Anomalous low-temperature heat capacity in antiperovskite compounds*

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The low-temperature heat capacities are studied for antiperovskite compounds AXM_3 (A = Al, Ga, Cu, Ag, Sn, X = C, N, M = Mn, Fe, Co). A large peak in $(C-\gamma T)/T^3$ versus T is observed for each of a total of 18 compounds investigated, indicating an existence of low-energy phonon mode unexpected by Debye T^3 law. Such a peak is insensitive to the external magnetic field up to 80 kOe (1 Oe = 79.5775 A·m^{-1}). For compounds with smaller lattice constant, the peak shifts towards higher temperatures with a reduction of peak height. This abnormal peak in $(C-\gamma T)/T^3$ versus T of antiperovskite compound may result from the strongly dispersive acoustic branch due to the heavier A atoms and the optical-like mode from the dynamic rotation of XM_6 octahedron. Such a low-energy phonon mode may not contribute negatively to the normal thermal expansion in AXM_3 compounds, while it is usually concomitant with negative thermal expansion in open-structure material (e.g., ZrW_2O_8, ScF_3).

Keywords: low-temperature heat capacity, low-energy phonon modes, antiperovskite compound, negative thermal expansion

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1. Introduction

Heat capacity is a fundamental property of material, which measures the ratio of the heat added to (or removed from) an object to the resulting temperature change. In the high temperature range, the heat capacity C of a solid can be very well described by Dulong–Petit law, i.e., $C = 3Nk_B$,[1] where N is the number of atoms in the solid and k_B is Boltzmann constant. At low temperatures, if electronic and other non-phonon contributions are subtracted, the specific heat obeys the Debye T^3 law.[2] In amorphous solid, an excess specific heat, a hump in C/T^3 versus T curve, was widely observed due to the low-energy vibrational modes that are not considered by the Debye model.[3] These modes are also seen in inelastic neutron scattering and Raman scattering, which is known as "boson peak" because in Raman spectrum the intensity of the related low-energy peak takes a Bose distribution with temperature.[4,5] The mechanisms responsible for these low-energy phonon modes have been discussed in terms of localized vibration "floppy modes", domain wall motions of the glassy mosaic structure, transverse phonon modes, etc.[6–9] Though rare, a peak in C/T^3 versus T curve has also been observed in crystal and attributed either to a specific structural feature,[10–12] or to the geometrical frustration,[13] or to Van Hove singularities of the phonon spectrum.[9,14]

In a compound with open framework in crystal structure, such as ZrW_2O_8[10,15,16] and ScF_3,[17,18] the low-energy vibrations manifested by excess specific heat were argued to be crucial to the occurrence of negative thermal expansion (NTE). Recently, Mn-based antiperovskite compounds $AXMn_3$ (A: transitional metals or semiconducting elements; X represents nitrogen or carbon) have attracted a lot of attention due to their multi-functionalities, such as NTE,[19–21] magnetocaloric and barocaloric effects,[22–25] nearly zero temperature coefficient of resistivity.[26–28] Although the antiperovskite compounds possess such rich physical properties,[29,30] they each have a simple crystal structure as shown in Fig. 1. The crystal symmetry of $AXMn_3$ compound (space group, Pm-3m) is the same as that of ScF_3, but the mechanism of NTE differs essentially from ScF_3. For $AXMn_3$, the gradual lattice contraction due to Γ^{5g}-type antiferromagnetic (AFM) to paramagnetic (PM) (AFM–PM) transition overcomes the usual thermal expansion upon heating, leading to a large NTE.[31] However, it has not yet been understood whether the excess of specific heat exists in $AXMn_3$ as it does in ScF_3 and how it contributes to the overall thermal expansion. Here we report the analysis of

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low-temperature specific heat data of a total of 18 Mn-, Fe-, and Co-based antiperovskite compounds. A large excess of low-temperature specific heat beyond the Debye contribution is observed in each of the compounds investigated. Moreover, after subtracting the electronic heat γT (γ is the coefficient of electronic heat), the peak in $(C-\gamma T)/T^3$ versus T curve shifts towards higher temperatures in the compound with smaller lattice constant, which is consistent with the "blue" shift of boson peak frequency with pressure.[32,33] The origin of low temperature specific heat anomaly is analyzed and discussed. Our results suggest the excess phonon mode in antiperovskite compound is not necessarily associated with NTE.

