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Deformation-induced grain coalescence in an electrodeposited pure iron sheet studied by in situ neutron diffraction and electron backscatter diffraction

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Abstract

The plastic deformation behavior of an ultrafine-grained electrodeposited pure iron sheet with a strong $\{111\}\langle hkl\rangle$ texture was studied by in situ neutron diffraction during tensile deformation at room temperature and by electron backscatter diffraction (EBSD). The combination of volume-averaged crystallographic orientation changes determined by neutron diffraction and the local orientation relationship determined by EBSD reveals a texture change to $\{111\}\langle 110\rangle$ and corresponding microstructural changes with tension deformation. Related to such grain rotation, grain coalescence on deformation was found using semi in situ EBSD. The results obtained are explained using a characteristic slip model, which also gives a reason for the ultrahigh Lankford value of this material.

Keywords: Electrodeposited pure iron; Tensile deformation; Grain coalescence; In situ neutron diffraction; Electron backscatter diffraction

1. Introduction

Grain growth (or coalescence) during deformation at both cryogenic and room temperatures has been reported to occur for several ultrafine-grained (UFG) and nanocrystalline (nc) materials. This issue has been extensively investigated in nc face-centered cubic (fcc) Al [1–4], Cu [5–9], Ni [10–12], and Pt [13] metals and Ni–Fe [14–16], Co–P [14,17], and Al alloys [18] through various experimental and computational studies (e.g. transmission electron microscopy (TEM) observations, X-ray diffraction (XRD) and molecular dynamics (MDs) simulations [19–21]). It is suggested that grain boundaries play an important role in the deformation of nanostructured materials. A number of mechanisms to explain this unusual phenomenon have been identified, either one or a combination of grain boundary sliding, curvature-driven grain boundary migration, and deformation-induced grain rotation. Twinning and dislocation slip may act as accommodating mechanisms. Wang et al. observed the complete processes of individual grain rotation and neighboring grain rotation/growth during in situ TEM tensile deformation in nc Ni. Deformation-induced grain rotation and growth in nc materials was revealed as one plastic deformation mechanism [22]. It has been demonstrated that the coupling of dislocation-mediated plasticity and the formation and growth of voids play a dominant role in the deformation of nanocrystalline Ni [23]. The Morris group have reported strain-induced grain coalescence by in situ nanoindentation of UFG Al in a transmission electron microscope at room temperature [24–26]. Stress-induced grain growth/coalescence was also demonstrated by in situ XRD and TEM analyses during the nanoindentation of UFG and nc Al films [27,28], and it was reported that different levels of impurity pinning atmospheres, e.g. light elements, lead to

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stress-driven rapid grain coarsening. The TEM observations of grain growth in nc Cu under an indent at ambient and cryogenic temperatures and in Pt nc films after microtensile experiments provide further evidence of the role of mechanical stress in initiating grain growth in nc metals [29,30]. However, the existence of such characteristic deformation mechanisms has not been sufficiently explained.

Yoshinaga et al. have reported [31,32] that an UFG electrodeposited pure iron sheet with a strong $\{1\,1\,1\}\langle h\,k\,l\rangle$ texture exhibits a Lankford value (R value) greater than 7.0. The R value is generally used to evaluate the formability of sheet steel. In a tension test it is defined as the ratio of width true strain to thickness true strain of a specimen cut from a sheet.

$$R = \ln(w_0/w_f)/\ln(t_0/t_f) \quad (1)$$

Here w and t denote width and thickness and the subscripts 0 and f denote the initial and final values, respectively. Most commercially available sheet steels exhibit R values less than 2.0. R values as high as 3.0 have been achieved in steel used for deep drawing, during which the crystalline texture is carefully controlled. When employing a conventional slip deformation model such an ultrahigh R value cannot be predicted (or explained) simply by the crystallographic texture, therefore, other hidden (or latent) factors, such as grain boundary sliding and the characteristic manner of slip associated with fine columnar grains, must be investigated [33,34]. The present authors have recently confirmed the existence of hydrogen in an as-deposited specimen by thermal desorption spectroscopy and small angle neutron scattering measurements [35]. Tiny hydrogen bubbles were suspected to intrude along grain boundaries and disappear on cold rolling at room temperature. Moreover, electron backscatter diffraction (EBSD) observations suggested that grain coalescence occurred during cold rolling [35].

