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Citation: *Journal of Applied Physics* **101**, 064107 (2007); doi: 10.1063/1.2713080

View online: <http://dx.doi.org/10.1063/1.2713080>

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Nanoparticle decoration of ferroelectric domain patterns in LiNbO₃ crystal

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(Received 6 July 2006; accepted 20 January 2007; published online 22 March 2007)

A scatheless visualization has demonstrated that the engineered and spontaneous domains of lithium niobate (LiNbO₃) crystal can be delineated by means of the nanoparticle decoration. These nanoparticles were monodispersed in nonpolar solvent and negatively charged under the existence of aerosol-OT (AOT or sodium di-2-ethylhexylsulfosuccinate). This method was based on the process of electrostatic interaction, in which the LiNbO₃ crystal wafers were heated in the presence of decorating colloid solvent. The charged nanoparticles deposited preferentially on the surfaces of those domains with the opposite charges. The delineated patterns corresponded with the polarization property of domain structures and consequently provided a domain contrast image with resolution down to micron scale. This decoration technique is effective, easily operated, nondestructive for sample, noninvasive for human body, and especially, is more suitable for the domain mapping of oxide ferroelectric crystals. In addition, the initial exploration of the model of such decoration process is also presented in this article. © 2007 American Institute of Physics.

[DOI: [10.1063/1.2713080](https://doi.org/10.1063/1.2713080)]

I. INTRODUCTION

The ferroelectric crystal, lithium niobate (LiNbO₃), is a mature material which has been widely applied in the field of optoelectronics and holographic storage due to its good electron-optic, acousto-optic, and nonlinear optical performances. Recently, periodically poled lithium niobate (PPLN) has attracted great attention for its potential application in quasi-phase matching (QPM).¹⁻³ It has been noticed that the fabrication of a domain pattern with good periodicity and flat domain walls plays a crucial role in those applications. Till now, many methods have been developed for the mapping of domain pattern. For instance, the chemical etching techniques,⁴ environmental scanning electron microscope (ESEM),⁵ scanning force microscope (SFM),^{6,7} x-ray morphology,⁸ etc. However, they are usually either destructive, inconvenient, or need some expensive instruments.

Pearson and Feldmann described a technique for observing surface domain structures in ferroelectric crystals.⁹ Although this method was developed some 50 years ago, it has been used very little. This is because it can only be used to delineate the large domains in range of millimeter and its decorating quality is not so good. In this article, we present an updated method to delineate both engineered and spontaneous domains structure pattern in LiNbO₃ crystal. In our method, the likely charged nanospheres, monodispersed in nonpolar solvent, were applied on the surface of sample. Owing to the electrostatic interaction, the nanoparticles with negative charges could deposit on the polarized surface of negative domains and were repelled by that of positive domains selectively. Therefore, the domain contrast image with

resolution down to micron scale could be nondestructively provided. Furthermore, the initial exploration of the model of such decoration process is also presented in this work.

II. EXPERIMENT

A. Preparation of crystal samples

It is well known that the spontaneous polarization P_s of LiNbO₃ can be reversed by a pulse electric field at room temperature.¹⁰ Using different electrode patterns, LiNbO₃ crystals can be engineered by field poling method into periodic and quasiperiodic domain structures, which have been widely used in acoustics, nonlinear optics, and portable solid-state laser devices. In our experiment, two Z-cut LiNbO₃ crystal wafers were used. One (sample 1) with a periodic domain structure was prepared by engineered polarization method and measured the widths of negative and positive domains in one period by an optical microscope (XJZ-6A, JNOEC). Under our experiment condition, their corresponding values are 4.0 ± 0.5 and 6.0 ± 0.5 μm , respectively, i.e., the duty cycle of sample 1 is about 1:1.5, which means that the positive domains are slightly wider than the negative ones as shown in Fig. 3. The other one (sample 2), with island-shaped spontaneous domain structures, was fabricated directly by the Czochralski growth method. Both of these samples were optically polished and cleaned, and no domain pattern could be observed using optical microscopy before decoration processes.

