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Articles

Bismuth Aluminate: A New High-T_C Lead-Free Piezo-/ferroelectric

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Ferroelectric materials, which have a reversible spontaneous polarization and generate an electric potential when subjected to a mechanical stress (piezoelectricity), have applications in nonvolatile random access memory devices and micro-electromechanical systems. For such applications, materials that remain ferroelectric up to high temperatures are desired. Bismuth aluminate has been predicted to be one such material. We present herein the first characterization of the dielectric, ferroelectric, and piezoelectric properties of the ceramic $\mathrm{BiAlO_3}$ synthesized by the high-pressure method. It is demonstrated that $\mathrm{BiAlO_3}$ is indeed a lead-free ferroelectric with a Curie temperature $T_\mathrm{C} > 520$ °C, a piezoelectric coefficient $d_{33} = 28$ pC/N, and a room-temperature remnant polarization $P_\mathrm{r} = 9.5\ \mu\mathrm{C/cm^2}$. P_r increases with temperature, reaching 26.7 $\mu\mathrm{C/cm^2}$ at 225 °C.

Introduction

Ferroelectrics (which are a subset of piezoelectrics) are materials that have a spontaneous polarization that can be reversed with the application of an appropriate electric field. This reversible polarization makes ferroelectric materials useful in memory devices[1–5] (nonvolatile ferroelectric random access memory, FeRAM). The polarizability of ferroelectrics means that they also tend to have very large dielectric constants, making them ideal materials for high-power-density capacitors.[1] Ferroelectrics only exhibit spontaneous polarization below the Curie temperature (T_C); once heated above that temperature, the spontaneous polarization disappears. Upon cooling, the domains (regions with the same polarization) reappear with a random orientation.[1] Thus, a ferroelectric memory is erased (and a poled ferroelectric ceramic loses its piezoelectricity) if it is heated above the T_C. Furthermore, the fundamental polarization–T_C relation for displacive-type ferroelectric materials ($T_\mathrm{C} = 0.303 \pm 0.0018 P_\mathrm{s}^2$, where the spontaneous polarization, P_s, is in units of $\mu\mathrm{C/cm^2}$ and the T_C is in units of K)[6] implies that high-T_C materials also have large polarizations. A high T_C is therefore desired of ferroelectric materials.

Currently, the ferroelectrics that exhibit the best piezoelectric and ferroelectric properties are lead-containing materials like $\mathrm{Pb(Zr_{1-x}Ti_x)O_3}$[4,7] and $\mathrm{(Pb_{1-y}La_y)(Zr_{1-x}Ti_x)O_3}$.[1] Because of the toxicity of lead, there is currently much interest in developing lead-free materials to replace them.[3,7–14] There are, however, only a limited number of lead-free ferroelectric materials with a T_C sufficiently high and the performance good enough for piezoelectric applications; the most promising of these materials are the $\mathrm{KNbO_3}$–$\mathrm{NaNbO_3}$ (KNN) ceramics and modified versions thereof.[8] For memory applications, where good piezoelectric properties are not required, there are more suitable lead-free options, such as $\mathrm{BiFeO_3}$[13,15] (BFO), $\mathrm{BaTi_2O_5}$,[16] $\mathrm{SrBi_2Ta_2O_9}$,[17–19] (SBT), and

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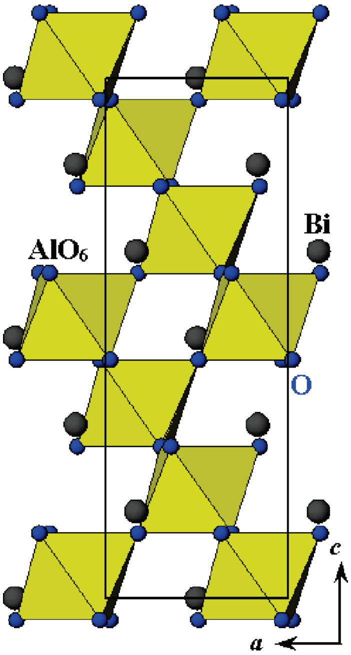

Figure 1. Crystal structure of $BiAlO_3$ (adapted from ref 10) which consists of a rhombohedral unit cell that is elongated along the *c*-axis, with the relevant lattice parameters[10] $a = 5.3755$ Å, $c = 13.3933$ Å (hexagonal axes) or $a = 5.4372$ Å, $\alpha = 59.25°$ (rhombohedral axes).

a few others. Of these, BFO[3,12] and SBT[4,5,17–19] have received much attention for the FeRAM applications.

