

Ultrasonic evidence for strong isotope effects on the local motion of H(D) in TaV₂H(D)_x

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Ultrasonic measurements were performed on the C15 Laves-phase compounds TaV₂H_{0.18} and TaV₂D_{0.17} over the temperature range 3–345 K. Ultrasonic loss peaks were observed in both materials at 250–275 K for measurement frequencies in the range of 1 MHz. These peaks were interpreted as H(D) hopping between interstitial *g*-site hexagons, the motion responsible for long-range diffusion. This motion exhibited a small isotope effect with the H hopping rate being somewhat faster than that for D. The activation energies and attempt frequencies were in good agreement with earlier measurements by other techniques. In contrast to the similarity of the TaV₂H_{0.18} and TaV₂D_{0.17} results at high temperatures, the low-temperature behavior of the two materials differed substantially. A weak loss peak was observed in TaV₂D_{0.17} at low temperatures. This peak was attributed to the local motion within *g*-site hexagons previously explored by nuclear magnetic resonance and quasielastic neutron scattering experiments. At a given temperature the rate of this motion is several orders of magnitude faster than that responsible for the high-temperature peak. No evidence of a low-temperature loss peak was found for TaV₂H_{0.18}. This strong isotope effect suggests that the local motion is highly dominated by quantum effects. The most likely reason for the absence of an attenuation peak in TaV₂H_{0.18} is that the local motion occurs at a rate much higher than that of the ultrasonic frequency (~ 1 MHz) throughout the temperature range explored.

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I. INTRODUCTION

A number of studies have shown unusual features for the motion of hydrogen isotopes in C15 Laves-phase materials. Nuclear magnetic resonance (NMR) and quasielastic neutron scattering (QENS) experiments give strong evidence for two frequency scales of the H(D) hopping rate among interstitial sites in these materials.^{1–8} Hydrogen isotopes occupy the interstitial *g* sites in these compounds,⁹ these sites forming a series of linked hexagons. The *g*-*g* distance for neighboring *g* sites *within* the same hexagon, r_1 , is usually shorter than the *g*-*g* distance for *g* sites on *different* hexagons r_2 . The present experimental evidence indicates that the slower, rate-limiting step for long-range diffusion involves H(D) hops between hexagons, while the faster, local motion involves hops within the same hexagon. There is much that is not understood about this local motion. Only a small fraction of H(D) atoms participates in the local motion, and this fraction appears to be both isotope and temperature dependent. There are similarities to the situation in the rare-earth metals where there are at least two frequency scales of motion, and where there is NMR evidence for a local motion in which only an isotope-dependent fraction of the H(D) atoms participate.¹⁰ NMR and QENS have been the primary techniques used to study dynamics of H(D) in the Laves-phase materials, although a preliminary ultrasonic experiment¹¹ gave evidence for an isotope effect in the local motion. Ultrasonic methods, and mechanical spectroscopy in general, are especially well suited for studying isotope effects in that the coupling between the ultrasound and the H(D) atoms is not sensitive to isotope mass, cross section, or spin, so that the measured isotope effects reflect the motion of the isotopes, independent

of the coupling mechanism. Furthermore, mechanical spectroscopy can explore a much lower frequency range than the other techniques. Because the motion is usually temperature dependent, the lower frequencies essentially permit the study of a different temperature range. As a result, it is possible that ultrasonic measurements at relatively low frequencies may reveal two attenuation peaks well separated in temperature due to the two types of motion.

