MS46 P01

Reconstruction of High-Temperature Deformation Processes in Zr-based Alloys Perlovich Yu.[a], Isaenkova M.[a], Krymskaya O.[a], Kropachev S.[b], Akhtonov S.[b] [a]*Moscow Engineering Physics Institute, Moscow,Russia.* [b] *Chepetckiy Mechanical Plant, Glazov, Russia.*
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Keywords: Zr-based alloys-1, high-temperature deformation-2, phase transformation-3.

Processes of high-temperature deformation are reconstructed as applied to commercial Zr-Nb alloys by features of diffraction lines, measured at room temperature. These alloys are characterized by phase transformations α↔β within a temperature interval 610° - 860°C, where the high-temperature β-phase and the low-temperature α-phase have crystalline lattices BCC and HCP, respectively. Forging at temperatures of β- and (α+β)-regions of the phase diagram are favorable for operation of various deformation mechanisms, among which there are crystallographic slip in α- and β-phases as well as mutual displacements of crystallites along interphase boundaries under phase transformation or along intergranular boundaries under dynamic recrystallization. Experimental study was conducted as applied to model samples and real billets from Zr-based alloys, subjected to forging by various temperature-rate regimes [1]. In order to reveal the concrete mechanisms, operating by different deformation regimes, changes of X-ray line profiles and the texture by passing from one regime to another were considered. It was ascertained, how sensitive are main parameters of X-ray lines relative to different substructure features.
Deformation at temperatures of (α+β)-region is inevitably associated with α→β phase transformation due to additional local heating near shear bands. Besides, deformation at the phase transformation temperature additionally reduces stability of the crystalline lattice, so that repeated cyclic phase transformations prove to be possible. Activation of the phase-boundary slippage, resulting in texture scattering, is most probable by equal contents of both phases. As the deformation temperature increases, a volume fraction of β-grains and their size grow, the phase-boundary slippage weakens and the intragranular crystallographic slip intensifies. Structure features of alloys at high temperatures determine the operating deformation mechanisms as well as the character of quenching effects, which control the physical broadening of X-ray lines, measured for α-matrix at room temperature.
Parameters of α-Zr crystalline lattice vary depending on the content of dissolved Nb and other alloying elements, on the dislocation density and, most of all, on the residual strain. The content of α-phase in the alloy at the deformation temperature decreases with growth of this temperature, and a volume reduction of the deformed sample due to β→α phase transformation by the following quenching becomes more uniform as the temperature of previous deformation increases. The phase transformation α→β develops with a volumetric expansion, and at high temperatures stresses due to interaction between residual α-grains and new-originated β-grains are relaxing. Therefore, when grains of modified α-phase, experienced α→β→α phase transformations, return by cooling to the initial volume, their crystalline lattice proves to be extended. This elastic strain depends on the deformation temperature, i.e. on the fraction of α-grains, participated in phase transformations, and on the development of deformation-induced phase transformations.

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MS46 P02

X-ray induced structural changes in organic and biological crystalline materials. F. Camus[a], C. Besnard[a], A. Dahlström[a], I. Margiolaki[b], P. Pattison[a,c], M. Schiltz[a], [a]*Laboratory of Crystallography, EPFL, Lausanne, Switzerland.* [c]*ID31, ESRF, Grenoble, France.* [c]*SNBL, ESRF, Grenoble, France.* E-mail: fabrice.camus@epfl.ch

Keywords: synchrotron powder diffraction, radiation chemistry, radiation damage studies

The investigation of radiation-induced processes in organic and biological molecules is of importance for gaining a better understanding of the fundamental mechanisms by which certain compounds (*e.g.* halogenated nucleotides) induce a radio-sensitizing action and can thus be used to improve anticancer radiotherapies since they allow to selectively enhance the therapeutic effectiveness of ionizing radiation by increasing tumor-cell killing and minimizing normal-tissue toxicity. Molecular damage to the DNA molecule induced by ionizing radiation in living organisms plays also a critical role in the development of tumors and substantial research efforts are underway in this field but relatively little is known about the chain of molecular and structural processes that leads from the initial radiation event to a DNA strand break. . Our project focuses on the component molecules of DNA – nucleobases and nucleosides.
We have carried out powder diffraction measurements to investigate structural changes as a function of X-ray irradiation in organic and biological crystals. In these experiments, synchrotron radiation is used to both irradiate the samples and collect diffraction data. Powder diffraction is employed to monitor changes in the unit cell and micro-structural parameters (crystallite size and lattice strain) in crystals of native and halogenated nucleobases and nucleosides under X-ray irradiation. Our aim in these studies is to investigate radiation-induced changes as a function of temperature, wavelength and X-ray dose rate. Attempts to interpret the observed unit-cell expansions in terms of radiation-induced structural modifications in the crystal will be discussed.