In an effort to search for new high-performance lead-free high-T_C piezo-/ferroelectrics, density functional theory calculations were performed,[9] and one of the compounds studied, $BiAlO_3$ (BAO), was predicted to have noncentrosymmetric and polar *R*3*c* symmetry, good piezoelectric properties, and a ferroelectric T_C of ~530 °C. Furthermore, $BiAlO_3$–$BiGaO_3$ (BAO–BGO) solid solutions were predicted to have excellent piezoelectric properties, on par with those of the $PbZrO_3$–$PbTiO_3$ ceramics.[9] Recently, BAO and BGO were synthesized[10] at high pressure (6 GPa), and subsequent diffraction experiments confirmed that the structure of BAO has the *R*3*c* symmetry. Like that of BFO,[20] the structure of BAO (Figure 1) consists of a rhombohedral unit cell that is elongated along the *c*-axis. On the basis of structural data, the spontaneous polarization was calculated[10] to be 32 μC/cm^2, which is lower than the predicted[9] value of 76 μC/cm^2. Note that neither of these values agrees with the fundamental relationship (if the T_C is the predicted 530 °C). This is because the ferroelectricity in BAO is not purely due to B-site cation displacement but is also largely due to the activity of the A-site Bi^{3+} ion with the polarizable lone electron pair.[9] Differential scanning calorimetry (DSC) experiments showed that the BAO ceramics decompose at 547 °C, and subsequent diffraction experiments showed that the products of this decomposition were Al_2O_3 and $Bi_{25}AlO_{39}$.[10]

The poor thermal stability of BAO limits its potential applications. However, we have previously shown that BAO–$LaAlO_3$ solid solutions (with up to 20 mol % BAO content) can be formed at high temperatures (1400 °C).[21] Thus, it may be possible to make BAO-based ceramic solid solutions with improved thermal stability. Since it has not yet been possible to make BAO ceramics at normal pressures and high-pressure synthesis is very costly, it would, in any event, be necessary to make BAO-containing solid solutions (at normal pressures) or BAO thin films (which could possibly be stabilized by film–substrate interactions) before BAO could be used in technological applications.

Previously, dielectric properties of a ceramic prepared by sintering a 1:1 Bi_2O_3:Al_2O_3 mixture at 4 GPa were reported by Mangalam et al.[22] However, their sample contained roughly 20% perovskite-phase $BiAlO_3$ and 80% impurities, displayed very weak hysteresis ($P_s \approx 0.03$ μC/cm^2), and had a very low dielectric constant $k \approx 0.1$ at room temperature (10^6 Hz). Because of the very low purity of their sample, however, no claims about the intrinsic properties of BAO could be made. Thus, those results should be rejected. No other information on the dielectric, piezoelectric, or ferroelectric properties of BAO is available to confirm the actual performance of this material.

In this work, we report the first characterization of the dielectric, ferroelectric, and piezoelectric properties of perovskite $BiAlO_3$ and demonstrate that it is indeed a lead-free piezo- and ferroelectric with a Curie temperature $T_C > 520$ °C. These properties are then compared with those of other lead-free high-T_C materials, in particular, SBT and BFO, to show that BAO is a potentially interesting material for ferroelectric applications. The piezoelectric properties of BAO are not good enough for such applications, but knowledge of the properties of BAO may lead toward the development of solid solutions which are useful piezoelectrics (e.g., the BAO–BGO solid solution).

