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### INTERNAL FRICTION ASSOCIATED WITH THE ALLOTROPIC TRANSFORMATION OF COBALT

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<u>Résumé</u> - Des mesures de frottement intérieur ont été effectuées dans du cobalt de haute pureté au voisinage de la transformation allotropique hc-cfc. A basse fréquence ( $\sim$  1 Hz), on observe un pic important qui est dû à un effet transitoire. En effet, ce pic disparaît en conditions isothermes. A haute fréquence ( $\sim$  5 kHz), le terme transitoire est absent. Le frottement intérieur varie de façon discontinue au cours de la transformation de phase. De plus, au chauffage, apparaissent un petit pic de frottement intérieur et une chute de la fréquence, qui sont probablement reliés à la transformation de phase.

Ces caractéristiques sont analogues à celles observées dans d'autres transformations martensitiques.

<u>Abstract</u> - Measurements of internal friction have been performed in highpurity cobalt in the temperature range of the allotropic hcp-fcc transformation.

At low frequency ( $\sim$  1 Hz), an important peak is observed which is due to a transitory effect. This peak, actually, disappears under isothermal conditions.

At high frequency ( $\sim$  5 kHz), the transitory term is absent and a discontinuous change of internal friction occurs during the transformation. Moreover during heating, a little internal friction peak and a frequency dip are observed, which are probably connected with the phase transformation. These features are similar to those observed for other martensitic transformations.

#### I. INTRODUCTION

The internal friction spectrum associated with first order phase transitions (specially martensitic transformations) can be decomposed into three terms :

$$Q_{Tr}^{-1}$$
,  $Q_{PT}^{-1}$  and  $Q_{Cl}^{-1}$ .

The first,  $Q_{T_{k}}^{-1}$ , is a transitory term which appears only during heating or cooling. It has been shown [1, 2, 3] that  $Q_{T_{k}}^{-1}$  is proportional to the amount of material transformed per cycle, and as a consequence

$$Q_{T_n}^{-1} = \alpha(\tilde{T}/f)$$

(1)

where  $\alpha$  is a constant, f is the frequency of the applied stress and  $\tilde{T}$  is the heating or cooling rate. The second term,  $Q_{PT}^{-1}$ , is also associated with the phase transition, but it does not depend on  $\tilde{T}$ . It is, on the contrary, correlated with the chan-

ge of the equilibrium state induced by the applied stress near the phase transition. Dejonghe et al. [3] and Koshimizu [4] have shown that  $Q_{P1}^{-1}$  can depend on the frequency and/or the amplitude of the applied stress. As pointed out by Koshimizu [4], this term should give important information on the dynamical properties of the phase transition.

The third term,  $Q_{-1}^{-1}$  is a classical term, which is not directly associated with the phase transition, <sup>C1</sup> but which corresponds to the appropriate internal friction due either to the low temperature  $(Q_{1-1}^{-1})$  or high temperature  $(Q_{1-1}^{-1})$  phase. Thus

$$Q_{\Gamma 1}^{-1} = m Q_{\Gamma T}^{-1} + (1 - m) Q_{\Pi T}^{-1}$$
 (2)

where m = m(T) is the proportion of the low temperature phase and can change more or less suddenly during the phase transition.

Since the microscopical mechanisms and, more precisely, the role of the dislocations are well known for the allotropic transformation of cobalt [5, 6], internal friction measurements were performed on this metal. In order to show the respective importance of the three terms, measurements were carried out in two frequency ranges (1 Hz and 5 kHz) and at  $\hat{1}$  different or equal to zero.

#### II. EXPERIMENTAL METHODS

The cobalt used was provided by Uohnson-Matthey in the shape of a 2 mm thick polycrystalline sheet. The nominal purity of the material was 99.99 % Co. The average grain diameter was about 0.1 mm.

From this material a 40 x 4 x 2 mm sample was cut by electroerosion, and then annealed 4 hours at 800  $^{\circ}$ C. Measurements were carried out in the high frequency range (5 kHz) using a free-free bar apparatus.

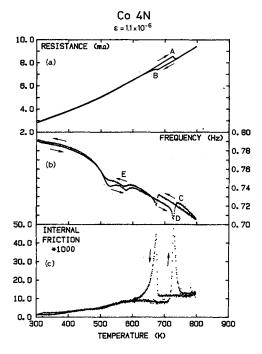


Fig. 1 - Co 4N, Temperature dependance of electrical resistance, frequency and internal friction at low frequency ( $\sim$  1 Hz).

Experiments were also performed at low frequency (1 Hz) using an inverted torsional pendulum. A 40  $\times$  4  $\times$  0.5 mm sample was cut from the former one in order to get a frequency close to 1 Hz. The torsional pendulum apparatus allowed simultaneous measurements of internal friction, frequency and electrical resistivity to be made as a function of temperature.