A series of studies⁸ have shown a strong correlation between the parameters of the two jump processes and the two *g*-*g* distances r_1 and r_2 . In comparison to a number of C15 Laves-phase compounds the ratio r_1/r_2 is least for TaV₂, while the ratio of the fast jump rate to the slow jump rate is greatest in this compound.⁵ Measurements of the temperature dependence of the nuclear spin-lattice relaxation rates for ¹H, ²D, and ⁵¹V in TaV₂H(D)_x showed two overlapping maxima due to H(D) motion, with the maximum at lower temperature being attributed to the local motion. While the high-temperature motion exhibited Arrhenius behavior, the lower-temperature motion was strongly non-Arrhenius. The measurements of the spin-lattice relaxation of ⁵¹V due to quadrupolar interactions with the moving H and D atoms revealed isotope effects.² The results indicated that the local motion (and indeed long-range motion) of D was somewhat slower than that of H for a given concentration and temperature. More striking, however, was the fact that the ⁵¹V relaxation rate due to D was considerably greater than that due to H. This effect was interpreted as the fraction of D atoms participating in the local motion being about three times that of H, both fractions increasing with temperature.

The present paper reports ultrasonic attenuation and elastic constant measurements of TaV₂H_{0.18} and TaV₂D_{0.17} over

the temperature range 3–345 K. These measurements in the 1-MHz frequency range reveal a large attenuation peak in both $\text{TaV}_2\text{H}_{0.18}$ and $\text{TaV}_2\text{D}_{0.17}$, which is associated with hexagon to hexagon hopping of H and D atoms, respectively. For the case of $\text{TaV}_2\text{D}_{0.17}$ a second, much weaker peak is observed at low temperatures that is well separated from the high-temperature peak. No such low-temperature peak is found in $\text{TaV}_2\text{H}_{0.18}$ indicating a strong isotope effect.

II. EXPERIMENTAL DETAILS

Polycrystalline ingots of TaV_2 were prepared by arc melting appropriate mixtures of the high-purity constituent elements in an argon atmosphere followed by various annealing procedures. The TaV_2 material was then loaded with H or D to obtain $\text{TaV}_2\text{H}_{0.18}$ or $\text{TaV}_2\text{D}_{0.17}$ in a manner described previously.¹ Resonant ultrasound spectroscopy^{12–14} (RUS) was used to measure the ultrasonic loss and elastic constants. With the RUS technique, piezoelectric transducers are used to excite a large number of the lowest-frequency vibrational eigenmodes in samples of well-defined shapes, such as rectangular parallelepipeds. Samples were prepared by cutting approximate parallelepipeds from the button ingots using a low-speed diamond saw. These were then hand polished into accurate rectangular parallelepipeds suitable for the ultrasonic measurements. The room-temperature sample edge dimensions of $\text{TaV}_2\text{H}_{0.18}$ and $\text{TaV}_2\text{D}_{0.17}$ were $0.2103 \times 0.1681 \times 0.1903$ and $0.1215 \times 0.1697 \times 0.1072$ cm³ with masses of 65.6 and 21.6 mg, respectively. Either lead zirconate titanate (PZT) or lithium niobate compressional-mode transducers were used to excite and detect the mechanical resonances. Measurements below room temperature were made using a commercial cryostat and temperature controller, with temperature resolution of ± 0.1 K. These measurements were made in a low vacuum (~ 3 mm of Hg) environment of helium gas. Measurements above room temperature were made at atmospheric pressure using a commercial oven and temperature controller. From measurements of the resonant frequencies, with the sample dimensions, density, and crystal structure known, the full elastic modulus tensor can be determined. The ultrasonic attenuation is found by measuring the quality factor Q of the resonant line shapes. The ultrasonic loss is then given simply as $1/Q$.

III. RESULTS AND DISCUSSION

Figure 1 shows the ultrasonic loss versus temperature over the range 3–345 K for $\text{TaV}_2\text{H}_{0.18}$ and $\text{TaV}_2\text{D}_{0.17}$. The frequencies of the modes are 1.058 and 1.149 MHz for $\text{TaV}_2\text{H}_{0.18}$ and $\text{TaV}_2\text{D}_{0.17}$, respectively. These peaks each depend solely on the aggregate shear elastic constant, not on the bulk modulus. Several features are immediately apparent. A large loss peak centered near 250–275 K is observed for both compounds. As will be discussed in more detail, these peaks are associated with H(D) hopping between hexagons, the relatively slow, rate-limiting motion responsible for long-range diffusion. The two peaks are of nearly equal amplitude, indicating that the coupling of the H(D) motion to the ultrasound depends little, if any, on the isotope. The peaks do

