Micro-Raman study of the microheterogeneity in the M A-M C phase transition in 0.67PbMg1/3Nb2/3O3-0.33PbTiO3 single crystal

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Micro-Raman study of the microheterogeneity in the M_A - M_C phase transition in $0.67\text{PbMg}_{1/3}\text{Nb}_{2/3}\text{O}_3$ - 0.33PbTiO_3 single crystal

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Polarized Raman spectroscopy has been employed to investigate the evolution of the microstructure of $0.67\text{PbMg}_{1/3}\text{Nb}_{2/3}\text{O}_3$ - 0.33PbTiO_3 (PMN-33%PT) single crystal in the temperature range from -195 to 300 °C. The M_A - M_C -cubic transition sequence was observed in the microareas with M_A -type (space group Cm) and M_C -type (space group Pm) monoclinic structures. Interestingly, the M_A - M_C phase transition temperature exhibited remarkable microareal dependence due to the spatial inhomogeneity of polar nanoregions (PNRs). The M_C -cubic phase transition took place at 155 °C in both microareas, which consisted well with previous reports. These results reveal that the phase transition in PMN-33%PT single crystal is closely related with the thermal dynamics of PNRs, which will be useful for understanding the microheterogeneity in this compound. © 2011 American Institute of Physics. [doi:10.1063/1.3574666]

I. INTRODUCTION

Relaxor ferroelectric single crystals $(1-x)\text{PbMg}_{1/3}\text{Nb}_{2/3}\text{O}_3$ - $x\text{PbTiO}_3$ (PMN- x PT) have garnered intense interest in the past decades owing to their giant piezoelectricity that has enabled applications in transducers and actuators.¹ Of the most interest are the PMN- x PT single crystals with compositions near the morphotropic phase boundary, where PT composition is in the range of $0.25 \leq x \leq 0.35$.²⁻⁴ Nanosized local inhomogeneities, such as polarized nanoregions (PNRs) and nanoscale chemical order regions (CORs), are widely believed to be essential in the intriguing properties of the relaxor ferroelectrics.⁵⁻¹⁰ In addition, the coexistence of microdomains with different structures arising from these nanosized inhomogeneities have been observed in PMN- x PT single crystals.¹¹⁻¹⁴ For example, Xu *et al.* proposed the coexistence of the rhombohedral and monoclinic phases in PMN-33%PT single crystal.¹² In recent years, especially, two different monoclinic phases, M_A -type monoclinic structure with space group Cm and M_C -type monoclinic phase with space group Pm , have been demonstrated to exist simultaneously in PMN- x PT single crystals.^{13,14} It has been demonstrated that monoclinic phases play an important role in the ultrahigh piezoelectric responds of PMN- x PT single crystals under poling electric field.^{4,15,16} However, the thermal dynamics of different monoclinic structures in PMN- x PT crystals is not yet well understood.

Combined with an optical microscope, a Raman spectrometer can collect the spectra in a small volume and identify the structure within a micro-sized area, enabling one to study the structural microheterogeneity of the sample. In the past two decades, Raman spectroscopy has proven to be a convenient method to investigate the dynamical properties of PMN- x PT.^{10,14,17-22} However, most of the previous reports

just concerned the Raman scattering from one microarea of PMN- x PT single crystals, while research focusing on different microareas in one sample is scarce. An investigation of the structural microheterogeneity of PMN- x PT single crystals will provide useful information for understanding the macroscopic properties of the relaxor system. Therefore, performing micro-Raman measurement simultaneously on different microareas of one PMN- x PT single crystal is crucial.

In the present work, we report the polarized micro-Raman scattering study of the temperature dependence of the microstructure in PMN-33%PT single crystal. The M_A - M_C -cubic phase transition was observed in different types of microareas while increasing the temperature from -195 to 300 °C. Strikingly, the M_A - M_C transition temperature is different from one microarea to another, whereas the M_C -cubic transition temperature is the same for different microareas. The mechanisms underlying these two phase transition processes are discussed.

II. EXPERIMENT

The PMN-33%PT single crystal studied in this work was grown using a modified Bridgman technique,²³ and was cut perpendicular to the pseudocubic [001] direction with the long edge orientations [100]/[010]. The dimensions of the crystal are $5 \times 5 \times 2$ mm³. Raman spectra were collected on the (001)-face of the crystal using a confocal micro-Raman spectrometer (Horiba/Jobin Yvon HR800). A solid state laser of 532 nm wavelength was used as the excitation source. The laser beam was focused using a $50\times$ objective with numeric aperture $NA=0.5$, and the spot size was about 1.5 μm . The polarization direction of the incident light was parallel to the y -direction. The spectra were recorded in the parallel (VV) $z(yy)\bar{z}$ and crossed (VH) $z(yx)\bar{z}$ polarization configurations, respectively. The sample was placed inside a cryostat cell (Linkam, THMS 600), and the temperature was changed from -195 to 300 °C with a stability of ± 0.1 °C.

