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Influence of hydrogen on dislocation self-organization in Ni

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ABSTRACT

The influence of hydrogen on the evolution of the deformation microstructure in Ni at the same specific macroscopic strain has been determined for the first time. The evolution of the microstructure was accelerated by hydrogen as evidenced by the formation of smaller dislocation cells and dense dislocation walls compared to only dislocation cells in the absence of hydrogen. This enhanced evolution is described in terms of the hydrogen-enhanced localized plasticity mechanism.

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1. Introduction

The rate of generation of dislocations is predicted by the hydrogen-enhanced localized plasticity (HELP) [1,2] as well as the thermodynamic defactant mechanism [3–5] to be increased by the presence of hydrogen. These mechanisms are based on different concepts. The HELP mechanism relies on hydrogen segregated to the stress fields of dislocations and other obstacles influencing the stress-field interactions. The resultant hydrogen shielding is directionally dependent with it increasing in some directions and decreasing in others [6]. The defactant concept relies on the reduction of the formation energy of defects to promote increased generation rates [3–5]. With respect to dislocation generation rate, it has been claimed that it is attributable to hydrogen decreasing the line energy [5] and to hydrogen decreasing the dislocation formation energy [7]. However, the magnitude of these reductions has not been estimated or measured, making it difficult to assess their significance. Direct evidence of hydrogen enhancing the rate at which dislocations are generated has been provided by experiments performed in-situ in a transmission electron microscope (TEM) equipped with an environmental cell [1,8], and indirectly through interpretation of stress relaxation tests [9,10] and nano-indentation tests [11,12].

Both mechanisms also predict that the presence of hydrogen causes an increase in the dislocation velocity. The HELP mechanism attributes this enhancement to the change in the stress-field interaction of dislocations with other obstacles, whereas the defactant mechanism attributes this to a reduction in the kink-pair formation energy [13]. The latter energy is small at room temperature in FCC metals and usually is considered to be negligible even in the absence of hydrogen [14,15]. Therefore, it remains a point of debate if the defactant mechanism is able to explain the enhanced dislocation velocity that has been observed experimentally. Direct experimental evidence for hydrogen enhancing the velocity of dislocations has been provided by the in-situ TEM experiments [1].

Taken together the increase in the dislocation generation rate and the enhancement of the dislocation velocity predict that the

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dislocation density should be increased in metals deformed in the presence of hydrogen [16,17]. Early attempts to quantify any variation in the dislocation density between deformed uncharged and hydrogen-charged materials yielded conflicting results [18–21]. For example, Robertson and Birnbaum reported no discernible differences in the dislocation structures in Ni deformed to failure in the absence and presence of hydrogen [18]. McInteer et al. [4] also showed no discernible differences in dislocation arrangement due to hydrogen at true plastic strains of 2.5%–10% of Ni, although the presence of hydrogen increased waviness of interior slip lines through slip line broadening. In contrast, Wilcox and Smith reported an increase in dislocation density and a change of the microstructural features in Ni in the presence of hydrogen but these were evident only at tensile strains of 6%–9% [20]. Matsui et al. reported the average dislocation density in Fe in the presence of hydrogen (3.0% tensile strain) is 5–10 times higher than in air (3.8% tensile strain), although the difference in the features of dislocation structure was not significant [21]. More recently, the dislocation microstructures in the absence and presence of hydrogen have been examined beneath fracture surfaces generated under tensile loading in Ni [16] and Fe [17] and fatigue loading of 316L stainless steel [22,23] as well as in cold rolled Pd [7,24]. The latter studies demonstrated that the complexity or the degree of evolution of the microstructure increased with increasing hydrogen content. Nanoindentation studies have found that the "pop-in" load decreased in the presence of hydrogen and this was interpreted as a decrease in the load to produce dislocations homogeneously [25,26]. This result was explained by the hydrogen-induced reduction in the formation energy of dislocations, which was attributed to a reduction of the shear modulus, an increase in the radius of the dislocation core, and a decrease in the stacking-fault energy. However, as noted by Kirchheim [5], the magnitude of the decrease in the shear modulus is insufficient to explain the decrease in the "pop-in" load. Furthermore, the experimental result is valid only for the homogeneous nucleation of loops and if no other sources for dislocation generation are present [27].