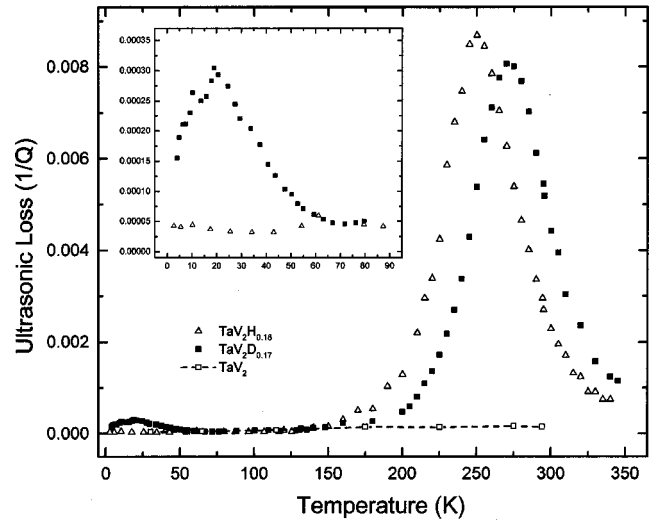


FIG. 1. Ultrasonic loss vs temperature in $\text{TaV}_2\text{H}_{0.18}$, $\text{TaV}_2\text{D}_{0.17}$, and TaV_2 over the range 3–345 K. The ultrasonic frequency was approximately 1 MHz for all modes shown.

indicate a small isotope effect for the H(D) hopping rate; the peak for $\text{TaV}_2\text{D}_{0.17}$ clearly occurs at higher temperatures for approximately the same measurement frequency. This indicates that the D hexagon-to-hexagon hopping rate is somewhat lower than that for H, a result consistent with the NMR measurements.²

More important, there is a small loss peak at low temperature for D, *but no such peak is observed for H*. By comparison with the NMR results, this low-temperature peak is associated with the rapid, local motion. The results immediately indicate a large isotope effect for the local motion. Figure 2 shows in more detail the low-temperature loss peaks in $\text{TaV}_2\text{D}_{0.17}$ for four measurement frequencies. The peaks shift slightly to higher temperatures for higher measurement frequencies. Neither the high-temperature peak nor the low-temperature peak was observed in the H(D)-free material, which had a low-level, almost temperature-independent attenuation.

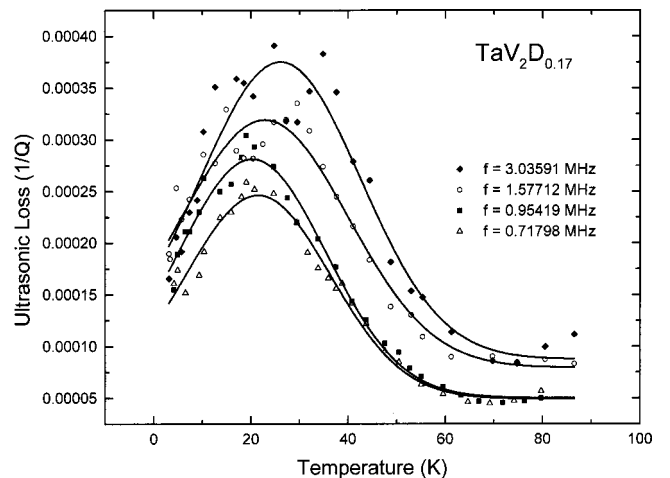


FIG. 2. Expanded view of the low-temperature ultrasonic loss peak in of $\text{TaV}_2\text{D}_{0.17}$ for four vibrational modes. The solid lines are guides to the eye and do not represent theoretical fits to the data.

