

MICROSTRAIN MEASUREMENT IN PLASTICALLY DEFORMED AUSTENITIC STEEL

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Some results of high-resolution neutron diffraction experiments resulting in determination of the microstrain in metallic materials are reported. The method of the diffraction line broadening analysis was verified in two materials, investigated earlier by X-ray diffraction: an austenitic steel prestrained up to 30% and a cold rolled aluminium magnesium alloy. For these materials, the mean-square microstrain and the dislocation density were determined, a good agreement was reached with the results of X-ray experiments. As an application of such analysis we investigated the influence of the shot-peening treatment on an austenitic steel. This treatment is commonly used to introduce compressive stresses in the surface of material, giving improved resistance to fatigue fracture. The microstrain distribution and the size of coherently diffracting blocks for a shot-peened sample of austenitic steel as a function of depth from the surface are determined by the above analysis and the results are presented.

Keywords: Austenitic steel; Peak profile analysis; Microstrain; Neutron diffraction

INTRODUCTION

In the last decade neutron diffraction has been extensively used as a non-destructive technique for the determination of internal elastic strain

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in polycrystalline materials. In fact due to the small attenuation of neutrons in most materials, bulk samples of irregular shapes and large dimensions can be analysed. The principle of the method is based on measurements of small lattice spacing variations resulting in shifts of the Bragg peaks. More recently, the neutron diffraction technique has also been applied for the determination of plastic strains which result from microstructural defects and lead to peak broadening (Macek *et al.*, 1996). Different approaches are described in literature allowing to relate the diffraction profile parameters obtained by refinement with the microstructural parameters (for a review see Balzar, 1993). In the present study Keijser's approach was used to separate the line broadening due to the domain size and the mean-square microstrains. Using these two parameters the dislocation density and the elastic stored energy can be estimated.

Neutron diffraction line broadening analysis requires generally high-resolution neutron diffractometer. In this work, we report a preliminary study on plastically deformed austenitic steel, performed on the conventional two-axis diffractometer G5.2 of the Orphée reactor, where a good instrumental resolution is achieved by a large monochromator take-off angle and a small incident beam divergence.

THEORETICAL

The measured profile, $h(x)$, is the convolution of an instrumental line profile, $g(x)$, with the microstructure broadened profile, $f(x)$. To obtain the original sample broadened profile, which contains the information about the microstructural parameters of the sample, different approaches can be used. The most rigorous one is the Stokes deconvolution method combined with the Warren–Averbach analysis, since no assumption about the analytical form of diffraction-peak shape is required in this case (see Warren, 1959). However, when peaks overlapping and sample broadening are comparable with the instrumental broadening, this method gives unstable solution or cannot be performed at all. To obtain reliable results, proper correction have to account for truncation effects, sampling and standards error bars (Balzar, 1993).

Therefore several methods have been proposed to separate size and strain broadening from single diffraction peak. The single-line

microstrain analysis developed by de Keijser *et al.* (1983) is based on the assumption that the sample broadened profile can be described by the convolution of the Gauss function and the Cauchy function, depending on the strain and the size, respectively:

$$G(x) = \exp(-\pi x^2/\beta_G^2), \quad L(x) = (1 + \pi^2 x^2/\beta_L^2)^{-1}, \quad (1)$$

where β_G and β_L denote the integral breadth of the Gaussian and Cauchy components. In particular most promising results were obtained by using a pseudo-Voigt function (pV), which is a linear combination of a Lorentzian L (size) and a Gaussian G (strain) ones:

$$\text{pV}(x) = \eta G(x) + (1 - \eta)L(x). \quad (2)$$

In this case, according to Keijser, the parameters of pV function β_G , β_L and η are related to the area-weighted crystallite size D and the mean-square strain $\langle \varepsilon^2 \rangle^{1/2}$ by the following formulas:

$$D = \lambda/(2C_1 \cos \theta), \quad (3)$$

with $C_1 = (1 - \eta)\beta_C^2/\beta$, where β the integral breadth of the pV(x), and

$$\langle \varepsilon^2 \rangle^{1/2} = \frac{(C_3 - (2/\pi)C_2)^{1/2}}{2(2\pi)^{1/2} \tan \theta}, \quad (4)$$

with $C_2 = (1 - \eta)\beta_C^3/\beta$, $C_3 = \eta\beta_G^3/\beta$.

