Deuterium motion in yttrium studied by ultrasonic measurements

R. G. Leisure

Department of Physics, Colorado State University, Fort Collins, Colorado 80523

R. B. Schwarz and A. Migliori

Los Alamos National Laboratory, Los Alamos, New Mexico 87545

D. R. Torgeson

Department of Physics, Iowa State University, Ames, Iowa 50011

I. Svare

Department of Physics, University of Trondheim, NTH, N-7034 Trondheim, Norway

I. S. Anderson

Institut Laue Langevin, Grenoble, France

Ultrasonic-attenuation measurements in $YD_{0.10}$ reveal broad, asymmetric attenuation peaks that are interpreted in terms of rapid motion of deuterium among interstitial sites in the Y metal lattice. The results are well described with a model involving tunneling among highly asymmetric sites. A weakly temperature-dependent relaxation rate, compatible with coupling of the sites to electrons, accounts for the relaxation up to about 70 K. At higher temperatures a strongly temperature-dependent relaxation rate becomes dominant. The major asymmetries that are responsible for the coupling of the tunneling centers to the ultrasound are attributed to the interaction between D atoms that lie on *different* c axes.

I. INTRODUCTION

The rare-earth–metal-hydrogen systems ($RH(D)_x$, $R = $ Y, Sc, Lu, Tm, Er, and Ho) have unusual thermodynamic, structural, and dynamical properties. Hydrogen (or deuterium) is retained in solid solution for x as high as 0.2–0.3, down to the lowest temperatures,[1] with no precipitation of the hydride phase. Neutron-scattering experiments[2] show that hydrogen isotopes occupy predominately the tetrahedral (T) sites in the hcp metal lattice with little occupancy of the octahedral (O) sites; moreover, hydrogen pairs are formed as the temperature is lowered. The pairs involve hydrogen occupancy of next-nearest-neighbor T sites along the c axis with a bridging metal atom between the hydrogens. In addition, the pairs forms chains[3,4] along the c axis, with some correlation between chains.[5,6]

The dynamical properties of this system are complex; diffusive motion has been observed on at least three different time scales. The relatively slow motion responsible for long-range diffusion involves T-O-T jumps with an activation energy of ≈ 0.6 eV. This motion has been studied by the Gorsky effect,[7] quasielastic neutron scattering,[8] and NMR.[9] Low-frequency (≈ 1 Hz) internal friction measurements[10] show a peak with approximately the same activation energy. A fast local motion, presumably involving motion between nearest-neighbor T sites, has been detected by NMR proton spin-lattice relaxation measurements[11] in ScH_x, with a slight indication of such a motion in YH_x. A very fast local motion between nearest T sites has been observed in YH_x (Ref. 12) and

ScH_x (Ref. 13) by quasielastic neutron scattering (QNS). The relation between the two types of local motion is unclear; they occur over the same temperature range, but the QNS derived rates are at least 100 times faster than the NMR derived rates. In addition, below 70 K the temperature dependences of the two rates are dramatically different, the neutron measured rate decreases with increasing temperature, while the NMR measured rate increases with increasing temperature. Finally, recent acoustic measurements[14] on YO_yH_x present evidence for a tunneling motion of H trapped near O.

In view of the rich variety of phenomena found in these $RH(D)_x$ systems and the possible fundamental role of quantum-mechanical effects in describing the motion, we have performed ultrasonic measurements on YD_x. According to the selection rules for anelasticity,[15] the symmetry of the T sites in the hcp lattice is such that an isolated hydrogen ion hopping between such sites will not give rise to ultrasonic attenuation. Hydrogen-hydrogen interactions will lower the site symmetry, however, and give rise to ultrasonic loss. Since such interactions are responsible for many of the interesting effects in these systems, it appears useful to use a probe dependent on these interactions. In addition, the frequency range of our measurements (≈ 1 MHz) is quite different from those described above, which may result in interesting phenomena being detected.