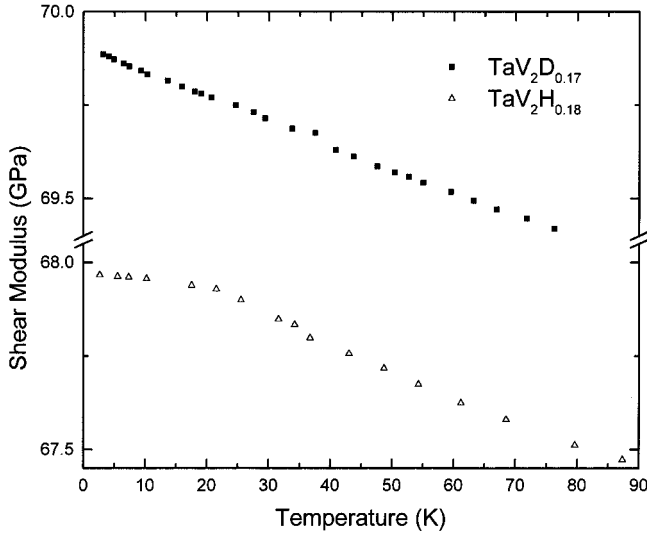


FIG. 3. Elastic shear modulus (G) for polycrystalline $\text{TaV}_2\text{H}_{0.18}$ and $\text{TaV}_2\text{D}_{0.17}$ in a range 3–90 K.

Figure 3 shows the aggregate elastic shear modulus, G , for these polycrystalline samples. Although a more detailed account of TaV_2H_x modulus measurements has been published elsewhere,^{15,16} the important point to notice in Fig. 3 is that the temperature dependence of G clearly differs for the two isotopes. The shear modulus of $\text{TaV}_2\text{H}_{0.18}$ deviates from an approximately linear temperature dependence below about 20 K, but this does not happen for $\text{TaV}_2\text{D}_{0.17}$. Due to uncertainties in sample dimensions and density, the absolute error in G is about 1%; thus the difference in absolute values of the shear modulus for the two materials is just marginally outside experimental error. The error in the temperature dependence is much smaller and is essentially given by the scatter in the data.

We now turn to a more detailed analysis of the data. The hopping of hydrogen between nearby interstitial sites has been studied in many systems by means of mechanical spectroscopy (i.e., internal friction, ultrasonic attenuation, etc.).^{17–26} Anelastic relaxation²⁷ occurs in cases where nearby interstitial sites are affected differently by an external stress, leading to a redistribution of the hydrogen occupancy of the sites. If the redistribution rate is comparable to the frequency of the applied stress, mechanical-energy dissipation occurs. The ultrasonic loss $1/Q$ due to relaxation is given by^{28–30}

$$\frac{1}{Q} = \frac{\Delta c}{c} \frac{\omega \tau_R}{1 + \omega^2 \tau_R^2}, \quad (1)$$

where $\Delta c/c$ is often referred to as the relaxation strength, $\omega/2\pi$ is the ultrasonic frequency, and τ_R is the relaxation time. The quantity Δc is the difference between the unrelaxed (c_U) and relaxed (c_R) elastic moduli; $\Delta c = c_U - c_R$. An explicit expression for the relaxation strength can be calculated for simple systems. An interstitial atom such as hydrogen, which may occupy either of two nearby interstitial sites, can be described as a two-level system. The energy splitting ΔE is given by

$$\Delta E = 2(E_T^2 + A^2)^{1/2}, \quad (2)$$

where $2E_T$ is the tunnel splitting and $2A$ is the difference in site energy (asymmetry). For this case it can be shown that the relaxation strength is of the form

$$\frac{\Delta c}{c} = \frac{n \gamma^2}{k_B T c} \text{sech}^2 \left[\frac{\Delta E}{k_B T} \right], \quad (3)$$

