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Room temperature magnetoelectric multiferroism through cation ordering in complex perovskite solid solutions

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Abstract

A strategy for developing ferrimagnetism through B-site cation ordering in thermodynamically stable ferroelectric complex perovskites is described, with the aim of realizing both a spontaneous polarization and magnetization at room temperature. Specifically, solid solutions of the complex perovskites $\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$, $\text{Sr}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$, and $\text{Ba}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$ with BiFeO_3 are considered. Preliminary data on the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic show promising results.

1. Introduction

Magnetoelectric multiferroic materials are multifunctional materials with simultaneous ferroelectric and magnetic properties [1–3]. Combining ferroelectricity and magnetism in a single-phase compound would obviously be of tremendous interest not only for practical applications but also for fundamental physics [1–8]. The intrinsic ability to couple the electrical polarization to the magnetization allows an additional degree of freedom in the design of conventional devices. A range of new applications is envisaged that takes advantage of this unique material characteristic. This great potential has launched an intensive worldwide research effort into new multiferroic materials and devices [1–8].

Experimental confirmation of magnetoelectric multiferroism in single-phase compounds was accomplished in the early 1960s [9, 10]. However, limited progress has been made during the last several decades for the following reasons. First, coupling in existing single-phase compounds was too weak to be of practical use. Secondly, there were very few compounds displaying the magnetoelectric coupling. Furthermore, the Curie or Néel temperature of most of the compounds was far below room temperature. Therefore, an alternative approach of combining magnetostrictive and piezoelectric compounds to form

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composites has been attempted [11]. In this situation, magnetoelectric coupling is realized through an extrinsic strain. The first artificial magnetoelectric composite was synthesized by unidirectional solidification of the eutectic composite of ferroelectric BaTiO₃ and magnetic CoFe₂O₄ compounds [12]. Recently, research on magnetoelectric composites has been focused on laminated structures, such as the lead zirconate titanate (PZT)/Terfenol-D laminates, in which giant magnetoelectric coupling was observed at room temperature [13–15].

However, the significant technological achievement in the laminated composites offers no insight into the intriguing intrinsic magnetoelectric coupling in single-phase compounds. Most importantly, there are technical concerns about the magnetoelectric composite approach. First of all, the giant magnetoelectric coupling effect can only be observed within a narrow frequency range centred at the natural resonance frequency of the structure [3, 13–15]. In addition, the magnetostrictive components used in these composites are electrically semiconductive or even conductive. This creates difficulties in retaining the electrical polarization developed in the ferroelectric phase and the composite structure would be too lossy when driven by ac electric fields. As a result, these composites have only been tested under ac magnetic fields [13–15].

Therefore, there remains a critical need to find new single-phase materials with strong magnetoelectric coupling at room temperature for practical applications. Recently, first principles calculations have shown that it is more realistic to have ferrimagnetism, instead of ferromagnetism, through B-site cation ordering in the multiferroic perovskite compounds [16]. In fact, the necessity of having cation ordering for the development of spontaneous magnetization, but at a temperature much lower than room temperature, has been experimentally demonstrated by Smolenskii *et al* [9]. A noticeable spontaneous magnetization at temperatures below 170 K in the solid solution of 0.7Pb(Fe_{2/3}W_{1/3})O₃–0.3Pb(Mg_{1/2}W_{1/2})O₃ was detected. The Mg²⁺ and W⁶⁺ cations in the complex perovskite Pb(Mg_{1/2}W_{1/2})O₃ are fully ordered. The magnetically active Fe³⁺ ions in the solid solution of (1 – x)Pb(Fe_{2/3}W_{1/3})O₃–xPb(Mg_{1/2}W_{1/2})O₃ are distributed in both B-site sublattices as



The compounds display a G-type antiferromagnetic super-exchange structure. Since the two sublattices contain a different number of Fe³⁺ cations, the compound as a whole exhibits a net magnetic moment.

In this paper, we examine three complex perovskite compounds with different degrees of B-site cation ordering. With the aim of realizing room temperature coupling, their solid solutions with BiFeO₃ are proposed. We have demonstrated the solid solution approach in the Pb(Fe_{2/3}W_{1/3})O₃–BiFeO₃ system.