Experimental Section

The $BiAlO_3$ ceramics were prepared under high pressure via a two-step method, following the process described in ref 10. First, Bi_2O_3 and Al_2O_3 were intimately mixed and heat-treated at 750 °C for 8 h to form a precursor mixture consisting of $Bi_{25}AlO_{39}$ and Al_2O_3. The precursor mixture was then calcined at 1000 °C for 40 min, at a pressure of 6 GPa (applied by a belt-type apparatus). After calcining, the samples were quickly cooled to room temperature, and the pressure was slowly released. The dense sample was then cut into (three) rectangular pellets (ranging from 2.9 × 2.2 to 3.4 × 3.8 mm^2 in area and 0.4 to 0.6 mm in thickness) using a diamond saw and polished with diamond papers.

Scanning electron microscope (SEM) images were taken using a FEI DualBeam Strata 235 apparatus on the surface of the as-prepared sample (the unpolished outer surface of the sample), which was sputtered with a thin layer of Au, in a pulsed sputtering mode, in an attempt to reduce the buildup of charges on the surface.

In preparation for dielectric spectroscopic measurements, the sample was sputtered with Au electrodes. Au wires were then attached with Ag paste, and the measurements were performed in the 10–10^6 Hz frequency range, from −130 to 350 °C, with a NovoControl broadband dielectric spectrometer. This measurement was repeated on two separate samples, and the results were found

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to be reproducible. The temperature range was limited by the heating unit on our NovoControl apparatus, which had an operational temperature limit of 350 °C.

Polarization vs electric field (*P*–*E*) hysteresis measurements were performed on a 110 μm thick sample (which had previously been heated up to 300 °C during impedance measurements) covered with Ag paste electrodes. Au wires were attached with Ag paste, and the measurements were performed at 9.7 Hz using a Radiant Technologies RT66A ferroelectric test system. For the measurements at high temperatures, the sample was placed in a silicone oil bath, the temperature of which was controlled using a Delta 9023 chamber.

Measurements of the piezoelectric coefficient d_{33} were performed by means of an IACAS ZJ-6B piezo d_{33}/d_{31} meter. According to the manufacturer's specifications the resolution of the measurement is 0.1 pC/N and the accuracy is 5%.

Prior to these piezoelectric measurements, the samples were sputtered with Au electrodes and poled in a silicone oil bath using a Stanford Research Systems P5350 high-voltage power supply. For high-temperature poling processes, the temperature was controlled by the Delta 9023 chamber.

Resonance–antiresonance measurements (in which one looks for the frequency at which the impedance is a minimum, which coincides with the resonance frequency of the sample) are often also used to explore the piezoelectric properties of materials like BAO. In such experiments, the resonance frequency is inversely proportional to the thickness and diameter (or length) of the sample. Our samples were very small, their size being limited by the cavity size of the belt-type apparatus used to make them. Furthermore, our NovoControl apparatus cannot perform impedance measurements above 10 MHz (and its accuracy decreases substantially above 1 MHz). Thus, we could not do resonance–antiresonance experiments and instead had to rely on our d_{33} meter for information about the piezoelectric properties of BAO.

DSC and DDSC (derivative differential scanning calorimetry) experiments were performed from −50 to 300 °C using a Seiko Exstar 6000 apparatus.

Results

Scanning electron microscope (SEM) images (Figure 2a,b) of the as-prepared ceramics showed that they have an average grain size of 10 ± 3 μm, a fairly homogeneous microstructure, and good densification, thus suitable for subsequent characterization.

Dielectric spectroscopic measurements reveal the frequency dependence of the real part of the permittivity (also called the dielectric constant, ϵ') and loss tangent of the material at room temperature (Figure 3). At 25 °C, the dielectric constant of BAO ranges from 142 to 94.2 over the 10–10^6 Hz frequency range. The loss tangent is relatively high at low frequencies ($\approx$0.6 at 10 Hz), indicating the contributions from slow mobile charges, and it drops quickly to <0.1 at 1 kHz and <0.05 at $f \geq 10$ kHz. These high losses will potentially limit the applicability of BAO ceramics. We have previously shown that BAO–$LaAlO_3$ ceramics can be made with very low loss tangents (a loss tangent of 0.004 at room temperature and 100 Hz was reported for a ceramic with 20 mol % BAO),[21] so it may be possible to reduce the loss tangent of BAO by making solid solutions.

