Quantitative local scale analysis by electron diffraction applied to nanocrystalline FeAl

C. Gammer[1], C. Mangler[1], C. Rentenberger[1] and H.P. Karnthaler[1]

1. Physics of Nanostructured Materials, University of Vienna,
Boltzmanngasse 5, 1090 Wien, Austria

christoph.gammer@univie.ac.at

Keywords: nanocrystalline material, electron diffraction, domain size, local information

X-Ray diffraction (XRD) is routinely used as an integral method for quantitative analysis of nanocrystalline materials however recent developments of nanostructured materials necessitate the quantitative characterization on a local scale. We show that peak analysis of selected area diffraction patterns (PASAD) can be used for a quantitative analysis of nanocrystalline materials on a local scale.

B2 ordered single crystalline intermetallic FeAl can be transformed into a bulk nanocrystalline material that is disordered using high pressure torsion (HPT). HPT deformation of Fe-45at.%Al was performed at room temperature with a pressure of 8 GPa. Transmission electron microscopy (TEM) samples were prepared from the HPT sample by electropolishing. TEM investigations were carried out with an acceleration voltage of 200kV.

The evolution of the nanostructure occurs by the formation of shear bands containing disordered nanocrystalline material. With increasing shear strain the volume fraction of the nanocrystalline material increases. Figure 1a shows that the sample consists of two different structural phases: a highly deformed ordered single crystalline phase and adjacent to it a nanocrystalline one. After a shear strain of about $\gamma=50$ the material is entirely nanocrystalline (cf. Fig. 1c). It is the aim to analyze locally the evolution of the nanocrystalline structure.

For the quantitative local analysis selected area diffraction patterns (SAD) were taken from the areas of interest (cf. Fig. 1b+d). The PASAD-software (cf. Fig. 2) [1] was used for deducing diffraction profiles from the SAD by ring integration (cf. Fig. 3). Peak broadening was studied with our software by fitting combined Voigt peak-functions. The instrumental peak broadening can be deduced from the width of diffraction spots of a single crystal. To compensate for this, the results were deconvoluted. From the half width at half height of a diffraction peak (Δg), the coherently scattering domain size (CSD) was calculated using the Scherrer equation ($D_{med}=0.443 \cdot (\Delta g)^{-1}$) [2]. The {200}, {211} and {220} peaks were taken into account. To obtain a good statistical analysis at least 10 SAD were analyzed for each sample and the mean value and the standard deviation were calculated.

Figure 4 shows the CSD sizes calculated from the profiles of Figure 3. As the CSD size does not change with γ, it is concluded that the nanograins are not further refined during the deformation. This means that the nanocrystalline structure locally formed during the early stages is the same as that of the homogeneous nanocrystalline structure finally reached. In the latter case our results can be compared with those from X-ray measurements of the CSD size, leading to a good agreement. Our work demonstrates that electron diffraction is a powerful tool for determining CSD sizes of nanocrystalline materials *locally* that could not be achieved by X-ray methods.