B. Preparation of decorating medium

Polystyrene (PS) nanospheres prepared by surfactant-free emulsion copolymerization were used as the decoration

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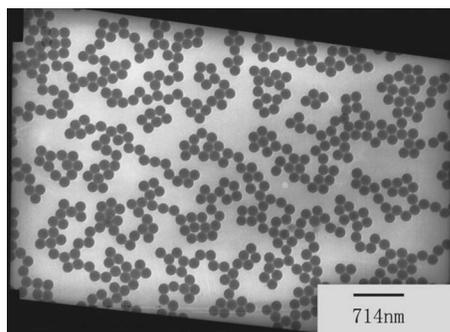


FIG. 1. TEM image of PS particles with diameter of 130 nm.

medium. The average particle diameter was measured using transmission electron microscopy (JEOL JEM-200CXII) and found to be 130 nm in the mean size value; the size distribution was less than $\pm 5\%$ in the diameter range of PS particles (Fig. 1). These nanospheres were cleaned via multiple cycles of centrifugation, removal of supernatant, and the precipitate was dried in vacuum desiccators for 24 h. To avoid the neutralization for the free charges on the sample surface when it was submerged in the colloid suspension, an appropriate nonpolar solvent with low dielectric constant and weak viscosity should be chosen to disperse these PS particles equally, so that the charged colloidal particles could move freely toward the ferroelectric domains with the opposite charges under the electrostatic attraction. Because the crystal samples would be heated up to 60°C during the following process of domain delineation, the instability of the liquid induced by volatilization would strongly influence the distribution of the charged nanoparticles. In order to reduce this negative effect, the solvent should have low volatilization rate and high boiling point. Although many organic liquids meet these requirements, we found dodecane ($\epsilon \approx 2$, $h = 1.5 \text{ mPa s}$, $T_{\text{boiling}} = 200^\circ\text{C}$) to be the most satisfactory.

Usually, the dried PS nanoparticles are neutral. In order to make these particles similarly charged in the dodecane, the aerosol-OT (AOT, or sodium di-2-ethylhexylsulfosuccinate), a surfactant with a sulfonate group on its polar head and two branched hydrocarbon tails,¹¹ was added into the mixture of PS particles and dodecane. The development of a net charge at the particle surface affects the distribution of ions in the neighboring interfacial region, resulting in an increased concentration of counterions—ions of charge opposite to that of the particle—close to the surface. Thus, an electrical double layer is formed in the region of the particle-liquid interface, known as the zeta potential. The zeta potential is a function of the surface charge of these colloidal particles with the nature and composition of the surrounding medium in which the particle is suspended, and has proven to be extremely relevant to the practical study for the control of colloidal stability and flocculation processes. In our experiment, the zeta potential and the average size of these 130 nm PS particles in that nonpolar solvent were measured and found to be $-25.10 \pm 4.0 \text{ Mv}$ and $252 \pm 10 \text{ nm}$, respectively. (Brookhaven Zeta-PALS: standard deviation, 18 measurements.) These data showed that the PS particles were negatively charged in the present of AOT. Furthermore, the diam-

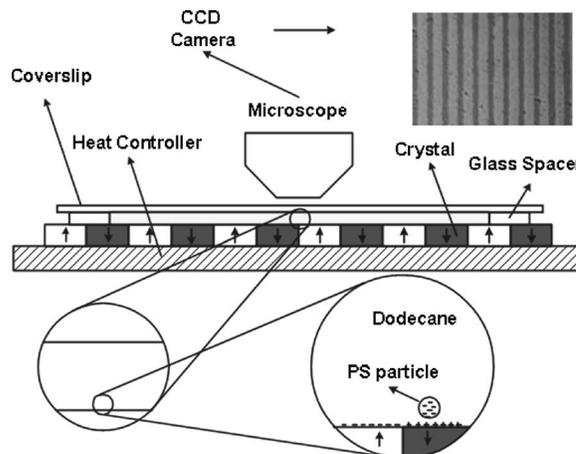


FIG. 2. Schematic diagram of the experimental setup.

eter change of PS spheres might be caused by the expanding effect in organic solvent or weak aggregation of these nanoparticles.

