Magnetically originated phonon-glass-like behavior in Tb$_2$Ti$_2$O$_7$ single crystal

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We report a study on the thermal conductivity ($\kappa$) of Tb$_2$Ti$_2$O$_7$ single crystals at low temperatures. It is found that in zero field this material has an extremely low phonon thermal conductivity in a broad temperature range. The mean free path of phonons is even smaller than that of amorphous materials and is 3–4 orders of magnitude smaller than the sample size at 0.3 K. The strong spin fluctuation of the spin-liquid state is discussed to be the reason of the strong phonon scattering. The magnetic-field dependence of $\kappa$ and comparison with Y$_2$Ti$_2$O$_7$ and TbYTi$_2$O$_7$ confirm the magnetic origin of this phonon-glass-like behavior.

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I. INTRODUCTION

Ultra-low thermal conductivity ($\kappa$) of crystal lattice or phonons is a long-term investigated topic.\textsuperscript{1} It is directly related to the applications of thermoelectric materials and thermal barrier materials. Introducing structural imperfections into the samples, such as impurities and defects, is always a useful method to increase the phonon scattering and decrease the phonon thermal conductivity.\textsuperscript{2} In an extreme case, the amorphous solids are known to have very weak heat transport of phonons.\textsuperscript{2} Because of the lack of the long-range periodicity of the atom positions in amorphous materials, the phonon mean free path is very short even at very low temperatures.\textsuperscript{2} However, in many cases, it is not a suitable way to obtain small phonon thermal conductivity by simply using amorphous materials. For example, the highly efficient thermoelectric materials should have simultaneous low thermal conductivity and high electric conductivity.\textsuperscript{3,4} This requirement cannot be met in those samples with significant crystal imperfections or in amorphous solids. In other words, to obtain very small phonon thermal conductivity in bulk single crystals is still a challenging task.

There may be some chances in magnetic materials since some recent works have revealed that the magnetic fluctuations could yield strong scattering on phonons. The magnetic excitations can work as either heat carriers or phonon scatterers.\textsuperscript{5} In those materials exhibiting long-range magnetic order, the strong magnetic fluctuations near the critical region of phase transition can scatter phonons rather strongly.\textsuperscript{5,6} A recent example is the hexagonal manganite HoMnO$_3$.\textsuperscript{7} The phonon thermal conductivity was found to be suppressed by two orders of magnitude at the regime of antiferromagnetic (AF) transition of Ho$^{3+}$ ions. However, such scattering is not effective at lower temperatures when the long-range order is well established.\textsuperscript{7,8} In addition, the phonon thermal conductivities of these materials are still much larger than those of the amorphous materials. Even stronger magnetic scattering effect is therefore to be expected in those materials with strong spin fluctuations and no long-range order. This is what the spin-liquid materials may exhibit. Another requirement for strong phonon scattering in magnetically disordered state is that the coupling between spin and lattice should be strong.

Geometrical spin frustration is very promising to result in a three-dimensional spin liquid.\textsuperscript{9} A well studied material is the rare-earth pyrochlore Tb$_2$Ti$_2$O$_7$.\textsuperscript{9,10} Although the ground state of this materials is still a controversial question, most of the previous studies evidenced that it is a cooperative paramagnet down to milli-kelvin temperatures, much lower than the energy scale of the AF interactions. The single-ion ground state is a degenerate doublet and the Tb$^{3+}$ spin has an Ising-like anisotropy with the easy axis along the local [111] axis.\textsuperscript{11} In addition, the peculiar crystal field effect yields a pronounced magnetoelectric effect, which was verified by X-ray diffraction and demonstrated that the coupling between spin and lattice is very strong.\textsuperscript{12} As a result, an anomalously strong structural fluctuation has been observed in zero field.\textsuperscript{12} Therefore, Tb$_2$Ti$_2$O$_7$ seems to meet the requirements for exhibiting strong spin-phonon scattering. In this work, the thermal conductivity of high-quality Tb$_2$Ti$_2$O$_7$ single crystals is measured down to 0.3 K. It is found that in a broad temperature regime the thermal conductivity is extremely small and comparable to that of the amorphous materials. In particular, the mean free path of phonons is found to be as low as 20 nm at 4 K and 3–4 orders of magnitude smaller than the sample size at subkelvin temperatures, and is even shorter than that of amorphous materials.\textsuperscript{2} This result shows an exceptional case of very low phonon thermal conductivity in an insulating single crystal. The strong spin fluctuation in the spin-liquid-like state is the main mechanism of the phonon scattering. Both the magnetic-field-induced increase of $\kappa$ and the comparison of Tb$_2$Ti$_2$O$_7$ with Y$_2$Ti$_2$O$_7$ and TbYTi$_2$O$_7$
also confirm the magnetic origin of this phonon-glass-like behavior.

