International Workshop

New trends in research of UFG materials produced by SPD

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New trends in processing metals by severe plastic deformation

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Conventional thermo-mechanical processing provides the potential for producing materials with very small grain sizes, typically of the order of ~3-10 μm. However, extensive experiments over the last twenty years have demonstrated that much smaller grain sizes may be attained, in the submicrometer or even the nanometer range, through the application of severe plastic deformation (SPD). Several different SPD processing techniques are now available but typical procedures are equal-channel angular pressing (ECAP) and high-pressure torsion (HPT). Very recently, alternative variations of these procedures were developed based on ECAP and HPT and it was shown that these new procedures provide an opportunity to achieve exceptional properties. This presentation describes examples of these alternative procedures.

UFG materials produced by SPD: multifunctional properties

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This report is devoted to research and development of a variety of nanostructured metals and alloys with advanced multifunctional properties, when their high mechanical properties (strength, fatigue, wear resistance) go with high physical (electrical conductivity, magnetic properties, etc.) or chemical (corrosion resistance, biocompatibility) ones. Physical nature of such unusual combination of properties in nanoSPD materials is discussed and the examples of innovative applications of such materials in engineering and medicine are considered as well.

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Experimental investigation of the influence of nanoscaled particles and GB segregations on the thermal stability of UFG Al alloys

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UltraFine Grained (UFG) Aluminium alloys are attractive because they could combine high strength, low density and for some of them also high electrical conductivity. However, due to the high driving force for grain growth, their thermal stability is often relatively poor. In this presentation, it is proposed to discuss recent in-situ annealing Transmission Electron Microscopy (TEM) results that have been collected on two model systems (Al-Rare Earth and Al-Zn) processed by Severe Plastic Deformation (SPD). A special emphasis will be given on the role of grain boundary segregations and nanoscaled particles to pin boundaries. The artefact resulting from the TEM thin foil surfaces will be discussed and finally it will be shown how such in-situ experiments could be used to tune and optimize UFG structures by short time annealing.

Biodegradable Metals

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After decades of developing strategies to minimize the corrosion of metallic biomaterials, there is now increasing interest in using intentionally corrodible metals in a number of critical medical device applications. A term “biodegradable metal”(BM) had been used internationally to describe these new kinds of degradable metallic biomaterials for medical application. In this paper, the definition of BM and its classification are given for the first time, along with the summary of the degradation mechanisms of BMs and its environmental influencing factors, which includes the degeneration of mechanical integrity and the metabolism of the degradation products. The recently-developed representative Mg-based BMs (pure Mg, Mg-Ca alloy, Mg-Zn alloy, etc.), Fe-based BMs (pure Fe, Fe-Mn based alloys, etc.) and Zn-based BMs (pure Zn , Zn-Ca alloy, Zn-Sr alloy, etc.) were comprehensively reviewed with emphases on their microstructures, mechanical properties and degradation behaviors, in vitro and in vivo performances, pre-clinical and clinical trials. Moreover, current approaches to control their biodegradation rates to match the healing rates of the host tissues with various surface modification techniques and novel structural designs are summarized. Finally, this paper comprehensively discusses the directions of future development and the challenges of transitioning BMs from raw materials to semi-products to final medical devices.

Giant straining by HPT to produce UFG materials

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In this study, giant shear strains up to 100,000 are introduced in different metallic materials by high-pressure torsion (HPT) method and evolution of microstucture and phase transformation are investigated. New phases are formed in many immiscible systems, while a real saturation of microstructure is hardly achieved even at very large strains. The mechanical properties of ultrafine-grained (UFG) materials produced by such large strains are discussed in this talk with a special attention on room-temperature superplasticity in an Mg-based alloy [1].

[1] K. Edalati, T. Masuda, M. Arita, M. Furui, X. Sauvage, Z. Horita, R. Z. Valiev, Room-Temperature Superplasticity in an Ultrafine-Grained Magnesium Alloy, Scientific Reports, in press (2017).

