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Excess oxygen ordering in the $La_2NiO_{4+\delta}$ system studied by low-frequency internal friction

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Different elastic spectra were found for $La_2NiO_{4+\delta}$ in the three ranges: $\delta < 0.110$, $0.110 < \delta < 0.145$, and $\delta > 0.145$. It is concluded that for $0.110 < \delta < 0.145$ the low-temperature phase consists of phases with three-dimensional (3D) ordering and one-dimensional (1D) ordering of interstitial oxygen; the change of the ordering state of excess oxygen from 1D to 3D with increasing oxygen content is not abrupt but smooth; and for $\delta > 0.145$ all excess oxygen is three-dimensionally ordered. A model of the 3D ordering clusters for $0.110 < \delta < 0.145$ is discussed. Glass transitions of the interstitials and glass dynamic phenomena were also observed.

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I. INTRODUCTION

The behavior of excess oxygen in a K_2NiF_4-type structure has attracted much attention of researchers since superconductivity was found in $La_2CuO_{4+\delta}$.[1–3] $La_2NiO_{4+\delta}$, which shows no superconductivity, has been studied as a good reference compound to $La_2CuO_{4+\delta}$ because of the larger excess oxygen content compared to $La_2CuO_{4+\delta}$. Excess oxygen atoms like to order at low temperature; the ordered arrangement of excess oxygen atoms leads to the ordered deformation of NiO_2 planes, which are expected to affect the potential energy acting on the holes in NiO_2 planes, and thus affect the nature of the spin and charge orderings. For $\delta > 0.110$ in $La_2NiO_{4+\delta}$ the magnetic ordering changes abruptly from commensurate to incommensurate (or long-period commensurate) and the charge ordering appears with a change of interstitial correlations, from one-dimensional (1D) staging to 3-dimensional (3D) staging order.[4–6] This leads to the question of the interplay of the spin, charge, and lattice degrees of freedom. Tranquada *et al.* suggested that the charge correlations are fluctuating about an average ordered configuration determined by the ordered interstitial oxygen.[7–9] The plateaus for the magnitude of the incommensurate splitting, ϵ, demonstrate that the charge stripes are coupled to the lattice[8] and Tranquada *et al.* suggested that the occurrence of the rational fraction of ϵ is the result of a competition of two length scales, one associated with the ideal spin-charge order and the other associated with modulation of the lattice potential due to the ordering of the oxygen interstitials.[6] The interplay remains unclear partly due to the limited knowledge of the state of the interstitials. Tranquada *et al.* proposed a model of a completely ordered arrangement of excess oxygen atoms corresponding to a stoichiometric value of $\delta = 2/15$ for a $La_2NiO_{4.125}$ single crystal, but the imperfect agreement between the observed and calculated intensities of the superlattice peaks and the presence of diffuse scattering at 10 K indicate that a certain disorder remains in the arrangement of the excess oxygen atoms even in the low-temperature ordered phase.[10] Tamura *et al.* found a first-order phase transition from the orthorhombic phase (*Fmmm*) to the tetragonal phase ($I4/mmm$) for $0.15 < \delta < 0.18$ and suggested that for $\delta > 0.15$ the *Fmmm* phase is correlated to the 3D ordering of the excess oxygen.[11,12] There are some discrepancies among the above results; thus, whether the ordering of the excess oxygen really exists and to what extent the disorder in the arrangement of the excess oxygen atoms remains in the low-temperature phase is an important issue. The internal friction technique, which in general measures the dissipation of elastic energy (Q^{-1}), has proved to give valuable information about defects in ceramic samples.[13–16] In this paper we present the results of the internal friction study of a series of $La_2NiO_{4+\delta}$ samples with different excess oxygen and discuss the relation of the state of the ordering of the excess oxygen and the internal friction spectra.

II. EXPERIMENT

The rectangular samples in form of $30 \times 4.2 \times 1.2\ \mathrm{mm}^3$ are prepared by the conventional solid-state reaction from a stoichiometric mixture of highly pure La_2O_3 and NiO powders. The content, δ of excess oxygen was adjusted by changing temperature and atmosphere of subsequent annealing or by the electrochemical oxidation method. The value of the overall oxygen content was achieved by iodometric titration with absolute random errors of ±0.003. The internal friction and shear modulus were measured in a computer-controlled automatic inverted torsion pendulum with the forced-vibration method with the maximum torsion strain amplitude kept at 2×10^{-5}.