where n is the concentration of hydrogen atoms contributing to the attenuation, $\gamma = \partial(\Delta E)/\partial \epsilon$ is the variation of the energy level splitting with respect to the ultrasonic strain ϵ , and c is an elastic constant. The formalism may be used even when the tunneling matrix element E_T is negligible. It is usually assumed that the variation of ΔE with respect to strain is due to the strain dependence of A , not E_T . It is seen that for γ to be nonzero, the two nearby interstitial sites must respond differently to strain; in other words, they are inequivalent. Except at very low temperatures it is usually the case that $k_B T \gg \Delta E$ and the $\text{sech}^2[\Delta E/k_B T]$ term approaches unity. In the present study it was found that the data for the high-temperature peaks could be fitted satisfactorily with Eq. (3) using this assumption. Figures 4(a) and 4(b) show more attenuation data for the high-temperature peaks, for both compounds, along with fits using thermally activated hopping for τ_R . For $\text{TaV}_2\text{D}_{0.17}$ the data for two different measurement frequencies (1.1490 and 2.2626 MHz) were fitted using the simple expression

$$\tau_R = \tau_{R_0} \exp(E_a/k_B T), \quad (4)$$

giving τ_{R_0} and E_a as 6.0×10^{-13} s and 0.29 eV, respectively. The solid lines in Fig. 4(a) are fits using these parameters. The agreement between experiment and theory is clearly rather good. This value for E_a is in reasonable agreement with the NMR results at higher temperatures and concentrations. Skripov *et al.*² found 0.26 eV for $\text{TaV}_2\text{D}_{0.50}$ in the 300–400-K range. While the data for $\text{TaV}_2\text{H}_{0.18}$ could be fitted reasonably well with the same approach, it was found that a consistently better fit was found by taking the hopping rate for H as the sum of two Arrhenius rates,

$$\tau_R^{-1} = \tau_{R_{01}}^{-1} \exp \left[\frac{-E_{a1}}{k_B T} \right] + \tau_{R_{02}}^{-1} \exp \left[\frac{-E_{a2}}{k_B T} \right]. \quad (5)$$

Figure 4(b) shows the fit of the data for $\text{TaV}_2\text{H}_{0.18}$ for four different measurement frequencies (0.8–1.4 MHz). The results of the fit gave $(9.6 \times 10^{-13}$ s, 0.27 eV) and $(6.0 \times 10^{-9}$ s, 0.12 eV) for $(\tau_{R_{01}}, E_{a1})$ and $(\tau_{R_{02}}, E_{a2})$, respectively. We follow the interpretation of Renz *et al.*^{31,32} associating process 1 with phonon-assisted tunneling through an excited state and process 2 with phonon-assisted tunneling between ground states. The attempt frequencies for the higher activation energy process are $\tau_{R_{01}}^{-1} \approx (1-2) \times 10^{12}$ s⁻¹, which are much lower than the typical H(D) vibration frequencies³³ of $\nu_{\text{HD}} \approx (2-4) \times 10^{13}$ s⁻¹; however, these attempt frequencies are closer to the Debye frequency, which we estimate as $\nu_{\text{D}} \approx 6 \times 10^{12}$ s⁻¹ for TaV_2 . The theory of quantum diffusion^{34,35} indicates that $\tau_{R_{01}}^{-1}$ should be of the