II. EXPERIMENTS

High-quality Tb$_2$Ti$_2$O$_7$, Y$_2$Ti$_2$O$_7$, and TbYTi$_2$O$_7$ single crystals were grown by the floating-zone technique. These crystals could be grown well under different oxygen pressures and growth rates. Tb$_2$Ti$_2$O$_7$ and TbYTi$_2$O$_7$ crystals were grown in 0.4 MPa pure oxygen with a growth rate of 2.5 mm/h and in 0.25 MPa pure oxygen with a rate of 3 mm/h, respectively. Y$_2$Ti$_2$O$_7$ crystal was grown in mixed oxygen and argon with a ratio of 4:1 and at a growth rate of 4 mm/h. The thermal conductivities were measured using a conventional steady-state technique and two different processes: (i) using a “one heater, two thermometers” technique in a $^3$He refrigerator and a 14 T magnet at temperature regime of 0.3 – 20 K; (ii) using a Chromel-Constantan thermocouple in a pulse-tube refrigerator for zero-field data above 5 K. Note that a careful precalibration of resistor (RnO$_2$) sensors is indispensable for the precise thermal conductivity measurements in high magnetic fields and at low temperatures. The specific heat was measured by the relaxation method in the temperature range from 0.4 to 30 K using a commercial physical property measurement system (PPMS, Quantum Design).

III. RESULTS AND DISCUSSION

Figure 1(a) shows the zero-field thermal conductivities of two Tb$_2$Ti$_2$O$_7$ samples with very different dimensions (2.91×0.66×0.16 mm$^3$ and 3.31×2.43×1.84 mm$^3$). The heat currents were applied along the length direction, which was along the [111] axis of crystal lattice. There are several remarkable features in these two curves that point to a phonon-glass-like behavior of these single-crystal samples. First, the magnitudes of $\kappa$ are very small and there is no any signature of the so-called phonon peaks at low temperatures. It is well known that the $\kappa(T)$ of insulators usually exhibits a pronounced peak at low temperatures (10–20 K), which is a characteristic of phonon heat transport. The absence of phonon peak in a high-quality single crystal is actually quite unusual and indicates a strong phonon scattering effect. In addition, the overall temperature dependence of $\kappa$ is very similar to what the amorphous solids exhibit, in which the phonons behave as a glassy state. Second, the mean free path of phonons is found to be extremely short in Tb$_2$Ti$_2$O$_7$ crystals. The phononic thermal conductivity can be expressed by the kinetic formula $\kappa_{ph} = \frac{1}{2} C v_p l$, where $C = \beta T^3$ is the phonon specific heat at low temperatures, $v_p$ is the average velocity and $l$ is the mean free path of phonons. Using the $\beta$ value obtained from specific-heat measurements, the phonon velocity can be calculated and then the mean free path is obtained from the $\kappa$. The inset to Fig. 1(a) shows the calculated $l$ at low temperatures of the big sample, which is only ~ 20 nm at 4 K. Even when lowering temperature to 0.3 K, $l$ is shorter than 1000 nm and is 3–4 orders of magnitude smaller than the geometrical size of sample. Note that the mean free path is so short that it is even smaller than the typical magnitudes in the amorphous materials. In contrast, the microscopic phonon scatterings in usual single crystals, such as the phonon-phonon scattering and scattering by various crystal defects, would be quenched at very low temperatures and the mean free path could reach the sample size. This is known as the phonon boundary scattering limit. It means that at temperatures as low as 0.3 K the phonon scattering in Tb$_2$Ti$_2$O$_7$ crystals is still very strong. Note that this cannot be caused by the crystal defects since the temperature is low enough. Third, the comparison of two samples with very different sizes also confirmed the
absence of phonon boundary scattering. In usual crystals, when the phonon mean free path is long enough to be comparable to the sample size, the thermal conductivity is proportional to the sample size. However, the two sets of data in Fig. 1(a) indicate that the κ values are essentially independent on the size, which also evidences that the mean free path of phonons must be significantly shorter than the sample size.

