third generation Al-Cu-Li alloys find extensive application in aerospace sector due to their superior properties like high specific strength, good corrosion resistance and high elastic modulus. Their specific strength is among the highest of all aluminum alloys. The primary reason for such high strength is precipitation strengthening caused by coherent $Al_2CuLi(T_1)$ precipitates. These precipitates are extensively formed only in presence of dislocations. Hence pre-stretching is a common industrial practice for such alloys before aging treatment. In the present work, role of dislocations is investigated through experimentation and modelling. AA 2195 samples are given 0, 5, 10, 15, and 20% pre-strain and subsequently aged for a range of duration at 150°C. Microstructural and mechanical characterisation is done using XRD, EBSD, TEM, DSC, tensile test and hardness. Modified Kampmann-Wagner numerical model incorporating the effect of heterogeneous precipitation on dislocations and non-spherical precipitate shape is developed and applied to simulate precipitation evolution kinetics. Dislocation-precipitation interaction is also studied by areal glide model which helps in predicting strength of the material.

Acknowledgments

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A full field crystal plasticity simulation study on the propensity of grain fragmentation in copper for strain path change in shear

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ABSTRACT

Popular severe plastic deformation techniques like equal channel angular extrusion and high-pressure torsion rely on imparting large strain to the workpiece without changing the dimensions by deformation in shear to achieve grain refinement. Change in strain path has been employed effectively for different routes like A, B, C and B_A and B_C of equal channel angular extrusion as well as forward and reverse loading for high pressure torsion. In the present investigation, we employ full field crystal plasticity simulations to decipher the role of change in strain path in shear on the grain fragmentation of polycrystalline face centered cubic copper. To this end, the synthetic microstructure of copper generated using Dream.3D software was subjected to a von Mises strain of 4 in simple shear as well as with different strain paths in shear to monitor the evolution of microstructure in terms of intragranular misorientation as well as geometrically necessary dislocation density and texture using crystal plasticity simulations based on fast Fourier transformation solver using the DAMASK (Dusseldorf Advance Material Simulation Kit) software. The critical role of strain path change in tailoring slip activity to promote higher geometrically necessary dislocation density aiding in grain refinement will be analysed.

An experimental and crystal plasticity simulation study to explain optimum strength ductility combination in gradient microstructure copper samples produced by surface mechanical grinding treatment

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Surface severe plastic deformation techniques offer a unique heterogeneous gradient microstructure of different microstructural attributes like grain size, dislocation density, and residual stress to offer optimum strength ductility combinations in metallic materials. In the present investigation, we developed a laboratory scale tool for surface mechanical grinding treatment (SMGT) and subjected plates of oxygen free high conductivity copper to six passes of SMGT to obtain a thin layer of ultrafine grained microstructure on the surface. Tensile deformation of the SMGT samples presented higher yield strength (102±20 MPa) and ultimate tensile strength (253±25 MPa) with comparable ductility (53±2%) compared to the as-received material (65±17 MPa, 189±5 MPa, 68±3%). Additionally, measurement of the hardness performed at the surface and center of the gradient sample showed higher hardness at the surface 2.1±0.2 GPa compared to the center 1.8±0.1 GPa. Higher hardness at the surface compared to the center is attributed to higher stored dislocations density and finer grain size. Crystal plasticity simulations using the fast Fourier transform solver in DAMASK (Dusseldorf Advanced Materials Simulation Kit) software was employed to explain the mechanical properties in different regions of the gradient microstructure. In addition, sample scale simulations were performed to explain the unique strength ductility composition for the SMGT sample by employing crystal plasticity simulations at a higher length scale. It is shown that judicious application of the crystal plasticity simulations at the micro and meso length scale can be utilized to explain the macroscopic properties of SMGT samples.

Validation of Thermomechanical Model for Stress Relaxation in Superelastic Shape Memory Wire using Experimental Observation

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ABSTRACT

The NiTi alloy is a member of the family of materials known as shape memory alloys and exhibits both superelasticity under stress-induced cycles and the shape memory effect during heat cycling. These lead to the usage of SMAs in a variety of mechanical, civil, and biomedical engineering applications where mechanical and thermal loadings are present. It is to be noted that when NiTi alloy is subjected to cyclic loading plastic strain accumulates due to plastic deformation in the material. In this work, we have investigated and modeled the successive stress relaxation behavior during cyclic loading.



