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Experimental study on the diffusion of hydrogen along individual grain boundaries in nickel



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ABSTRACT

Whether hydrogen diffusion can be accelerated by grain boundaries (GBs) still remains controversial. In this study, the impact of different types of GBs within the directionally solidified polycrystalline nickel specimens on hydrogen diffusion was directly observed at room temperature by combining Electron Back Scattered Diffraction (EBSD) and scanning Kelvin probe force microscopy (SKPFM). The results suggest that the coherent Σ 3 twin GB provides a fast path for hydrogen diffusion, while the random GB cannot significantly accelerate the transport of hydrogen atoms. In addition, this study confirms that SKPFM is a reliable technique to allow the detection of the local diffusion and distribution of hydrogen in metals with high lateral resolution down to the GBs.

1. Introduction

Hydrogen embrittlement (HE) is caused by interactions between hydrogen and a metallic matrix and is one of the most common reasons for the loss of mechanical properties and ultimate failures of those affected metals [1]. To improve the understanding of the HE mechanism, hydrogen diffusion behaviors in materials should be studied. It is generally accepted that grain boundaries (GBs) can act as a tunnel for hydrogen transport in polycrystalline materials [2]. Unfortunately, although extensive studies have been conducted, the effect of GBs on hydrogen diffusion still remains unclear.

Electrochemical permeation [3,4] is a commonly used experimental method for the study of hydrogen diffusion along GBs. With this technique, many researchers [5–7] demonstrated that hydrogen movement could be accelerated because of the short-circuiting effects along GBs. However, using the same method, Yao et al. [8,9] proposed that GBs acted as trap sites rather than fast paths for hydrogen transport. Furthermore, Oudriss et al. [10,11] reported that the nature of the GBs determined their effects on hydrogen transport and that random GBs could provide a rapid tunnel for hydrogen diffusion. These reports are contradictory because the electrochemical permeation method can only provide the average characteristics of all the GBs within a sample. Although difficult in practice, studying a specific type of GB can obviously provide much more information [12].

Scanning Kelvin probe force microscopy (SKPFM) is a novel tool for detecting hydrogen and is capable of both directly and dynamically In this work, hydrogen transport along different individual GBs has been directly observed by SKPFM technique, and influences of different types of GBs on hydrogen diffusion have been determined.

2. Materials and methods

2.1. Material preparation and EBSD analyses

High purity nickel (99.99% Ni) was used in the present study. The Ni sample was directionally solidified in order to obtain grains that were large enough for observation of a single GB. The obtained cylindrical sample was cut perpendicular to the axial direction into discoid specimens. Because grains grew along the axial direction of the cylinder and the GB lengths along the axial direction could reach several centimeters, GBs could easily run through the specimen thickness. Electropolishing was applied to both sides of all foil specimens, leading to a final thickness from 140 to 200 μm .

Electron Back Scattered Diffraction (EBSD) analyses were carried out to assess the GB characters. Fig. 1(a) illustrates a random GB with a

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detecting the diffusion of hydrogen [13]. Senöz et al. [14] first reported SKPFM as a powerful method to measure hydrogen permeation within a metal matrix with extremely high sensitivity and lateral resolution down to a single GB. Using this method, many researchers [15–24] have investigated the diffusion and concentration of hydrogen in various metals and strongly demonstrated the validity and superiority of this method when applied to hydrogen detection.

