

## ABSTRACTS

### **Horst Hahn**

*Nanoglasses and cluster-assembled metallic glasses: some results and future prospects*

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Nanoparticles and cluster ions have been used as building blocks to obtain metallic glasses with atomic structures and properties distinctly different from those of melt-spun amorphous materials. The consolidation of the building blocks in the size range from a few hundred atoms to 20 nm is performed in controlled UHV environments using mechanical compaction and energetic impact of the nanoparticles and clusters, respectively. The resulting structures are characterized by large fluctuations of the free volume and of the chemical composition on the length scale of the primary particles and by substantial differences of the atomic structures compared to melt-spun metallic glasses. Extensive structural characterization, using synchrotron-based techniques, electron microscopy, Mössbauer spectroscopy, positron lifetime spectroscopy, atom probe tomography, and others lead to the conclusion that the structural motifs and the medium-range order of the cluster- and nanoparticle-assembled glasses are different from those of melt-spun ribbons. These changes of the atomic structures lead to different thermodynamic, mechanical, electronic, magnetic and biocompatibility properties of the resulting metallic glasses.

### **Julia Ivanisenko**

*Microstructure and mechanical behaviour of ZrCu nanoglass*

Yulia Ivanisenko, Sree Harsha Nandam, Xiaoke Mu, Di Wang, Reda Chellali, Horst Hahn

The talk will give an overview of the structure and mechanical properties of nanoglasses taking Cu<sub>50</sub>Zr<sub>50</sub> as an example. Typical features of nanoglasses such as chemical inhomogeneity/interfacial segregations of some elements, deviation of the crystallization temperature, hardness, Young's modulus and more homogenous plastic deformation of nanoglasses compared to the rapidly quenched counterparts will be presented and discussed.

### **Ruslan Valiev**

*Novel effects in amorphous alloys subjected to HPT*

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In recent years the study of the effect of severe plastic deformation (SPD) on the atomic structure and behaviour of amorphous alloys (metallic glasses) attract growing interest because the properties of these materials are expected to improve significantly, including ductility.

This report presents the results of recent studies performed by the Russian team of the joint DFG-SPbSU project on the influence of high-pressure torsion (HPT), one of the most popular SPD techniques, on structural states and properties of amorphous alloys of several compositions. The effect of HPT regimes (temperature, strain and strain rate) on the formation of nanocluster

structure, free volume evaluation, shear bands formation was studied. Mechanical tests were also performed using nanoindentation and uniaxial tension testing, including strain rate sensitivity measurements, strength and ductility test as well as fracture toughness.

The obtained results demonstrate HPT as a promising technique to control atomic structure and improve the properties in metallic glasses. However, a number of questions still remain open – in particular those dealing with the formation of shear bands and nanoclusters – requiring discussion and further research.

### **Leonardo Velasco Estrada**

#### *Shear band characterization on bulk metallic glasses after HPT by using TEM and APT*

It has been proposed that by using severe plastic deformation on bulk metallic glasses it will be possible to generate amorphous structures comprise of medium and long range ordering, that is, analogous to nanoglass materials a core-shell type amorphous structure. Vitreloy 105 bulk metallic glass pellets (15 mm diameter and 0.75 mm thickness) were deformed by using high pressure torsion to study the possible microstructural and compositional changes that can occurred after such severe deformation. Several features that can be consider as shear bands are observed on the cross-sectional view of the sample. To study these features, the extraction of sample lamella from the region of interest was conducted by using focused ion beam (FIB), a first examination of the sample using transmission electron microscopy (TEM) shown no presence of shear bands. However step features could be observed on the top surface of the sample. After 2 weeks of sample storing in a glove box ( $O_2$  content  $<0.05$  ppm) the sample lamella was examined one more time in the TEM and reveled that following the step features there were shear bands. To understand these findings, the following experiments were conducted: (i) preparation of a TEM lamella with FIB follow by immediate observation in the TEM and chemical evaluation with atom probe tomography (APT), (ii) observation of the sample in TEM after storing for 3 weeks in a glove box and APT analysis after 1 week sample storing in air. This presentation will highlight the results obtained by TEM and APT techniques, described the microstructural and chemical changes at the shear band.

### **Askar Kilmametov**

#### *Density measurements of metallic glass solids using a new technique for small weight samples*

In this study a novel approach for precise density measurement of samples with a small weight in the range of 1–3 mg is presented. A new kind of displacement method has been developed, by which a solid object is dropped into a semi-confined cylindrical cavity, which is filled with a nonvolatile liquid, to determine the volume of the sample. Monitoring of the liquid level is performed by confocal laser microscopy with  $\mu\text{m}$ -resolution. A straightforward procedure is developed to build-up a mass/volume linear dependence. This enables to determine the density of small solids with an accuracy of  $\leq 0.5\%$ . The method appears to be very sensitive to deviations of the density due to microstructural peculiarities.

