Determination of grain size and grain size distributions from the nano to the micro scale by means of EBSD

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Physical properties of crystalline materials often strongly depend on the grain size of the crystallites. Mechanical properties like the yield strength may be the most prominent ones but also electrical and magnetic properties can be strongly influenced by grain boundaries [1]. Only in very rare cases one has a single grain size throughout the sample. Depending on their synthesis route, polycrystalline materials generally have a more or less pronounced distribution of grain sizes. This means, that not only the average grain size but also the distribution of grain sizes will affect the properties of the investigated material. Exemplarily, we use an inert-gas condensed iron sample which is nanocrystalline (D = 11 nm) in the as-prepared state. By annealing, it is possible to coarsen the microstructure of this material. In this study, we want to show that electron backscatter diffraction (EBSD) is a suitable tool to analyse the grain size and the grain size distribution of microstructures in the range from several tens of nanometres up to the micrometre regime without the need of a sample destruction as it would be necessary with transmission electron microscopy.
In the limit of very small grain sizes (D < 50-100 nm), i.e. the case of a real nanocrystalline structure, wide-angle x-ray scattering is a common method for the determination of the average grain size of the microstructure under investigation. Advanced methods like the Warren-Averbach analysis [2] allow the determination of a volume and an area averaged mean grain size and so enable to calculate the width of the grain size distribution whenever its general functional shape, usually a lognormal distribution, is known. Figure 1 shows the grain size distribution of the as-prepared igc-Fe sample. The width was determined by the differently weighted average grain sizes evaluated by x-ray peak analysis according to the Warren-Averbach method. Depending on the instrumental equipment and the investigated material the accessible range of grain sizes usually does not exceed 50-100 nm.
For a few years now, EBSD is becoming established as a reliable tool to quantitatively analyse average grain sizes and grain size distributions of polycrystalline materials [3]. The domain of accessible grain sizes ranges between 100 and 1000 nm which is otherwise difficult to access by standard means like x-ray diffraction or optical microscopy. As matter of fact, EBSD is able to close this gap but care must be taken when comparing average grain sizes measured by different methods. The mean grain size evaluated by x-ray diffraction is a volume average while the data measured by EBSD represent an average over a cross-sectional area, i.e. it is an area averaged value. In contrast to x-ray diffraction, EBSD yields direct evidence of the grain-size distribution and thus offers the possibility to calculate all moments of the distribution function [4].
Our measurements were done in a JEOL 7000 F scanning electron microscope equipped with an EDAX Trident EBSD analysis system. Different average grain sizes of our sample were obtained by consecutive annealing at different temperatures in a reducing atmosphere (95vol% N_2, 5vol% H_2). Figure 2 shows a typical EBSD scan (Inverse Pol Figure) and the appropriate grain size distribution of the igc-Fe sample after annealing at 600°C.

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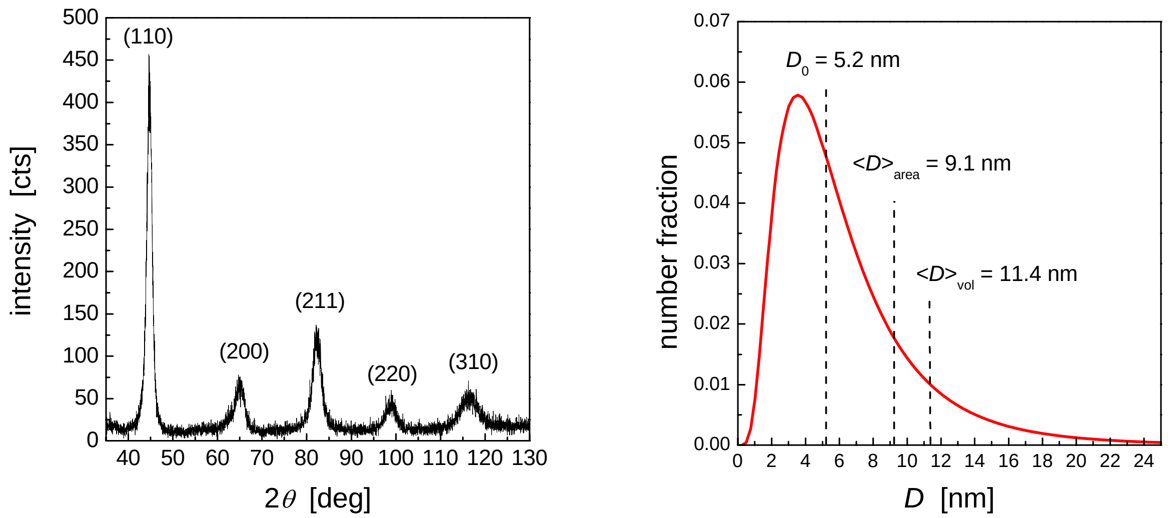

Figure 1. Wide-angle x-ray pattern of igc-Fe ($Cu_{K\alpha}$ - radiation). Shown on the right-hand side is the grain size distribution and values of average grain sizes differently weighted

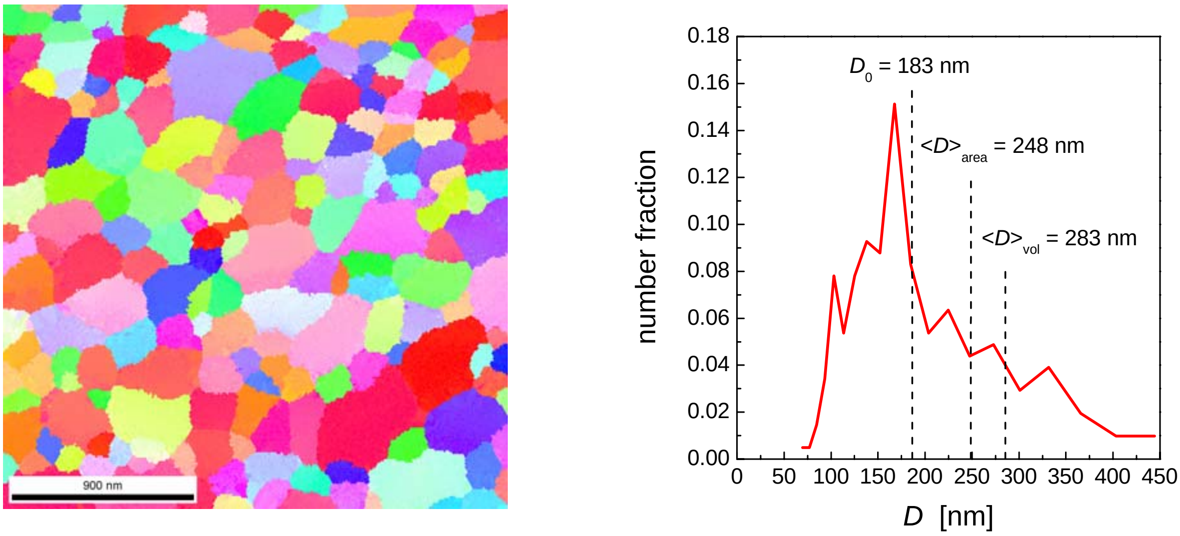

Figure 2. EBSD scan (Inverse Pol Figure) and the appropriate grain size distribution of the igc-Fe sample after annealing at 600°C

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