II. EXPERIMENTAL DETAILS

The single-crystal samples were grown at the Ames Laboratory, Iowa State University, from high-purity ma-

terials. Deuterium was loaded from the gas phase in the usual way.[8,16] Rectangular parallelepipeds, ≈ 2 mm on an edge, were cut from larger samples using an electric discharge machine. Vacuum fusion analysis of one of the YD_x crystals showed $x = 0.10$, and 260 ppm of N and 2890 ppm of O impurities. Whereas qualitative results were obtained on two different samples, the data reported below were obtained on a single, rectangular-parallelepiped sample of room-temperature dimensions 1.82×1.92 mm $\times 2.06$ mm. The long axis of the parallelepiped was parallel to the c axis to within $0.5°$, as determined by x-ray diffraction. Since a crystal of hexagonal symmetry is elastically isotropic in the basal plane, the orientations of the other crystalline axis were not determined relative to the parallelepiped axes.

Resonant ultrasound spectroscopy[17] was used to measure the ultrasonic-attenuation and elastic constants. With this technique a single-crystal, rectangular-parallelepiped sample is placed corner-to-corner between two piezoelectric transducers, one transducer being used for generation and the other for detection of ultrasonic vibrations. By sweeping the excitation frequency, a large number of the lowest vibrational eigenfrequencies can be investigated. The eigenfrequencies are related to the elastic constants, while the inverse of the Q of the resonances is related to the vibrational energy loss in the sample. Using the method of Visscher *et al.*,[18] we have identified the lowest 31 eigenfrequencies of one sample by fitting the measured frequencies to a set of elastic constants. This identification is needed for a microscopic interpretation of the results as discussed below.

III. RESULTS AND DISCUSSION

Figure 1(a) gives results for $1/Q$, for a single-crystal $YD_{0.10}$ sample, obtained at a frequency of 0.81 MHz, over the temperature range of 15–325 K. Three general features are apparent. The loss increases at the highest temperatures; this increase may be associated with the long-range diffusion process, or other processes discussed below. There is a small attenuation effect near 160 K, in the temperature range where resistance anomalies have been observed and associated with hydrogen ordering.[19] The attenuation anomalies in this temperature range were found to depend on the rate and direction of temperature change and have not been well characterized. The main effect, of course, is the large, broad, asymmetric attenuation peak with the maximum near 87 K. The solid line is a theoretical fit to the data to be described below. Figure 1(b) gives data similar to that of Fig. 1(a), but for a different vibrational eigenmode, the difference to be discussed below. The major difference between the data sets for the two eigenmodes is in the magnitude of the low-temperature peak. Figure 2 gives additional data at other frequencies for the low-temperature peak. A *temperature-independent* background $(1/Q)_{bg}$ was subtracted from the two highest-frequency curves before plotting the data in Fig. 2. $(1/Q)_{bg} = 0.00007$ and 0.0001 for the 1.4- and 2.7-MHz curves, respectively. Since the peak for the lowest frequency was much higher relative to the background, no
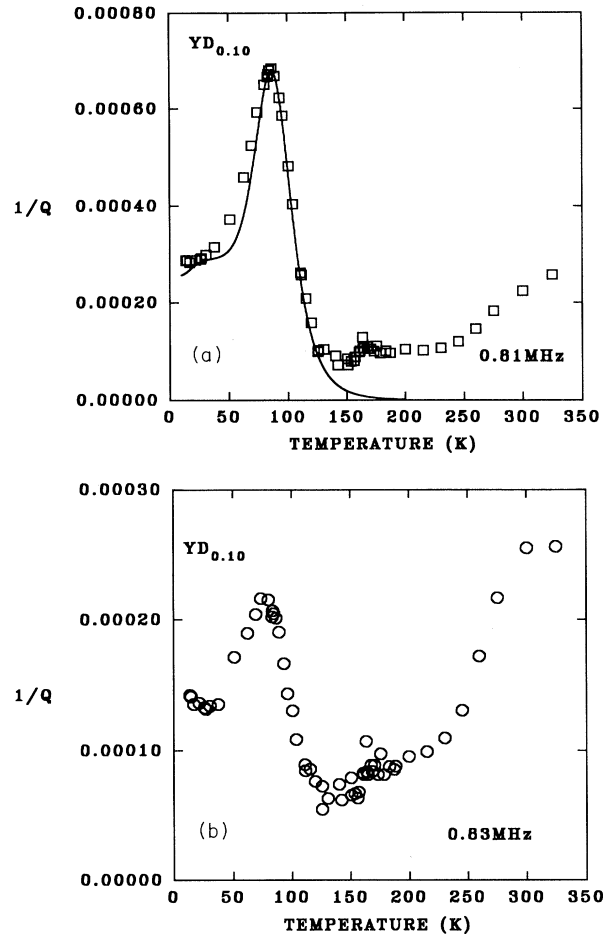

