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Materials Letters 59 (2005) 3406 – 3409

materials letters

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# Microstructure–microhardness relation of nanostructured Ni produced by high-pressure torsion

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Received 4 January 2005; accepted 26 May 2005
Available online 1 July 2005

**Abstract**

X-ray diffraction line broadening, TEM and microhardness investigations were carried out on Ni samples produced by the high-pressure torsion (HPT) technique. Distribution of grain size, microstrain (dislocation density) along the radial direction of the as-pressed Ni disk were quantified. Gradient in microhardness from the center to the edge of the deformed Ni disk is rationalized in terms of the grain size, dislocation density and grain boundaries at the corresponding region.


*Keywords:* XRD; TEM; Ni; Microstructure; Mechanical property; High-pressure torsion

## 1. Introduction

Ultrafine-grained materials prepared by severe plastic deformation (SPD) have attracted increasing interest due to interesting modulation of properties [1,2]. High-pressure torsion (HPT) has been demonstrated to be one of the powerful SPD methods to refine the grain sizes of alloys and pure metals to produce pore free nanostructured materials with bulk shape. The HPT processing technique has the advantage of allowing a continuous accumulation of strain without interruption, comparing with many other SPD methods.

Recently, a series of papers [3–7] described the experimental parameters influencing the microstructures during the HPT training. It was claimed that, after a sufficiently high torsional strain under a sufficiently applied pressure, a homogeneous microstructure of equiaxed grains was developed in alloys and pure metals [3]. However, a notable phenomenon for the HPT-processed materials is that the microhardness at the central region is lower than that at the periphery region, and this difference could not be completely eliminated by increasing the HPT straining [5,7]. Therefore, further studies are required to clarify the microstructure–property relation for the HPT-processed materials. In the present study, investigations were carried out on Ni samples treated by the HPT technique to reveal the microstructure–property relation, since Ni has been demonstrated to be an ideal model material for use in experiments of SPD [8].

## 2. Experimental procedures

High purity nickel (99.98 at.%) sheets 0.3 mm thick and 10.0 mm in diameter were deformed by high-pressure torsion for 5 rotations, corresponding to a true logarithmic strain of about 6. A load of 7 GPa was applied at room temperature. The samples were annealed at 650 °C for 15 min to obtain microstructures with an initial grain size of ~30 μm before the HPT straining.

The XRD measurements were performed with a Panalytical MRD diffractometer equipped with a Co tube, and a diffracted beam graphite analyzer set to select the Co $K\alpha_{1,2}$

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doi:10.1016/j.matlet.2005.05.077

radiation. By inserting a mask with a width of $W$ mm and setting a constant length of observation of $L$ mm, the illuminated area on the sample will be $W*L$ mm$^2$ by the incident X-ray. In case of switching on the spindle of the sample holder, illumination of an area within a circle with the diameter d equal to the diagonal of the rectangle could be realized. In the present study, a mask size of 5 mm was chosen; three areas with diameters $d$ of ~5.4, 8.2 and 10.0 mm were investigated by changing the observation length $L$. Values of grain sizes and microstrain were derived from the integral breadths of the diffraction peaks using the Williamson–Hall (WH) method [9]. The instrumental broadening was determined from the measurement of a Si standard.

Samples for transmission electron microscopy (TEM) observations were prepared by twin-jet electropolishing using a solution of $CH_3OH:HNO_3=2:1$ by volume at −30 °C. The TEM observations were carried out on a Philips CM 20 transmission electron microscope operating at 200 kV.

The Vickers microhardness Hv was measured using a Leitz Wetzlar microhardness tester equipped with a diamond pyramidal indentor. A load of 100 g was applied for 15 s. Each reported microhardness value is the average of 10 individual measurements taken with an incremental step of about 100 μm.

