Correlation of Fractal Dimensionality with Physical and Mechanical Properties of Deformed Cu

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It is known that severe plastic deformations (SPD) allows to get materials with submicro and nano-structures [1, 2], possessing principally new complex of properties. However, physical principles of such materials creation haven’ t been exposed completely. On this account it is actually to explore the evolution of materials structure in the SPD processes. In material science, the fractal analysis and multifractal parametrization are actively used [3]. Because of deformed metals structures represent the kind of a self-similar set with grains and grain-boundary generates the network. Such network can be interpreted as the fractal image of the structure. As is generally known [4], such image is characterized by fractal or the so-called Hausdorff dimension which is an important quantitative description of the explored objects.

A basic hypothesis in this work is that the evolution of structure of the metals takes place in a self-similar manner by forming fractal structures that substantially simplifies description of structures [5]. Assuming that successive set of high-angular boundaries of crystallites, are prefractals of one and the same fractal, we have the following estimation for the area of the given set in the unit volume of metal: $S \sim d^{-\nu}$, where $d$ - characteristic size of the fragment, $\nu = D - 1$, and $D$ - fractal dimension of the set of lines on the plane crossing high-angular boundaries. In this case value of $D$ is within the limits of 1<$D$<2. If the sizes of elements of prefractals are distributed in a wide range, the dimension of fractal substantially differs from 2. If the sizes of elements are approximately identical, the dimension of fractal verges towards 2. On the self-similar stage of structural fragmentation of metals value of $\nu = \text{const}$ and as a result of aforesaid it lies between 0 and 1.

In this paper, the changing of fractal dimension of copper in SPD processes is explored and compared with physical and mechanical properties. It is shown that for the undeformed metals the stochastic network of boundaries of structural elements has the fractal dimension $D=1.60\pm0.03$. SPD leads to increasing of fractal dimension. Possible reasons of changes in fractal dimension of metal structures in the SPD processes are analyzed.


17 August

Oral session
Deformation Mechanisms and Their Transformation
Nanostructured materials usually have high strength but low ductility. To improve their ductility, we need to first understand their deformation physics. Past attempts to increase the ductility usually led to a decrease in strength. In this presentation, I’ll first present the deformation physics in nanostructured materials and how they affect the ductility. I’ll then present several strategies to tailor the structures and deformation physics of nanostructured materials with the aim of improving the ductility without sacrificing the strength. We have been able to simultaneously increase the *ductility and strength* using these strategies.
Invited report

Plastic Deformation Mechanisms of Fully Dense Nanocrystalline Material

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We present results on the fundamentals of plastic deformation in fully dense bulk nanocrystalline metals with a mean grain size between 10 and 30 nm prepared by an innovative and new combination of Inert Gas Condensation (IGC) and subsequent Severe Plastic Deformation (SPD) technique. The model system investigated is Pd (fcc) at the first stage and several single-phase Pd alloys. The experiments focus on mechanical tests using new testing equipment for miniaturized specimens with stress state conditions, strain rate and temperature (at room temperature and below) as the variables. The aim is to obtain a significantly improved database of materials behaviour for these alloys at this very small grain size, since this has previously only been explored only sporadically, and to elucidate and describe the microscopic mechanisms that mediate the deformation. Special emphasis will be addressed to \textit{in situ} investigation of mesoscopic effects in plastic flow of nanocrystalline materials, such as formation of shear bands and/or cooperative grain boundary sliding, and quantitative and qualitative fractography investigations. The obtained results are important not only for fundamental material science, but also allows to elaborate the principles of development of technically feasible nanostructured materials providing the highest level of strength and ductility of these materials. Several recent results will be discussed also in comparison with nanocrystalline materials prepared under different conditions.
Structural-Scaling Transitions and Mechanisms of Deformation in Meso- and Nanostructural Materials