2. Cation, dipole and spin order

The strategy of introducing magnetic behaviour into ferroelectric compounds is to incorporate magnetically active species, such as Cr³⁺, Mn³⁺, Fe³⁺, Co²⁺, Ni²⁺, and rare earth ions into the lattice [1–10]. Of particular importance are the compounds with the perovskite ABO₃ crystal structure, such as Pb(Fe_{2/3}W_{1/3})O₃ [17–21], Sr(Fe_{2/3}W_{1/3})O₃ [22, 23], and BiFeO₃ [24–31] and the Ba(Fe_{2/3}W_{1/3})O₃ compound with the hexagonal perovskite structure [32, 33]. Their magnetic and ferroelectric properties are summarized in table 1. Obviously they display high transition temperatures for both ferroelectric and magnetic behaviour.

It should also be noted that Pb(Fe_{2/3}W_{1/3})O₃ displays a nanoscale 1:1 B-site cation order similar to that observed in the well characterized system Pb(Mg_{1/3}Nb_{2/3})O₃ [34]. Sr(Fe_{2/3}W_{1/3})O₃ displays partial 1:1 cation ordering on a length-scale greater than that of Pb(Fe_{2/3}W_{1/3})O₃. Ba(Fe_{2/3}W_{1/3})O₃ exhibits a significant degree of long-range ordering in

Table 1. Magnetic and ferroelectric properties of several pure perovskite compounds. (Note: AFM—antiferromagnetic; FIM—ferrimagnetic; RFE—relaxor ferroelectric; AFE—antiferroelectric; FE—ferroelectric; T_C —Curie temperature; T_N —Neél temperature.)

Compounds	Pb(Fe _{2/3} W _{1/3})O ₃	Sr(Fe _{2/3} W _{1/3})O ₃	BiFeO ₃	Ba(Fe _{2/3} W _{1/3})O ₃
Structure	Cubic	Tetragonal	Rhombohedral	Hexagonal
Magnetic behaviour	AFM	FIM	AFM	FIM
	$T_N = 370$ K	$T_C = 370$ K	$T_N = 650$ K	$T_C = 210$ K
Ferroelectric behaviour	RFE	AFE	FE	AFE
	$T_C = 170$ K	$T_C = 470$ K	$T_C = 1120$ K	$T_C = 570$ K
B-site order	Nanoscale order	Partial order	N/A	Complete order

which the face-sharing oxygen octahedra are occupied by both Fe³⁺ and W⁶⁺ and the corner-sharing ones are exclusively occupied by Fe³⁺, resulting in hexagonal symmetry [33]. As a result, Pb(Fe_{2/3}W_{1/3})O₃ shows antiferromagnetic behaviour while the partially ordered Sr(Fe_{2/3}W_{1/3})O₃ and completely ordered Ba(Fe_{2/3}W_{1/3})O₃ display ferrimagnetic behaviour with a spontaneous magnetization.

As expected, BiFeO₃ has the highest magnetic critical temperature because it is the most concentrated compound with the magnetically active Fe³⁺ species. Dilution of the magnetic species by one-third in the compounds leads to a decrease of 280 K in the critical temperature, as is evident in both Pb(Fe_{2/3}W_{1/3})O₃ and Sr(Fe_{2/3}W_{1/3})O₃. The decrease in Ba(Fe_{2/3}W_{1/3})O₃ is even more dramatic since it has a different crystal structure from the other three compounds [33].

The ferroelectric behaviour in these four compounds is also distinct from each other. Pb(Fe_{2/3}W_{1/3})O₃ is a relaxor ferroelectric with a strong frequency dispersion in its dielectric response [17]. Both Sr(Fe_{2/3}W_{1/3})O₃ and Ba(Fe_{2/3}W_{1/3})O₃ are antiferroelectric and only BiFeO₃ is a normal ferroelectric. Due to its high critical temperature, BiFeO₃ has received extensive attention in recent years [24–31]. Unfortunately it is an antiferromagnetic compound with a negligible spontaneous magnetization at room temperature. In addition, the ferroelectric Curie temperature is too high and the spontaneous electric polarization is very hard to switch at room temperature. Furthermore, the electronic conductivity is relatively high even in the phase pure BiFeO₃ compound, making it very difficult to sustain the high electric fields necessary for polarization switching [31].

