



*Advanced Research Workshop*



**Nano  
HP  
SPD**

# **Nanostructured Materials by High-Pressure Severe Plastic Deformation**

## **FINAL PROGRAM**



September 22-26, 2004  
Donetsk, UKRAINE

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## September 22, 2004

p.m.: Participants' Arrival and Registration.

## September 23, 2004

a.m.

8<sup>00</sup> – 9<sup>10</sup>: Participants' Registration.

### **Mechanical Behaviours**

(Chairman - Viktor Varyukhin, Yuntian T. Zhu)

9<sup>00</sup> – 9<sup>10</sup>: ARW opening remarks

9<sup>10</sup> – 9<sup>35</sup>: Cheng Xu<sup>1</sup>, Minoru Furukawa<sup>2</sup>, Zenji Horita<sup>3</sup> and Terence G. Langdon<sup>1</sup>,

*<sup>1</sup>Departments of Aerospace & Mechanical Engineering and Materials Science,*

*University of Southern California, Los Angeles, U.S.A.; <sup>2</sup>Department of*

*Technology, Fukuoka University of Education, Munakata, Fukuoka, Japan;*

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*Kyushu University, Fukuoka, Japan,*

***”The deformation characteristics of pure aluminum processed by equal-channel angular pressing”***

It is well-established that the process of equal-channel angular pressing (ECAP) leads to substantial grain refinement in polycrystalline metals and to the production of materials having unusual and useful properties including high strength and a capability for superplastic forming. This paper examines the fundamental deformation characteristics associated with the application of ECAP to samples of pure aluminum. Results are presented describing the development of a homogeneous microstructure in polycrystalline aluminum and the characteristics associated with the pressing of single crystals.

9<sup>35</sup> – 10<sup>00</sup>: Yan Beygelzimer, *Donetsk Phys. & Tech. Institute of the NAS of Ukraine,*

*Donetsk, Ukraine,*

***”Grain Refinement and Viscous Fracture of Metals at Severe Plastic Deformation”***

In the presentation I discuss several important questions that arise when producing ultrafine grain materials using severe plastic deformation techniques.

The main questions are:

- Is there a limiting grain size? or Can SPD give amorphous metals?
- If there is a limit, what does it depend on?
- What deformation processes are more effective for grain refinement?
- How to accumulate deformation without fracture?

In order to answer these questions, we propose and advocate some hypotheses, and (based on them) a model of fragmentation and fracture of metals.

**10<sup>00</sup> – 10<sup>25</sup>: Sergey A. Firstov, *Frantcevykh Institute for Problems of Materials Science of the NAS of Ukraine, Kiev, Ukraine,***

**“Ultimate structural States of Metals and their ultimate mechanical Properties”**

The stages of deformation hardening, the sequence of the structural types produced under severe plastic deformations, the ultimate values of yield stress are discussed. This sequence of stages is connected both with a change of the deformation substructure type and deformation hardening parameters. Specific sizes of the structural elements corresponding to the changes of hardening mechanism are estimated. The structural elements size  $d$  decreases with deformation  $e$  according to the equation:

$$d(e) = K / (s + k \cdot e)$$

where  $K$ ,  $k$  and  $s$  - are constants. This equation describes the experimental data better than the equation:

$$d(e) = d_0 \exp(-e/2).$$

Creation of ultra fine grained structures of deformation origin is followed by changing hardening mechanism. Two characteristic grain sizes could be distinguished when considering hardening mechanism changes. The first one ranges from 0.5  $\mu\text{m}$  to 1  $\mu\text{m}$  and causes the changes of  $m$  - exponent ( $s = kd^m$ ) from 0.5 to 1. The second one is connected with the transition to nanosizes. It has been also demonstrated that the severe plastic deformation results not only in the high strain hardening of the bcc metals, but also in decreasing of the temperature of the brittle-ductile transition.

For single - component materials transition to nanosizes is followed either by saturation of strength or even by strength decrease. As a rule, hardening of pure metals does not exceed  $1/150$  even after severe plastic deformations ( $e > 5$ ). At the same time, the strength of multi - component materials can be much higher. In particular, real strength of iron - based alloys can reach the level of  $1/30 - 1/40$ . Reaching such a strength level with using of volume hardening methods is quite a difficult task. It's much easier to create such structures using local deformation. That is why surface hardening and/ or formation of gradient structures seems to be perspective direction in elaboration of gradient nanostructured materials. Idea of using "useful" impurities and alloying elements, which can increase essentially density of grain boundaries and grain cohesion by grain boundary segregation, is discussed. An important direction of further investigations is chemicothermal treatment of nanostructured materials. In general, it is necessary to improve the technologies of controlling composition and structure of grain boundaries of nanomaterials.

**10<sup>25</sup> – 10<sup>40</sup>: Ibrahim Karaman<sup>1</sup>; G. Guven Yapici<sup>1</sup>; Yang Cao<sup>1</sup>; Zhiping Luo<sup>2,1</sup> *Texas***

*A&M University, Dept. of Mech. Eng., College Sta., USA; <sup>2</sup>Texas A&M*

*University, Microscopy & Imaging Ctr., College Sta., USA*

**“Microstructure and Mechanical Behavior of Severely Deformed 316L Stainless Steel Polycrystals and Hadfield Steel Single Crystals”**

The present work focuses on the microstructural evolution and deformation behavior of AISI 316L austenitic type stainless steel and high manganese Hadfield steel. Equal channel angular extrusion (ECAE) equipped with a 90 tool angle is utilized. While, bulk 316L stainless steel bars are processed at temperatures of 450 C, 550 C, 600 C, 700 C and 800 C; Hadfield steel single crystals were extruded at room temperature. Microstructure and mechanical properties of successfully extruded billets are investigated using light microscopy, electron microscopy, and mechanical testing. X-ray analysis is conducted to monitor macro-texture evolution in 316L stainless steel while orientation imaging microscopy (OIM) is used for tracking micro-texture evolution in Hadfield steel single

crystals. High strength levels even in the 316L samples deformed at 800 C was attributed to the relatively high volume fraction of nanoscale deformation twins formed during ECAE. Observed tension/compression asymmetry in the yield strength values and strain hardening was attributed to the deformation induced directional back stress. Activation of twinning at such high temperatures (0.65Tm) was attributed to the effect of the high stress levels on the stacking fault energy. The goal is to produce desired end microstructures where deformation twinning is stabilized at high temperatures forming nanostructured AISI 316L stainless steel, leading to improved mechanical properties. Single pass extrusions of Hadfield single crystals demonstrated that twinning was the main deformation mechanism. One objective of this study is to see the effect of initial orientation and severe deformation on the activation of mechanical twinning in single crystals and on the resulting mechanical properties.

### Coffee-Break

11<sup>00</sup> – 11<sup>25</sup>: **Amiya K. Mukherjee, Nathan Mara, Alla V. Sergueeva**, *Department of*

*Chemical Engineering & Materials Science, University of California, One Shields Avenue, Davis, USA,*

#### **”Elevated Temperature Deformation Characteristics”**

High strain rate and/or low temperature superplasticity was observed in 1420 aluminum alloy and in Ni<sub>3</sub>Al. The enhanced plasticity demonstrated in these materials is attributed to a refinement in grain size to the nano or near-nano range through the use of High Pressure Torsion (HPT) prior to elevated temperature tensile testing. Grain sizes after HPT were approximately 100 nm for the 1420 aluminum alloy, and 50 nm for Ni<sub>3</sub>Al. Tensile testing of the alloys was carried out at strain rates up to 10<sup>-1</sup> and temperatures up to 725°C. Tensile curves typically exhibit high flow stresses and a degree of strain hardening that cannot be adequately described by conventional recovery mechanisms such as grain growth. The phenomena of cooperative grain boundary sliding, grain rotation, and grain boundary migration may play a role in explaining this difference between the flow stress observed in nanocrystalline and microcrystalline microstructure. Room-temperature TEM *in-situ* straining experiments reveal little dislocation activity, and no evidence of dislocation pile-ups. It is suggested that the dominant deformation mechanism for elevated temperature plasticity in this study is grain boundary sliding and rotation. (NSF-DMR-0240144)

11<sup>25</sup> – 11<sup>50</sup>: **Yu. M. Podrezov**, *Frantsevych Institute for Problems of Materials Science of the NAS of Ukraine, Kiev, Ukraine,*

#### **“Structural sensitivity of mechanical properties for nanocrystalline materials”**

During the last decade different techniques have been successfully used for the fabrication of nanostructure in various metals. This report presents several results from recent investigation of structural sensitivity of mechanical properties of this class of materials. Consideration is given to the distinction between nanocrystal strengthening and Hall-Petch grain size strengthening which varies linearly with  $d^{1/2}$ . The important role of grain boundary structure in the strengthening formation of nano materials is emphasized. The base elements of segregation theory is proposed to use for improvement of structure and mechanical properties of nanocrystalline materials.

Static and dynamic recoveries are the main reasons limiting the minimal size of structural elements under deformation in high deformed materials. The postulate of mechanics that deformation stress being achieved in a material at repeated deformation corresponds exactly to deformation stress achieved in it at unloading moment at initial deformation does hold well for materials which have not susceptibility to a recovery process. But this postulate does not hold for materials which have inclination to recovery. Structural relaxation proceeding

by the recovery mechanism both during plastic deformation and during unloading causes loss of strengthening in high deformed materials. At the initial stage of repeated deformation the recovered cell structure interacts with moving dislocations in a special way. In the micro-deformation level hardening stress is practically independent on previous deformation degrees in wide interval of deformation. The usual increase of strengthening with rise of deformation is observed only from the yield point.

The critical deformation degree  $\epsilon_c$  characterizes the beginning of considerable activation of the recovery process. The change of mechanical behavior near the critical deformation  $\epsilon_c$  was observed in high deformed Fe-Armco after ECA pressure. The growth of the fracture toughness for specimens with cracks introduced into the plane perpendicular to the plane of deformation was shown, and the decrease of the fracture characteristic for specimens with cracks introduced into the plane parallel to the plane of deformation was observed. The increase of the deformation degree ( $\epsilon > \epsilon_c$ ) promotes the change of the failure mechanism as follows: quasi-cleavage  $\rightarrow$  quasi-cleavage with delamination.

**11<sup>50</sup> – 12<sup>15</sup>: Nobuhiro Tsuji**, *Dept. of Adaptive Machine Systems, Osaka University, Suita, Japan*,

***“Unique Deformation Behaviors of the Ultrafine Grained Aluminum Alloys Fabricated by Accumulative Roll Bonding”***

The accumulative roll-bonding (ARB) is a severe plastic deformation process using rolling. It can produce bulky sheets filled with the ultrafine grains whose grain size is smaller than 1 micrometer in various kinds of metals and alloys. Subsequent annealing can change the grain sizes of the ARB processed materials continuously, which enables us to clarify the various properties of the ultrafine grained materials systematically. The author has been clarifying the mechanical properties, i.e., strength, ductility, and fatigue, of aluminum alloys systematically as a function of the grain size. Quite unique deformation behaviors of the ultrafine grained materials, such as yield-drop-like phenomenon in pure aluminum and increasing ductility with increasing ARB strain in Al-Fe-Si alloy, will be introduced in the keynote talk.

**12<sup>15</sup> – 12<sup>20</sup>: P. Gabor, N. Gupta, H. J. Maier, I. Karaman**, *LWK, University of Paderborn, Germany*

***“Fatigue Behavior of Ultrafine Grained Copper”***

In recent years, ultrafine grained materials have received increasing interest because of the superior combination of high strength and ductility. Although there are several recent studies that have focused on the monotonic properties, only few ones addressed fatigue behavior of these materials. Thus, the present study focuses on the cyclic stress-strain response and fatigue life of ultrafine grained copper obtained by equal channel angular extrusion (ECAE). Microstructural analysis using scanning and transmission electron microscopy was employed to shed light on the governing microstructural mechanisms.

Oxygen-free high-conductivity copper was ECAE-processed using routes 8A, 8E and 16E. Dog-bone shape samples with a rectangular cross section of 2 mm x 3 mm and a gauge length of 8 mm were machined from the 25.4 mm x 25.4 mm cross-section ECAE bars. The samples were fatigued (a) in the as-ECAEed condition and (b) after an additional heat treatment (170 $\pm$  C, 2h). All fatigue tests were conducted in symmetric push-pull loading in total strain control.