1.   PASAD-software. www.univie.ac.at/pasad
2.   F. Fultz and J.M. Howe, "Transmission Electron Microscopy and Diffractometry of Materials", Springer Verlag, Berlin Heidelberg, 2008.
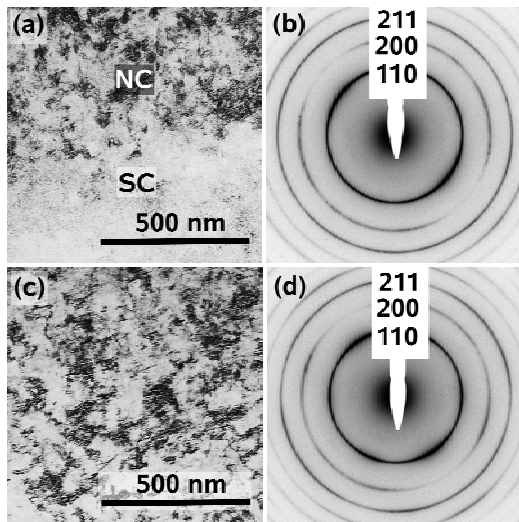
Figure 1. (a) TEM bright-field image of HPT-deformed FeAl (γ=23) showing two structural phases: the severely deformed single crystal (SC) and the nanocrystalline material (NC). (b) SAD corresponding to NC. (c) TEM bright-field image of homogeneous NC (γ=71). (d) SAD corresponding to (c).
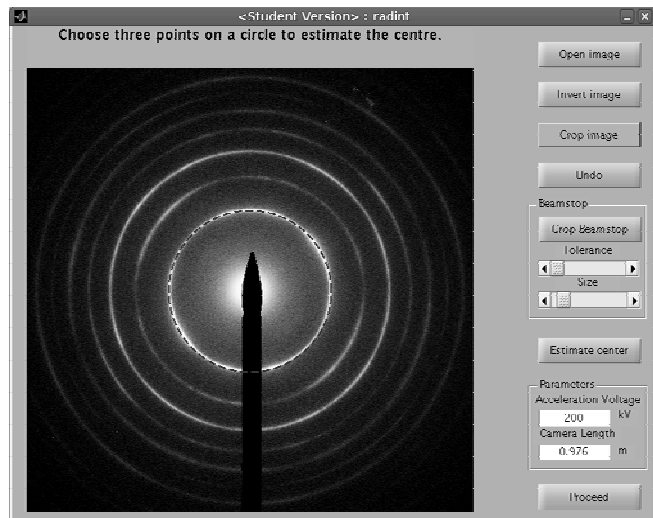
Figure 2. PASAD software. Radial integration of the SAD is done with high precision; the center of integration is refined automatically. The resulting diffraction profile is automatically background subtracted, the peak positions are detected and combined Voigt peak-functions are fitted to the diffraction profile [1].
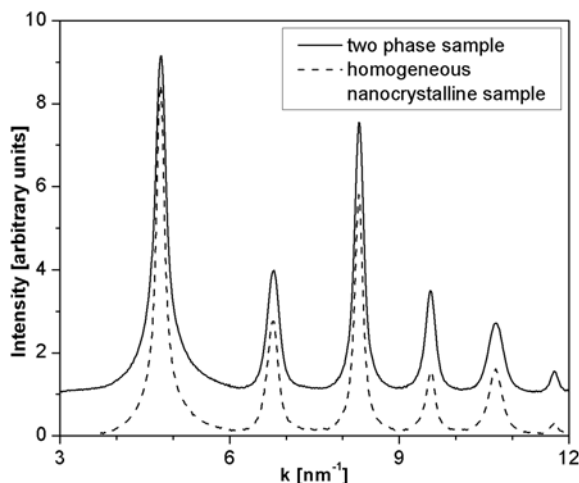

Figure 3. Diffraction profiles of nanocrystalline HPT deformed FeAl (γ=71) and of the NC volume present in the sample with two structural phases (γ=23).
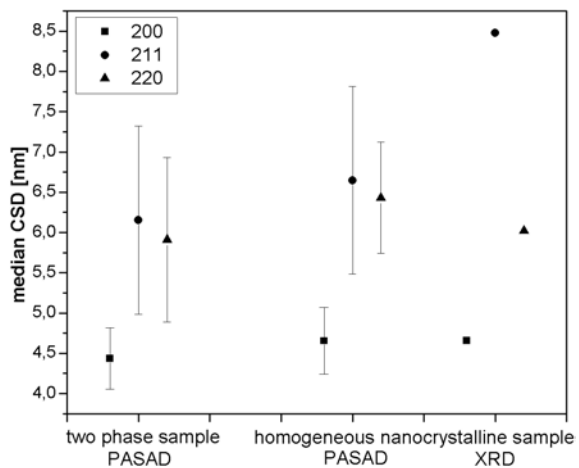
Figure 4. Comparison of the coherently scattering domain sizes (CSD) of the NC volumes: in the sample with two phases (γ=23) using PASAD and in the homogeneous nanocrystalline sample (γ=71) using both PASAD and XRD.