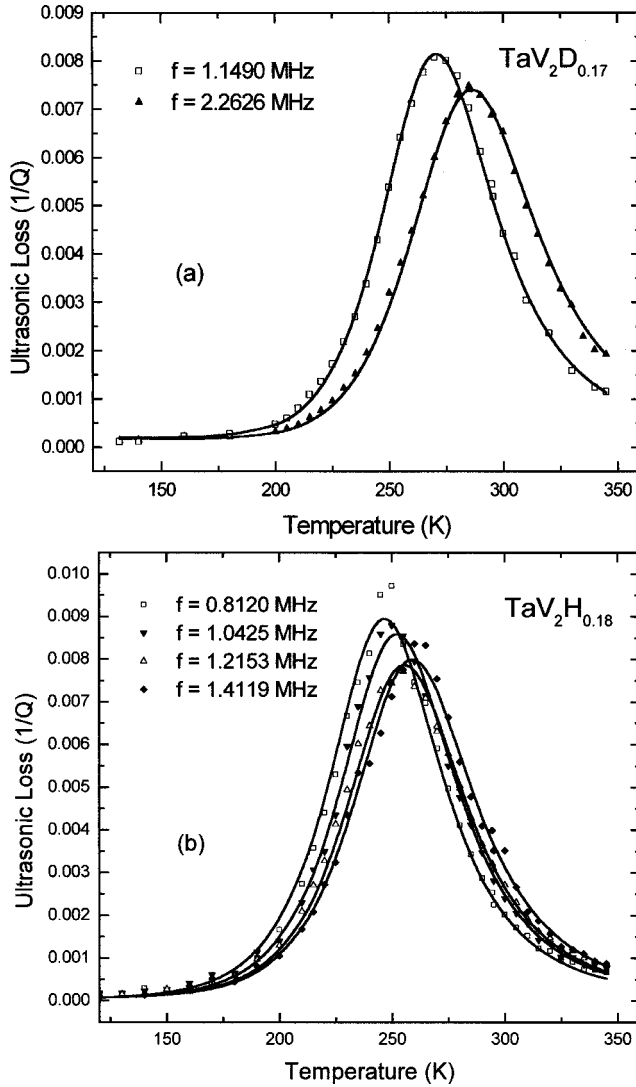


FIG. 4. Expanded view of the high-temperature ultrasonic loss peaks: (a) $\text{TaV}_2\text{D}_{0.17}$. The solid lines represent theoretical fits to the data using Eqs. (1), (3), and (4). (b) $\text{TaV}_2\text{H}_{0.18}$. The solid lines represent theoretical fits to the data using Eqs. (1), (3), and (5).

order of the Debye frequency. This theory also accounts in a natural way for the absence of process 2 for D. Tunneling between ground states is expected to be especially sensitive to the mass of the tunneling particle; process 2 may be suppressed for D due to the larger mass. The slightly higher value of E_a^D as compared to E_a^H is in agreement with Skripov *et al.*,² who found a slightly higher activation energy for D as compared to H for comparable concentrations.

In contrast to the high-temperature peak in $\text{TaV}_2\text{D}_{0.17}$, which is well described with an Arrhenius-type process, the low-temperature peak behaves quite differently. An activation energy of ~ 10 meV would be required to describe the shift of the peaks with the ultrasonic measurement frequency and also to reconcile the present results with the earlier NMR results. However, an activation energy of this value gives loss peaks that are too narrow, and have the wrong shape, to describe the actual data. Thus, it seems that a simple Arrhenius process will not describe the low-temperature peak; this

conclusion is in agreement with the NMR results obtained at higher temperatures. The broad, asymmetric loss peak could no doubt be fitted with a distribution of activation energies, but there seems little justification for such an approach, especially given that the high-temperature peak requires only a single activation energy. In fact, the NMR results are also inconsistent with a broad distribution of frequencies of the localized motion. Skripov *et al.*² were able to fit the low-temperature spin-lattice relaxation with an expression $\tau_L = \tau_{L0} \exp(-T/T_0)$ for the hopping time at low temperatures, where T_0 and τ_{L0} are fitting parameters. Such an expression has some justification from the theory of quantum diffusion.³⁶ Using this expression for the relaxation time in Eq. (1), good fits to the low-temperature peaks were achieved for each *particular* frequency. However, this expression cannot successfully account for the shift of the peaks with measuring frequency.

Further complicating the attempt to give a theoretical description of the low-temperature peak is the fact that NMR and QENS results indicated that only a fraction of the D atoms participate in the local motion, and that this fraction is temperature dependent. As a result, the temperature dependence of the relaxation strength $\Delta c/c$ is not clear at this point. However, certain observations are possible. Clearly, the low-temperature peak is much weaker than the high-temperature peak. To proceed further we assume that the relaxation strength varies as $n\gamma^2/T$ where now n is the concentration of participating D atoms. Furthermore, for a crude estimate we assume that γ is the same for the high-temperature and low-temperature peaks. This assumption may be reasonable because g sites are apparently involved in both cases.