FIG. 1. (a) Ultrasonic loss, $1/Q$ vs temperature for $YD_{0.10}$ at a frequency of 0.81 MHz. The solid line represents a theoretical fit to the data described in the text. This vibrational eigenmode depends almost purely on C_{44}. (b) Ultrasonic loss, $1/Q$, vs temperature for $YD_{0.10}$ at a frequency of 0.83 MHz. This vibrational eigenmode depends almost purely on C_{66}.
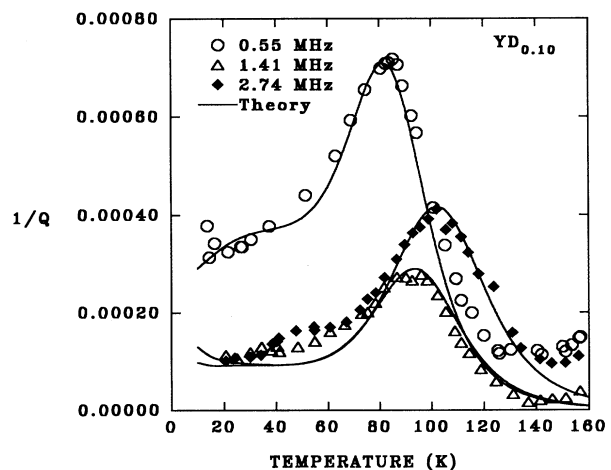

FIG. 2. Ultrasonic loss, $1/Q$, vs temperature at three different frequencies. The solid lines represent theoretical fits to the data described in the text.

background subtraction was performed. Neither was any background subtracted from the data of Fig. 1. Note that the peak positions shift to higher temperatures with increasing frequency.

We attribute the ultrasonic-attenuation peaks to relaxation attenuation due to the motion of hydrogen ions. Such motion, resulting in ultrasonic attenuation, has been described in terms of classical barrier hopping[20,21] in PdH_x and quantum-mechanical tunneling[22-24] for hydrogen trapped at impurities in Nb. An attempt was made to fit the data from this work with a classical, thermally activated relaxation time. The shift of the peak position with frequency, and the *high-temperature side* of the peak, could be fit reasonably well with an activation energy of 60 meV and an attempt frequency of 2×10^{10} s^{-1}. The low-temperature side of the peak did not fit well with this approach, even with a Gaussian distribution of activation energies. In any case, these values seem unphysical when compared with neutron-scattering measurements[25] of hydrogen vibrations in the T sites of Y: 60 meV is less than the energy of the first excited state for H vibration in the T site; and, 2×10^{10} s^{-1} is a factor of 10^3 lower than the optical frequency measured by neutron scattering. Such low apparent values of activation energies and attempt frequencies are often an indication of quantum-mechanical tunneling.[26] Thus, rather than use a description based on classical barrier hopping, which is in conflict with the neutron-scattering results, we turn to a picture based on quantum-mechanical tunneling as was done for the NMR spin-lattice relaxation data.[27]

Considering the neutron and NMR results, it seems likely that the low-temperature motion is due to movement between the two closely spaced T sites arranged in pairs along the c axis. The movement of hydrogen between nearby interstitial sites can be described as a two-level system[22-24] (TLS) with an energy splitting $E = (E_T^2 + \Delta^2)^{1/2}$, where E_T is the tunnel splitting and Δ is the difference in energy of the two wells (asymmetry). In principal Δ may include contributions from the different zero-point energies of the two wells if the wells have different shapes. In cases where tunneling is not a factor $E_T = 0$, but the model may still be used to describe the ultrasonic losses. The ultrasonic loss is given by[28]

$$\frac{1}{Q} = \frac{n_0 D^2}{4 C k_B T} \text{sech}^2 \left[\frac{E}{2 k_B T} \right] \frac{\omega \tau}{1 + \omega^2 \tau^2} , \qquad (1)$$

