

Tunneling of H and D trapped by O(N) in niobium by anelastic relaxation measurements

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Deuterium trapped by oxygen (nitrogen) in niobium gives rise to two anelastic relaxation processes at 1.5 and 4 K at 20 kHz. The process at higher temperature and the analogous one observed in Nb-O(N)-H have been interpreted in terms of a two-level tunneling system (TLS) interacting with phonons or electrons. Two-phonon and electron interactions in the superconducting state give nearly equivalent fits to the data. In both cases the tunneling matrix element is found to be about 0.9 meV without appreciable isotope effect; the coupling parameters of the TLS to electrons and phonons have also been estimated.

I. INTRODUCTION

The recent extension below 4 K of the measurements of the thermal conductivity,¹ specific heat,²⁻⁵ ultrasonic attenuation,⁶⁻⁹ internal friction,¹⁰⁻¹² neutron spectra¹³ and sound velocity¹⁴ in metal-hydrogen (deuterium) systems has displayed evidence for the tunneling of hydrogen at very low temperatures. Hydrogen is confined around heavier interstitial or substitutional impurities, which act as trapping centers inhibiting its precipitation, and it is accepted that it is delocalized in tunnel systems constituted by trapping sites coordinated with the impurity trap. At present there is disagreement on the configuration of the tunnel systems. It was first proposed that a tunnel system in Nb-O(N)-H(D) is constituted by a ring of four tetrahedral sites near the O(N) atom,^{6,7} and transitions between the different levels occur via two-phonon processes. In contrast, it has been pointed out¹⁵ that such a geometry is unlikely to form a four-level system due to the large difference between the site energies (100 meV) with respect to expected splitting (1 meV). The authors of Refs. 13 and 15 propose a two-level system (TLS) in which H tunnels between two equivalent sites symmetrically arranged around the oxygen trapping center. Subsequent ultrasonic attenuation measurements⁸ have shown that the relaxation peak disappears upon application of a magnetic field which destroys the superconducting state; the authors conclude that electronic interactions are operative and directly influence the relaxation rates of the TLS.

In view of the existing controversy and the lack of data at low frequency, the present authors have extended the experiments on the superconducting Nb-O(N)-H(D) sys-

tems in the kHz range; the results have been analyzed and compared with existing data.

II. EXPERIMENTAL AND RESULTS

The specimen was a polycrystalline disc (30 mm in diameter and 2.5 mm thick) of 99.9% pure Nb supplied by MRC, containing 0.13 at. % O and 0.015 at. % N, as estimated by the height of the Snoek peaks. Dislocation annealing was performed at 1500°C in a vacuum better than 10^{-7} mbar, and doping was made at 550°C in an atmosphere of 99.9% pure deuterium. The deuterium concentration c_D , as estimated by the weight variation both after doping and vacuum extraction, was 0.14 at. %.

The internal friction measurements in the deuterium-charged specimen were carried out at two vibration frequencies (20 and 73 kHz) from room temperature to 1.2 K. It was checked that deuteride precipitation never occurred, as it was expected, since $c_D = c_O + c_N$. The relaxation curves of the same hydrogenated specimen have already been reported in Ref. 11.

On cooling, a peak around 100 K was first observed (result not reported here), which is attributed to the stress-induced reorientation of D around O(N);¹⁶ a small maximum due to traces of hydrogen was superimposed to that peak. Below 8 K two more peaks were observed and labeled as *P1* and *P2* at lower and higher temperature, respectively. Figure 1 presents the experimental data and the estimated contribution of hydrogen (solid and dashed lines) which has been subsequently subtracted; the correction is not significant. The peak temperature rises with increasing frequency for both processes and correspondingly the height of *P1* remains unchanged, whereas that of *P2* markedly increases.