It is necessary to discuss the role of magnetic excitations in the heat transport of Tb$_2$Ti$_2$O$_7$. Although the nature of the ground state is not fully understood, it is most likely a spin-liquid-like disordered state, in which the magnetic excitations are short-range correlated spin fluctuations and were evidenced by the low-energy spectra of neutron measurements. From the extremely low thermal conductivity of Tb$_2$Ti$_2$O$_7$, it can be easily concluded that the spin fluctuations strongly scatter phonons rather than act as heat carriers.

Figure 1(b) compares the thermal conductivity of Tb$_2$Ti$_2$O$_7$ with Y$_2$Ti$_2$O$_7$ and ThbYTi$_2$O$_7$ single crystals. Y$_2$Ti$_2$O$_7$ has the same crystal structure with Tb$_2$Ti$_2$O$_7$ but is nonmagnetic. It is clearly seen that the thermal conductivity of Y$_2$Ti$_2$O$_7$ (having size of 4.51×0.63×0.15 mm$^3$) behaves exactly like the usual insulating crystals, with a phonon peak at about 15 K. For a more quantitative comparison, the phonon mean free path of Y$_2$Ti$_2$O$_7$ crystal was also calculated and shown in the inset to Fig. 1(a). At low temperatures, it is 2–3 orders of magnitude larger than that of Tb$_2$Ti$_2$O$_7$ and at 0.3 K it reaches a large value (~2×10$^5$ mm), close to the averaged sample width, suggesting that the boundary scattering limit is almost established. Apparently, the small difference of lattice parameters of Y$_2$Ti$_2$O$_7$ and Tb$_2$Ti$_2$O$_7$ cannot lead to so large difference of phonon transport. The main difference between these two materials is their magnetisms. Therefore, it is naturally concluded that the magnetic scattering, due to the strong magnetic fluctuations in the quantum-spin-liquid state of Tb$_2$Ti$_2$O$_7$, is the main mechanism for the extremely strong phonon scattering. The heat transport of another material, ThbYTi$_2$O$_7$, seems to support this point. ThbYTi$_2$O$_7$ is stoichiometrically a mixture of Tb$_2$Ti$_2$O$_7$ and Y$_2$Ti$_2$O$_7$, which would be expected to exhibit a weaker heat transport than two parent compounds. The high-temperature (>10 K) data indeed show such a result. However, at very low temperatures the κ of ThbYTi$_2$O$_7$ is clearly larger than that of Tb$_2$Ti$_2$O$_7$. Since the dilution of Tb$^{3+}$ ions by the nonmagnetic Y$^{3+}$ ions would suppress the spin correlations, the magnetic scattering on phonons is reasonably weakened.

If the strong phonon scattering of Tb$_2$Ti$_2$O$_7$ single crystals is magnetically originated, the magnetic field is expected to be able to strongly affect the heat transport, which is confirmed by the field dependence measurements of κ. Figure 2 shows how the magnetic field, parallel to either the [111] direction or the vertical one, changes the thermal conductivity. A striking finding is that at very low temperatures the κ can be enhanced by 30–40 times with increasing field. To our knowledge, there has been no report of such a strong magnetothermal conductivity effect in any other magnetic materials. In principle, it is understandable that the magnetic field can suppress the spin fluctuations and therefore weaken the phonon scattering. In this sense, a 30–40 times increase of κ in fields is actually far below the expectation. Considering that the mean free path of phonons is 3–4 orders of magnitude smaller than the geometrical size of the sample, one may expect more than 1000 times enhancement of κ if the magnetic field is strong enough to smear out all the magnetic scattering on phonons.

It is notable that the field dependencies are quite different for two field directions. For $H \perp [111]$, the very-low-T κ is nearly field independent for $H < 2$ T and then increases monotonically with field. It seems that the magnetic field along this direction just simply suppresses the spin fluctuations. The effect of magnetic field along [110] direction had been well studied, in which case the Tb$^{3+}$ spins were thought to align on two sets of chains parallel to the field direction (so-called α chains) align along the local [111] axis with a component parallel to the magnetic field. Whereas the spins on chains perpendicular to the field direction (the β chains) favor an AF order at high fields. This field-induced order had already been revealed by the neutron scattering. It was
found that the field-induced elastic neutron scattering intensity increased gradually above 2 T and at subkelvin temperatures, which is qualitatively consistent with the increase of $\kappa(H)$ above 2 T. At higher temperatures (> 3 K), the field-induced magnetic order was hardly to be observed$^{29,30}$ correspondingly, the $\kappa$ shows much weaker enhancement with increasing field.

