PHYSICAL REVIEW B VOLUME 23, NUMBER 8 15 APRIL 1981

Glassy behavior of crystalline solids at low temperatures

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(Received 24 October 1980)

Measurements of low-temperature specific heat C, thermal conductivity κ, and dielectric dispersion have been made on crystalline samples of the superionic conductor Li_3N, the oxygen conductor Y_2O_3-stabilized ZrO_2, and the relaxation ferroelectric $Pb_3(MgNb_2)O_9$. In each case, the behavior is similar to that found in amorphous materials. Data on other crystalline materials are reviewed to emphasize that the low-temperature phenomena found in glasses can occur in crystals. It is shown that a peak in C/T^3 appears to be correlated, in both amorphous and crystalline materials, with a nearly temperature-independent regime (plateau) in κ.

I. INTRODUCTION

It is now established that most amorphous materials harbor localized excitations which dominate many of the low-temperature properties.[1] These excitations may be represented by a ground state plus an excited state of energy E which is well separated from any states of higher energy. The energy distribution $n(E)$ is very broad, is only weakly dependent on E, and is of roughly the same magnitude for most glassy materials. A phenomenological model[2,3] assumes that the two-level excitations arise from the quantum-mechanical tunneling of some entity between potential-energy minima. This model has been useful in the correlation of various low-temperature measurements.

From the time that this low-temperature behavior was first indicated by Zeller and Pohl,[4] the question has been asked as to whether similar excitations exist in crystalline materials. If they did, it might be easier to identify the physical nature of the localized excitation. In one early measurement, for example, it was shown[5] that a compositionally disordered crystal of KBr-KI did not exhibit glasslike behavior in thermal conductivity or specific heat. Thus, it appeared that topological disorder is needed. In this paper we present data on four crystalline materials, and review briefly other published experimental data. It will be noted that "glassy" behavior is well established for three crystalline solids, namely, β-alumina, Li_3N and neutron-irradiated SiO_2, and probably occurs in a variety of other crystalline materials.

We first indicate more explicitly what is meant by "glassy" behavior. The specific heat C includes a contribution C_{ex} which, in magnitude and temperature dependence, is roughly 10^{-5} T (J/K cm^3). This contribution is most evident at temperatures below $\approx$1 K. The thermal conductivity κ is roughly quadratic in T at temperatures below 1 K, $\kappa \approx 10^{-4}$ T^2(W/cm K). The dielectric constant ϵ is strongly temperature and frequency dependent relative to the dependence in a pure crystal. The dielectric constant ϵ passes through a minimum at a temperature which is determined by the measuring frequency, but this typically occurs near $\approx$0.1 K at $\approx 10^4$ Hz. It is generally assumed that the anomalous behavior of the thermal conductivity and other properties is associated with the same excitations, or some subset of the excitations, which cause the "linear" term in the specific heat. Finally, the thermal conductivity is nearly temperature independent over a range of temperatures near 10 K. This feature has been referred to as a "plateau," and may or may not be directly related to the other properties discussed above.

The crystal which has been studied most extensively is the two-dimensional superionic conductor M β-alumina. This is a layered material with the mobile cations M lying on monatomic planes separated by $\approx$10-Å-thick layers of alumina crystal. The conducting planes are highly nonstoichiometric having an excess of 15–30% cations. As the crystal is cooled, the cations remain disordered, unlike those in most other superionic conductors. Early specific-heat measurements[6] above 1 K suggested the presence of a term linear in T typical of a glass. The glasslike behavior was confirmed by specific-heat measurements to lower temperatures,[7] and by measurements of thermal conductivity,[8] dielectric dispersion,[9,10] and ultrasonic dispersion.[11] The localized excitations have been studied by electron-spin relaxation,[12] and the two-level character verified by the observation of the saturation of dielectric attenuation.[10]

In brief, the β-alumina exhibit the same low-temperature properties as glasses, except certain properties are anisotropic. From the anisotropic dielectric behavior[9] it is known that motion associated with the excitations is directed parallel to the conducting plane. From the sensitivity of the thermal conductivity to the cation present,[8] it is likely that the motion involves the cations rather than some other entity in the crystal. It would now be desirable to reduce the disorder in the conducting plane by improving the stoichiometry, but such crystals are difficult to grow.