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The combination of high hardness and plasticity in nanocrystalline materials is explained by pronounced scale phenomena related to the refining of grains and qualitative new behavior of grain boundary defects. Nanostructural materials represent special case of nonequilibrium metastable state, when the properties of polycrystalline systems depend on the collective behavior of grains, grain boundary defects and the latter reveal the increased energy. There is the key question: does the sharp borrow exist between conventional polycrystalline and nanocrystalline state under the pass of some critical grain size. There is also “thermodynamic” formulation of problem: does the transition from polycrystalline to nanocrystalline state have the features of the first kind of phase transition. Statistical theory of typical mesoscopic defects allowed the establishment of new class of critical phenomena – structural-scaling transitions - related to the collective properties of defect ensembles and to propose the explanation of properties of nanocrystalline state. Specific feature of structural-scaling transition is the existence of additional order parameter – structural scaling parameter, that depends on the scale characteristics of material with defects and plays the role of the “effective temperature” of non-equilibrium state induced by defects. Statistically based phenomenology was developed as generalization of the Ginzburg-Landau approach and allowed one to establish the qualitative different dynamics of structural-scaling transitions in the terms of collective modes of defects that are characteristic for quasi-brittle (blow-up dissipative structures), ductile (auto-solitary waves)and fine grain (breathers) state. These modes, being generalized variables of mentioned states, determine the spectrum of thermodynamic and dynamic properties of materials. The understanding of links between the types of collective modes and physics and mechanics of characteristic material states allowed the explanation of the transitions between quasi-brittle, ductile and fine grain states, the role of scale characteristics of materials in these transitions, the link of dynamics of these modes in relaxation properties and failure. It was shown that the transition to the nanocrystalline state leads to the qualitative change of the type of collective modes in the ensemble of grain boundary defects: the transition from auto-solitary waves, providing the momentum transfer
under the plastic (yield) flow, to the breather ensemble – spatially localized finite-amplitude modes of defects to form in the material the “nonequilibrium dislocation lattice”, that changes qualitatively the symmetry, thermodynamic and kinetic (relaxation) properties of system. The transition from polycrystalline to the fine grain state in the terms of collective modes is accompanied by the degeneration of collective orientation modes that has the structural image as localized shear areas. The formation of “nonequilibrium dislocation lattice”, degeneration of collective orientation modes of defects changes the mechanisms of momentum transfer (the violation of the Hall-Petch law) and diffusion. The method of characterization of materials state in terms of the “effective temperature” was proposed using the generalization of the fluctuation-dissipation theorem for out-of-equilibrium states.

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Oral report

X-ray Diffraction Analysis of Planar Faulting in SPD-Metals

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Recent investigations of nanocrystalline materials showed an increased appearance of twin boundaries especially when the nanosized grain structure has been produced by methods of Severe Plastic Deformation (SPD), but has also been observed after the deformation of nanocrystalline material produced by classical methods such as inert gas condensation [1, 2]. This indicates that deformation twinning gets an important deformation mechanism below a critical grain size even for high stacking fault energy materials. One of the first models of deformation twinning suggests that the critical stress increases slower with decreasing grain size for moving twinning partials than for lattice dislocations [3]. Experimental data for stacking fault energies in different metals and alloys [4], however, do not support this model. A more sophisticated model suggests that an optimum grain-size range exists in which the critical stress for the nucleation of deformation twins is lowest [5, 1]. Zhu et al. [1] model the creation of large deformation twins in copper produced by high pressure torsion (HPT) schematically, showing how a leading and a trailing partial dislocation forms a deformation twin.

The X-ray Line Profile Analysis (XPA) has been applied successfully in analysing the microstructure of nanostructured materials in numerous experiments [6-10]. With this method the arrangement and density of the dislocations as well as the grain/subgrain-size and its distribution can be characterized quantitatively. Further improvements enable the verification of planar faults such as stacking and twin faults in parallel to the hitherto parameters. Numerous diffraction patterns were calculated for intrinsic and extrinsic stacking faults due to the theoretical description and have parametrized as function of the density and type of the planar faults. A whole profile fitting procedure, previously worked out for determining the dislocation structure and crystallite size distributions, is extended for planar fault by including these parameter into the evaluation algorithm [11].

Using this method Cu, Ni and Ag representing materials with very different stacking fault energies have been subjected to high pressure torsion (HPT) and investigated by XPA. The results of these investigations will be compared with those from the actual literature, in terms
of the defect structure – dislocations and stacking/twinning faults –, grain size and mechanical properties.

We give a brief overview of theoretical models [1-6] describing physical mechanisms of plastic deformation and fracture in bulk ceramic nanocomposites and nanocrystalline metals. The special attention is paid to theoretical models that describe (a) heterogeneous generation of partial dislocations and deformation twins at grain boundaries (GBs); (b) stress-driven GB migration; (c) generation of lattice and GB dislocation loops by nanoscale shear events; (d) heterogeneous generation of nanocracks/nanovoids at GBs and their triple junctions; (e) the effects of misfitting inclusions on crack growth; and (f) competition between nanocrack generation and diffusional mass transfer at GBs and their triple junctions in bulk ceramic nanocomposites and nanocrystalline metals.

