**PR.17.00.17 INVESTIGATION OF THE DISLOCATION STRUCTURE PARAMETERS IN SINGLE CRYSTALS BY X-RAY METHOD.** Sergey V. Ulshin and Olga P. Karasevskaya. Institute of Metal Physics,Ukrainian Academy of Sciences, 36,Vernadsky pr.,252142 Kiev,Ukraine

The basis for this method is the kinematic theory of X-ray scattering by non-ideal crystal with dislocations. This X-ray method allows to obtain three-dimensional intensity distribution from of I(q) in the cases of homogeneous and nonhomogeneous dislocation structure (DS), pole figure, average meanings of I(q) and their dispersion and to compare theoretical and experimental of I(q) for real crystals and identify DS.

But real crystals very often contain mixture of different DS with numerous parameters, that's why their analyze is complicated.In our paper we modeled several X-ray reflexes and found out that for crystals with different DS reflexes parameters strongly depend on their {hkl} and diffraction vector orientation. Therefore it is necessary to select reciprocal lattice sites, which can allow us to obtain the smallest error for structure parameters. The mathematical criterion for the choosing of the sum of sites which are suitable for investigation have been proposed by us. The investigations of I(q) in the cases of different types DS being in the crystal have shown that I(q) differs essentially for different sites and is very sensitive to types the DS. This makes it possible to develop the express-method for qualitative determination of the DS evolution during the process of single crystals deformation of new kind of the dislocations.

The parameters DS in BCC single crystals were studied. As a result of our experiments the change of the density of screw and edge dislocations {110},{112},{123}<111> and their distribution during the creep in the temperature interval (0,3-0,6) Tm established. At the high temperature creep multilevel DS is formed. The screw dislocations density really does not increase. At the initial stage of deformation correlation of the edge dislocations density 9:3:1 for the {110},{112},{123}<111> is formed.Than after 3-6% deformation depending on the single crystals orientation one sign surplus dislocation density essentially increases. At the low temperature creep dislocations density increase linearly with deformation.Correlation of the edge dislocations density 20:2:1 for the {110},{112},{123}<111> is formed.

## Theory, Techniques And Instrumentation

**PS17.01.01 SIMULATION OF SYNCHROTRON WHITE BEAM TOPOGRAPHS: APPLICATION TO THE STUDY OF PIEZOELECTRIC DEVICES** . Y. Epelboin*, B. Capelle* & J. Détaint**, *LMCP, URA 009 CNRS, University P.M.Curie, Paris, France, **CNET, PAB, dept BAG/MCT/CMM, 92220 Bagneux, France

We present a new package which allows to simulate synchrotron white beam topographs for any kind of defect. It is split in two parts: a first program which computes the derivatives of the deformation and which must be written by the user for each model. A second general purpose simulation program takes these data and computes the image (Y. Epelboin, 1996, accepted in J. Appl. Cryst.). This program is valid for all studies. This allows to rapidly check new models for the defects without having to rewrite a whole simulation program.

A new algorithm for the integration of Takagi-Taupin equations has been written for massively parallel computers and multiple processors machines. It computes in parallel different lines of the image. The numerical method (C.A.M. Carvalho & Y. Epelboin (1993) Acta Cryst. A49, 460-467) has been modified to be able to vectorize the computation and to make it as efficient as possible. The simulation of an image needs from one up to three hours using one processor on a Cray C9x. It may be reduced by a factor equal to the number of available processors thus to less than one hour in all cases.

The study of the propagation of acoustic waves in piezoelectric devices is given as an example of the possibilities of this new program. The devices are circular plates from a quartz AT-cut crystal. The experimental topographs have been recorded by B. Capelle, J. Détaint & A. Zarka (1995). We consider an ideal case where the acoustic vibration mode is a pure thickness-shear mode. Two models have been investigated. describing the deformation inside the crystal either as exponentially decreasing cosine functions (Stevens & Tiersten, (1986) J. Acoust. Soc. Am. **79**, 1811-1826) or as a development with Bessel functions (Détaint & al. (1991) Proc. 45th Annu. Frequency Control Symp., pp 166-180).

The agreement between the experiments and the simulation is good except in the center of the resonator, which may be explained by imperfections in the geometry of the devices.

**PS17.01.02 A NEW LIGHT MICROSCOPY TECHNIQUE FOR EXAMINING BIREFRINGENT MATERIALS.** J.G. Lewis & A.M. Glazer, Clarendon Lab., Dept. Of Physics, Univ. Of Oxford, Oxford OX1 3PU, UK

A new imaging technique based around a polarising microscope is demonstrated (U.K. Patent Application Number BG9604785.7) [1]. Color coded images of optically birefringent materials are produced where colour represents | sin δ | (δ relative retardation) or ϕ the orientation of the cross section of the indicatrix of the specimen. Thus the contract seen in direfringent materials between the usual crossed polars setting is seperated out into its components. With this instrument the two values (ϕ and | sin δ |) can be easily quantified and monitored spatially with the resolution of a traditional light microscope.