Effects of solutes on strength and stability of nanotwinned UFG metals

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We consider the effects of solute atoms on plastic deformation through de-twinning processes mediated by incoherent twin boundaries (ITBs) in nanotwinned metallic alloys under a mechanical load. Within the model, the regions near ITBs contain the segregations of solute atoms formed during the fabrication of nanotwinned alloys in the stress fields of the dislocations composing ITBs. We calculate the critical parameters that characterize de-twinning in Cu-based nanotwinned alloys containing low concentrations of Ni, Zn or Al. By comparison of these critical parameters with those of pure nanotwinned Cu, it is concluded that solute atoms hinder detwinning, thus improving the stability of deformed nanotwinned solids. Also, in the case of highly nonequilibrium ITBs, where detwinning contributes to plastic flow, solute atoms strengthen nanotwinned materials.

In addition, we theoretically examine two plastic deformation mechanisms in deformed columnar-grained nanotwinned metals loaded parallel to coherent twin boundaries. The first mechanism represents the formation and expansion of split dislocation loops within individual twins, while the second one assumes the emission of split jogged dislocations from grain boundaries into grain interiors. Within the model, both split dislocation loops and jogged dislocations are generated at pre-existent full dislocations at grain boundaries. Under these assumptions, the critical stresses for the nucleation and expansion of split dislocation loops and jogged dislocations are calculated. It is demonstrated that at a specified grain size, the critical stress for the formation and motion of jogged dislocations is always smaller than that for the expansion of split dislocation loops across twin lamellae.

Grain refinement by friction-stir processing

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In this work, grain refinement efficiency of friction-stir processing (FSP) was examined. To this end, the effect of FSP on microstructure, misorientation distribution and crystallographic texture was studied in number of materials and grain refinement mechanisms were identified.

FSP was found to be feasible for production of reasonably homogemeous microstructure with mean grain size of ~1 μm. Fraction of high-angle boundaries may vary from ~50 to 90 pct, depending on particular material and processing conditions. FSP was shown to normally produce a simple shear texture and texture distribution in stir zone is fairly inhomogeneous.

Microstructure evolution during FSP was found to be a relatively complex process. In all cases, it involves geometrical effect of strain but the grain refinement mechanisms is principally dependent on stacking fault energy (SFE). In high-SFE materials, the grain-structure development was shown to be dictated by grain subdivision mechanism. In low-SFE materials, in addition to the grain-subdivision, the microstructure evolution was also essentially enfluenced by discontinuous recrystallization and the related annealing twinning.

Superior strength of ultrafine-grained Al and Fe-based alloys provided by grain boundary segregations

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An Al-6Mg alloy and an austenitic stainless steel were nanostructured by high pressure torsion (HPT) at different temperatures. As a result different ultrafine-grained (UFG) states were observed in the investigated alloys characterized by different grain size, dislocation density and the other features. The Al alloy processed at room temperature and the 316 steel processed at 400°C were characterized by significantly enhanced strength, which exceeded the value expected for the given grain size according to the Hall-Petch relation. This extra-strength was explained by formation of heterogeneous strain-induced segregations of solute atoms at grain boundaries detected in both alloys by 3D atom probe tomography (see Fig.1a for the case of the UFG Al-Mg alloy). Interestingly, the ultrafine-grained steel produced by HPT at room temperature did not show a trend for grain boundary segregation and, respectively, its strength did not exceed the Hall-Petch predictions. Moveover, annealing at temperatures ranging from 400 to 650°C leaded to a notable increase in strength for the case of the steel produced at room temperature, accompanied with the formation of irregular grain boundary segregations. Estimations for the strengthening contribution in terms of the extra-stress required for the dislocation to overcome the segregated boundary were found to be in a reasonable agreement with the experimentally observed values. These results can be used in understanding of mechanical performance of Fe-Cr alloys under investigation.

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Electrochemical impedance spectroscopy of PEO coatings on UFG magnesium alloy Mg-1Ca

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Magnesium alloys promise development of biodegradable implants for treatments in traumatology. However, insufficient strength and corrosion activity inhibit their applications in surgery. Advanced material and surface engineering via nanostructuring and plasma electrolytic oxidation (PEO) opens wide possibilities to control mechanical and corrosion properties of the alloys. This contribution uncovers biodegradation mechanisms for coarse grain and ultrafine grain (UFG) Mg-1Ca alloy, both coated and uncoated, using advanced method of electrochemical impedance spectroscopy (EIS).