Therefore, the most plausible approach to realize room temperature coupling of a high remanent electric polarization and spontaneous magnetization is to form solid solutions of Pb(Fe_{2/3}W_{1/3})O₃, Sr(Fe_{2/3}W_{1/3})O₃, and Pb(Fe_{2/3}W_{1/3})O₃ with BiFeO₃. It should be noted that solid solutions in these systems have not been reported before in the literature. In many applications utilizing ferroelectrics, the solid solution approach has been widely used for many decades to adjust the Curie temperature, enhance the normal ferroelectric behaviour, and manipulate the B-site cation order [34, 35]. In the compound Pb(Fe_{2/3}W_{1/3})O₃, a solid solution with Li(Fe_{1/3}W_{2/3})O₃ strengthens the B-site ordering dramatically [19]. In addition, Fe-rich stoichiometries in the Pb(Fe_{2/3}W_{1/3})O₃ system have been found to favour B-site cation ordering [20]. By incorporating BiFeO₃ into Pb(Fe_{2/3}W_{1/3})O₃ the overall composition becomes Fe-rich. Based on the previous results, an enhancement in cation ordering would be expected in the solid solution Pb(Fe_{2/3}W_{1/3})O₃–BiFeO₃. Also, B-site cation ordering can be engineered in the solid solution of Ba(Fe_{2/3}W_{1/3})O₃ with BiFeO₃. Since Ba(Fe_{2/3}W_{1/3})O₃ displays complete B-site ordering and a distorted perovskite (hexagonal) crystal structure, solid solutions of (1 – x)Ba(Fe_{2/3}W_{1/3})O₃–xBiFeO₃ should have a morphotropic phase boundary (MPB) and a composition-dependent degree of B-site cation ordering.

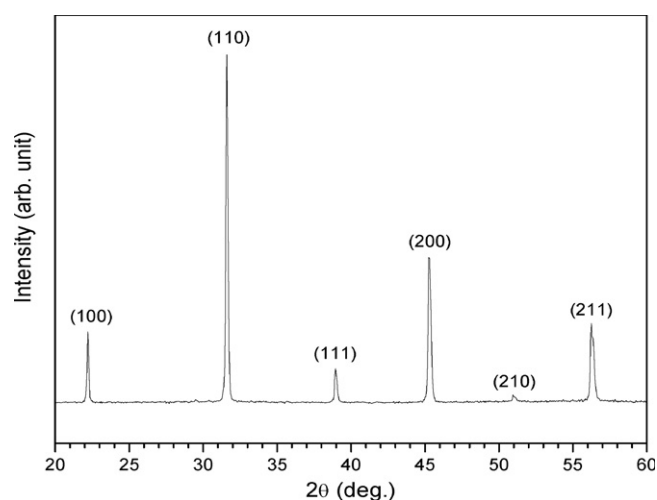


Figure 1. X-ray diffraction pattern of the sintered $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic.

3. Experimental procedure

The solid solution approach described above was demonstrated experimentally with a ceramic of composition $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$. Starting with high purity (>99.9 wt%) PbO , Bi_2O_3 , Fe_2O_3 , and WO_6 powders, a two-step method was utilized to synthesize phase pure ceramics [36]. Stoichiometric amounts of Fe_2O_3 and WO_6 powders were first mixed and milled with zirconia media in a plastic bottle for six hours. After drying in an oven at 150°C for 24 h, the mixture was calcined at 1000°C for 4 h. The A-site oxides PbO and Bi_2O_3 were then mixed with the calcined B-site compound and the mixture was milled again for 6 h. After drying, the mixture was calcined at 890°C for 2 h. Finally, pellets were cold-pressed and sintered at 910°C for 2 h.