III. RESULTS AND DISCUSSION

Through chemical analysis, the excess oxygen of the as-prepared sample is 0.140; the excess oxygen of the samples annealed at N_2 atmosphere at different temperature are 0.071, 0.110, 0.121, 0.135; the excess oxygen of the sample achieved by electrochemical oxidation is 0.147.

Figure 1 shows the temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.110}$ with various frequencies at a heating rate of 2 K/min. An internal friction peak was observed around 250 K, accompanied by a corresponding decrease on the shear modulus. The peak shifts to higher temperature with increasing frequency, which demon-

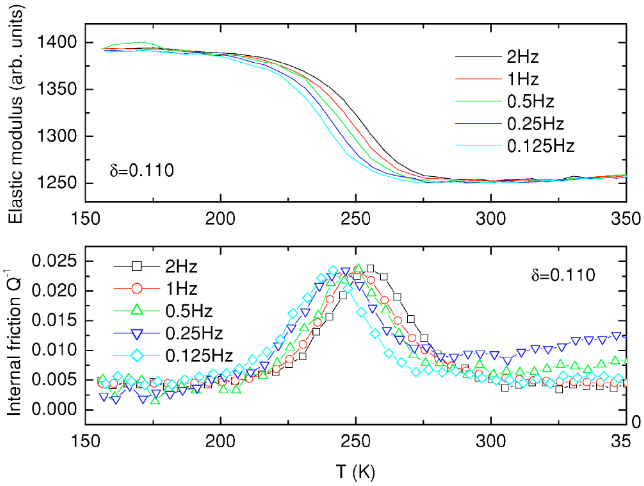

FIG. 1. (Color online) Temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.110}$ with various frequencies at a heating rate of 2 K/min.

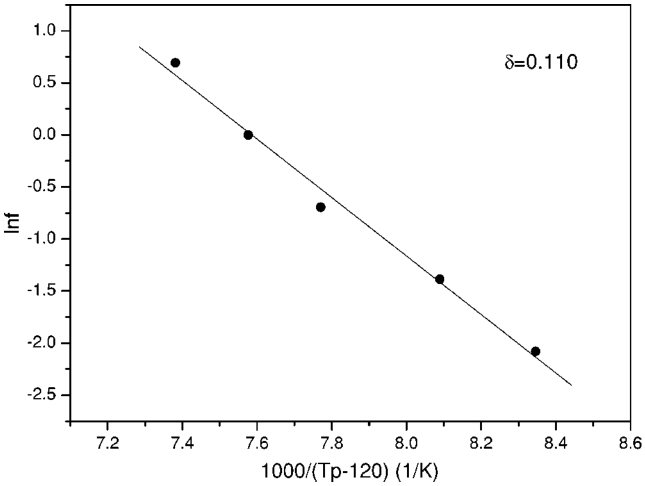

FIG. 2. $\ln f$ vs $1/(T_P-T_0)$ curve.

strates that the relaxation rate of the process is thermally activated. For a thermal relaxation process, we have the Arrhenius relation of $\tau=\tau_0\exp(E/kT)$, where τ is the relaxation time, τ_0 is the relaxation time extrapolated to infinite temperature and E is the activation energy. For the case of a Debye peak, the condition that

$$\ln(\omega\tau)=0 \tag{1}$$

at the peak gives $\ln(\omega\tau_0)+E/kT_P=0$, where T_P is the temperature at the peak. Substituting $\omega=2\pi f$ into the above equation, we have

$$\ln(2\pi f\tau_0)+E/kT_P=0, \tag{2}$$

where f is the vibration frequency of the specimen. Through the $\ln f$ vs $1/T_P$ curve, we obtained $E=0.9$ eV, $\tau_0=8\times10^{-20}$ s for $La_2NiO_{4.110}$. That τ_0 is so low compared to the values characteristic of atomic hopping which are $\tau_0\sim10^{-14}-10^{-12}$ s, strongly indicating that there is a glass transition process of the hopping of interstitial oxygen.[17] For a glass transition process, the relaxation time follows the Vogel-Fulcher law