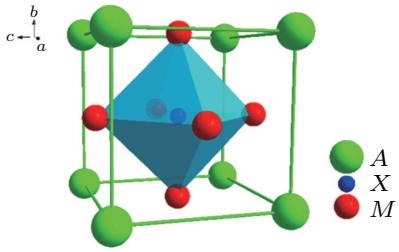


Fig. 1. (color online) The structure of antiperovskite compound AXM_3 for room temperature. Atoms A (transitional metals or semiconducting elements) occupy the cubic corner sites. Interstitial atom X (C, N) is at the body-centered position and transitional metals X atoms (Fe, Co, Mn) occupy the face-centered sites. The X and M atoms form an XM_6 octahedron.

2. Materials and methods

All polycrystalline samples were made by standard solid state reaction and the structural characterizations and their physical properties have been reported elsewhere.[34–43] Take antiperovskite compound $GaCMn_3$ for example, the starting materials including powders of manganese (4N), graphite (3N), and gallium (5N) were mixed in a stoichiometric proportion and sealed in an evacuated quartz tube. The sample was sintered in a box furnace at 1073 K–1123 K for 8 days, then cooled down to room temperature. After it was fully ground, the powder was pressed into pellets, sealed in an evacuated quartz tube and sintered at 1073 K–1123 K for another 8 days sequentially.[41] Meanwhile for $ANMn_3$ sample, the precursor Mn_2N must be prepared first, and the following steps are the same as those of synthesis for $ACMn_3$.

X-ray diffraction patterns at room temperature were collected in a Philips X'pert PRO X-ray diffractometer with Cu Ka radiation. For these samples, x-ray diffraction peaks can be indexed with the cubic antiperovskite structure (space group, $Pm-3m$).[34–43] In some $AXMn_3$ compounds, a small amount of MnO impurity (less than 3%) may exist,[38,43] which does not affect the specific heat analysis. The heat capacity measurements were performed by a relaxation method using a commercial Quantum Design Physical Property Measurement System (PPMS) (1.8 K$\leq T \leq$ 800 K, $0 \leq H \leq$ 90 kOe). Heat specific data for most samples were reported in previous work,[34–41] and all the original heat capacities are plotted in supplementary materials.

3. Results and discussion

The antiperovskite compounds are metallic and have good electrical and thermal conductivities. Thus specific heat arising from conduction electrons cannot be ignored. The coefficient of electronic heat γ can be obtained by a linear fitting to the C/T–T^2 plot ($\leq$ 10 K). The typical fitting profiles for $Ga_{0.7}NMn_{3.3}$, $CuNMn_3$, and $AgNMn_3$ can be found in Fig. 2 as examples. In Fig. 3, the measured specific heat is plotted as $(C-\gamma T)/T^3$ versus T for each of the compounds investigated. For a normal metal, if other contributions are not taken into account, the low-temperature $(C-\gamma T)/T^3$ should take a constant value at low temperatures since the electronic contribution is subtracted. However, a clear peak is observed for each of the samples, though the peak height and position vary from case to case. Such an excess specific heat obviously exceeds the anticipation of Debye model.

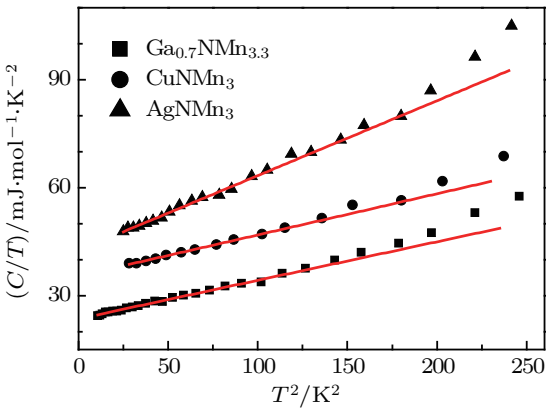


Fig. 2. (color online) Linear fittings (solid lines) to C/T versus T^2 data below 10 K for $Ga_{0.7}NMn_{3.3}$, $CuNMn_3$, and $AgNMn_3$.

