

Analysis of Internal Friction Peaks in High Purity Molybdenum by a Viscoelastic Procedure Independent of the Relaxation Strength

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Abstract. In this work we present a novel procedure, involving linear viscoelastic analysis, to discriminate the two possible contributions of the observed damping peak which appears around 840 K – 1050 K in mechanically deformed high purity single-crystalline molybdenum. An interesting feature of the procedure is that, for low damping samples, it can efficiently resolve experimental peaks that result from the superposition of different processes independently of the ratio between their relaxation strengths. This allows us to confirm that two different relaxation processes appear in molybdenum in the temperature range about 0.3 T_m , one around 840 K, and the other one near 1050 K. These can be related to diffusion and to a coupled mechanism involving creation and diffusion of vacancies, respectively.

Introduction

Molybdenum, a group VI transition metal has a melting point of 2883 K, high specific heat, and good corrosion and creep resistance. Among the metals useful for high-temperature applications, the melting point of molybdenum is exceeded only by tungsten and tantalum. Molybdenum is ductile at room temperature, with a brittle-ductile transition temperature significantly lower than that of tungsten. Molybdenum has also good strength at high temperatures, being lighter than tungsten and tantalum [1, 2]. These qualities make molybdenum attractive for the use in the nuclear industry [3 - 7].

Mechanical spectroscopy, referred to also as the internal friction method, is a non-destructive technique and is a fundamental tool for studying the movement of dislocations and their interaction with point defects [8, 9]. It involves the simultaneous measurement of the damping or internal friction, Q^{-1} , and the elastic modulus as a function of temperature.

We have reported recently that molybdenum exhibits a damping peak at about 840 K – 1050 K, which is developed in deformed samples after annealing at temperatures above that of the vacancy migration [10, 11]. The intensity of the damping peak depended on the degree of plastic deformation at room temperature, but it was not affected by a bias stress. Moreover, the peak temperature and activation energy of this relaxation process increased with the temperature of the previous annealing of the sample; and it was independent of the crystal orientation. For instance, the activation energy increases from 1.6 eV for peak temperature at around 840 K to 2.7 eV for a peak at 1000 K. Also, the shape of the peak when it appears at temperatures around 1000 K is markedly asymmetrical. It has been proposed that the vacancy-dislocation interaction mechanism controls this peak [10, 11].

The Modified Relaxation Time (MRT) function and its applications, derived from a general linear viscoelastic formalism, are a very useful tool to determine if more than one overlapping

relaxation processes are taking place. In fact, it is well known that relaxation processes involving dislocations and non-dilute concentration of point defects originate damping peaks that are wider in comparison with the Debye model. Also, in most cases the observed peaks are asymmetrical. The MRT function clearly describes the width and symmetry characteristics of relaxation processes, as it will be later shown.

In this work, the MRT is applied to the study of the relaxation damping peaks at high temperatures in deformed and deformed plus irradiated molybdenum. The dependence with temperature of experimental data from these relaxation processes is adequately described by a Havriliak-Negami (HN) function, and the MRT makes possible to find a relation between the parameters of the HN function and the activation energy of the process.

The analysis allows us to relate the relaxation peak appearing at temperatures below 900 K, to a physical mechanism involving vacancy-diffusion-controlled movement of dislocations. In contrast, when the peak appears at temperatures higher than 900 K, the damping is controlled by a coupled mechanism of diffusion and creation plus diffusion of vacancies in the dislocation line.

Theoretical Background

The dynamical response of a linear viscoelastic material is usually described in terms of the complex modulus G^* (or the complex compliance J^*) as functions of the circular frequency ω and the temperature T . The complex modulus is generally presented in terms of its real and imaginary parts, that is, $G^*(T, \omega) = G'(T, \omega) + i G''(T, \omega)$, where G' is the storage modulus, G'' is the loss modulus and i is the imaginary unit [12].

The internal friction (or loss tangent) Q^{-1} is defined as the quotient between the imaginary and real part of the complex modulus.

$$Q^{-1}(T, \omega) = \frac{G''(T, \omega)}{G'(T, \omega)}. \quad (1)$$

It is useful to define a dimensionless magnitude Δ , usually called the *relaxation strength*, in terms of the characteristic parameters of the modulus:

$$\Delta(T) = \frac{\delta G(T)}{G_r(T)} = \frac{G_u(T) - G_r(T)}{G_r(T)}, \quad (2)$$

where the zero-frequency limit $G_r(T) = G^*(T, \omega = 0)$ is the called *relaxed modulus*, and the high-frequency limit $G_u(T) = G^*(T, \omega \rightarrow \infty)$ is the *unrelaxed modulus*.