II. EXPERIMENTAL RESULTS

A layered superionic conductor[13] having a nonstoichiometry of only $\approx 1\%$ is Li_3N. Like β alumina there is no phase transition indicating ordering of the mobile ions. Again, early specific-heat measurements[14] above 1 K suggested a low-temperature term linear in T, and dielectric measurements on small crystallites indicated "glassy" behavior.[15] We have therefore measured the specific heat, thermal conductivity, and dielectric dispersion of lower temperatures on polycrystalline samples. The specific-heat data are shown in Fig. 1 plotted as C/T^3 to emphasize the large departure below 10 K from the $C \propto T^3$ Debye behavior. The low-temperature data make a reasonable extrapolation to the higher temperature data of Ref. 14. Below 1 K, $C_{ex} \approx 2 \times 10^{-5}\ T^{0.8}$ (J/cm^{3}K), a behavior not unlike that found in glasses. Dielectric data are presented in Fig. 2. The frequency and temperature dependence of ϵ is also similar to that observed in glasses such as BK7.[16]

The thermal conductivity of our Li_3N samples is shown in Fig. 3. The temperature dependence is

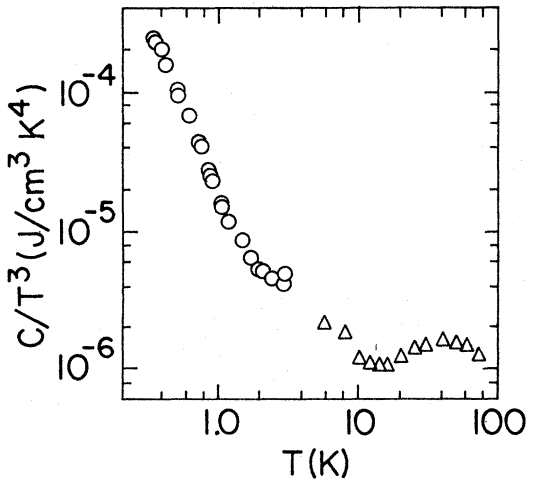

FIG. 1. Specific heat of Li_3N. The data have been divided by T^3 to emphasize departure from a T^3 Debye behavior. ○, present data; Δ, data from Ref. 14.

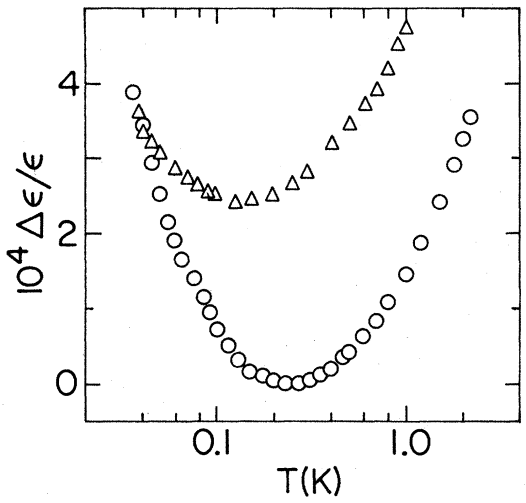

FIG. 2. Variation of dielectric constant ϵ of polycrystalline Li_3N with frequency and temperature. Δ, 1.3×10^3 Hz; ○, 10^4 Hz.

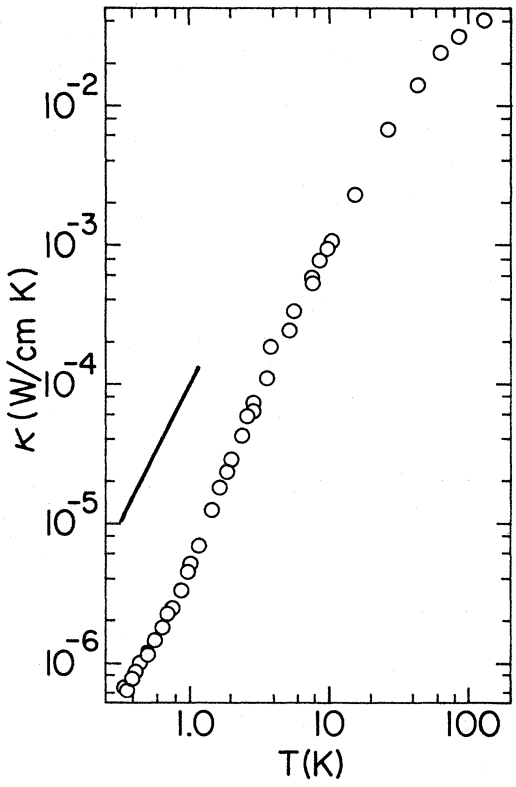

FIG. 3. Thermal conductivity of Li_3N. The solid line is $\kappa = 10^{-4}\ T^2$ (W/cm K), which is typical for glassy materials at $T \lesssim 1$ K. The polycrystalline Li_3N sample was mounted using a drybox. After these data were obtained, the sample was exposed to moist air for 2.5 days. There was no change in thermal conductivity or in specific heat.