For $H \parallel [111]$, both the field and temperature dependencies show much more complicated behaviors, compared to the case of $H \perp [111]$. The 0.36-K $\kappa(H)$ curve shows a three-peak (at 2.5, 8.5, and 11.5 T) or three-dip (at 0.5, 6, and 10.5 T) feature between 0 and 14 T (see Fig. 3(d) also). In principle, the field-induced enhancement of $\kappa$ in this case is likely to have the same mechanism as the case of $H \perp [111]$, i.e., some field-induced magnetic order. However, it is hard to imagine so complicated magnetic transitions if each anomaly on the $\kappa(H)$ curves corresponds to a magnetic transition. There actually has been few experimental studies on the possible field-induced changes of magnetic properties for $H \parallel [111]$. An early neutron scattering experiment had found that the magnetic field along the [111] axis could also induce an AF order$^{31,32}$ The elastic neutron scattering intensity was found to increase with field up to 2–3 T and become nearly saturated, which seems to have some correspondence with the sharp increase of $\kappa$ at 1–2 T. However, either the nature or the spin structure of this ordered phase were not resolved$^{31}$. Magnetic susceptibility data taken at several tens of millikelvin temperatures also indicated a shoulder-like anomaly at about 1.2 T, which is likely to be related to a field-induced order. More recent experiments indicated that this anomaly exists and shows a weak temperature dependence at temperature scale of several hundred millikelvins$^{32,33}$. However, such susceptibility measurements did not reveal any other clear anomaly for fields up to 14 T$^{33}$. Based on these existing experimental results, it is not likely that there are some multiple magnetic transitions in high fields along the [111] axis. A recent susceptibility study has revealed that Tb$_2$Ti$_2$O$_7$ is likely to exhibit a quantum-spin-ice ground state and the magnetic field along the [111] axis can drive transitions very similar to those in the spin-ice material Dy$_2$Ti$_2$O$_7$.$^{34}$ However, these transitions between the “quantum-spin-ice”, the “quantum-kagomé-ice”, and the “three-in-one-out” states occur in rather low fields (< 1 T) and at very low temperatures (< 150 mK).$^{32}$ Therefore, these low-field transitions are apparently irrelevant to the $\kappa(H)$ behaviors in Fig. 2(c). There should be some other reasons for the complicated $\kappa(H)$ behavior besides the field-induced orders or transitions.

Similar field-dependence measurements have been done on the Tb$_2$Ti$_2$O$_7$ single crystal, as shown in Fig. 3. The effects of magnetic field in this sample are also very strong, but weaker than those in Tb$_2$Ti$_2$O$_7$. It is understandable if the low thermal conductivity of Tb$_2$Ti$_2$O$_7$ is due to the strong magnetic scattering on phonons and Tb$_2$Ti$_2$O$_7$ has weaker magnetic fluctuations. Nevertheless, there are several remarkable phenomena in the transport properties of Tb$_2$Ti$_2$O$_7$. First, as shown in Fig. 3(b), the high-field induced enhancement of $\kappa$ for $H \perp [111]$ is several times weaker in Tb$_2$Ti$_2$O$_7$ than that in Tb$_2$Ti$_2$O$_7$. Since half of the Tb$^{3+}$ ions are replaced by Y$^{3+}$, the exchange interactions between magnetic ions are weakened and it is probably difficult to form the field-induced magnetic order of the $\beta$ chains. In this sense, the stronger high-field enhancement of $\kappa$ in Tb$_2$Ti$_2$O$_7$ has some relationship to the spin orders, which can result in weaker magnetic scattering than the isolated magnetic ions in Tb$_2$Ti$_2$O$_7$. Second, the $\kappa(H)$ isotherms for $H \parallel [111]$ show comparably large magnetothermal conductivity but rather different field dependencies, compared with those of Tb$_2$Ti$_2$O$_7$. In particular, each low-$T$ $\kappa(H)$ curve displays two rather broad peaks at about 3 and 11.5 T and a shallow valley at 8–9 T. However, the two peak positions are nearly the same as two of the Tb$_2$Ti$_2$O$_7$ peaks, as shown in Fig. 3(d). This indicates that the $\kappa(H)$ behaviors in $H \parallel [111]$ may have close relationship to the single-ion properties, like the crystal-field excitations.