The Debye function, a process characterized by an single relaxation time, $\tau_D(T)$, is often employed to describe relaxation processes; due to its conceptual simplicity. In this case, the internal friction is written as

$$Q^{-1}(T, \omega) = \frac{\Delta(T)}{\sqrt{1 + \Delta(T)}} \frac{\omega \tau_D(T)}{1 + (\omega \tau_D(T))^2}. \quad (3)$$

On the other hand, if the process cannot be represented by a single relaxation time, different approaches have been developed for analyzing the problem [9, 12]. This work presents a different, novel way to represent the loss tangent and, therefore, re-analyze the magnitude and its characteristic parameters.

The Modified Relaxation Time (MRT) function. The internal friction represented by Eq. 3 can be written in a different manner. In previous works [13, 14] it has been rigorously demonstrated that the dependence of Q^{-1} on T and ω can be expressed in terms of two functions, $\Lambda(T)$ and $\tau_i(T, \omega)$ as follows:

$$Q^{-1}(T, \omega) = \Lambda(T) \frac{\omega \tau_i(T, \omega)}{1 + [\omega \tau_i(T, \omega)]^2}, \quad (4)$$

where $\Lambda(T)$ is the envelope function and $\tau_i(T, \omega)$ is the *modified relaxation time function (MRT function)*, formerly called the integrated distribution function in [13, 14]. The function τ_i represents a local perturbation of the Debye process in the (T, ω) domain, and its frequency dependence is critically related to the existence of multiple relaxation processes, that is, a relaxation times distribution function. For the case of only one relaxation time, the MRT function is reduced to $\tau_D T$ and the envelope function is the pre-factor in Eq. 3.

In a measurement of internal friction as a function of frequency, at constant temperature, which usually gives a single-peaked function, $\Lambda(T)$ represents simply the double of the peak value, since the loss tangent takes its maximum value at the frequency ω_m where $\omega_m \tau_i(T, \omega_m) = 1$. On the other hand, in a measurement of internal friction as a function of temperature at constant frequency, $\Lambda(T)$ represents the envelope of the family of Q^{-1} vs. T curves, since $Q^{-1}(T, \omega) \leq \Lambda(T)/2$ for all ω [13].

Analysis of Thermally Activated Processes Using the MRT

The MRT function can be used to analyse the relaxation distribution involved in thermally activated processes. Usually, the shift of the peak temperature in the internal friction peaks with frequency has been widely used to determine the activation energy in this kind of processes [8, 9]. In fact, assuming that the characteristic relaxation time of the present distribution function depends exponentially on temperature as

$$\tau(T) = \tau_0 \exp(H / kT) \quad (5)$$

and at the peak temperature is such that $\omega\tau(T) = 1$, the mean activation energy, H , can be obtained from a linear regression between the measurement frequency at the peak temperature and the peak temperature. This is the so called Arrhenius plot [8, 9]. In Eq. (5) k is the Boltzmann constant and τ_0 is the mean pre-exponential factor. However, this procedure does not give any information about the distribution function that describes the internal friction peak, and in particular, whether there are or not several closely spaced relaxation times around the principal one.

In order to show the advantages of the MRT formalism the Havriliak-Negami (HN) parametrical expression for dynamical modulus will be used. They are defined by [15, 16]

$$G^*(T, \omega) = G_u(T) - \frac{\delta G(T)}{\left[1 + (i\omega \tau(T))^{\alpha(T)}\right]^{\beta(T)}}. \quad (6)$$

This function has been used to generate different Q^{-1} vs. T curves, for several frequencies and relaxation strength values. The values of α and β are the characteristic parameters of HN function, while relaxation time $\tau(T)$ has been simulated by using Eq. (5) for different values of H . α and β are phenomenological parameters that describe the symmetrical and asymmetrical broadening of the peak, respectively. In fact, these behaviours are related to the loss modulus (the imaginary part), which could lead to slightly differences in Q^{-1} . For simplicity, α , β and Δ will be considered independent of temperature in the following discussion. In addition, it is convenient to mention that α and β are introduced as phenomenological parameters to describe the broadening of the relaxation peak (in comparison to the Debye model) and in consequence do not have, up to the present, a clear physical interpretation.