The Keijser's method (de Keijser *et al.*, 1983) also implies that the resolution function of diffractometer should be described by the Voigt, pseudo-Voigt or Pearson VII functions, since only under this condition the analytical deconvolution of the resolution function is possible.

In the present work we use the indirect deconvolution method. In this case the measured profile $h_{\text{obs}}(x)$ was assumed to be a pV function numerically convoluted with one-dimensional instrumental resolution profile $g(x)$:

$$h_{\text{cal}}(x) = \sum_k g_k * f_{j-k}. \quad (5)$$

By minimizing the weighted sum of the square differences between the experimental measured profile and its analytical expression (5), six parameters were refined for broadened profile: the scale factor, three

parameters of the pV function (β_G, β_C, η), the peak position and the flat background (no background slope was detected in the experimental profile). From these parameters it was then possible to determine the crystallite size D and the mean-square strain (see Eqs. (3) and (4)). The instrumental resolution profile was determined by a procedure described below and can be well described by the Gaussian function. (We note, however, that since the convolution is performed numerically it can be applied for any other function or even for the resolution given as a set of experimental points.)

Generally, the resulting microstructural parameters D and ε are often used for deducing the dislocation density in metals. Such estimate can be derived from a comparison of the single dislocation energy and stored energy of the lattice (Williamson and Smallmann, 1955),

$$\rho_\varepsilon = \frac{k \langle \varepsilon^2 \rangle}{F b^2}, \quad (6)$$

where b is the Burgers vector, F is the factor describing interaction between dislocation ($F = 1$ in the simple case) and k can be approximated by $k \sim 12A$ ($A \sim 2$ for profiles close to the Cauchy distribution; $A = \pi/2$ for Gauss distribution).

EXPERIMENTAL PROCEDURE

Neutron diffraction measurements were carried out on the two-axis diffractometer G52, situated on the cold neutron guide of the Orphée reactor at the LLB (Saclay). The monochromatic neutron beam of the wavelength of $\lambda = 2.84 \text{ \AA}$ is produced by reflection from pyrolytic graphite (PG 004). The diffracted beam is detected by a position sensitive detector (PSD) with a spatial resolution of about 1.5 mm. The instrumental profile was determined from the (111) reflection of silicon powder ($\Delta d/d = 1.9 \times 10^{-3}$). The typical counting time was about 40 min per diffraction spectrum.

RESULTS AND DISCUSSIONS

The first experiment was carried out on the samples of austenitic steel (AISI 316, the chemical composition 0.03% C, 2% Mn, 1% Si, 12% Ni,

17% Cr, 2% Mo), which were annealed at 1150°C and deformed by tension at room temperature up to 30% elongation. The neutron diffraction profiles of the (111) reflection were measured from the small gauge volume ($2 \times 2 \times 10 \text{ mm}^3$) in the centre of the samples. Examples of the (111) profile of the deformed steel and of the etalon profile are shown in Fig. 1. First, the integral breadth was plotted as a function of the tensile deformation degree (Fig. 2). The integral breadth, which is a good qualitative measure of plastic strain (independent of a further data treatment method), increases with prestrain degree and for annealed sample it is approximately equal to the resolution width. The above described analysis was then performed and the results of separation of strain and size contribution from experimental profiles are summarized in Fig. 3, where the size of mosaic blocks and the microstrain parameter obtained by neutron experiment are shown.

As seen from Fig. 4, the fraction of the Cauchy component increases with increasing tensile strain, which shows that the dislocation density in the crystallites increases as well. These results are in agreement with those obtained earlier by TEM (Fig. 5). The dislocation density of the annealed sample is very low, then it increases considerably for 5% prestrained sample and then stabilizes at deformation about 15%.