$$\tau=\tau_0\exp[E/k(T-T_0)], \tag{3}$$

where T_0 is the freezing temperature. Analogically, combined with Eq. (1), we have

$$\ln(2\pi f\tau_0)+E/k(T_P-T_0)=0. \tag{4}$$

We assume the freezing temperature of the hopping of the interstitials is about 120 K (with $\tau\sim10^9-10^{12}$ s calculated from Ref. 17), which is used in the $\ln f$ vs $1/(T_P-T_0)$ curve in Fig. 2. Then we obtained $E=0.24$ eV, $\tau_0=1\times10^{-11}$ s for $La_2NiO_{4.110}$. Although the E and τ_0 we obtained are not the real values because of the uncertain T_0, the analysis above clearly confirms the existence of the glass transition process. Kyômen *et al.* also found a glass transition phenomenon due to freezing-in of the diffusion process of excess oxygen over a wide temperature range of 150–300 K for $La_2NiO_{4.094}$.[17] Considering E and τ_0 obtained by the above analysis and in addition the fact that the interstitial oxygen remains mobile down to 150–200 K,[17,18] we suggest that the relaxation peak is due to the hopping of single interstitial oxygen in *ab* plane.[16,18] It is reported that a peak due to oxygen pairs locates just at the high-temperature side of the peak due to single interstitial oxygen in the $La_2CuO_{4+\delta}$ system,[16,18] which is also observed by the low-frequency internal friction method.[19] The absence of the peak due to oxygen pairs in the $La_2NiO_{4+\delta}$ system indicates that most of the excess oxygen forms large stable clusters in the *ab* plane, which do not contribute to internal friction. This also indicates that in the 1D staging order there is some kind of structure of interstitial oxygen in the occupied interstitial layers even though the density of the interstitials is very low. This kind of structure is believed needed to provide a force for the phase separation process to different 1D staging order in the $La_2NiO_{4+\delta}$ system.[4] Similar relaxation peaks with the same relaxation strength were observed in the cooling process for $La_2NiO_{4.110}$ and the height of the peak does not change in the cooling process from two different temperatures, room temperature and 450 K. This means that the peak is not affected by 1D ordering, and also indicates that the clusters of interstitial oxygen are stable at high temperature, which is also suggested by Tranquada *et al.* to explain the negligible change in the orthorhombic strain for $La_2NiO_{4.105}$.[4] The elastic spectra observed for $La_2NiO_{4.071}$ are similar compared to $La_2NiO_{4.110}$, which is not shown in this paper.

It is reported that a change of interstitial correlations, from a 1D staging to a 3D staging order occurs at $\delta=0.110$.[4–6] Now we discuss $La_2NiO_{4+\delta}$ samples in the range of $0.110<\delta<0.145$ with different elastic spectra. The temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.135}$ and $La_2NiO_{4.140}$ with various frequencies in a cooling process from 450 K is shown in Fig. 3. For $La_2NiO_{4.140}$ there is a frequency-independent softening of the shear modulus, which is caused by the 3D ordering process of interstitial oxygen,[4–6] while the absence of this softening for $La_2NiO_{4.135}$ indicates that the 3D ordering process starts at a lower temperature. This also shows that the samples with larger excess oxygen have a stronger ability to form 3D ordering clusters. Wochner *et al.* found measurable effects of

