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Internal Friction and Frequency Measurements in Molybdenum Containing Oxygen and Nitrogen

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Abstract. We have measured internal friction and frequency as a function of temperature in molybdenum containing oxygen and nitrogen in solid solution. These measurements were performed by a torsion pendulum operating in the temperature range of 300 K to 700 K with oscillation frequency about 1.0 Hz. The results showed the complex relaxation process identifying the stress induced ordering of oxygen and nitrogen atom around the molybdenum atoms of the metallic matrix.

1. INTRODUCTION

Impurity interstitial atoms as O and N dissolved in solid solution causes strong alterations in the anelastic behaviour in bcc metals. The first one to study this effect was Snoek [1], who postulated that these atoms reordered by an external applied stress. This reordering gives peaks in the internal friction spectrum as a function of temperature, called Snoek peaks. Later, several works were made with the objective of understanding this relaxation process in various types of metals, as Nb, Ta and V (see, for a review Berry [2], Wert [3], Szkopiak [4] and Weller [5]). This work presents the Snoek relaxation studied in single crystal molybdenum samples, containing oxygen and nitrogen in solid solution, by internal friction and frequency measurements as a function of temperature. The results showed a relaxation structure that was resolved in two Snoek peaks, one due to oxygen and other due to nitrogen.

2. EXPERIMENTAL PART

2.1. Samples

The single crystal molybdenum samples were produced by electron beam melting in Rice University (Houston, TX). This samples contain oxygen and nitrogen residuals of the fusion. Chemical analyses to determine the oxygen and nitrogen contents were performed using a Leco TC-136 analyser. The oxygen and nitrogen contents were estimated in 0.060 wt. % of N and 0.009 wt. % of O. The accuracy of these measurements were respectively 0.007 and 0.002 wt %.

2.2. Internal Friction and Frequency Measurements

Measurements of internal friction and frequency were obtained as a function of temperature by means of an inverted torsion pendulum of Kê type [6] between 300 and 700 K. A heating rate of 1.0 K/min. was employed. The data were acquired by an automatic system which measured the angular velocity of the pendulum about the equilibrium point. The maximum strain amplitude was about 10^{-5}. The internal friction and frequency spectra were resolved into their constituents, associated with relaxation processes represented by a Debye peak or step, analysed by Successive Subtraction Method [7], using the Jandel Peak Fitting software.

3. RESULTS

The internal friction curve as a function of T^{-1} for the sample measured with frequency of 1.5 Hz (sample Mo15) is shown in Fig. 1 and was resolved into two Debye peaks by the method of successive subtraction. Two stress-induced thermally activated relaxation processes are evident. The following metal-interstitial interactions are proposed: Mo-O (505 K) and Mo-N (441 K), the first one is due the stress induced ordering of O atoms around the Mo atoms of the metallic matrix and the second is due the stress induced ordering of N atoms around the Mo atoms of the metallic matrix. The amount of interstitials present in the alloy can be estimated from the Q^{-1} peak heights; a clear predominance of nitrogen over oxygen is observed. An internal friction result measured with a frequency about 3.8 Hz (sample Mo38) as a function of T^{-1} is given in Fig. 2. The peaks observed reflect the same kinds of interaction with temperatures different from sample Mo15. The internal friction results measured with frequency about 6.0 Hz (sample Mo60) as a function of T^{-1} are given in Fig. 3. The peaks observed reflect at the same kinds of interaction, but the temperatures were different from those found in sample Mo15 and Mo38.

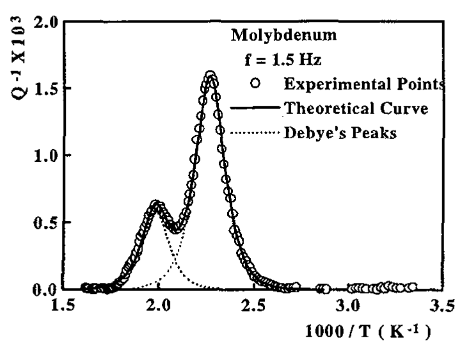

Figure 1. Internal Friction as a function of inverse of temperature for the Mo sample measured with 1.5 Hz.

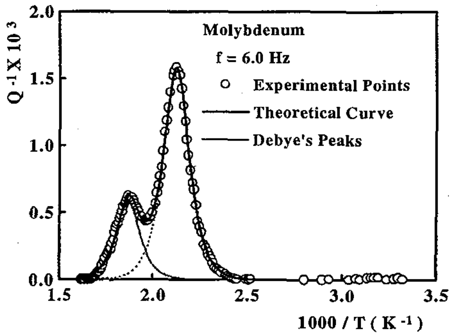

Figure 3. Internal Friction as a function of inverse of temperature for the Mo sample measured with 6.0 Hz.

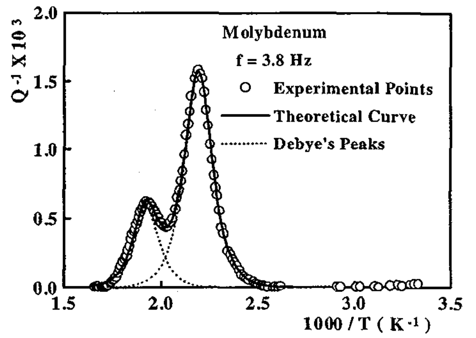

Figure 2. Internal Friction as a function of inverse of temperature for the Mo sample measured with 3.8 Hz.