Fig. 1 shows a few typical examples of internal friction peaks and the corresponding MRT functions vs. temperature curves. In all cases, even though the relaxation strength, Δ , varies over three orders of magnitude, it is evident that the MRT functions are independent of Δ .

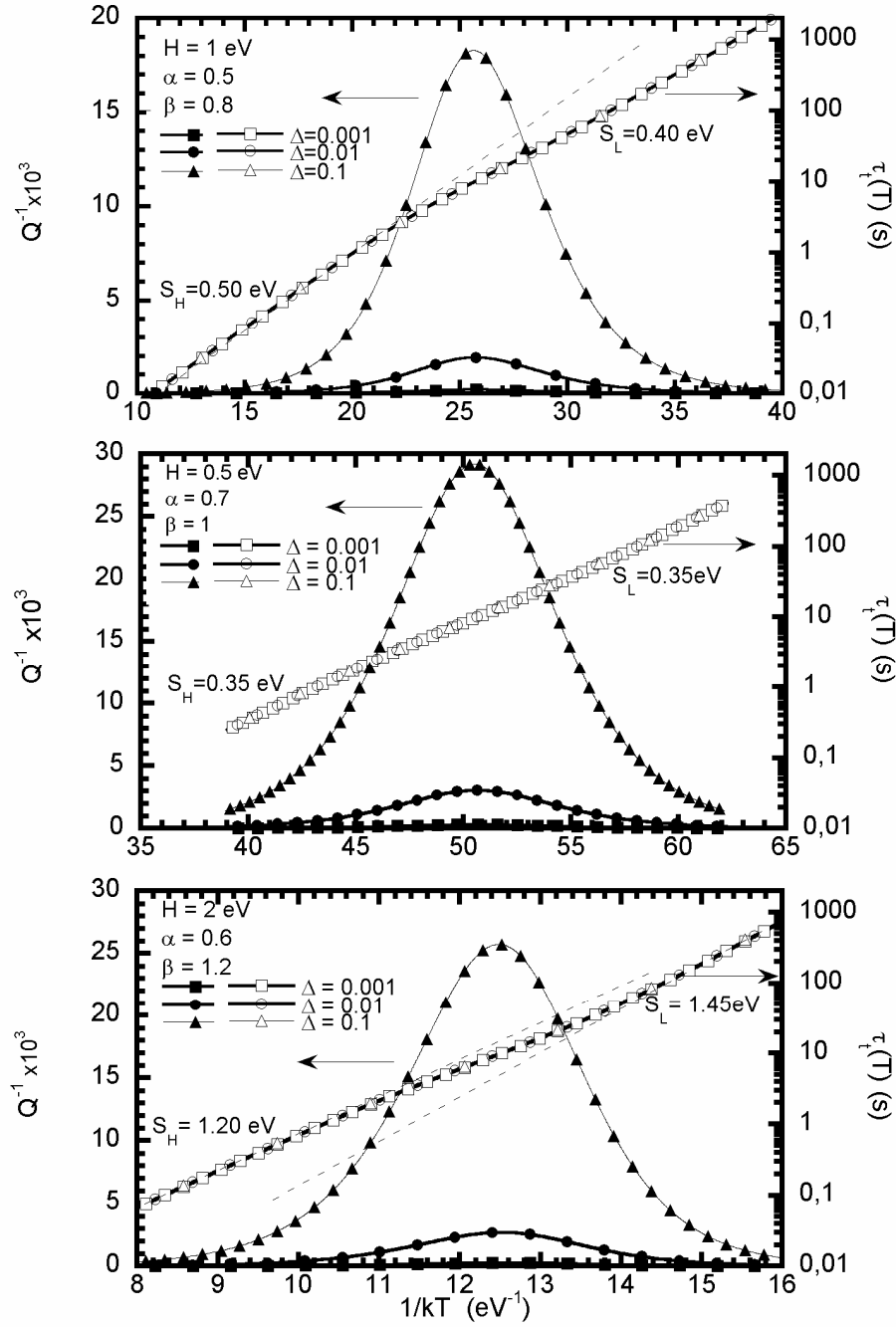


Fig. 1. Internal friction peaks (full symbols) and MRT functions (empty symbols) for the case of a HN function, with different relaxation strengths.

