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INTERNAL FRICTION OF DEFORMED IRON CONTAINING HYDROGEN

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Abstract. Internal friction of the deformed and hydrogen doped \( \alpha \)-Fe shows three peaks near 130K, 170K and 330K at 1 Hz. The 130K and 170K peaks are hydrogen cold work peaks and the 330K peak is a \( \gamma \)-peak. Activation energies of 0.22 eV and 0.35 eV were obtained for the 130K and 170K peaks, respectively. The 130K peak may be due to the interaction between nonscrew dislocations and hydrogen atoms and the 170K peak due to the interaction between nonscrew dislocations and aggregated hydrogen atoms on the nonscrew dislocations. Electrolytic hydrogen charging experiments indicate that the dislocation structure alters during the charging.

1. Introduction. The hydrogen cold work peak (H-CWP) of \( \alpha \)-Fe has been studied using internal friction (IF) [1,2] and magnetic after effect [3] techniques. In most previous studies hydrogen was introduced by electrolytic charging in acids at room temperature, and the H-CWP was observed near 130K at 1 Hz. However, we found another distinct peak near 170K at 1 Hz using a discharge method [4] which enables us to dope hydrogen from room temperature to below 100K and reported that there exist two H-CWPs [5]. The conditions of the appearance of the peaks are confirmed and a model for the 130K peak is proposed.

2. Experimental Procedures and Specimens. The specimens were wires 0.65 mm in diameter and 100 mm in length of MRC pure polycrystal iron containing about 20 wt ppm metallic impurities. The specimens were purified by heating in pure hydrogen gas [6].

IF, \( Q^{-1} \), and frequency, \( f \), were simultaneously measured from 78K to 400K at a strain amplitude of \( 3 \times 10^{-6} \) by an inverted torsion pendulum near 1 Hz in an axial magnetic field of 100 Oe.

The specimens were deformed 3\(^\circ\)5 \% in tension, mounted on the apparatus and deformed in torsion slightly near 300K before hydrogen charging. Some specimens were in situ deformed 2 \% in torsion additionally near 150K after hydrogen charging.

Two hydrogen charging methods were employed: an electrolytic method (1mA \( \sim \) 300mA) and a discharge method (20\( \mu \)A \( \sim \) 1mA) [5].
3. Experimental Results.

3-1. Repeated electrolytic hydrogen charging. The electrolytic hydrogen charging was repeated as follows: (1) 1mA × 0.5 h, (2)~(7) 10mA × 0.5 h, (8) 10mA × 3 h, (9) 100mA × 0.5 h, (10) 300mA × 0.5 h. $Q^{-1}$ and $f$ were measured after each hydrogen charging. In order to avoid other effects than hydrogen dope, the charging was done on the apparatus without dismounting the specimen. Results are shown in Fig. 1. Peaks grew and saturated after the 8th run. During the charging at 10mA for 3 h before the 8th run, a zero point shift of the pendulum (shear strain of $\approx 10^{-4}$ at the specimen surface) was observed in the inverse direction of the torsional deformation of the specimen.

3-2. Analysis. The IF maxima in Fig. 1 are decomposed into two peaks as shown in Fig 2(a) by a computer fitting. Each component can be described by four parameters: relaxation strength, peak temperature, activation energy, and $\beta$ parameter. The $\beta$ parameter is a measure of the width of a Gaussian distribution in logarithmic relaxation times [7]. We put two assumptions: (1) A plot of logarithmic background vs. $T^{-1}$ is linear; (2) Activation energies for two peaks are 0.22 eV and 0.35 eV, respectively (Fig. 4 and 5). Results are summarized in Fig. 2(b).

3-3. In situ deformation at 150K and hydrogen charging from 300K to 150K. After hydrogen charging at 300K, the specimen was deformed 2% in torsion on the pendulum at 150K and cooled to 78K, and two measuring runs (the 3rd and 4th runs in Fig. 3(a)) from 78K to room temperature were done. The IF spectrum in the 3rd run shows no distinct peak and that in the 4th run shows a well developed peak at 175K with a subsidiary peak around 130K. The modulus defect around 250K in the 3rd run is the same as that of the hydrogen free specimen after the low temperature deformation [6] and it is related to the point defects generated by the 150K deformation.