roughly T^2 as observed for glasses, but the magnitude is a factor of ≈ 10 too small and there is no plateau for $T \geq 10$ K. From the change in temperature dependence seen near 1 K, we suspect that thermal phonons are being scattered by voids[16a] of size ≈ 100 Å producing a frequency-independent phonon mean free path of ≈ 400 Å for $T > 1$ K. This assumed porosity would give an apparent mass density for the sample of 1.08 g/cm^{-3}, which is indeed close to the measured density of 1.065 g/cm^3. The x-ray density is 1.29 g/cm^3.

Dielectric and ultrasonic measurements have been made recently[17] on a single crystal of Li_3N. In the ultrasonic measurements, glassy behavior was observed only if the acoustic wave caused atomic motion in a direction parallel to a Li_2N layer. Hence the behavior is similar to that of β-alumina. It has been suggested[17] that the localized excitation in Li_3N may involve the quantum-mechanical tunneling of hydrogen impurities.

Zirconium oxide ZrO_2, when stabilized in the fluorite structure with ≈ 8 wt.% Y_2O_3, is an oxygen-ion conductor[18] and contains a disordered array of oxygen vacancies. Specific-heat measurements[19] above 1 K showed a low-temperature term linear in T. We have therefore measured the specific heat, thermal conductivity, and dielectric dispersion to much lower temperatures. The specific-heat data of Fig. 4 agree with the earlier measurements at higher temperatures. Below 1 K, $C_{ex} \approx 6 \times 10^{-5}$ T (W/cm^3 K). Hence the magnitude of the density of states $n(E)$ is somewhat larger than in many glasses, yet $n(E)$ is nearly independent of E as for glasses. Indeed the dielectric constant, Fig. 5, has a temperature and frequency dependence like that of a glass.

The thermal conductivity data for ZrO_2:Y_2O_3 are shown in Fig. 6. Over most of the temperature range $\kappa \approx 10^{-4}$ T^2 (W/cm K) as for a glass. Near 30 K there occurs a plateau as for a glass, though at a higher temperature. Below 0.3 K the temperature dependence of κ approaches T^3, which is probably caused by phonons scattering from voids having a diameter larger than the phonon wavelength. Indeed, when the sample was fractured, voids of average radius ≈ 1 μm were observed in a scanning electron microscope. The measured mass density of 5.68 g/cm^3 versus an x-ray mass density of 6.03 g/cm^3 leads to a void density of $\approx 3 \times 10^{10}$ cm^{-3}, or a phonon mean free path of ≈ 20 μm. This is close to the mean free path deduced from the data of Fig. 6, namely, ≈ 40 μm. The thermal conductivity corresponding to this mean free path is indicated by the dotted line in Fig. 6.

We had been studying the magnetic behavior of a polycrystalline perovskite material $Pb_2(MnTa)O_6$, or PMT. The magnetic susceptibility[20] and large specific heat[20,21] are indicative of spin-glass behavior similar to that observed in dielectric MnO glasses.[22,23] The thermal conductivity is shown in Fig. 7. Below 1 K, $\kappa \approx 10^{-4}$ T^2 (W/cm K) as for a glass. We had not expected to observe such "glassy" behavior since the PMT sample consisted of 5 μm grains of a single cry-