The microstructural analysis revealed an ultrafine grain structure with a high density of dislocations in all the samples. The annealed specimen showed a bimodal structure with a uniform density of dislocation in the large grains.

The samples in the as-ECAEed condition showed Masing behavior and stabilized rapidly in incremental step tests (IST) but displayed some deviation from Masing behavior in conventional low-cycle fatigue tests. By contrast, the annealed specimens showed perfect Masing behavior in both IST and constant amplitude test.

The LCF test data revealed a shorter fatigue life in the as-ECAEed samples as compared to the annealed ones. This is attributed to the bimodal structure, which deflects short fatigue cracks. Thus, a tortuous crack path results and overall small crack growth rate is decreased. The effects of the actual ECAE process routes on fatigue behavior will be discussed, and the ramifications of the results obtained with respect to optimization of ultrafine grained materials will be addressed.

p.m.

## Mechanical and Thermal Properties

(Chairman – Ruslan Z. Valiev, Michael J. Zehetbauer)

14<sup>00</sup> – 14<sup>25</sup>: **Viktor Varyukhin**, *Donetsk Phys. & Tech. Institute of the NAS of Ukraine, Donetsk, Ukraine*

### ***“High Pressure Effects and Large Plastic Deformation”***

Donetsk physics and technology institute of the NASU has more than 35-years experience of utilization of high pressure liquid in metal forming technologies. In the institute it was developed following processes: hydroextrusion, hydromechanical forging, hydrostatic drawing, etc. Here technology plasticity of metals increase due to high pressure liquid guarantee both low friction and high level of hydrostatic pressure in a deformation enclosed area.

New methods of SPD based on high pressure liquid using developed in the Donetsk Physics and technology institute of the NASU are analyzed in the report. They are bale hydroextrusion, equal channel multiangular extrusion and twist hydroextrusion. It is shown that these methods allows to perform UFG structure in different materials. The materials performed are characterized by both high strength and high ductility.

14<sup>25</sup> – 14<sup>50</sup>: **Terry C. Lowe**, *Los Alamos National Laboratory, Los Alamos, USA*,

### ***“Comparison of Alternative Routes to Producing Bulk Nanostructured Metals”***

Bulk nanostructured metals can be produced by a growing number of methods. In this work the most recently developed methods are summarized and compared. Limitations and unique differentiating characteristics are identified to suggest the most appropriate applications of alternative nanomaterials processing methods.

14<sup>50</sup> – 15<sup>15</sup>: **K.T. Hartwig and S.N. Mathaudhu**, *Texas A&M University, Department of Mechanical Engineering, College Station, USA*,

### ***“Microstructural Refinement of Bulk Nb and Ta by Severe Plastic Deformation for Composite Superconductor Applications”***

Pure niobium and pure tantalum are used in composite Nb<sub>3</sub>Sn superconductors as a precursor filament in the case of Nb for Nb<sub>3</sub>Sn, and as a diffusion barrier to protect stabilizer copper from tin contamination. Because both Nb and Ta are stronger and work harden at a higher rate than Cu (the matrix material for Nb<sub>3</sub>Sn filamentary conductors), they may not co-deform well with pure annealed Cu. The tendency to co-deform well during multifilamentary superconductor wire fabrication depends on the microstructure of the three materials: a fine

uniform microstructure promotes better co-deformation behavior. The purpose of the work reported here is to develop highly refined and uniform microstructures in bulk Nb and Ta, to form these materials into superconductor precursor rod or sheet and retain the fine and uniform microstructure, to demonstrate that these materials will co-deform better than commercially available materials within a pure Cu matrix to form fine filament and thin diffusion barrier regions, and most importantly, to demonstrate that longer length and/or higher critical current density wire are possible with SPD processed materials. A considerable amount of work to date has shown that extremely fine grained and uniform microstructures can be achieved in bulk Nb and Ta via ECAE processing, which is the topic of this presentation. Results will be reported on processing 25 X 25 X 150 mm bars of cast pure Nb and Ta via multipass ECAE. Extrusions were performed in a right angle die at room temperature to strains in excess of 9. Severely strained material shows a substructure with features on the order of 50-100s of nanometers. The recrystallized grain size after four passes is less than 20 microns. The choice of which ECAE multipass route to use for development of a uniform recrystallized microstructure will be discussed. Comparisons to commercially processed Nb indicate that ECAE may be a viable method for manufacturing fine-grained low-texture high-homogeneity Nb and Ta for superconductor applications.

15<sup>15</sup> – 15<sup>30</sup>: **N.Ammannati**<sup>2</sup>, **P.Cavaliere**<sup>1</sup>, **E. Cerri**<sup>1</sup>, **P. Leo**<sup>1</sup>, **E.Martellucci**<sup>2</sup>; <sup>1</sup>*INFN-*

*Dept. of Ingegneria dell Innovazione, Engineering Faculty, University of Lecce, Lecce, Italy;* <sup>2</sup>*Research Centre Europa Metalli, Fornaci di Barga (LU),*

**“Microstructural and mechanical evolution of ultra-pure copper processed by ECAP”**

The mechanical and microstructural properties of ultra pure copper processed by Equi Channel Angular Pressing (ECAP) were analysed in the present work. The material under investigation was subjected to 1, 2, 3, 4 and 12 pressings with a rotation of 90° between each pressing (route Bc) and then prepared for mechanical testings and optical and Transmission Electron Microscopy in order to study the evolution of grain refinement of the induced nanocrystalline structure. After 4 ECA pressings the material started to exhibit large zones of equiaxed grains of dimensions lower than 300 nm, after 12 passes the material appears with a uniform structure with very small grains with dimensions around 100 nm. Tensile tests were performed on the material after 4 and 12 pressings and then compared with the data corresponding to as-received one, the ultra-pure copper, analysed in the present study, revealed a yield strength of 340 MPa after 4 pressings and 465 MPa after 12 pressings. The High Cycle Fatigue (HCF) behaviour was studied for the material processed with 12 ECA pressings in order to analyse the effect of the strong grain refinement on the mechanical properties of the material. The stress amplitude corresponding to 107 cycles was 265 MPa. The fatigue properties of the material subjected to 12 pressings result more and more higher respect to those exhibited by the commercial ultra-pure copper.

15<sup>30</sup> – 15<sup>45</sup>: **V.V. Stolvarov**<sup>1</sup>, **R.Z. Valiev**<sup>1</sup>, **T.C. Lowe**<sup>2</sup> and **Y.T. Zhu**<sup>2</sup>; <sup>1</sup>*Institute of*

*Physics of Advanced Materials, Ufa State Aviation Technical, Ufa, Russia;* <sup>2</sup>*Los Alamos National Laboratory, Los Alamos, USA,*

**“Structure and properties of ultrafine-grained Ti-based alloys processed by ECAP”**

Ultrafine-grained bulk billets from various sorts of CP Ti and TiNi based alloy have been processed by ECAP and the following post-deformation or thermal treatment. Structure features and properties including mechanical, fatigue, corrosion, tribology and special functional ones were investigated. Advantages of ECAP processed alloys both before and after post deformation and annealing in comparison with coarse grained counterparts are demonstrated.

15<sup>45</sup> – 16<sup>00</sup>: **M. Berta, P.J. Apps, and P.B. Prangnell**, *Manchester Materials Science Centre, University of Manchester/UMIST*,

***“Effect of second phase particles on grain growth kinetics of ultrafine grained aluminium alloys produced by ECAE”***

The effect of fine and coarse second phase particles on the kinetics and mechanisms of recrystallization in severely deformed aluminum alloys has been investigated using a high purity binary Al-0.25wt.%Sc and a AA8079 alloy containing fine 20nm Al<sub>3</sub>Sc and coarse 2µm Al<sub>13</sub>Fe<sub>4</sub> particles, respectively. The alloys were deformed by ECAE to an effective strain of ten. Results have been compared to those obtained from a single-phase Al-0.13Mg alloy, deformed under identical conditions. The materials were annealed for 1 hour at several temperatures; the resulting structures were quantitatively analyzed using high resolution EBSD orientation mapping. The presence of the particles was found to affect the annealing behavior both directly by particle pinning and indirectly by altering the initial deformation structure. Coarse particles led to a more uniform ultrafine grain structure after deformation and uniform coarsening was observed, until the grain size reached that of the interparticle spacing. In comparison, the fine coherent Al<sub>3</sub>Sc dispersoids reduced the level of grain refinement and resulted in a more heterogenous deformation structure. However, the high density of particles proved to stabilize the ultrafine grained structure until temperatures around 500° C, when some discontinuous recrystallisation followed by abnormal grain growth was observed. This rapidly lead to a very coarse grains being formed.

## Coffee-Break

16<sup>20</sup> – 16<sup>45</sup>: **Hael Mughrabi, Heinz Werner Hoepfel and Martin Kautz**, *Institut für Werkstoffwissenschaften, Universität Erlangen-Nürnberg, Erlangen, Germany*,

***“Microstructural Aspects of Fatigue Strength and Ductility of Ultrafine-Grained Metals”***

In earlier work, it has been shown that the fatigue performance of ultrafine-grained (UFG) materials produced by severe plastic deformation via the equal channel angular pressing (ECAP) technique is generally enhanced in the high-cycle fatigue (HCF) regime but lowered in the low-cycle fatigue (LCF) range. In a total strain fatigue life diagram, this behaviour is easily understood in terms of the generally increased strength and the reduced ductility prevailing in UFG materials after ECAP. The microstructural reason for the deterioration of the LCF fatigue behaviour has been found to lie in a pronounced cyclic softening caused by dynamic grain coarsening even at low homologous temperatures and/or severe cyclic strain localization in macroscopic shear bands. Dynamic grain coarsening which has been shown to occur through a thermally activated process is more pronounced in high-purity than in commercial purity materials. Macroscopic shear banding occurs in high-purity and in commercial purity materials but differs in microscopic details.

Through an appropriate annealing treatment, the UFG microstructure can be rendered more stable, and the ductility can be improved at the expense of a loss of strength. As a consequence, an improved LCF behaviour can be achieved which is, however, not always as good as that of conventional grain size materials. The objectives of the current studies are to optimize the overall fatigue behaviour of different engineering metals and alloys through suitable heat treatments and to explore which microstructures produced by ECAP and subsequent heat treatment are most desirable for high fatigue performance.

16<sup>45</sup> – 17<sup>10</sup>: **Yu. R. Kolobov, Aleksei Lipnitskii**, *Institute of Strength Physics and Materials Science, SB RAS, Tomsk, Russia*,

**“Grain boundary diffusion and plasticity of polycrystalline and nanostructured metals and alloys”**

The regular features of nanostructure formation and mechanical properties in polycrystalline metals and alloys produced by severe plastic deformation in comparison with coarse-grained counterparts are considered. The diffusivities of substitution impurities were measured by direct experimental methods. It has been found that the grain -boundary diffusivities obtained for nanocrystalline (Ni) and nanostructured metals (Ni, Cu, Ti) far exceeds (by one to four orders) those of the coarse-grain counterparts. The probable physical causes for the above distinguishing features manifested by the diffusion in nanostructured materials are addressed [1]. An investigation was performed for a range of polycrystalline, fine-grained and nanostructured metals and alloys, e.g. Mo, Fe, Ni, etc., which allowed to reveal a hitherto unknown activation effect, i.e. initiation of grain -boundary sliding by diffusion impurity fluxes from the environment. Using the Mo(Ni) and Fe(Ni) systems by way of an example (the element in brackets stands for the diffusant), it was shown that the action of grain boundary diffusant fluxes from an external source might cause superplastic state to be realized in the material [2].

The role played by the diffusion-controlled processes occurring along the grain boundaries in the realization of low-temperature and/or high-rate superplasticity was investigated for nanostructured Ni and Al-Mg-Li-Zr(Sc) alloy. The role played by directional grain -boundary diffusant fluxes from the environment or an internal source in the manifestation of superplasticity in fine-grain and nanostructured metals and alloys is discussed. Characteristic features of failure development in nanostructured metals and alloys at ambient and high temperatures (under superplastic conditions) are considered. Characteristics of mechanical properties of nanostructured materials (resistance to creep, to fatigue, etc.) which are very important for practical applications are discussed.

