

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  $hk0$  ( $h+k=\text{odd}$ ) reflections streaked in the  $a^*$ -direction; while the other reflections ( $h+k=\text{even}$ ) 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 ( $\omega$ -scanning) method is successfully used for misorientation measurements of single crystals. The electron channelling patterns (ECP) method use to measure of grain orientation of polycrystals 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 quantitative procedures for microstructure characterization on the base  $\omega$ -scanning technique are developed for polycrystals with grain size more than 20  $\mu\text{m}$ . 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  $\omega$ -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  $\omega$ -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  $\omega$ -scanning patterns, for example a fraction of the  $\gamma'$ -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 considered 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,  $\text{TiB}_2$ ,  $\text{Al}_2\text{O}_3$ , 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<sup>3</sup>B<sup>5</sup> SINGLE CRYSTALS BY A X-RAY DIFFUSE SCATTERING METHOD.** Kirill D. Chitchebatchev and Vladimir T. Bublik, Moscow State Institute of Steel and Alloys, Dept. of Semiconductor Materials & Devices, Box 034, Leninskiy pr. 4, 117936 Moscow, Russian Federation

A method of stoichiometry control for low dislocation density ( $N_d < 10^{10} \text{cm}^{-2}$ ) A<sup>3</sup>B<sup>5</sup> 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 nonequilibrium 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 first one is the association of point defects which became nonequilibrium 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 stoichiometry. We used the method to control stoichiometry 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 ( $n = 1.5 \div 13.8 \cdot 10^{17} \text{cm}^{-3}$ ). 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<sub>2</sub>Te<sub>3</sub> (the sample GaSb(Te)  $n = 13.8 \cdot 10^{17} \text{cm}^{-3}$ ). Hence the obtained results can be solid background for creation of highly informative and nondestructive method of stoichiometry control.