The surface layers of the sintered discs were removed by mechanical grinding and x-ray diffraction was used to verify the phase purity. Fresh fracture surfaces were examined with a scanning electron microscope (SEM) to reveal the grain morphology. The ferroelectric domain structure as well as cation ordering was examined with a Phillips CM30 transmission electron microscope (TEM) operated at 300 keV. Dielectric characterization was performed with an LCR meter (HP-4284A, Hewlett-Packard) in conjunction with an environmental chamber (9023, Delta Design). A heating rate of 3°C min^{-1} was used during measurement. The polarization hysteresis measurement was carried out with a standardized ferroelectric test system (RT-66A, Radiant Technologies). A Quantum Design PPMS 9T equipped with the vibrating sample magnetometer VSM option with furnace was used for magnetic characterization.

4. Results and discussion

The as-sintered ceramic is phase pure and possesses a pseudocubic perovskite structure with the lattice parameter of 4.0011 \AA , as evidenced by the x-ray diffraction spectrum shown in figure 1. SEM examination indicates that the ceramic is dense, with grain size around $5 \mu\text{m}$ (see figure 2). TEM analysis reveals that the primary microstructure feature is mottled polar domains, typical of relaxor ferroelectric oxides (figure 3). The inset in figure 3 shows the $\langle 1\bar{1}0 \rangle$ zone axis selected area electron diffraction pattern. Very faint and diffused contrast is noticed at $\{1/2, 1/2, 1/2\}$ -type superlattice spot positions. Two examples are pointed out

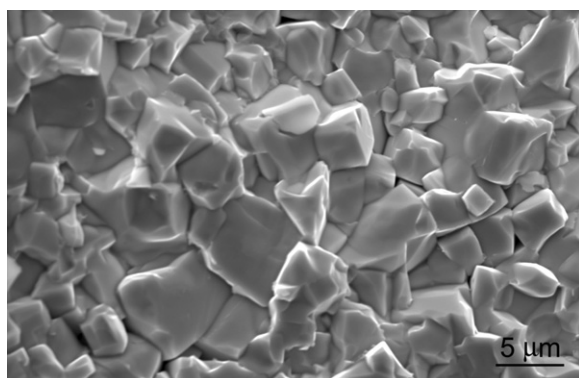


Figure 2. Scanning electron microscope image of the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic.

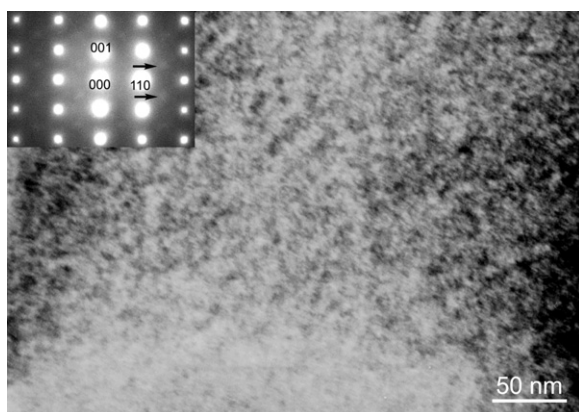


Figure 3. Transmission electron microscope image of the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic. The inset shows the $\langle 1\bar{1}0 \rangle$ zone axis selected area electron diffraction pattern. Diffuse contrast at $\{1/2, 1/2, 1/2\}$ -type superlattice positions is indicative of weak B-site cation ordering.

by the dark arrows. These $\{1/2, 1/2, 1/2\}$ -type superlattice spots were too weak to allow dark field imaging. In complex perovskite compounds, such superlattice spots are indicative of 1:1 B-site cation ordering or oxygen octahedra tilting [34, 35, 37, 38]. However, from the x-ray diffraction pattern shown in figure 1, the ceramic has a cubic structure with one ABO_3 formula unit in the unit cell with a lattice parameter around 4 Å. It is not likely that oxygen octahedra are tilted in the structure. Therefore, the B-site cations are ordered in the as-prepared $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic, but to a very limited extent. Further optimization of processing conditions and/or chemical compositions is needed to enhance such cation order.

