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Stress Induced Ordering Due to Interstitial Elements in Nb and Ta by Frequency Analysis

C.R. Grandini and M.M. Shukla

Departamento de Física, UNESP, 17.033-360, Bauru SP, Brazil

Abstract. It is well known that the interstitial elements present in solid solution in metals interact with the matrix by a relaxation process known as stress induced ordering. Traditionally this relaxation process is observed in the internal friction measurements. It is a common practice that researchers present the results of the frequency together with internal friction without giving any analysis. In this work we apply an expression which relates the variation of frequency with temperature and analyse the experimental results cited in the literature of the relaxation process due to the stress induced ordering of oxygen and nitrogen present in niobium and tantalum.

1. INTRODUCTION

For the last few decades there has been extensive theoretical as well as experimental studies on the variation of internal friction with temperature on anelastic internal friction studies (see review articles of Szkopiak [1], Weller [2] and Wert [3]). The fact that the relaxation rate varies by an Arrhenius law in term of the activation energy and the absolute temperature of the solid gives an analytical expression for the variation of internal friction with the temperature. The vibration frequency of the solid is also related indirectly to the internal friction and the variation of internal friction with temperature also gives rise to variation of vibration frequency, f , with the temperature. As far as our knowledge goes no one in the literature has given a straight forward expression to relate the variation of vibration frequency with temperature although Weller *et al.* [4] and Hermida e Povolo [5] have given very tedious expression to achieve this goal. In this work we are presenting a direct expression to show the variation of f^2 with temperature. We also present here the experimental measurements done by other authors in the samples of niobium and tantalum containing oxygen in solid solution to verify our theoretical predictions.

2. THEORY

As a matter of fact the theory derived by us is not a new result but rather some mathematical manipulation of the expression given in the book of Nowick and Berry [6]. According to Nowick and Berry, the variation of modulus M with $\omega\tau$ can be expressed by:

$$\frac{M(\omega\tau)}{M_0} = 1 - \frac{\Delta}{(1 + \omega^2\tau^2)} \quad (1)$$

Where M_0 is the unrelaxed modulus and Δ is the relaxation strength. As is well known that the square of the frequency f is proportional to M , thus we have:

$$\frac{M(\omega\tau)}{M_u} = \frac{f^2}{f_u^2} = 1 - \frac{\Delta}{(1 + \omega^2\tau^2)} \quad (2)$$

Expressing the relaxation time by an Arrhenius type and noting that $\omega\tau = 1$ for $T = T_p$, where T_p is the peak temperature and substituting this expression in equation (2), we have:

$$f^2 = f_0^2 \left\{ 1 + \frac{\Delta}{2} \exp \left[\frac{E}{k} \left(\frac{1}{T} - \frac{1}{T_p} \right) \right] \text{sech} \left[\frac{E}{k} \left(\frac{1}{T} - \frac{1}{T_p} \right) \right] \right\} \quad (3)$$

where f_0 is the value of f at room temperature.

Now, we would like to introduce the variation of Δ with the temperature. Following Weller *et al.* [4], it is given by:

$$\Delta = \frac{2Q_p^{-1}T_p}{T} \quad (4)$$

Substitution of equation (4) in (2) and (3) gives the final expression of the variation of the frequency with the temperature, as given by:

$$f^2 = f_0^2 \left\{ 1 + \frac{Q_p^{-1}T_p}{T} \exp \left[\frac{E}{k} \left(\frac{1}{T} - \frac{1}{T_p} \right) \right] \text{sech} \left[\frac{E}{k} \left(\frac{1}{T} - \frac{1}{T_p} \right) \right] \right\} \quad (5)$$

3. EXPERIMENTAL MEASUREMENTS AND COMPARISON WITH THE THEORY

The samples utilized for the verification of our theory was that of niobium (sample 289 [9]) and tantalum (sample 237 [9]) containing oxygen in solid solution. Figures 1 and 2 shows the variation of f^2 with the inverse of temperature for the above samples.

Figure 1. Relative square frequency for the niobium sample.

Figure 2. Relative square frequency for the tantalum sample.

Table 1 gives the relaxation parameters obtained by fitting our equation and that found in the literature, by internal friction analysis.

A critical analysis of the figures 1 and 2 shows that we have obtained a good fit between the theory proposed here and the experimental results for f^2 versus temperature. There is a good agreement between two results in the entire course of the figures.

Table 1. Relaxation Parameters for Nb-O and Ta-O relaxation process

System	Process	Analysis	T(K)	E (eV)	Reference
Nb	Nb-O	Frequency	438	1.17	this
		Internal Friction	424	1.165	[9]
			430	1.15	[8]
			427	1.165	[10]
			425	1.16	[11]
Ta	Ta-O	Frequency	423	1.11	this
		Internal Friction	423	1.105	[9]
			415	1.10	[12]
			445	1.09	[13]

4. DISCUSSION AND CONCLUSION

The basic aim of this paper was to give a direct expression between f^2 and T which we have achieved. As an example, we utilized the experimental measurements of the internal friction available in the literature for niobium and tantalum containing oxygen in solid solution [9] to test our expression.

A look at figure 1 shows that we have obtained a good fit with the experimental data on the theoretical equation presented here.

We are further aware of some of the short coming of our model in the sense that the real and imaginary part of the mechanical compliance are not independent in a rigorous treatment but are related to the Kramers-Krönig relations. Our treatment is quite simple as it is based on classical approach of the dynamical response function together with a single Debye peak. This treatment is only valid when the loss tangent is very small.

We would like to point it out here that there exists a way to calculate the activation energy from the temperature derivative of f^2 (see Iwasaki *et al.* [14]). But, we think that this result would not reflect anything new in this work as our basic aim was to give an expression to calculate the temperature variation of f^2 .

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