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III. RESULTS AND DISCUSSION

Figure 1 presents the polarized Raman spectra for the microareas with M_A - and M_C -type monoclinic structures (hereafter denoted as μA -A and μA -C), respectively, measured at selected temperatures. As can be seen in Figs. 1(a) and 1(b), the spectra for these two microareas resemble the spectral features of M_A - and M_C -type monoclinic structures that were previously reported.¹⁴ The VV and VH spectra of μA -A are nearly the same, while the spectra of μA -C exhibit remarkable dependences on the polarization configurations. On the basis of group theoretical calculation, the monoclinic structure of PMN-33%PT gives rise to 12 Raman-active modes: $\Gamma_{\text{Raman}} = 8A' + 4A''$. Although it is hard to identify the symmetry of every Raman mode due to the broadening and overlapping of modes arising from the complex B -site substitution in this compound, several Raman modes can be assigned according to their intensity dependences on the polarization configurations. For example, the Raman modes located at 570 and 780 cm^{-1} are assigned as A' modes, while the modes at 270 and 520 cm^{-1} are attributed as A'' modes. One can see in Figs. 1(a) and 1(c), the spectra of μA -A do not show significant difference between the room and low temperatures. In contrast, the low-temperature spectra of μA -C are obviously different from the room temperature spectra. As exhibited in Fig. 1(d), the line shapes of the VV and VH spectra become nearly the same, similar to those of μA -A. Combining the results shown in Figs. 1(c) and 1(d) elucidates that μA -C has transformed into M_A -phase at low temperature. This M_C - M_A phase transition took place at -85°C , evidenced from the Raman spectra obtained while cooling the crystal (not shown here).

Figure 2 shows the polarized Raman spectra of μA -A over the temperature range from -195 to 300°C . It should be noted that the VV and VH Raman spectra are nearly the same at the temperatures below 105°C . Then, with further

FIG. 1. (Color online) Polarized Raman spectra for microarea μA -A measured at (a) room temperature (23°C) and (c) low temperature (-195°C). Polarized Raman spectra for microarea μA -C measured at (b) room temperature (23°C) and (d) low temperature (-195°C).

FIG. 2. (Color online) Temperature-dependent Raman spectra for μA -A in PMN-33%PT single crystal measured in the (a) VV and (b) VH polarization configurations.

increasing temperature, significant changes take place in the VH spectra. The intensity of the peak at around 570 cm^{-1} (A_{570}) and the band centered at 780 cm^{-1} (A_{780}) decrease quickly and disappear at 155°C . It is reported that the dielectric maximum temperature of PMN-33%PT is 155°C calculated using a semiexperimental equation.²⁴ And a phase transition to cubic phase is observed in PMN-33%PT at around 160°C using dielectric spectroscopy.^{25,26} Therefore, based on previous experimental and calculation results, the disappearances of A_{570} and A_{780} in the VH geometry obtained here can be assigned as the indicator of the monoclinic-cubic phase transition. According to Raman selection rule (RSRs), first-order Raman-active modes are forbidden in the cubic phase with $Pm3m$ group. However, intense peaks are still seen clearly in the Raman spectra when the crystal is heating upon 155°C , which is in contrast to RSRs. Thus, the intense Raman modes observed at high temperature implies the existence of the local structures with lower symmetries in PMN-33%PT.

The temperature dependence of the polarized Raman spectra of μA -C is shown in Fig. 3. Upon heating, the intensities of A_{570} and A_{780} in the VH spectra decrease gradually, whereas no obvious change is observed in the VV spectra. As temperature increases to 55°C , A_{570} and A_{780} only exhibit high intensity in the VV geometry, presenting obvious polarization dependence. It is noteworthy that the spectra at this temperature recover the features of the room-temperature spectra that are shown in Fig. 1(b). When the temperature increases to 155°C , A_{570} and A_{780} disappear in the VH geometry, reflecting the lattice structure of μA -C transforming into cubic phase.