It was shown that the grain size variation using nanostructuring via high pressure torsion technique and annealing helps to increase the alloy corrosion resistance in a simulated body fluid. The alloy corrosion rate decreases with the grain refinement; this helps to increase the sample disintegration time from 3-4 days to more than 30 days. Application of a biocompatible coating using plasma electrolytic oxidation further decreases the corrosion rate more than twice due to formation of a Ca-, P-containing oxide layer with porosity decreasing towards the alloy grain refinement.

The EIS shows that the equivalent circuit can have a Voight structure, and it includes at least one R-CPE branch standing for a double layer and Re for the electrolyte. For come cases where two semicircles in the spectra are clearly identified, two R-CPE branches were used. The first Rct-CPE-d branch can be assigned to a double layer representing the alloy corrosion products and the PEO coating. The second Rb-CPE-b branch has high values of the capacitance Qdl suggesting its small thickness; therefore, this branch can be assigned to a barrier layer. This branch appears only for nanostructured and UFG alloys which have the best corrosion resistance. All the circuits have a finite length Warburg element indicating the influence of the diffusion in the system.

The mechanism underlying the controllable biodegradation is based on the balance between the anodic dissolution in local corrosion galvanic cells and the oxide/hydroxide formation contributing towards barrier layer development. This balance is controlled by the secondary phase Mg2Ca particle size and distribution uniformity, and the barrier layer development and protection with the PEO coating having adjustable pore size distribution. Among the tested samples the best corrosion resistance is provided by UFG Mg-1Ca alloy with grain size 0.9-1.3 μm and having the PEO coating 9-11 μm thick. These parameters fortunately correlate with the necessity to anneal the alloy nanostructure and release the internal stresses in order to improve the ultimate strength and ductility to trade-off values achieved at the same grain size. Finally, the proposed structure and surface treatments contribute to production of future biodegradable Mg implants suitable for surgery.

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Modification of ultrafine-grained titanium by chemical etching and atomic layer deposition to produce bioactive implant surfaces

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The samples of ultrafine-grained titanium modified by chemical etching in NH_4OH/H_2O_2 or H_2SO_4/H_2O_2 and Atomic Layer Deposition (ALD) were successfully prepared. The surface topography and morphology have been studied by means of scanning electron microscopy (SEM), atomic-force microscopy (AFM) and the spectral ellipsometry. The composition of the samples has been determined by X-Ray fluorescence analysis (XRF), energy dispersive X-ray- scanning electron microscopy (EDX-SEM) and X-Ray Photoelectron Spectroscopy (XPS). The energy characteristics of the surface were evaluated by measuring the wetting angles. The bioactivity and biocompatibility were studied "in vitro" (adhesion, viability, proliferation, differentiation of osteoblast cells MC3T3-E1) and "in vivo".

The relationship between morphology, topography, composition of the samples surface and cytological response of MC3T3-E1 osteoblasts cells were studied in detail. Regardless of the type of surface treatment, all the samples showed good adhesion and cell viability, however, coating the surface of the sample with titanium dioxide leads to an increase in hydrophilicity and also a significant improvement in the proliferation and differentiation of cells in the osteogenic direction. «In vivo» study have shown that the samples are non-toxic, successfully implanted in rabbits and have prospects for successful application as bone implants. Among the samples studied, the one that was etched in NH_4OH/H_2O_2 solution within 2 hours and modified by ALD is particularly prominent. This sample showed the best values of the cell differentiation «in vitro» (2.5-3 times higher than the control sample) and engraftment «in vivo».