Fig.1: Hysteresis loops under experimental strain rate (successive stress relaxation) and corresponding model validation

In order to investigate the stress relaxation processes in SMA wires, we have developed a thermomechanical model based on the approach presented by Khadkhodai *et al.* [1]. In this model, the heat transfer coefficient is assumed to be temperature dependent and the transformation of latent heat and convective heat transfer between the material and the environment are taken into consideration. When a shape memory alloy wire is loaded and unloaded, the model estimates the change in stress for the subjected strain rate. The SMA Wire used in this experiment had a diameter of 0.6 mm, and a length of 100 mm. Using 1% step increments for loading and unloading up to 8% capacity. After each 1% loading and unloading, a 20-second period of relaxation is given. Both loading and unloading occur at a strain rate of 400%/second. Experimental and model responses of hysteresis loops under successive stress relaxation during cyclic loading are illustrated in Fig.1. The thermomechanical model's validity in the successive stress relaxation experiment of SMA wire and experimental results have a fair agreement. One can observe that the discrepancies between the experiment and model results may be caused by omitting the wire's radiation and conduction heat. Efforts are being made to improve the model by including radiation and conduction heat, with specifics to be presented at a conference.

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ATOMISTIC APPROACH FOR UNDERSTANDING DYNAMIC STRAIN AGEING IN AL-MG ALLOYS

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ABSTRACT

In the present work, force field based atomistic modelling techniques assisted by evolutionary algorithms were adopted to simulate solute (Mg)-dislocation interaction in Al-Mg alloys. Firstly, the segregation behavior of Mg towards dislocation was evaluated by calculating the segregation energy as a function of solute concentration. Evolutionary algorithm was used to select the energetically most favorable substitutional sites for a particular concentration. The segregation energy was found to be negative for all the concentrations. It indicates that segregation of Mg to dislocation core is energetically favorable. It was also observed that Mg atoms prefer to go to the tension side of the dislocation core. However, beyond 4 wt.% Mg, the solutes start substituting in the compression side as the energetically favorable sites on the tension side get exhausted. Furthermore, differential binding energy (ΔW) of solute atoms and activation enthalpy (ΔH_{cross}) required for solute atoms to jump across the dislocation core were computed using atomistic simulations. The differential binding energy per Mg atom was found to be independent of the Mg concentration. The results obtained from atomistic simulations were then used as input in a phenomenological work hardening model to simulate the macroscopic stress-strain response.

A comprehensive study on the implications of cluster characteristics on work hardening, strain rate sensitivity, formability and paint-response of AA 6082 sheets

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ABSTRACT

In the present work, the role of natural aging and preaging on uniaxial and biaxial deformation behavior was investigated to develop a holistic understanding of formability of an automotive grade Al-Mg-Si alloy (AA 6082). The uniaxial and biaxial deformation tests were performed in conjunction with digital image correlation to obtain the temporal evolution of strain and unambiguously establish the uniform and non-uniform deformation regimes. It was observed that the natural aged sample showed a higher uniform strain compared to preaged sample. The strain hardening rate was also higher for the natural aged sample compared to preaged sample. The fracture surface of the natural aged sample revealed a ductile type failure characterized by the presence of dimples. On the other hand, the preaged sample showed flat shear type failure. Nonetheless, the paint bake response of naturally aged sample was observed to be poor with a hardening increment of ~ 10 MPa. It was observed that preaging at 100° C for 3 hours provides the best compromise between formability and paint bake response.

Acknowledgments

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A new physical based framework capturing the role of dynamic strain aging on work hardening behavior of an Al-Mg alloy

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ABSTRACT

A new physical based framework is proposed to predict the work hardening behavior of Al-Mg alloy. Uniaxial tensile tests were performed on solution treated AA5083 alloy at different strain rates spanning from 1 X 10^{-4} s⁻¹ to 2 X 10^{-1} s⁻¹ at room temperature. It was observed that the type of serration changed from A type to C type with decrease in strain rate, characterized by changed in stress drop histogram from power law distribution to narrow peaked distribution. Furthermore, the strain hardening rate was observed to be enhanced with decrease in strain rate. Upon analyzing the flow curves using the traditional one internal state variable (1IV) model based on forest dislocation density, it was observed that the model overpredicts the early and underpredicts the later portion of the flow curves. The agreement between experimental and simulated curves was improved upon integrating the 1IV model with strengthening contribution from dynamic strain ageing (DSA). Nonetheless, the integrated model failed to reproduce the later stages of work hardening in a satisfactory manner. Upon considering the role of solutes in hampering the cross-slip propensity in combination with strengthening contribution from DSA, a significantly better agreement between experimental and simulated flow curves were observed. The simulation results were validated in terms of dislocation density measurements using convolutional multiple line profile analysis and electron back scatter diffraction.