where n_0 is the concentration of TLS's, $D = \delta E / \delta \epsilon$ is the variation of the energy-level splitting with respect to the ultrasonic strain ϵ, C is an elastic constant, $\omega / 2\pi$ is the ultrasonic frequency, and τ is the relaxation time. It is usually the case[29] that $\delta \Delta / \delta \epsilon \gg \delta E_T / \delta \epsilon$ so that

$$D = \frac{\Delta}{E} \frac{\delta \Delta}{\delta \epsilon} = \gamma \frac{\Delta}{E} . \qquad (2)$$

In amorphous materials it is normally assumed that both Δ and E_T are characterized by distributions, but in the present case it may be reasonable to assume that E_T is single valued, since we are dealing with a crystalline material, while Δ is described by a distribution due to interactions with the neighboring hydrogen. Such a model

was used to describe the NMR spin-lattice relaxation in ScH_x (Ref. 27) and ultrasonic attenuation in the Nb-O-H system.[24] The asymmetric attenuation peaks in Figs. 1 and 2 appear similar to the asymmetric spin-lattice relaxation curves in ScH_x.[11] Thus, we are led to use Eq. (1) with a distribution of asymmetries.

Equation (1) must be integrated over the chosen distribution of asymmetries. The difficulty lies in not only finding the proper distribution, but in finding the proper expressions for the relaxation time, τ, which in general depends on the asymmetry. Many different expressions have been given for coupling of TLS's to electrons[30-34] and phonons.[23,27,35] In general the electron and one-phonon rate are slowly varying functions of temperature, while the multiple-phonon rates are much more strongly dependent on temperature. The low-temperature sides of the attenuation peaks in Figs. 1 and 2 appear to be due to a slowly varying relaxation time and perhaps also due to the thermal population of the energy levels having a splitting comparable to $k_B T$, while the shift of the peak position and the high-temperature side of the attenuation peaks appear to be due to a much more rapidly varying relaxation time.

The fits shown in Figs. 1 and 2 were obtained with a Lorentzian distribution of asymmetries. Gaussian distributions were also explored, but the fits were somewhat better with Lorentzian distributions. For relaxation due to the electrons we have used the expression[27]

$$\frac{1}{\tau_{\text{el}}} = \frac{\pi K E_T^2}{\hbar E} \coth \left[\frac{E}{2 k_B T} \right] , \qquad (3)$$

where K is a dimensionless parameter representing the coupling to the electrons. We have been unable to find a good fit to the data using the theoretical two-phonon relaxation rates. Thus, we have fit the data using a phenomenological expression for the high-temperature relaxation time. The expression we have used is

$$\frac{1}{\tau_p} = \frac{F T^n}{E^2} , \qquad (4)$$

where F represents the strength of the relaxation. The total relaxation rate used in Eq. (1) is just the sum of the two relaxation rates, $\tau^{-1} = \tau_{\text{el}}^{-1} + \tau_p^{-1}$.

The parameters used to calculate the curves shown in Figs. 1 and 2 are, apart from the magnitude of the attenuation, as follows: The Lorentzian distribution of asymmetries is centered at $\Delta_1 / k_B = \pm 60$ K with a width $\Delta_0 / k_B = 15$ K, the phenomenological relaxation time is characterized by $n = 8$ and $F / k_B^2 = 4 \times 10^{-6}$ K^{-6} s^{-1}; the strength of the electron relaxation is given by $K E_T^2 / k_B^2 = 1.4 \times 10^{-5}$ K. If a typical value of $K = 0.04$ is used, then the inferred tunnel splitting is $E_T / k_B = 0.019$ K. This value for D tunneling in Y seems reasonable as compared to values for H tunneling in Nb (Ref. 36) and Sc (Ref. 27).

From Eqs. (1) and (2), the magnitude of the attenuation peaks depends on $n_0 D^2 / 4C \approx n_0 \gamma^2 / 4C$. In general the various eigenmodes will depend on a combination of elastic constants, so that C is not a single elastic constant.