As seen in Fig. 3(a), the peak in $(C-\gamma T)/T^3$ versus T curve is sensitive to the element at A site in $ANMn_3$, i.e., the peak becomes progressively higher in magnitude and shifts towards lower temperatures as the atomic mass of A site increases. Similarly, when Ga (Ag) is partly replaced by Mn in $GaNMn_3$ ($AgNMn_3$), the peak shifts towards higher temperatures with a reduction of peak height as shown in Fig. 3(b). Figure 3(c) shows that partially or totally substituting the body-centered C with N also leads to a shift of the peak in $(C-\gamma T)/T^3$ versus T towards lower temperatures and enhances the peak height. As shown in Fig. 3(d), however, the peak is less sensitive to the atom at the face-centered positions than to those at A and X sites. Particularly, for $GaCM_3$ (M = Fe, Co or Mn) the peak position is almost invariable when M changes.

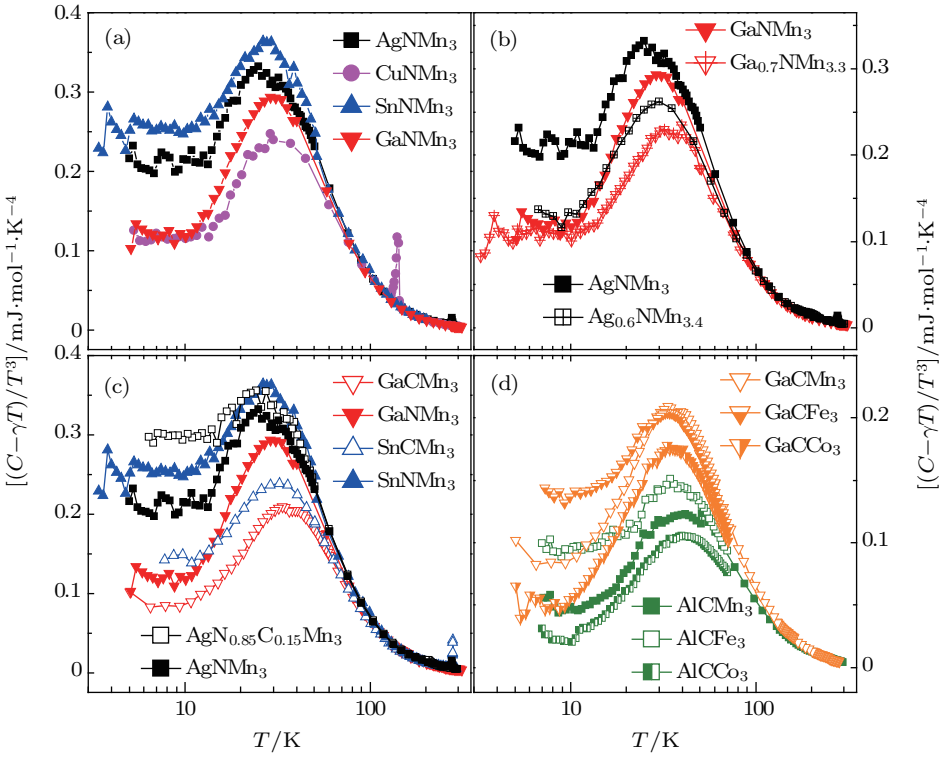


Fig. 3. (color online) Specific heat plots of $(C-\gamma T)/T^3$ versus T for antiperovskite compounds, $ANMn_3$ (A = Ag, Sn, Cu, Ga) (a), $Ga_{1-x}Mn_xNMn_3$ ($x = 0$, 0.3), and $Ag_{1-x}Mn_xNMn_3$ ($x = 0$, 0.4) (b), GaXMn$_3$, SnXMn$_3$, and AgXMn$_3$ (X = N, C) (c), and GaCM$_3$ and AlCM$_3$ (M = Fe, Co, Mn) (d).