The dielectric response of this phase pure pellet was also examined and the result is shown in figure 4. It is interesting to note that a broad peak appears around 320 K. The maximum relative permittivity is measured to be 1550 at 10 kHz. A strong frequency dispersion is noticed both below and above the dielectric peak temperature, different from that of typical relaxor ferroelectric ceramics. The dielectric loss ($\tan \delta$) measurement indicates a value of less than 0.1 within 220–475 K under both 10 and 100 kHz. At room temperature (300 K), $\tan \delta$ was measured to be 0.014 under 10 kHz and 0.021 under 100 kHz. Therefore, incorporating BiFeO_3 into $\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$ raised the ferroelectric transition temperature to above room temperature. However, the frequency dispersion behaviour does not resemble that of a typical relaxor ferroelectric.

The low dielectric loss measured from the ceramic pellet implies a high electrical resistivity that could sustain high electric fields for polarization switching. The electric polarization versus

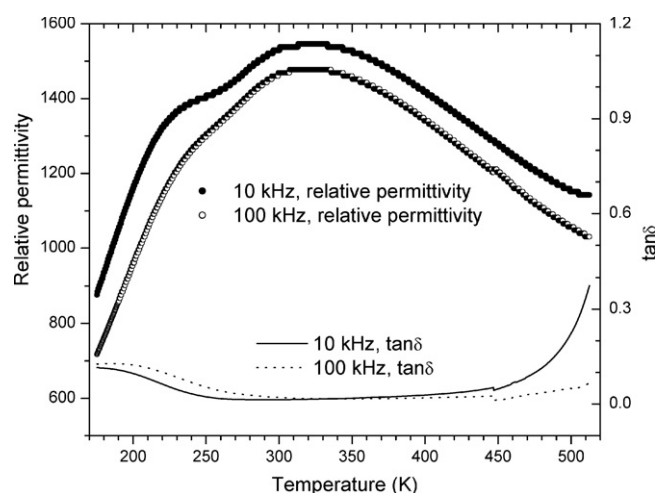


Figure 4. Relative permittivity and dielectric loss of the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic as a function of temperature at 10 and 100 kHz.

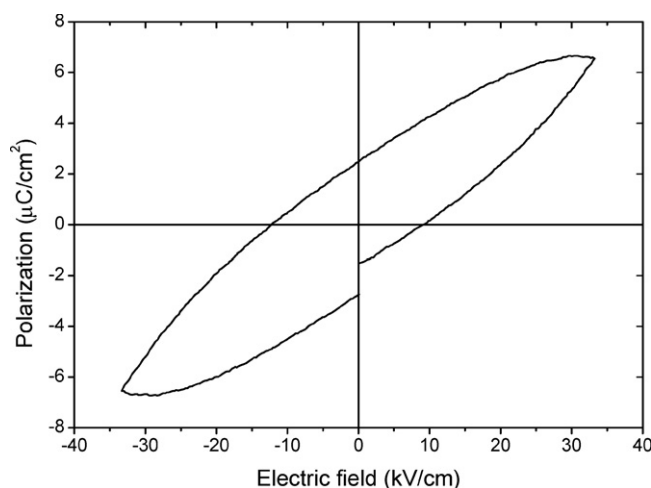


Figure 5. Polarization versus electric field hysteresis loop measured at 4 Hz at room temperature in the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic.

electric field hysteresis loop was evaluated at 4 Hz at room temperature. As shown in figure 5, an unsaturated loop was recorded. An electric polarization of $6.6 \mu\text{C cm}^{-2}$ was measured at the peak electric field 33 kV cm^{-1} . Therefore, the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ composition demonstrated that the solid solution approach is effective in adjusting the ferroelectric transition temperature and developing spontaneous polarizations.