Comparing the peak heights at 250 and 20 K leads to an estimate of $n_l = 3 \times 10^{-3} n_h$, where n_l and n_h refer to the concentration of D atoms participating in the low- and high-temperature peaks, respectively. Assuming that essentially all the D atoms contribute to the high-temperature peak it is estimated that only 0.3% of D atoms participate in the local motion at 20 K. Skripov *et al.*⁵ estimated the fraction of H atoms participating in the local motion by QENS measurements over the temperature range 80–300 K. Extrapolating their results for $\text{TaV}_2\text{H}_{0.6}$ down to 20 K gives 3.7% as the participating concentration of H. However, NMR measurements² at much higher temperatures indicated that the fraction of D atoms participating in the local motion is about three times that of H. Thus, the QENS and NMR results at higher temperatures extrapolated to 20 K give an estimate of about 10% participating D. This is much larger than our estimate of 0.3%. This disagreement may indicate that the extrapolation is invalid, or that one of the assumptions about $\Delta c/c$ is incorrect. Also, the QENS and NMR measurements were at higher H(D) concentrations than the present measurements.

The most remarkable feature of the present results is the absence of a low-temperature peak for the hydrogen-containing material. There are at least two possibilities for this absence. First, the number of participating H atoms may be too low to result in an observable peak. However, if the factor of 3 found from NMR measurements holds for the

present case a low-temperature peak should be observed. Figure 1 indicates that a peak approximately a factor of 10 weaker than that found for D would just be observable. Second, it may be that the local motion remains at a rate well above the ultrasonic frequency even down to 3 K so that the condition $\omega\tau_R \ll 1$ holds throughout the temperature range of the measurements. This high value of the local relaxation rate is consistent with the NMR measurements. Some support for this second viewpoint is also found from the modulus measurements. Tunneling motion can lead to a resonant decrease in the sound velocity at low temperatures.^{29,37} In brief, H would be expected to have a larger tunneling matrix element than D due to the mass difference. The different temperature dependencies for the shear modulus of TaV₂H_{0.18} and TaV₂D_{0.17} could be due to the onset of the resonant process for H as the temperature is lowered. Thus, rapid tunneling by H could account for both the absence of a low-temperature peak for H (the relaxation rate remains well above the ultrasonic frequency) and the different temperature dependencies for the moduli. This downturn depends on both the tunneling matrix element and any asymmetries that exist among the interstitial sites. The present data are insufficient to determine these parameters. Experiments at lower temperatures are planned to obtain more data and test this idea.

IV. CONCLUSIONS

The unusual motion reported for H(D) in C15 Laves-phase compounds has been investigated in TaV₂H_{0.18} and TaV₂D_{0.17} by means of ultrasonic measurements. An ultrasonic loss peak at 250–275 K for measurement frequencies in the range of 1 MHz was observed in both materials and

interpreted as H(D) hopping between *g*-site hexagons, the motion responsible for long-range diffusion. This motion exhibited a small isotope effect with the H hopping rate being somewhat faster than that for D. The activation energies and attempt frequencies were in good agreement with earlier measurements. In contrast to the similarity of the TaV₂H_{0.18} and TaV₂D_{0.17} results at high temperatures, the low-temperature behavior of the two materials differed substantially. A weak loss peak was observed in TaV₂D_{0.17} at low temperatures. This peak was attributed to the local motion within *g*-site hexagons previously explored by NMR and QENS experiments. The rate for this motion is several orders of magnitude faster than that of the high-temperature peak. No evidence of a low-temperature loss peak was found for TaV₂H_{0.18}. This strong isotope effect suggests that the local motion is highly dominated by quantum effects. The most likely reason for the absence of an attenuation peak in TaV₂H_{0.18} is that the local motion occurs at a rate much higher than that of the ultrasonic frequency (~ 1 MHz) throughout the temperature range explored (down to 3 K). To explore these ideas further, future experiments at lower temperatures and higher H concentrations are planned.

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