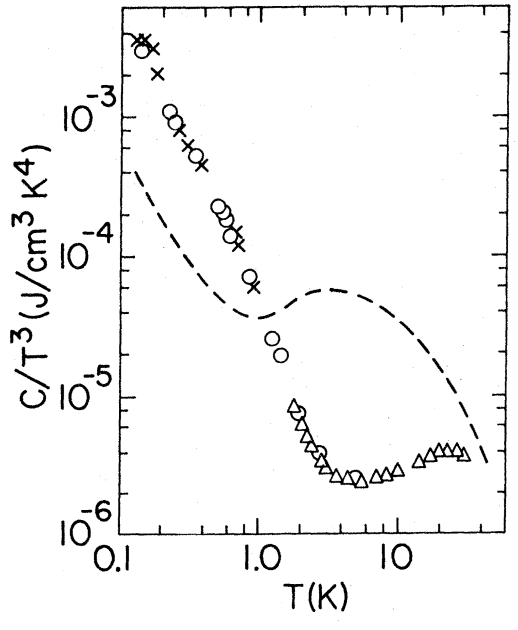

FIG. 4. Specific heat, divided by T^3, of Y_2O_3—stabilized ZrO_2. X, O, present data; Δ, data from Ref. 19. Data represented by O were obtained with an adiabatic calorimeter, while data represented by X were obtained with a diffusive technique. The dashed line shows the specific heat of an amorphous polymer, from Ref. 51.

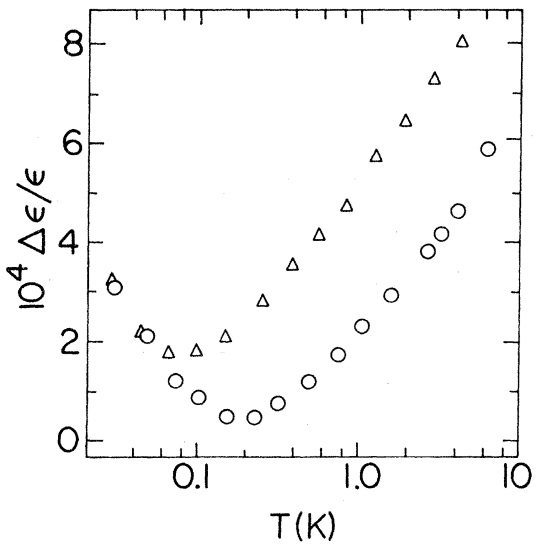

FIG. 5. Variation of dielectric constant of ZrO_2:Y_2O_3 with frequency and temperature. Δ, 10^3 Hz; O, 10^4 Hz.

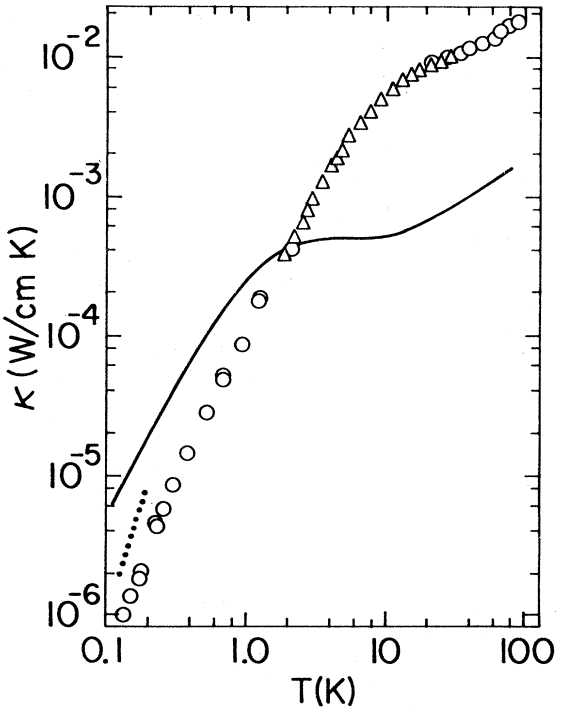

FIG. 6. Thermal conductivity of ZrO_2:Y_2O_3. The two sets of data were obtained in different laboratories. The dotted line represents the thermal conductivity if phonons were scattered only by the voids present in the sample. The thermal conductivity of an amorphous polymer is shown by the solid line, from Ref. 51.

stalline phase, with no x-ray evidence for the presence of a glassy phase. Dielectric data over the temperature range 1–200 K indicate that PMT is a relaxation ferroelectric.[20]

To determine if the thermal transport in PMT was associated with magnetic or nonmagnetic behavior,

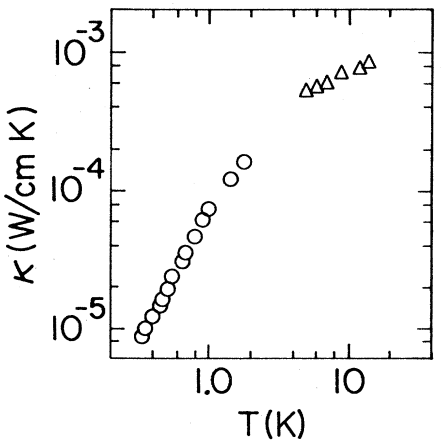

FIG. 7. Thermal conductivity of PMT. ○, present data; Δ, data from Ref. 21.