17<sup>10</sup> – 17<sup>35</sup>: **Z. Mróz<sup>1</sup> and A. Baltov<sup>2</sup>**, *<sup>1</sup>Institute of Fundamental Technological Research, Warsaw, Poland; <sup>2</sup>Institute of Mechanics, Sofia, Bulgaria*,

**“Modeling hardening effects and grain size evolution in metals induced by severe plastic deformation”**

The present work is concerned with formulation of the phenomenological model specifying both deformational response and grain size evolution of metals in the process of large plastic deformation. It is assumed that the shear band pattern is developed with the accompanying recrystallization process governed by the stored free energy with its volumetric and grain boundary portions. The incremental relations are formulated specifying both hardening moduli and grain microstructure, in particular boundary porosity and shear band density. The resulting model predictions are compared with the available experimental data

17<sup>35</sup> – 17<sup>50</sup>: **D. Orlov<sup>1</sup>, Y. Beygelzimer<sup>1</sup>, A. Reshetov<sup>1</sup>, Yu. Milman<sup>2</sup>, D. Lotsko<sup>2</sup>, O. Sirko<sup>2</sup>, V. Voropaiev<sup>2</sup>**, *<sup>1</sup>Donetsk Phys.&Tech. Institute of the NAS of Ukraine, Donetsk, Ukraine; <sup>2</sup>Frantsevych Institute for Problems of Materials Science of the NAS of Ukraine, Kiev, Ukraine*,

**“Twist Extrusion As Tool For Grain Refining In Al-Mg-Sc-Zr Alloys”**

The influence of severe plastic deformation (SPD) by means of twist extrusion (TE) in a temperature interval of 20-300 °C and subsequent annealing on the structure and mechanical properties of Al-3Mg-0.3Sc (wt. %) alloy additionally alloyed with Zr was studied.

The starting condition of alloys was ingots with the grain size about 100  $\mu\text{m}$  without additional thermal treatment. In ingots containing Zr in the amount of 0.1 wt. % and higher a precipitation of primary intermetallic  $\text{Al}_3(\text{Sc,Zr})$  particles of 1-2  $\mu\text{m}$  in size was registered.

TEM investigation of extruded samples has shown the formation in them of a very fine wrought structure that was somewhat different in the center and at the edges of the extrudants. In the layer at the extrudate edges of several millimeters in thickness a clear cellular structure with somewhat elongated cells has been formed. The average cross size of cells depended on the deformation temperature and was of about 1  $\mu\text{m}$  in the case of extrusion at 280-300  $^{\circ}\text{C}$  and of about 0.3  $\mu\text{m}$  in the case of extrusion at 20  $^{\circ}\text{C}$ . In the latter case a large amount of dislocations in cell body was observed. In the center of the extrudate areas with cellular structure co-existed with areas of structures with continuous bending of the crystalline lattice with a wave length of about 0.5  $\mu\text{m}$ .

TEM registered no secondary coherent  $\text{Al}_3(\text{Sc,Zr})$  particles after extrusion at temperatures up to 300  $^{\circ}\text{C}$ . These particles became visible after annealing extruded samples for 1 h at temperatures of 450  $^{\circ}\text{C}$  and higher. Still literary data show that at least after extrusion at elevated temperatures such particles do exist, but particles to 4-5 nm in size are invisible in TEM image due to their coherency. In extrudants manufactured at room temperature such particles are most probably absent, and a high level of their strength is formed by a superfine cellular structure together with strain fields from dislocations inside of cells.

Annealing has shown a rather good thermal stability of the cellular structure. After annealing at 400, 450, and 500  $^{\circ}\text{C}$  the average cross size of cells increased to 0.55  $\mu\text{m}$ , 0.7  $\mu\text{m}$ , and 1  $\mu\text{m}$ , respectively.

The results of measuring hardness and tensile characteristics of samples under investigation will be also presented in the report.

17<sup>50</sup> – 18<sup>05</sup>: Alexander Zhilvaev, *Naval Postgraduate School, USA*,

***“The Microstructural Characteristics Of Ultrafine-Grained Nickel”***

A detailed investigation was conducted to evaluate the microstructural characteristics in samples of pure nickel processed using three different procedures of severe plastic deformation (SPD): equal-channel angular pressing (ECAP), high-pressure torsion (HPT) and a combination of ECAP and HPT. Several different experimental techniques were employed to measure the grain size distributions, the textures, the distributions of the misorientation angles and the boundary surface energies in the as-processed materials. It is shown that a combination of ECAP and HPT leads both to a greater refinement in the microstructure and to a smaller fraction of boundaries having low angles of misorientation. The estimated boundary surface energies were higher than anticipated from data for coarse-grained materials and the difference is attributed to the non-equilibrium character of many of the interfaces after SPD processing.

**September 24, 2004**

a.m.

## **Nanomaterial Fundamentals**

**(Chairman – Terence G. Langdon and Zenji Horita)**

**9<sup>00</sup> – 9<sup>25</sup>: Yuntian T. Zhu, Los Alamos National Laboratory, Los Alamos, USA**

### ***“Deformation Mechanisms of Nanostructured Materials”***

Nanostructured materials deform via mechanisms not accessible to their coarse-grained counterparts. Partial dislocation emission from grain boundaries, stacking faults and deformation twinning may occur in metals such as Al, which does not deform by twinning in its coarse-grained state. In this presentation I'll discuss several deformation mechanisms in nanomaterials as well as their formation conditions.

**9<sup>25</sup> – 9<sup>50</sup>: Vladimir Kopylov<sup>1</sup>, Vladimir Chuvildeev<sup>2</sup>, <sup>1</sup>*Physico-Technical Institute of the National Academy of Sciences of Belarus, Minsk, Belarus; <sup>2</sup>Nizhny Novgorod State University, Nizhny Novgorod, Russia,***

### ***“Ultimate Grain Refinement by ECAP: Experiment and Theory”***

Equal-channel angular pressing (ECAP) is an effective method for controlling microstructure of metals. A multi-pass ECAP process is widely used to produce nanostructured materials. Nowadays, it is the only available method for obtaining nanostructures in the bulk. To achieve optimal grain refinement, the ECAP mechanics ought to provide correct control over the homogeneity of plastic deformation, required value of hydrostatic pressure, strain and strain rate during every pass of the ECAP treatment. The final characteristic size of the microstructure depends on numerous deformation parameters such as temperature, the intensity and route of repeated deformation as well as on the properties of the material itself such as crystallographic symmetry, number of slip systems, stacking fault energy, impurities, phase composition, etc.

For attaining profound grain refinement down to nanoscale level, most important is the development of optimal routes for microstructure controlling. At present, in multi-pass ECAP processing different technological routes (A, B<sub>A</sub>, B<sub>C</sub> and C) are used, which include different combinations of sample rotations about the longitudinal axis between successive passes and produce a variety of final microstructures. However, the number of such routes may be much larger, and their influence on grain refinement can be much more effective. The goal of this paper is to develop efficient multi-pass ECAP routes for microstructure control.

Microstructural investigations performed on a wide range of metals and alloys have revealed the fundamental relationships between the pattern of simple shearing during each pass and resulting structure formation. These results lead me to a definite conclusion that multi-pass ECAP is actually based on only two basic routes with respect to microstructure formation (hereinafter referred to as “microstructural routes”): one producing equiaxed grains and the other giving elongated oriented grains. On a wide range of metallic materials (Cu, Ni, Fe etc.) it has been demonstrated that applying each of these basic routes separately results in substantially different plastic properties and ductility of as-obtained nanostructural billets.

An optimal combination of two microstructural routes permits obtaining advanced mechanical properties: the yield stress increases by the factor of about 3 without sacrificing ductility. For example, 18-10 stainless steel after multi-pass ECAP possesses high strength ( $\sigma_B=1550$  ???,  $\sigma_{0.2}=1340$  ???) and enhanced plasticity ( $\delta=27\%$ ).

Thus, microstructure control for optimal grain refinement by multi-pass ECAP consist in the art of choosing an appropriate combination of two basic microstructural routes.

9<sup>50</sup> – 10<sup>15</sup>: **G.A. Salishchev** *Institute for Metals Superplasticity Problems of the Russian Academy of Sciences, Ufa, Russia,*

**“Mechanisms of submicron grain formation in titanium during large strain warm working”**

The “abc” warm deformation is a method for refining the grain structures of conventional alloys to submicron levels. In this paper the mechanisms by which grain refinement takes place over the extreme strain range attainable in severe deformation processing are studied, using an example of commercial pure titanium. Large strains were obtained during successive compression of sample along three orthogonal directions at 400°C. It is shown formation of submicrocrystalline structure with grain size of 0.3 μm. Grain refinement occurs through grains subdividing by deformation induced high angle boundaries (DIHAB) and interaction between them that leads to formation of submicron-grained structure and strengthening. Structure development on different scales of plastic flow (macroscopic, mesoscopic and microscopic) was studied.

10<sup>15</sup> – 10<sup>20</sup>: **Nina Noskova**, *Institute of metal physics, Russian Academy of science, Ural branch,*

**“Structural Features, Strength, and Mechanisms of Deformation of Nanocrystalline Materials”**

Results of recent original studies of structure and properties of nanocrystalline metals and alloys produced by severe plastic deformation. High resolution transmission electron microscopy, scanning electron microscopy, and in situ deformation in the column of an electron microscope were used to analyze the structures and the mechanisms of plastic deformation of nanocrystalline materials. An increase in the grain size to dozens of nanometers in nanocrystalline metals and alloys is accompanied by an increase in their strength and hardness.

Based on the results of the investigation of deformation of fcc, bcc, and hcp nanocrystalline materials in situ in the column of an electron microscope, we can apparently assume for all types of crystal structures that, as the nanograin size decreases in a nanocrystalline material, rotational deformation modes arise upon deformation by tension, which lead to the development of mesoscopic deformation shears because of their cooperative nature. In the hcp nanocrystalline titanium, unlike the fcc and bcc nanocrystalline materials, deformation microtwinning is also developed additionally.

## Coffee-Break

10<sup>50</sup> – 11<sup>15</sup>: **Zenji Horita<sup>1</sup>, Takeshi Fujita<sup>1</sup>, Kenji Kaneko<sup>1</sup> and Terence G. Langdon<sup>2</sup>,**

<sup>1</sup>*Department of Materials Science and Engineering, Faculty of Engineering,*

*Kyushu University, Fukuoka, Japan;* <sup>2</sup>*Departments of Aerospace & Mechanical*

*Engineering and Materials Science, University of Southern California, Los*

*Angeles, U.S.A.,*

**“Precipitation behavior in age-hardenable alloys after severe plastic deformation”**

Equal-channel angular pressing (ECAP) is a useful technique to produce ultrafine-grained structure in metallic materials through the introduction of severe plastic deformation. When the ECAP technique is combined with an aging process, it is also possible to control the

morphology and distribution of second-phase particles. In this study, ECAP is conducted on age-hardenable Al alloys and the precipitation behavior is examined in association with the subsequent aging process. The microstructures are examined using transmission electron microscopy. It is shown that severe plastic deformation introduces unusual phenomena in the precipitation process.

**11<sup>15</sup> – 11<sup>40</sup>: M. Shtern, V. Skorokhod, A. Ragulya, Frantcevykh Institute for Problems of Materials Science of the NAS of Ukraine, Kiev, Ukraine,**

**”Straining Path Effect on Coarsening Control and Pore Distribution During Sintering of Nano – Materials”**

The coarsening control, especially at the finishing stage of sintering of Nano – Ceramics, is one of key problems of nano – technology. Accompanying problem – is the control of pore size distribution, which may be inhomogeneous because of preliminary agglomeration. Among other ways how to solve the given problems the loading use seems to be rather effective. The theoretical aspects of straining combined with the sintering are considered here. Modeling is based on rheological conception of sintering which is sensitive to both grain size effect and bimodal pore distribution. Various loading paths are considered. The combinations of deviatoric and hydrostatic stresses and straining history providing needed characteristics are defined for some cases of sintering.