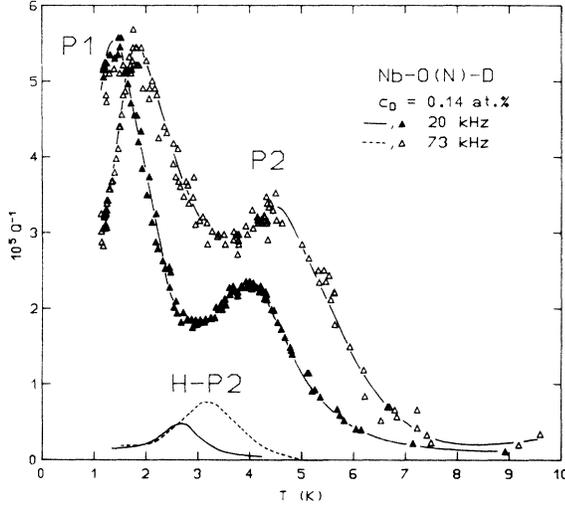


FIG. 1. Internal friction of Nb-O(N)-D at two vibration frequencies. The solid and dashed lines represent the estimated contribution of hydrogen traces which has been subsequently subtracted.

The relative variations of frequency referred to an arbitrary frequency are reported in Fig. 2. It is important to note that the relative frequency change associated with *P*2 is about equal to the value of the peak height, and this indicates that the process is characterized by nearly a single relaxation time. Instead, peak *P*1 does not seem to be single, as displayed by the magnitude of its modulus defect.

III. DISCUSSION

A. Anelastic relaxation due to a two-level system

The data presently obtained will be interpreted in terms of tunneling of hydrogen or deuterium in sites coordinated with the O(N) trapping centers. It is shown in the fol-

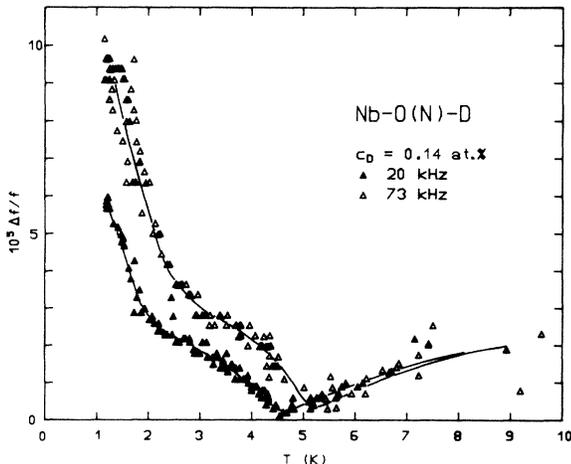


FIG. 2. Relative variation of the vibration frequencies referring to the same measurements of Fig. 1.

lowing that the data are adequately explained by a TLS, the theory of which has been developed for the amorphous materials without considering the particular tunneling species (for the theory of the TLS's see for example Ref. 17). The two levels may arise from two traps symmetrically arranged around oxygen, as proposed recently.^{13,15} The tunneling splits the energy levels of the two sites and the splitting is given by the tunneling matrix element Δ_0 . When interactions between the various defects are operative the two trap sites may differ in energy by Δ ; in this case the energy splitting E is given by:

$$E = (\Delta_0^2 + \Delta^2)^{1/2}.$$

The application of an external stress to the sample may change this splitting, giving rise to anelastic relaxation. In the theory of TLS it is found that the change in E with strain e is the following:

$$D = \frac{dE}{de} = \frac{\Delta_0}{E} \frac{d\Delta_0}{de} + \frac{\Delta}{E} \frac{d\Delta}{de} = 2 \left[\gamma_0 \frac{\Delta_0}{E} + \gamma \frac{\Delta}{E} \right]; \quad (1)$$

Eq. (1) implies that in the tunneling anelastic relaxation can occur even if the two site energies are equally perturbed by the external stress for symmetry reasons ($\gamma=0$). In fact, in this case the variation in E is supplied by Δ_0 , which does not require symmetry conditions to be influenced. On the contrary, in the classical case where $E=\Delta$ and $\Delta_0=0$, the nonequivalence of the sites with respect to the applied stress is a prerequisite for relaxation to occur. Generally it is assumed that γ_0 is negligible with respect to γ according to an argument which is not convincing because it seems to neglect the effect of strain on the height of the energy barrier between the two sites. Furthermore, it will be shown in subsection D that the assumption of $\gamma_0=0$ leads to inconsistencies with our results.

The relaxation strength of such a TLS is the following:

$$\delta(T) = \frac{nD^2}{4cE} \beta E \operatorname{sech}^2 \left(\frac{\beta E}{2} \right) = \delta_0 \beta E \operatorname{sech}^2 \left(\frac{\beta E}{2} \right), \quad (2)$$

where c is the stiffness, n the concentration of the TLS's, and $\beta=1/kT$. The relaxation peak is given by the Debye formula:

$$Q^{-1}(T) = \delta(T) \frac{\omega \tau}{1 + (\omega \tau)^2}, \quad (3)$$

being ω the angular frequency of vibration and $\tau(T)$ the relaxation time of the TLS. The relaxation time can be governed by interactions with phonons and/or electrons, and both cases will be discussed in what follows.