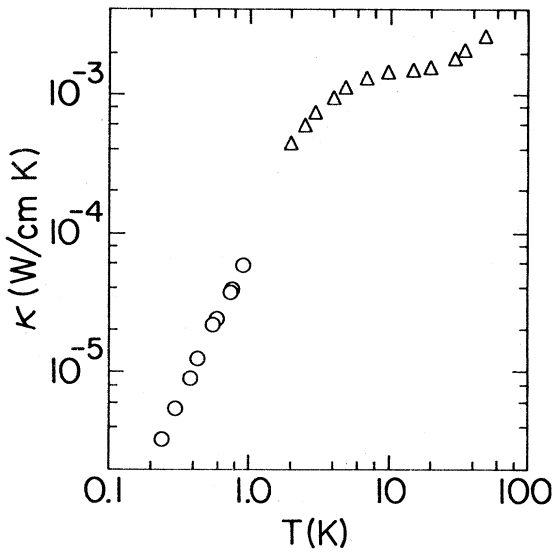

FIG. 8. Thermal conductivity of PMN. The two sets of data were obtained in different laboratories.

we have investigated another relaxation ferroelectric, $Pb_3(MgNb_2)O_9$ or PMN.[24,25] The magnetic MnO of the PMT has essentially been replaced by nonmagnetic MgO in PMN. The thermal conductivity, Fig. 8, again is that of a glass with $\kappa \approx 0.6 \times 10^{-4}\ T^2$ (W/cm K) below 1 K and a plateau near 15 K. Below 1 K, $C_{ex} \approx 4 \times 10^{-5}\ T$ (J/cm^3 K) as shown in Fig. 9. The temperature dependence and magnitude of C are close to those found in glasses.

The low-temperature dielectric behavior of PMN is shown in Fig. 10. The frequency and temperature dependence are typical of a glass.[26] However, the magnitude of ϵ near 0.1 K is $\approx$300. Thus $\Delta\epsilon$ is

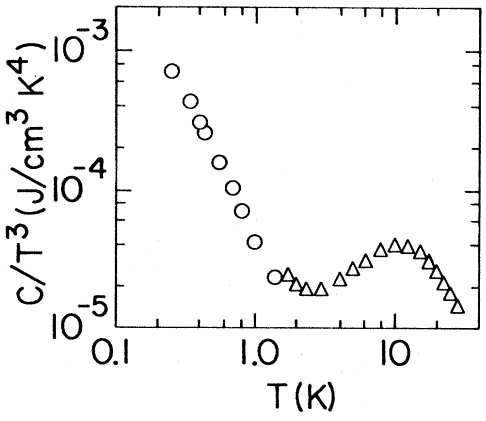

FIG. 9. Specific heat, divided by T^3, of PMN. The two sets of data were obtained in different laboratories. The low-temperature data (○) were measured with a diffusive technique, and thus may be in error by a constant, multiplicative factor of $\approx$1.

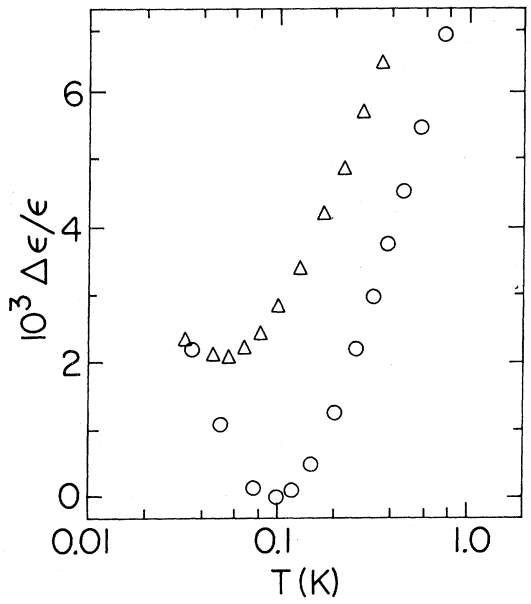

FIG. 10. Variation of dielectric constant of PMN with frequency and temperature. Δ, 10^3 Hz; ○, 10^4 Hz.

much larger than in glasses or even the β-aluminas. The obvious implication is that the basic electric dipoles of this ferroelectric are involved in the motion associated with the localized excitations. A "two-phase" model has been suggested[24,25] to explain the dielectric behavior of PMN, and the excitations may be related to fluctuations between these "phases."

