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Quantitative local structure analysis of nanocrystalline FeAl by electron diffraction

Rentenberger C.¹, Gammer C.², Karnthaler H. P.¹

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University of Vienna, Physics of Nanostructured Materials, Vienna, Austria,

2

National Center for Electron Microscopy, LBNL, Berkeley, California, USA

Email of the presenting author: christian.rentenberger@univie.ac.at

Profile analysis by X-ray diffraction has been proven to be able to obtain microstructural parameters averaged over a large sample volume ($>10\mu\text{m}^3$). In nanocrystalline materials it is frequently the case that local information is required. This can be achieved by local quantitative analysis based on selected area electron diffraction (SAED). Using the method of PASAD [1] that provides profile analysis of SAED patterns we show that structural parameters can be deduced of volumes on a submicrometer scale ($<0.01\mu\text{m}^3$).

Nanocrystalline B2 ordered FeAl with a mean grain size of about 35nm was made by high pressure torsion (HPT) followed by a heat treatment [2]. The achieved nanocrystalline material was exposed to a further HPT deformation (3 turns, 8 GPa). SEM studies indicate that the deformation occurs inhomogeneously in the form of shear bands. TEM studies were carried out using 200kV.

Fig. 1 shows a bright field image of a nanocrystalline FeAl sample after further deformation by HPT. The complex contrast variations are caused by orientation variations of individual grains and by lattice defects. The darker band in the middle of the image corresponds to a shear band (SB). The density of the dislocations is so high that it is not possible to determine it. Therefore, we use an alternative method. Fig. 2 shows an SAED pattern taken from the encircled area (cf. Fig. 1). The pattern consists of concentric rings. Using PASAD-tools [1] an intensity profile as a function of the diffraction vector g is obtained by integration along the rings (cf. inset Fig. 2). The broadening of the peaks (half-width at half maximum, HWHM) corrected for instrumental broadening was studied by fitting combined Voigt peak-functions. Since broadening by grain size and strain has different effects on the peak profiles both of them can be determined using the method of modified Williamson-Hall plots [3]. This is shown in Fig. 3(a) taking the contrast factors C of dislocations (slip system $\langle 111 \rangle \{110\}$) into account. The slope of the curve is proportional to the square root of the dislocation density. The values of the slope were calculated from 35 SAED patterns arranged in a 5x7 array within the area indicated in Fig. 1. Fig. 3(b) shows a contour plot of the slope values as a function of the SAED positions. The values indicate that even in a nanocrystalline material the dislocation density within a shear band can be up to a factor 4 higher than in the neighbouring area.

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[2] C. Mangler, C. Gammer, H. P. Karnthaler, C. Rentenberger. *Acta Mater* 58 (2010) 5631.

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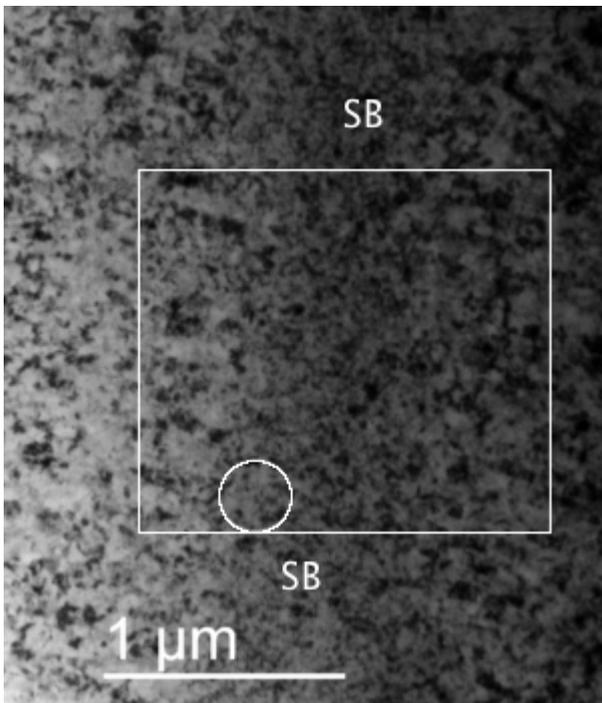


Fig. 1: TEM bright-field image of nanocrystalline FeAl deformed by HPT. Structural parameters were measured by profile analysis of SAED patterns of 35 circular areas placed within the marked rectangle.