The 5th run in Fig. 3 shows the IF spectrum after the charging from 300K to 150K. The subsidiary peak around 130K in the 4th run is suppressed. The IF spectrum and the modulus curve in the 6th run are identical to those in the 4th run below 300K. These results indicate that the excess hydrogen charged between 300K and 150K by the discharge method escaped from the specimen during the warm up to 295K in the 5th run. The 330K peak in the 6th run is $\gamma$-peak. After heating up to 395K, the H-CWPs disappear due to the hydrogen outgassing [2,5].

3-4. Activation parameters for the 130K and 170K peaks. The 170K peak (the 4th or 6th run in Fig. 3) after the 150K deformation is stable for the heating up to 300K and enable us to measure activation
Fig. 1: Internal friction and modulus spectra of the specimen deformed 5% in tension and 0.05% in torsion near 300K, aged at 395K for 20 min, and hydrogen charged repeatedly by the electrolytic method. An elastic modulus is normalized at 78K.

Fig. 2(a): Decomposition of internal friction maximum in the 7th run in Fig. 1. Dashed line shows an assumed background.

Fig. 2(b): Evolution of peak heights, peak temperatures and $\beta$ parameters as functions of charge time. The number of the experiment run is indicated on the top.

the 130K peak,
the 170K peak.
parameters. As shown in Fig. 1, the 130K peak is well developed and the 170K peak is suppressed by the electrolytic charging. In such conditions, Arrhenius plot for the 130K peak was obtained. Activation energies of $0.35 \pm 0.05$ eV and $0.22 \pm 0.04$ eV were obtained for the 170K and 130K peaks, respectively (Fig. 4 and 5).

3-5. Aging experiments for the 130K peak. Figure 6 shows the relation between the peak height and the peak temperature of the 130K peak.

Fig. 3(a): Internal friction and modulus spectra of α-iron deformed 3% in tension and 0.1% in torsion near 300K. (1) After aging at 395K for 10 min; (2) After hydrogen charging at 200 μA for 1 h at 300K; (3) + 2% torsional deformation at 150K; (4) After (3); (5) After hydrogen charging from 300K to 150K. (b): (6) After (5); (7) After (6).
Fig. 4: Arrhenius plot for the 170K peak.

Fig. 5: Arrhenius plot for the 130K peak. (1) After hydrogen charging; (2) After partial outgassing. The inset shows the 130K peak near 0.38Hz.

After successive agings at 295K. It should be noted the the curve in Fig. 6 has a fine structure.

4. Discussion. From the elastic modulus measurements on the specimens charged with hydrogen using the discharge method, it has been concluded that both the 130K peak (P1) and the 170K peak (P2) are due to the interaction between hydrogen atoms and nonscrew dislocations [5].

The activation energy for P2 is higher than that for P1. When the excess hydrogen is doped at low temperatures, P1 is suppressed as shown in Fig. 3(a). These experimental results suggest that P1 is due to the interaction between hydrogen atoms and nonscrew dislocations and that P2 is due to the interaction between nonscrew dislocations and aggregated hydrogen atoms (probably, small clusters) with low mobility on the nonscrew dislocations.

P1 grew and increased to the well developed peak; P2 grew and decreased with increasing the time of the electrolytic charging (Fig. 2 (b)). The zero point shift observed before the 8th run indicates that the dislocations move and the dislocation structure changes slightly during the charging. The decrease of P2 and the increase of P1 observed after this large zero point shift may be related to the change of dislocation structure. The alteration of the dislocation structure...
depends on the charging conditions: the charge current density, the charge time (cf. Fig. 2(b) and [2]).

6. Model. Schoeck model [8] well interprets the behavior of CWPs in BCC metals but can not explain the impurity concentration dependence of the relaxation strength. A model taking a distribution of the binding energy between dislocations and hydrogen atoms into account is proposed. A detail formalism of our model will be described elsewhere. One of the model calculation results is shown in Fig. 7. The curve in Fig. 7 agrees sufficiently with that in Fig. 6.

References.