Determination of Burgers vector populations in single crystals and strongly deformed materials with X-ray diffraction

Theses of Ph.D. dissertation

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Introduction

The lattice defects influence the different behavior of the materials. These defects can be investigated by X-ray measurement on various methods. Duo to the lattice defects the X-ray line profiles can shift and broaden. This broadening can be divided with two main parts, size and strain broadening respectively. The X-ray line broadening caused by dislocation is strongly anisotrop, it means that the broadening of reflections with different hkl indices is very different on a characteristic manner. In the case of randomly textured policrystalls or so called powder samples, averaging can be applied so that the averaging must be done all of the permutation in accordance the multiplication of the selected hkl indices. The averaging has as a result that we can only determine, from anisotrop line broadening of different hkl indices, the fraction of the edge and screw dislocation in the sample. Although this can be also very useful information about the real microstructure of the materials, but it is very insufficient for even the determination of the active slip systems or the investigation the Burgers vector population. The determination of these microstructural properties is possible only with investigation of even strongly textured or single crystal samples. I have chosen my investigation, my different samples and the developed processes in accordance with the above summarized concepts. I developed simple methods and models using the dislocation induced X-ray line broadening, in order to determine the Burgers vector population and dislocation density of the investigated samples. I have shown the effectiveness of these models in the following cases:

- I have determined the active slip systems and the Burgers vector population in plastically deformed germanium single crystals, the samples were compressed at high temperature. I have shown the inhomogenity of deformation.

- I have determined the dislocation densities of different grain populations in strongly textured and deformed NiAl samples.

- I have determined the active slip systems and the Burgers vector population in Cu-Nb multilayers.

Applied methods

The x-ray line profiles were measured on a special double crystal fine focal x-ray tool. This Nonius FR591 fine focus x-ray equipment had the following settings: 40 kV and 70 mA. I measured the x-ray profiles with two different detectors, with a linear position sensitive gas flow detector (OED 50 Braun, Munich) and with imaging plate two dimensional detector (Fuji BASH). The instrumental effect was smaller than 10 % of the physical broadening in both cases. In the case of gas detector the sample detector distance was 420 mm. In the case of imaging plates this distance was 700 mm. By recording the reciprocal space mapping the single crystal was measured 15 different angle "ω" position along rocking curve. The footprint of the beam on the sample surface was 0.1x0.6 mm^2. This small beam size enabled the scanning of the sample with moving the sample itself. By these measurements I oriented my samples so that the longer dimension of the beam was parallel to the visible slip lines on the sample surface. This geometry enabled the investigation of the inhomogeneous deformation and the fluctuation of the microstructure.

The evaluation of the measured profiles I have made with the help of "multiple whole profile" (MWP) software developed by Ungár and Ribárik.

Theses

1. The results of my investigations in germanium single crystals[1]:

   (a) I have measured germanium single crystals oriented single slip geometry, the samples were plastically deformed at high temperature. I have determined the active slip systems as well as the Burgers vector population in deformed Ge single crystals. I have found that the number of the active slip systems decreases with increasing deformation. This implies that in the investigated deformation range increasing the deformation the deformation takes place with the help of only one slip system. This is the slip system, which has the largest Schmid factor.

   (b) I have determined that the observed deformation is strongly inhomogeneous. I have shown this inhomogenity on two different scales. It can be also seen that toward to the edge of the samples the dislocation density is increasing. With the help of the reciprocal space mapping and scanning the samples, I could reveal that the samples are broken into mosaic blocks. This is because of the deformation and the boundary conditions of the deformation.

2. Results of the investigation on NiAl samples with <100>, <110> and <111> texture, which were deformed by 6 and 30% respectively. [2]:

   (a) Crystallites belonging to different texture components behaves like "soft" and "hard" regions depending on the direction of the deformation axes. I have determined the dislocation densities in different, "soft" and "hard", texture components with the help of different behavior of the x-ray line profile broadening measured two face of crystal, one face was parallel, and another was perpendicular to the deformation axis. I have calculated the individual contrast factors of the measured x-ray line profiles using the "multiple whole profile" (MWP) whole profile fitting procedure.

I determined the dislocation densities in different grain populations with compare the measured and calculated individual contrast factors.

(b) I have plotted together the x-ray line profile broadening of different grain population on a so called pseudo Williamson-Hall plot. With the help of this kind of plots I have shown the qualitative fraction of the dislocation densities in different grain population.

(c) I have found that in the so called hard grains with <100> orientation, the dislocation density is approximately half of that in the soft ones, with the exception of the sample with 6 % deformation and <111> texture. In that sample the orientation of soft particles is random. In this texture two different processes hinder the activation of <111> Burgers vectors: the first is the small Schmid factor, the second is the large absolute value of Burgers vector.

(d) I have investigated the stored energy of the dislocation and its relationship with the dislocation density. I have found that the Schmidt rule and the slip geometry define the dislocation density and so the stored energy, instead of the minimum of the stored energy. This result means that during plastic deformation the free energy of the system play not so important role than the geometrical constraints of the deformation.

3. Results of investigation on CuNb multilayers [3]:

(a) The dislocation densities in the initial state of Cu-Nb multilayers are very high about 2x10^{16} m^{-2} in both layer types. This is in accordance with the fact that in the strong textured layers, which are fitted together by Kurdjumov-Sachs rules, the large misfit is relaxed with the help of dislocation.

(b) I have determined that the large initial dislocation density cannot be increased by increasing the deformation of the multillayers. The dislocation densities of both layers are not changing significantly due to the further rolling. These phenomena can be explained with the followings: however huge dislocation flux goes through the material but because of the continuously annihilation of the dislocations the total dislocation density cannot grow further. During the deformation the system is in stationer state aspect of the dislocation density.

(c) With the help of Burgers vector analysis I have found that in the initial state the Burgers vectors lie in the foil plane in both layer types, and it does not change in Cu layers after deformation. However in Nb layers the Burgers vectors become randomized due to the deformation and new Burgers vectors appear which do not lie in the foil plane. I have shown that these Burgers vectors can be born with dislocation reaction induced by deformation.

Publications

1. **K. Nyilas**, C. Dupas, T. Kruml, L. Zsoldos, T. Ungár, J.L. Martin: "Dislocation structures and mechanical behaviour of Ge single crystals deformed by compression." Mat. Sci. Eng. A 387-389 25-28 (2004)

2. T. Ungár, **K. Nyilas** , W. Skrotzki , "Dislocation densities in soft and hard oriented grains of compressed NiAl polycrystals" Int. J. Mater. Res. 99 725-733 (2008)

3. **K. Nyilas**, A. Mistra and T. Ungár: "Micro-strains in cold rolled Cu-Nb nanolayered composites determined by X-ray line profile analysis" Acta Materialia 54, Issue 3, Pages 751-755, February 2006