The dielectric constant decreases with increasing frequency, which can be attributed to a reduced ionic contribution to the polarization at high frequencies. Ionic polarization has a relatively slow response to an applied field; thus, at high frequencies, it can no longer keep up with the changing electric field, and its contribution to the polarization is diminished. A similar effect was previously observed in SBT.[23]

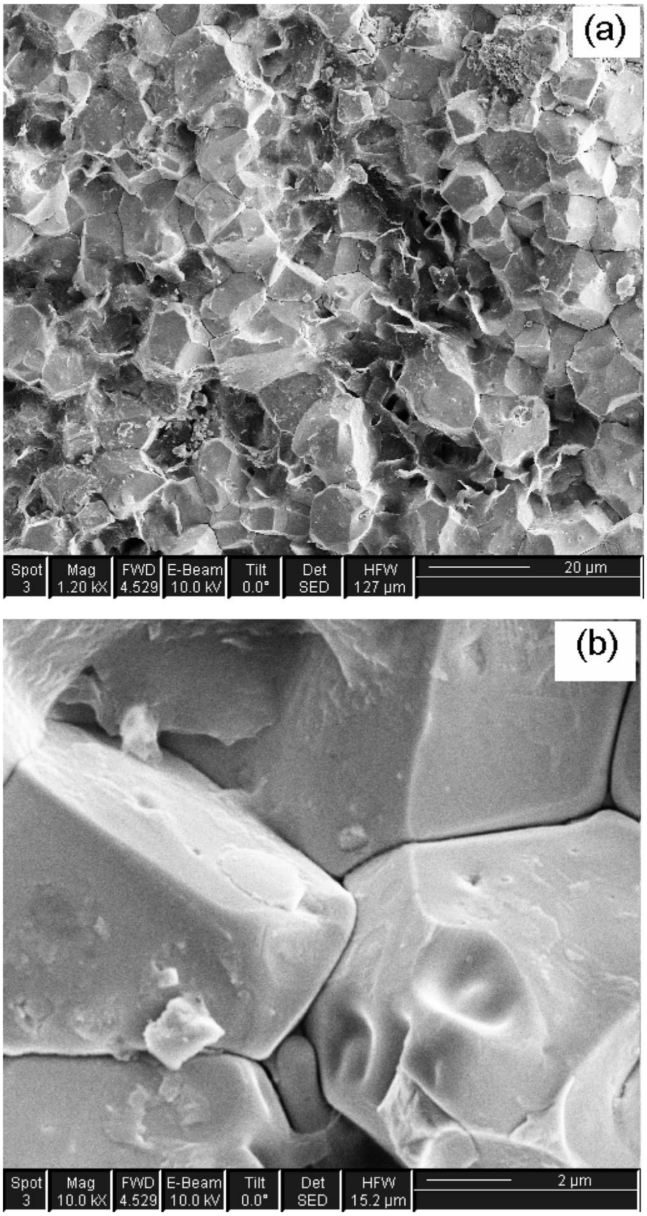

Figure 2. Scanning electron microscope (SEM) images of the as-prepared $BiAlO_3$ ceramics, which show an average grain size of 10 ± 3 μm (a) and a fairly good densification (b).

The variations of the dielectric constant and the loss tangent of BAO upon heating at 10^2–10^6 Hz are shown in Figure 4. The dielectric constant peaks at about 200 °C (Figure 4a), but only at low frequencies. The data collected during the cooling of the sample did not show this impedance peak, and neither did impedance data collected on a previously heated (to 350 °C) ceramic. This suggests that there is some irreversible change occurring in the sample at around 200 °C. The DSC data of Belik et al. (in the online Supporting Information associated with ref 10) show a tiny

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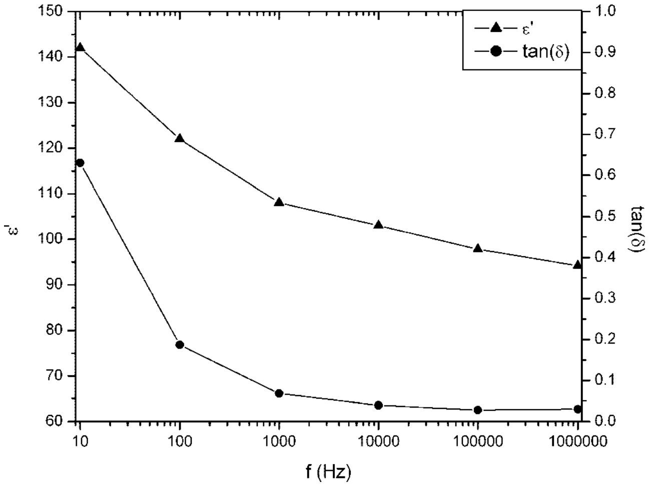