Most of the antiperovskite compounds are magnetic in the ground state. As is well known, the heat capacity due to magnetic contribution becomes significant at very low temperatures where the phonons are frozen or near transition temperature.[2] But the observed peak temperature related to the excess specific heat is neither too low nor near the transition temperature in our study, indicative of a non-magnetic mechanism. We measure the specific heat under high magnetic field of 50 kOe for GaCMn$_3$ and 80 kOe for GaNMn$_3$ (Fig. 4). It is found that the external magnetic field has little influence on the specific heat. For both compounds, the $(C-\gamma T)/T^3$ versus T measured under high magnetic fields overlaps well with the zero-field curve. The immunity of low-temperature specific heat to external magnetic field indicates again that the excess specific heat is not associated with magnetism.

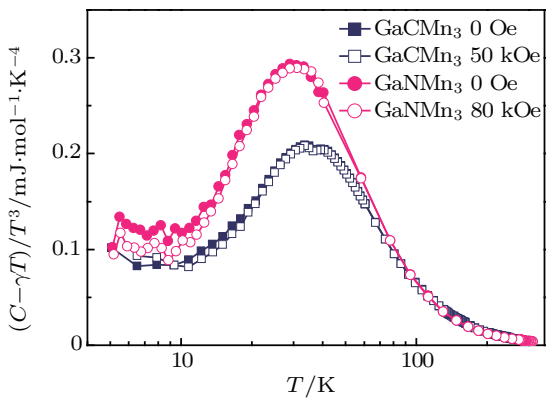


Fig. 4. (color online) A comparison of specific heat plot of $(C-\gamma T)/T^3$ versus T for GaCMn$_3$ and GaNMn$_3$ between in zero field and under high magnetic field (50 kOe or 80 kOe).

The peak-like feature in $(C-\gamma T)/T^3$ versus T can be treated as an Einstein contribution with single frequency as has been reported for other materials with boson peak.[10,13,17] In order to separate the Debye contribution ($C_{\rm D}$) from the Einstein one ($C_{\rm E}$), a fitting taking both contributions into account is carried out with the formulation, $C(T) = p_{\rm D}C_{\rm D} + p_{\rm E}C_{\rm E}$,[15] where the p coefficients are the oscillator strengths per unit cell. $C_{\rm D}$ takes the expression of

$$C_{\rm D} = 3Nk_{\rm B}\left(\frac{T}{\Theta_{\rm D}}\right)^3 \int_0^{\Theta_{\rm D}/T} \frac{\xi^4 {\rm e}^{\xi}}{({\rm e}^{\xi}-1)^2}{\rm d}\xi, \qquad (1)$$

where Debye temperature $\Theta_{\rm D} = \hbar\omega_m/k_{\rm B}$, $\xi = \hbar\omega/k_{\rm B}T$, $\hbar$ is the Planck constant, ω is the vibration frequency, and $\omega_{\rm m}$ is cut-off frequency. $C_{\rm E}$ is determined by

$$C_{\rm E} = Nk_{\rm B}\frac{(\hbar\omega_{\rm E}/k_{\rm B}T)^2\,{\rm e}^{\hbar\omega_{\rm E}/k_{\rm B}T}}{({\rm e}^{\hbar\omega_{\rm E}/k_{\rm B}T}-1)^2}, \qquad (2)$$

where $\omega_{\rm E}$ is the Einstein frequency.[1]

Typical fitting profiles can be found in Figs. 5(a) and 5(b) for GaNMn$_3$ and Ag$_{0.6}$Mn$_{3.4}$, respectively. A sum (solid line) of $C_{\rm D}$ (dashed-dotted line) and $C_{\rm E}$ (dotted line) explains the experimental curve (open circle) well. As shown in Fig. 5(a), for GaNMn$_3$, the Debye contribution provides a flat table below $\sim$ 40 K, whereas an Einstein contribution, $C_{\rm E}$, is responsible for the peak at $\sim$ 29 K ($\hbar\omega_{\rm E} = 12.5$ meV) in $(C-\gamma T)/T^3$ versus T. For Ag$_{0.6}$Mn$_{3.4}$, the best fitting yields $\Theta_{\rm D} = 422$ K, and $\hbar\omega_{\rm E} = 12.8$ meV. Figure 6 presents a summary of $\hbar\omega_{\rm E}$ as a function of lattice constant (a_0) for all 18 antiperovskite compounds. Roughly, $\hbar\omega_{\rm E}$ is inversely proportional to a_0, namely,