In particular, it is theoretically shown that formation of anomalously wide stacking faults between partial dislocations in nanocrystalline Al is directly caused by high stresses but not by small grain size as was supposed in previous models. Overlapping stacking faults form thick lamellae of deformation twins. When their generation occurs in the stress field of GB disclinations, it may be characterized by the absence of any energy barrier under reasonable values of external stress. Growth of twin lamellae in thickness needs some increase in the external stress, thus causing strain hardening of nc-metals. A disclination model of stress-driven GB migration predicts two main regimes of the migration process: a stable regime under relatively low stress values, when propagation of GBs is determined by the level of the external stress, and an unstable regime under relatively high stress values, when GB migration does not depend on the stress level. Theoretical estimates show that stress-induced GB migration can substantially contribute to plastic deformation of nanocrystalline materials. A theoretical analysis of the generation of nanocracks at GB disclinations in a nanocomposite material demonstrates that the nanocrack can change its direction at the nanoinclusion/matrix interface or propagates straight into the bulk of the nanoinclusion. The probability of nanocrack generation increases near the nanoinclusions with negative (compressive) dilatation eigenstrain. The decrease in size of a nanoinclusion diminishes the probability of nanocrack
growth along the interface, if the eigenstrain is negative, and increases this probability, if the eigenstrain is positive (tensile). It is also shown that GB diffusion decreases the magnitude of the stresses of dislocations accumulated at or near triple junctions in the course of plastic deformation in nanocrystalline materials. This leads to a shrinking of the length interval at which GB nanocracks formed at triple junction dislocations are favored to grow. As a result, the plastic strain at which such nanocracks start to form increases. The suppressing effect of diffusion on nanocrack generation is especially pronounced at high temperatures and small strain rates.
Invited report

The Contribution of Phase Transformations, Dynamic Segregations and Heterophase Boundaries on the Grain Size Refinement by SPD

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During the last decade, severe plastic deformation (SPD) processes have been extensively applied to a large number of metals and alloys to reduce their grain size and improve their mechanical strength or superplastic properties. Materials processed by SPD typically exhibit a grain size in a range of 100 nm to 1 μm and only in few cases below 50 nm. Some of the mechanisms involved in these later cases would be reviewed in this paper. A special emphasis would be given on the role of alloying elements, heterophase boundaries and phase transformations that could occur during SPD. These features have been investigated thanks to transmission electron microscopy and three-dimensional atom probe in various materials processed by torsion under high pressure. In a 6061 aluminum alloy, the dynamic segregation of Mg and Si along dislocation cell boundaries during SPD seems to play a key role in the grain size reduction. In pearlitic steel, free carbon atoms resulting from carbide decomposition exhibit a very similar behaviour. In multi-phase materials (for instance Cu-Cr and Cu-Fe composites), it is shown that heterophase boundaries help to reduce the grain size down to 50 or even 20 nm. It is worth noticing, that some non-equilibrium intermixing occurred in these materials during SPD which gives rise to an excellent combination of high strength and ductility.
Grain Boundary Phase Transitions in Bulk Nanostructured Materials

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The nature and behavior of grain boundary (GB) phases is very important since they can control strength, plasticity, resistivity, grain growth, corrosion resistance etc., especially in nanocrystalline materials. For nanocrystalline Al-based light alloys extremely high plasticity has been observed in restricted temperature and concentration intervals close to the solidus line. This phenomenon is not fully understood. It can be explained by formation of GB phases not included in the bulk phase diagram. Therefore, the structure of GB phases, as well as thermodynamic conditions for their existence has to be carefully studied. In this work the structure and composition of GBs and GB triple junctions in Al–5 at. % Zn polycrystals annealed in the temperature region above and below the bulk solidus line were studied by high-resolution electron microscopy and analytical transmission electron microscopy. Evidence has been obtained that a thin layer of a liquid-like phase exists in GBs and GB triple junctions slightly below the bulk solidus line.

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Ultrafine Grained Structure Development and Deformation Behaviour of
Aluminium Processsed by CGP

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The intense plastic straining technique, named constrained groove pressing (CGP) was used to impose the severe plastic deformation to pure aluminium of commercial purity. The principle of CGP is that a material is subjected to the repetitive shear deformation under plain strain deformation condition by utilizing alternate pressing with the asymmetrically grooved die and flat die constrained tightly by the cylinder wall. In this experiment the Al specimens were pressed with maximum of 32 pressings and cumulative effective strain $\varepsilon_{\text{eff}}$ becomes $\sim 9.3$ throughout the sample. The impact of the repeated groove pressing was investigated upon microstructure changes with transmission electron microscopy of thin foils. The changes in mechanical properties measured by tensile tests and by hardness were related to microstructure development. Hardness values measured in different sites of the deformed plates indicated some non-homogeneity in strain distribution in all specimens regardless the number of pressings performed. The formation of banded grains with subgrain structure of submicron size was common feature observed in all deformed plates subjected to different passes. Small nuclei of polygonal grains with high angle boundaries in the deformed substructure have been observed already after two pressings. The high angle boundary fraction increases continuously with equivalent strain. The substantial impact of strain upon the tensile strength increase was observed after the first pressings. The further increase of the tensile strength with increasing number of pressings passes was less marked. Only small enhancement of the strength was detected with increasing number of the pressings. The concomitant loss of ductility was observed in all CGP processed plates. The thermal treatment of deformed plates was applied to recover the plastic ability of structure.
Oral session