## 3. Results and discussion

The XRD line broadening is a function as the convolution of microstructural (such as grain size, microstrain and strain gradients etc.) and instrumental broadening. The microstructural broadening can be worked out by removing the instrumental broadening using a deconvolution procedure [10]. The microstructural broadening will be analyzed by the WH method below, to deduce the grain sizes $D$ and microstrain $\varepsilon$. Fig. 1a shows the WH plot for the measurement with a diameter of 8.2 mm, $\beta^*$ versus $d^*$, according to

$$\beta^* = 1/D + 2\varepsilon d^*, \quad (1)$$

where $\beta^*=\beta\cos\theta/\lambda$ denotes the integral breadth in reciprocal space, $\beta$ the physical integral breadth of the measured peak, $\theta$ the diffraction angle, $\lambda$ the wavelength, and $d^*=2\sin\theta/\lambda$ the reciprocal lattice spacing [9,10]. It can be seen from Fig. 1a that the line broadening is strongly anisotropic with respect to the crystallographic directions <hkl> for the HPT Ni, which can usually be contributed to anisotropic elasticity [11,12] for deformed metals. To check this idea, an alternative treatment of the measured data by substituting $d^*$ with $d^*/E_{hkl}$, where $E_{hkl}$ is the elastic modulus at the crystallographic direction <hkl> [13], see Fig. 1b. The treated data distribute nicely close to a fitting straight line in Fig. 2b, showing that the microstresses are approximately equal along different <hkl> directions, and thus the anisotropy in the line broadening can be largely related to the anisotropic elasticity of Ni. It must be pointed out that other microstructural factors also have effect on the anisotropic peak broadening [14], which can explain the phenomenon that the experimental data do not lay perfectly

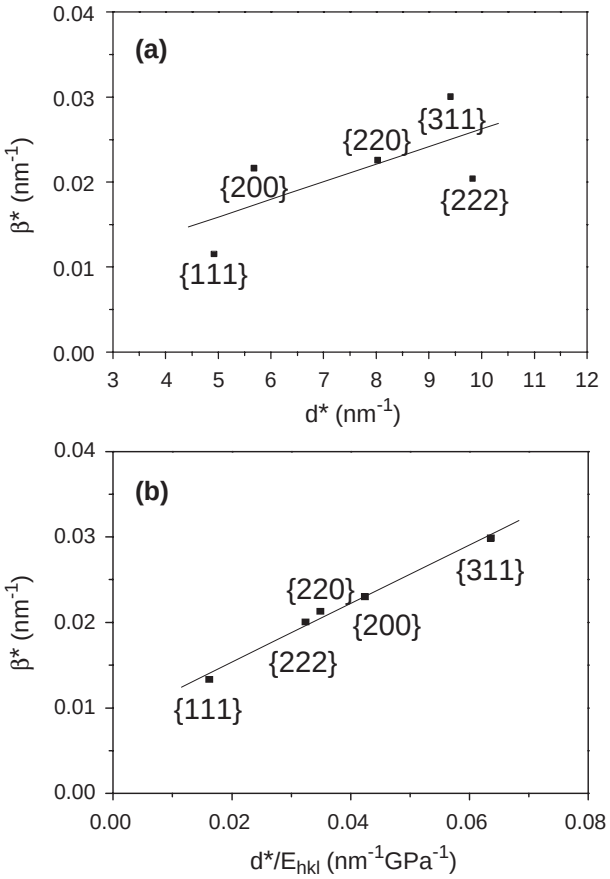


Fig. 1. WH analysis for the 8.2 mm XRD measurement, (a) standard WH plot showing obvious anisotropy in line broadening, (b) experimental data lying almost linearly in the alternative WH plot.

on a straight line even after the alternative treatment. The resulting grain sizes and microstrain for the three measurements are summarized in Table 1. The results demonstrate clearly that the outer region has the smallest mean grain size. The measured microstrain increases first and then decreases with increasing the illuminated area, which is not consistent with the fact that the applied straining increases from the center to the edge of the Ni disk for the HPT technique.

Fig. 2 shows TEM bright field images for (a) the central region and (b) the outer region, with insets showing the corresponding selected-area electron diffraction (SAED) patterns. The GBs are mostly vague and curved in both images, making estimation of the grain sizes difficult. Alternate distribution of grains showing obviously different diffraction contrast can be observed in both images, indicating the formation of high angle GBs throughout the sample. The microstructures of the periphery region are more homogeneous than those of the central region, in the sense of the grain misorientation distribution. In addition, obvious concentration of diffraction spots forming a short piece of arc can be observed on the diffraction rings in the SAED patterns, which reveals that there is still higher fraction of low angle GBs in the sample relative to a real homogeneous microstructure. For example, electron diffraction analyses demonstrated that the lower part shown in Fig. 2(a) was dominated by low angle GBs. Although no significant difference in grain sizes could be observed based on the TEM images, a careful comparison between two sets