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To cite this version:

K. Tanaka, T. Atsumi, M. Yamada. AN INTERNAL FRICTION PEAK DUE TO HYDROGEN-DISLOCATION INTERACTION IN NICKEL. Journal de Physique Colloques, 1981, 42 (C5), pp.C5-139-C5-144. <10.1051/jphyscol:1981521>. <jpa-00221063>

HAL Id: jpa-00221063
https://hal.archives-ouvertes.fr/jpa-00221063
Submitted on 1 Jan 1981

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AN INTERNAL FRICTION PEAK DUE TO HYDROGEN-DISLOCATION INTERACTION IN NICKEL

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Abstract.- Effects of hydrogen on the internal friction in deformed Ni are investigated at a frequency of about 30kHz. A relaxation peak develops after hydrogen charging at the expense of another relaxation peak induced by deformation, but it decays on subsequent ageing (outgassing). A simplified description of the cold-work peak which takes into account a nonuniform distribution of hydrogen in a specimen explains well the observed behaviour of this peak.

1. Introduction

Studies on the behaviour of hydrogen in metals have attracted growing scientific and technological interests in recent years. Among others, much attention has been paid to studies revealing the interaction between hydrogen atoms and lattice defects in irradiated or deformed metals. Nickel is one of the materials that have often been subjected to these investigations. In Ni, evidence has been given of the trapping of hydrogen by dislocations through phenomena such as serrated yielding[1], strain ageing[2], deformation-assisted hydrogen transport[3], positron Doppler broadening[4], and so on. In our previous internal friction studies on cold-worked Ni[5,6], a relaxation peak ($P_{dH}$) was shown to develop after hydrogen charging at the expense of another peak ($P_d$) produced by the deformation. A good proportionality was found to exist between the height of $P_{dH}$ and that of $P_d$ in a fixed charging condition. Such a hydrogen-induced peak was not observed in fully annealed and hydrogen-charged Ni. $P_{dH}$ peak was interpreted to be the cold-work peak which involved the interaction between hydrogen atoms and dislocations, and $P_d$ peak to be an intrinsic dislocation peak (Bordoni peak) in Ni.

The present paper describes a further study of the effects of hydrogen charging and subsequent ageing upon these peaks.

2. Experimental Procedure

Cylindrical Ni rods of 99.95% purity, 4.5mm in diameter, were annealed at 900°C for 3h, deformed in tension at room temperature and subjected to cathodic hydrogen charging for 10 to 320h with a current density of 100mA/cm² in a 1N H₂SO₄ + H₂O solution kept at 50°C.
As shown later, the hydrogen concentrations were far from being uniform in as-charged specimens. The averaged concentration \((H/Ni)\) after 320h charging evaluated from a weight increment was \(\bar{c} = 0.0034\). The internal friction was measured employing the longitudinal vibration at a frequency of about 30kHz in a dc magnetic field of 500 Oe close to saturation to minimize the ferromagnetic damping[7].

3. Results

Figure 1 shows internal friction spectra of a specimen deformed by 4.0\% and subjected to successive hydrogen charging up to 320h. It is obvious from the figure that \(P_{dH}\) peak which occurs around 300K grows progressively with increasing charging time and reaches its maximum height after 320h, while \(P_d\) peak at 140K is suppressed gradually and disappears finally. The peak temperature of \(P_{dH}\) tends to shift upward during charging, while that of \(P_d\) does not. Upon subsequent isothermal ageing at 100\(^\circ\)C, this \(P_{dH}\) peak decays monotonically and shifts downward significantly, as shown in Fig. 2. When it almost anneals out after ageing for 4096h, the suppressed \(P_d\) peak begins to increase again.

The ageing characteristics of the internal friction in less charged specimens are somewhat different from those presented above. Figure 3 shows an example for a specimen deformed by 10.9\% and charged with hydrogen for 10h. In this case, \(P_{dH}\) peak first increases significantly on ageing shifting toward lower temperature, reaches its maximum height after 16h and then decays. On the other hand, \(P_d\) peak is temporarily suppressed during ageing, but recovers to the original height when \(P_{dH}\) peak anneals out completely after 1053h.