B. Interaction with phonons

For one-phonon transitions one obtains (see for example Ref. 17);

$$\begin{aligned} \tau_{\text{ph}}^{-1} &= \frac{2\pi}{\hbar} \sum_a |M_{12}^{(a)}|^2 \frac{E^3}{4\pi^2 \hbar^3 \rho v_a^5} \coth \left(\frac{\beta E}{2} \right) \\ &= \tau_0^{-1} \coth \left(\frac{\beta E}{2} \right), \end{aligned} \quad (4)$$

where the sum is extended over the three acoustic phonon modes, ρ is the density, v the sound velocity of the material, and $M_{12}^{(\alpha)}$ is the coupling of the TLS to the phonons of the α mode for a transition between the first two levels. Taking into account the variation of Δ_0 with strain, $M_{12}^{(\alpha)}$ is given by:

$$M_{12}^{(\alpha)} = \frac{\Delta}{E} \gamma^{(\alpha)} - \frac{\Delta_0}{E} \gamma_0^{(\alpha)}. \quad (5)$$

At high temperature $\tau_{1\text{ph}}^{-1}$ depends linearly on temperature.

For two-phonon transitions we can apply the results of Orbach¹⁸ who calculated the rate of phonon-assisted transitions for the spin-lattice relaxation in rare-earth salts:

$$\begin{aligned} \tau_{2\text{ph}}^{-1} = & \frac{2\pi}{\hbar} \sum_{\alpha\alpha'} \left| \sum_n \frac{M_{1n}^{(\alpha)} M_{n2}^{(\alpha')}}{E_n} \right|^2 \frac{(\beta/2)^{-7}}{2(4\pi^2 \hbar^3 \rho)^2 v_\alpha^5 v_{\alpha'}^5} \\ & \times \cosh \left[\frac{\beta E}{2} \right] \int_0^\infty dx \frac{x^3}{\sinh x} \\ & \times \frac{(x + \beta E/2)^3}{\sinh(x + \beta E/2)}, \end{aligned} \quad (6)$$

$$\tau_{2\text{ph}}^{-1} = \tau_0^{-1} (\beta E/2)^{-7} \cosh(\beta E/2) I(\beta E/2),$$

where $M_{1n}^{(\alpha)} M_{n2}^{(\alpha')}/E_n$ is the effective matrix element for a transition between the first two levels through a third state n of energy E_n . Equation (6) is obtained under the condition that E_n is greater than the Debye energy E_D of

$$\tau_{\text{el}}^{-1} = \frac{\pi}{4\hbar} |v_\perp n(\epsilon_F)|^2 \int d\epsilon_1 \int d\epsilon_2 g(\epsilon_1) g(\epsilon_2) \left[1 - \frac{\Delta_s^2}{\epsilon_1 \epsilon_2} \right] f(\epsilon_1) f(-\epsilon_2) [\delta(\epsilon_2 - \epsilon_1 - E) + \delta(\epsilon_2 - \epsilon_1 + E)], \quad (8)$$

where $\Delta_s(T)$ is the Bardeen-Cooper-Schrieffer (BCS) energy gap, $g(\epsilon) = |\epsilon| / (\epsilon^2 - \Delta_s^2)^{1/2}$ for $|\epsilon| > \Delta_s$ and $g(\epsilon) = 0$ otherwise is the density of states of the electrons (quasiparticles) in the superconducting state, $f(\epsilon) = 1 / (\epsilon \beta E + 1)$, and δ is the Dirac function. In the normal state, when $\Delta_s = 0$, Eq. (8) becomes Eq. (7), while for $\Delta_s \neq 0$ Eq. (8) has to be numerically evaluated.