Different types of metallic glass solids, namely as rapidly quenched melt-spun ribbons, bulk nanoglass states processed by inert-gas condensation and severely deformed metallic glasses of altered compositions were used for comparative analysis of their bulk density measurements. Obtained results are discussed in frames of specific microstructures formed in dependence on the given processing route.

**Dmitri Gunderov**

*BMG Vit105 processed by HPT: SEM observations of shear bands, X-ray studies and the three-point bending tests*

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The effect of high-pressure torsion (HPT) on the structure and mechanical properties of the Zr-based BMG Vit105 alloy has been investigated. We used an original procedure for this study. Vit105 disks were first cut into two segments (halves), which were then jointly subjected to HPT processing. The procedure that we used enabled establishing the sequence of accumulation of shear bands during HPT processing with different strains using SEM. It is shown that under the used regimes of HPT processing, a high density of shear bands forms in the structure, observable by SEM on the previously polished cross-cut ends of the halves. With increasing number of revolutions, the density of shear bands increases ever more, and after HPT processing for  $n=5$  the distance between the bands is 0.5  $\mu\text{m}$ . At the same time, the XRD data show that the structure of the amorphous phase in the Vit105 BMG changes noticeably as a result of HPT processing, the radius of the first coordination sphere,  $R_1$ , and the free volume increase. In tensile tests BMG samples before and after HPT are brittle under a stress of about 2000 MPa. For the first time, the three-point bending tests of a BMG after HPT were performed. In the three-point bending tests of the initial BMG and the BMG after HPT, strength exceeds 3000 MPa and the plastic flow is fixed. A slight increase in the strength of the BMG Vit105 after HPT is observed, according to the results of the three-point bending tests.

**Vasily Astanin**

*Use of AFM for analysis of the shear bands in HPT-processed Vit105 BMG*

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Surface relief formed by shear bands in bulk metallic glasses (BMG) Vit105 under various types of loading, including high-pressure torsion (HPT), has been investigated by the method of atomic force microscopy (AFM). Shear bands after microindentation were also investigated using AFM accompanied by scanning electron microscopy (SEM) imaging. The height of the deformation steps was determined, and their fusion in the joints was also shown.

**Andrey Bazlov**

*Phase separation in amorphous Zr62.5Cu22.5Fe6Al10 alloy during thermo deformation processing*

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In recent years, metallic glasses, including bulk metallic glasses (BMG), have attracted great interest among the scientific community, thanks to their defect-free structure, which provides high strength properties. However, the almost complete absence of plasticity at room temperature makes it impossible to use them as construction materials. The deformation of metallic glasses occurs through the nucleation and propagation of shear bands. The main cause of the low ductility of these materials is the localization of deformation in one shear band. There are several ways to increase the plasticity of BMG, which consists either in creating homogeneously distributed surface defects by deformation processing, or by creating homogeneously distributed inhomogeneities. In this work, we analyzed the effect of thermo deformation processing on the structure and properties of an amorphous alloy Zr<sub>62.5</sub>Cu<sub>22.5</sub>Fe<sub>5</sub>Al<sub>10</sub>.

The ribbons of amorphous alloy were rolled at different temperatures with different strain up to 40%. The rolled ribbons were heat treated at temperatures below the glass transition temperature with different annealing times. Structure of the alloy before and after thermo deformation processing was studied using XRD, TEM and SEM. The mechanical properties of the alloy were evaluated using microhardness test and simple bending test.

The formation of a double-phase structure after thermo deformation processing was found. The phase released during the annealing process is not crystalline and has an amorphous structure. The second phase has a homogeneous distribution and its average particle size is about 7 nm. It is shown that an increase in the exposure time during annealing affects only the volume fraction of the precipitated phase, but not its size. After the thermo-deformation treatment, the alloy is not brittle and at the same time there is a significant increase in hardness (about 30%). The paper analyzes the effect of the strain and the annealing temperature on the structural parameters and mechanical properties of the alloy.