Modeling of Nanostructured Materials
Invited report

**Finite Element Simulation of Severe Plastic Deformation Methods**

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In recent years, severe plastic deformation (SPD) processing was developed as a new method of manufacturing bulk specimens having ultrafine grained microstructure or nanostructure. Equal channel angular pressing (ECAP) is a unique and relatively simple metal forming process to the other metal forming processes, but still complex under coupled effects with multi-factors, such as geometric factors, material factors and processing conditions. Investigating the plastic deformation behavior in the deformation zone during ECAP is very significant for predicting the metal flow, microstructural evolution, controlling the quality of deformed workpiece and optimizing the ECAP processing. In this chapter simulation of ECAP using various numerical methods (finite element method, upper bound theorem and slip line field theory) are reviewed. The papers from literature analyzing ECAP and modified ECAP processing by the finite element method (FEM) are summarized and compared in terms of software, mesh size, dimension and analyzed results. It can be found that not only stress, strain, velocity, temperature fields and fracture tendency but also their distributions as a function of the effects of the processing variables (speed, size, friction, die geometries, temperature etc.) can be correctly predicted. A state-of-art for microstructural consideration using dislocation cell model is introduced for the prediction of evolution of microstructures, grain (dislocation cell) size and misorientation angle and their distributions.

![Figure 1. Mesh system of 3-dimensional: initial mesh, deformed mesh and strain distribution](image)

"Figure 1. Mesh system of 3-dimensional: initial mesh, deformed mesh and strain distribution"
Figure 2. High pressure torsion simulation


Oral report

Analysis of the Deformation Behavior of Nanostructured Metals by Means of Kinetic Modeling

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Kinetic modeling is one of strong tools successfully applied to analyze the peculiarities of the evolution of microstructure, deformation behavior and deformation mechanisms in bulk nanostructured materials [1, 2]. As is known [3], the deformation behavior of bulk nanostructured materials is characterized by rather specific features, among which absence of strain hardening [4], simultaneous exhibition of high strength and ductility [5], low-temperature or high-rate superplasticity [6] etc. should be mentioned. The causes of the mentioned peculiarities are unusual structural states which form in these materials as a result of application of the severe plastic deformation (SPD) technique. An extremely small grain size of tens and hundreds of nanometers, a sizeable volume fraction of the grain-boundary phase, non-equilibrium high-angle grain boundaries with a high density of introduced grain-boundary dislocations are typical of the formed nanostructures. As a result processes of dynamic recovery and recrystallization [4], grain-boundary diffusion [7], twinning with emission of partial dislocations by grain boundaries [8] etc. activate.

This report presents the results of development of the kinetic dislocation model and its application for analyzing the processes accompanying the plastic deformation of bulk nanostructured metals. Cu with fcc lattice and Ti with HCP lattice were chosen as materials for study. Experimental true deformation curves typical of the mentioned metals after SPD were investigated.

Contribution of various possible plastic strain mechanisms into formation of these curves was analyzed. The cases of activation of conservative and non-conservative crystallographic slip, crystallographic twinning and vacancy migration were considered. Conclusions about the influence of a crystalline lattice type, strain rate, temperature, accumulated strain degree on the character of the evolution of microstructure, deformation behavior, active deformation mechanisms were made.

Grain boundaries (GBs) are primary crystal lattice defects in bulk nanostructured materials (BNMs). Numerous experimental studies of the structure and properties provided an evidence that the GBs in BNMs produced by severe plastic deformation (SPD) have a specific, nonequilibrium structure caused by extrinsic dislocations and disclinations introduced during deformation [1,2]. Earlier we have shown that dislocation / disclination models of nonequilibrium GBs are capable to account for the elastic strains, excess energy and volume expansion observed in SPD-nanomaterials [2]. However, understanding the role of nonequilibrium GB structure in the properties of BNMs such as strength, plasticity, diffusion etc. requires a use of atomistic simulation methods.

In the present paper recent results of molecular dynamics simulations of nonequilibrium GBs will be reviewed. Two important issues have been addressed by these simulations: 1) the formation of interfacial cracks from GB junction disclinations and competing stress release mechanisms and 2) the effect of extrinsic dislocations and disclinations on the GB diffusion coefficient in BNMs.

The first issue, relaxation of stress concentrations, has been studied by means of a combination of atomistic computer simulations and linear elastic theory analysis for a bicrystalline cylinder of Ni and Ti containing a negative wedge disclination. The main finding of this research is that for both metals there exists a critical disclination strength, $\omega_c$, above which an interfacial microcrack is nucleated near the disclination core. This strength decreases with the cylinder radius and depends on the type of GB. These calculations allowed the estimates of the upper limit for the strength of discinations that can exist in bulk nanostructured metals: about $6^\circ$ to $4^\circ$ for the grain size in the range 100 to 200 nm. These data are in good agreement with direct observations of disclinations in heavily deformed metals [3]. In Ti alternative competing relaxation mechanisms such as a new grain formation and disordering of the disclination core were observed. These processes retard the crack formation that results in an increase of the critical disclination strength. An example of an amorphized disclination core in a (1128) tilt boundary of a 20 nm diameter nanowire at $T = 600$ K is
presented in the figure. The disclination strength in this example is \( \omega = 18.9^\circ \), slightly lower than the critical strength \( \omega_c \).