the Einstein peak shifts towards higher temperatures (higher $\hbar\omega_{\rm E}$) when the lattice constant becomes smaller. This is in good agreement with the trends shown in Figs. 3(a) and 3(b) that a smaller $\hbar\omega_{\rm E}$ corresponds to a heavier A atom which has a larger atomic radius and thus a larger lattice constant. This effect can be considered as a chemical pressure effect, similar to the "blue" shift of boson peak frequency under pressure in glass.[32] The pressure changes the elastic constant and the density which can influence the position of the excess heat capacity peak.[14]

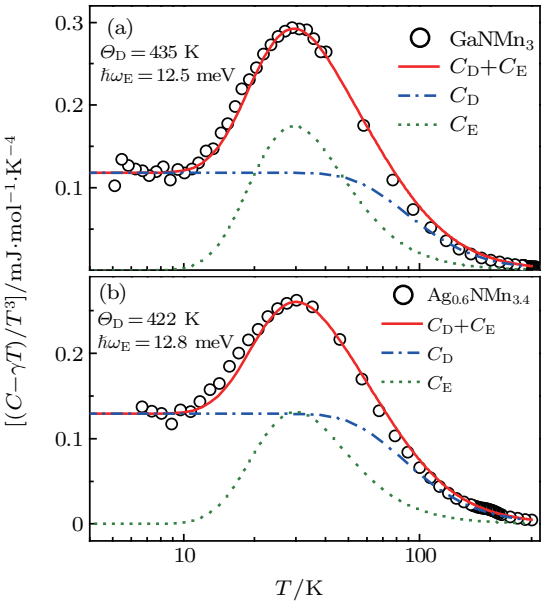


Fig. 5. (color online) Plots of $(C-\gamma T)/T^3$ versus T with a combination of both Debye and Einstein models for GaNMn$_3$ (a) and Ag$_{0.6}$NMn$_{3.4}$ (b). The solid line refers to the total contribution from both models, and the dashed-dotted and dotted lines represent the individual contributions from Debye ($C_{\rm D}$) and Einstein ($C_{\rm E}$) terms, respectively. The open circles denote the experimental data.

For materials with open or flexible crystal structures, the linkages and rotations of the polyhedral/octahedral network are likely to accommodate transverse vibrational modes, which can lead to significant negative contributions to the thermal expansions.[15–18] In ScF_3, the rotation of ScF_6 octahedron was proposed to be responsible for the NTE over a temperature range of 1000 K.[17] Accordingly, the low-temperature heat capacity of ScF_3 shows a significant excess beyond the Debye model due to the low-energy phonon modes responsible for NTE.[17,18] Although the phonon spectrum for $AXMn_3$ is not available, it was estimated that the lowest optical phonons which can be taken as the vibration of CNi_6/NNi_6 octahedron at the center zone for $MgCNi_3$ and $CuNNi_3$ are both more than 10 meV.[44,45] These energies are comparable to $\hbar\omega_{\rm E}$ values as shown in Fig. 6. Experimentally, static rations of NMn_6 octahedra were verified in local structures of $Cu_{1-x}Ge_xNMn_3$ and $Cu_{1-x}Sn_xNMn_3$ via pair distribution function,[46–48] though the average structure was kept cubic. This suggests the dynamic rotations of XM_6 octahedra may exist in AXM_3 compounds. Alternatively, in crystal, the abnormal peak of plot C/T^3 versus T can be attributed to Van Hove singularities of vibration density of states.[9,14] The vibrational modes which disperse and pill up at the Brillouin boundary are responsible for excess vibration states.[9,49,50] According to the phonon spectrum of $MgCNi_3$, the energies of the three optical modes due to light atom (C) are all very high (>77 meV) and the heaviest atoms (Ni) provide the most low-frequency acoustic branches.[44] In this scenario, heavy A atoms of $AXMn_3$ should result in a flattening and downshift of relevant low-energy acoustic mode.[51] So, it is possible that both the strongly dispersive acoustic branches and the optical-like modes hybridize and account for the excess specific heat observed in our work.

