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Strain mapping in a deformation-twinned nanocrystalline Pd grain

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Abstract

Strain in a deformation-twinned nanocrystalline Pd sample of about 24 nm grain size was mapped by means of geometric phase analysis based on an individual high-resolution transmission electron microscopy image. The in-plane components of the strain tensor were calculated and charted. Strains with magnitudes of about 0.8% were found in the grain interior. Twins and matrix were significantly distorted relative to each other (by about 3° on average) and showed a strong rotation gradient from top to bottom, revealing that the whole grain is bent. An estimate of the strain energy stored in the Pd grain yielded a value of $E_{\text{strain}} = 4.157\ \text{J g}^{-1}$. Based on the strain distribution observed, a temporal deformation scenario has been developed. In our judgement, deformation twins had formed first and subsequently dislocations were activated, most likely by the misfit strain/stress concentrations generated by the twins themselves. The interaction of the dislocations with the twin boundaries left behind Shockley partials and this accounted for the strain concentrations finally observed along the twin boundaries. It is concluded that in the temporal evolution of deformation in a nanocrystalline material, twinning must be by far the fastest deformation mode, which accounts for the abundance of deformation twins, especially at high strain rates.

Keywords: Strain mapping; Nanocrystalline plasticity; Twinning; High-resolution electron microscopy

1. Introduction

Characterization of the microstructure of nanocrystalline materials usually provides information on grain size, orientation and defects (dislocations, twins, stacking faults, etc.) and involves the use of various diffraction techniques, e.g., X-ray line broadening (in X-ray diffraction), or transmission electron microscopy (TEM). Recent investigations have shown that the micro-strain at grain boundaries/interfaces or in the interior of the grain is significant and the former increases with decreasing grain sizes [1–4]. Imaging methods were first applied to visualize/quantify micro-strain at twin boundaries in ultra-fine-grained Cu [4] using the geometric phase analysis (GPA) [5]. In the present study, GPA was applied to a deformation-twinned nanocrystalline Pd grain. Strain maps displaying the in-plane components of the symmetric strain tensor were generated showing that considerable strain is stored in the grain interior. Based on the strain analysis, a temporal evolution of deformation in nanocrystalline materials is suggested.

2. Experimental

In this study, the strain stored in a deformation-twinned nanocrystalline Pd sample of about 24 nm grain size was measured using the GPA. The nanocrystalline material was synthesized by inert-gas condensation, deformed by cold rolling and subsequently characterized by high-resolution-TEM. The inert-gas condensation took place in a

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vacuum vessel (starting pressure $\sim 10^{-7}$ mbar) where a He atmosphere (6 mbar) was established. Pieces of Pd (99.95%) were thermally evaporated at ~1700 °C out of a molybdenum boat with an Al_2O_3-ceramic inlay. Nanoparticles formed by condensation and agglomeration in the He atmosphere were then collected by a liquid-nitrogen-cooled cold finger, which led to the growth of a loose layer of black agglomerated powder on it. The powder was filled into a sleeve, which is a movable part of a compaction press, by scraping it off the cold finger. The compaction took place between two tungsten carbide anvils at a compaction pressure of ~2 GPa. The resulting sample was disc-shaped with a diameter of 8 mm and a thickness of 235 μm. The average grain size in the as-prepared state was 14 nm (determined by XRD peak broadening) and the initial density was 91.7% (determined by the Archimedes method) of the theoretical density (12.02 g cm^{-3}). Deformation took place in a one-pass thickness reduction of 32% (true strain) using a motorized laboratory rolling mill. The average strain rate during the deformation was 0.3 s^{-1}. After rolling the average grain size had slightly increased to 16 nm while the density had increased to 96.4%. Thin electron-transparent foils were prepared by low-angle (6°) ion-milling (beam energy 3.5 keV) using a PIPS (Gatan). TEM investigations were performed in an FEI Tecnai F20 G^2 (field-emission gun, super-twin lens, $C_s = 1.2$ mm) operated at 200 kV. The high-resolution micrographs were taken using conditions in which delocalization effects due to the spherical aberration of the objective lens [6] were minimized (near Scherzer defocus). Particular care was taken to align the specimen such that the deformed nanocrystalline Pd grain was oriented exactly along the [0 1 1] zone axis. Fourier filtering was applied in two cases (Bragg filtering in Fig. 1b and Wiener filtering in Fig. 1c) to improve the image quality [7]. Further experimental details about the synthesis, rolling conditions and transmission electron microscopy are described in Ref. [8]. TEM investigations showed a few large (>1 μm) grains, in which a high dislocation density was found. It is conceivable that the deformation had induced or accelerated grain growth. However, in contrast to the large grains, the small grains displayed a considerable number of deformation twins, some of them even showing multiple twins. The local strain variations in such a grain constitute the focus of this study.