The second issue has been studied in terms of the concentration of vacancies in the stress fields of extrinsic grain boundary dislocations (EGBDs) and disclinations. An EGBD was inserted into a \( \Sigma = 5 \) (310) special tilt boundary; positive and negative wedge disclinations with the strength 5° were inserted into a cylinder of 100 nm radius containing either a \( \Sigma = 5 \) (210) tilt boundary of a general twist GB. Vacancy formation energy, \( E_v \), in all crystallographically non-equivalent sites of these GBs has been calculated for GB periods located on different distances from the EGBD and disclination core. A significant effect of the stress fields of defects on \( E_v \) was observed. Analysis of the results shows that both EGBDs and disclinations can cause up to 2 orders of magnitude increase of the average vacancy concentration in the GBs of nanomaterials.

Oral report

**Computer Simulation of Low-Angle Grain Boundaries**
**During Plastic Deformation**

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The computer model based on the method of molecular dynamics for sub-boundary nucleation and formation is developed. This model takes into account elastic interaction of dislocations between each other and with disclinations and processes of dislocations generation, annihilation and sink. Regularities of formation of broken sub-boundaries in elastic field of disclination during plastic deformation are investigated. Two typical cases are investigated: the first, when external stress field $\sigma_e$ which provide plastic deformation is more than maximal value $Dw/2$ of stress field of disclination ($\sigma_e > Dw/2$) and the second, when $\sigma_e < Dw/2$. It is shown that in both cases sub-boundaries nucleate along the line going through the disclination perpendicular to dislocations sliding planes. At $Dw/2 < \sigma_e$ dislocation walls appear and destroy right here by dislocations flow. As a result the rare sub-boundary consists of a few quasi-equilibrium walls appear. This sub-boundary is unstable and destroys at material unloading ($\sigma_e = 0$). At $Dw/2 > \sigma_e$ stationary narrow sub-boundary formation is observed which remain at $\sigma_e = 0$. The average misorientation of these sub-boundaries $\theta_{st}$ is about one half of disclination power. The accommodative mechanism of sub-boundaries formation is investigated within the framework of this model. Selected parameters of dislocation kinetics are corresponding to the absence of dislocations multiplication at the established value of external stress and absence of disclinations. It is shown that linear increase of disclination power $\Omega$ during deformation leads to the appearance of dislocation sources in the body of grain. At the same time sub-boundary misorientation $\theta_{st}$ linearly increase with deformation also and is equal to about one half of disclination power ($\theta_{st} \cong \Omega/2$)(fig.1).
The formation of sub-boundaries in model polycrystals consist of several grains in self-consistent statement of problem is analysed. In this case disclinations appear in grain boundary triple junctions just during plastic deformation due to accumulation of misorientation misfit dislocations in grain boundaries. It is shown that in every case the appearance of sub-boundaries is due to different rates of dislocations loops nucleation in the elastic field of disclination and hence asymmetry of value of strain accumulated in grain regions to the left and right sides from sub-boundary. It is shown that sub-boundaries formation lead to equalizing of strain rates throughout the volume of grain.
Oral report

Microstructure and Texture Evolution at ECAP Described by a Combined Viscoplastic Selfconsistent - Disclination Model

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The deformation behaviour of f.c.c. metals is studied using the polycrystal model, viscoplastic self-consistent (VPSC), which has been updated to account for the grain shape effect and grain-to-grain interaction [1], important for large strain behavior. Recently this approach was coupled with a disclination criterion for substructure formation and grain subdivision [2] to consider its influence on texture evolution during the large plastic straining imposed by the equal channel angular pressing (ECAP) process. Several models for deformation history (a view of velocity gradient tensor) in the channels’ intersection area have been employed for approximating the complex plastic flow during ECAP. ECAP texture predictions from these models in combination with VPSC were compared against measurements obtained by neutron diffraction [3]. It is demonstrated that the coupled subgrain-VPSC model provided better agreement with the experimental texture data regardless of the form of the velocity gradient tensor. A multi-scale simulation scheme - a combination of the subgrain-VPSC model with a deformation history provided by 3D macromodeling by variation-difference method [4] - is shown to achieve the best agreement to experimental data.

Oral report

**Numerical Investigations of 3 Pass Accumulative Stress and Strain Distributions in Twist Extrusion Process for Ti-6Al-4V**

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Twist Extrusion (TE) process is a rather recently proposed method of Severe Plastic Deformation (SPD) in order to produce bulk nano-structured materials. On the other hand, finite element method (FEM) as a powerful mean to predict optimized mechanical and geometrical parameters has rarely been used for TE except in a few and limited papers [1,2].

In this study, the simulation of three consecutive clockwise twist extrusion setup (Fig. 1) with a twisted channel die (Fig. 2) of Ti-6Al-4V samples is presented using an explicit finite element method.