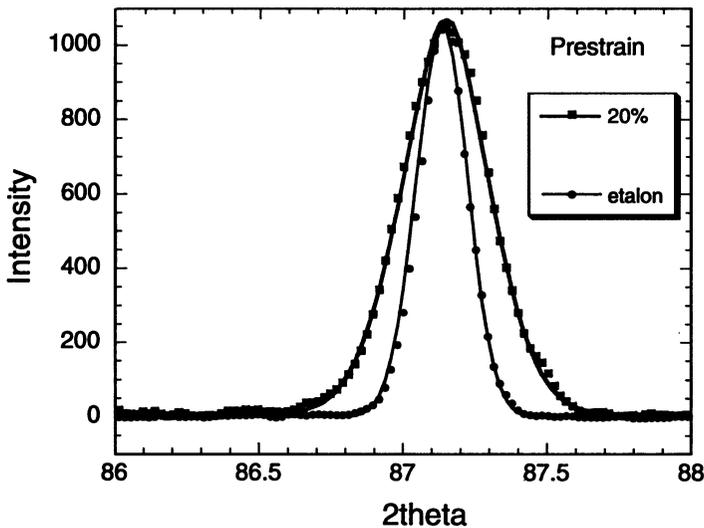


FIGURE 1 Examples of measured profiles.

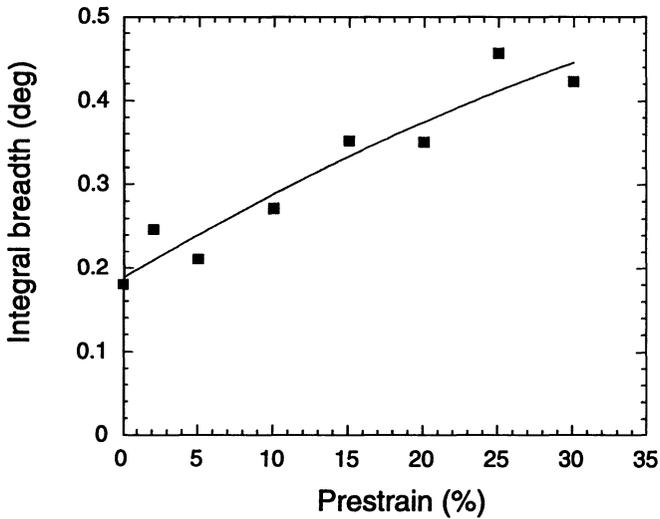


FIGURE 2 Integral breadth vs prestrain degree of the AISI 316 sample.

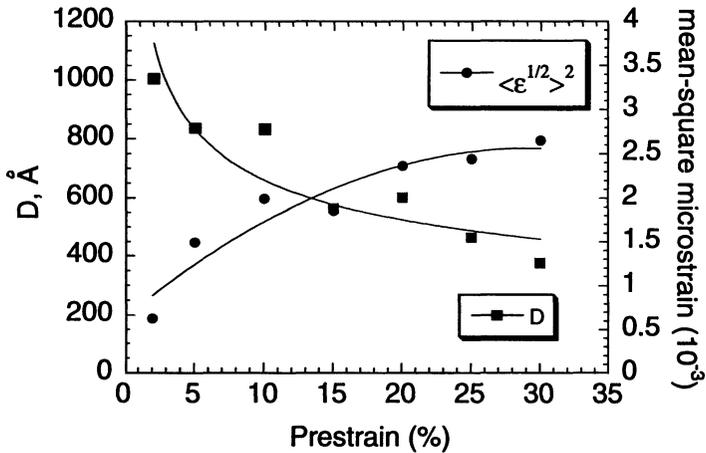


FIGURE 3 Size of mosaic blocks (D) and mean-square strain ($\langle \epsilon^{1/2} \rangle^2$) vs prestrain degree for the AISI 316 sample.

This level corresponds to the formation of the cellular structure (Braham, 1984).