Also, it is important to note that the MRT functions show a linear behaviour on both sides of the peak, but the slopes of the linear sections on each side of the peak have a different dependence on the activation energy and on the parameters of the HN function. On the high temperature side, the slope of the MRT function, S_H is found to be very close to

$$S_H = \alpha H \quad (7)$$

and therefore it is independent of the value of β . In addition, on the low temperature side, the slope S_L is found to be nearly equal to

$$S_L = \alpha \beta H \quad (8)$$

Eqs. 7 and 8 have been verified through extensive numerical computations spanning all the physically meaningful range of the variables α , β , Δ and H . This remarkable, novel result

highlights the usefulness of the MRT formalism for the analysis of experimental data of relaxation processes.

When β is different from 1, there is a change in the slope of the MRT function, and the quotient between S_L and S_H is directly the value of the parameter β . Moreover, both slopes are proportional to the activation energy H . In the Debye case, that is, when $\alpha = 1$ and $\beta = 1$, the function is a straight line and its slope is directly the value of H , as it could be expected.

It should be emphasized that in the novel procedure described above for the analysis of relaxation processes, the Modified Relaxation Time (MRT) function depends only on the relaxation times distribution function, and in consequence it is independent of the relaxation strength and the relaxed modulus.

Moreover, for processes described by the Havriliak-Negami function, the slope of the MRT, as a function of temperature at a given frequency, is proportional to the shape parameters, α and β , and the activation energy H . Therefore, the graphical representation of the MRT makes much easier identify the characteristic parameters related to the shape and symmetry of the distribution function of relaxation times involved in the process.

Experimental

Samples. The single crystals used in this work were prepared from zone refined single-crystal rods of molybdenum in A.E.R.E., Harwell, UK. The residual resistivity, RR, of the samples was about 8000, the main residual impurity being tungsten. Samples with $\langle 110 \rangle$ crystallographic tensile axis were annealed and then deformed in extension, followed by torsion at room temperature. Two types of deformed samples were studied: type I (elongation: 5 %, torsion: 1 %) and type II (elongation: 3 %, torsion: 1 %). Further experimental details are given in Ref. [11].

After the room temperature deformation some samples of type II were neutron irradiated. Low flux neutron irradiation were performed at room temperature, at the Siemens SUR 100 nuclear reactor, RA-4, of the National University of Rosario and National Atomic Energy Commission of Argentina. The RA-4 was operated at 0.7 W. The thermal- and fast-neutron fluxes were about 5.2×10^7 n/cm²s, and their energies were of 0.025 eV and 10 Mev, respectively. Samples were irradiated at two different doses, 64 Gy and 127 Gy, and in this work are indicated as samples b and c, respectively. A more detailed description of the neutron irradiation procedure is given elsewhere [17].

Mechanical Spectroscopy Measurements. Damping and natural frequency were measured in an inverted torsion pendulum, under a vacuum of about 10^{-5} Pa. The equipment can also apply a bias stress or “in situ” deformation. The maximum strain on the surface of the sample was 5×10^{-5} and the measurement frequency was around 1 Hz, except for the determination of the frequency dependence of the peak temperature. The heating and cooling rates employed in the test were 1 K/minute. A heating ramp and its corresponding cooling run will be called hereafter a thermal cycle. There was no hold time once the maximum temperature had been achieved [11].

Results and Discussion

Fig. 2 shows the damping peaks for the samples of type I and II, after background subtraction [18]. Curves A, B and C correspond to damping peaks obtained from a stabilised damping spectrum measured in a type II sample at a natural frequency of about 0.2 Hz, for thermal cycles with maximum temperatures of 1040 K, 1100 K and 1155 K, respectively; see reference [11]. The resulting peak temperatures were around 900K, 940 K and 960 K, respectively. Peaks labelled D and E correspond to the sample of type I after stabilization during thermal cycles up to temperatures of 1050 K and 1230 K, respectively. The resulting peak temperatures were 817 K and 949 K, respectively. In this figure the relaxation peaks measured in $\langle 110 \rangle$ samples have been summarized, in order to study the response of the MRT formalism, see for more details Ref. [18].

The calculated curves of the logarithm of the MRT as a function of $1/kT$ (that is, $\ln(\tau_i(\omega, T))$ vs. $1/kT$), corresponding to peaks in Fig. 2 are given in Fig. 3. For the sake of clarity, only part of the

points calculated with the MRT function have been plotted. For the same reason the curve corresponding to the peak B has not been included, but it has a similar behaviour to the plotted curves, E and C. Vertical arrows in the figure indicate the temperature T_p corresponding to the maximum value of damping (Q_p^{-1}). S_H and S_L indicate the slope at high temperature and low temperature of the fitted straight lines (full lines), as defined in Eqs. 7 and 8.