**11<sup>40</sup> – 12<sup>05</sup>: V.V. Novikov, Odessa National Polytechnical University, Odessa, Ukraine,**

**“Viscoelastic Properties of Inhomogeneous Nanocomposites”**

The analysis of theoretical methods for definition of viscoelastic properties for inhomogeneous materials with chaotic structure has been introduced in the report. Fractal model for chaotic structure has been described. Comparison of theoretical and calculation data has been carried out.

Hierarchic model of structure [V. V. Novikov, K. W. Wojciechowski, D. V. Belov and V. P. Privalko, Phys. Rev. E **63**, 036120 (2001)] is generalized and applied to study viscoelastic properties of a two-component inhomogeneous medium with chaotic, fractal structure and with one of the components exhibiting a negative shear modulus. It is shown that similarly to the results obtained recently in frames of the Hashin-Strikman model [R. S. Lakes, Phys. Rev. Let. **86**, 2897 (2001)], the present model predicts possibility to obtain composites of the effective shear and dumping coefficient much higher than those characterizing both the component phases. The viscoelastic properties of the fractal medium are, however, qualitatively different from the properties of the Hashin -Strikman medium.

**12<sup>05</sup> – 12<sup>20</sup>: Marcello Cabibbo, Enrico Evangelista, Dipartimento di Meccanica - Universita' Politecnica delle Marche,**

**“Deformation mechanisms inducing microstructure refinement in commercially pure aluminium processed via ECAP: comparison to Cold-Rolling and Hot-Torsion”**

The subgrain and grain evolution with the strain of an equal channel angular pressed commercially pure aluminium alloy has been investigated by means of transmission electron microscopy techniques. The deformation was carried out through route C. The process was capable of producing a microstructure decorated mainly by nano-scale grains and very fine subgrains in the grain interior, after a true strain of 8. The deformation mechanisms, induced by equal channel angular pressing, were investigated and discussed in terms of early

subgrain formation and subsequent fine grain formation. The fraction of high-angle grain boundaries, reached after 8 passes, was of 72%. The microstructure evolution was compared to the one induced by cold rolling. With this respect, and on the basis of the microstructural inspections, the role of geometrically necessary boundaries and incidental dislocation boundaries were compared and clarified in the two cases. The substructure parameter was practically the same, although, geometrically necessary boundaries and incidental dislocation boundaries, in the material subjected to equal channel angular pressing, showed a lower increasing slope with the strain compared to cold rolling, basically due to a different deformation mechanism. The equal channel angular pressing microstructure evolution was also compared to the one occurring during hot torsion. The two mechanism differentiated essentially in the formation and propagation process of geometrically necessary boundaries and incidental dislocation boundaries. Tangle dislocations and cells formation and evolution was discussed for all the three severe plastic deformation methods.

**p.m.**

**14<sup>20</sup>: Tour Orthodox Monastery and surrounding area**

**18<sup>30</sup> : Workshop Banquet**

**September 25, 2004**

a.m.

**(Chairman – Amiya K. Mukherjee, Nikolay V. Novikov)**

**9<sup>00</sup> – 9<sup>25</sup>: Ruslan Z. Valiev, *Inst. of Physics of Advanced Materials, Ufa State Aviation***

*Technical University, Ufa, Russia*

***“Applications of Severe Plastic Deformation for Materials Nanostructuring  
Aimed at Advanced Properties”***

This paper shows that extraordinary mechanical properties, i.e. very high strength and ductility, as well as low temperature and high strain rate superplasticity, can be observed in nanostructured metals produced by severe plastic deformation (SPD). However, the appearance of these advanced properties is originated not only from the presence of fine grains, but also from the structure of grain boundaries and internal stresses, as well as disperse precipitations of second phases. The origin of these advanced properties is considered and discussed in this work based on an analysis of performed mechanical tests and thorough microstructural studies. These advanced mechanical properties are of great engineering importance, in particular for developing nanostructured materials for new structural and functional applications. Examples of such applications in medicine and engineering are considered and discussed in the present paper.

**9<sup>25</sup> – 9<sup>50</sup>: S.V. Dobatkin<sup>1</sup>, L.Lev<sup>2</sup>, R. Verma<sup>2</sup>, <sup>1</sup>*Moscow State Institute of Steel and Alloys***

*(Technological University), Moscow, Russia; <sup>2</sup>General Motors Corporation,  
Warren, Michigan, USA,*

***”Development of Continuous Processes of Severe Plastic Deformation for Strip  
Production”***

Grain refinement during severe plastic deformation (SPD) accompanied by substantial material property improvements has been well established. However, the industrial applications are hindered by the absence of effective continuous SPD processes. The potential of development of continuous SPD process based on extrusion processes such as "conform", "linex" and "extrolling" is considered. Existing continuous SPD processes for strip production such as "conshearing", continuous confined strip shearing (C2S2) and accumulative roll bonding, are analysed. New methods of continuous SPD processing of sheet stock are discussed.

**9<sup>50</sup> – 10<sup>15</sup>: Yuri Estrin<sup>1</sup>, Eugen Rabkin<sup>2</sup>, Ralph Hellmig<sup>1</sup>, M. Kazakevich<sup>2</sup> and**

***Aikaterini Zi***<sup>1</sup>, <sup>1</sup>*Institute of Materials Science and Technology, Clausthal  
University of Technology, Clausthal, Germany; <sup>2</sup>Department of Materials  
Engineering, Technion, Haifa, Israel,*

***“New Applications of the SPD concept: mSPD”***

Extreme grain refinement by severe plastic deformation (SPD) is an established processing approach broadly applied to bulk materials. The dimensions of specimens or workpieces used are typically in the centimetre range. We propose to adopt an analogue of equal channel angular pressing in the context of microforming. Using sub-millimetre sized channels

(equal channel vias') in a process similar to the conventional forcefill technique, fibres or wires with ultrafine grain sizes can be manufactured. Their cross-sectional dimensions can be in the micrometer range. An alternative technique leading to ultrafine grained fibres or wires is pressing of metals through porous 'filters' with submicrometer scale open porosity. First results demonstrate the viability of this approach, which we refer to as *mSPD*.

**10<sup>15</sup> – 10<sup>30</sup>: A.Babushkin, O.Savina, I.Sukhanov, Ural State University, Russia,**

***“Application of diamond anvils techniques for studying nanostructure formation in massive materials directly during severe plastic deformations”***

Progress in understanding of properties of materials at high pressures in many respects is connected to diamond anvils technique. In transparent anvil cell structures and phase transitions in many dielectrics and semiconductors is investigated. We developed and investigated a technique of megabar range pressure valuation, applicable in the opaque high pressure diamond anvil cell (DAC) with anvils of the (rounded cone-plane (Verchagin-Yakovlev) type made of synthetic carbonado-type diamonds, consisting of dielectric grains of synthetic diamonds in layers of conducting materials. These anvils are good conductors, permitting measurement of the electrical properties of samples placed between the anvils in the DAC by using the anvils as the electrical contacts to the sample. Researches of dielectrics and semiconductors are traditional.

We for the first time have applied this technique to research of formation of new states in metals and metals alloys. As structurally sensitive parameters we use a thermo electromotive force (TEMF). At compression of a metal sample in anvils, there are severe plastic deformations. These deformations conduct to change of a microstructure of a material. Therefore, at measurement a TEMF directly it is possible to study dynamics of changes occurring in a sample during deformation. It is possible to study dependence of structure on size of deformation and on time. These techniques represent not only scientific interest, but also can be interesting for technology.

We report the results of an investigation of the TEMF of nanostructured materials (zirconium dioxide, Ti-alloys) directly during severe plastic deformations in DAC (pressure range between 15 and 50 GPa and room temperature).

**Coffee-Break**

**10<sup>50</sup> – 11<sup>15</sup>: M.J.Zehetbauer<sup>1</sup>, L. Zeipper<sup>1,2</sup>, E. Schafner<sup>1</sup>, <sup>1</sup>Institute of Materials Physics,**

*University of Vienna, Wien, Austria, <sup>2</sup>Austrian Research Center Seibersdorf GmbH, Seibersdorf, Austria;*

***“Modelling Mechanical Properties of SPD Materials during and after Severe Plastic Deformation”***

In developing new techniques to achieve ultra-fine and/or nanosized grains by plastic deformation, it is necessary to design models consisting of constitutive equations which describe not only the evolution of stress but also that of microstructure as a function of imposed strain. While it has been well known for some time that the temperature of deformation, the alloy content and the presence of precipitates rule the fragmentation process and thus the final grain size, the number of experiments on the effect of hydrostatic pressure yet is comparably little although this seems to be the most important feature of Severe Plastic Deformation. A model has been developed by the author which successfully reflects in-situ measured quantities from High Pressure Torsion (HPT), by taking into account the deformation induced generation and migration of vacancies. In accordance with the model,

the maximum possible vacancy concentration in solid pure metal crystals is reached when the pressure level approaches a value of about 5 GPa, for a deformation temperature of about  $T \sim 0.2 T_m$  ( $T_m$  is the melting temperature in K). This explains recent measurements done at the same pure metals showing that the strength, the dislocation density and also the grain size obtained after HPT deformation do not increase anymore although the pressure is increased further.

11<sup>15</sup> – 11<sup>40</sup>: **Hyoung Seop Kim**, *Department of Metallurgical Engineering, Chungnam National University, Daejeon, KOREA,*

***“Process Modelling of Equal Channel Angular Pressing for Ultrafine Grained Materials”***

Recently several methods of severe plastic deformation, such as equal channel angular pressing (ECAP), high pressure torsion straining, multiple forging, accumulated roll bonding, etc., have been developed to process bulk materials with ultrafine grained microstructures. Among them, the ECAP process is a convenient forming procedure to extrude material by use of specially designed channel dies without a substantial change in geometry and to make an ultrafine grained material. The properties of the materials are strongly dependent on the plastic deformation behaviour during pressing, which is governed mainly by die geometry (channel sizes, a channel angle and corner angles), material properties (strength and hardening behaviour), and process variables (temperature, lubrication and deformation speed). Because the evolution of microstructures and the mechanical properties of the deformed material are directly related to the amount of plastic deformation, the understanding of the phenomenon associated with strain development is very important in the ECAP process.

It is clear that there is a need for modelling techniques which may permit a wider study of some of the effects observed for better process control and the understanding of process related phenomena. In this study, we describe a range of our continuum modelling results of the ECAP process in order to illustrate the modelling applicability. Firstly, the finite element results of ECAP modelling for various geometric factors are described. Secondly, the inhomogeneous deformation due to the hardening property of the material is explained. Lastly, modelling the effects of process variables during the ECAP process is presented.

11<sup>40</sup> – 12<sup>05</sup>: **O. Lyeshchuk**, **M. Novikov**, **O. Borimsky**, *V. Bakul Institute of Superhard Materials of the NAS of Ukraine, Kiev, Ukraine,*

***“Advancement of equipment and technology of superhard materials synthesis in solid-state high pressure apparatuses”***

The action of high pressures and temperatures on various substances allows us to change their physico-mechanical properties and structure and is accompanied by phase transitions and formation of new materials. Synthesis of superhard materials (SH?) (diamond and cubic boron nitride) and sintering of polycrystals on the basis of them are the most prominent example of the efficient use of high pressure-high temperature technique.

We consider the basic types of solid-state high pressure apparatuses (HPA) (recessed-anvil, cylindrical, multianvil), which have been designed in the ISM for SHM synthesis, and sintering of polycrystals on their basis at a pressure of 4-9 GPa and temperature of 1500-2800 K. HPA for scientific investigations, which allow the pressure reaching up to 40 GPa to be created, are discussed. The basic technical characteristics of HPA are given and the most efficient applications of them are grounded. Special presses of the force up to 50 MN, which are used for the HPA operation, are considered.

A procedure for computer-aided modeling of stressed-strained state of HPA components and optimization of their design, a procedure for calculation of crystallization zones of diamond

of various habits have been developed depending on the temperature and pressure created in HPA reaction cells at synthesis. Numerical modeling of the diamond crystallization in a HPA, which is accompanied by the coupling of electrical and heat conduction, thermoelastoplastic deformation and phase transitions, is considered. The problem of determination of fields of temperature, pressure and concentration of diamonds has been solved for the HPA reaction volume and local diamond-melt-graphite system.