A similar suggestion was advanced to explain the glasslike properties of a single-crystal sample of superconducting ZrNb. Both the thermal conductivity and specific heat of ZrNb had temperature dependencies and magnitudes close to those found in amorphous dielectrics.[27] The excitations responsible for this behavior were believed to arise from fluctuations between the β-phase matrix and inclusions of a ω phase formed by quenching the sample from elevated temperatures. Some doubt was cast on this interpretation when similar behavior in supreconducting TiNb, which also contained the ω configuration,[28] and in superconducting V_3Si (Ref. 29) was attributed to normal inclusions within the superconductor. Nevertheless, it has been demonstrated more recently that the ultrasonic attenuation of polycrystalline ZrNb containing the ω configuration is very similar to that for an amorphous superconducting alloy.[30]

For completeness we note that crystalline SiO_2, having some disorder introduced by neutron irradiation, has also been studied carefully. Measurements above 1 K showed that the thermal conductivity was reduced and approached that of fused silica with increasing irradiation.[31,32] Measurements on thermal conductivity extending to lower temperatures,[33] as well as measurements of specific heat,[33] the saturation of ultrasonic attenuation,[34] and the observation of phonon echoes[35] have confirmed the glassy behavior of neutron-irradiated SiO_2 crystals. It should be noted that the crystals were not irradiated to the point of losing their discrete x-ray diffraction characteristics. The localized excitations seem to reside within microscopic volumes of heavy damage, and are not distributed uniformly throughout the crystalline sample.

We also note that a single crystal of YB_{66} had a low- temperature thermal conductivity[36] of $\approx 10^{-4}\ T^2$ (W/cm K) as for a glass. The authors state that the crystal was disordered, that certain boron groups had a choice of positions. Other properties, which would help confirm the "glassy" behavior, appear not to have been measured.

It is unfortunate that many of the measurements discussed in this paper have been made only on polycrystalline samples. Specific-heat measurements[37] on GeO_2 and other polycrystalline or partially crystallized samples[38,39] at temperature above 1.5 K exhibit a term linear in T which is of nearly the same magnitude in both vitreous and crystalline samples. It has been suggested[38,39] that the excitations responsible for this specific heat may exist at the grain boundaries. Even in totally amorphous materials, it has been proposed that the tunneling states may occur at boundaries on ordered regions of microscopic size.[40]

Nevertheless it would be expected that less disordered material would appear in crystallized samples leading to a reduction in the average specific heat from this contribution. Measurements on other polycrystalline materials[5,41–43] to temperatures below 1 K provide no definitive evidence for "glassy" behavior. Thus, if grain boundaries do support localized excitations, the exictations occur only for select materials. In the present measurements, if the excitations did occur at grain boundaries, the excitations would be physically separated by only ≈ 3 Å for 5-μm crystallites assuming the distribution $n(E)$ extended to $E = 10$ K. This is a much higher density of excitations than observed, for example, on the conducting planes of the β-aluminas. Thus we suspect that the excitations are occurring within the crystalline material.

III. DISCUSSION, $T \lesssim 1$ K

It has been noted above that "glassy" behavior does exist in crystalline β alumina, Li_3N and neutron-irradiated SiO_2, and that there is strong evidence that glassy behavior also exists in ZrNb, ZrO_2:Y_2O_3, PMN and possibly other crystalline materials such as PMT and YB_{66}. The phenomenon would appear to be rather pervasive. Although the microscopic description of a localized excitation must

be different for different crystals, just as it must be different for different amorphous materials, the macroscopic behavior is essentially the same. Hence the same theoretical formulation, such as the tunneling-states model of Refs. 2 and 3, should apply to either amorphous solids or to disordered crystalline solids.

