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ON THE TWIST EFFECT OF V-H ALLOYS ASSOCIATED WITH THE PRECIPITATION OF HYDRIDES

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Abstract.- A vanadium wire containing hydrogen begins to twist by itself, when cooled, at the onset of hydride precipitation. The magnitude of the twist is greatly increased, if the specimen is plastically deformed in torsion prior to observations. The effect of the elastic stress is systematically studied by observing the zero point drift on cooling and heating under the applied torque; both the precipitation and the dissolution processes are found to be affected by the external stress.

Hydrides in vanadium is known to precipitate in the form of platelets along particular crystallographic planes. The twist effect can be interpreted as the preferential precipitation (dissolution) of one type of platelets over others under the effect of external or internal stresses.

1. Introduction.- The behaviour of hydrogen or deuterium in V, Nb and Ta has been studied extensively by various experimental methods. Recent research activities are well summarized in a monograph "Hydrogen in Metals" edited by Alefeld and Völk1[1]. The present authors have been studying the internal friction of V, Nb and Ta with and without hydrogen[2-7]. On cooling specimens containing hydrogen, an abrupt rise in internal friction is observed at a temperature where the precipitation of hydrides begins to occur; the internal friction shows a maximum as a function of temperature. The peak is usually referred to as the precipitation peak, and has been utilized to determine the hydrogen solvus boundaries.

The precipitation of hydrides can also be monitored by observing the drift of the zero point of a torsion pendulum; the specimen wire twists by itself at the onset of the precipitation. The phenomenon was first noticed by Owen and Scott[8], who referred to the effect as the Poynting effect; they stated in their paper:

"Because the mechanism of the rotation was not immediately apparent it was labelled the Poynting effect after the second-order elasticity coupling of shear and normal strains. Regardless of the mechanism, this phenomena affords a very sensitive indication of hydride formation and dissolution."

In fact, the sensitivity is confirmed and utilized in our previous
investigations[3,6].

The phenomenon has also been investigated by Ferron and Quintard[9], who ascribed it to the movement of dislocation loops punched-out by hydride precipitates; anisotropic distribution of precipitates is assumed as a consequence of textural anisotropy of wire specimens.

In the present paper, we report the effect of stress on the direction and the magnitude of the twist effect in V-H alloys, and discuss the mechanism of the phenomenon.

2. Experimental.- Vanadium wires of 0.6 mm in diameter and 70 mm in length are used in this investigation. The details of specimen preparation, deoxygenation treatment etc. were described elsewhere[2,3,6]. Hydrogen charging was made by equilibrating specimen wires with an appropriate amount of vanadium-hydride powder (VH$_{0.45}$) for 1 week at 500°C. The experimental results reported in this paper are all for specimens with 1 at.% H (V-1H).

The internal friction was measured in an inverted torsion pendulum over a temperature range from 90 K to 300 K. The zero point drift of the torsion pendulum was optically detected, and observed visually or recorded on a chart recorder. The apparatus used is the same as that used in previous studies[2-7].

3. Results and Discussion.- Figure 1 shows the internal friction and the zero point drift as a function of temperature. The twist of the specimen is expressed in terms of the surface shear strain. On cooling, the specimen begins to twist at a temperature where the internal friction shows an abrupt rise. Except for the first thermal cycle, the torsional behaviour is nearly reversible. This trend is in agreement with the observation by Ferron and Quintard[9].

After plastic deformation in torsion (1 turn ~ 3% in the surface shear strain) at room temperature, the magnitude of the self twist becomes about eight times as large as that before deformation (Fig.2). This observation led us to examine the effect of elastic stress on the torsional behaviour.

Figure 3 shows the zero point drift of a virgin specimen in three cooling runs at a rate of 1.7 K/min, first without torque, then with a positive (defined arbitrarily) torque and finally with a negative torque of the same magnitude, where the surface shear stress is calculated from the surface elastic strain by using the value of the shear modulus, $G = 4.66 \times 10^{10}$ N/m$^2$. Note that the specimen twists in the direction of the applied torque. The different magnitudes of the
self twist under the positive and negative stress suggest the presence of hysteresis or a memory effect.

A series of experiments to be described below were made on another virgin specimen with the same hydrogen concentration; the internal friction was no longer measured so that the specimen was left at rest during thermal cycles for the observation of the torsional behaviour.