The magnetization as a function of temperature for the as-prepared ceramic is given in figure 6. There is a clear transition from paramagnetic to ferrimagnetic at $\sim 660 \text{ K}$. Heating the sample above 800 K in the measurement vacuum results in the introduction of a significant amount of a second magnetic phase with an ordering temperature of about 850 K . As a result, it is not possible to analyse the behaviour of the magnetization above T_c in terms of a Curie–Weiss law. The room temperature magnetic hysteresis loop of the as-prepared ceramic is shown in figure 7. The magnetization at 5 T is 0.958 emu g^{-1} , which corresponds to $0.0589 \mu_B$ per formula unit or $0.0785 \mu_B$ per Fe. The remnant magnetization of the randomly oriented ceramic sample is 0.276 emu g^{-1} ($0.017 \mu_B$ per formula unit) at room temperature, comparable to those of other perovskite multiferroic compounds [3]. The material exhibits a room temperature

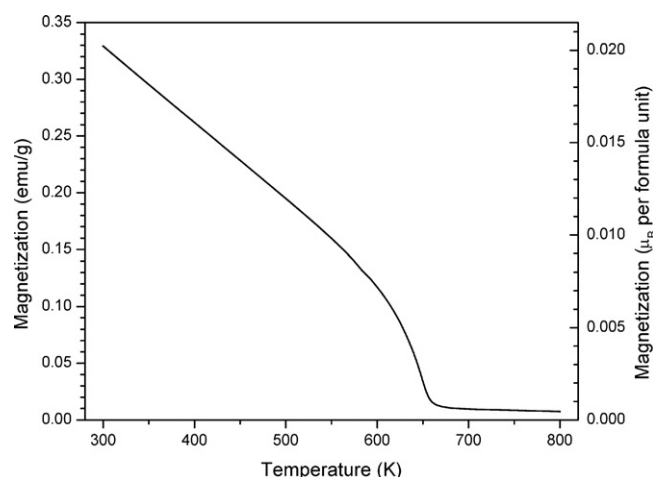


Figure 6. Magnetization versus temperature during cooling from 800 K to room temperature under 1 kOe in the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic.

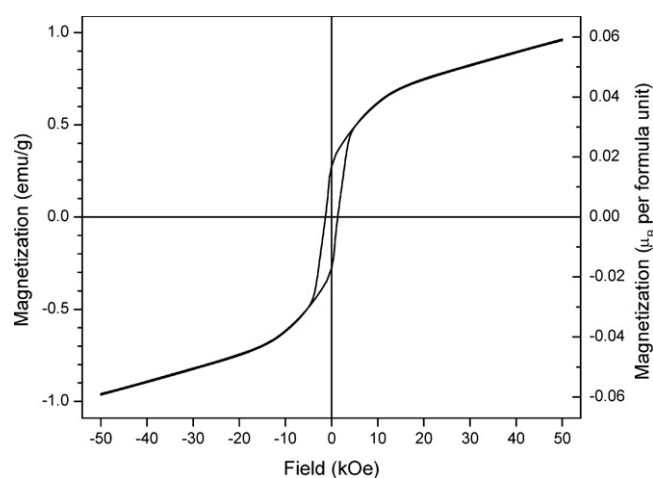


Figure 7. Magnetization versus magnetic field hysteresis loop measured at room temperature in the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic.

coercive field of 1.35 kOe. Again, the solid solution approach proves to be capable of adjusting the magnetization transition temperature and developing spontaneous magnetization. A much higher spontaneous magnetization is expected if the cation ordering is further enhanced in the oxide ceramic.

5. Conclusions

Solid solutions of $\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$, $\text{Sr}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$, and $\text{Ba}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3$ with BiFeO_3 are proposed as suitable candidates to achieve room temperature magnetoelectric multiferroism. By manipulating the B-site cation order in these complex perovskite-based ceramics, large spontaneous electric polarization and magnetic moments can be achieved at room temperature. The net magnetization is realized through uncompensated antiferromagnetic moments due to cation ordering. This approach is demonstrated in the $0.75\text{Pb}(\text{Fe}_{2/3}\text{W}_{1/3})\text{O}_3-0.25\text{BiFeO}_3$ ceramic, where weak cation ordering is present. The ceramic undergoes a ferroelectric transition at ~ 320 K and a magnetic transition at ~ 660 K, with a spontaneous magnetization of $0.017 \mu_B$ per formula unit at room temperature.

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