Ti-6Al-4V alloy is important due to high strength to weight ratio and resistance to corrosion. Moreover, this alloy is considered as one of the hard-to deform materials to achieve the flow behavior under the severe plastic deformation process. Twist extrusion of Ti-6Al-4V alloy were performed at 900°C. On the other hand, the difficulty of the process requests a deliberate perspective and prediction by simulation before carrying out any experimental procedure. Therefore a suitable constitutive equation is used to describe the behavior of Ti-6Al-4V at high temperature during the deformation. The flow of three simultaneous clockwise twists is presented using von-Misses stress and effective plastic strain contours at 4 different selected
elements alongside of the billet (Fig. 3). The positions of the maximum and minimum values of effective strain and von-Misses stress are determined and their profile histories are obtained. The maximum and minimum values of equivalent plastic strains at the end of three simultaneous clock wise passes are found to be approximately 2.6 and 1.2, respectively. The results show that the von-Misses stress and equivalent plastic strain are reached a maximum value at the end of the first pass and increased slightly to the end of second and third passes (Fig. 4). So the results show that in 2 and 3 passes, the values of stress and strain are not the integer coefficients of that of in one pass because the material is affected by the softening mechanisms in high temperatures during the deformation.


Special Oral Session for Young Scientists
(PhDs and PhD Students)
Oral report

**Radiotracer Investigation of Interface Diffusion in Bulk Nanocrystalline and Ultrafine Grained Materials**

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Nanostructured materials reveal often hierarchical microstructures. In nanocrystalline $\gamma$-Fe-Ni alloy produced by powder metallurgy, the nanosized crystallites were found to be clustered in micrometer-large agglomerates (see a schematic picture in Fig.1), with the grain boundaries between nanocrystallites and the interfaces between agglomerates revealing fundamentally different kinetic properties [1-3]. On the other hand, low-angle and high-angle boundaries typically co-exist in material after severe plastic deformation run. Diffusion investigations in such materials demand a special care. A complete and consistent model of diffusion in such a material was elaborated [2,3] that allows systematic experimental investigation of self- and solute diffusion in all possible kinetic regimes.

The radiotracer technique is applied for measuring grain boundary diffusion of Ni, Fe and Ag in the nanocrystalline Fe-40Ni alloy produced by powder metallurgy and of Ni and Fe in nanostructured copper prepared by equal channel angular pressing (ECAP).

The boundaries between nanograins in $\gamma$-Fe-Ni alloys were found to reveal diffusivities which are similar to those in their coarse grained counterparts. On the other hand, these are inter-agglomerate boundaries which show much enhanced diffusivities with significantly smaller activation enthalpies of self- and solute diffusion.

The penetration profiles for diffusion of Ni and Fe in ultra fine grained Cu prepared by severe plastic deformation revel two types of short-circuit diffusion paths with different kinetic properties, too. The majority of grain

![Figure 1. Schematic presentation of hierarchical microstructure of the bulk nanocrystalline Fe-Ni alloy](image-url)
boundaries in ECAP Cu shows diffusion rates similar to those of general high angle boundaries in coarse grained Cu.

The origin of fast diffusion paths is discussed with respect to a possible contribution of non-equilibrium grain boundaries produced by severe plastic deformation.

Oral report

Fatigue Behaviour of UFG Al: Influence of the ECAP Route and the Magnesium Allo ying Content

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The fatigue behaviour of ultrafine-grained (UFG) Aluminium-Magnesium alloys AlMg0.5, AlMg1, AlMg1.5 and AlMg2, produced by Equal Channel Angular Pressing (ECAP), has been investigated. For this purpose, total strain controlled fatigue experiments in the LCF regime and stress controlled fatigue experiments in the HCF regime have been performed. In comparison to the conventionally grain-sized (CG) condition, for all alloys an increase of the fatigue lives by several orders of magnitude has been observed with respect to the stress level. The cyclic deformation behaviour depends on the alloying content of the material: While the lower alloyed AlMg0.5 shows cyclic softening, a cyclically stable deformation behaviour is found for the higher alloyed AlMg2. This can be related to the difference in microstructural stability, which has been investigated by transmission electron microscopy and X-ray diffraction.

Interestingly, as it is shown in Fig. 1 the ECAP route A leads to a significantly higher fatigue life compared to the ECAP route B_C. Since no significant differences of grain size and grain

![Wöhler (S-N) plot of UFG AlMg1.5 after 8 ECAP passes with ECAP route A and B_C](image-url)
morphology could be found, the stronger <110> texture of the samples processed via route A is supposed to be the reason for the observed difference.

Also the influence of the number of ECAP passes will be discussed. It is shown, that a reduction of the number of ECAP passes from 8 to 4 leads to a much more pronounced softening and decreased fatigue life in case of AlMg0.5.