## **Evgeniy Boltynjuk**

*Analysis of strain rate sensitivity of metallic glasses subjected to HPT processing*

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Severe plastic deformation (SPD) is a promising method for transformation of structural and mechanical properties of metallic glasses. In this study Zr-based bulk metallic glass (BMG) and TiNiCu amorphous ribbons were subjected to high-pressure torsion (HPT) processing. The main attention was paid to the study of mechanical properties by means of nanoindentation. Using such technique values of hardness, Young's modulus and strain rate sensitivity were determined. Previously observed on Zr-based BMG tendency to enhanced values of strain rate sensitivity after HPT processing was confirmed on TiNiCu alloy. This observation may be interpreted as a general regularity of mechanical properties modifications of amorphous alloys by means of SPD. Values of shear transformation zones (STZs) of Zr-based BMG and TiNiCu ribbons were estimated using cooperative shear model of W.L. Johnson and K. Samwer [1] and approach of

D. Pan and coauthors [2]. It was found that HPT leads to a decrease in volumes of STZs of both amorphous alloys. The observed lack of serration flow, lower activation barrier for STZs activities along with the decreased sizes of STZs after HPT processing are indicators of more homogeneous deformation behavior of amorphous alloys processed by HPT. Further studies are required to better understand the deformation behavior and its correlation with the structural transformations of amorphous alloys caused by severe plastic deformation.

[1] Johnson W.L., Samwer K. Phys. Rev. Lett. 95 (2005) 195501.

[2] Pan D., Inoue A., Sakurai T., Chen M.W. Proc. Natl. Acad. Sci. U.S.A. 105 (2008) 14769-14772.

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### **Gleb Iankevich**

*Cluster ion beam deposition: synthesis of cluster-assembled metallic glasses.*

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Metallic glasses exhibit unique properties but the research on cluster-assembled metallic glasses and the correlation between their structure and properties are still lacking. The synthesis and research of such materials remains to be a complicated task for scientists. Cluster ion beam deposition (CIBD) is a technique which opens the opportunity to synthesize size controlled cluster-assembled metallic glasses under ultra-high vacuum conditions with outstanding precision and reproducibility in terms of cluster size, cluster amount, impact energy, and sample homogeneity.<sup>1</sup> The system is based on a combination of magnetron sputtering and inert gas condensation processes. *In situ* mass spectrometry allows to precisely measure the mass distribution of the formed clusters. The system already showed its significant capabilities in the case study of Fe<sub>80</sub>Sc<sub>20</sub> metallic glasses.<sup>2</sup> It was shown that the variation of cluster impact energy can lead to a significant change of the local atomic structure and magnetic properties. The Curie temperature changes by as much as 60 K when the deposition energy is increased from 50 to 500 eV per cluster. Further study of other metallic glass systems such as CuZr and FeB is planned. In my talk, I will focus on the system itself, experimental conditions, technological features and prospects of the CIBD system.

#### References:

1 A. Fischer, R. Kruk, and H. Hahn, Review of Scientific Instruments 86, 023304 (2015).

2 C. Benel, A. Fischer, A. Zimina, R. Steininger, R. Kruk, H. Hahn, and A. Léon, Mater. Horiz. 6, 727 (2019).

### **Nikita Kazarinov**

*Solid particle erosion testing. Perspectives to studies of amorphous materials.*

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We are going to talk about solid particle erosion setup that can be used to test and compare materials (for example sample after HPT) in solid particle erosion conditions. The setup is of a wind tunnel type: erosive particles are mixed into the air flow which is accelerated due to air

pressure in the system. The following parameters can be altered: the flow velocity, experiment duration, type of abrasive particles. Mass loss and surface degradation can be measured after the experiments allowing for estimates of the threshold particle velocities for particular material and conditions of the experiment. Using this technique, data on the abrasive wear of amorphous specimens subjected HPT can be obtained.

## POSTER SESSION

**Yuriy Mitrofanov**

*Shear modulus controlled heat release and absorption upon structural relaxation and crystallization of metallic glass*

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In the present time, physical nature of thermal effects occurring upon heating of metallic glasses remains unknown. These effects are observed as i) heat release below the glass transition temperature  $T_g$ , ii) heat absorption above  $T_g$  and iii) crystallization-induced heat release [1].

In the framework of the Interstitialcy theory, the heat flow  $W$  (heat per unit time and unit mass) is given as [2]

$$W = \frac{\dot{T}}{\beta\rho} \left[ \frac{G_{rt}}{G_{x,rt}} \frac{dG_x}{dT} - \frac{dG}{dT} \right], \quad (1)$$

where  $G$  and  $G_x$  are the shear moduli of glass and maternal crystal, respectively,  $G_{rt}$  and  $G_{x,rt}$  are the shear moduli of glass and maternal crystal at room temperature, respectively,  $\dot{T}$  is the heating rate,  $\rho$  is the density of glass and  $\beta$  is the shear susceptibility. According to Eq. (1), thermal effects are determined by shear moduli relaxation of glass and maternal crystal. For experimental verification of this equation, we performed high-precision *in situ* shear modulus measurements on a typical Zr-based metallic glass in the wide temperature range, from room temperature to the point of crystallization. We found that Eq. (1) provides a very good description all thermal effects – heat release below  $T_g$ , heat absorption in the  $T_g$ -region due to structural relaxation and heat release upon crystallization.