The second experiment was carried out on a cold rolled aluminium magnesium alloys for the (111) and (200) reflections. Typical samples

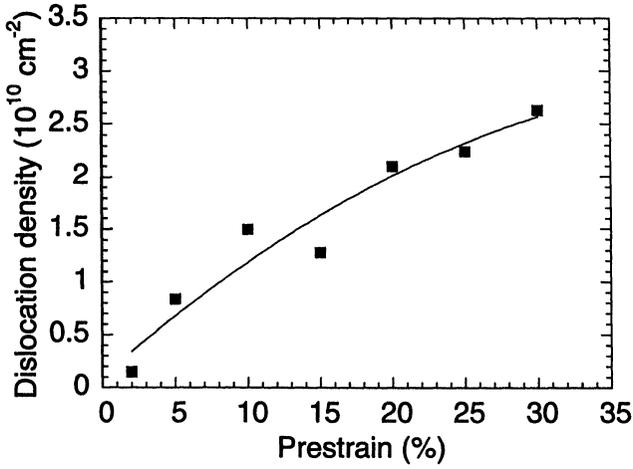


FIGURE 4 Dislocation density in the AISI 316 sample as a function of the prestrain degree.

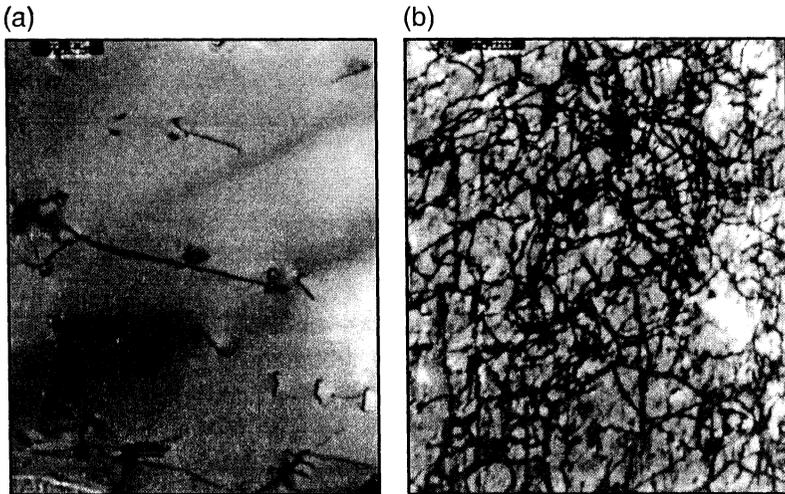


FIGURE 5 The observations by TEM of the AISI 316 annealed sample (a) and of the 5% prestrained sample (b).

were in the form of thin sheets of $10 \times 10 \text{ mm}^2$. The equivalent plastic strain ratio varied from 0.1 to 5.0 (Figs. 6–8).

The peak breadth increases for ε located between 0.1 and 1.0. In the second stage of deformation (for $\varepsilon > 1.0$) the peak breadth is stabilized

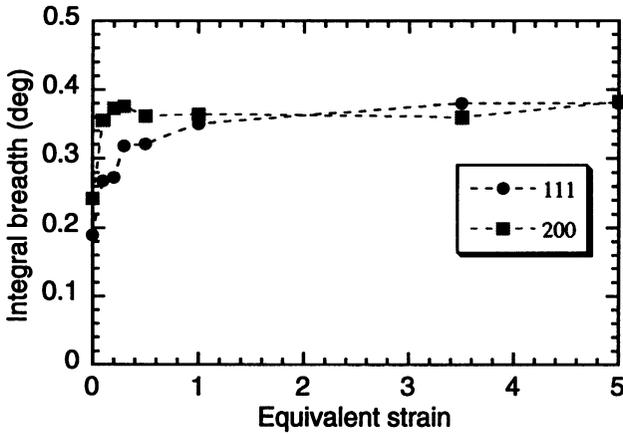


FIGURE 6 Integral breadth of the Al-Mn sample (111) and (200) reflections as a function of the equivalent strain.