C. Visualization and analysis

The experimental process of delineating the ferroelectric domain structure contained two steps. First, the colloid suspension was confined in a microcell, between a glass coverslip and the sample wafer, as shown in Fig. 2. The function of the coverslip was to decrease the instability of the colloid suspension caused by the solvent's volatilization and the air convection. The microcell between the coverslip and crystal sample was isolated by two glass spacers with separated distance of roughly $50 \mu\text{m}$.

After the colloidal suspension was confined in the cell, a semiconductor heat controller placed under the bottom of the cell was used to heat the sample up to 60°C in several minutes. Then, the selective sedimentation of PS particles on the sample surface could be observed by our optical microscope. Moreover, finer pictures were obtained by removing the coverslip after dodecane was all vaporized out. These pictures indicated the domain configuration of our LiNbO_3 samples clearly. The display quality depended largely on the appropriate concentrations of PS particles and added AOT. Optimum contrast images were obtained under the following conditions: PS particle volume density, 0.06%; AOT concentration, 10 mM; and the crystal sample was heated from room temperature ($\sim 25^\circ\text{C}$) to 60°C in several minutes.

The color digital image of the domain patterns was recorded by a charge-coupled device camera attached to our optical microscope. In the image of the periodic domain structure, the repeat distance of the stripe patterns was analyzed by two-dimensional fast Fourier transform (FFT). Measuring the distance between the center and the prominent points in the transformed image, the periodicity in the original image can be calculated. The distance in the transformed image can be related to the distance in the original image using the equation $d = N/r$. As Figs. 3(a) and 3(b) illustrate, d is the center-to-center line spacing in the original image, N is the total pixel numbers of that image in the x direction, and r is the distance in pixels from the center of the transformed

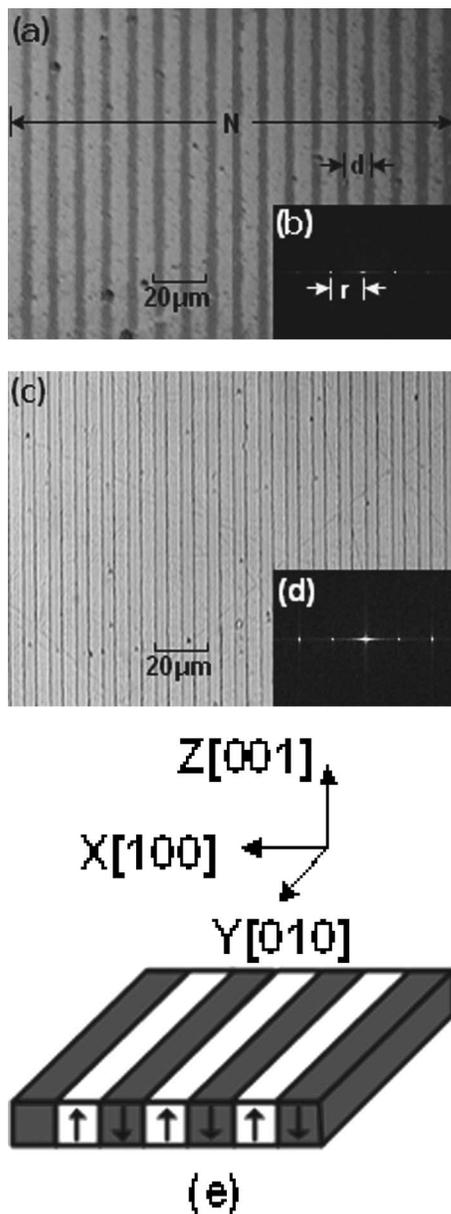


FIG. 3. Comparison of domain images of surface portion in sample 1. (a) Polished surface with PS particles decoration. (b) FFT of the pattern (a). (c) Etched surface. (d) FFT of the pattern (c). (e) The relation between coordinate and orientation of sample 1.

image to the center of the prominent points (all of these variables are in the unit of pixel).¹² To confirm the experimental results, these two samples were etched in a 2:1 mixture solution of HNO_3 and HF at room temperature as usual. The domain patterns on the etched surface of samples were observed as comparison with those patterns obtained from the nanoparticle decoration technique.