Figure 4 shows the specific heat of Tb$_2$Ti$_2$O$_7$ and Tb$_2$Ti$_2$O$_7$ single crystals at low temperatures and in the [111] fields. Our zero-field specific heat data of Tb$_2$Ti$_2$O$_7$ are essentially consistent with an earlier work$^{35}$. Two broad peaks or humps at about 0.7 and 6 K could be related to the ground-state and the first-excitation doublets of Tb$^{3+}$ crystal-field levels, broadened by the ex-
change interactions. In magnetic fields along the [111] axis, the two anomaly peaks react sensitively to the field. In high fields, only the high-T peak remains and shifts to higher temperatures, while the low-T one disappears at fields above 2 T. Note that the lowest-T upturns of specific heat in high fields is likely some nuclear spin contribution. In TbYTi2O7, the low-T anomaly moved significantly to ~2 K and the high-T one moves slightly to ~8 K and becomes much weaker in the magnitude. Nevertheless, the specific-heat data do not show any obvious signature for the possible magnetic order and the field-induced changes are mainly related to the crystal-field excitations.

Let us discuss about the possibility of the crystal-field effects on the exotic field dependencies of $\kappa$ in the [111] field. Since the low-field transition to some AF order state can significantly weaken the phonon scattering, a monotonic increase of $\kappa$ with increasing field is expected, similar to the case of $H \perp [111]$. Thus, the high-field behavior of $\kappa$ can occur if there are resonant phonon scatterings by the crystal-field excitations at those particular fields like 6 and 10.5 T, and probably another one higher than 14 T. In this regard, the crystal-field levels of Tb2Ti2O7 were rather clearly determined at least at 4 K. The ground state and the first excitation are non-Kramers doublets with ~18 K separation, and higher levels are singlets. Considering the Zeeman splitting of the doublets, one can figure out two possible phonon resonant scatterings at 0.36 K. The first one is that the splitting of the ground-state doublet can produce a resonant scattering at about 0.2 T, when the energy splitting is about $3.8k_BT$. The second one is expected when the lower branch of the first-excitation level becomes close enough to the ground-state level (with interval of $3.8k_BT$), which occurs at about 16 T. Note that due to the strong anisotropy of the Tb3+ spins, the Landé factor is about 10–14 for the [111] direction and zero for the direction perpendicular to the [111] axis. Therefore, the resonant scattering by the crystal-field excitations can work only for fields along [111]. It is reasonable that only with $H \parallel [111]$ the $\kappa(H)$ and high-field $\kappa(T)$ shows the dip-like features, which could be due to the resonant scattering. However, the above estimated resonant scattering fields are significantly different from the dip fields in $\kappa(H)$.

Another possible reason for the anisotropic field dependence of $\kappa$ is related to the magnetostriction, which was experimentally found to be strongly anisotropic also. The existing data indicated that the longitudinal and transverse magnetostrictions only showed smooth functions of field up to 6 T at 4 K. However, since the magnetoelastic coupling is quickly enhanced with lowering temperatures, some drastic field dependencies of magnetostriction can be expected at subkelvin temperatures. When the remarkable changes of lattice parameters and elastic constants are induced by the magnetic field, lowering crystal symmetry and structural phase transitions could occur in strong field. Such effects on phonon heat transport was recently observed in another rare-earth pyrochlore Dy2Ti2O7. Nevertheless, very-low-T measurements of the magnetostriction would be useful for clarifying the mechanism of the magnetothermal conductivity effect in Tb2Ti2O7.

IV. SUMMARY

An extremely low thermal conductivity was observed in Tb2Ti2O7 single crystals. The short mean free path of phonons points to a phonon-glass-like behavior of this material. Both the field dependencies of $\kappa$ and the comparison with Y2Ti2O7 and TbYTi2O7 indicate that the phonons are strongly scattered by the spin fluctuations, whereas the field-induced changes of magnetism are remained to be investigated.

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Our specific heat data of $Y_2Ti_2O_7$ show a simple lattice contribution at low temperatures. Fitting the data to the formula $C = \beta T^3 + \gamma T^5 + \delta T^7$, with $\delta = 1.55 \times 10^{-3}$ $J/K\cdot mol$. This value is considerably smaller than that of $Lu_2Ti_2O_7$. It seems to be also smaller than an earlier result of $Y_2Ti_2O_7$, which gave $\delta = 2.36 \times 10^{-4}$ $J/K\cdot mol$ if one used the same fitting formula. Even though this $\delta$ value corresponds to a phonon mean free path 3 times smaller than that in Fig. 1(a), our conclusion is essentially unchanged, considering the difference between $Tb_2Ti_2O_7$ and $Y_2Ti_2O_7$ is several orders of magnitude.

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