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Shape and size of non-spherical particles in single crystals, investigated by SAXS

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Abstract

The analysis of small-angle scattering (SAS) from single crystalline alloys provides a high quality of information on decomposing systems: size, shape and orientation of particles can be determined from three dimensional SAS data of single crystals. Adequate evaluation procedures for SAS data from single crystals are, e. g. , simulations of two dimensional (2D) scattering patterns and evaluation of slices along special directions in reciprocal space. These techniques, which allow the full exploitation of the instrumental resolution, are demonstrated with the examples of ellipsoidal and plate-like precipitates in alloys. In favourable cases the upper sensitivity limit can be increased up to 4000 Å at a sample to detector distance of only 1m.

1 Introduction

Small-angle scattering (SAS) is used in various materials to study inhomogeneities of sizes in the nanometer range. Important applications of the method in materials science are studies of decomposing alloys [1,2]. SAS can be used to characterize the time-evolution of such systems; i. e. the development of size, shape and crystallographic orientation of precipitates.

The SAS signal from (ideal) polycrystalline samples is spherically averaged by superposition of intensities from many differently oriented grains. Considerable loss of information takes place for non-spherical particles due to this averaging process. This disadvantage can be avoided at least for coherent inclusions by the use of single crystals [1]. In this case there are usually only a few orientations of particles according to well defined crystallographic relationships between precipitates and matrix. The strict alignment of non spherical particles along well defined directions or planes leads to reduction or increase (depending on the shape of particles) of the SAS intensity along corresponding directions of reciprocal space. A method, based on the deviations from circular symmetry for the determination of dimensions and shapes of particles is presented in this work. It requires a pinhole SAS camera to avoid the smearing out of the data (like from slit geometry), and two dimensional detection of the SAS intensity.

We demonstrate methods of data evaluation for 2D SAS patterns, using precipitates in Ti- and Cu-alloys as model cases. In b.c.c. Ti-Mo alloys particles with *small* aspect ratio were investigated: in this material the decomposition process starts via precipitation of metastable, coherent ω -phase particles of ellipsoidal shape [2]. After long-time or high-temperature annealing plate-like particles of α -Ti appear, at the expense of ω -particles [5]. Data evaluation for particles of *large* aspect ratio is demonstrated with oxide precipitates in Cu-Fe alloys.

2 Data evaluation

2.1 Particles with small aspect ratios

Figure 1: (a) SAS intensity (in logarithmic scale vs. scattering vector k) in the (110)-plane from ellipsoidal ω -phase precipitates in single crystalline Ti-12at%Mo after 96h at 450°C. (b) Simulated SAS-intensity from ellipsoids of revolution with an aspect ratio of 2:1.

SAS from particles with small aspect ratio deviates only slightly from spherical symmetry. As a consequence, the scattering from particles of different orientations is not separated into distinct regions of reciprocal space and superposition has to be taken into account. In Fig.1a 2D SAS from precipitates of ω -phase is shown in the (110)-plane of b.c.c. Ti-12at%Mo [9]. Iso-intensity contours are plotted in a logarithmic scale vs. scattering vector k . $[1\bar{1}0]$ is horizontal, $[001]$ is vertical in this and in the following 2D figures. The nearly spherical particles give rise to a SAS intensity, which is of the same order of magnitude along every direction. The only remarkable deviation is a flattening of the contours along $\langle 111 \rangle$, due to the elongation of particles along these directions; i.e. ω -precipitates are prolate ellipsoids with elongated rotation axes along a cubic $\langle 111 \rangle$ direction.

The shape of the 2D SAS was used to determine the aspect ratio of the ellipsoid-like particles. To this end simulations of the data were performed with the scattering function of ellipsoids [5,7]. The measured intensity results from a sum over all equivalent particle orientations. In addition a size distribution of particles has to be taken into account in order to obtain the smooth curves presented in Fig.1b. For slightly anisometric particles the shape of the SAS pattern is very sensitive to changes of the aspect ratio. For ω -particles in Ti-Mo an aspect ratio of 2:1 was found to give the best fit of the data (Fig.1b). The size of a semi-axis of the ellipsoids can be determined from evaluation of a radial slice in the 2D SAS pattern. This evaluation is similar to the "Guinier-evaluation" of spherically averaged data [4,6]. Unlike the case of polycrystals, the size of both semi axes can be determined by this procedure, since the aspect ratio of particles is known from the shape of 2D SAS patterns. The semi axes of the ω -particles in Ti-Mo corresponding to Fig.1a were determined to be 40 $\text{\AA}$ and 80 $\text{\AA}$.