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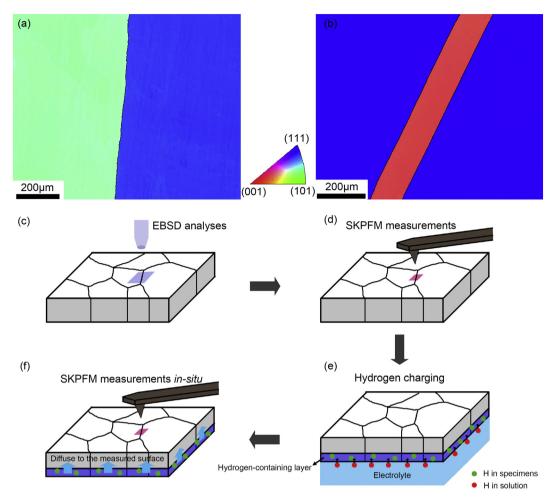


Fig. 1. Results of EBSD analyses: (a) a random GB with a misorientation of 43° ; (b) two coherent $\Sigma 3$ twin GBs. (c)–(f) Schematic illustrations of the present experimental procedure. (c) EBSD analyses for an individual GB. (d) SKPFM measurements at the GB area before hydrogen charging. (e) Hydrogen charging on the reverse side of SKPFM measured surface. (f) Continuous SKPFM measurements *in-situ* after cathodic charging.

misorientation of 43°, and Fig. 1(b) shows two coherent twin GBs, which are a type of typical $\Sigma 3$ GB. These two specimens represent the two most common types of GBs in Ni.

2.2. Electrochemical hydrogen charging and SKPFM measurements

Surface potential was obtained *in-situ* by SKPFM at the same position around a GB before and after hydrogen charging. Hydrogen charging on one side of the specimen was performed at a constant current density of $10\,\mathrm{mA/cm^2}$ in a solution of $0.1\,\mathrm{M}$ NaOH + $0.22\,\mathrm{g/L}$ thiourea at room temperature. The other side of the specimen was measured by SKPFM. The charging time for all specimens was 30 min. According to the hydrogen diffusion coefficient in Ni [25], hydrogen atoms could diffuse in the specimen but could not permeate to the SKPFM measured surface during the 30 min charging period. Any hydrogen that spontaneously diffused to the measured surface with the duration of time after hydrogen charging could be continuously detected by SKPFM. The measured change in surface potential could give some information about the effects of GBs on hydrogen diffusion. Fig. 1(c)–(f) schematically show the procedure of present experiments.

All SKPFM measurements were performed in air at room temperature. A tapping/lift mode was used. Probes were double-sided Pt-coated silicon tips with a resonant frequency of 43–81 kHz. The contact potential difference (CPD) (i.e., the surface potential) is defined as $V_{\rm CPD} = (\phi_{\rm tip} - \phi_{\rm specimen}) / e$, where $\phi_{\rm tip}$ and $\phi_{\rm specimen}$ are the work functions of the tip and specimen, respectively, and e is electronic charge. Therefore, a lower work function from the hydrogen

concentration on specimen surface corresponds to a higher potential value in all surface potential images, as mentioned in [19].

3. Results and discussion

Fig. 2(a)–(1) show the measured evolution of the surface potential images before and after hydrogen charging around a GB ((a)–(f): the random GB, (g)–(l): the coherent Σ 3 twin GB). It is very clear that in both cases the surface potential rises gradually over time. In particular, Fig. 2(i)–(l) show that the surface potential of the coherent Σ 3 twin GB area is brighter than the crystal interiors at a period of time after hydrogen charging. This observation corresponds to a higher hydrogen concentration at the twin GB site. However, this does not occur in the random GB case.

Fig. 2(m) and (n) illustrate the distribution of potential across the random GB and the coherent $\Sigma 3$ twin GB (along the dotted line shown in Fig. 2(b) and (h)) at various times. This further supports that successive hydrogen emergence on the surface can remarkably increase the surface potential. Fig. 2(m) shows that the stepped-shape change of the surface potential at the random GB site, which is caused by the difference in the work function of the disparate grains on either side of this GB, does not change significantly. On the other hand, Fig. 2(n) clearly shows that the potential-distance profile across the $\Sigma 3$ twin GB is similarly characterized by a step change in the initial stage after hydrogen loading; however, nearly 100 min after cathodic charging, it starts to present a potential peak at the $\Sigma 3$ twin GB area. As described above, this corresponds to a higher hydrogen concentration at this GB site.

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