Images of crystals, minerals and biological birefringent specimens are shown and analysed in both a qualitative and quantitative manner.

[1] Glazer, A.M. Lewis, J.G. & Kaminsky, W. *Nature* (awaiting publication).
[2] Wood, I.G. & Glazer, A.M. *J.Appl. Cryst.*, 133, 217-223 (1980).

**PS17.01.03 X-RAY CHARACTERIZATION OF TEXTURE IN THIN FILMS USING A TWO DIMENSIONAL POSITION SENSITIVE DETECTOR** P.D. Moran, Siemens Energy and Automation, Inc., Analytical Instrumentation, 6300 Enterprise Lane, Madison WI. 53719-1173, USA

By employing a low-noise, two-dimensional position sensitive x-ray detector it is possible to record pole figures in less than an hour from films that are as thin as 200 Angstroms. As well, "Area Detector" techniques allow one to accurately characterize sharply textured films, such as those seen in thin film optoelectronic and superconducting materials, with a resolution not practical with conventional point-detector based texture instruments. Third, by simultaneously collecting data from the substrate, one can also investigate the relationship of texture in the film to the crystallography of the substrate on which it was grown. The ability to accomplish these three tasks is primarily due to the ability of an area detector to simultaneously measure data from a large amount of reciprocal space at the same time. Of course, the measurements discussed above require different data collection and analysis strategies. The dependence of the strategy on the degree of texture present is discussed through practical examples of texture measurements from broadly and sharply textured thin films. Finally, the strategies employed using Area Detector techniques are examined through a comparison with the strategies that would be employed using a conventional point detector-based texture instrument to accomplish the same goals.

**PS17.01.04 QUANTIFICATION OF STACKING FAULTS IN SYNDIOTACTIC POLYSTYRENE SINGLE CRYSTALS.** Masatoshi Tosaka, Noritaka Hamada, Masaki Tsuji, Masahiro Fujita, Shinzo Kohjiya, Institute for Chemical Research, Kyoto University

Syndiotactic polystyrene (sPS) crystals have a unique character; i.e., they have inherent stacking faults in beta-form single crystals (orthorhombic $P2_1P2_1P2_1$, a=2.87nm, b=0.88nm, c(chain axis)= 0.51nm)[1]. In this study, we tried to determine the proportion of these faults from electron diffraction (ED) patterns and to investigate the dependence of the proportion on crystallization and annealing temperatures. The reli-

ability of estimated values was examined by comparison with the directly counted number of these faults recorded in high-resolution TEM images. Existence of stacking faults is readily deduced from ED patterns of sPS single crystals. They are characterized by certain hk0 (h+k=odd) reflections streaked in the a*-direction; while the other reflections (h+k=even) are spot-like. From the analysis of these patterns, we have already proposed a model for the faults; the faults are defined as successive (irregular) sequence in the regularly alternating sequence of two motifs, each of which is made up of two molecular layers spread along the b-direction [1]. Based on the model, the proportion of the fault can be determined by converting the half-breadth of streaked hk0 reflections. Before the conversion, a component due to instrumental broadening was subtracted from the breadth, assuming that their profiles are Gaussian and instrumental broadening is reproduced on spot-like reflections.

The estimated proportion showed a maximum at 165deg.C. Then, single crystals grown at this temperature, which are to have a maximum proportion of the faults, were annealed for one hour at various temperatures. The proportion of the faults decreased with annealing temperature, but annealed crystals still had more faults than those crystallized at the same temperatures. In the assumed model, these faults can be cancelled only when they move in the a-direction until they reach to a crystal edge or when two faults collide with each other. Thus the small decrease of the proportion by annealing suggests rather small mobility of these faults.

[1] Tsuji et al., MSA Bulletin, 23, 57(1993).

**PR17.01.05 DEVELOPMENT OF ROCKING CURVE METHOD FOR POLYCRYSTALLINE MATERIALS.** S.Ya. Betsofen, Baikov Inst. of Metallurgy, Russian Academy of Sciences, 49 Leninsky pr. Moscow 117334 Russia

The rocking curve (ω-scanning) method is successfully used for misorientation measurements of single crystals. The electron channelling patterns (ECP) method use to measure of grain orientation of polycrystalls with a variety of grain size. The time consuming procedure of this method is limited for its application for industrial scale specimens with nonuniform microstructure. In the present paper the various quantative procedures for microstructure characterization on the base ω-scanning technique are developed for polycrystals with grain size more than 20 μm. The principal advantages of the method is that it allows to make the non-destructive measurement and also it gives more statistic validity. The technique allows to measure a volume fraction of recrystallized grains and its grain size distribution for the different grain orientations (in accordingly to a number of X-ray reflexes under measurement). It permits to assess the actually texture formation mode for the recrystallization process. The application of the ω-scanning method for development of the production technology of the textured Ti parts is demonstrated in the present paper. The dynamic and static recrystallization behaviour of a commercial grade Ti is studied on the basis of texture mode changes during industrial processing steps. The damage accumulation under loading in form subgrain fragmentation is obtainable from the ω-scanning patterns for structural materials which are used in recrystallized state, such as Ni-superalloys. On this base the non-destructive method of residual lifetime forecast was proposed for the aircraft engine discs. A volume fraction of a precipitated phase in recrystallized matrix also can be estimated from the ω-scanning patterns, for example a fraction of the γ'-phase in the Ni-superalloys.