III. RESULTS AND DISCUSSION

In ambient conditions, the spontaneous polarization charges on the surface of the ferroelectric crystal are effectively compensated by free-charge carriers from the environment; correspondingly, they cannot affect the distribution of the charged PS nanospheres.⁶ In order to get the domain contrast image, the samples were heated up to 60 °C from

room temperature during the decorating process. Because of the pyroelectric property of LiNbO_3 crystal, the surface of negative domains bore positive charges and that of positive ones had negative charges. Consequently, the PS particles with the negative charges were just deposited on the region of negative domains, and thus the domain contrast image could be observed by optical microscope. Figure 3 shows the comparison between the nanoparticles' decorating image and the chemical etching image, and clearly indicates the domain patterns of sample 1 with antiparallel periodic domain structures. The locations and shapes of the contrast patterns in Fig. 3(a) match the etching patterns in Fig. 3(c) very well. This confirms that the contrast stripes originated from the antiparallel periodic domains. From Figs. 3(a) and 3(b), from Sec. II C as mentioned, the measured values are 230 for the N and 17 for the r , respectively. From the equation of $d = N/r$, the period of original image, d , is calculated to be 13.53. According to the scale bar, there are 27 pixels in the range of 20 μm. Therefore, the period of original image, d , is calculated to be 10.2 ± 0.1 μm. Direct measurement from the etching image [Fig. 3(c)] shows that the period of domain pattern is 10.0 ± 0.5 μm. This result shows good agreement with the FFT calculation, although it is only the statistical mean result because the domain structures cannot be manipulated so perfectly by the current experimental technique.

Sample 2 has irregular island-shaped spontaneous domain structures. In order to further confirm our experimental result, sample 2 was etched slightly (4 min in hydrofluoric acid) before the decoration process was employed. Figure 4(a) shows the etching picture of sample 2. It is found that the domain walls have been clearly designated by the atrous lines, and the contrast between negative domains and positive ones is very weak due to the short etching time. Figure 4(b) shows the contrast patterns using our nanoparticle decoration method. It is obvious that almost all particles have been confined in the area of negative domains, and the domain structure patterns have been clearly revealed by PS nanoparticle decoration. Figure 4(c) shows the relation between coordinate and the orientation of sample 2.

As mentioned above, the negative-charged nanoparticles played a governing role in our decoration method. According to traditional theory, however, the electrostatic barrier in nonpolar solvents ($\epsilon \approx 2$) to charging is 40 times larger than in aqueous systems ($\epsilon \approx 80$) and much greater than $k_B T$. The common expectation is that charge effect for nanoparticles in nonpolar solvent is inconsequential. But, due to the presence of certain surfactants, such as the AOT, charge exists in many nonpolar colloids as in aqueous systems. Recently, measurements with the surface force apparatus and atomic force microscope have demonstrated repulsive electrostatic forces between surfaces in Refs. 13 and 14. Hsu and Dufresne *et al.* have also given a framework for understanding charging behavior in nonpolar colloids.¹¹ According to their conclusion, the surfactants form nanoscale reverse micelles when the concentration of AOT is above the critical micellar concentration. Furthermore, the thermal energy enables these micelles to ionize and form negative and positive ions in the nonpolar solvent. Under the drive of entropy, the colloid particles are negatively charged.