The method of GPA, developed by Hÿtch et al. [5], provides a powerful tool for measuring strains quantitatively as revealed in high-resolution-TEM (HR-TEM) micrographs. It has been shown that strain measurements obtained by this method agree well with theoretical calculations based on linear anisotropic elastic theory to an accuracy of about 0.2% at a spatial resolution of 2–3 nm [9,10]. However, the measurements are restricted to the in-plane components and are exterior to dislocation cores. The robustness of GPA itself has been demonstrated in several case studies by comparison with other strain mapping algorithms, e.g., pair-peak analysis [11–16], and theoretical considerations [17].

Within the GPA method, the displacements in the Fourier-filtered local image pattern relative to an internal reference area is analyzed with respect to a shift in the atomic positions. The in-plane strain tensor components, ε_{ij}, and the in-plane rigid-body rotation, ω_{ij}, can thus be obtained from the local structural displacements u_{ij} by numerical/partial differentiation [5,9]:

$$\varepsilon_{ij} = \frac{1}{2}\left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i}\right); \quad \omega_{ij} = \frac{1}{2}\left(\frac{\partial u_j}{\partial x_i} - \frac{\partial u_i}{\partial x_j}\right) \tag{1}$$

A commercial version, GPA Phase 1.0 (HREM Research) [18], which is based on the formalism given in Ref. [9] and implemented in Digital Micrograph (Version 1.71.38, Gatan) as a plug-in, was used to calculate the in-plane components of the symmetric strain tensor, ε_{ij}, and the rigid-body rotation, ω_{ij}, [9,10] using a high-resolution micrograph of a deformation-twinned Pd grain [8,19,20]. The coordinates were chosen such that the x-axis was aligned parallel to the twin boundaries. Strain maps were plotted with respect to an internal reference lattice by $\mathbf{g}_1 = [-1, 1, -1]^{\text{matrix/twin}}$ and $\mathbf{g}_2 = [2, 0, 0]^{\text{matrix}}$ for the matrix and separately for the twinned area by $\mathbf{g}_1 = [-1, 1, -1]^{\text{matrix/twin}}$ and $\mathbf{g}_3 = [-1, -1, 1]^{\text{twin}}$ using Lorentzian masks with a diameter of 0.4 nm^{-1} (in reciprocal space). The reference lattice regions of the matrix and the twin used for all strain calculations are indicated by the white frames in Fig. 2a. The individual maps obtained for matrix and twins were then stitched together to display the complete strain distribution, i.e., matrix and twin, in a single image [10]. The strain profile analyses were performed using Digital Micrograph (Version 1.71.38, Gatan). Moreover, the commercial GPA version allows for the correction of image distortions introduced by the projector lens [21] and/or the imaging filter (Gatan GIF 200). For this correction an unstrained Si lattice was used so that the experimental results presented in the next section should not be affected by any systemic/methodical flaws.

It should be mentioned here that a preliminary strain analysis of the nanocrystalline Pd grain (Fig. 1a) has already been published [20]. However, the investigation in Ref. [20] was incomplete because only the matrix was analyzed and the image distortions were neglected when using the NCEM Phase Extensions Routines (created by Kilaas and Hÿtch).

3. Analyses and results

Fig. 1a shows a lattice image of a deformation-twinned nanocrystalline Pd grain in [0 1 1] projection, which is clearly bordered by the adjacent grains (rotation angle $\theta \approx 14°$) at the top and bottom. The lateral grain boundaries are undefined since these grains are not oriented in the Bragg condition. The following strain analysis is confined to the central Pd grain only. Thin twin lamellae, indicated in the figure, had formed in the grain interior as a result of the cold rolling. A detailed analysis using Fourier