**PR17.01.06 X-RAY METHODS TO THE CHARACTERIZATION OF THIN COATINGS.** S. Betsofen* and L. Petrov**. *Baikov Inst. of Metallurgy, Russian Academy of Sciences, 49 Leninskiy pr. Moscow 117334 Russia; **National Inst. of Aircraft Technology

The features of characterization methods such as texture, residual macrostress, microhardness and X-ray fluorescence thickness measurement are considered. The X-ray fluorescence method is considered in comparison to the other methods. The sensitivity, accuracy and the limits of thickness measurement are cosidered for the various combinations of the coating and the substrate. The predominant orientations of Ti monolayer coatings are observed: {1010}, {1120} and {1011} in depend of deposition condition. The different orientations of TiN monolayers are observed in the range from {110} for low of both the nitrogen pressure and arc current to {111} for higher of its. The semicoherent Ti-TiN interface is obtained in the case multilayer coatings. In this case the orientation of layers are {111} for TiN and {0001} for Ti. The orientations of {1010} and {1120} for Ti and {111} for TiN are obtained in the case of diffuse interface. The features of macrostresses measurement concern with thin high textured coatings are considered. The contribution of both the texture and elastic anisotropy to macrostress measurement are calculated in terms of elastic anisotropy theory. The X-ray reflection for which elastic anisotropy lead to linear or nonlinear dependences of X-ray strain vs. $\sin^2\Psi$ are obtained. The effects which are associated with nonuniform distribution of macrostrain in a graines having a different orientation are considered. The macrostress phenomenon concern with mismatch of thermal extension coefficient (TEC) of coating and substrate, deposition parameters and layer sequence in the multilayer coatings are discussed. The texture of alternating layers like Ti, $TiB_2$, $Al_2O_3$, AlN and SiC, which have the TEC anisotropy ($\alpha_c \neq \alpha_a$), can be used for governing of macrostresses by the formation of specific textured layers.

## Applications

**PS17.02.01 ON POSSIBILITY OF STOICHIOMETRY CONTROL FOR SEMICONDUCTOR $A^3B^5$ SINGLE CRYSTALS BY A X-RAY DIFFUSE SCATTERING METHOD.** Kirill D.Chtcherbatchev and Vladimir T.Bublik, Moscow State Institute of Steel and Alloys, Dept. of Semiconductor Materials & Devices, Box 034, Leninskii pr. 4, 117936 Moscow, Russian Federation

A method of stoichiometry control for low dislocation density ($N_d<10^4 cm^{-2}$) $A^3B^5$ single crystals based on a measurement of a X-ray diffuse scattering (XRDS) by microdefects is proposed. Point defect clusters of various sizes, shapes and nature (for example, dislocation loops, inhomogenities with diffuse boundary, nuclei of metastable and stable phases etc.) can be determined as microdefects (MD). The feature of a stoichiometry composition is a high probability of an annihilation of nonequilibrum interstitials and vacancies in each sublattice without MD formation. Hence the principal criterion of the state is a minimum of XRDS. The formation of MD may proceed in two independent ways. The fist one is the association of point defects which became nonequilibrum during the post-crystallization cooling. The second one is the formation of MD during the decomposition of supersaturated by A or B component solid solution. A contribution of the second process in MD formation increases with increasing of a deviation from stoihiometry. We used the method to control stoihiometry in two systems. The first one is undoped InSb single crystals grown from a melt with various contents of Sb (50, 51 and 52%). And the second one is Te-doped GaSb ($n=1.5 \div 13.8 \cdot 10^{17} cm^{-3}$). We analyzed both Huang and asymptotic diffuse scattering by microdefects. Using of the latter one proved to be preferable under the conditions of simultaneous presence of MD both of negative (vacancionic) and positive (interstitial) sign of dilatation. This method gave an opportunity to study the MD which were not revealed by TEM and selective etching. We found out that the crystal grown from the melt with 51% at. Sb had the most stoichiometric composition. We also managed to fix a transfer through a pseudobinary section GaSb-$Ga_2Te_3$ (the sample GaSb(Te) $n=13.8 \cdot 10^{17} cm^{-3}$). Hence the obtained results can be solid background for creation of highly informative and nondestructive method of stoichiometry control.