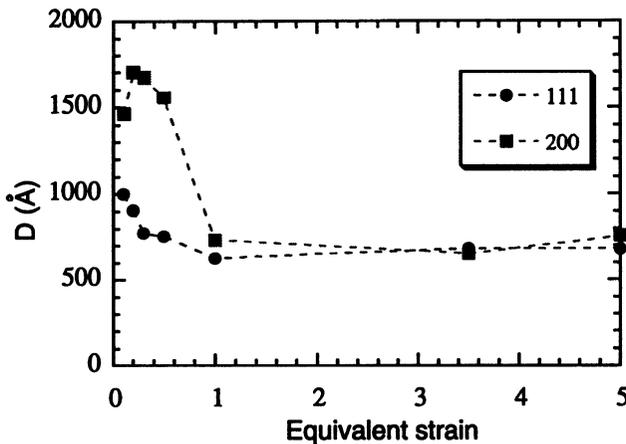


FIGURE 7 Size of mosaic blocks (D) vs equivalent strain for the Al-Mn sample.

(Fig. 6). This result is in a good agreement with the results from X-ray experiments carried out by Ji (1989), who used the Warren–Averbach method for (111) and (222) reflections. The first stage of deformation corresponds to progressive and homogeneous dislocation multiplications, which lead to the increase of dislocation density and to continuous formation of dislocation cells. This process gives the broadening of

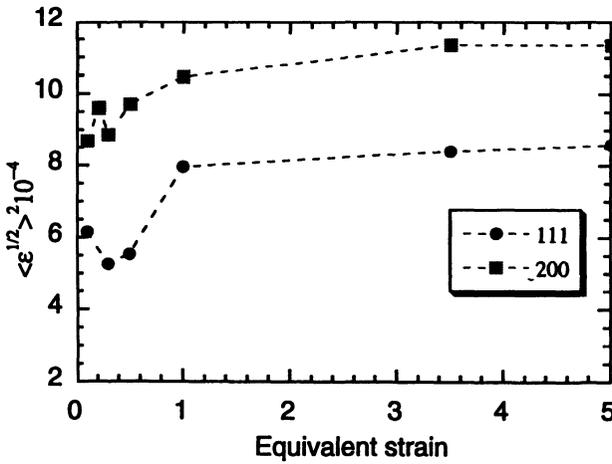


FIGURE 8 Mean-square microstrain ($\langle \epsilon^{1/2} \rangle^2$) vs equivalent strain for the Al-Mn sample.

TABLE I Dislocation density (10^{10}cm^{-2}), estimated from X-ray and neutron experiments. L is the distance normal to the planes of interplanar spacing d

Strain	X-ray		Neutron
	$L = 50A$	$L = 100A$	
0.1	2.922	2.06	1.41
0.3	2.98	2.107	1.03
0.5	3.027	2.14	1.14
1	3.046	2.154	2.34
3.5	3.085	2.182	2.62
5	2.953	2.088	2.73

profiles. The cells then become completely structured and in the second stage of deformation, which is characterized by the formation of cellular microstructure, the dislocation density tends to be stabilized. In Table I the comparison of the value of dislocation density estimated from X-ray and neutron experiments is presented. The values obtained with both techniques are in reasonable agreement.

As an application of such an analysis we investigated the influence of the shot-peening treatment on the austenitic steel. This treatment is commonly used to introduce compressive stresses in the surface of

material, giving improved resistance to fatigue fracture. The measurement was performed on a shot-peened samples of austenitic steel (AISI 304, the chemical composition is: 0.03% C, 2% Mn, 1% Si, 10% Ni, 18% Cr). To obtain the through thickness microstrain profile the sample was moved through the beam in steps of 0.1 mm close to the surface and in greater steps elsewhere. Measurements were made in transmission direction. Cadmium masks were inserted in both the incoming and the diffracted beam to define the gauge volume of $0.3 \times 0.3 \times 10 \text{ mm}^3$. Figure 9 shows the peak breadth variation with the depth. The plastic deformation takes place only at the distance less than 0.7 mm from the surface of the sample. We note that the broadening due to the large strain gradient (elastic macrostrain) was neglected because of the small gauge volume used.

The microstrain distribution and the size of coherently diffracting blocks as a function of depth from the surface are determined by the above analysis (see Fig. 10) and the dislocation density was estimated using formula (6).