Figure 3. Frequency dependence of the dielectric constant and loss tangent of $BiAlO_3$ at 25 °C. The solid lines are to guide the eye.

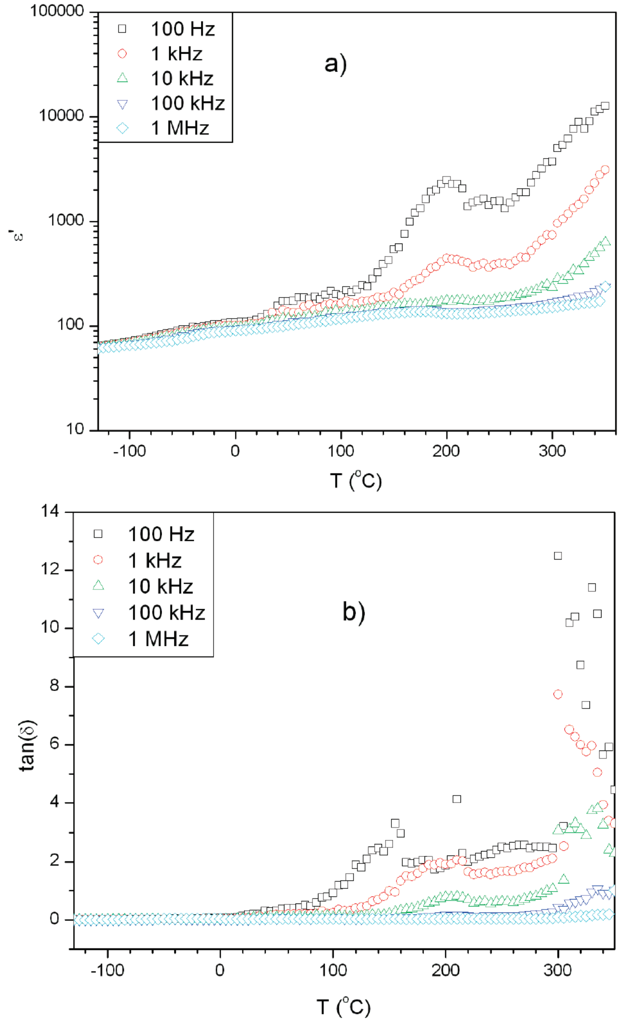

Figure 4. Dielectric constant (ϵ') (a) and tan δ (b) of $BiAlO_3$ as a function of temperature measured at 10^2–10^6 Hz.

peak at around this temperature upon heating, but not cooling. Our DSC and DDSC (derivative differential scanning calorimetry) data collected on a previously heated (to 350 °C) sample did not show this peak. One possibility is that there are charged (positive and negative) defects present in the sample after synthesis, which become more mobile at increased temperatures and eventually recombine. The decrease in the magnitude of the dielectric anomaly with increasing frequency suggests that these defects must be relatively slow-moving. A similar dielectric effect has previously been observed in $Ba_{1-x}Pb_xTiO_3$ ceramics.[24] In that case, it was shown that the dielectric anomaly was caused by the movement of charged defects and that it could be made to disappear by annealing the sample or changing the synthetic conditions.