Acknowledgments

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Microstructure and texture evolution during uniaxial compression of Mg-Zn-Ca alloy

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Lightweight biocompatible Mg-Zn-Ca alloys have a high potential to be used as biodegradable structural bio-implants. In this study, Mg–1Zn–0.2Ca (wt%) alloy was fabricated by casting followed by plane-strain forging at 450°C. Uniaxial compression tests were performed at room temperature along the flow direction normal to the forging axis. The tests were stopped at intermediate strains to carry out microstructural analysis and texture evolution using the electron backscatter diffraction (EBSD) technique. The initial texture has a profound effect on the microstructure, and texture evolution of the Mg-Zn-Ca alloy. The microstructural examination indicates the evolution of various twins and their interactions influence the texture.

Keywords: Mg-Zn-Ca alloy, Uniaxial Compression, EBSD, Plane strain Forging

Effect of grain size gradient on mechanical properties of FSPed AZ91

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ABSTRACT

Magnesium alloys have attracted attention for structural applications due to its high strength to weight ratio, low density and high stiffness. AZ91 (Mg–9Al–1Zn) is one such Mg alloy which is widely used in aerospace, automobile, electronic industries and biomedical field due to its excellent castability, wide range of room temperature mechanical properties and high corrosion resistance. However, due to dendritic microstructure and scarcity of active slip systems, its room temperature formability is poor. Friction stir processing (FSP) is a relatively newer solid state microstructure refining technique capable of bulk processing of sheet metals. FSP was employed on as-cast AZ91 Mg alloy to break the dendritic cast structure and reduce the grain size to few microns. FSP parameters were optimized based on the formation of fine and defect free microstructure. A grain size gradient was observed consisting of very fine grains on the top layer (close to the tool shoulder) of the nugget zone which subsequently grew as we inched towards the bottom of the nugget zone. Microhardness tests were performed and an increase in hardness was observed post FSP.



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Understanding the micromechanics of precipitation distribution in WE43 alloy using Nye tensor

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Abstract

The micromechanics of precipitation distribution in WE43 alloy was studied in detail using the electron back-scattered diffraction (EBSD) and Nye tensor method. It was observed that the dislocation density increases during deformation and aging leading to a decrease in the dislocation density of the sample. Further, it was found that low dislocation density is required for grains of near prismatic orientation $(50^{\circ} - 90^{\circ})$ compared to grains of near basal orientation $(20^{\circ} - 40^{\circ})$ to form the precipitates in grains under identical aging conditions. The orientation distribution functions (ODFs) showed that shifting of texture components along φ_1 axis is more for near prismatic orientated grains compared to near basal orientated grains suggesting higher activity of screw dislocations in the former one. The higher activity of screw dislocations for near prismatic orientated grains is reflected in the formation of more dislocation debris of $\langle c \rangle$ type on the (1010) planes. The dislocation debris of $\langle c \rangle$ type is reflected in the higher values of α_{31} and α_{32} components in the Nye dislocation tensor matrix compared to near basal orientated grains. The high density of dislocation debris accelerates the pipe diffusion resulting in the formation of more precipitates in the prismatic oriented grains compared to near basal oriented grains and the said precipitates are present mostly near the grain boundaries. The number of precipitates is more in the prismatic oriented grains compared to near basal grains.