The relaxation frequencies of the described phonon and electron processes normalized with respect to τ_0^{-1} , are plotted in Fig. 3; the relaxation rates used in Fig. 3 and in all the fits to our data have been numerically calculated from Eqs. (6)–(8) without any simplification. The curves for the phonon processes are valid for all values of E , while that for the electron interaction has been plotted for a TLS with an energy splitting $E/k_B = 10$ K in Nb [$T_c = 9.2$ K and $\Delta_s(0) = 17.7$ K]. In the normal state the latter curve coincides with the one-phonon curve and has a discontinuity when $2\Delta_s(T) = E$, because the creation and annihilation of quasiparticles by the TLS becomes impossible when $2\Delta_s > E$.

D. Analysis of the experimental data

In order to analyze $P2$, the contribution of $P1$ has been subtracted interpolating its experimental points by a De-

the crystal (24 meV for Nb). In a TLS like that supposed here the levels above the first two originate from the excited states of the Harmonic oscillator associated to the H(D) atom vibrating in one site. In niobium the energies of the excited sites of hydrogen and deuterium exceed those of the ground state of about 100 meV;¹⁹ therefore the condition $E_n > E_D$ is fully satisfied here. When βE tends to zero the relaxation frequency approaches:

$$\tau_{2\text{ph}}^{-1} \simeq 0.0437 \tau_0^{-1} (\beta E/2)^{-7}.$$

C. Interaction with electrons

The interaction with the conduction electrons is also possible;^{17,20} considering the inelastic scattering of the conduction electrons by the TLS, one obtains the following relaxation in the normal state:

$$\tau_{\text{el}}^{-1} = \frac{\pi}{4\hbar} |v_\perp n(\epsilon_F)|^2 E \coth \left[\frac{\beta E}{2} \right] = \tau_0^{-1} \coth \left[\frac{\beta E}{2} \right], \quad (7)$$

where v_\perp is the off-diagonal coupling of the TLS to electrons, and $n(\epsilon_F)$ is the electronic density of states at the Fermi level. It is worth noting that the temperature dependence is identical to that of the one-phonon process.

The extension to the case of a superconducting system has been worked out by Black and Fulde.²¹ The expression for the relaxation frequency is the following:

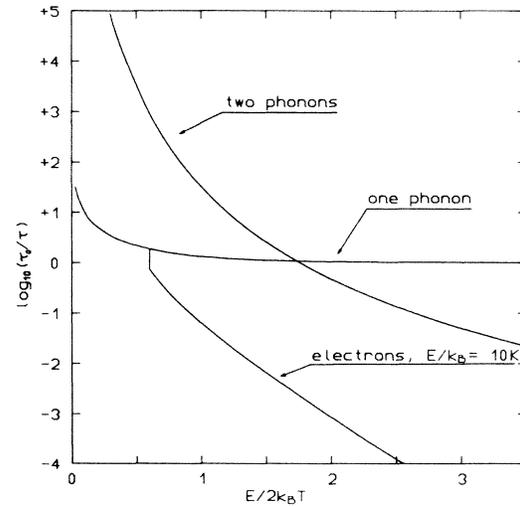


FIG. 3. Relaxation frequencies of a TLS vs $E/2k_B T$ assuming one-phonon, two-phonon, and electron interactions normalized with their respective τ_0 's. The curves for the phonon processes are valid for all values of the parameters, while that for electron transitions below the critical temperature are plotted for the case of a TLS with an energy splitting $E = 0.86$ meV ($E/k_B = 10$ K) in Nb ($T_c = 9.2$ K, $\Delta_s(0)/k_B = 17.7$ K).

bye function with a relaxation time following the law $\tau^{-1}=(T_M/T)^n$. The best fit resulted from the values $n=3.7$, $T_M=1.52$ K at 20 kHz and $n=3.0$, $T_M=1.85$ K at 73 kHz (solid lines of Fig. 4). At present we do not attribute any physical meaning to the function used to describe process *P1*. After subtraction, the experimental points for both frequencies have been plotted in Fig. 5. Figure 6 reports the experimental data (without any correction) of the analogous relaxation reported in the same specimen doped with H.¹¹ Attempts have been made to interpret peak *P2* in terms of one- or two-phonon processes or electron interactions. While one-phonon transitions [Eq. (4)] give curves exceedingly broad with respect to the measured peaks, two-phonon [Eq. (6)] and electron [Eq. (8)] interactions better fit the experimental data. From this fit the values of δ_0 , E , and τ_0^{-1} have been derived for both mechanisms, and the results are reported in Table I.