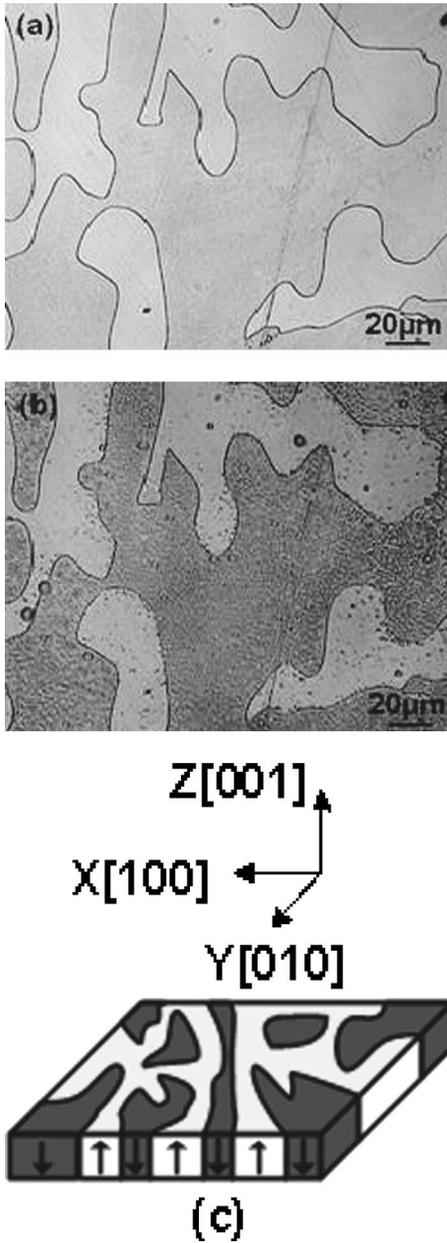


FIG. 4. Comparison of domain images of surface portion in sample 2. (a) Slightly etched pattern surface. (b) The same surface with PS particles decoration. (c) The relation between coordinate and orientation of sample 2.

To semiquantitatively understand the decoration process, a simple model for the case of periodic domain structure (sample 1) is discussed in the following text. Taking into account the influence of the periodically distributed surface charge, the total electrostatic potential in the vicinity of the surface of sample 1 can be calculated as¹⁵

$$\begin{aligned} \varphi(x, z) = & 4\lambda p \times \Delta T \{ [1 + (\varepsilon_a \varepsilon_b)^{1/2}] \varepsilon_0 \}^{-1} \\ & \times \sum_{n=0}^{+\infty} \{ (2n+1)\pi \}^{-2} e^{-(2n+1)\pi(z/\lambda)} \sin[(2n+1)\pi(x/\lambda)], \end{aligned} \quad (1)$$

where x and z are the coordinates, respectively, in the non-polar and polar directions, as show in Fig. 3(e). Here, for simplicity, we assume the width of the positive domains is

the same as the negative domains, namely, the half period of the domain structure, $\lambda (=5 \mu\text{m})$. $p \times \Delta T$ gives the surface charge density, where $p (= -1.6 \times 10^{-3} \text{ C m}^{-2} \text{ K}^{-1})$ is the pyroelectric coefficient of LiNbO_3 crystal, $\Delta T (=35 \text{ K})$ is the temperature change value. $\varepsilon_a (=30)$ and $\varepsilon_b (=84)$ are the relative dielectric constants in the x and z coordinates, respectively, and ε_0 is the vacuum dielectric constant. From Eq. (1), it is found that the surface potential of sample 1 is also periodically distributed, which is consistent with its domain structure characteristic. The absolute value of the surface potential in the x - y plane is almost uniform ($|\varphi_0| = 300 \pm 10 \text{ V}$, $z=0$) when the sample is heated up from 25°C to 60°C ; the electrostatic potential in the area of positive domain surface is $|\varphi_0|$ and in the area of negative domain is $-|\varphi_0|$, respectively. The zeta potential of a charged surface, which was submerged in the colloid suspension, is given by the Gouy-Chapman theory, which describes the effect of a static surface charge on the semi-infinite surface in the plane.¹⁶ A negative surface charge leads to formation of a double layer, since positive ions in solution tend to balance the negative surface charge. For simplicity, we will only consider external surface charge. This is the relevant case for most experiments, where ion substitutions are only made in the external solution. According to this theory, there are three different conditions: $Ze|\varphi_0|/K_B T \ll 1$, $Ze|\varphi_0|/K_B T \sim 1$, and $Ze|\varphi_0|/K_B T \gg 1$. In our experiment $|\varphi_0| = 300 \pm 10 \text{ V}$, so the condition of $Ze|\varphi_0|/K_B T \gg 1$ is satisfied. As a result, the potential distribution near the negative domain's surface can be expressed as $\varphi_s^- = (4K_B T / Ze) e^{-\kappa z}$, and near the positive domain's surface is given as $\varphi_s^+ = -(4K_B T / Ze) e^{-\kappa z}$, which is also given by the Gouy-Chapman theory (z is the distance away from the sample's surface, $Z (=1)$ is the charge number of one reverse micellar, $\kappa (=1 \mu\text{m}^{-1})$ is the screening length of colloid when the concentration of AOT is 10 mM).¹¹ Taking the periodical domain pattern into account, the surface potential of sample 1 submerged in nonpolar colloid is given by the following equation:

$$\begin{aligned} \varphi(x, z)_s = & (16K_B T / Ze) \times e^{-\kappa z} \\ & \times \sum_{n=0}^{+\infty} \{ (2n+1)\pi \}^{-1} \sin[(2n+1)\pi \times x/\lambda]. \end{aligned} \quad (2)$$

Figure 5 shows the diagram of the surface potential of sample 1 which was submerged in the colloid suspension. We have not plotted the Y axis owing to the uniform surface potential in this direction.

Furthermore, the interaction between a nanoparticle and an isolated surface can be given by Derjaguin-Landau-Verwey-Overbeek (DLVO) theory, which is widely used to explain colloidal stability and other phenomena arising from the charging of particles and the surfaces in polar liquids. Here, the sphere-shaped particle is assumed to be a point charge. So, depending on Coulomb's law, the charge of this particle can be given by $q = 4\pi\varepsilon_r\varepsilon_0 a\varphi_p$, where a and φ_p represent the radius and zeta potential of the particle, respectively. As a result, the interaction between the particle and charged wall can be simply expressed as^{17,18}

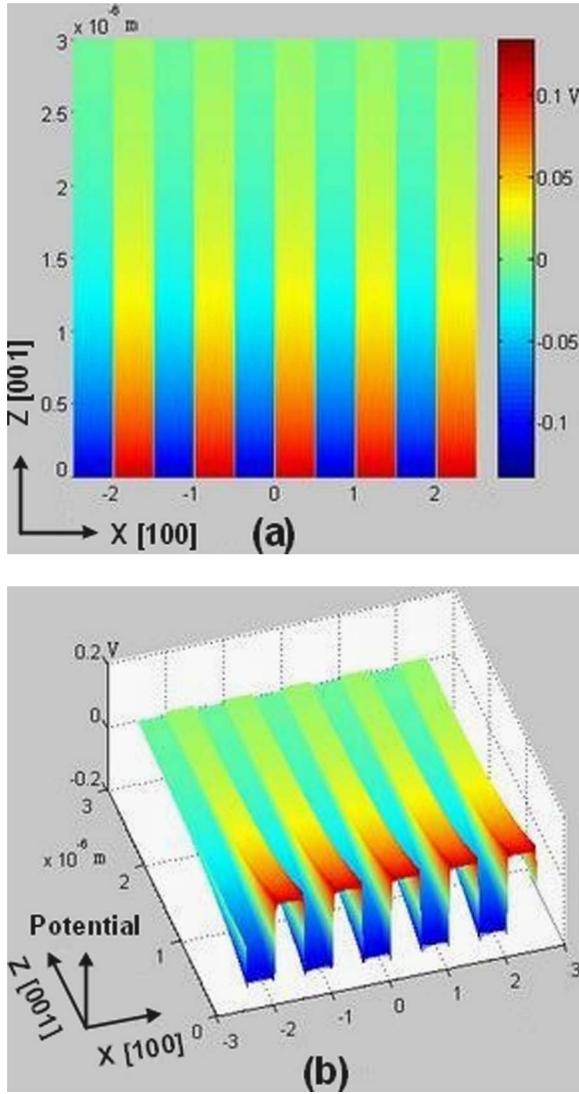


FIG. 5. (Color online) Diagram of the surface potential of sample 1. (a) 2D figure of the surface potential, Z-axis denotes the distance away from the sample's surface, X-axis denotes the location of positive and negative domains, and color column denotes the electrostatic potential value. (b) 3D figure of the surface potential; the potential value is denoted by potential axis.