Deformation mechanisms in UFG and nc materials have primarily been studied by TEM observation, where only a few grains in a volume-limited specimen can be identified. It is necessary to find new reliable and systematic microstructural characterization methods for volume-averaged information during deformation. Neutron diffraction is an effective probe to evaluate stress and is widely applied to fundamental studies of materials. Because the crystallographic response can be correlated with the bulk properties of the material, neutron diffraction is suitable to investigate lattice (elastic) strain in differently oriented grains and texture evolution during plastic deformation [36–40]. On the other hand, EBSD analysis provides local crystallographic information such as misorientation angles. However, the information is limited to free surface deformation. An electrodeposited specimen with a grain size of several hundred nanometers is an appropriate sample to determine deformation behavior by both in situ neutron diffraction and EBSD [41,42], which will provide comprehensive information, i.e. bulk average and microscopic information. Hence, the plastic deformation behavior of a UFG electrodeposited pure iron sheet was studied by in situ neutron diffraction and EBSD observations during tension testing at room temperature. The purpose of this paper was to investigate how neutron diffraction and EBSD results come together to illustrate the reasons for grain coalescence and the exceptionally high R value.

2. Experimental procedures

The electrodeposited pure iron sheets (approximately 1.5 mm thick) used in this study were manufactured by Toho-Zinc Co. Ltd. The iron purity estimated from electroresistivity measurements was greater than 99.995 wt.%. The definition of specimen directions is presented schematically in Fig. 1. Tensile test specimens were cut from the sheets. The dimensions of the parallel gage portion were $30 \times 5 \times 1.5$ mm for the conventional tension test and $50 \times 5 \times 1$ mm for the neutron diffraction experiment. The tensile properties of the as-deposited specimen were examined.

In situ neutron diffraction measurements during tensile loading were conducted using the TAKUMI time-of-flight (TOF) diffractometer for engineering material research at MLF/J-PARC [43,44]. The schematic arrangement of the specimen with respect to the incident and diffracted neutron beams is presented in Fig. 2. As shown in Fig. 2, the angle between the incident neutron beam and the tensile axis is 45°, therefore, diffraction patterns axial and transverse to the loading direction can be measured simultaneously by the south and north detector banks, respectively, with a scattering angle of ±90°. The tensile specimen was set in such a way that either the tensile (or axial) direction (AD) and the transverse direction (TD) data (case 1) or AD and the deposition (or normal) direction (ND) data (case 2) could be obtained. Schematic illustrations of these two settings are shown in Fig. 2b and c, respectively. The tensile load was increased stepwise with a holding time of 300 s to acquiring a diffraction profile, which provides sufficient statistical data for analysis.

The intergranular strain of the $\langle h\,k\,l\rangle$ grain family can be evaluated from the shift in the diffraction profile. The neutron diffraction profiles were then analyzed by single peak

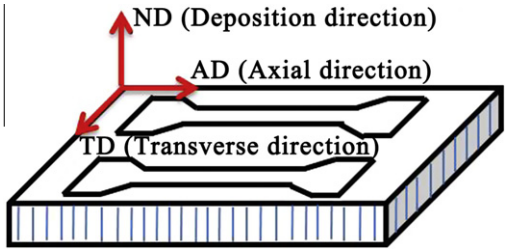

Fig. 1. Schematic diagram of the definitions of specimen directions: the tensile direction (AD), the transverse direction (TD), and the deposition direction, which is defined as the normal direction (ND).