As seen from Fig. 11, the dislocation density is very high close to the surface and decreases rapidly inside the sample, which gives clear evidence of strong microstrains induced at the surface by the shot-peening process.

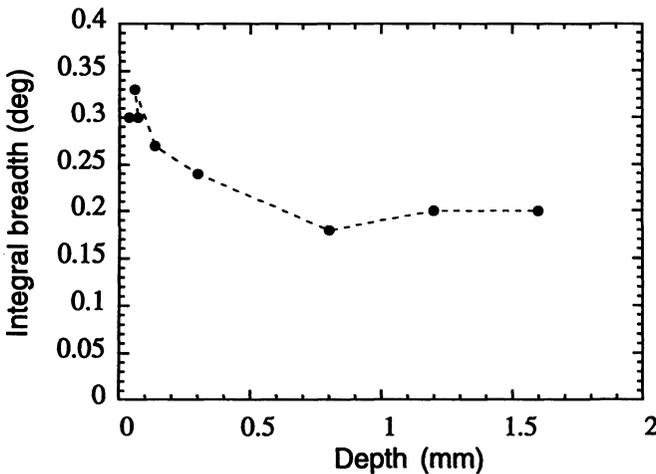


FIGURE 9 Integral breadth variation with the depth from surface for the shot-peened steel sample.

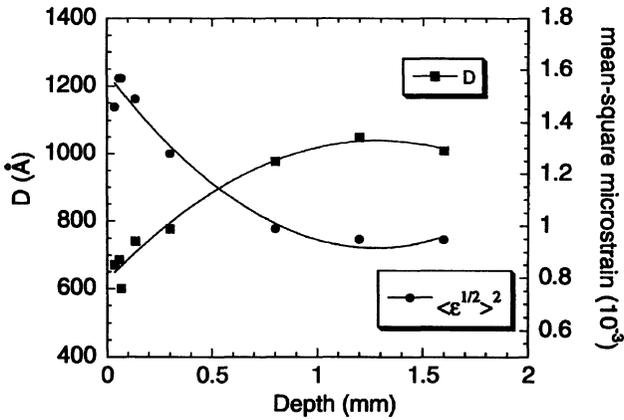


FIGURE 10 Domain size (D) and mean-square microstrain ($\langle \epsilon^{1/2} \rangle^2$) variation with the depth from surface of the shot-peened steel sample.

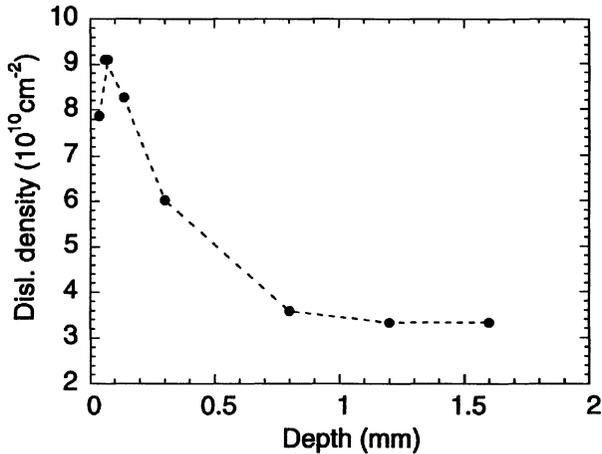


FIGURE 11 Dislocation density variation with the depth from surface of the shot-peened steel sample.

CONCLUSIONS

Method of the single peak analysis with indirect deconvolution was verified using the austenitic steel specimens. Results are in agreement with the X-ray and TEM experiments which show that the dislocation

density increases considerably for deformations less than 15% and then stabilizes at the achieved level.

This method was also applied to shot-peened austenitic steel and the microstrain distribution and the size of coherently diffracting blocks as a function of depth from the surface were determined. The dislocation density was found to be very high close to the surface and decreased considerably inside the sample.

In conclusion the method of the single-peak analysis provides useful information which allows to evaluate non-destructively the effect of plastic deformation on the microstructure of materials.

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