The transition from ω - to α -particles leads to a strong increase of the 2D SAS intensity along $\langle 111 \rangle$ -directions; i.e. along directions where flattening has been observed for ω -particles (compare Fig.1a with Fig.2a). From this fact the α -particles were determined to be flattened along $\langle 111 \rangle$ and to be extended within the $\{111\}$ -habit planes of particles. Superposition of scattering from different orientations had to be considered also in this case, since the intensity streaks are not very well pronounced according to the low aspect ratio of the α -plates.

Figure 2: (a) 2D SAS intensity in the cubic (110)-plane from α -Ti plates in single crystalline Ti-12at%Mo after 40h at 500°C. The habit plane of particles is {111}. (b) Simulated SAS for disks with this habit-plane, diameter 190 Å and thickness 70 Å.

The scattering function for cylinders was used to determine the thickness and the diameter of α -particles [4,6]. From radial slices along a streak - i.e. perpendicular to the cross-section plane of the correlating particle - the average thickness of plates was determined. Their diameter was determined by slices across the streaks, i.e. from their width: the larger the diameter, the sharper the intensity concentration along the streaks. This kind of slices was performed at finite distance from the direct beam ("off-center") in order to keep clear off the direct beam, where superposition of scattering from differently oriented particles occurs [9]. The resulting size parameters (diameter 190 Å and thickness 70 Å) were used for the simulation, presented in Fig. 2b.

2.2 Particles with large aspect ratio

In Fig. 3a the 2D SAS of platelike oxides in a Cu-1at%Fe single crystal is plotted in the cubic (110)-plane. Due to the high aspect ratio of these particles the SAS intensity is concentrated along sharp streaks in {111} and {100} directions. Additional measurements of the samples in other orientations confirmed, that these streaks are produced by two families of plate-shaped particles, lying on {111} and {100} habit planes [9]. Compared with Fig. 2a the streaks are much narrower, due to the much higher anisotropy of the oxide particles. This concentration of the scattering from strongly anisometric particles into small regions of the reciprocal space leads to the separation of scattering from differently oriented precipitates. In fact, superposition of scattering occurs in Fig. 3a only near the direct beam and need not be considered: not only the intensity *across* a streak is due to only one orientation of plates (as in the last example) but also the intensity *along* a streak can be interpreted as resulting from only one orientation of platelike particles (except the region of very low scattering vectors).

After correcting the data with the resolution function of the apparatus [4,8] the diameters of two families of oxide plates were determined to be 2500 Å for {111}-habit and $d > 4000$ Å for {100}-habit planes. Evaluation of slices along the streaks gives as average thickness 195 Å for plates with habit plane {111} and 330 Å for the {100}-habit. This information was used to simulate the 2D data (Fig. 3b). Intensity oscillations along the streaks in Fig. 3b come from the monodisperse distribution of particles. The thickness of the streaks is smaller - compared to the measured data - since the instrumental resolution has not been taken into account for the simulations.

Figure 3: (a) 2D SAS of oxide plates in a Cu-1at%Fe single crystal. (b) Simulated SAS intensity for disklike particles on $\{111\}$ and $\{100\}$ habit-planes.

This example shows that in favourable cases - for strongly anisometric particles - dimensions up to $\approx 4000 \text{ \AA}$ may be determined. Compared to conventional evaluation of Guinier radii from polycrystals [9], the resolution increases almost by one order of magnitude. This is possible due to the exploitation of the instrumental resolution by evaluating "off-center" slices: While radial slices (e.g. for Guinier evaluations) usually suffer from parasitic scattering near the direct beam and from the cut-off due to the beam-stop, slices across intensity streaks at finite scattering vectors are not restricted by these apparative handicaps.

In conclusion, size and orientation of anisometric particles in single crystals can be determined from the shape of the 2D-SAS in several (well chosen) planes of reciprocal space. For slightly anisometric particles the shape of the 2D scattering patterns is a useful measure for the aspect ratio. The dimensions of strongly anisometric inclusions can be determined by evaluation of slices along and across the correlated intensity streaks in reciprocal space.

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