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STUDY OF THE hcp-fcc PHASE TRANSITION IN COBALT BY INTERNAL FRICTION AND ELASTIC MODULUS MEASUREMENTS IN THE kHz FREQUENCY RANGE

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Abstract

Internal friction and dynamic elastic modulus measurements were carried out in the vicinity of the hcp-fcc phase transition in pure cobalt. The vibration frequency was high enough (kHz) to eliminate the transitory internal friction. A peak of internal friction accompanied by an anomaly of the elastic modulus was observed.

It is shown using thermal cycling near the transition temperature that the modulus anomaly is the superposition of the two effects: a) a step-like decrease due to the change of elastic constants hcp-fcc. b) a dip of anelastic origin. The dip is associated with the internal friction peak.

No pretransitional effect was observed. The anomaly of the modulus is clearly related to the simultaneous presence of the two phases hcp and fcc. This suggests that the anomaly of the modulus and the internal friction peak can be associated with the hcp-fcc interfaces.

INTRODUCTION

The Internal friction (IF) observed in the vicinity of a first order phase transition is composed of the three terms: IF_{tr} (Transitory IF), IF_{PT} (Phase Transition IF), IF_{cl} (Classical IF) [1,2,3,4].
The first term, $IFT_T$, is a transitory term. It appears only during heating or cooling (heating or cooling rate $\dot{T} \neq 0$). In Delorme's model $IFT_T$ is proportional to the amount of transformed phase during one cycle of vibration. Consequently:

$$IFT_T = \frac{\partial m}{\partial T} \frac{\dot{T}}{f} \tag{1}$$

where $m$ is the amount of phase transformed, $\dot{T}$ the heating rate and $f$ the frequency of vibration [1].

The second term, $IF_{PT}$, accounts for the peak which is anyway observed when the transitory IF is absent, that is at constant temperature ($T=0$) or high frequency. Different models have been developed which can explain this internal friction. They involve either interface motion [5], dislocation motion [6,7], or localized soft modes [8]. Some of these models concern mainly the pretransitional domain (soft modes) while other apply to the phase transition itself (interfaces). As pointed out by Koshimizu [7], $IF_{PT}$ should provide very useful informations on the mechanism by which the transformation proceeds.

The third term, $IFT_I$, accounts for the background in the two phases.

In the present study, we show that $IF_{PT}$ is found in cobalt and is associated with an elastic modulus anomaly. Then, we present thermal cycling experiments. These experiments were made to determine if $IF_{PT}$ is a pretransitional effect or rather an effect occurring during the transformation process. By means of thermal cycling, we detect the temperature at which the hcp-fcc hysteresis appears. Assuming that this temperature is the phase transition temperature, it is possible to distinguish between pretransitional effects and transitional effects.

**EXPERIMENTAL**

The cobalt used in these experiments was a 99.98% Co polycrystal provided by Johnson Matthey (impurities: Cu 200 ppm, Fe 2 ppm, others < 1 ppm). The average grain size was 1 mm. The sample was spark-cut to approximate dimensions 40*4*1.4 mm. Then, the internal friction and the elastic modulus were measured using a free-free bar apparatus working at the frequency of the fundamental flexural mode (3 KHz) [9]. As will be shown, this frequency is high enough to eliminate the transitory internal friction.
RESULTS

A typical result obtained at the vibration frequency of nearly 4 KHz is shown on Fig 1.

In the vicinity of 700 K, the hcp (low temperature phase) to fcc (high temperature phase) transition gives rise to important modifications of the internal friction and frequency. The frequency (related to the elastic modulus E by the relation \( E = f^2 \)) decreases suddenly when the hcp\(\rightarrow\)fcc phase transition occurs. The curve goes through a dip (A, Fig 1) and then follows again a normal linear dependence. On cooling, the curve is linear in temperature down to 670 K, then it drops, goes through a minimum (B), and afterwards increases slowly to reach a value corresponding to the hcp elastic modulus.

On heating, the internal friction curve exhibits a peak (C). The increase of the internal friction corresponds to the frequency drop. By contrast, no pronounced peak was found on cooling but rather an increase of internal friction. This increase of internal friction (D) is correlated with the frequency drop.

The height of the peak does not depend on heating or cooling rate \( \dot{T} \). Therefore, internal friction cannot be related to the transitory effects as it was the case at low frequency [4]. This is in accordance with equation (1) which predicts a 1000 times lower internal friction at the KHz frequency than at the Hz frequency. As the peak height is roughly 50*10^{-3} at 1 Hz, the transitory IF must be negligible at frequencies higher than the KHz. This confirms that the KHz frequency range is suitable for studying the term IF_{PT}.