4. Analysis and Discussion

The above features of \(P_{dH}\) and \(P_d\) peaks may be related with non-uniform distributions of hydrogen which change significantly with charging and ageing times in the specimens. Utilizing the diffusion coefficient of H in Ni given by[8]

\[
D = 4.8 \times 10^{-3} \exp(-0.408eV/kT) \quad (\text{cm}^2/\text{s}),
\]

where \(k\) is Boltzmann's constant and \(T\) the temperature, and assuming that the hydrogen concentration at the surface of a specimen \((r = a)\) is given by \(c = c_0\) during charging and \(c = 0\) during ageing, the radial distribution of hydrogen in the specimen can be calculated from Fick's equations[6,9], as shown in Figs. 4-6. The \(c_0\)-value estimated from the total H-content after 320h charging is 0.0078. Since the diffusivity of H in Ni is relatively small and the specimen radius employed is large, it takes about 320h for hydrogen introduced at the surface
to arrive at the center of a specimen (Fig. 4). This must be a reason why $P_d$ peak is suppressed so gradually by $H$-charging. The hydrogen is removed, changing its distribution, upon ageing after 320h and 10h charging (Figs. 5, 6).

Now, we attempt to explain the behaviour of $P_{dH}$ peak based on Schoeck's model\cite{10} of the cold-work peak with certain modifications. It is assumed that the relaxation strength ($\Delta$) and relaxation time ($\tau$) of the cold-work peak change with the hydrogen concentration, as shown in Fig. 7. Namely,

$$\Delta = \begin{cases} \frac{C}{c_s} \Delta_0 & (c < c_s) \\ \Delta_0 & (c \geq c_s) \end{cases}$$

and

$$\tau = \tau_0 \exp \left( \frac{H}{kT} \right),$$

where $\Delta_0$ and $\tau_0$ are some constants that depend on the dislocation loop length. The internal friction is calculated from

$$Q^{-1} = 2 \int_0^1 \Delta \frac{\omega \tau}{1 + \omega^2 \tau^2} \left( \frac{\tau}{\tau_0} \right) \left( \frac{\tau}{\tau_0} \right), \quad (1)$$

where $\omega$ is the angular frequency. Putting into Eq. (1)

$$\omega \tau = \frac{C}{c_p} \exp \left[ \frac{H}{kT} \left( \frac{1}{T} - \frac{1}{T_p} \right) \right],$$

where $H = 0.75eV[6]$, $T_p = 348K$ and $c_p/c_0 = 0.8$ are chosen, and setting $c_s/c_0 = 0.1$, we obtain $Q^{-1}$ versus $T$ curves shown in Figs. 8-10.

Though the calculated internal friction spectra depend strongly upon the choice of $c_s/c_0$, they appear to conform, in general, to the observed spectra of $P_{dH}$ peak after charging (Fig. 1) and ageing (Figs. 2, 3). The temperature shifts of this peak are reproduced less satisfactorily by the calculation. However, it predicts smaller shifts during charging than during ageing, in agreement with the experiment.

5. Summary

(a) The internal friction in cold-worked Ni suffers from a great change when hydrogen is introduced into the lattice: $P_d$ peak is reduced but $P_{dH}$ peak is enhanced. Hydrogen interacts with dislocations, suppressing the former by pinning them at low temperatures and causing the latter by being dragged at elevated temperatures. A complete outgassing of hydrogen during subsequent ageing ($c < 10^{-7}$) well reproduces $P_d$ peak and eliminates $P_{dH}$ peak.

(b) The variations of the peak height and temperature of $P_{dH}$ peak
can be explained, at least qualitatively, by a simplified model calculation of the cold-work peak which takes into account nonuniform distributions of hydrogen in the Ni specimens.

References


Fig. 1: Effect of successive H-charging on Q-1 in 4.0% deformed Ni.

Fig. 2: Effect of isothermal ageing on Q-1 in 4.0% deformed Ni after 320h H-charging.
Fig. 3: Effect of isothermal ageing on $Q^{-1}$ in 10.9% deformed Ni after 10h H-charging.

Fig. 4: Radial concentrations of hydrogen with charging times indicated, calculated for a Ni rod of radius $a = 2.25$ mm.

Fig. 5: Variation of the hydrogen concentration during isothermal ageing after H-charging for 320h.

Fig. 6: Variation of $H$-concentration during ageing after charging for 10h.
Fig. 7: Relations between the relaxation strength ($\Delta$) and the relaxation time ($\tau$), and the hydrogen concentration ($c$).

Fig. 8: Calculated spectra of $Q^{-1}$ corresponding to the hydrogen distributions shown in Fig. 4.

Fig. 9: Calculated spectra of $Q^{-1}$ corresponding to the hydrogen distributions shown in Fig. 5.

Fig. 10: Calculated spectra of $Q^{-1}$ corresponding to the hydrogen distributions shown in Fig. 6.