#### Low frequency experiments

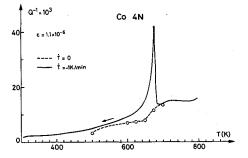
Fig. I shows typical results obtained during heating and cooling at  $\tilde{T} = 1$  K/min. In the vicinity of the transformation ( $\sim$  700 K), anomalies of the measured quantities are observed [6, 7]. On heating, a sudden decrease of the electrical resistance is observed (A, fig. 1(a)) due to the transformation. The reverse effect appears on cooling (B, fig. 1(a)) with a temperature hysteresis of about 50 degrees. Similar effects occur for the frequency, except that the jump (c, fig. 1(b)) is preceded by a dip (D, fig. 1(b)). This dip is often observed during structural phase transitions and is generally attributed to a softening of the shear modulus [8]. As is the case for many martensitic transformations, the internal friction exhibits an important peak near the transformation temperature [9]. In order to display the transitory component  $(Q_{1}^{-1})$ , measurements were carried out also under isothermal conditions. The internal friction peak, which appears when the temperature is varied (fig. 2, full curve), is no more present under isothermal conditions (fig. 2, dashed curve). As a consequence, this peak is due to a transitory effect  $(Q_{T_{\infty}}^{-1})$ . Since no maximum of internal friction is observed in the isothermal conditions, it seems that the second term  $Q_{PT}^{-1}$  is small or in any case difficult to be observed in the low frequency range. The correlation between this curve and the resistivity measurements shows that the change of internal friction observed during the trans-

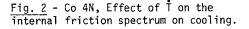
formation is due to the classical component  $Q_{-1}^{-1}$ . As for the internal friction, the frequency dip which is present when  $t \neq 0$  also disappears completely when t = 0. This means that this effect is strictly correlated with the effect responsible for the internal friction peak and should not be attributed to a softening of the shear modulus. The same behaviour was observed during heating.

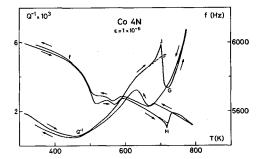
In the 500 K to 600 K temperature range, the frequency shows an anomalous decrease, the curve going through two minima (E, fig. l(b)). This effect is probably related to the drop of elastic constants observed by Wallace [10] in the vicinity of 250°C, the origin of which is attributed to a change of the easy axis of magnetization.

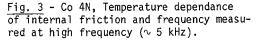
#### High frequency experiments (5 kHz)

Fig 3. shows results obtained in the kHz frequency range during heating and cooling.





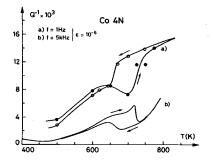




In the vicinity of the allotropic transformation, anomalies are observed on the in-

ternal friction and frequency curves. Measurements done at different heating and cooling rates or under isothermal conditions ( $\tilde{T} = 0$ ) show no effect on the internal friction spectrum. This result is in accordance with equation 1 and shows that the transitory term is completely absent in this frequency range. As a consequence,

this frequency range is more suitable for studying the two other terms,  $Q_{\text{PT}}^{-1}$  and  $Q_{c1}^{-1}$ . It is not easy, but nevertheless important, to show the influence of each of these two terms. Certainly, the big drop (F to G, fig. 3) observed on the internal friction curve is due to the classical term  $Q_{c1}^{-1}$  and should be attributed to the appropriate internal friction of the low or high temperature phase (eq. 2). But, during heating the dip observed on the frequency curve (H, fig. 3) is certainly correlated with a small peak appearing on the internal friction curve (J, fig. 3). These features should be systematically studied in order to understand better how they are associated with the phase transition  $(Q_{pT}^{-1} \text{ term})$ . Note that during cooling, these features are not observed on the internal friction and the frequency curves. Finally, figure 4 shows a comparison between the behaviors of the last term  $Q_{C1}^{-1}$ in the low and high frequency ranges. Significant differences are observed :



 The internal friction is much smaller for high frequency measurements than for low frequency measurements.

- The jump of internal friction appearing during the phase transition occurs in an opposite sense, i.e. for low frequency measurements on heating, the internal friction increases at the phase transition, whereas for high frequency measurements, it decreases. As a consequence the physical mechanisms responsible for the internal friction should be different for the two frequency ranges.

Fig. 4 - Co 4N. (a) low frequency internal friction ( $\sim$  1 Hz) under isothermal conditions ( $\dot{T}$  = 0). (b) High frequency internal friction ( $\sim$  5 kHz).

#### III. CONCLUSION

From a general point of view, the internal friction measured during the allotropic transformation of high-purity cobalt shows quite similar effects as for other martensitic transformations. In the low frequency range, the transitory term  $Q_{-1}^{-1}$  is predominant and masks  $Q_{PT}^{-1}$  completely. On the other hand, in the kHz range, the tran-sitory term disappears. The change in the classical term  $Q_{C1}^{-1}$  is the most important effect, but during heating a frequency dip (H, fig. 3) and an internal friction peak (J, fig. 3) occur, which is probably related to the phase transformation  $(Q_{DT}^{-1})$ .

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