Impact toughness of ultrafine-grained CP titanium for medical application

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CP titanium and titanium alloys are considered as one of the best groups of biomaterials for implantable device applications. As is known, high tensile strength and high endurance limit under cyclic loading of titanium are achievable due to severe plastic deformation. But, along with the improved fatigue resistance, a plasticity and impact toughness (as ability of a metal to absorb energy and deform plastically) is the one of major mechanical requirements for implants (in particular for osteosynthesis). The UFG structure was performed by ECAP-Conform with subsequent drawing and annealing. UFG structures with various parameters (size and form of grains, dislocation density, condition of boundaries and etc.) were formed by varying treatment regimes. The tensiletests and impact toughness tests of specimens with a V-shaped notch with various types of the CP-4 Ti structure in a coarse-grained (CG) and in an UFG states are carried out.The results are demonstrated that intensive refinement of grains with high dislocation density, their elongated form obtained as a result of drawing at room and enhanced temperatures (up to 400°C), leads to the decrease in the impact toughness of Ti Grade 4. A possibility to improve the impact toughness of UFG Ti with non-significant decrease of strength via short annealing at 400-450° C, contributing to the decrease in dislocation density and transformation of low-angle boundaries into the high-angle ones as a result of recovery and redistribution of dislocations is demonstrated. The dependence of the impact strength on the UFG Ti ability on strain hardening is discussed.

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TiNi shape memory foams produced by self-propagating high-temperature synthesis

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The aim of the work is a study of the structure, martensitic transformation and functional properties in porous TiNi shape memory alloys. The TiNi forms were produced by self-propagating high-temperature synthesis using the Ti and Ni powders mixtures with different Ni concentration. It was found that the TiNi foams consisted of the TiNi, Ti_3Ni_4 and Ti_2Ni phases moreover, the TiNi phase was characterized by the heterogeneous Ni element distribution and this led to the martensitic transformation were observed in a wide temperature range. The method to determine the volume fraction of the TiNi phase with different Ni concentration was found. The influences of the Ni concentration in powders mixture and the parameters of the post-production heat treatment on the structure and the Ni distribution in the TiNi phase were found. The mechanical behaviour and the shape memory effects were studied in the TiNi foams with different structure. It was found that the presence of the TiNi phase volumes with the 50.0 at. % Ni concentration assisted to the plastic deformation of the TiNi foams instead of the deformation via phase transformation or the martensite reorientation. It decreased the value of the recoverable strain and increased the contribution of the irrecoverable strain to the total strain variation observed on heating or thermal cycling. A decrease in the TiNi phase volumes with the 50.0 at. % Ni concentration and increase in the TiNi phase with 50.7 at. % Ni concentration improved the functional properties of the TiNi foams.

Improvement of the mechanical and biomedical properties of implants via the production of nanocomposite based on nanostructured titanium matrix and bioactive nanocoating

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In this presentation, we report on our studies of bioactivity of ultrafine grained (UFG) titanium by means of bioactive coatings. We will analyze the geometrical modification, namely, titanium oxide coating by sol-gel dip coating technique, the production of the two-level surface relief hierarchy (due to the structuring of the film by means of the shock-drying or introduction of polymeric dopants). Also, we will report on the results of the development of the composite coatings titanium oxide/calcium phosphate and of the coatings based on the titanium-organic groups. The cytological results will be discussed. Also, the prospects of the new direction will be presented, namely, assignment of the antibacterial properties to the coatings by means of the covalent grafting of antimicrobial drugs, creation of the bioresorptive implants with the coatings that control the rate and the mechanism of the resorption, development of the methods of the coating of the bioactive titanium-organic groups and other coatings by means of ALD on the substrates of various shape.

Microstructure, strength, electrical conductivity and heat resistance of an Al-0.4Mg-0.2Zr alloy after ECAP-CONFORM and cold drawing

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This work describes the routes to process conducting materials based on the Al-Mg-Zr system combining high strength (σ_{UTS} = 267 MPa), electrical conductivity (over 57 % IACS) and heat resistance (up to 150 °C). These properties in the alloy with 0.4 Mg and 0.2 Zr (wt. %) are achieved through the formation of ultrafine-grained microstructure by treatment, including annealing at 385 °C, severe plastic deformation (SPD) via equal channel angular pressing-Conform (ECAP-C) at room temperature followed by cold drawing (CD). The mechanical strength is enhanced by the formation of ultrafine grains during ECAP-C and their additional refinement through CD. Also, ECAP-C and CD result in the material strengthening due to increased dislocation density. The annealing of the alloy prior to SPD is used to provide a good heatresistance and high electrical conductivity via formation of nanoscale Al_3Zr precipitates with a metastable $L1_2$ structure and, correspondingly decreasing the concentration of Zr atoms in the Al solid solution.