Our experimental and calculated results of diamond synthesis in various HPAs are considered depending on the volume of a reaction cell, thermodynamic parameters of the process, composition and the scheme of an arrangement of reaction charge components. Comparison between calculated and experimental results has confirmed potentiality and expediency of the efficient use of computer-aided modeling for the development of new technologies of diamond synthesis.

The examples are given of the use of the developed numerical technique for calculation of crystallization zones of cubic boron nitride, modeling of temperature conditions in the reaction cell during crystallization of gallium nitride, determination of conditions of efficient sintering of cBN-based polycrystalline materials, modeling of thermostressed state and optimization of parameters of thermobaric treatment of high-temperature superconducting ceramics in a HPA. Perspective directions of the development of high pressure equipment are determined, i. e. increase in HPA working volume and range of achievable pressures and temperatures.

**12<sup>05</sup> – 12<sup>20</sup>: Tatjana Konstantinova, *Donetsk Phys.&Tech. Institute of the NAS of Ukraine*,**

***“Mesoscopic mechanisms of fragmentation at severe plastic deformation”***

The idea about declinations and a rotary plasticity is key when discussing the fragmentations deformed solids so far. At the same time some particularities of the rotary plasticity stay unexplained within the framework. Questions about a rising high degrees discrete disorientation hadn't considered. Reasons responsible for the size of fragments hadn't ascertained. Not clear, why the boundaries of banded structure have not a determined crystallographic orientation, as a rule, and alternately in size and sign indemnify each other. On the base of analysis of own and literary data the local elastoplastic bends of crystalline lattice observed in metallic alloys with high dislocation density are offered to consider as an independent three-dimensional defect of the mesoscopic level of the plastic deformation. As the declinations the using of mesoscopic level relieves a description complex dislocation structures.

Results of studies deformed Fe-Ni martensite aging steel by the method positron annihilation have revealed a changing a correlation of a edge and screw dislocation on the initial stage of forming local bend, that means increasing their mobility on comparison with their condition in a initial martensite structure. According to models the conversion from structures with fluent change an orientation to discrete one is possible because of a reorganization dipole flat and tilted rows in a dipole dislocation walls under the action of stresses at the severe deformation.

New descriptions allow more clearly describe an evolution of the dislocation structure at developed plastic deformation. Jump in orientation appears in the region of compressions and strains of a local bend area. The size and configuration of the local bend area of a crystalline lattice define size and form of the fragment. Banded structure appears at forming the cylindrical fields of curvature.

Under severe plastic deformation the fragmentation of solids occurs by different ways, depending from the initial structure of material and deformation mechanisms. Three main types of structures separated on the degree disorientation are formed at the dislocation nature of plastic flow on the micro level: nondisoriented structures, with fluent change and with discrete disorientation. These structures evolve on different ways in the process of deformation and following heating. At study Fe-Ni martensite aging alloys it was established that in systems with high density of dislocations the plastic deformation can be accomplished

by the way of local dipole bends of the crystal lattice. These bends are considered as volumetric defects of the mesolevel and the deformation modes can be considered as bending ones. The method of determination of the bending- torsion tensor components was developed for local bend. The dislocation model of a new mesodefekt is built. The physical principles of high-strength state formation in multicomponent alloys and methods of their realization are developed. Possibilities of creation the nanostructures under twinning and martensite conversion are discussed.

**12<sup>20</sup> – 12<sup>35</sup>: M.R. Shagiev, G.A. Salishchev, *Institute for Metals Superplasticity Problems of the RAS, Russia*,**

**“Low-Temperature Sheet Rolling of TiAl Based Alloys”**

Titanium aluminides are considered as promising materials for aerospace applications because of their low density, reasonable oxidation resistance and high strength at temperatures up to 800<sup>0</sup>C. Recently, a significant interest has been developed to use these alloys in sheet forms for applications such as thermal protection or exhaust honeycomb structures. However, these materials generally have limited ductility and can be processed only at temperatures above 1000<sup>0</sup>C. For example, sheet rolling of these materials is currently being performed at temperatures of 1200-1300<sup>0</sup>C, which requires expensive tool and can materials. Moreover, such sheets have rather coarse-grained structure, and high temperatures are required for secondary forming operations, particularly for superplastic forming.

**p.m.**

**14<sup>00</sup> – 15<sup>30</sup>: Poster session**

**Coffee-Break**

**16<sup>00</sup> – 18<sup>00</sup>: Round-table discussion, Moderator: Viktor Varyukhin, Yuntian T. Zhu**

**Note: This will be free style. Anyone in the audience can raise a question and answer someone else’s question.**

**September 26, 2004**

**a.m.**

**Departure of participants.**

**p.m.**

## Poster presentations

**P-01. Yu. Bazhenova;** *Institute of Physics of Advance Materials, Ufa state Aviation Technical University, Ufa, Russia;*

***Insitute of Physics of advanced materials USATU: International Cooperation in the field of nanostructured materials produced by SPD***

The Institute of Physics of Advanced Materials of Ufa State Aviation Technical University (IPAM USATU) is a scientific and technical center focusing on fabrication of bulk nanostructured materials using severe plastic deformation techniques.

In collaboration with Russian partners from research centers in Tomsk, Ekaterinburg and others cities, IPAM has been participating actively for several years in numerous joint international projects funded through the ISTC, NATO, CRDF and INTAS. This has allowed IPAM to accumulate solid experience in scientific and technical cooperation with research centers from other countries (USA, Europe, Asia).

The given paper reviews the topics and the main research directions covered in these projects. Taking the projects as examples, the author discusses the objectives of research cooperation between Russia and other countries. The problem of increasing the efficiency of management and as a result productivity of international research projects is addressed.

**P-02. V. Bengus<sup>1</sup>, S. Smirnov<sup>1</sup>, E. Tabachnikova<sup>1</sup>, V. Romanchenko<sup>1</sup>, V. Natsik<sup>1</sup>, D.**

**Gunderov<sup>2</sup>, V. Stolyarov<sup>2</sup>, R. Valiev<sup>2</sup>;** <sup>1</sup>*B. Verkin Institute for Low Temperature Physics & Engineering, NASU, Kharkov, Ukraine;* <sup>2</sup>*Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa, Russia;*

***Nanostructured Titanium Anomalies Of Plasticity Under Cooling From 300 Down To 4,2 K***

It is known that low temperature mechanical properties of polycrystalline titanium are characterized by the anomaly of macroscopic plasticity: under cooling below ambient temperature the ultimate plastic deformation before the failure of a coarse grained titanium is not decreases, but increases and achieves a great maximum at 40 - 80 ++. This peculiarity distinguishes titanium from other polycrystalline metals, which naturally decrease their plasticity under cooling, due to a thermally activated character of plastic deformation.

This work is devoted to the experimental study of the plasticity of commercial purity nanostructured titanium, processed by equal channel angular pressing (ECAP) from initial grain size 15 +? down to 0.3 and 0.1 +? (after additional 75% cold rolling and 1 hour annealing at 300 C) and to the theoretical explanation of obtained results. Samples were deformed with a 4+10-4 s-1 strain-rate uniaxial tension, at the machine of 3 +?N/mm elastic stiffness, at 300, 77 and 4.2 K up to the beginning of necking and subsequent failure. It is found that for nanostructured titanium, under cooling from 300 to 4,2 ++, plastic deformation value eneck before beginning of necking, increases. I. e. the plasticity anomaly, which earlier had been known for a polycrystalline titanium, is observed also for the nanostructured one. It is established that at a given temperature, eneck depends on the average grain size d and decreases with decreasing of d.

Changing of eneck with temperature depends also on the average grain size. In the coarse grained titanium it is the most strong (the maximum value of eneck = 45% and is achieved at 77 K). For the nanostructured titanium increasing of eneck under cooling is essentially lower than for the coarse grained one (at 4,2 ++ eneck is 20% for d =0,3 +? and only 10% for d =0,1 +? ).

Observed eneck values increasing of nanostructured and polycrystalline titanium under

cooling can be understood with taking into consideration thermal anisotropy internal stresses, which arise under cooling. Obvious expressions are obtained for the complete stress tensor components, which take into account thermal anisotropy internal stresses in Ti. Obvious expressions for the number of grains deformed by twinning and slip are obtained. Theoretical analysis of the possible integral contribution to plastic deformation of a sample of dislocations from all available slip  $\langle 110 \rangle \{100\}$  (prismatic) and  $\langle 113 \rangle \{101\}$  (a+c) systems, and also from twinning dislocations of all  $\langle \cdot \cdot \cdot \rangle \{ \cdot \cdot \cdot \}$  systems, shows that consideration of its temperature dependence allows to understand plasticity anomalies of titanium under cooling. This work have being carried out within the program of the INTAS-01-0320 Project

**P-03. Varyukhin V.N., Belousov N.N.,** *Donetsk Phys.&Tech. Institute of the NAS of Ukraine, Donetsk, Ukraine,*

**“Structural-Kinetic Approach To Produce Nanostructural Materials By High Pressure Torsion”**

The idea of in-situ technology consists in continuous registration of external deforming parameters and establishment of connection with internal submicro- and nanostructural parameters. To realize the deformation by torsion under pressure a modified chamber was developed. The chamber allowing forming structure directly in a special device of a testing machine with computer registration of structure - sensitive parameters. To investigate structural changes directly in deformation conditions an advanced X-ray chamber of high pressure with diamond anvil was used. As the basic material of investigation high nitrogen steel was taken as less investigated in the aspect of formation of submicro- and nanocrystalline (SMC and NC) structures.

The results of investigation have shown that the curve of dependence of compression force on value of deformation is of a stage-like character consisting of: a) a stage of elastic behaviour; b) a stage of plastic deformation with fragmentation of elements of the structure; c) a stage of structure hardening and secondary elastic behaviour. Crystal lattice parameter monotonously decreased with the growth of compressive pressure. Width and intensity of X-ray lines non-monotonically changed, that correlates with the beginning of fragmentation of structural elements and confirmed by non-monotonous behavior of temperature, electroresistance and increasing of intensity of acoustic emission. At a pressure of more than 20 GPa the diffuse scattering has considerably increased. The quantitative processing of X-ray diffraction peak by profile analysis using the Multiple Whole Profile Fitting Method has shown the presence of nanodimensional elements – 80-150 nm. It is shown that the severe plastic deformation at the fragmentation stage is accompanied by structural - kinetic changes. This promotes the formation of elements and complexes with submicro- and nanocrystalline structures.

**P-04. I.G.Brodova<sup>1</sup>, V.V.Stolyarov<sup>1</sup>, R.Lapovok<sup>2</sup>;** *<sup>1</sup>Institute of Physics of Advance Materials, Ufa state Aviation Technical University, Ufa, Russia;* *<sup>2</sup>School of Physics and Material Engineering, Monash University;*

**Structure and properties of Al-Fe alloy by ECAP with backpressure**

Equal channel angular pressing (ECAP) with a different number of passes (n=1-16) and backpressure levels (P) from 40 to 275 MPa was used for obtaining submicrocrystalline (SMC) binary Al-5 wt.% Fe alloy. The sample structure and phase composition were controlled by TEM, SEM and X-ray methods. Tensile tests and Vickers microhardness measuring performed at room temperature. Microstructural refinements, strength and ductility improvements as functions of P and n were determined.

It was established that the material subjected to ECAP (P=275 MPa, n=16) had SMC composite structure with grain size of 325 nm in the Al-based matrix and dispersed

aluminides of less than  $10^{-1}$  m. The measurements of the Al phase lattice parameter showed that ECAP causes the additional increase in the content of Fe into Al-solid solution. Maximum solubility of 0.6 wt.% Fe was obtained. The mechanical properties (strength, ductility) and microhardness of deformed material were significantly enhanced. The ECAP alloy is thermal stability to 2500C. Heating of the deformed alloy leads to decomposition of supersaturated solid solution. It was shown that the further increase in mechanical properties was observed after subsequent artificial ageing. Increasing backpressure promotes improvement in workability and enhancement of ductility of the alloy processed by ECAP.