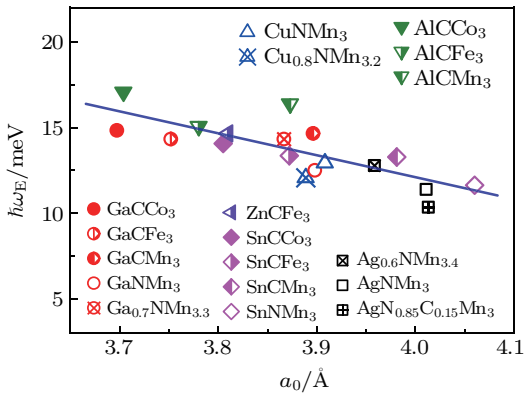


Fig. 6. (color online) Energies of Einstein model corresponding to the peak in $(C-\gamma T)/T^3$ versus T curve as a function of lattice constant for antiperovskite compounds.

Under high pressure, the structure of ZrW_2O_8 becomes amorphous from cubic through orthorhombic structure, consequently the softening of low-energy phonon modes which account for the NTE is observed.[52,53] So, the excess specific heat observed in antiperovksite AXM_3 compound is unrelated to NTE since the chemical pressure leads to the stiffening of the lower-energy modes. Moreover, only some Mn-based compounds (i.e., Ga$_{0.7}$NMn$_{3.3}$ and Ag$_{0.6}$NMn$_{3.4}$) among the 18 compounds show NTE. The average linear thermal expansion coefficient α values are -16.7 ppm/K (175 K–212 K) and -25 ppm/K (206 K–279 K) for Ag$_{0.6}$NMn$_{3.4}$ and Ga$_{0.7}$NMn$_{3.3}$, respectively.[42,43] In $ANMn_3$ compounds, the NTE is related to the ordering of Γ^{5g}-type AFM phase. Such a unique AFM configuration does not exist in Fe- nor Co-based antiperovskite compounds that show normal positive thermal expansion rather than NTE.[34–36,40] In fact, the height of peak in $(C-\gamma T)/T^3$ versus T in antiperovksite compound (≤ 0.35 mJ·K^{-4}·mol^{-1}) is lower than in ScF_3 (~ 0.55 mJ·K^{-4}·mol^{-1}).[54] The relevant peak positions of antiperovskite compounds (between 10 K–30 K) are much higher than that of ScF_3 (a few K). So the low-energy modes are significantly stiffened in antiperovskite compounds compared with in ScF_3. This indicates again that the excess phonon modes in antiperovskite compounds are not able to make a considerable negative contribution to the overall thermal expansion. Consistently, an earlier theoretical calculation claimed that the transverse motion from the rigid

NMn_6 octahedron had no contribution to the observed NTE of $Cu_{0.5}Ge_{0.5}NMn_3$,[55] though details were not reported. In AXM_3, M atoms are chemically bound by A atoms which are at the corners of the cubic lattice. Therefore, NM_6 octahedron is less flexible than ScF_6, which is responsible for the smaller excess of specific heat observed in antiperovskite compound than in ScF_3.

In a perfect perovskite-type cubic structure all crystallographic sites have inversion symmetry, thus the first order Raman scattering is forbidden.[44] In order to shed light on the nature of excess specific heat observed in an antiperovskite compound, theoretical study or inelastic neutron scattering is desirable.

4. Conclusions and perspectives

In the present study, large excess specific heat is observed in each of the investigated Mn-, Fe-, and Co-based antiperovskite compounds due to low-energy phonon modes exceeding the contribution from the Debye model. A chemical pressure effect is observed, i.e., as the lattice shrinks by full or partial chemical doping, the characteristic energy of low-energy mode is increased. In addition, it is found that the low-energy mode is not necessarily coupled with NTE. The strongly dispersive acoustic branches of A atoms and the optical-like modes by the dynamic rotation of XM_6 octahedra may account for the low-energy thermal vibrations, while further studies, both experimental and theoretical, are needed.

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