Keywords: WE43 alloy, Dislocation density, Nye tensor, EBSD

Fabrication nanostructure during the heat treatment of Ti2AlNb intermetallic

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ABSTRACT

Ti₂AlNb intermetallics are promising next-generation aerospace materials. Manufacturing products from these alloys is carried out using different technologies, including additive manufacturing, and necessarily includes heat treatment at various temperatures below beta-transus. During heat treatment, a wide range of nanostructures can be obtained, which have a significant effect on the mechanical properties of materials and alloys. In this work, the influence of temperature-rate processing modes on the structure, phase composition and hardness of the Ti-21Al-24Nb-1.7V alloy was studied. The method of differential thermal analysis (DTA) was used for heating up to the temperatures of 830, 870, 910, 950 °C with cooling rates of 10, 20, 37, 50 and 80 °C/min, for this. The research showed at a temperature of 950 °C in the investigated range of cooling rates (10...80 °C/min) an increase in the volume fraction of the dispersed nano-platelets of O-phase is observed. The decomposition during cooling from heating temperatures contributes to an increase in the values of the lattice parameter of the β -phase. The change in hardness is affected by the following factors: the ratio of β - and O-phases, the presence and fraction of nano O-precipitates, the alloying of the β-phase with Al, and ordering in the β-phase. The decrease in hardness with an increase in the heating temperature from 830 to 910°C is determined primarily by a reduction in the volume fraction of the O-phase and an increase in the fraction of the disordered β-phase. The spread of hardness values at a heating temperature of 950 °C is determined primarily by the presence of secondary precipitates with different thickness from 32 nm to 330 nm. An increase in the thickness of the precipitates of the O-phase leads to an increase in hardness and possible higher strength and lower ductility of the material.

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Development of Al₃BC reinforced novel in-situ Al-based metal matrix composites via warm-extrusion

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Abstract

The work involves developing feasibility of synthesizing in-situ Al₃BC/Al nanocomposites via solid reactions from mixed blended powders of amorphous Boron (B), Aluminum (Al), and graphene nano-plates (GNPs) using warm-extrusion. Prescribed route of preparing this composite is addition of highly reactive amorphous B and GNPs into Al powder via mechanical milling in a planetary ball mill and consolidation via warm extrusion under inert atmosphere, to get the final bulk composite. This processing route tailors advantages both solid reactions and severe plastic deformation, yields homogeneous distribution of phase within the final product. The addition of GNPs will provide smaller dimension carbon particles that dispersed uniformly and react more efficiently in the composites than graphite. Furthermore, the evolutions of plasticity, deformation and strengthening mechanism will be explored using nanomechanical characterizations.

Keywords: Metal matrix composite; in-situ Al₃BC; Graphene nano-plates; Severe plastic deformation; and Warm Extrusion.

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Crystallization kinetics on melt spun and HPT processed Zr₆₂Cu₂₂Al₁₀Fe₅Dy₁ metallic glass

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ABSTRACT:

Influence of conventional and accumulative high pressure torsion (HPT) processing on structural properties of $Zr_{62}Cu_{22}Al_{10}Fe_5Dy_1$ metallic glass is examined by X-ray diffraction (XRD) technique. The XRD results showed that HPT processing leads to free volume enhancement in the metallic glass sample. Free volume increased with number of anvil turns in conventional HPT process, while highest free volume increment was observed in accumulative HPT processed sample. Further, influence of Dy addition on the crystallization kinetics is investigated under non-isothermal conditions using differential scanning calorimetry (DSC). Minor Dy addition resulted in increased activation energy and lower nucleation rate during crystallization of Zr-Cu-Al-Fe metallic glass. Strong correlation was observed between latent crystalline phases and intermetallic compounds in devitrified $Zr_{62}Cu_{22}Al_{10}Fe_5Dy_1$ metallic glass.

Comparison of multi-axial forging of Ta and Ta-10wt% W

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ABSTRACT

The presented work compares the multi-axial forging (MAF) of pure tantalum and tantalum-10wt% tungsten carried out in a closed die keeping the sample of size $10 \times 9.9 \times 5$ mm³ between top and bottom plug. The sample was forged to 50% height in a channel with cross section 10×10 mm². After each forging pass, the sample was rotated such that the dimension of the sample with 9.9 mm goes into the channel die. The samples were forged till 3, 6, and 9 passes to accumulate an equivalent strain of 2.4, 4.8, and 7.2 respectively. Microstructural and mechanical characterization were carried out in both the Ta and Ta-10% wt W samples and their results were compared. Microstructural examination using EBSD scans reveal wavy nature of grain boundaries (GBs). Large grains with wavy GBs ping to form small grains. Sliding, mobility and interaction of equilibrated GBs in Ta were studied using molecular dynamics (MD) simulations. MD results showed that the low angle GBs are mobile while high angle GBs require ledges or dissociation to attain mobility. Wavy nature of GBs was observed in both experimental results and simulations. The effect of W on microstructure and mechanical properties were quantified.