It was found, that the fatigue cracks of the UFG alloys were always stage I cracks, closely associated to the shear plane of the last ECAP pass. The cyclic deformation behaviour and damage mechanisms of the investigated alloys are discussed with respect to the microstructural changes.
Oral report

Superplastic Behaviour in a ZK60 Magnesium Alloy

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Several papers have shown the occurrence of superplasticity in magnesium alloys processed by ECAP [1] and the ZK60 alloy exhibits some of the largest elongations. An earlier report [2] showed that elongations higher than 900% can be obtained in this alloy after processing by the \textit{EX-ECAP process} [3] when testing at a temperature of 473 K in the strain rate range of $10^{-5}$-$10^{-3} \text{ s}^{-1}$. This study focuses on the characteristics of the superplastic behavior of the ZK60 alloy.

The previous study showed that an increase of only 20 degrees in the temperature caused the loss of the superplastic capability in the ZK60 alloy [2]. It is shown in the present study that significant grain growth occurs in this material at 493 K but grain growth is limited at 473 K thereby explaining the pronounced difference in the behavior at these two temperatures.

Another characteristic of the superplastic behavior of the ZK60 alloy processed by ECAP and tested at ~473 K is the occurrence of pronounced strain hardening which is observed in the first stage of deformation. This strain hardening is not readily noted when plotting nominal/engineering stress against nominal/engineering strain [2,4,5] but it becomes clear when the curve is transformed into true stress versus true strain as in a recent report [6]. This unusual hardening can be explained by grain growth during deformation. The present study shows the true stress-true strain curves for the ZK60 alloy tested under superplastic conditions and the grain structures at different stages of deformation that provide experimental evidence of grain growth during the hardening stage and dynamic recrystallization in the final stage.

It is also shown that the evolution of the strain rate sensitivity with the testing temperature exhibits excellent agreement with the elongation to failure. For lower strain rates the optimum testing temperature is 450 K and at higher strain rates the optimum temperature is increased to 473 K.


Oral report

Fatigue Behaviour and Damage Mechanisms of Ultrafine-Grained Ti-6Al-4V ELI Alloy for Medical Application

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Due to their excellent strength and corrosion characteristics titanium alloys and, in particular, the Ti-6Al-4V ELI alloy, are widespread as the materials for hard tissue replacement. It is well known that medical implants are subjected to high complex loadings, which lead to fatigue crack formation and failure of implants and, consequently, to repeated surgical procedure and traumas of patients.

Therefore, static and cyclic strength of the alloy are the most essential properties for hard tissue replacement and development of new generation implants requires their enhancement.

Many scientific investigations have been dedicated to the strength increase of titanium and its alloys and one of the perspective approaches to enhance strength characteristics is formation of nanocrystalline and ultrafine-grained (UFG) structures in metals and alloys by means of severe plastic deformation (SPD) [1-2]. With ECAP an UFG structure with an average grain size of 0.8 µm and an ultimate tensile strength rise by 40% are obtained for Ti-6Al-4V ELI alloy [3].

Since fatigue properties are very critical in case of medical application of the alloy this work has been devoted to investigation of fatigue lives and damage mechanisms depending on its microstructural states. Cyclic deformation behaviour of the UFG Ti-6Al-4V ELI alloy has been studied under strain and stress controlled conditions. Results of fatigue tests have been generally presented by the Coffin-Manson plot and the Wöhler (S-N) diagram (Fig. 1) and reflect enhanced fatigue behaviour of the UFG alloy. Fatigue tests have been supplemented by structural investigations showing different crack propagation behaviour in coarse-grained und UFG Ti-6Al-4V ELI alloys.
Figure 1. Wöhler S-N Diargams of UFG and CG Ti-6Al-4V ELI alloy

Oral report

Regularities of Nanocrystalline Structure Formation and Functional Properties of Long-Sized Samples of Ti-Ni Shape Memory Alloys

Using Electroplastic Deformation

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In our recent work, a nanocrystalline structure was obtained in Ti-50.0%Ni and Ti-50.7 %Ni shape memory alloys as a result of annealing after severe cold rolling (true strain e=1.9 – 1.7). Such structure allowed obtaining a record combination of the functional properties of Ti-50.0% Ni alloy: maximum recovery stress of 1400 MPa and maximum completely recoverable strain of 8 %. However, these results were obtained in a thin band (the cross-section area mach less than 1 mm²). To transform to larger cross-section, it is necessary to improve a technological ductility of samples. This aim was achieved using electroplastic deformation (EPD, i.e., plastic deformation under impulses of a high electrical current density). The EPD of Ti-50.7 % Ni alloy band having 2×8 mm cross-section increased the limiting accumulated (before rupture) true strain from 0.6 (after usual cold rolling) to 0.8-0.9 in the case of controlled current density. That allowed obtaining mixed amorphized and nanocrystalline structure directly after EPD (Fig. 1 a), and a nano-size grain/subgrain structure upon post-deformation annealing (Fig.1 b). That resulted in a better combination of the functional properties (maximum recovery stress and completely recoverable strain) as compared with the same properties of the same alloy annealed after usual cold rolling and contained mainly polygonized substructure.