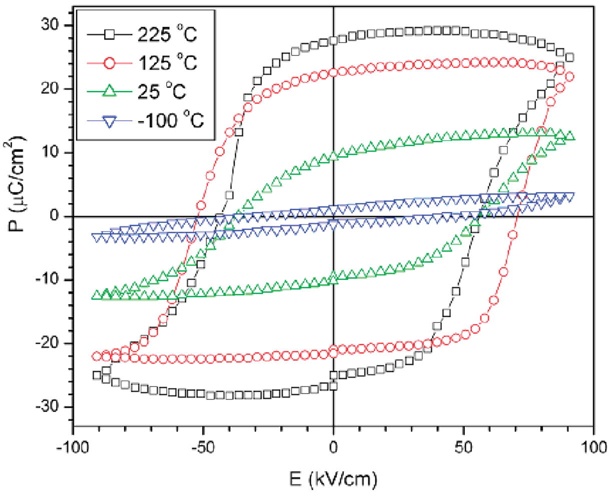

Figure 5. Polarization vs electric field hysteresis loops measured on a 110 μm thick $BiAlO_3$ sample at four different temperatures. The P_r increases from 1.1 to 26.7 $\mu C/cm^2$ as the temperature is increased from −100 to 225 °C. The solid lines are to guide the eye.

The polarization vs electric field (*P*–*E*) hysteresis is displayed in Figure 5. It clearly demonstrates the ferroelectric nature of BAO. Several interesting properties of BAO can be deduced from these *P*–*E* loops, including the temperature dependence of polarizability, conductivity, and internal bias fields. The *P*–*E* loops measured at different temperatures show an increase in remnant polarization (P_r, the polarization of the material once the external electric field has been removed) and saturation polarization (P_s, the maximum polarization attained in the *P*–*E* loop) with increasing temperature. This may, at first glance, appear contrary to the fundamental temperature–polarization relationship (i.e., at high temperatures, where the cation displacement should be reduced, we are reporting increased polarization). However, our data are limited by the electric field that we can apply (in all loops, the maximum field was the same, 91 kV/cm). At none of the experimental temperatures are our *P*–*E* loops fully saturated. While the spontaneous polarization must decrease with temperature, the dipoles become easier to switch at high temperatures, so we still see an increase in measured polarization. This suggests that, if a larger field could be applied to the samples (especially at lower temperatures), larger P_r values may be obtained. Also, if we could saturate the *P*–*E* loops, we would expect to see a decrease in P_r with increasing temperature.

The slightly curved tips of the hysteresis loops (the regions of maximum positive and negative applied electric field) displayed at 125 and 225 °C indicate that the conductivity of the samples becomes significant at higher temperatures (as seen

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in Figure 4b). In *P–E* hysteresis loops, conductivity causes a decrease in polarization with increasing electric field strength. This happens because some leakage currents are established when the samples are placed in a large electric field, thereby reducing the measured polarization. In our measurements, this effect can be attributed primarily to ac conductivity: our ability to apply large dc voltages across the sample when poling the sample for piezoelectric measurements, even at 225 °C (the maximum temperature at which *P–E* loops were displayed), implies that the dc conductivity must remain very low even at this elevated temperature.

In Figure 5, it can be seen that the *P–E* loops are not symmetric about the polarization (*Y*) axis. Instead, they are shifted slightly toward the positive *X*-direction, suggesting that an internal bias field is present in the sample. An internal bias field can be caused by preferential orientation of the grains in a particular direction, which may be caused by the high pressure used to synthesize the material. It should be noted that bias fields can also be created by the movement of defects or the pinning of domain walls within the sample.[5]

Since $BiAlO_3$ is ferroelectric, it must also be piezoelectric. The piezoelectric coefficient d_{33} (the polarization generated along the same direction as the applied force per unit applied force) was measured in the ceramics poled under several different regimes. When the poling was performed at room temperature under $E = 50$ kV/cm, the sample had a d_{33} value of 7(0.4) pC/N. A d_{33} value of 28(1) pC/N is found at room temperature after poling at 225 °C under $E = 21$ kV/cm. The fact that the BAO ceramic poled at a higher temperature and a lower field exhibits a higher d_{33} value confirms that more dipoles have switched, as demonstrated in the *P–E* hysteresis loops.