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X-ray Powder Diffraction Analysis of Sigma-Phase in Welded Fe-Cr-Ni Alloys Jorge L. Garin, Rodolfo L. Mannheim, *Department of Metallurgical Engineering, Universidad de Santiago de Chile, Santiago, Chile.*
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Keywords: x-ray diffraction, sigma phase, Fe-Cr-Ni alloys

Commercial Fe-Cr-Ni alloys such as cast heat-resistant steels are an important class of elevated-temperature materials currently being considered for welding applications in the metallurgical and mining industry; however, the metallurgical characterization of fusion welds of these materials in service at elevated temperatures, has shown the precipitation of intermediate

complex phases, where sigma-phase is the most significant compound in the microstructure, due to its detrimental influence on the mechanical properties of the alloy. This phase is a complex intermetallic compound of Fe and Cr, based upon an ideal stoichiometric composition AX_2, Pearson's code tP30 and space group P_2/mnm.
Owing to the usually complex diffraction patterns, which disclose many overlapping reflections, and the strong textures caused by the welding process, the Rietveld method was used to resolve those difficulties in to representative welded joints, such as HC-type (25Cr-3Ni) and HD-type (30Cr-6Ni). The Rietveld refinements were performed based upon typical measurement and global parameters. The powder diffraction patterns of the weldments resulted in strong preferred orientation effects due to the uniaxial solidification of the weld metal-pool, which was corrected in the Rietveld refinement by using the March-Dollase function. The pseudo-Voigt function was used for the simulation of the peak shapes, while the background was modeled by a 3rd order polynomial in 2θ with refinable coefficients. A total of five phases, namely ferrite (Cr,Ni), austenite (Ni,Cr), sigma phase, $Cr_{23}C_6$ and Cr_7C_3 were identified and considered in the quantitative analysis. The results obtained revealed that a quantitative microstructural characterization of the weld in heat resistant steels is properly achieved by Rietveld processing of the x-ray diffraction data.

MS46 P04

Characterization of elastic properties of nanostructured thin films by X-rays G. Geandier[a] P.-O. Renault[a], Ph. Goudeau[a], E. Le Bourhis[a], O. Castelnau[b], [a]LMP – Université de Poitiers, France, [b]LPMTM - *Université Paris 1, France*
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Keywords: X-ray diffraction, strain determination, thin films

Understanding the mechanical behaviour of nano-structured thin films in relation with their microstructure is of high importance for the development of technological applications. The present approach to the problem consists to prepare thin films with controlled microstructure, then to characterize the mechanical response of these films thanks to the development of diffraction based techniques, and finally to analyse experimental results using mechanical modelling of elastic grain interaction. Our research was mainly oriented on the investigation of the elastic properties of single phase thin films (W or Au) [1, 2]. We have shown the feasibility of such experimental techniques and verified that the mechanical behaviour is related to the gold thin film microstructure, i.e. the texture [3]. We are now focussing our research on nanometric multilayer such as W/Au and W/Cu systems elaborated by ion beam sputtering techniques in order to study the mechanical behaviour for different period thicknesses. These metallic multilayers are supported by a (thin) polyimide substrate. Multilayer mechanical response of the two diffracting phases is characterized experimentally through in situ tensile testing in diffractometers available at our laboratory and at synchrotron beamlines. Results are interpreted by an appropriated mechanical modelling accounting for the material microstructure, based on homogenization schemes [4].

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MS46 P05

Microstructure of Metals after Severe Plastic Deformation Studied by Different Methods. Radomír Kužel[a], Viktoria Cherkaska[a], Zdeněk Matěj[a] Miloš Janeček[a], Jakub Čížek[a], Milan Dopita[a,b]. [a]*Faculty of Mathematics and Physics, Charles University in Prague, Ke Karlovu 5, 121 16 Praha 2Czech Republic*[b]*TU Bergakademie Freiberg, Institute of Materials Science, Gustav Zeuner Str 5, D-09599 Freiberg, Germany. E-mail: kuzel@karlov.mff.cuni.cz.*

Keywords: XRD line profile analysis, texture, severe plastic deformation

Severe plastic deformation is an effective tool for production of compact sub-microcrystalline materials of high purity and no residual porosity. In principle, there are two basic techniques – equal channel angular pressing (ECAP) and high-pressure torsion (HPT). In present work, samples prepared by both techniques were studied. Copper and copper composites with different amounts of Al_2O_3 and Zr were selected for studies by different techniques - X-ray powder diffraction (PXRD), transmission electron microscopy (TEM), positron life-time spectroscopy and electron back-scattered diffraction (EBSD). Conventional powder diffraction was performed with the aid of Seifert-FPM diffractometer XRD7 and with Panalytical X'Pert Pro. The evaluation consisted mainly in the line profile analysis for the estimation of dislocation density and crystallite size. Pole figures were measured with Panalytical MRD equipped with the Eulerian cradle and polycapillary in the primary beam.
Line broadening analysis showed that the HPT samples (6 GPa, 7 rotations) have smaller crystallite size compare to the ECAP samples in the range 100 – 300 nm, the dislocation densities are similar – of the order of $1\times10^{15}m^{-2}$. In samples with Al_2O_3 smaller crystallites below 100 nm were found. There are only small changes in the mean dislocation density with the increasing number of passes for ECAP. This fact is well confirmed also by positron annihilation. TEM pictures discovered that after the first pass, the dislocation cells strongly elongated along {111} planes and with low-angle boundaries appeared. After the second pass the grain size is slightly reduced and after the fourth pass, the fraction of equiaxed subgrains increased and the larger proportion of high angle grain boundaries was observed. After eight passes almost homogeneous microstructure with equiaxed subgrains separated by mostly high-angle grain boundaries was observed.
Pole figures obtained from HPT samples indicate fiber texture (often mixed (111)-(100)). ECAP samples have quite complicated textures that are changing with number and type of passes. The measurements were performed in the plane transversal to the direction of pressing. The dominant component is (111) slightly inclined to the surface. With increasing number of passes more components appear and they are broader. After 8 passes, the (110) component is the strongest one. It seems that in