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UFG structures and mechanical properties of two phase Al-Zn alloys

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This work demonstrates that processing by SPD leads to the formation of UFG structure in the Al-Zn alloys and leads to improved ductility. The supersaturated solid solution in alloys is decomposed during SPD processing resulting in nucleation and growth of a secondary phase precipitates, formation of segregations of alloying elements. It is proposed that the atomic mobility could be significantly enhanced during SPD especially thanks to the high vacancy concentration, solute drag by dislocations, pipe diffusion along dislocations or grain boundary diffusion. We have systematically investigated such dynamic precipitation phenomena in Al–Zn alloys with various ZN-content at different SPD processing regimes.

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The strength of submicrocrystalline aluminum alloys at wide range of strain rates

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The structure and the mechanical properties of A7075, A3003 and A5083coarse grained and submicrocrystalline aluminum alloys were studied in a wide range of strain rates (from static to dynamic). Uniaxial tension tests were conducted at room temperature and at strain rate of 10^{-3} s^{-1}. Dynamic compression was carried out by means of a splitHopkinson bar at strain rates of 4×10^3 and 6×10^3 s^{-1}. The deformation behavior of the materials was also investigated in the dynamic strain rate rangeof$(1.2–3)\times10^5$ s^{-1} achieved during shock-wave compression loading.

SMC structure was produced by the Dynamic Channel Angular Pressing (DCAP). Coarse grained (CG) rods 14 and 30 mm in diameter were used as the initial samples for deformation. The average subgrain size was 2 μm. During DCAP, a matrix extrusion die with channels intersecting at an angle of 90° was used. The initial sample velocity (V) in the matrix extrusion die was specified by the propellant powder mass and varied from 50 to 300 m/s. The number of pressing cycles N was varied from one to four. All passes were performed according to route Bc.

The most typical structure of the aluminumalloys prepared by DCAP is a fragmented nonequilibrium SMC structure with a high dislocation density and an increased level of internal stresses, where high-angle crystallite boundaries predominate.The fragmented structure forms via shear androtational plastic deformation mechanisms. An increase in the strain rate of the alloys with a high dislocation mobility calls for one more mechanism of elastic strain energy relaxation, namely, dynamic recrystallization.

For the aluminum alloys prepared by DCAP,the yield strength as a functionof the strain rate in the range from 10^{-3} to 10^5 s^{-1} exhibits complex behaviorand depends on the type of SMC structure.The decrease in the yield strength of A7075 alloy found in a ranges of strain rates of $4\text{-}6\times10^3$ s^{-1}, when the strain rate increases indicates a change in the dislocation mechanisms of deformation in SMC aluminum alloys.The dynamic yield strength of SMC aluminum alloys after DCAP increases at all strain rates of 10^5 s^{-1} compared to coarse grained alloys.

Development of UFG NiTi alloys by ECAP-Conform for medical application

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NiTi alloys are well-known thanks to their remarkable properties–enhanced strength, corrosion resistance, and especially pseudoelasticity and shape-memory effect. Due to this, they are very promising for many structural and functional applications in engineering and especially in medicine. The equal channel angular pressing (ECAP) technique allows to refine structure of bulk samples of NiTi-based alloys and significantly increase their strength and shape memory effect parameters. However, for practical applications continuous ECAP-"Conform" method is more promising technique. This approach allowing to receive a long-length semi-products such as rods. Present work shows possibility of ultrafine-grained (UFG) structure formation with a grain size of about 250 nm in NiTi alloys using ECAP-"Conform". The influence of ECAP-"Conform" processing on parameters of the microstructure, phase transformations and mechanical properties of NiTi alloy have been investigated. Results of comparative pseudoelastic cycling of the alloy NiTi in coarse-grained and UFG states are presented and discussed.