In order to deeply study the temperature evolution of these two monoclinic structures in PMN-33%PT single crystal, the Raman spectra for μA -A and μA -C are deconvoluted using Gaussian/Lorentzian mixed function and compared. The intensity ratio of peak A_{570} to the peak at 520 cm^{-1} in the VH geometry ($I_{570}^{\text{VH}}/I_{520}^{\text{VH}}$) and the depolarization ratio of A_{780} ($I_{780}^{\text{VH}}/I_{780}^{\text{VV}}$) for these two microareas are plotted in Fig. 4

FIG. 3. (Color online) Temperature-dependent Raman spectra for $\mu\text{A-C}$ in PMN-33%PT single crystal measured in the (a) VV and (b) VH polarization configurations.

as a function of temperature. As shown in Fig. 4(a), the $I_{570}^{\text{VH}}/I_{520}^{\text{VH}}$ of $\mu\text{A-A}$ increases slightly with increasing temperature to 0 °C, and then decreases quickly but not linearly. Two sudden drops at 55 and 155 °C can be seen clearly in the slope of $I_{570}^{\text{VH}}/I_{520}^{\text{VH}}$. Figure 4(c) exhibits the depolarization ratio $I_{780}^{\text{VH}}/I_{780}^{\text{VV}}$ of $\mu\text{A-A}$, where three obvious changes can be seen clearly at 55, 105, and 155 °C, respectively. Combining the results shown in Figs. 4(a) and 4(c), three structural changes, including one structure deformation and two phase transitions, are inferred. The structural change at 55 °C is attributed as a structure deformation, since no obvious change is observed in the spectra [see Fig. 2(b)]. The first phase transition is the $M_{\text{A}}-M_{\text{C}}$ phase transition, occurred at 105 °C. The second phase transition takes place at 155 °C, attributed as the transition from M_{C} phase to cubic phase ($M_{\text{C}}-\text{C}$). These two phase transitions are also inferred from the changes in the line shapes of spectra shown in Fig. 2(b). In the case of $\mu\text{A-C}$, $I_{570}^{\text{VH}}/I_{520}^{\text{VH}}$ and $I_{780}^{\text{VH}}/I_{780}^{\text{VV}}$ decrease slightly with increasing temperature to 45 °C, as illustrated in Figs. 4(b) and 4(d).

FIG. 4. (Color online) Temperature evolutions of (a) $I_{570}^{\text{VH}}/I_{520}^{\text{VH}}$ and (c) $I_{780}^{\text{VH}}/I_{780}^{\text{VV}}$ for the microarea $\mu\text{A-A}$. Temperature evolutions of (b) $I_{570}^{\text{VH}}/I_{520}^{\text{VH}}$ and (d) $I_{780}^{\text{VH}}/I_{780}^{\text{VV}}$ for the microarea $\mu\text{A-C}$.

It can be seen that $I_{570}^{\text{VH}}/I_{520}^{\text{VH}}$ and $I_{780}^{\text{VH}}/I_{780}^{\text{VV}}$ drop abruptly at 55 and 155 °C, corresponding to the occurrence of the $M_{\text{A}}-M_{\text{C}}$ and $M_{\text{C}}-\text{C}$ phase transitions, respectively.

Comparing the temperature evolutions of the intensity ratio for these two monoclinic microareas, several notable features should be highlighted here. Firstly, the lattice structure of $\mu\text{A-C}$ transfers into M_{A} phase at -85 °C, suggesting the structure of the whole crystal belongs to M_{A} phase. This is in good agreement with previous reports, in which only the M_{A} phase was observed at low temperature.²⁷ Such phase transition is expected to relate to the nanoinhomogeneous, i.e., PORs or CORs, in the crystal. It has been indicated that the $M_{\text{C}}-M_{\text{A}}$ phase transition under external electric field arises from the polarization switching mechanism.²⁸ Nevertheless, CORs provide no contribution to the polarization of the crystal because they belong to the nonpolar space group $Fm\bar{3}m$. Hence, PNRs are considered to take the responsibility for the appearance of the M_{A} phase at low temperature. However, the structure of PNRs is still ambiguous so far. It is reported that PNRs belong to the rhombohedral symmetry^{6,29} or the tetragonal symmetry.¹⁸ Recently, Slodczyk *et al.* proposed that PNRs in PMN- x PT with PT component of $0.28 \leq x \leq 0.38$ have monoclinic structure at low temperature.²² From the results shown in our work, the symmetry of PNRs in PMN-33%PT single crystal cannot be identified. Detailed describing the structure of PNRs is attractive, which warrants further investigation.

In addition, it was suggested that the volume density of PNRs increases on cooling,^{29,30} and the polarization of PNRs reorient during the growth process.^{10,19,31} With decreasing temperature, PNRs grow and result in the phase transition in PMN- x PT.²² According to these previous reports, therefore, the low temperature M_{A} phase observed in our work is closely related the thermal dynamic of PNRs without regarding of the local symmetry of PNRs.