It has to be remarked that for curves A and D two linear zones with the same slope were found in the MRT plot, in spite of the scatter of the calculated data. For temperatures far from the peak temperature the linearity is lost, as it could be expected due to the uncertainties introduced in the data by the background subtraction procedure. It was shown in the theoretical background section that the distribution function of relaxation times should be symmetrical for this kind of behaviour of the MRT. In contrast, for spectra B (not shown in Fig. 3), C and E two clearly linear zones with different slopes were found. The behaviour of the MRT function indicates that these relaxation peaks should be asymmetrical, in agreement with the experimental results. Therefore, for these peaks it can be proposed that at least two overlapped relaxations occur; where each one can be described through its corresponding distribution function of relaxation times. It should be emphasized that, as it was indicated in the theoretical background section, no assumptions or restrictions were made about the shape of the distribution function of relaxation times for the calculation of the MRT curves plotted in Fig. 3.

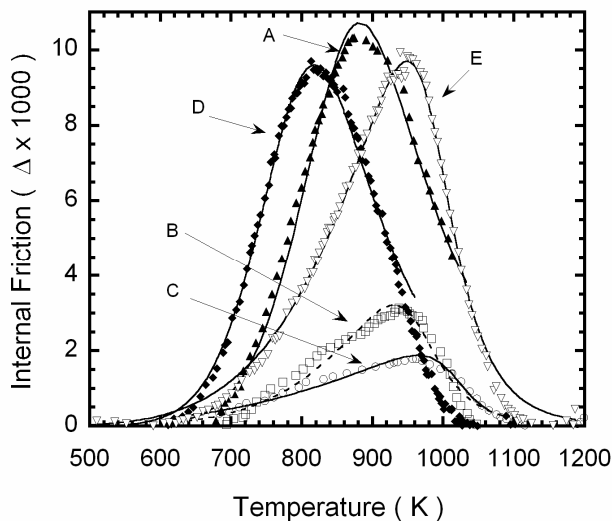


Fig. 2. Internal friction peaks after background subtraction (symbols) for the molybdenum samples. Spectra A, B and C: type II. Spectra D and E: type I. Full lines represent the numerical fitted peaks.

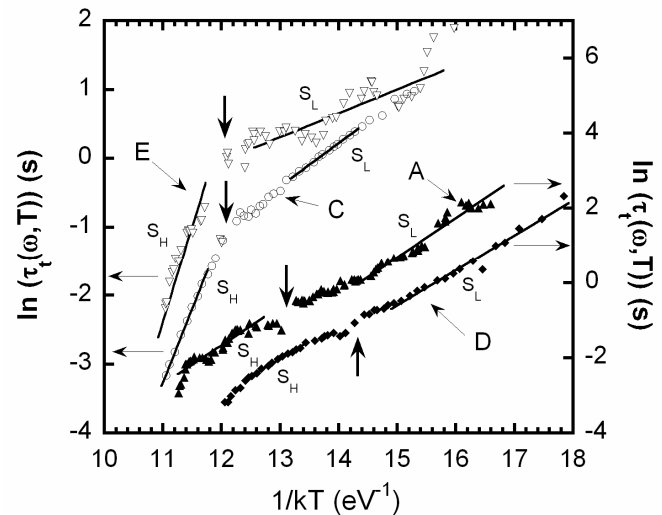


Fig. 3. Natural logarithm of MRT as a function of $1/kT$ for the peaks plotted in Fig. 2. Vertical arrows indicate the position of the peaks on the $1/kT$ axis. Full lines, S_H and S_L are defined in the text.

From the slopes of the straight segments fitted to the $\ln(MRT)$ vs. $1/kT$ curves (full lines in Fig. 3), the parameters of the HN distribution function can be obtained by means of Eqs. 7 and 8. Table 1 gives the calculated parameters for the HN function together with relevant experimental information related to each damping peak.

The damping peaks calculated theoretically by means of Eqs. 1 and 6 using the fitted parameters of the HN function are also shown in Fig. 2 by means of full lines. The activation energy for the calculation of Eq. 6, using Eq. 5, is the value that we obtained previously from the usual Arrhenius procedure, see ref. [11] and Table 1. The use of this value of the activation energy for the HN function is supported by the fact that, even if there is a distribution of relaxation times, the experimentally measured $\tau(T)$ (Eq. 5) corresponds to the mean value of the distribution function. The peak temperature and peak height for the calculated relaxations are also listed in Table 1.