The structure and its mechanical properties of Mg-Zn-Ca alloys during severe plastic deformation

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There is a considerable interest in application of magnesium alloys for manufacturing of surgical implants. However, conventional magnesium materials possess rather weak mechanical properties. Recent studies have demonstrated that the chemical composition of the alloy significantly affect the formation of a nanocrystalline structure during high pressure torsion (HPT) technique. It is shown that after HPT alloy Mg-1% Zn-0,005% Ca average grain size was 250 nm. HPT alloy with a high content of up to 0.2% Ca in the alloy leads to the formation of the nanostructure with an average grain size of 90 nm. It was found that it was shown that all the samples subjected to high values HPT microhardness. The formation of fine particles enhances thermostability Mg_2Ca alloy structure. The dependences of mechanical properties on the structural features of UFG Mg-Zn-Ca alloys are analysed.

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Physical and Mechanical Properties of Cu - 2Be Alloy Processed by HPT

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An influence of thermal treatment and high pressure torsion (HPT) processing on secondary phase particles formation and mechanical properties of Cu-2Be alloy was studied. A homogenized solid solution treated alloy was subjected to three main thermomechanical treatment regimes: a coarse-grained alloy HPT with subsequent aging, coarse-grained alloy aging with subsequent HPT and aging during HPT at the temperature range from 150°C to 250°C. The results of X-Ray diffraction (XRD) over obtained materials showed that thermomechanical treatment regime affect on grain size, internal stress values and secondary phase CuBe particles location in the parent matrix material. According to XRD, the secondary phase particle in HPT processed alloy can locate both at the grains boundaries and in grains interiors. Mechanical properties investigations provided through uniaxial tensile tests showed that good combination of strength and plasticity was achieved in the alloy subjected to aging at the temperature 325°C for 10 hours with subsequent HPT processing. Fractography analysis via scanning electron microscopy (SEM) reveals a ductile-brittle fracture behavior of nanostructured Cu-2Be alloy. Results of this work shows that selection of thermomechanical treatment regime can be used as a driving factor for desirable phase formation kinetics with following mechanical properties.

Influence of SPD on fatigue behavior of UFG and nanostructured Ti-6Al-4V alloy

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This work was invoked to study an influence of severe plastic deformation techniques on fatigue properties of ultra-fine grained (UFG) and nanostructured Ti-6Al-4V alloy. Fatigue experiments data was carried out using designed mini-sized specimen geometry. An unique experimental equipment for fatigue testing, compatible with standard grips of fatigue testing machine, was designed and manufactured. Fatigue test with R = 0, and frequency of 30 Hz was provided using this experimental technique. A relevant Wöhler curves for coarse grained (CG) hot rolled, equal-channel angular pressing (ECAP) processed (8 passes) and high-pressure torsion (HPT) processed Ti-6Al-4V alloy was obtained. Fatigue curves curve can be characterized by good statistical dispersion, what is aims on applicability of proposed technique for fatigue testing of sub-sized specimens. As a result of conducted experiments the fatigue limit based on 10^7 cycles was estimated. It is equal to 350 MPa, 425 MPa and 300 MPa for CG, ECAP-processed and HPT processed alloy correspondingly. Obtained results can be useful to scale up the fatigue behavior from small specimens to big ones and to predict the fatigue behavior of bulk nanostructured materials. Fatigue tests and fractography over HPT-processed alloy showed that this state is brittle and elevated temperatures should be used during processing to avoid macrodefects (cracks, pores) formation during HPT.

Microstructure evolution of titanium alloy VT8M-1 with globular-lamellar structure during deformation in temperature range of 650–800 °C

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The VT8M-1 alloy is used in the production of the heavy-duty parts and units of aviation engines, including compressor disks and blades that operate under service conditions of high static and dynamic loading, erosion, corrosion, and temperature exposure. A promising way of enhancing the mechanical properties of titanium alloys is the formation of an ultrafine-grained (UFG) structure in them using severe plastic deformation (SPD) techniques. As is known, two-phase titanium alloys are hard-deformed, therefore SPD processing are usually performed in the temperature range 600 - 800°C. Herewith the formation of a UFG structure in titanium alloys is a complex process of structural transformations. The completeness of the two-phase structure transformation upon plastic deformation is determined by a temperature, strain rate, and the type of initial structure. For example, for Ti-6Al-4V alloy earlier it was shown that lamellar and globular modes of α-phase are significantly different in evolution kinetics during SPD processing. The aim of this work is a study the structure evolution and the mechanical behavior of the VT8M-1 alloy with an initial globular-lamellar structure during plastic deformation in the temperature range of 650–800°C.