Peak *P2* seems to be a process with a single relaxation time, as already reported for H,¹¹ and this is confirmed by the magnitude of the modulus defect associated with the process (Sec. II). It means that all TLS's in the sample have nearly the same values of the parameters τ_0^{-1} and E . Consequently, the asymmetry Δ , which would be zero for noninteracting TLS's, must be negligible with respect to Δ_0 , and $E \cong \Delta_0$; thus the values reported in Table I are also indicative for Δ_0 .

Due to the large mass ratio, the values of the tunneling matrix element of D are expected to be remarkably lower than those of hydrogen.²² Instead it is seen from Table I that there is no appreciable isotope effect on Δ_0 , independently of the relaxation mechanism assumed; on the other hand, within the framework of the TLS model, the experimental data clearly show that E must have values close to those given in Table I because: (i) a lower limit of about 0.6 meV for E is imposed by the fact that the maximum of $\delta(T)$, which occurs at $k_B T=0.65E$ [Eq. (2)], must lie at a temperature higher than those of the peaks, since they grow with rising temperature at the frequencies tested; (ii) the values of the energy splitting in Table I can be considered as an upper limit because a higher value of E

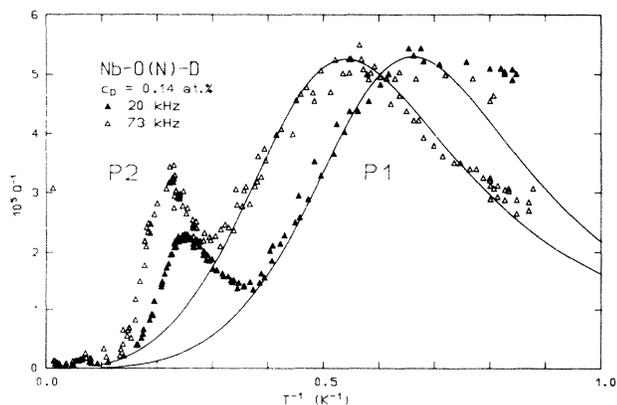


FIG. 4. Internal friction of Nb-O(N)-D after subtraction of the H contribution. The solid lines, which are fits to peak *P1*, have been subtracted to analyze peak *P2*.

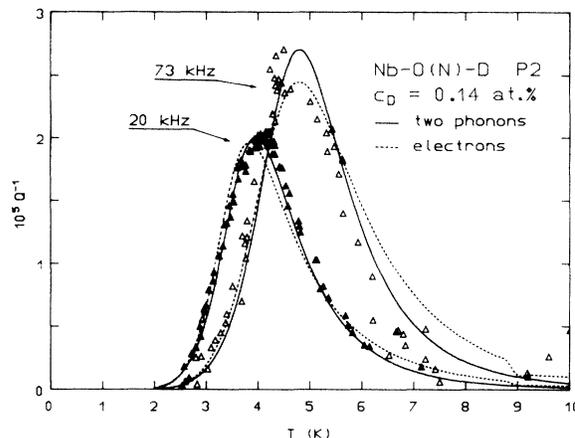


FIG. 5. Peak *P2* in Nb-O(N)-D after subtraction of the contribution of the H traces and of *P1*. The solid and dotted lines are the best fits assuming two-phonon and electron interactions; the values of the parameters used for the fit are given in Table I.

would further broaden the theoretical curves with respect to the experimental points (Figs. 5 and 6).

Apart from the isotope effect the reported values of Δ_0 are of the same order of magnitude as those theoretically estimated for the H tunneling in Nb,²² but remarkably different from those reported by specific heat and neutron scattering experiments^{5,13} and tentatively attributed to the mechanisms originating peak *P1*.⁸ Those measurements reveal the presence of TLS's with energy splitting of 0.19 and 0.02 meV in Nb-O-H and Nb-O-D, respectively. This suggests that there exist at least two different microscopic mechanisms giving rise to tunneling systems and in fact the relaxation spectrum displays two main peaks.