In the case of EPD of Ti-50.7%Ni alloy under constant current value, a true strain value of 1.8 was reached. However, the deformation was accompanied by developed dynamic softening, coarser grain structure formation (Fig. 1 c) and deterioration of the properties.
This work was supported by the Agency on Science and Innovation, Russian Federation, project No. 02.442.11.7526.

Figure 1. Structures and electron diffraction patterns of Ti-50.7 \%Ni alloy directly after EPD, $e=0.8$ under controlled current density (a), after subsequent post-deformation annealing at 400 °C (b), and directly after EPD, $e=1.8$ under constant current value (c)
Oral report

Investigation of Hardening Behavior and Die Geometry on the Deformation Performance of Equal Channel Angular Pressing by Finite Element Analysis

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Severe plastic deformation methods are widely employed to obtain ultrafine grained microstructures. There are several processes which introduce severe plastic deformation to a material, of which, “Equal Channel Angular Pressing (ECAP)” is the most common one. Since ECAP is suitable for production of ultrafine grained bulk samples of a wide range of metals and alloys without porosity, there is a great potential for employing the method in industrial and commercial applications.

In this study, finite element method was employed to analyze the deformation performance in ECAP process. Numerical experiments were carried out in order to observe the effect of strain hardening behavior and die geometry on the process performance and deformation homogeneity. During the construction of the model, the process was considered to be isothermal, and to simplify the problem, plane strain condition was assumed. The numerical results showed that both strain hardening behavior and die geometry strongly affect the deformation homogeneity and process performance. The results were found to be in good agreement with those of the theoretical studies and other data in literature.
Oral report

Microstructure Evolution of Two-Phase Titanium Alloys During Warm Deformation

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Microstructure refinement caused by heavy deformation at a relatively low temperature improves the mechanical properties of titanium and its alloys. However, information on the microstructure evolution of lamellar microstructure of two-phase titanium alloys during deformation at low temperatures is insufficient.

This work was aimed to investigate the microstructure evolution and mechanical behavior of α+β Ti-6Al-4V and pseudo-β Ti-5Al-5Mo-5V-1Cr-1Fe titanium alloys during uniaxial compression at 800 and 600°C to a high strain of 70%. The microstructure was examined by using an optical microscope, a JEOL JEM-2000EX transmission electron microscope and a JEOL JSM-840 scanning electron microscope equipped with an EBSD-device.

Initial microstructure of the alloys was lamellar with the width of α-lamellae of 1.6 and 1.3 μm for Ti-6Al-4V and Ti-5Al-5Mo-5V-1Cr-1Fe alloys, respectively. Mechanical behavior of each material at 600 and 800°C is described by σ-ε curves with strengthening, softening and steady state flow stage. Microstructure refinement of alloys at 800°C was found to be associated with dynamic recrystallization within α-lamellae with the formation of high-angle boundaries after compression of 70%. At lower temperature shear deformation plays an important role in breaking down of α-lamellae and β-laths. In addition α-lamellae were found to be divided by transversal dislocation walls/subboundaries, which increased their misorientation by accumulation of dislocations. However, in some thick α-lamellae longitudinal deformation-induced boundaries were observed. After deformation to 70 % microstructure of Ti-6Al-4V was found to be refined to a grain size of about 0.7 μm and 1.3 μm after deformation at 600 and 800°C, respectively. Ti-5Al-5Mo-5V-1Cr-1Fe alloy had a microstructure with grain size of 0.8 and 2 μm after deformation at 600 and 800°C, respectively.

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Oral report

A Study for Multi-pass Equal-Channel Angular Extrusion with Split Dies for Manufacturing Bulk-Nano Materials

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Equal channel angular extrusion (ECAE) is one of the important methods to produce bulk-nano materials by accumulating plastic strain into the workpiece without changing its cross-sectional shape in the multi-pass processing. As this forming technique greatly refines the grain size, it is an effective method of obtaining materials with high strength and toughness.

Many researches have been done for the study of multi-pass ECAE process and material behaviour using conventional solid die. In the experiments using conventional solid die, the forming load is very high because of severe friction and dummy specimen.

In this study, split die was designed to reduce the forming loads and improve the accuracy of a material shape in the multi-pass ECAE process. Experiments with commercially-available pure aluminum alloy (AA1050) specimen of a square cross-section were carried out using the split die for route A and C, up to four passes. The forming simulation tool, CAMPform3D, based on the rigid thermo-viscoplastic approach and constant shear friction model, was currently used to simulate the multi-pass ECAE process through rotating three-dimensional model. The accuracy of finite element simulation results was verified through comparison of numerical data with experimental findings.

The effect of routes and number of passes was currently examined using finite element analysis in terms of the prediction of forming loads and deformation behaviour. Compression test and Vickers hardness test were carried out to investigate the variation of yield strength and hardness of specimens during the multi-pass ECAE process. Also the microstructure of specimens was investigated to find the reduction of grain size.