$$u_{pls}(z) = 4\pi\epsilon_r\epsilon_0 a\varphi_p\varphi_s. \quad (3)$$

In the condition of our experiment, $\epsilon_r(=2)$ is the dielectric constant of dodecane, z ($=126$ nm, equal to the radius of PS sphere a) is the distance between the sample's surface and the center of PS sphere, $T(=333$ K) is the temperature of heat controller, $\varphi_p(=-25.10\pm 4$ mV) is the zeta potential of the PS nanoparticles when the AOT concentration is 10 mM, $|\varphi_s|(=95\pm 1$ mV) is the absolute surface potential of sample 1 which was submerged in the colloid suspension. The energy difference between the positive and negative domains in the vicinity of the surface of sample 1 is given by $\Delta u = 8\pi\epsilon_r\epsilon_0 a\varphi_p|\varphi_s| = 0.79\pm 0.2$ eV. According to the Boltzmann distribution, $n_{\text{neg}}/n_{\text{pos}} \approx 6.8 \times 10^{11}$ (n_{negative} is the probability of PS particles appearing on the negative domains; n_{positive} is the probability of PS particles appearing on the positive domains). This means almost all the PS particles with negative charges deposited on the negative domains section during

our experiment procedure, and agrees very well with the phenomenon shown in Fig. 3(a).

From the expression of the crystal's surface electrostatic potential [Eq. (2)], we can conclude that the electric field along with axis Z is also nonuniform in the vicinity of the surface of sample 1. In this case, the particles will be attracted by both the negative and positive domains due to the surface polarization arisen from the nonuniform electric field. For simplicity, we assume that a single charged spherical particle suspended in an electrolyte is subjected to a nonuniform electric field. According to dielectrophoretic (DEP) theory,¹⁹ the dielectrophoretic force F_{DEP} acting on the particle is then given by the following general expression:

$$F_{\text{DEP}} = 2\pi\epsilon_m r^3 \text{Re}[b] \nabla |E|^2, \quad (4)$$

where r is particle radius, ϵ_m is the dielectric constant of the suspension medium, E is the applied electric field, ∇ is the del vector operator, and $\text{Re}[b]$ is the real part of the dipole factor (also called the Clausius-Mossotti factor, which is the frequency-dependent complex). It is shown from Eq. (4) that the F_{DEP} is directly proportional to the particle volume ($\sim r^3$); therefore, as the particle size is decreased, the DEP force experienced by the particle becomes smaller. The minimum particle size required for dielectrophoretic motion is estimated to be around 400–600 nm under reasonable physical conditions.²⁰ In our experiment, the nanoscale PS sphere with diameter of ~ 100 –200 nm was chosen, so the weak DEP force acting on the particles could be neglected and our results of the nanoparticles decoration were found to be satisfactory.

IV. CONCLUSION

The contrast images of the periodic domains and the island-shaped domains of LiNbO_3 crystals have been successfully revealed by the nanoparticle decoration technique. This method shows a higher domain resolution than the traditional one due to the use of fine nanoparticles. This delineating technique is also very promising since it allows us to directly observe microscale domain structures in an unetched sample surface. The decoration process to obtain the contrast image is scatheless for the sample, and does not need any complicated instruments. Some primary considerations for the decorating process had also been carried out, depending on a relative simple electrostatic interaction model. There are several effects that have been ignored, for instance, the surface potentials of the samples may be affected due to the existence of the charged PS particles, and so on. A detailed study is in progress.

ACKNOWLEDGMENTS

We are thankful for helpful discussions and suggestions from Wang Zhen-Lin, Xue Qi, Zhu Yong-Yuan, Wang Guang-Hou, Ma Yu-Qiang, Han Ming, and Li Ai-Dong. This work was supported by the National 863 and 973 Projects Foundation of the People's Republic of China.

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