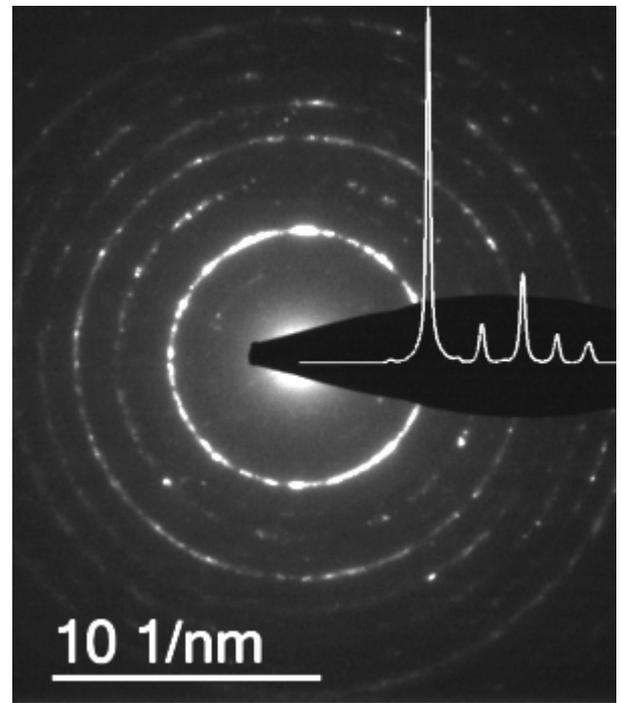


Fig. 2: TEM selected area electron diffraction pattern of the encircled area indicated in Fig. 1. The inset shows the corresponding intensity profile.

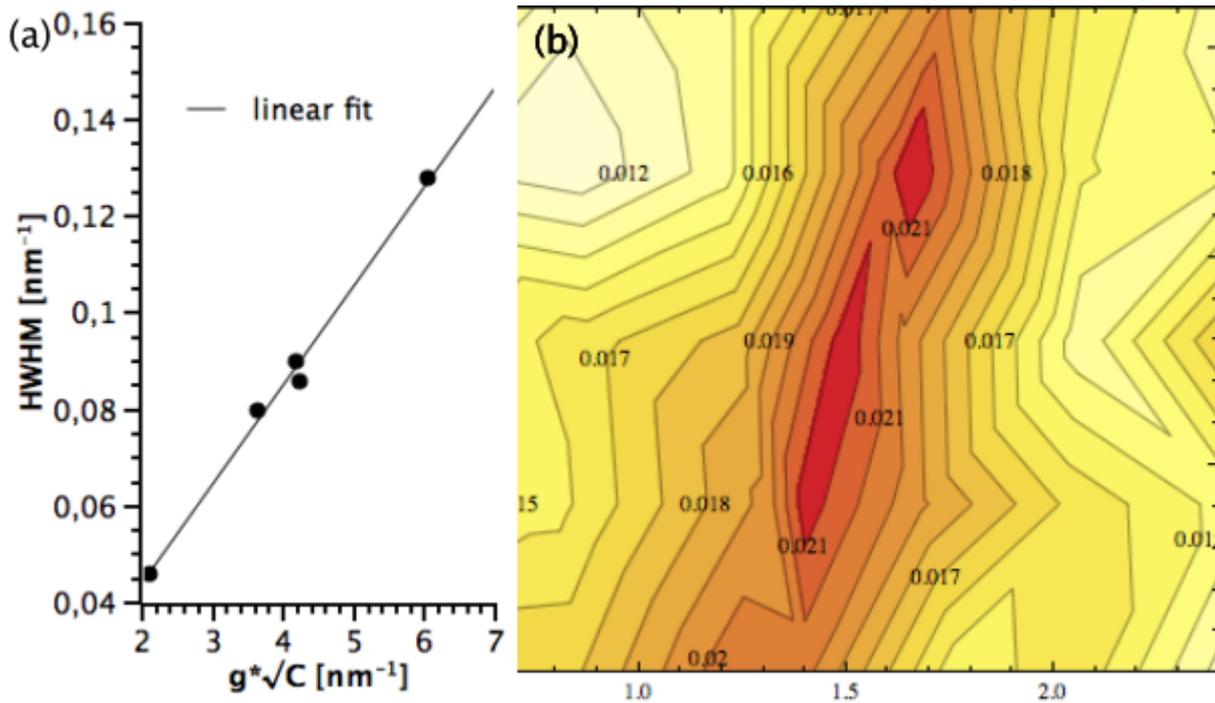


Fig. 3: (a) Modified Williamson Hall plot obtained from the intensity profile shown in Fig. 2. (b) Contour plot drawn from the slope values of the modified Williamson-Hall plots obtained from a 5x7 array of SAED patterns (of the area marked in Fig.1). The values are proportional to the square root of the dislocation density.