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Y. Bertin, J. Gacougnolle, S. Sarrazin, J. de Fouquet. INTERNAL FRICTION TRANSIENT EFFECTS OF TITANIUM HYDROGEN SOLID SOLUTION. Journal de Physique Colloques, 1981, 42 (C5), pp.C5-175-C5-180. 10.1051/jphyscol:1981527 . jpa-00221069

HAL Id: jpa-00221069

<https://hal.science/jpa-00221069>

Submitted on 1 Jan 1981

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INTERNAL FRICTION TRANSIENT EFFECTS OF TITANIUM HYDROGEN SOLID SOLUTION

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Abstract. - The internal friction of pure Ti between 500K and 700K is constant at 10^{-4} when measured during a continuous increase of temperature. In opposite when the temperature is increased step by step, each increase of 10K induces immediately a raising up of $4 \cdot 10^{-4}$ internal friction. During the following stage at the new temperature the internal friction falls slowly down to its initial value.

This behaviour can be attributed to either the sudden modification of the thermodynamic equilibrium of pure titanium or the presence of a small amount of hydrogen trapped by the specimen during the experiment. This paper compares the internal friction of pure titanium and titanium containing 7 w.ppm of hydrogen.

The presence of hydrogen has three consequences :

- i - the internal friction background is increased from 10^{-4} up to $30 \cdot 10^{-4}$.
- ii - the transient maximum of internal friction due to the temperature steps is 10 times as high as the corresponding value obtained with pure titanium.
- iii - at temperatures higher than 700K large instabilities both of damping and modulus are recorded and it is not possible to reach an equilibrium value.

It can be concluded that the transient maximums of internal friction are specific of the metal, the presence of hydrogen increases the amplitude of the effects.

Introduction. - The spectrum of damping of titanium at 1 Hz is determined by increasing the temperature, step by step, from 200°C to 475°C(1). Each step gives rise to a sudden increase of the damping, and once the temperature is stabilized, the internal friction decreases slowly to reach its equilibrium value for this temperature. The maximum value of the transient internal friction, is twice the equilibrium value for a change of temperature of 10°C. This transient effect is observed for an increase or a decrease of the temperature greater than 5°C.

This transient effect has been observed on high purity titanium, hence it is difficult to attribute it to chemical instabilities; nevertheless its very low activation energy (0.2 eV) could indicate the contribution of hydrogen to this process. Hydrogen could come from a superficial chemical reaction between titanium and residual oil vapour in vacuum during the heat treatment which follows the mounting of the

specimen of pure titanium on the pendulum. In order to verify this hypothesis the transient effects on pure titanium and Ti containing 0.15 at. % H are compared.

Experimental procedure.— The specimens are platelets (80x8x1 mm) of high purity titanium with major impurities (at.ppm): O, 20 ; N, 20 ; C, 6 ; Fe, 2 ; Mn ; Mg ; Si < 1. The high degree of purity is confirmed by the very low elastic limit : 40 MPa. Hydrogen is added to titanium by electrolysis in $H_2 SO_4$ (4N) with a current density of 20 mA/cm² for 1 mm.

The internal friction is measured in an inverted torsion pendulum by the free decay method at a frequency of about 1 Hz. The maximum amplitude is 5×10^{-6} and the accuracy of measurement of damping ΔQ^{-1} is $\pm 10^{-5}$. The temperature is maintained constant within $\pm 0.25^\circ C$ as long as necessary.

After mounting on the pendulum, the specimens of pure titanium are annealed at $800^\circ C$ for 1 hour in vacuum of 5×10^{-6} torr. The specimens of Ti-0.15 at % H do not receive this heat treatment to avoid the loss of hydrogen in vacuum (2) as three specimens heat treated at $800^\circ C$ in ultra-vacuum behave like pure titanium.

Transient effects of pure titanium.— The transient of damping is shown on figure 1.

After several hours at $359^\circ C$ the specimen exhibits a nearly stable damping : $Q^{-1} = 2 \times 10^{-4}$. The temperature is then brought to $370^\circ C$, this change of temperature takes one hour. In the figure 1 it can be checked that the period of the pendulum increases simultaneously with the temperature indicated by the thermocouple.

As soon as the temperature of the specimen increases its internal friction rises . The maximum value $Q_M^{-1} = 4.2 \times 10^{-4}$ is reached 20 mn after the start of the heating and then the damping decreases exponentially. The equilibrium value $Q_\infty^{-1} = 2 \times 10^{-4}$ is reached after 6 hours following the relationship (fig. 2) :

$$Q^{-1}(t) = Q_\infty^{-1} + (Q_M^{-1} - Q_\infty^{-1}) \exp - \frac{t}{\tau^*} \quad |1|$$

The transient effect is defined by 2 parameters : its magnitude $(Q_M^{-1} - Q_\infty^{-1})$ and its decreasing rate related to the characteristic time τ^* .

As shown in figures 3 and 4 these two parameters are functions of the temperature : the value $(Q_M^{-1} - Q_\infty^{-1})$ increases slowly from 1×10^{-4} at $230^\circ C$ to 3.5×10^{-4} at $437^\circ C$, then decreases rapidly and the transient

effect vanishes at 470°C. The characteristic time first decreases from 5h at 230°C to 2.5h at 430°C, and it rises suddenly up to 5.5h at 437°C then decreases again to 1 h at 465°C. So two temperature ranges must be distinguished : one below 430°C with an activation energy of 0.2 eV and another above 430°C with an activation energy 2.6 eV.

