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

samples with zirconium the texture components are sharper.
The above findings were more or less confirmed by the EBSD analysis. The analysis of the deformed and recrystallized parts indicate the changes from deformed part after one pass to about 70% recrystallized fraction after four and eight passes.

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**MS46 P06**

**Structural and ferroelectric characteristics in the $Ba(Ti_{1-x}Zr_x)Li_{xy}O_{3(1-xy)}F_{3xy}$ solid solution** K. Taïbi[a], A. Kerfah[a], A. Guehria-Laidoudi[a], A. Simon[b] and J. Ravez[b]
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**Keywords: lead-free, ferroelectric, transition**

Relaxor are a special class of ferroelectrics with very interesting properties. Relaxor materials actually used are lead-based ceramics which present a disadvantage due to the toxicity of PbO. The actual evolution of research is oriented to environment-friendly application. In this way, we have previously investigated new lead free compositions. Dielectric studies performed on $Ba(Ti_{1-x}Zr_x)Li_{xy}O_{3(1-xy)}F_{3xy}$ solid solutions showed three kinds of behaviour. For compositions very close to $BaTiO_3$ the three phase transition were retained as was the case in the classical ferroelectric $BaTiO_3$. For compositions $Ba(Ti_{1-x}Li_x)O_{3(1-x)}F_{3x}$ and $Ba(Ti_{1-x}Zr_x)O_3$, it appears one broad peak with frequency dispersion characteristic of relaxor behaviour. For compositions close to $Ba(Ti_{1-x}Zr_x)O_3$ only one diffuse phase transition, without frequency dispersion was evidenced. In this latest domain, it is possible to observe the spontaneous phase transition from ferroelectric to relaxor state just by thermal change.
To understand the origin of this behaviour much works based on structural and physical models are performed. It has been found that the frequency and the temperature $T_m$ can be described by using the Vogel-Fulcher relationship [3, 4]. In the present work we fitted all the dielectric data to this Vogel-Fulcher equation and correlated the results with structural data obtained by powder diffraction study. The fitting parameters and the upper limit of the composition for ceramics with relaxor state were defined. The plot of Tm(f)/Tm (1 kHz) versus the logarithm of the frequency allows to determine the limit of composition between the ferroelectric and the relaxor state which is close to 0.155.
Powder XRD patterns indicates a particular behaviour for $y = 0.25$ where two kinds of single domain phases are observed: tetragonal, for $0 \le x < 0.15$ and cubic for $0.15 \le x \le 0.25$. The latest domain corresponds to the relaxor phase. The change from tetragonal to cubic symmetry related to the disappearance of the (200) and the appearance of the (111) reflexions, characteristic of the cubic perovskite structure. The relaxor to normal ferroelectric transformation and the structural phase transition are strongly related to the domain morphology evolution. The presence of a local disorder related to nanoscale heterogeneities is responsible of the relaxor behaviour when the composition deviates from the well known $BaTiO_3$ [3, 4].

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**MS46 P07**

**Structural studies of crystallization and growth of magnetron deposited $TiO_2$ thin films by X-ray diffraction and reflectivity** Kužel Radomir, Zdeněk Matěj[a]., Jan Šícha[b], Jindřich Musil[b], [a]*Department of Condensed Matter Physics, Faculty of Mathematics and Physics, Charles University in Prague, Ke Karlovu 5, 121 16 Praha 2*, Czech Republic, [b]*Department of Physics, Faculty of Applied Sciences, University of West Bohemia in Pilsen, Czech Republic*. E-mail : nichtova@gmail.com

**Keywords: thin films; $TiO_2$, anatase; dc magnetron sputtering; microstructure**

$TiO_2$ films are nowadays widely used because of their interesting photocatalytic and self cleaning properties. Complex X-ray scattering studies were performed on sets of titanium dioxide thin films sputtered by dual dc magnetron [1]. Three sets of nanocrystalline and amorphous $TiO_2$ thin films magnetron deposited on glass and silicon substrates have been studied. Phase analysis and X-ray line broadening were studied by X-ray powder diffraction in parallel beam optics; the residual stresses were measured with the aid of the Eulerian cradle and surface roughness determined by X-ray reflectivity measurement. Microstructure parameters were extracted from XRD measurements by individual peak profile fitting and also by whole powder pattern modelling [2] approach (MAUD [3], modified FOX[4]).
By both thickness dependence of XRD patterns of nanocrystalline films and depth profiling measurements it was found that rutile phase growths on the substrate and it is transformed to anatase with increasing distance from the substrate. This may be caused by temperature gradient during deposition. Another set of amorphous films with different thickness was studied after annealing and also by in-situ measurements during the heating.
It was found that the crystallization temperature started at about 250 °C for thicker films but it was higher for thinner films (< 200 nm) and reached about 350 °C. Thinner films were single phase (anatase) while thicker films above 1200 nm contained also a smalll amount of nanocrystalline rutile. The crystallite size of these samples immediately after crystallization was larger than 100 nm by contrast to the nanocrystalline films which did not show any significant changes after annealing in this temperature range and their crystallites remain small under about 10 nm. Annealing at temperatures above 500 °C leads to increase of crystallite size and transformation of anatase into rutile.
Simple uniaxial tensile stress and only a weak texture were found for the amorphous films after crystallization. Only for the thinnest films (~ 100 nm), the 101 texture (anatase) was found. In case of nanocrystalline films the stress was low but complicated. This can be related to significantly stronger and more complicated texture due to dual magnetron geometry.