Table 1. AT: annealing temperature. Q_p^{-1} : damping value at the maximum. T_p : temperature for the Q_p^{-1} . α and β : parameters of the HN function. H : activation energy, taken from Ref. [11].

Peak name / sample type	AT (K)	T_p (K)	$Q_p^{-1} \times 10^3$	H [eV]	α	β	Calculated	
							T_p (K)	$Q_p^{-1} \times 10^3$
A / II	1040	880	10.3	1.8	0.58	$\rightarrow 1$	880	10.7
B / II	1100	940	3.0	2.0	$\rightarrow 1$	0.3	935	3.2
C / II	1155	960	1.7	2.75	$\rightarrow 1$	0.12	960	1.9
D / I	1040	817	9.5	1.6	0.6	$\rightarrow 1$	817	9.6
E / I	1230	949	9.8	2.1	$\rightarrow 1$	0.25	949	9.8
A / b (irradiated 64 Gy)	973	615	4.5	1.6 (*)	0.33	$\rightarrow 1$	615	4.5
B / b (irradiated 64 Gy)	1230	972	2.8	2 (*)	$\rightarrow 1$	0.31	980	2.5
A / c (irradiated 127 Gy)	973	695	3.6	1.7 (*)	0.39	$\rightarrow 1$	695	3.6
B / c (irradiated 127 Gy)	1230	842	3.7	1.7 (*)	0.50	0.54	872	3.7
		929	3.7					
C / c (irradiated 127 Gy)	1230	1067	1.7	3 (*)	$\rightarrow 1$	0.32	1060	1.6

(*) calculated value from the fitting of the HN function (see the text).

As it can be seen from Fig. 2, the agreement between the experimental and calculated peaks is good, indicating that the calculated parameters of the HN function (α and β) are appropriate for describing the relaxation peaks in molybdenum at temperatures about 0.3 Tm. Therefore, from the study of the behaviour of α and β parameters as a function of temperature valuable information can be obtained about the physical process controlling the relaxation peaks.

As it can be noted from Table 1 the peaks A and D were mainly symmetrical ($\beta \rightarrow 1$) but broader ($\alpha < 1$) than a Debye peak, in agreement with previous works [10, 11], where the half-width of the peaks was evaluated by means of the traditional analysis [8, 9]. Indeed, the peaks which appear at temperatures well below 900 K, obtained during annealing up to temperatures smaller than about 1100 K, can be described by a symmetrical distribution function of relaxation times, with an average activation energy represented by the value obtained from the Arrhenius plot. In addition, a good agreement was also found for relaxation peaks during thermal cycles up to 973 K in type I samples with a Q_p^{-1} of about 20×10^{-3} . This indicates that the same physical mechanism occurs when the sample is heated below 1100 K [18].

For peaks B, C and E (with $T_p > 900$ K), which correspond to samples heated above 1100 K, the α and β parameters are: $\beta < 1$ and $\alpha \rightarrow 1$, revealing that the peaks are asymmetrically broadened. This means that there is an overlapping of relaxation processes leading to an asymmetrical broadening of the peak. It must be remembered that α and β are the phenomenological parameters that describe the symmetrical and asymmetrical broadening of the peak, respectively, and in consequence, if $\beta \neq 1$, the limit $\alpha \rightarrow 1$ does not lead to a Debye peak.

Moreover, C and E peaks, which have close peak temperature (≈ 950 K) and very different relaxation intensities (by a factor about 5), exhibit similar α and β parameters, indicating an effect only on the relaxation strength, in agreement with the explanation given above for the peak at lower temperature.

However, the physical mechanism controlling these asymmetrical peaks at higher temperatures is different to the physical mechanism giving rise to the symmetrical peaks found at lower temperatures. The change in the α and β parameters, depending of the peak temperature, (see Table 1) leads to the conclusion that there exist two relaxation processes in molybdenum in the temperature range of 0.3Tm. The first relaxation process is related to the symmetrical peak which

appears between 820 K - 900 K, and the other to the asymmetrical peak which appears at higher temperatures, above 950 K [18].