In an effort to estimate the upper limit for the T_C of BAO, a poled sample with a d_{33} of 28(1) pC/N was held at 520 °C for 15 min, after which the d_{33} was remeasured. The d_{33} value was found to be 25(1) pC/N after this heating process, indicating that thermal depoling had not occurred. A ferroelectric ceramic heated above the T_C and then cooled to room temperature would have randomly oriented domains. (The domains disappear at temperatures above T_C, and when they re-form upon cooling, they re-form with random orientations to minimize the energy of the system.) Thus, any piezoelectric properties displayed by the crystallites would average out to zero over the whole ceramic (which consists of many, randomly oriented grains). Therefore, the presence of piezoelectricity after heating suggests that the sample has not been heated above the T_C. The small decrease in d_{33} after heating can be attributed to the increased rate of tunneling of dipoles through the energy barrier (back to a lower energy, less fully poled configuration) with increasing temperature.

We can therefore conclude that BAO must have a T_C higher than 520 °C. Differential scanning calorimetry (DSC) measurements[10] previously showed that there are no detectable thermal events between 520 and the 547 °C decomposition temperature of BAO. This suggests that if the transition from the ferroelectric to the paraelectric phase of BAO is first-order (as is the case with isostructural compound BFO[13,15]), then it must occur at a temperature above 547 °C, which is in agreement with the prediction of 530 °C in ref 9. Therefore, we have proven that $BiAlO_3$ is a high-T_C ferroelectric material.

Table 1. Dielectric and Ferroelectric Properties of $BiAlO_3$ Compared with Those of Other Lead-Free High-T_C Ferroelectric Ceramics

compound	d_{33} (pC/N or pm/V)	P_r at 25 °C (μC/cm^2)	T_C (°C)
BAO (this work)	28 (1)	9.5	>520
BFO[a]	2.5	12	820–836
SBT[b]	21	6	325–330

[a] References 13, 15, 20, and 27. [b] References 17 and 18.

Discussion

Table 1 summarizes the values of d_{33}, P_r, and T_C of BAO ceramics along with those of other high-T_C ferroelectric materials BFO and SBT.

The only d_{33} value reported for BFO ceramics is much lower than that of BAO. This is due to the difficulties encountered in poling the material, owing to its high conductivity.[20,25] This conductivity arises from the oxygen vacancies created by the variable oxidation state of iron (both Fe^{2+} and Fe^{3+} ions tend to be present in BFO[20,25,26]). No such problems occur with BAO because the Al^{3+} ion has a definite oxidation state, which preserves the stoichiometry. This gives BAO a relatively low conductivity, which has allowed us to perform high-temperature hysteresis measurements (such measurements have not been possible for pure BFO ceramics).

The piezoelectric properties of BAO are, however, much worse than those of KNN-based ceramics (which have d_{33} values as high as 416 pC/N for the textured ceramics).[8] Thus, BAO is not very promising as a piezoelectric material. However, solid solutions containing BAO may very well prove to be high-performance piezoelectrics (as predicted by Baettig et al.[9]); this is left for future work.

From the comparison of ferroelectric properties, it can be seen that the P_r of BAO is competitive with those of other lead-free materials (BFO and SBT) that are being considered for memory applications; it is higher than that of SBT and lower than that of BFO. In BFO, the high conductivity poses a serious limitation on its ferroelectric performance.[20,25] This is, however, not the case with BAO.

The squareness of the BAO hysteresis loops is also similar to those of BFO[20,25] and SBT[17] ceramics. Loop squareness is important for memory materials. A square (or rectangular, to be more accurate) hysteresis loop has only two polarization states $+P_s$ and $-P_s$; thus, it is a truly binary system.

From this comparison, BAO performs as well as, or better than, SBT and BFO, and it may be a promising material for memory applications.

Conclusions

In summary, we have characterized the dielectric, ferroelectric (hysteresis), and piezoelectric properties of $BiAlO_3$ ceramics prepared by high-pressure synthesis, confirming

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some of the predictions of Baettig et al.[9] We have demonstrated that $BiAlO_3$ is indeed ferroelectric and that it has a T_C that is higher than 520 °C. The dielectric, ferroelectric, and piezoelectric properties of BAO are comparable to those of BFO and SBT, making it a promising new high-T_C lead-free ferroelectric material for memory applications. There are, however, two main factors—the poor thermal stability and extreme synthesis conditions—that limit the usability of BAO in technological applications. Nevertheless, the properties of BAO are such that, in solid solution with other materials, it may very well prove to be technologically useful.

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