Secondly, the $M_{\text{A}}-M_{\text{C}}$ transition temperature ($T_{M_{\text{A}}-M_{\text{C}}}$) is different from one microarea to another. As shown in Fig. 4, the $M_{\text{A}}-M_{\text{C}}$ phase transition takes place at 105 and 45 °C, respectively, in $\mu\text{A-A}$ and $\mu\text{A-C}$. The areal dependence of the rhombohedral-tetragonal transition temperature in PMN-35%PT single crystal observed by Jiang *et al.*, which was attributed to the dynamic of PNRs.¹¹ The areal discrepancy of $T_{M_{\text{A}}-M_{\text{C}}}$ observed in our work could be associated with the dynamic of PNRs as well.

Thirdly, the lattice structure in these two types of monoclinic microareas undergoes drastic variations at the vicinity of 50 °C. In the case of $\mu\text{A-C}$, the $M_{\text{A}}-M_{\text{C}}$ phase transition occurs due to the reorientation of the polarization directions of PNRs, which is testified in the previous section. Of noteworthy the room-temperature structure of $\mu\text{A-C}$ is in the M_{C} phase before cooling [see Fig. 1(d)], whereas it is in the M_{A} phase in the heating process, and does not transform into M_{C} phase until 45 °C [see Figs. 4(b) and 4(d)]. This phenomenon could be associated with the thermal hysteresis behavior. For example, the thermal hysteresis has been observed in the normal ferroelectric BaTiO₃ during both the ferroelectric-ferroelectric and ferroelectric-paraelectric transitions.³² Moreover, the relaxor ferroelectric PZN-9%PT single crystal witnessed a thermal hysteresis of ~ 30 °C in the rhombohedral-tetragonal

phase transition process.³³ Hence, the hysteresis of the M_A - M_C phase transition in μA -C is attributed to the thermal hysteresis property of the PMN-33%PT crystal.

Unlike μA -C, no phase transition occurs in μA -A at around 50 °C. But a structure deformation takes place in μA -A, as evidenced in Figs. 4(a) and 4(c). These changes in the spectra only come from μA -A rather than μA -C, as the diameter of the laser spot focused on the sample surface is less than 1.5 μm , which is smaller than the size of the microareas (3–5 μm). Because the microareas with M_A and M_C phases exist alternately in PMN-33%PT single crystal, the lattice of μA -A will be squeezed while the M_A - M_C phase transition occurs in the surrounding μA -C microareas. Consequently, the relative intensities of the Raman modes for μA -A change, and an anomaly of $I_{570}^{VH}/I_{520}^{VH}$ is observed at 55 °C in Fig. 4(a).

Last but not least, the M_C -C transition occurred at 155 °C in both μA -A and μA -C microareas. Since the structure of these two microareas have been in the same lattice symmetry (M_C phase) above 105 °C, the transition to cubic phase is expected to occur at the same temperature. As presented in Figs. 2 and 3, intense Raman modes are observed in the spectra of PMN-33%PT above M_C -cubic transition temperature (T_{M_C-C}), and A_{780} exhibits obvious dependence on the polarization configurations. Some researchers attributed the high temperature Raman spectra to the existence of the CORs with $Fm\bar{3}m$ structure.^{19,34} The other researchers proposed that the Raman peaks at high temperature come from PNRs due to the local distortions in the crystal.^{22,35,36} For example, Kania *et al.* have studied the PMN- x PT single crystals with high PT-content ($x = 50\%$ and 64%) and inferred that the high frequency mode originate from PNRs rather than CORs.³⁵ Moreover, it is demonstrated that A_{780} consists of two peaks, located at 730 and 800 cm^{-1} , arising from the Ti–O–Ti and Nb–O–Mg stretching modes, respectively.³⁷ The appearance of the TiO_6 octahedral mode suggests that low symmetry structures do exist in PMN-33%PT. According to above discussions, we can draw the conclusion that PNRs with low symmetries are responsible for the Raman modes of PMN- x PT crystals at the temperature up T_{M_C-C} .

IV. CONCLUSION

In summary, the lattice structure of PMN-33%PT single crystal was systematically studied by analyzing the polarized Raman spectra as a function of temperature. The M_A - M_C -C phase transition was observed in the temperature range from –195 to 300 °C. The M_A - M_C transition temperature exhibited areal divergence, which is associated with the spatial inhomogeneous of PNRs. The M_C -C transition occurred at the same temperature in different microareas, and the existence of the Raman modes at cubic phase was attributed to the PNRs with lower symmetry. Our results imply that the structural inhomogeneous in PMN-33%PT single crystal closely relates with the temperature evolution of PNRs.

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