The behaviour of the damping peaks and the MRT curves measured in irradiated samples are in good agreement with the analysis given above. Figures 4 and 5 show the damping peaks for samples (b) and (c) after background subtraction, after some thermal cycles up to 973 K. In this case, during the cooling part of the first thermal cycle, after heating to 973 K, the damping peaks appear at lower temperature and have smaller intensity than the one for non-irradiated samples, as a consequence of the excess of vacancies, out of thermodynamic equilibrium, promoted by the neutron irradiation [17]. Successive thermal cycles up to 973 K did not modify the peak temperature and intensity of the peaks (see spectra A in the Figures).

After the first thermal cycle up to 1230 K a wide peak appears within the temperature interval 600 K – 1100 K, which is composed by more than one elementary peak. This situation is shown in the spectrum B for the sample (c) in Fig. 5. Indeed, the extra amount of vacancies produced by irradiation in the deformed samples makes it possible, at least once, to observe the two relaxations in the same spectrum [17].

Subsequent thermal cycles up to 1230 K lead to a strong shift in the peak temperature and to a decrease in the peak height, in a similar fashion as the non-irradiated sample (see peak marked B in Fig. 4 and peak C in Fig. 5).

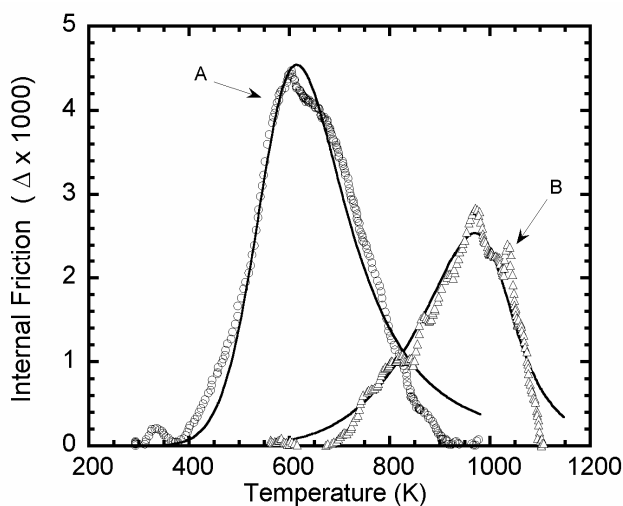


Fig. 4. Internal friction peaks after background subtraction (symbols) for molybdenum (b) samples (deformed and irradiated 64 Gy). A: After thermal cycles up to 973K. B: After several thermal cycles up to 1230K. Full lines represent the numerically fitted peaks.

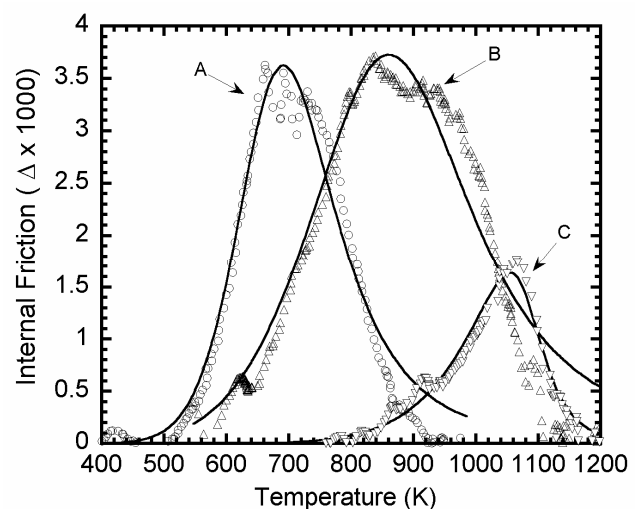


Fig. 5. Internal friction peaks after background subtraction (symbols) for the molybdenum (c) samples (deformed and irradiated 127 Gy). A: After a thermal cycles up to 973K. B: After first heating up to 1230K. C: After several thermal cycles up to 1230K. Full lines represent the numerically fitted peaks.

The behaviour of the calculated MRT values for the peaks plotted in Figures 4 and 5 is shown in Figures 6 and 7. As it can be inferred from these Figures, MRT results are in agreement with experimental data. This clearly shows that, for peaks measured after annealing at temperatures higher than 1100 K, the relaxation process must be described by more than a single relaxation time; see also Table 1. In fact, curves B for sample (b) (Figure 6) and curves B and C for sample (c) (Figure 7) have two linear zones, indicated as above by S_L and S_H , with markedly different slopes. In contrast, peaks obtained from thermal cycles performed up to temperatures lower than 1100 K exhibited the same slope in the straight lines S_L and S_H .