Effect of initial grain size on Recrystallization texture of cold rolled Ti-15V-3Al-3Cr-3Sn Beta Titanium Alloy

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In the present work, the effect of initial grain size on the deformation and recrystallization behaviour of beta titanium alloy (Ti-15V-3Al-3Cr-3Sn) was investigated. Initially sample were solution annealed at 800°C for 30 min and 1000°C for 15 min to obtain two different equiaxed initial grain sizes. The initial average grain size after solution annealing at 800°C was ~87 μ m, which increased to ~117 μ m for solution annealing at 1000°C. Both the samples were then cold rolling up to 80% thickness reduction. After cold rolling recrystallization was done at 780°C for varying times. After cold rolling, elongated grains were observed in the rolling direction (RD) with the occurrence of shear band Formation of shear bands in the microstructure was more prominent for smaller grain size sample i.e., the frequency of occurrence was more. Further, these bands were found to have orientation dependence, they were significantly more in γ -fiber (ND// <111>) grains. Cold rolled samples showed the presence of partial α -fiber (RD// <110>) and complete γ -fiber (ND// <111>). Recrystallization produced a strong discontinuous γ -fiber (ND// <111>) with maxima centered around {111} <112> component for both the samples.

Keywords: Beta titanium, EBSD, recrystallization, shear bands, texture.

Plastic Flow in Small Cavities of Nanocrystalline CuZn30 Introduced by Nanoimprinting

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ABSTRACT

Metallic nanoimprinting is a new technique to form robust surface structures on metallic materials on various length scales. Besides the dimensions of the nanoimprinting die, the mechanical behavior of the imprinted material and its microstructure also influence the shape and size of the surface features. A multi-length-scale approach was applied by varying the cavities of the imprinting die (width between 20 nm and 2.76 μ m), as well as the grain sizes of the sample material. The examined CuZn30 alloys have grain sizes between 100 nm and 277 μ m, thus covering a wide range of different hardening behaviors and ratios of grain size to cavity width. Experimental results indicate the major influence of the work hardening behavior and the subgrain or grain size on the forming characteristics during nanoimprinting. It can be stated that an ideal plastic behavior leads to the largest extrusion heights. Regarding materials with a strong work hardening behavior, low extrusion heights for all cavity widths could be measured. Based on these investigations, further researches have been carried out to upscale this process. For that purpose, a new imprinting tool with periodic structures in the micrometer regime has been manufactured, which is subsequently incorporated into the sample material. The evaluation of the results leads to the conclusion that a good transferability of the structures of the punch geometry is possible on nanocrystalline materials, as well as on cold-formed microstructures. These results show the potential of highly deformed and nanocrystalline microstructures for the application in industry.

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Effect of B Addition on the Phase Transformation and Mechanical Behavior of Nanocrystalline Ti-Fe Alloy

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ABSTRACT

As the strongest eutectoid β stabilizing element, Fe is constantly used to design low-cost titanium alloys due to its cheap price, which could also improve mechanical properties simultaneously through the solid solution strengthening effect. Severe plastic deformation (SPD) can be considered as an alternative technology for improving the properties of Ti-based alloys. SPD always initiates the strong grain refinement and can lead to various phase transitions.

In the previous studies, the $\alpha \rightarrow \omega$ and $\beta \rightarrow \omega$ phase transformations generated in NC binary Ti-xFe alloy produced by high pressure torsion (HPT), which occupied 80% after 5 rotations under 6GPa pressure. Subsequently, the ultimate tensile strength of NC Ti-xFe (x=0.04~1) alloy to 1~1.4GPa, but the ductility is less than 2% due to abundant of hard and brittle ω phase.

Recently, it has been shown that the addition of trace amounts of B (up to 0.1 wt.%) is beneficial in improving the mechanical properties. Together, these benefits have the potential to make Ti alloys much more affordable. In the present work, effect of 0.1 wt.% B addition on the phase transformation and mechanical behavior of NC Ti-2Fe alloy by HPT was investigated, Result indicated that the ultimate tensile strength of nanocrystalline Ti-2Fe-0.1B exceed 1.6 GPa, and the ductility is around 8%, which is much better than binary Ti-2Fe alloy. The influence of B addition on the phase transformation of Ti-2Fe under HPT was studied and the strengthening and ductile mechanism was discussed.

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