In this study, the dislocation structure produced in Ni at the same level of shear strain is compared in the absence and the presence of hydrogen. The uncharged and hydrogen-charged samples were subjected to high-pressure torsional processing and the dislocation structures at different strain levels were assessed by using a scanning transmission electron microscope (STEM). It will be demonstrated that the microstructure is more evolved in the presence of hydrogen. In addition, it will be demonstrated for the first time that the evolution pathway and interactions of dislocation are identical in the absence and presence of hydrogen and that the influence of hydrogen is simply to accelerate them.

2. Experimental procedures

The Ni used in this study was purer than 99.76%. The main impurities were C (0.026 mass%) and Fe (0.16 mass%, in average) as determined by combustion method and inductively coupled plasma mass spectrometry, respectively. The as-received Ni was cut into 10 mm-diameter discs with a thickness of 1.0 mm by electric discharge machining, and annealed at 700 °C for 8 h. The grain size in the annealed discs was between 100 and 500 μm. Hydrogen was introduced by thermal charging in an autoclave filled with high-pressure hydrogen gas at 200 °C for a duration of 160 h under a gas pressure of 120 MPa. For these specimen dimensions and hydrogen-charging condition, a uniform hydrogen distribution was assumed to have been established in the discs [28]. The hydrogen-charged specimens were subsequently stored in liquid nitrogen and then equilibrated with ambient condition before subjected to high-pressure torsion (HPT) processing [29,30].

The uncharged and hydrogen-charged samples were subjected to the HPT processing at a pressure of 6 GPa, with a rotation speed of 0.107 rotation per minute to either 1/16 rev (total loading time: 35 s) or 1/32 rev (total loading time: 18 s). The shear strain rate, $\dot{\gamma}$, at a site r (mm) from the center of the axis of rotation, can be estimated from the rotation speed, ω (deg/s), according to:

$$\dot{\gamma} = \frac{2\pi r\omega}{360l} \tag{1}$$

Here l is the thickness of HPT sample, 1 mm. The corresponding shear strain can be estimated from the time integration of Equation (1). These experimental conditions are shown in Table 1 along with the equivalent shear strain rate and equivalent von-Mises strain ($\varepsilon_{VM} = 2\pi rN/\sqrt{3}l$, where N is the total number of revolutions) at 2.0 mm away from the rotation center. To determine the hydrogen concentration of the hydrogen-charged sample, the gas chromatograph thermal desorption analysis method was used. Hydrogen desorption rate was measured from room temperature to 800 °C at a constant rate of 200 °C h^{-1}. The thermal desorption spectrum showed only one peak from room temperature to 492 °C and the hydrogen concentration was determined to be 1479 appm (25.20 mass ppm). Focused-ion beam (FIB) machining was used to extract samples at distances of 0 and 2 mm from the center of the disc; a Zeiss 1540XB CrossBeam workstation was used. These electron transparent samples were 15 μm long along the radial direction and 10 μm deep in the surface normal direction. It should be clear that a variation in the strain gradient of HPT samples exists along the radial direction, however, the variance along the length of the extracted sample is less than 0.17%, and its influence on the dislocation microstructure is assumed to be negligible. Observation of dislocation structures was performed using a FEI Tecnai TF-30 field emission scanning/transmission electron microscope (S/TEM), which was operated at an accelerating voltage of 300 kV.

3. Experimental results

The initial dislocation structure generated by the compression associated with conducting the HPT processing can be determined by examining samples from the center of the discs, as these should have experienced zero shear strain during the torsional loading. Examples of these microstructures obtained at the rotation center in the 1/16 rev samples are compared and contrasted for the uncharged and hydrogen-charged samples in Fig. 1. These STEM micrographs show the microstructure as a function of distance from the free surface. In comparing these micrographs, it is clear that there is no discernible difference in the initial microstructure due to the presence of hydrogen; both materials show the early stages of formation of a dislocation cell structure in which the cell size varies broadly and the dislocation walls contain a low density of loosely

Table 1
HPT processing conditions and the equivalent strain rate and strain at a distance, r, of 2.0 mm away from the center of the disk.

Revolution (rev)	Rotation speed (rpm)	Test duration (s)	Shear strain rate at $r = 2.0$ mm (s^{-1})	Von-Mises strain at $r = 2.0$ mm
1/32	0.107	18	0.022	0.23
1/16	0.107	35	0.022	0.45

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