From the values of τ_0^{-1} of Table I it is possible to estimate the coupling parameters of the TLS to the phonons or electrons. In the case of electron interaction, using the definition of τ_0^{-1} given in Eq. (7), it is derived that

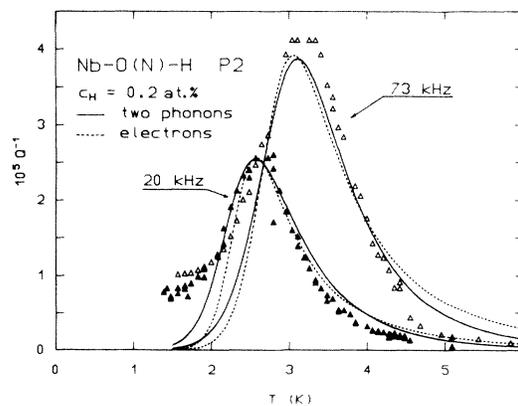


FIG. 6. Peak *P2* in Nb-O(N)-H (measurements of Ref. 11). The increase below 2 K is due to the process at lower temperature (*P1*). The solid and dotted lines are the best fits assuming two-phonon and electron interactions; the value of the parameters used for the fit are given in Table I.

TABLE I. Parameters of the TLS derived from the fit of peak *P2* assuming two-phonon or electron interactions.

	Deuterium				Hydrogen			
	δ_0 (10^{-5})	E (meV)	E/k_B (K)	τ_0^{-1} (Hz)	δ_0 (10^{-5})	E (meV)	E/k_B (K)	τ_0^{-1} (Hz)
Two phonons	8	1.0	12	7.0×10^4	14	0.78	9	1.9×10^5
Electrons	6	0.86	10	1.0×10^7	18	0.86	9	1.7×10^8

$v_{1n}(\epsilon_F) = 1.3 \times 10^{-2}$ and 3.1×10^{-3} for H and D, respectively. In metallic glasses, where the nature of the TLS's is still unknown, a typical value of $v_{1n}(\epsilon_F)$ is 0.1.²⁰

The case of two-phonon interactions is less straightforward because τ_0^{-1} contains a sum over the phonon modes, but taking into account the fact that ultrasonic measurements on Nb-O-H(D) single crystals show relaxation only in the c' mode^{6,7,9} (whose sound velocity is 2.6×10^5 cm/sec) and assuming that only the first pair of excited states ($n=3,4$) are involved as intermediate states, we have only two terms in the sum and obtain

$$\sum_{n=3}^4 M_{1n} M_{n2} / E_n \simeq 35 \text{ eV}$$

for H and 8 eV for D. Taking $E_{1n} = 0.1$ eV (Ref. 19) and assuming $M_{1n} = M_{n2}$ we get $M_{1n} \simeq 1.3$ eV for H and 0.63 eV for D.

In what follows we will show that the TLS model with the tunneling matrix element Δ_0 independent of strain ($\gamma_0=0$) is inconsistent with our data. In fact from the values of δ_0 it is possible to estimate D [Eq. (2)]; then assuming relaxation of only the c' mode and introducing in Eq. (2) a geometrical factor of value 0.5 to take into account that we have flexural vibrations of a polycrystalline plate, we find that $D=730$ K for H and 510 K for D. Now, if $\gamma_0=0$, it is necessary to introduce a distribution function for Δ which is so narrow not to affect the shape of the peak but yields a finite D [Eq. (1)]. If this distribution is a Gaussian of width σ_Δ , then $(\langle D^2 \rangle)^{1/2} = \sqrt{2} \gamma \sigma_\Delta / E$ and a lower limit for γ is imposed, because it must be $\sigma_\Delta \ll E = 10$ K; even choosing $\sigma_\Delta = 5$ K one obtains $\gamma \geq 800$ K. If this value of γ is introduced in Eqs. (4) and (5), one finds that the one-phonon relaxation would be the predominant one, with a transition rate ($\tau_{1ph}^{-1} > 5 \times 10^8 \text{ sec}^{-1}$) which is much higher than the vibration frequency of the specimen, and therefore is in contrast with the experimental relaxation curves. If, on the contrary, $\gamma_0 \neq 0$ the need of choosing high γ to obtain the correct relaxation strength is removed and there can be a partial cancellation of the contributions of γ and γ_0 in M_{12} [Eq. (4)], which further reduces the one-phonon relaxation rate.