**P-05. V.D. Blank, S.G. Buga, G.A. Dubitsky, V.M. Prokhorov; Federal State**

*Institution Technological Institute for Superhard and Novel Carbon Materials,  
Troitsk, Russia;*

***Sintering of metal-coated diamond powders with fullerenes at nonhydrostatic  
HPHT***

Industrial diamonds are used very extensively in various tools. In cutting and drilling instruments polycrystalline, single crystal and sintered powder diamonds are employed. Bound-free sintering requires very high pressure and temperatures; the target aggregates usually have poor cohesion and their strength is worse compare to polycrystalline and single crystal diamonds. Metal bounds fill the intergrain pores, provide carbide chemical bonds and thus relatively high strength of such composite material to be used in tools.

New nanostructured superhard carbon materials on the basis of fullerenes can be competitive with diamonds in some industrial applications. They possess high hardness and fracture toughness. But the limiting factor is their poor thermal conductivity, approximately the order of magnitude below that of diamonds. Fullerites can be used in tools in composition with other superhard materials with high thermal conductivity and stability. Solid C60 fullerene is van der Waals crystal and can effectively migrate in diamond intergrain voids at moderate pressure and temperature. Going to high pressure and temperature above 9 GPa, 700 K we obtained strong 3D-polymeric fullerene matrix with embedded diamonds. Different kinds of sintered composite diamond materials have been obtained varying the diamond/fullerene mass ratio. We used metal-coated (niobium) synthetic diamond powders of 100/80 nm grain size for sintering with fullerene. Toroid-type high-pressure apparatus have been employed for HPHT treatment. Using different pressure-transmitting media and varying the shape of a specimen it is possible to vary shear stresses in the apparatus. The target structures of superhard fullerites and thus their physicochemical properties strongly depends on the shear stresses and deformations. We investigated cylindrical samples of different sizes, obtained at the same load of the press and temperatures of synthesis. Generally, the structure of samples was not uniform. Samples with more uniform structure and properties have been obtained at the diameter/height ratio of about 1.5 ? 2. But the strongest materials were obtained at the same load and temperature in the central zone of inhomogeneous samples with the reverse dimensions ratio: 0.75 ?0.5. This takes place due to the effect of severe shear deformation on phase transition in fullerene under pressure. The bulk homogeneity of samples and the rates of sound waves have been investigated using scanning acoustic microscopy method. The elastic modules of samples have been evaluated using the acoustic rates and the data for densities. Sintering of metal-coated diamond powders with C60 fullerene steadily increases the bulk elastic module of the material. Its gain of 50-60% was obtained at 1:1 weight content of diamonds and fullerene. For practical purpose the method must be optimized to get maximum weight and structure of samples with desirable diamond-fullerene ratio taking into account the stress and deformation fields in the chamber.

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***Properties and microstructure of 316LVM steel subjected to severe plastic deformation by Max Strain and transverse rolling***

Recently growing interest is observed in materials processed by SPD methods which enables production of relatively large bulk nanomaterials with a significant grain size refinement due to the plastic strain at a range of  $\epsilon=10$ . In this work for the 316L steel, which is important for medical applications has been subjected to Max Strain and transverse rolling. Max Strain unit has been used also to conduct thermoplastic treatment. Plastic strain was applied cyclically along three principal axes. All samples have been deformed up to logarithmic strain approaching  $\epsilon = 40$ . Three strain intervals have been used ( $\epsilon_j=0,2;0,4;0,6$ ).

The DUO rolling process is an original technology protected by a patent. Sample deformation system assures full control of rolling conditions the amount of plastic strain is. The of SPD samples have been shown by light microscopy and TEM. Microhardness measurement were also carried The result show that both method produce nano-scale structure with very high mechanical properties.

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***Surface nanostructure and tribological properties of metallic materials***

With metals in sliding friction, in the surface layer ( $\approx 10$  nm), ultradisperse structures are formed, which to a considerable extent determine the tribological properties of the surfaces. The structures can be considered to be nano-crystalline (NCS): micro-fragments with large-angle boundaries are 0.005 to 0.1  $\mu$ m in size. The objects of investigation were the nitrogen-containing Fe-Mn-(Cr) alloys: Fe-19% Cr-10% Mn-2% Si-0.7% N-0.07% C and Fe-18% Cr-20% Mn-0.8% N-0.08% C. To compare the obtaining results, the use was also made of the tribological characteristics of different alloys: Fe-10% Mn-0.05% C, Fe-20% Mn-0.05% C, Fe-11% Cr-13% Mn-0.03% C, Fe-40%Mn-0.05% C and Fe-18% Cr-12% Mn-0.5% N 0.07% C differing in phase composition, structure and value of the stacking fault energy (SFE). Tribological test were done at laboratory-scale plants in conditions of sliding friction for the steel-alloy and abrasive-alloy pairs. The NCS  $\gamma$  -alloys and NCS nitrogen-containing  $\gamma$  -alloys possess low friction coefficients K and increased of abrasive wear  $\epsilon$ . In this alloys, the developed basal or planar slip characterized by a smaller quantity of the slip systems, condition the lowering of resistance NCS fragments rotation and, as a result, the improvement of tribological properties.

Thus, the essential lowering of friction coefficient K and intensity of adhesion wear  $I_h$ , as well as the increase in the abrasive wear  $\epsilon$  of NCS HCP-  $\epsilon$ -alloy and nitrogen-containing NCS FCC  $\gamma$  -alloy are mainly related to decreasing the quantity of slip systems in nanocrystals of these alloys ( $n = 3$ ). As a result, the dislocations travel to the boundaries of nanocrystals more easy.

- P-08. B. M. Efros<sup>1</sup>, V.A. Ivchenko<sup>2</sup>, N.B. Efros<sup>1</sup>, E.V. Popova<sup>2</sup>, L.V. Loladze<sup>1</sup>, T.P. Zaika<sup>1</sup>**; <sup>1</sup>*Donetsk Phys. & Tech. Institute of the NAS of Ukraine, Donetsk, Ukraine;* <sup>2</sup>*Institute of Electrophysics of RAN, Ekaterinburg, Russia;*  
***Structure and hardening of ultrafine grained nickel after severe plastic deformation***

Method of packet hydroextrusion was used to investigate the effect of severe plastic deformation on defect structure and hardening of polycrystalline nickel (99.97 wt.%). The grain-boundary component of the hardening is shown to be due to the increase in the defects density. The formation of sub blocks in the structure of ultrafine grained nickel subjected to severe plastic deformation has been detected by the field ion microscopy method.

- P-09. A.A. Nazarov<sup>1</sup>, N.A. Enikeev<sup>1</sup>, A.E. Romanov<sup>2</sup>, T.S. Orlova<sup>2</sup>, I.V. Alexandrov<sup>1</sup>, I. J. Beyerlein<sup>3</sup>**; <sup>1</sup>*Institute of Physics of Advanced Materials, Ufa State Aviation University, Ufa,* <sup>2</sup>*Ioffe Physico-Technical Institute, Russian Academy of Sciences, St. Petersburg, Russia;* <sup>3</sup>*Los Alamos National Laboratory, Los Alamos, USA*  
***Modeling of grain subdivision during severe plastic deformation by VPSC method combined with disclination analysis***

Mechanisms and regularities of the microstructure refinement during severe plastic deformation is a matter of a great theoretical and practical importance, particularly with regard to the preparation of ultrafine grained materials using equal-channel angular pressing (ECAP). Dislocation modeling of this process is a challenging task due to the complex nature of the processes of dislocation multiplication, interaction and annihilation in real crystals. In the last decades, the disclination approach has been shown to be very helpful for the description of elementary processes of grain subdivision [1,2]. It has been proposed that grains are divided by low-angle boundaries growing due to a conservative motion of partial disclinations generated at grain boundaries and grain boundary junctions. However, this formalism alone cannot be used to analyze the whole process of grain subdivision, since it neglects the distribution of plastic deformation on slip systems and does not consider important crystallographic and geometric factors influencing the grain refinement. On the other hand, micromechanical models accounting for these factors, neglect the non-uniformities of the stress and strain in grains that lead to the subdivision. Therefore, the use of disclination models in combination with micromechanical modeling seems to be a powerful tool for modeling of the microstructure development during large plastic deformation.

In the present work we present a model for the incorporation of the disclination approach into a visco-plastic self-consistent (VPSC) model for polycrystalline materials [3]. In the latter method, each grain is considered to deform in a homogeneous medium composed of all other grains. The stresses under which the grain is deformed are calculated from a difference of strain rates in a self-consistent way. This method has proven advantageous for simulations of texture development and mechanical response. Using this method, one can calculate the difference between the strain rate tensors of any grain and its neighbors. This difference determines the strength of disclinations accumulated in triple junctions of grains. When a disclination of certain strength is accumulated, the junctions are assumed to build up low-angle boundaries dividing the grain. This subdivision event can be analyzed in terms of either energetic, or force criteria. In the force criterion it is considered that the junctions generate boundaries, if the strength of junction disclinations exceeds some critical value of the order of 1°. Parameters of the newly nucleated grain boundary can be determined in terms of the energetic approach. Successive generation of disclinations results in the increase of the misorientation angle of these boundaries. At some value of misorientation, the newly

formed subgrains are considered to become grains and are included to the VPSC simulation scheme.

Using this combined model, simple shear of a polycrystal containing initially randomly oriented grains has been simulated. The results show that the ability of a grain to subdivide strongly depends on its orientation with respect to the neighbors. The slower is the strain rate of a grain as compared to its neighbors, the higher is its subdivision rate. The crystallographic and morphological textures after simple shear strain of a value 1, corresponding to strain accumulated during the 1<sup>st</sup> ECAP pass are analyzed. Implications of the results to microstructure development during ECAP are discussed.

- P-10. R.K.Islamgaliev<sup>1</sup>, N.F.Yunusova<sup>2</sup>, R.Z.Valiev<sup>2</sup>; <sup>1</sup>*Institute of Mechanics Russian Academy of Sciences, Ufa, Russia;* <sup>2</sup>*Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa, Russia;***

***The influence of the SPD regimes on the superplastic behavior of aluminum alloys***

Recent development in processing of bulk nano- and submicrocrystalline metals and alloys using severe plastic deformation (SPD) has provided new opportunities in investigations of enhanced superplasticity in metallic materials. These investigations were started in two directions: firstly, fabrication of large billets with submicrocrystalline structure with a grain size of less than 1 μm by equal channel angular pressing (ECAP); secondly, processing of nanocrystalline structures in small samples by high pressure torsion (HPT). It was demonstrated experimentally that the SPD alloys have a great potential for achieving superplastic characteristics at low temperatures and high strain rates. Although superplasticity has been extensively studied in a number of SPD alloys, there has been limited experimental data about microstructural features leading to the exciting superplastic properties. The present paper illustrates the significance of the SPD regimes for microstructure and superplasticity of the 1420 and 1421 aluminum alloys.

From experimental results obtained in the present paper using both *in-situ* heating and tensile testing one can conclude that a very important pre-requisite of the enhanced superplasticity in the UFG materials, in addition to the strong refinement of the microstructure by SPD, is the morphology of precipitated phases and the formation of non-equilibrium high angle grain boundaries. Apparently that these microstructural parameters depend strongly on the SPD regimes, which should be controlled carefully, in order to attain enhanced superplastic properties. Regarding the grain size effect, one can see that grain refinement down to the submicron range contributes to the enhancement of superplastic properties and to attaining low temperature and/or high strain rate superplasticity. However, further decrease in the grain size to the nanometer range can lead to intensive grain growth at elevated temperature of tensile testing. Therefore, the exciting superplasticity for nanocrystalline materials may be observed mainly at low temperatures. Probably there exists a critical grain size (perhaps within the range of 100-300 nm) below which further investigations of superplasticity should be carefully performed.

- P-11. Dominika Klassek<sup>1</sup>, Halina Garbacz<sup>1</sup>, Malgorzata Lewandowska<sup>1</sup>, Waclaw Pachla<sup>2</sup>, Krzysztof J. Kurzydowski<sup>1</sup>; <sup>1</sup>*Division of Materials Design, Faculty of Materials Science and Engineering, WUT, Warsaw, Poland;* <sup>2</sup>*High Pressure Research Center, Polish Academy of Sciences UNIPRESS, Warsaw, Poland;***

***The influence of hydrostatic extrusion on microstructure and mechanical properties of austenitic stainless steel for biomedical applications***

Stainless steels are widely used for fracture fixation and dental devices, hip implants and piercing studs, due to their good biocompatibility and adequate mechanical properties.