It should be also mentioned that in irradiated samples the activation energy values given in Table 1, were also determined from the numerical procedure of fitting the HN function. Despite the scatter in the obtained values, the agreement is fairly good.

Finally, full lines in Figures 4 and 5 are the calculated peaks using the parameters determined through the MRT function, as it was done above for deformed samples. The agreement between experimental and theoretical results is good.

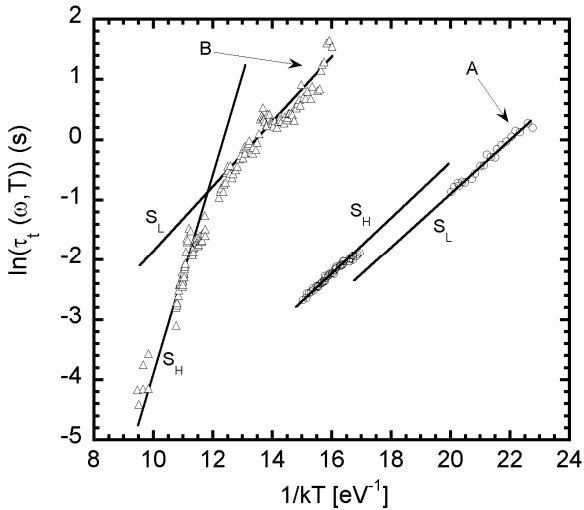


Fig. 6. Natural logarithm of MRT as a function of $1/kT$ for the peaks plotted in Fig.4. Full lines, S_H and S_L are defined in the text.

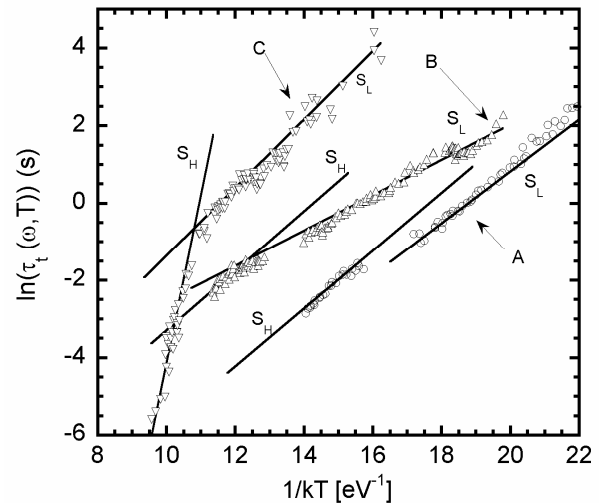


Fig. 7. Natural logarithm of MRT as a function of $1/kT$ for the peaks plotted in Fig.5. Full lines, S_H and S_L are defined in the text.

In order to relate the information obtained from the MRT function with the physical driving force which gives rise to the damping peaks discussed above, the following facts must be taken into account. The low temperature peak is developed both in deformed and deformed plus irradiated samples after annealing at temperatures above that of vacancy diffusion, and the higher temperature peak appears at temperatures near to $0.3 T_m$ [10, 11, 17, 18]. In addition, the measured activation energy for the lower temperature peak is 1.6 eV [11]. Therefore, this peak can be related to the dragging of vacancies by the dislocation line controlled by a diffusion mechanism. In contrast, when the peak appears at higher temperature, results show that it is controlled by another mechanism. This mechanism is consistent with the creation and diffusion of vacancies in the dislocation line, in agreement with the activation energy measured for the high temperature peak (2.7 eV). In addition, it has been proposed that the high temperature peak is involving both diffusion and creation plus diffusion of vacancies in the dislocation line [17, 18].

Conclusions

A novel method has been presented to analyze relaxation processes in terms of the Modified Relaxation Time function (MRT). The slope of the MRT when applied to processes described by the Havriliak-Negami function, as a function of temperature, is shown to be proportional to the shape parameters, α and β and the activation energy H . Also, the MRT is independent of the relaxation strength and the unrelaxed modulus.

The Modified Relaxation Time function procedure was applied to a Havriliak-Negami fit of experimental results from mechanical spectroscopy in high purity single-crystalline molybdenum, with good results. The analysis indicates that the distribution of relaxation times is symmetrical for peaks below 900K and asymmetrical at higher temperatures. The lower temperature relaxation was related to vacancy-diffusion-controlled movement of dislocations and the higher temperature peak was related to a process controlled by both the diffusion and the creation plus diffusion of vacancies in the dislocation line.

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