The transient effect is influenced by several parameters : the purity of titanium, the content of oxygen, the microstructure and also the conditions of the temperature changes. All the parameters will be considered in detail in a future article (7). In this paper we study the influence of hydrogen only.

Transients effects of Ti-0.15 at % H.- Measurements have been made raising the temperature step by step from 20°C up to 450°C this temperature range was covered twice on each sample. During the first heating the specimen behaves like the pure material but the values of Q_M^{-1} and Q_∞^{-1} are increased by a factor 2. In the figure 5 the values of Q_M^{-1} and Q_∞^{-1} are respectively 12.5×10^{-4} and 4×10^{-4} at 355°C instead of 4.2×10^{-4} and 2×10^{-4} for the pure metal. The sample is held for 6 hours at 400°C and then cooled on the pendulum.

During the second heating the transient maximum and the equilibrium value are increased : at 365°C the values of Q_M^{-1} and Q_∞^{-1} are 95×10^{-4} and 35×10^{-4} , 20 times their values on the pure metal, but the characteristic time $\tau^* = 2h$ remains identical (fig. 6). The maximum of the transient is reached 80 mn after the beginning of the change of the temperature, so the damping continues to increase for 30 mn after the temperature is stabilized.

Above 420°C a new kind of instability appears during the holds at constant temperatures ; three examples are given below :

- i) At 432°C (fig 7a-7b) the decrease of Q^{-1} which follows the maximum is scattered, $\Delta Q^{-1} = \pm 10 \times 10^{-4}$, compared to the scattering at 365°C which is only $\pm 2 \times 10^{-4}$. Moreover after 4 hours at 432°C the damping raises suddenly from 100 to 200×100^{-4} whereas the modulus remains constant. During the first 4 hours the period decreases simultaneously with the damping but the amplitude effect is inversed after 2 hours.
- ii) During the hold at 462°C (fig. 8a-8b) it was not possible to analyze the variations of the damping. We could only observed that $Q^{-1} < 100 \times 10^{-4}$. The period measurements are also scattered. They vary from 4.5 to 4.85 s, which corresponds to a modulus defect

$\frac{\Delta G}{G} = 15 \times 10^{-2}$. This high modulus defect cannot be directly related to the damping.

iii) At 471°C (fig. 9a-9b) the simultaneous variations of the damping and the period occur every 90 mn after the first maximum but these variations are in contradiction : after 3 hours at 471°C, Q^{-1} decreases from 60 to 40×10^{-4} whereas the increase of the period shows a modulus decrease of 180×10^{-4} instead of a modulus increase.

Discussion.— The solubility of hydrogen in titanium is approximately 0.1 at % at 20°C and 10 at % at 300°C (3) (4) (5), but the original Ti-H diagram, given by Lenning et al, is not established between 350°C and 600°C.

When the hydrogen ions go into the specimen they precipitate close to the surface because of the low solubility of hydrogen at room temperature. These hydrides are dissolved during the first heating at 400°C, then hydrogen is entirely in solid solution for temperatures higher than 200°C.

The transient phenomena in pure titanium are attributed to the modifications of the equilibrium position of the dislocations in the Frank network as this equilibrium network is different for different temperatures. Some dislocations are temporarily unpinned and can contribute to the damping until they are pinned again.

The two temperature ranges below and above 430°C which are described for pure titanium exist also for the titanium-hydrogen solid solution. Below 400°C the presence of hydrogen magnifies the transient effects, increases the equilibrium values Q_{∞}^{-1} and delays the time necessary to reach Q_M^{-1} after the temperature change but the characteristic time τ^* is not modified. The interaction of unpinned dislocations with the numerous isolated hydrogen atoms contributes certainly in the increase of the damping as suggested by Richtie et al.

For temperatures higher than 400°C, the sudden and opposite variations of the damping and the modulus during the holds at constant temperature can be explained only by chemical instabilities. Bertin has shown that the vacancies are mobile at temperatures higher than 420°C and that the diffusion of vacancies is the controlling process of the characteristic time τ^* in pure titanium for this temperature range. In the case of the titanium-hydrogen solid solution the variations of Q^{-1} are so high that the characteristic time has no significance. C.W. Schoenfelder et al (2) have shown that hydrogen leaves titanium in vacuum for temperatures higher than 400°C by a process

of diffusion through a gaseous boundary layer on the surface of the specimen but they do not give any information on the nature of this layer. The action of this layer on the damping and the modulus of the specimen would be a good way of investigation.

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Fig. 1 : Variation of period and internal friction of pure titanium due to an increase of the temperature.

Fig. 2 : Verification of the relationship |1| for different temperatures.

Fig. 3 : Variation of $(Q''_M - Q''_∞)^{-1}$ with the temperature.

Fig. 4 : Variation of τ^* with the temperature.

Fig. 5 : Transient effect of Ti-0.15 at % H at 355°C during the first heating.

Fig. 6 : Transient effect of Ti-0.15 at % H at 365°C during the second heating.

Fig. 7a : Internal friction of Ti-0.15 at % H at 432°C.

Fig. 7b : Period of Ti-0.15 at % H at 432°C.

Fig. 8a : Internal friction of Ti-0.15 at % H at 462°C.

Fig. 8b : Period of Ti-0.15 at % H at 462°C.