Recent development of processing methods for bulk nanostructured metals opens possibility to obtain novel properties in such steels. The paper presents results of investigations of the properties and microstructure of 316LVM stainless steel subjected to severe plastic deformation, SPD, via hydrostatic extrusion. Final values of logarithmic plastic strain in the range from 1,4 to 1,8 at different process temperatures were obtained. Microstructures of the processed steel were investigated by light and transmission electron microscopy. The initial and post-SPD material grain size was estimated using special software MICROMETER [1]. Mechanical properties were described in terms of hardness, microhardness and uniaxial compression tests. Considerable refinement of grain size was observed for specimen extruded at higher temperature. Low temperature extrusion caused a substantial decrease in the size of deformation twins. Both types of microstructures bring about a significant increase in hardness, microhardness and yield strength.

**P-12. Alexander Korshunov;** *RFNC-VNIIEF, Technology Division;*

***Response of mechanical properties along the workpiece section to ECA-pressing***

Nonuniformity of mechanical properties along the workpiece section depending on the number of ECA-pressing operations was studied using annealed M1 Cu (min. Cu content 99.9 %) as a sample material. The samples under examination had a square section with a side length of 8 mm and were cut out from an 8-mm thick sheet along the rolling direction. Microhardness distribution along the sample section was determined. Mechanical properties at drawing (conventional yield strength, ultimate strength, relative elongation and contraction) were determined at 9 points along the section using microsamples with a diameter of 1.5 mm cut out along the pressing direction. Strain diagrams were drawn. The investigations were carried out after 1, 2 and 4 pressing operations along the BC and C routes.

**P-13. B.M.Efros<sup>1</sup>, V.P. Pilyugin<sup>2</sup>, A.M. Patselov<sup>2</sup>, S.V. Gladkovskii<sup>3</sup>, N.B. Efros<sup>1</sup>, L.V. Loladze<sup>1</sup>;** *<sup>1</sup>Donetsk Phys.&Tech. Institute of the NAS of Ukraine, Donetsk,*

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***Martensitic transformations in nanocrystalline Fe-Cr-Ni and Fe-Mn alloys***

Influence of pressure on the structural and martensitic transformations in nanocrystalline Fe-Cr-Ni and Fe-Mn systems has been studied. The obtained experimental data show that the character of phase transitions and, thus the quantity of structural-phase components in metastable systems are defined by both the internal (chemical and phase compositions, structure, etc.) and external (scheme and degree of deformation, pressure, temperature, etc.) factors.

**P-14. Varyukhin V.N.<sup>1</sup>, Pashinskaya E.G.<sup>1</sup>, Pashinsky V.V.<sup>2</sup>, Tkachenko V.M.<sup>1</sup>, Tishenko I.I.<sup>1</sup>;** *<sup>1</sup> Donetsk Phys.&Tech. Institute of the NAS of Ukraine, Donetsk, Ukraine; <sup>2</sup>Donetsk National Technical University, Donetsk, Ukraine;*

***Changes in copper submicrostructure as a result of combination twist extrusion and conventional metal forming processes***

Recently a high attention is being paid to the problems of producing and studying the materials of a specific submicrocrystalline (SMC) structure, the so-called nanostructural materials [1,2]. A new method of severe plastic deformation (SPD) by torsional hydroextrusion (THE) has been proposed at Donetsk Physico-Technical Institute named after A.A.Galkin. This method permits to attain hard plastic deformations on bulk specimens and the formation of a typical submicrocrystalline structure.

Since under the rolling of copper after the THE in directions parallel and normal to THE axis there are similar changes in longitudinal and cross sections of the specimens, we may speak of the observed fact as a regular phenomenon in dicating that after the THE a specific nonanisotropic structure of copper has been formed capable of stress relaxation at further loading by the grain-boundary slippage mechanism.

The carried out investigations show that the THE method results in the formation of fine-crystalline fragmented structure in copper. The produced specimens are notable for a high-uniform deformation over the cross-section. Principal changes in structure and properties are observed for  $\epsilon=2.7$ , there are no noticeable changes with further decreasing the degree of deformation. It is shown that with the increasing total deformation the properties of copper are changing nonmonotonously, the submicrostructure is formed in several stages. After the THE the grain shape is like an ellipsoid rotated about THE axis an angle of 45 degree. The peculiarities of the formed submicrostructure (broad boundaries, fine grain, etc) enable the grains to be subsequently rotated by an angle around the rolling axis. The analysis of different cross-sections shows that the structure is highly uniform over the section. So, to obtain a material of highly uniform properties, the THE combined with rolling can be recommended. Peculiarities of the structure formed as well as a high level of the strength and plasticity characteristics can be utilized in realizing subsequent production operations, such as rolling, drawing, etc.

**P-15. Y.E. Beygelzimer, V.N.Varyukhin, O.V. Prokofyeva, B.M. Efros; Donetsk**

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***Grain Refinement And Failure Of Metal Materials Under The Large Plastic Deformations: Prerequisites For Modelling***

Basis on the experiments of a large plastic deformation of metal materials the generalizations are made and physical prerequisites for a structure evolution model are formulated. It is based on the interrelation between processes of grain refinement and failure of the material which are offered to consider as additional to each other processes. Physical reasons and experimental data are given for substantiation the hypothesis laid in a basis of the model. The influence on the deformation structure of the material formation by factors, such as high hydrostatic pressure and non-monotone deformation is investigated. The basic scripts of a grain refinement are considered: for the materials with high and low energy of defects of packing. The interrelation with the fractal theory is founded out and the opportunity of its application to the researched processes is proved. Conclusions regarding design the large plastic deformation processes directing on the ultra fine-grained materials reception are made.

**P-16. Beygelzimer Y., Reshetov A., Synkov S., Orlov D., Synkov A.; Donetsk**

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***Foundations of manufacturing technology of long size ultra-fine grained bar profiles based on twist, spread and direct extrusion processes***

Scheme for long size ultra-fine grained bar profiles manufacturing is introduced. This scheme consist form:

- 1) As-received rolled circular billets is extruded to profile for twist extrusion (TE);
- 2) TE by turns with a spread extrusion is being performed. This allow to develop ultra-fine structure in a billet volume;
- 3) Final forming to achieve profile specified is being performed by direct extrusion.

In order to manage structure formation process plastic flow during each processing steps is investigated by experimentally-calculating method.

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***Evolution of structure and mechanical properties in Ti-alloy at high pressure deformation***

Titanium alloys have a broad usage in different areas of industry, in aircraft and space engineering because of the good combination of specific weight, strength and heat resistance. There are possibilities of perfecting for alloy properties by means of structured minimization although titanium alloys are intensively studied. A submicro- and nanocrystal structure creation essentially improves the mechanical and other characteristics of material. High hydrostatic pressure (HP) is one of the methods of treatment, promoting reduction of the structure in the course of the material processing.

Peculiarities of structure evolution in Ti-5Al-5% V industrial alloy are investigated in the present work depending on HP - deformation and the Phase State. Two initial structural states of the alloy were studied: the mono-phase  $\beta$ -state (BCC-structure) and two-phase  $\alpha + \beta$  state (HCA + BCC). Prepared samples were deformed by high hydrostatic pressure (HP) up to  $\epsilon = 50\%$ . The structure control included Transmission Electron Microscopy (TEM), Optical Metallography (OM) and X-ray structure analysis. Mechanical characteristics ( $\sigma_{0.2}$ ,  $\sigma_s$ ,  $\sigma_b$ ,  $\epsilon_b$ ) were defined at strains of standard samples.

The development of  $\beta$ -deformation to  $\epsilon = 5 - 15\%$  in mono-phase alloy results in the appearance of big amount of narrow layers, which, as seen by X-ray, were orthorhombic  $\alpha''$ -martensite. As a rule, the martensite plates have a complex "packet" structure with internal enclosure of smaller disoriented packages, the least of which reaches 20 nm (TEM data). When the HP-deformation increases up to more than 30%, X-rays do not observe the martensite already. Probably, a  $\alpha''$ -martensite formation occurs under severe plastic deformation ( $>30\%$ ) and then a quick inverse conversion takes place. The initial  $\beta$  - phase structure is restored completely at the inverse conversion, but martensite plate boundaries are saved, and as a result,  $\beta$  -grain is greatly fragmented. Formation of amount of areas on the place of former martensite layers containing grains of nanocrystalline size with big disorientation contributes to the 50% growing of breaking point, 20% growing of yield point. A usual dislocation sliding occurs at the HP deformation  $\epsilon < 20\%$  in two-phase  $\beta + \alpha$  - alloy. Then, the growing disordered orientation within grains is observed at the achievement of high dislocation density ( $\epsilon \sim 30\%$ ) confirmed by TEM. However, deformation reveals itself in continuous change disorientation within a grain, unlike discrete change of the orientation at deformation of a single-phase alloy. So the rotational mode of deformation in single-phase alloy is replaced with the bended mode in two-phase alloy. New possibilities of governing of alloy structure were opened by knowledge of mechanisms of deformation. The combination of annealing and following HP -deforming provides raising the strength limit of alloy on 35-40 % at the conservation of plasticity.

**P-18. I.P. Semenova, G.I. Raab, L.R. Saitova, R.Z. Valiev; *Ufa State Aviation Technical University;***

***Ti-6Al-4V alloy mechanical properties enhancement by means of severe plastic deformation***

The current work studies the ways of strength enhancement in the two-phase Ti-6Al-4V alloy, applying severe plastic deformation (SPD) and thermomechanical treatment. Equal-channel angular pressing (ECAP) was used for refining bulk billets up to 40 mm in diameter. Extrusion was applied as an additional thermomechanical treatment.

The conducted research allowed to study the influence of the initial structure and degree of accumulated strain on the features of microstructure formation in the Ti-6Al-4V alloy and mechanical properties evolution at various stages of deformation. It was determined that ECAP played the leading role in the formation of homogeneous ultrafine-grained (UFG) state in the alloy,

providing a good technological ductility of a billet during subsequent thermomechanical treatment. Low-temperature extrusion of ECAP-billets was shown to result in the additional refining of structural constituents, intensive accumulation of defects of crystalline lattice, which stipulated the alloy strengthening by nearly 40% in comparison with the initial annealed state.

- P-19. I.P. Semenova<sup>1</sup>, V.V. Latysh<sup>2</sup>, G.H. Sadikova<sup>1</sup>, R.Z. Valiev<sup>1</sup>;** <sup>1</sup>*Institute of Physics of Advanced Materials, Ufa State Aviation Technical University, Ufa, Russia;*<sup>2</sup> *Scientific Design Technical Office "Iskra", Ufa, Russia;*

***Microstructure and mechanical properties of long-sized semi-finished articles with ultrafine-grained structure***

The current work presents the research results of combined thermomechanical treatment effect on microstructure and mechanical properties of semi-finished articles out of CP Ti. The combined technological process includes preliminary structure preparation, equal-channel angular pressing (ECAP) and hardening thermomechanical treatment. The application of this deformation scheme enabled to process ultrafine-grained (UFG) billets out of VT1-0 and CP Ti (Grade 2) 6.5 mm in diameter and up to 1000 mm long.

The features of UFG structure formation, its homogeneity along the whole billet length, and mechanical properties evolution at various stages of technological process are studied in the present work. It was revealed that the formation of homogeneous UFG structure in semi-finished articles out of CP Ti with a-grains size about 150 nm allowed to considerably enhance strength characteristics, which stay the same along the whole billet length. For example, the values of yield stress ( $\sigma_{0.2}$ ) amounted up to 900 MPa, which is almost two times as much as the same value of initial annealed state. At that, ductility constituted  $\geq 10 \dots 15\%$ .

- P-20. Beygelzimer Y., Synkov S., Orlov D., Reshetov A., Synkov A.;** *Donetsk Phys. & Tech. Institute of the NAS of Ukraine, Donetsk, Ukraine;*

***Technological Aspects Of Twist Extrusion***

This work aimed to develop technology of the twist extrusion (TE). In order to achieve this trajectories of particles- plastic flow in volume of a deforming billet are experimentally investigated. This allowed to track uniformity of strain distribution in the billet during the TE.