An analysis of our results reveals, however, that several modes depend almost entirely on a single elastic constant. We found three modes that were almost pure C_{44}; the 0.81-MHz data of Fig. 1(a), the 0.55-MHz data of Fig. 2, and a mode at 0.79 MHz which is not shown. These three curves were fit with the *same* value of $n_0\gamma^2/4C$. Using our value of 2.56×10^{10} Pa for C_{44}, we find $n_0\gamma^2 = 2.6\times10^{-13}$ $J^2 m^{-3}$ for these modes. We do not know what fraction of the deuterium atoms participate in the ultrasonic attenuation. NMR spin-lattice relaxation measurements on ScH_x were fit[27] with 7% of the hydrogen ions contributing. For a crude estimate of γ we will use the 7% number here. With this figure we find $\gamma_{44} = 0.22$ eV, without regard to sign. Our analysis shows that the mode of Fig. 1(b) is almost entirely dependent on C_{66}. Following the above analysis we find $n_0\gamma^2 = 6.6\times10^{-14}$ $J^2 m^{-3}$ for this mode, and $\gamma_{66} = 0.11$ eV. These results for γ are somewhat larger than was found[24] for deuterium motion trapped near N in Nb, but comparable to those found for amorphous materials.[29]

The modulation of the asymmetries, which is responsible for the coupling between the ultrasound and the TLS's, is assumed to be due to D-D interactions. In view of the strong pairing between deuterium atoms located along the c axis, e.g., T sites 3 and 4 in Fig. 3, one might, at first, expect large ultrasonic losses to be associated with strains which change the separation of such pairs. It is interesting to note that the strains associated with C_{44} and C_{66}, namely ϵ_4 and ϵ_6, *do not* change this separation. Therefore, the loss peaks shown in Fig. 1 and the lowest frequency of Fig. 2 do not depend on a modulation of the distance between pairs lying along the c axis. (The vibrational eigenmodes associated with the two higher frequencies of Fig. 2 have not been identified). To explain the TLS-ultrasound coupling responsible for these peaks, we are led to consider interactions between D atoms occupying T sites such as 1 and 3 in Fig. 3. These sites are separated by about 2.6 Å. The change in the distance $r_{1,3}$
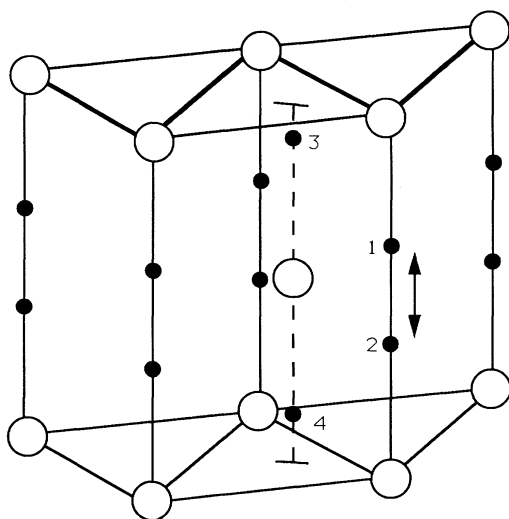

FIG. 3. Crystal structure of α-phase YD_x. The open circles represent Y atoms and the filled circles represent T sites which are partially occupied by D.

due to an ultrasonic strain is larger for ϵ_4, and equivalently ϵ_5, than for ϵ_6, in qualitative agreement with the results of Fig. 1. To investigate most directly the effects of the modulation of c-axis pairs on the ultrasonic attenuation, it would be desirable to have an eigenmode which depends solely on C_{33}, since the corresponding strain, ϵ_3, changes the separation of c-axis pairs. Unfortunately, among the modes investigated none depends solely on C_{33}. Several modes, however, do have a substantial contribution from C_{33}. Although a detailed analysis of the contributions to the ultrasonic attenuation for such modes is difficult, none of these modes show a particularly high loss peak. The result is that the interaction between second-neighbor c-axis pairs does not play a dominant role in the asymmetries responsible for the coupling between the TLS's and the ultrasonic vibration. Our interpretation of this result is that such pairs are locked in position at the temperature of our measurements, and for that reason do not play a role in the ultrasonic attenuation. This interpretation is consistent with our assumption above that only a small fraction of the D atoms contribute to the attenuation.