In this study was used a hot-rolled rod 25 mm in diameter made of VT8M-1 alloy (VSMPO-AVISMA Corporation) with the composition, wt %: Ti–5.54Al–4.13Mo–1.21Zr–1.3Sn–0.16Fe. Billets were subjected to heat treatment by heating of the alloy to a temperature of 940°C, subsequent quenching in water, annealing for an hour at 700°C, and cooling in air to obtain a globular-lamellar structure. Then, the cylindrical samples 7 mm in diameter and 8.5 mm high were compressed to strain $\varepsilon = 50\%$ in the direction of sample height at a strain rate of 1.4×10^{-3} s^{-1} under isothermal conditions in the temperature range of 650–800°C in air using an Instron testing machine. Microstructural examination was carried out using optical microscope, a scanning and transmission electron microscopes.

The globular-lamellar structure consisted of α-phase lamellae divided by β-phase interlayers and the primary α-phase of a globular morphology. The fraction of the globular α-phase was about 25%. The average globule size was 2.7 μm and the average thickness of the α-lamellae was 0.12 μm. A study of the evolution of the microstructure of VT8M-1 alloy during plastic deformation showed that when compressed in the temperature range 650-800°C, the primary globular α-phase is elongated along the direction of material flow, whereas the lamellar fraction of the structure undergoes fragmentation and recrystallization. An increase in the deformation temperature from 650 to 800°C results in the intense development of recrystallization. The lamellar fraction transforms into the globular one owing to globularization and recrystallization processes, resulting in formation of an ultrafine-grained structure upon plastic deformation in the VT8M-1 alloy. The average size of recrystallized α- particles increases from 0.25 to 0.73 μm.

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Mechanical behavior of ultrafine-grained Ti-6Al-4V alloy with a protective coating (Ti-V)N

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Development of modern branches of mechanical engineering imposes much higher requirements to the quality and service properties of structural materials and items produced from them. In many cases combination of different properties is very important: for example, high mechanical and fatigue strength at low specific weight, corrosion and erosion resistance, thermal stability. This is topical for Ti alloys applied for manufacturing of such vital items as gas-turbine engine blades operating in extremely severe conditions of thermal, cyclic loads in aggressive environment.

Recent studies over the last two decades demonstrate that the formation of ultrafine-grained (UFG) structures by severe plastic deformation techniques is an effective way to increase physical and mechanical properties of commercial metals and alloys. To date, different SPD methods have been applied to process bulk UFG samples from the alloy Ti-6Al-4V alloy with a strength of up to 1200...1500 MPa, whereas the strength of a conventional hot-rolled alloy does not exceed 1000 MPa. Special attention is paid to enhancement of such service properties of structural materials as corrosion and erosion resistance. In this work a combined approach was used to the Ti-6Al-4V alloy that has been successively subjected to severe plastic deformation for the formation of bulk UFG structure. In the subsequent vacuum-plasma application of a multilayer coating (Ti-V)N, the formation of a refractory vanadium nitride compound in the process of plasma chemical reaction during condensation additionally contributes to wear resistance. The mechanical behavior of the Ti-6Al-4V alloy subjected to combined treatment was studied at room and elevated temperatures for evaluation of its performance properties.

The investigations were carried out on the Ti-6Al-4V alloy (Ti-basis, Al – 6.6%; V – 4.9%; Zr – 0.02%; Si– 0.033%; Fe – 0.18%; C – 0.007%; O_2– 0.17%; N_2– 0.01%; H_2– 0.002%). The initial samples with a diameter of 20 mm and 105 mm long were subjected to processing following the pre-developed technique consisting of preliminary heat treatment by quenching from T=950 °C (heating during 20 min) and consequent annealing at 675°C during 4 hours, equal-channel angular pressing (ECAP) on a die-set with the channels intersection angle φ=120° at a temperature of 700°C and further extrusion at 300°C. The PVD coating (Ti-V)N was applied on the samples in the installationWATT-900 3D simultaneously from two arc evaporators. The values of residual stresses are averaged over the entire depth of the X-ray transmission (up to 10 microns). Cylindrical samples with a gauge length 15 mm and diameter 3 mm were subjected to tensile testing on an Instron tensile machine at temperatures of 20...700°C and strain rate $\acute{\varepsilon} = 10^{-3}\ s^{-1}$.