Figure 7 displays the vibration frequencies as a function of the reciprocal peak temperature in hydrogenated and deuterated niobium from various contributors. The data from both the kHz and the MHz ranges display two peaks and it is likely that the peaks at higher frequency correspond to those at low frequency labeled as *P1* and *P2*. The process *P1* for H at low frequency is not reported in Fig. 7 because only its tail could be observed.¹¹ Two mechanisms have been proposed to explain *P1*: (i) an

Orbach-type two-phonon relaxation of the tunnel system constituted by the O trap and the H delocalized over four trap sites;⁷ (ii) a relaxation via conduction electrons of a two-site tunneling system;⁸ support to this hypothesis is considered the fact that the peak disappears when the sample is forced to the normal state by a magnetic field. It must be noted that the former mechanism is different from the two-phonon process considered here, because it has four low-energy levels originated from the ground states of the four sites; therefore the energy of the intermediate level E does not satisfy the relation $E_n > E_D$ and this leads to a different expression of τ^{-1} .

We tried to analyze our data of *P1* for deuterium and found that the peak is too narrow to be due to one-phonon transitions and too wide for two-phonon or electron relaxations. However, *P1* does not seem to arise from a single-time relaxation, as indicated by the magnitude of the modulus defect (see Sec. II). So, it cannot be excluded that two-phonon or electron interactions may adequately describe the experimental curves, if distributions of the values of the TLS parameters are considered.

The high-temperature side of Fig. 7 presents the data for H and D referring to *P2* by us and Ref. 9. In the same Fig. 7 are data that are also plotted on a relaxation process observed in deformed niobium and attributed to

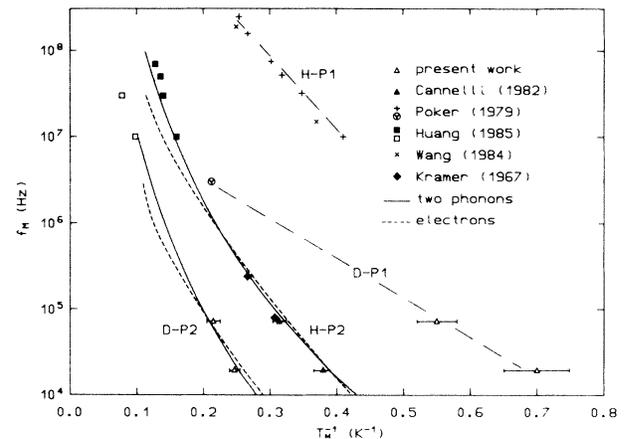


FIG. 7. Vibration frequency versus reciprocal maximum temperature of the peaks observed in Nb-O-(N)-H(D) by various authors at low temperature. The solid and dashed lines are the theoretical curves obtained assuming two-phonon and electron interactions of the TLS using the parameters of Table I. The dashed lines relative to H-P1 and D-P1 are only guides to the eye.

dislocations.²³ Because the features of that peak are strikingly similar to those of process *P2* reported for hydrogenated niobium,¹¹ it should be concluded that their samples contained unwanted quantities of hydrogen, and the peak is the same as *P2*. The same authors of Ref. 23 reported an abrupt shift of the peak to lower temperature under the application of a magnetic field establishing the mixed state.

The solid and dashed lines of Fig. 7 are the theoretical curves obtained assuming two-phonon and electron interactions of a TLS with the parameters of Table I. The curves relative to the electron interactions are interrupted near the critical temperature of Nb ($T_c = 9.2$ K) because the discontinuities of the relaxation time in that region introduce distortions in the internal friction curves and make the definition of the maximum temperature of the peak meaningless.

It must be noted that in Nb-O-D the two-phonon process cannot explain the shape of the peak measured by Huang *et al.*⁹ at 10 and 30 MHz, even if the corresponding theoretical curve in Fig. 7 satisfactorily fits their maximum temperatures.

IV. CONCLUSION

The Nb-O(N)-H(D) system displays two anelastic relaxation processes at liquid-helium temperature. The peak at higher temperature has been analyzed in terms of relaxation of a TLS via interactions with phonons or conduction electrons. The shape of the peak can be accounted for by two-phonon or electron interactions; in both cases the tunneling matrix element is estimated to be about 0.9 meV, without appreciable isotope effect. Although the two-phonon process gives a slightly better fit, it cannot explain the shape of the peak measured at 10 and 30 MHz in Ref. 9, which should correspond to our peak.

If the measured peaks are interpreted in terms of TLS, then the usual assumption of a tunneling matrix element independent of strain cannot be made, because the one-phonon relaxation rate deduced from the intensity of the peaks would be much higher than the observed relaxation frequency.

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