Our results suggest that all heat effects occurring upon heating of a Zr-based metallic glass have common structural origin, which can be understood as relaxation in the system of dumbbell interstitial-type “defects”, which can be treated as elastic dipoles [3].

### References

1. A.L. Greer, in Physical Metallurgy, D.E. Laughlin, K. Hono, Eds (Elsevier, Oxford) **I**, 305 (2014).
2. Yu.P. Mitrofanov, A.S. Makarov, V.A. Khonik, A.V. Granato, D.M. Joncich, S.V. Khonik. Appl. Phys. Lett. 101, 131903 (2012).
3. Yu.P. Mitrofanov, A.S. Makarov, D.P. Wang, W.H. Wang, V.A. Khonik. Sci. Rep. 6, 23026 (2016).

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## **Evgeniy Ubyivovk**

*TEM studies of amorphous alloys after HPT*

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Influence of high pressure torsion (HPT) processing on structural change of amorphous alloys was investigated. Using high-resolution transmission electron microscopy (HRTEM) and scanning transmission electron microscopy (STEM) it was shown previously [1], that HPT treatment may leads to various structural transformations such as shear banding, nanocrystallization and formation of cluster-type amorphous structure. However, peculiarities of such structural transformations and spatial distribution of above mentioned “events” are still insufficiently studied and unclear. In this work, on the basis of experimental data, we have tried to conduct a comparative analysis and to establish the influence of different parameters of HPT on the appearance or non-appearance of various structural features in amorphous alloys.

[1] E.V. Ubyivovk, E.V. Boltynjuk, D.V. Gunderov, A.A. Churakova, A.R. Kilmametov, R.Z. Valiev, Materials Letters 209, 327-329, 2017

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## **Diana Khalikova**

*Non-uniformity of microhardness distribution in the BMG Vit105 prior to and after HPT processing*

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We study the microhardness distribution at different points on the surface of a sample from the amorphous alloy Vit105 prior to and after HPT processing. According to the obtained data, in the initial sample the values of Hv are uniformly distributed across the sample, and the difference in the values from the upper and lower surfaces is within the measurement error. In the initial sample, on average, higher values of Hv are recorded than in the HPT-processed sample, i.e. HPT processing leads to a decline in microhardness. Microhardness in the HPT-processed sample is distributed non-uniformly. Hv is the highest on the end surface of the sample, it is slightly lower on the lower surface, and even lower on the upper surface of the HPT-processed sample. Such a microhardness distribution could be related to internal stresses arising in the process of HPT. Previously, the non-uniformity of microhardness distribution in a Zr-based BMG processed by HPT was reported in the paper [1].

[1] Nozomu Adachi, Yoshikazu Todaka, Yoshihiko Yokoyama, Minoru Umemoto, Cause of hardening and softening in the BMG Zr<sub>50</sub>Cu<sub>40</sub>Al<sub>10</sub> after HPT, Mat. Sci. & Eng. 2015, 627, C. 171–181

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**Dmitri Gunderov**

*Three-point bending tests of the Vit105 BMG processed by HPT*

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The effect of different types of treatment on the mechanical properties of the Zr- based BMG Vit105 alloy is investigated by three-point bending tests. The facility for three-point bending tests of microspecimens (9x1x0.5 mm) was designed in Ufa. These specimens can be produced from HPT-disks. Our studies show that both the initial Vit105 alloy and the HPT-processed alloys exhibit some ductility during three-point bending tests. Ductility expectedly increases with decreasing thickness of a tested specimen. After HPT processing, strength is slightly higher and certain ductility is preserved, although we do not observe any increase in ductility yet. Further, we can vary the HPT processing regimes (temperature, strain, groove shape) and establish the optimum HPT processing regimes providing an increase in the ductility of the BMG. Low-temperature annealing with the relaxation of free volume leads to an expected decline in ductility. Treatment by thermal cycling in a temperature range of -160 C to +180 C with the number of cycles equal to 1 leads to an increase in the ductility of Vit 105. A further increase in the number of cycles of thermal cycling results in a decline of ductility. These studies need to be further analyzed and continued.