The dependence of the magnitude of the self twist on the applied stress is examined by successive thermal cycles between 90 K and 300 K with increasing or decreasing the torque for each cycle; the result is summarized in Fig.4. In the first sequence, the torque is increased to a maximum value, in the positive direction (○), then decreased to a negative maximum value (●), and again increased (▲). For small stresses, the magnitude of self twist increases almost linearly, but seems to reach a saturation level at high stresses. It should be added here that the maximum stress is larger than the yield stress of vanadium at room temperature, so that the strain involves, at higher stresses, the contribution due to conventional plastic deformation.

Figures 5 and 6 show the effect of an applied stress on the dissolution process of hydrides. In this experiment, the specimen is cooled down to 90 K without torque; due to the memory of the preceding experiments, the specimen twists in the positive direction. Then, the specimen is heated under the application of either a negative (Fig.5) or a positive torque (Fig.6); the stress clearly affects the dissolution of hydrides.

The observations described above can be reasonably understood as follows. Hydrides in metals usually precipitates in the form of platelets along certain crystallographic planes. Anisotropic strains are produced either by precipitates themselves or by dislocations punched out from them. If the specimen is free from external and internal stresses, the total strain would be isotropic. However, if there exists any unidirectional stress, one type of precipitates would predominate over others so as to produce macroscopic shear strain, as schematically illustrated in Fig.7.

The effect of shear stress on the dissolution process, shown in Figs.5 and 6, is explained as follows. For simplicity, we assume that there are only two types of precipitates, one contributes to the positive strain and the other to the negative. If the internal stress is in the positive direction, the volume fraction $v_1$ of "positive" precipitates would be larger than that of "negative" precipitates, $v_2$. The magnitude of the self twist in the positive direction is proportional to the difference in the two volumes, $v_1 - v_2$. On cooling
without the external stress the specimen will twist in the positive direction, as schematically illustrated in Fig.8. On heating with a positive stress, "the negative" precipitates would preferentially dissolve so as to produce positive strain.

It is interesting to compare the strain versus temperature curves, in the same way as Ferron and Quintard[9], with the volume fraction of hydride calculated from the temperature dependence of hydrogen terminal solubility. The best fit was obtained with values of the heat of solution, 0.15 eV and 0.11 eV for the curves in Fig.3 with positive and negative shear stresses, respectively; if the magnitude of the twist is proportional to the volume of hydride precipitates, one should obtain the same value. Therefore, one should not expect a simple proportionality between the two quantities; for the same amount of precipitates the magnitude of twist would change with sizes and the distribution of precipitates. On the assumption that the twisting curves should provide a precise indication of the amount of precipitated hydrogen, Ferron and Quintard performed a quenching experiment, and interpreted that hydrogen can be quenched and the equilibrium is regained only on heating up to about 200 K. In view of the high diffusivity of hydrogen in vanadium, such an interpretation seems unlikely. Differences in sizes and the distribution of hydride particles on cooling at different rates could be responsible for the observed characteristics.

As mentioned in Introduction, Owen and Scott[8] labelled the self twist effect as the Poynting effect. Early in this century Poynting found that, when a metal wire is twisted, the length of the wire increases; the increment of the length is proportional to the square of the twist angle[10,11]. This phenomenon has been considered to be a direct manifestation of the second order effect of elasticity. In the light of the present observation, however, it seems not appropriate to refer to the self twist effect associated with the hydride precipitation as the Poynting effect.

References

Fig. 1: Zero point drift and internal friction as a function of temperature.

Fig. 2: Zero point drift and internal friction as a function of temperature after 3% deformation in torsion.

Fig. 3: Zero point drift under an external torque. $\sigma$ is the surface shear stress evaluated from the surface shear strain.

Fig. 4: The magnitude of twist associated with hydride precipitation as a function of the external stress (the surface shear stress).
Fig. 5: Zero point drift on heating with a negative torque. Dashed line indicates the drift on the preceding cooling run without torque.

Fig. 6: Zero point drift on heating with a positive torque.

Fig. 7: Distribution of precipitate platelets with and without shear stress (schematic).

Fig. 8: The change in the volume fraction of two types of hydrides as a function of temperature on cooling without an external stress and heating with an external stress. The magnitude of twist is considered to be approximately proportional to $v_1 - v_2$. 