Between 500 and 600 K, an anomalous decrease in frequency is observed (E, Fig 1), the curve going through two minima. This anomaly of frequency is correlated with a small increase of IF (F). It has been shown that these effects are due to a change of the easy axis of magnetization from parallel to perpendicular to the C hexagonal axis [10].
In order to interpret the internal friction peak and the frequency drop appearing at the hcp-fcc phase transition, it is essential to know what must be attributed to pretransitional effects in the parent phase and what deals with the transformation itself. This means that we have to determine the transition temperature (Cooling: Ms, heating: As). To do that, we will use thermal cyclings and we will detect the temperature at which hysteresis appears for the first time on the frequency curve. We will assume this temperature as the transition temperature. The pretransitional effects should not produce thermal hysteresis, unlike the transitional effects.

**Thermal cycling near Ms:**
Series of thermal cycling were made in the vicinity of the fcc→hcp phase transition temperature (Ms) (Fig 2). During these cyclings, the resonant frequency of the sample was measured.

![Graph showing thermal cycling](image)

Fig. 2. Co 99.98%, thermal cycling near the fcc→hcp transition temperature (Ms), strain amplitude ε=5*10^-6, dashed line: complete transformation.

The first cycle was made entirely in the fcc phase so that no hysteresis appeared. Since the second cycle, the temperature was lowered enough to induce the transformation of a little amount of hcp phase. An hysteresis loop was observed on the frequency curve which became larger at every subsequent cycle in proportion to the amount of phase transformed. In the third cycle, the frequency drop occurring at the beginning of the transition (B) was not recovered immediately on heating. The frequency remained low until the temperature of the reverse transformation was reached (near point A). This suggests that the frequency drop (B) does not correspond to some catastrophic break away related to the creation of the hcp phase. It should be more likely associated with an anelastic effect in the stable state of the two-phased material. The reverse transformation gives rise to a dip (A). This dip is less pronounced on subsequent cycles because a step-like change of frequency is
superimposed on the dip when a larger amount of material transforms. This step-like change of frequency is naturally explained by the fact that the fcc elastic modulus is lower than the hcp elastic modulus.

It is now clear that the frequency drop (B) occurring at the beginning of the cooling phase transition results from the transformation and not from a pretransitional phenomenon. As shown on fig 3 the Ms temperature must be placed just prior to the frequency drop.

Fig 3. Co 99.98%, temperature dependence of internal friction (IF) and frequency (f) showing the position of Ms and As, strain amplitude ε=10^-6, T=1.2 K/min.

Fig 4. Co 99.98%, thermal cycling near the hcp→fcc transition temperature (As) strain amplitude ε=10^-6, dashed line: complete transformation.

**Thermal cycling near As:**

Similar experiments were made near the hcp→fcc transition temperature (Fig 4). A first cycle was performed entirely in the hcp phase. Though the cooling curve is not superimposed on the heating curve, the characteristic phase transition hysteresis loop was not observed. In contrast, in the next cycles, the temperature was raised beyond the temperature (A) of the frequency drop. An hysteresis appears which indicates that the phase transition has already occurred.

Here also, the frequency drop is not a precursor effect for the phase transition. The As temperature has to be placed before the frequency drop (see Fig 3).
DISCUSSION

The experimental results show that thermal hysteresis is present since the beginning of the frequency drop. Therefore, this anomaly and the internal friction increase are connected with the phase transition and more precisely with the state when the two phases coexist. The internal friction could be attributed to the hcp-fcc interfaces. IF could be produced by the motion of the interfaces taken as a whole or by the individual partial dislocations which constitute the interface.

The step-like change of the elastic modulus is most likely due to the change of the elastic constants. This is in agreement with the neutron scattering measurements of Frey et al [11] which have found a drop of 25% in the hexagonal elastic constant $C_{44}$ at the hcp→fcc phase transition.

CONCLUSION

An internal friction peak associated with an elastic modulus anomaly was found at the hcp-fcc phase transition in cobalt which is not of transitory nature.

By means of thermal cycling near the phase transition temperature, it was shown that this internal friction was associated with the transformation rather than with pretransitional effects.

This suggests that the internal friction can be associated with the interphase boundaries.

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