The existence of low-energy excitations associated with impurities or defects in crystals is now new. Excitations associated with the tunneling of impurities[44,45] or the motion of lattice defects[46] are well established. But these excitations generally occur with discrete energy levels, whereas to explain the behavior of glassy materials a broad, nearly energy-independent spectrum is required. It is possible that the broad spectrum arises from electric or elastic interactions with other localized excitations or with different defects. The broad spectra found in β-alumina[6] and in ZrO_2:Y_2O_3 (Ref. 19) have been attributed to random electric fields created by the ions and/or charge-compensating defects. The possibility of elastic interactions has also been discussed.[47]

Although it is not our intent to provide a quantitative comparison between the tunneling-states model and the present data, we will comment briefly on the coupling constant γ between a phonon and a localized excitation. An estimate[48] of γ may be obtained from the density of states $n(E)$ as deduced from C (where $C \propto T$) and from the phonon mean free path as deduced from κ (where $\kappa \propto T^2$). The result is $\gamma \approx 0.5$ eV for both ZrO_2:Y_2O_3 and PMN. This is roughly the same magnitude as found for the β aluminas,[9] or even for various amorphous polymers,[49,50] and adds further to our conclusion that the low-temperature behavior of ZrO_2:Y_2O_3 and PMN is in fact similar to that observed in amorphous materials.

IV. DISCUSSION, $T \gtrsim 1$ K

The most distinguishing feature of amorphous materials at $T > 1$ K has been the plateau in the thermal conductivity. It now appears that a plateau may also occur in those crystals otherwise showing evidence of glassy behavior. Furthermore, the plateau for both amorphous and crystalline materials appears to be correlated with a maximum in C/T^3. This has become more evident in light of the present data because the plateaus for crystalline solids can occur at higher temperatures than in most amorphous materials. To provide an example, the specific heat and thermal conductivity of an amorphous polymer[51] are shown in Figs. 4 and 6, respectively. The temperatures at which the peak in C/T^3 or the plateau in κ occur are summarized in Table I for this polymer as well as for silicate glass,[52] amorphous As,[53] and a glassy metal.[54,55] Also included in Table I are several crystalline materials.

The correlation found in Table I between the plateau in κ and a peak in C/T^3 is interesting since the plateau has yet to be explained, although several suggestions have been advanced.[53] The peak in C/T^3 may have a satisfactory explanation[56] in a low-frequency phonon mode which can be approximated by an Einstein oscillator.[6,57] The implication is that the dispersion causing the peak in C/T^3 is intimately related to the strongly frequency-dependent phonon scattering[58] which causes the plateau. Dispersion has been incorporated into a theoretical attempt to explain the plateau,[59] and it would appear that further work in this direction should be encouraged. The plateau in κ cannot be viewed, as was suggested some years ago,[60] simply as arising from the low-frequency modes which resonantly scatter acoustic

TABLE I. A comparison between the temperature T_c at which a peak in C/T^3 occurs, and the temperature T_κ at which the plateau in κ occurs, for both amorphous and disordered crystalline materials.

Material	T_c (K)	T_κ (K)	Ref.
Amorphous epoxy	3.5	4	51
Silicate glass	10	10	52
Glassy As	5	5	53
Glassy PdSi	$\approx$10	15	54,55
Ag β-alumina	7	7	6,8
Na β-alumina	8,20	$\approx$20 (broad)	6,8
K β-alumina	30	(possible depression near 30)	6,8
Li β-alumina	(none below $\approx$40)	(inflection near 50)	6,8
PMN	11	$\approx$12	. . .
ZrO_2:Y_2O_3	24	$\approx$30	. . .
Li_3N	40	. . .	14
YB_{66}	. . .	100	36

thermal phonons, as the scattering has been observed to be inelastic.[61,62]

In summary, it has been shown through a review of existing data and the presentation of new data that the low-temperature properties common to nearly all amorphous materials also occur in a variety of crystalline materials. In a crystalline material it should be easier to identify the localized excitation responsible for these properties. It has also been shown that a correlation appears to exist, for both amorphous and crystalline materials, between a peak in C/T^3 and a plateau in the thermal conductivity.

ACKNOWLEDGMENTS

The authors thank L. E. Cross of Pennsylvania State University for supplying the PMN sample, D. Thompson of Corning-Zircoa Corporation for supplying the ZrO_2:Y_2O_3 sample, R. O. Bach of Lithium Corporation of America for supplying the Li_3N samples, and C. F. Clark of Lake Short Cryotronics for technical assistance. This work was supported in part by the U.S. Department of Energy under Contract No. DE-AC02-76ER01198 and by the National Science Foundation under Grant No. DMR-8006941.

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