It was created special device for correct estimation of friction. This device allowed to study factor of friction dependent on sub lubricating coating and lubricants. The investigations done contribute to faster determination of optimal combinations of sub lubricating coatings and lubricants for various materials in order to reduce friction during the TE processing. Installation for the TE with back pressure is described. Energy-power parameters of the TE are investigated by the strain measurement method.

- P-21. E. Tabachnikova<sup>1</sup>, V. Bengus<sup>1</sup>, V. Natsik<sup>1</sup>, K. Csach<sup>2</sup>, J. Miskuf<sup>2</sup>, D. Gunderov<sup>3</sup>, V. Stolyarov<sup>3</sup>, R. Valiev<sup>3</sup>;** <sup>1</sup>*B. Verkin Institute for Low Temperature Physics & Engineering, Kharkov, Ukraine;*<sup>2</sup> *Institute of Experimental Physics, Kosice, Slovakia;*<sup>3</sup> *Ufa State Aviation Technical University, Ufa, Russia;*

***Low Temperature Deformation And Failure Of Nanostructured Ti-6Al-4V Alloy***

This work is devoted to comparative experimental study of the low temperature mechanical behavior of the nanostructured Ti-6Al-4V alloy processed by severe plastic deformation through equal channel angular pressing (ECAP) and polycrystalline Ti-6Al-4V alloy.

Two structural states were investigated: initial polycrystalline alloy with the average grain size  $d = 6 \text{ mm}$ , hot pressing and ECAP alloy with the average grain size  $d = 0.5 \text{ mm}$ , produced by 12 ECAP passes at  $700^\circ\text{C}$  of the initial alloy. Prismatic ( $2 + 2 + 7 \text{ mm}$ ) samples

were cut from bars both parallel ( $\parallel$ ) and perpendicular ( $\perp$ ) to the ECAP axis and subjected to uniaxial quasi-static compression (with the 0.0004 s<sup>-1</sup> strain-rate) at 300-4.2 K.

It is discovered that decreasing of grain size by ECAP processing of Ti-6Al-4V alloy from 6  $\mu$ m to 0.5  $\mu$ m have resulted in 25% increasing of the yield stress under compression at 300-4.2 K. Obtained high strength state of ECAP processed Ti-6Al-4V alloy (with the 1.28 GPa yield stress at 300 K and 2.47 GPa at 4.2 K) is combined with a high enough plasticity of the material down to 4.2 K.

Deformation was of a serrated character at 4.2 K. At 77 and 4.2 K the failure took place by the catastrophic plastic shear sliding-off of one part of the sample relative to another at the compressive failure stresses up to 2.8 GPa.

Shear failure surfaces were oriented nearly at 45 degree to the compression axis. SEM fractography revealed vein patterns at all observed shear failure surfaces of nanostructured Ti-6Al-4V alloy. Non-uniform space distribution of veins and its dependence on the structural state of samples are observed. This phenomenon is considered as a consequence of a non-uniform internal stress distribution and non-uniform adiabatic heating along the surface of shear and failure in studied specimens.

Established low temperature peculiarities are analyzed with taking into consideration the influence of internal stresses and contribution of grain boundary dislocations into the low temperature plastic deformation of nanostructured Ti-6Al-4V alloy. This work have being carried out within the program of the INTAS-01-0320 Project.

**P-22. N.V. Tokiy, V.N. Varyukhin, T.Ye. Konstantinova;** *Donetsk Phys. & Tech. Institute of the NAS of Ukraine, Donetsk, Ukraine;*

#### ***Dislocation Model Of Local Bend***

Our approach is to gain a fundamental understanding of mezo- and microstructure evolution during plastic deforming. In our previous paper [1,2] the structure of local bend was investigated in crystal. We was obtained information about the local bend by studying the electron microscopic extinction contour measurements [3]. Kroupa [4] solved the corresponding elastic problem for the deformation of a plate caused by an edge dislocation lying in the central plane. He considered the total angle of tilt. In contrast of Kroupa [4] the object of the present paper is local angle of bend.

On the basis of the mechanics of deformed solids and theory of dislocations, simulation of the local bend caused by flat uniform systems of the edge dislocations has been performed in thin foil. Equation for the angle of the local bend is found. Dependence of twist-bend tensor components in foil on distance  $S$  to defect center is obtained from the results of simulation in thin plate for the flat row of the edge dislocation of one sign. The modeling of central region of the local bend is carried out. It is represented in a form of two rows of the edge dislocations of opposite signs tilted with respect to the neutral surface at a some angle. Simulation results is obtained for the dependence of local rotation angle on distance  $S$  to centre of dipole formed by two flat rows of edge dislocations of opposite sign. Gradient of the angle change can be compared with parameters of the bending-torsion tensor determined by the electron microscopy. Dependence of twist-bend tensor components on distance  $S$  to dipole center is obtained from the results of simulation in thin plate for the dipole of flat rows of edge dislocations of opposite sign.

Dependence of twist-bend tensor components on distance  $S$  to dipole center is obtained from the results of simulation in unlimited crystal. Parameters of local-bend dislocations model have been determined by comparing the results of simulation with the electron microscopic measurements. Stress fields generated by the local bend within the infinite crystal are estimated. Our goal is to investigate the evolution of these stresses and relate them to the microstructure and processing conditions. Once a fundamental knowledge about stress evolution is obtained, it will also be possible to manipulate these stresses to enhance materials properties.

**P-23. Pawel Widlicki<sup>1</sup>, Halina Garbacz, Malgorzata Lewandowska, Waclaw Pachla<sup>2</sup>,**

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***Microstructure and mechanical properties of 6082 aluminum alloy processed by hydrostatic extrusion***

Severe plastic deformation has become a popular method for fabrication of ultra-finegrained metals. The most widely used methods for such a processing are: equal channel angular pressing (ECAP), torsion under compression (TUC) and multi-step forging (MSF). Large plastic strain may also be achieved by a hydrostatic extrusion which results in ultrafine and homogenous structure in the entire volume of the material. The aim of this work was to show that hydrostatic extrusion can be used to obtain nanostructured aluminum alloys.

Hydrostatic extrusion process of 6082 aluminum alloy was investigated. The material in the initial state was fully recrystallized. The specimens were subjected to the hydrostatic extrusion in consecutive steps up to the cumulated true strain of 3,75. The extruded specimens were cooled in water at the die exit. The microstructure of the alloy was examined using light and electron transmission microscopy and image analysis. Microhardness measurements and tensile tests were performed to characterize the mechanical properties. Hardness, microhardness and tensile tests showed that mechanical properties of the alloy significantly increase due to hydrostatic extrusion. The process also result in grain refinement, as evidenced by TEM microscopy.

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***Anomalous Nitrogen Solubility in Gradient Nanostructured Layer Formed in the Surface of Bulk Iron by Severe Plastic Deformation under Friction***

Gradient nanostructured surface layers determined by applying both surface severe plastic deformation and nitrogen diffusion simultaneously were studied. Specific features of composition and nanostructure of gradient layer were investigated using X-ray diffraction analysis and transmission electron microscopy. It was found that diffusion under friction results in anomalous nitrogen solubility in bcc-Fe ( $a_N$  – phase). Nitrogen content in gradient nanostructured layer of  $a_N$  – phase was found to be higher by 3 times compared to that indicated by state diagram for system Fe–N in high-temperature state. Essential increase of nitrogen content in  $a_N$  – phase was attributed to the increase of dislocation density arranged by great extension of block boundaries, which were formed due to the grain refinement.

# Sponsors



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# **Co-sponsoring Organizations**



General Electric Company Global Research Center



## Science&Technology Center in Ukraine

In 1993, Canada, Sweden, Ukraine and the United States of America concluded the "Agreement to Establish a Science and Technology Center in Ukraine". The goal of such a Center was to aid unemployed or underemployed scientists and engineers who had previously worked on the development of weapons of mass destruction and their means of delivery by converting their acquired WMD skills towards peaceful purposes. This goal satisfied the Ukrainian authorities since it would help in preventing a 'brain drain' thus preserving the countries scientific potential and wasto the benefit of Western nations who feared that Ukrainian scientists and engineers could be used for sinister purposes by countries unfriendly to the West.

The Science & Technology Center in Ukraine (STCU) was finally opened in 1995 when the first projects were financed. As an intergovernmental, non-profit organization with the status of a diplomatic mission, the STCU is rather unique. Sweden has subsequently been replaced by the European Union and Azerbaijan, Georgia and Uzbekistan have also become signatories to the agreement.

Thus one of the important results of the STCU's activities is the formation of a good and professional technology transfer team for the benefit of Ukraine, Uzbekistan, Georgia and Azerbaijan. In Ukraine, this team could form a future national organization for research and development.

The STCU's original, and still existent, method of aiding former WMD scientists is through funding so-called regular projects. Scientists submit a proposal for a project which then goes through an approval process, first by the Ukrainian government and then by the STCU Governing board. Those projects which have been funded have included every known field of science. Projects ranging from molecular biology to space research have been accepted for STCU funding. In total, the STCU has funded 661 peaceful scientific projects since 1995 worth a total of more than US \$93 million dollars. This funding has supported the work and livelihood of more than six thousand scientists and engineers throughout Ukraine, Georgia and Uzbekistan. With Azerbaijan's joining the STCU, these numbers will only increase. With time the STCU has managed to evolve to meet the changing needs of the scientists that it works with. Since 1997, the Center has developed new programs to meet these needs foremost of which is the *Partnership Program* which has a number of different aspects including Matchmaking, Promotional Missions, Seminars and Workshops, Partnership Projects, Travel Grants and Patenting and Conference Support. The Partnership Program was created in order to attract financing from governmental agencies and private sector non-governmental organizations to help the STCU's mission of engaging former weapons scientists.

The promotion of opportunities to prospective partners through *Matchmaking* visits to the STCU by interested businessmen, and outreach missions to Western countries to meet face-to-face with potential investors has had a positive effect on the Center's recipient state institutes.

The main aim of the *Promotional Missions* initiative is to increase awareness of the STCU's work and activities in Europe and to find new partners in EU countries. The next mission is planned to be held in Brussels jointly with the European Commission and the Delegation of Ukraine to the European Commission on November 3, 2003.

The *Seminars and Workshops* program has been instrumental in supporting networking between CIS and Western scientists and the dissemination of ideas on an international arena. Last years' highly successful workshop entitled *Ecological and Health Threats Associated with Environmental Contamination* attracted 146 experts and guests from 10 different recipient and donor countries.

The offering of *Travel Grants* is an enhanced program for STCU Project participants and proponents from Ukraine, Georgia, Uzbekistan, and Azerbaijan supported by the governments of the U.S.A., Canada and the European Union. STCU funding has been instrumental in inventing various new technologies and further lends a hand in patenting these new technologies through its *Patenting Support* program. Since its inception, this program has helped the Center's scientists receive 90 Ukrainian patents and 3 USA patents.

The Science and Technology Center in Ukraine sponsors *Conferences* on a competitive basis, regional scientific conferences on a topic in the basic or applied sciences. There has been already 82 conferences funded by STCU.

The STCU's major initiatives and efforts for the next several years "will be to promote the Center, the scientists we work with and some of the key core competencies of Ukraine, Azerbaijan, Uzbekistan and Georgia, in terms of their scientific capabilities, to non-governmental organizations of all kinds, in Europe, the USA and Canada" states Mr. Carmel, STCU Executive Director. He furthermore states that the Center "will act as an instrument to secure and safeguard the investment that an organisation may wish make here in Ukraine. We feel that this is an important element since the investment climate in our recipient countries still requires approval when it comes to investments. Therefore Western organisations are very prudent before making any investments in this region. However, there are real tangible opportunities for Western organisations to get access to excellent science and technologies. Because the Center is a diplomatic mission which has a strong organisation supported by our funding parties, we are an instrument that can be used to safeguard the investments of non-governmental organisations so that they can achieve their goals of getting access to the science and technology and make sure that their money is properly taken care of."

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# NOTES

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