Recent acoustic measurements[14] in the frequency range 2–32 kHz on $YO_{0.0027}H_{0.016}$ revealed a series of loss peaks attributed to H trapped near O. Although our sample has a comparable O concentration, we do not believe that the peak we observe is as reported in Ref. 14 for two reasons: (1) The only candidate seems to be the peak labeled $P2$ in Ref. 14, but an extrapolation of the data for $P2$ to the frequency range of our measurements indicates a much higher temperature for $P2$ than we observe. The measurements in Ref. 14 are for *hydrogen,* while our measurements are for deuterium. It appears likely that deuterium moves more slowly than hydrogen, so the corresponding peak $P2$ would occur at an even higher temperature. This effect may contribute to the absorption increase we observe near room temperature. (2) The attenuation peaks we observe in Y, and have also observed[37] in Sc, appear similar to the maxima in the spin-lattice relaxation rates observed by NMR in these materials, especially ScH_x. The NMR results would not be expected to be strongly influenced by H trapped at low concentrations of O.

It seems likely that the ultrasonic-attenuation peaks we observe are due to deuterium atoms moving between nearest-neighbor T sites. Those pairs of sites with low or zero asymmetry have a very short relaxation time and contribute very little to the ultrasonic attenuation ($\omega\tau \ll 1$). Most of the sites, however, have high enough asymmetry that $\omega\tau > 1$ at the lowest temperature. The attenuation at the lower temperatures is due to sites with moderate asymmetry, which have $\omega\tau \approx 1$ and for which the upper level is thermally populated. At higher temperatures the rate τ_p^{-1} dominates the relaxation and the condition $\omega\tau = 1$ is satisfied for many centers in the 90-K temperature range, resulting in the attenuation peak the position of which shifts with frequency. Those sites in the wings of the distribution with low or zero asymmetry cause the theoretical curves to show an upturn as the temperature approaches zero. This increase occurs because such TLS's have a very fast relaxation rate, so that

the condition $\omega\tau\approx 1$ is satisfied at low temperature where the factor $(1/k_B T)\text{sech}^2[E/2k_B T]$ is large. Unfortunately, the experimental arrangement for the present work did not permit measurements below about 10 K. Measurements at lower temperatures would be desirable as a check of the distribution of asymmetries.

The measurements of Ref. 14 for H trapped near O in Y concluded that multiphonon relaxation processes dominated the relaxation above a few K. Our results are different. We find that $\tau_{el}\approx\tau_p$ at approximately 70 K. The disparity may be due to the different isotopes (H vs D) or different lattice sites occupied by the isotopes.

No doubt some of the parameters of our model could be changed, while still achieving a good fit to the data. The key features which yield a good fit to the data are the following: (1) a weakly temperature-dependent relaxation rate which dominates at low temperature. Another expression than the one we have used would probably fit the data with a somewhat different distribution of asymmetries. Most of the deuterium atoms have relaxation rates less than the ultrasonic frequency at low temperature. (2) a strongly temperature-dependent relaxation time which dominates the relaxation above about 70 K. This rate should vary approximately as T^8 in the temperature range of 80–140 K to fit both the shift of the peak with frequency and the high-temperature side of the attenuation peak.

Our analysis is based on the assumption of a temperature-independent distribution of asymmetries, but in fact the distribution may have a temperature dependence. The excitation of the deuterium to a higher level in one set of wells may reduce the asymmetry in a neighboring set of wells. The very rapid temperature dependence of the relaxation rate may be partly the result of the distribution changing to smaller asymmetries with faster rates.

IV. CONCLUSIONS

We have presented ultrasonic evidence for a rapid motion of D in Y in the temperature range of 15–140 K. The most likely origin of this motion, at least in the lower temperature range, appears to be tunneling between nearest-neighbor T sites with a distribution of asymmetries. A weakly temperature-dependent relaxation rate, probably due to coupling to electrons, dominates the relaxation up to about 70 K. Above this temperature a much more strongly temperature-dependent relaxation process becomes dominant. Interactions between D atoms lying on *adjacent c axes, not pairs lying along the same c axis,* appear to play the dominant role in providing the asymmetries involved in coupling the tunneling centers to the ultrasound.

ACKNOWLEDGMENTS

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