The formation of the UFG structure with a mean grain/subgrain size of 260 nm in the alloy Ti-6Al-4V processed through ECAP and subsequent extrusion resulted in a higher strength of 30% at room temperature, and of no less than 15% at elevated temperatures up to 400 °C in comparison with the coarse-grained structure. Mechanical tests of the UFG samples with and without coating at temperatures of 300, 350, and 400°C demonstrated that the samples with coating exhibit a higher strength compared to the samples without coating, which is, apparently, related to the occurrence of the «barrier effect» in the near-surface layer of the metal close to the coating.

The results of this work reveal significant prospects for improving the service properties of Ti alloys through the formation of UFG structure by means of SPD processing and subsequent application of vacuum-plasma coatings on the surface.

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Strengthening mechanisms and grain refinement in commercial-purity titanium subjected equal-channel angular pressing

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Due to its excellent biocompatibility, commercial-purity titanium is a very promising material for biomedical applications. Until recently, however, its widespread use was limited by insufficient mechanical strength. Developed in the early 1980s, equal channel angular pressing (ECAP) can provide significant material strengthening via the formation of an ultrafine-grain structure, thus enabling new opportunities for this material. Extensive research has conclusively demonstrated the excellent potential of ECAP for microstructure refinement in titanium and the concomitant enhancements in strength. However, the corresponding microstructure-strength relationship has not yet been fully-quantified. The current work attempted to fill this gap in scientific literature. To this end, high-resolution electron backscatter diffraction (EBSD) was used for microstructure characterization.

The principal directions of ECAP geometry are denoted below as the longitudinal direction (LD), the transverse direction (TD), and the normal direction (ND). Microstructure characterization was performed primarily by EBSD. EBSD analysis was conducted with a JSM-7800F field-emission-gun, scanning electron microscope (FEG-SEM) equipped with a TSL OIM™ EBSD system. A 15° criterion was applied to differentiate low-angle boundaries (LABs) and high-angle boundaries (HABs). The term ‘grain’ in the present work was applied to denote a crystallite bordered by a continuous HAB perimeter. Accordingly, a ‘fragment’ was defined as a crystallite delimited by any EBSD-detected boundaries (i.e. those having misorientations of at least 2°).

The microstructural study has revealed that ECAP-Conform promotes intensive microstructural refinement down to a mean grain size of ~0.3 μm. Further SPD results mainly in the enhancement of structural homogeneity.

Material strengthening during ECAP-Conform results from grain boundary evolution and is governed by three primary mechanisms, i.e. dislocation, LAB, and HAB hardening. At relatively low strains, all three mechanisms operate simultaneously and promote a relatively-high rate of strain hardening. With an increase in total strain, however, the dislocation density and grain-boundary density reach saturation levels, and, thus, the hardening efficiency of ECAP drops markedly.

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Microstructure evolution and mechanical behaviour of VT8M-1 titanium alloy during hot plastic deformation

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Recent studies show that one of the most effective ways to increase the mechanical properties of titanium alloys is to form ultrafine-grained (UFG) structures in them using methods of severe plastic deformation. At the same time, the behaviour of the UFG structure and mechanical properties during plastic deformation at elevated temperatures is of great interest, which is due to technological processes of shaping products from such alloys.

In this paper, studies of the evolution of the microstructure and the mechanical behaviour of the VT8M-1 alloy with the UFG structure in the process of hot plastic deformation were made. Deformation was carried out in the temperature range 650-800°C by uniaxial compression. Microstructural studies were carried out by raster and transmission electron microscopy.

It is established that the most significant conditional yield stress falls in the temperature range from 650 to 750°C. With a subsequent increase in the deformation temperature, a flow pad appears. Studies of the microstructure showed that the average size of the globules of a finely dispersed mixture of α + β phases in the temperature range 650-700°C does not change significantly. With a further increase in the deformation temperature to 750 and 800 °C, it doubles (up to 1 μm).

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