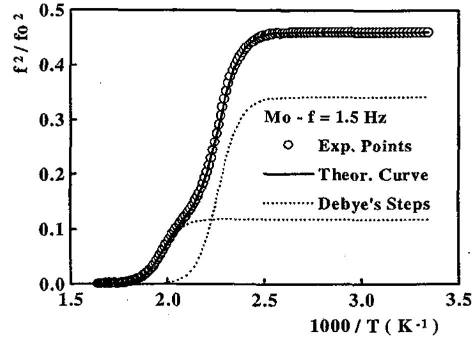

Figure 4. Relative square frequency as a function of inverse of temperature for the Mo sample measured with 1.5 Hz.

The square of the frequency, which is proportional to the elastic modulus as a function of T^{-1} is illustrated for samples Mo15, Mo38 and Mo60, in Figs. 4 , 5 and 6, respectively. These curves were analyzed by the same computer program as the one used above, yielding a family of step curves each one of which corresponded to a component peak in the internal friction spectrum.

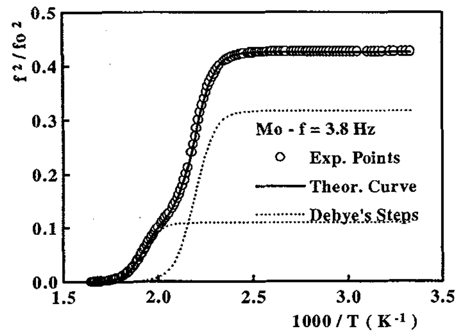

Figure 5. Relative square frequency as a function of inverse of temperature for the Mo sample measured with 3.8 Hz.

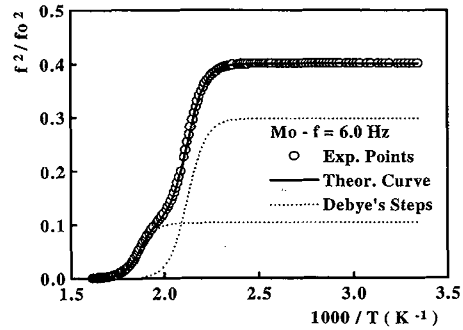

Figure 6. Relative square frequency as a function of inverse of temperature for the Mo sample measured with 6.0 Hz.

4. DISCUSSION

The theoretical fundation for anelastic relaxation is not the objective of this paper since it is clearly presented in the papers of Nowick [8-10]. In this case more than one relaxation process prevails so the internal friction is given by [8] :

$$Q^{-1} = \sum_{i=1}^{n} Q_{mi}^{-1} \,\mathrm{sec\,h}\left[\frac{E_i}{k}\left[\frac{1}{T_{mi}} - \frac{1}{T}\right]\right]$$

where: Q_{mi}^{-1} is the maximum internal friction for the peak corresponding to the i'th relaxation process; E_i is the activation energy of the i'th relaxation process and T_{mi} is the temperature at which the i'th relaxation process occurs.

The Kramers-Krönig relations [11] provide a means by which the frequency can be obtained from the internal friction data, and vice-versa, inasmuch as the frequency is proportional to the real part of the elastic constant and the internal friction is proportional to its imaginary part. Hence,

$$f^2 = \sum_{i=1}^{n}\left\{ f_{oi}^2 \left\{ 1 + \frac{Q_{mi}^{-1} T_{mi}}{T} \exp\left[\frac{E_i}{k}\left[\frac{1}{T_{mi}} - \frac{1}{T}\right]\right] \mathrm{sech}\left[\frac{E_i}{k}\left[\frac{1}{T_{mi}} - \frac{1}{T}\right]\right]\right\}\right\}$$

where: f_{oi} is the frequency at room temperature of relaxation process i.

There exists a relation between the Debye peaks with the internal friction curve and the step curves that make up the curve of the square of the frequency. This makes it possible to confirm the intensity of relaxation, which equals twice the value of the internal friction maximum and which also equals to the intensity of the steps of the curve of the square of the frequency. The relaxation parameters obtained by analyzing the curves shown in Figs. 1 through 6 are given in Table 1. The relaxation parameters are in excellent agreement with the data published by Yamane and Matsumoto [12], Haneczock et al. [13,14] and Kushnareva and Snejko [15], confirming the presence of relaxation processes that involved more than one type of interstitial.

5. CONCLUSIONS

1. Internal friction and frequency measurements were made for Molybdenum with oxygen and nitrogen in solid solution.
2. The multiple relaxation spectra were resolved into elementary peaks.

3. The relationship between the real part of the compliance, obtained by means of frequency measurements and the its imaginary part, obtained by means of internal friction measurements are confirmed. This relationship allows us to confirm that the observed peaks are really due to the anelastic relaxation induced by applied stress.
4. The relaxation parameters were calculated for each one of the identified processes obtained from the internal friction peaks and frequency curves.

Table 1. Relaxation parameters for the relaxation processes found in this work.

Process	Analysis	f (Hz)	T (K)	E (eV)	Reference
Mo-O	Internal Friction	1.5	506	1.35	this work
		3.8	521	1.35	this work
		6.0	537	1.35	this work
		1.5	489-522	1.345	[12]
		4.5	493	1.31	[15]
	Frequency	1.5	507	1.35	this work
		3.8	521	1.35	this work
		6.0	536	1.35	this work
Mo-N	Internal Friction	1.5	442	1.24	this work
		3.8	456	1.24	this work
		6.0	471	1.24	this work
		1.5	433-489	1.26	[12]
		1.0	438	1.25	[13]
		5.5	529	1.25	[14]
	Frequency	1.5	441	1.24	this work
		3.8	456	1.24	this work
		6.0	471	1.24	this work

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