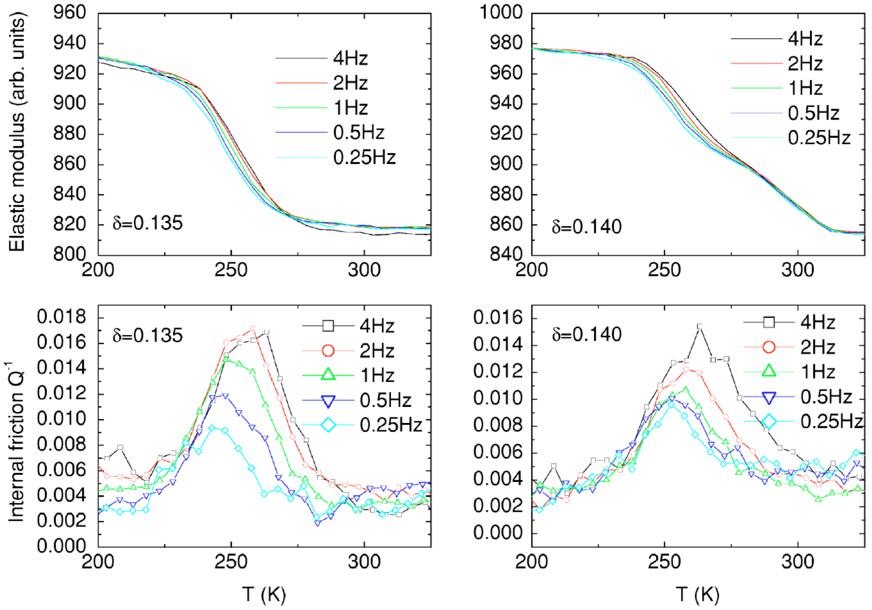

FIG. 3. (Color online) Temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.135}$ and $La_2NiO_{4.140}$ with various frequencies in a cooling process from 450 K.

the cooling rate on the interstitial order for $La_2NiO_{4.125}$ while not for $La_2NiO_{4.133}$,[8] which also confirms this point. The internal friction curves are more complex to explain. The low-temperature side of the peaks for both of the two samples is independent of frequency, and the peak height increases considerably with temperature in the frequency range from 0.25 to 2 Hz for $La_2NiO_{4.135}$ and from 1 to 4 Hz for $La_2NiO_{4.140}$. All these features are typical characters of the dispersion of the imaginary susceptibility of spin glasses, mixed ferroelectric/antiferroelectric system,[20] or relaxor ferroelectrics,[21,22] and Cordero *et al.* also found this phenomenon in the elastic spectra of Ca-doped $LaMnO_3$ system.[23] Therefore the phenomena we observed are glass dynamics, which seems to originate from the 3D ordering process mentioned above, leading to an extremely broad distribution of relaxation times. Then the whole internal friction peaks can be explained for $La_2NiO_{4.135}$ and $La_2NiO_{4.140}$, because they are affected by the beginning and tail of the 3D ordering process separately, so the peak height changes only a little for $La_2NiO_{4.135}$ with $f=4$ Hz and $f=2$ Hz and for $La_2NiO_{4.140}$ in the frequency range from 0.25 to 1 Hz. To confirm this point, we annealed the sample at room temperature for a long time and performed a measurement in the cooling progress from room temperature. As shown in Fig. 4, the peak height changes only a little for different frequency, which shows that the 3D ordering process is weak or even already finished at room temperature after sufficient time.[4] The peak height is lower in the cooling process from room temperature compared to the value from 450 K, which indicates that the number of interstitial oxygen atoms that contribute to the internal friction peak is smaller because of the 3D ordering process, and also indicates that the interstitial oxygen atoms in the stable 3D ordering clusters do not contribute to the internal friction peak because they are pinned.

In order to study the state of interstitial oxygen in the low-temperature phase further, we cooled the $La_2NiO_{4+\delta}$ samples slowly to low temperature and performed measurements in the heating process. Figure 5 shows the temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.121}$ with various frequencies at a heating rate of 2 K/min. There is a relaxation peak accompanied by a corresponding decrease on the modulus. The similarity of the peak compared with $\delta<0.11$ indicates the same origin for it. Tranquada *et al.* found that the diffuse scattering at 10 K for a $La_2NiO_{4.125}$ single crystal is similar to the type of scattering observed in $La_2NiO_{4+\delta}$ with $0.05<\delta<0.11$, where the interstitials order one dimensionally in a staged fashion.[10] We suggest that the peak of $La_2NiO_{4.121}$ is due to the hopping of single interstitials in the 1D ordering phase at low temperature. The frequency-independent softening of the shear modulus is due to the disordering process of the 3D ordering clusters. The disordering process always occurs after the interstitials have the high mobile ability, indicating that the disordering process takes place by the diffusion process of the interstitials. No glass dynamics was observed for $La_2NiO_{4.121}$ in the heating process, indicating that the disor-

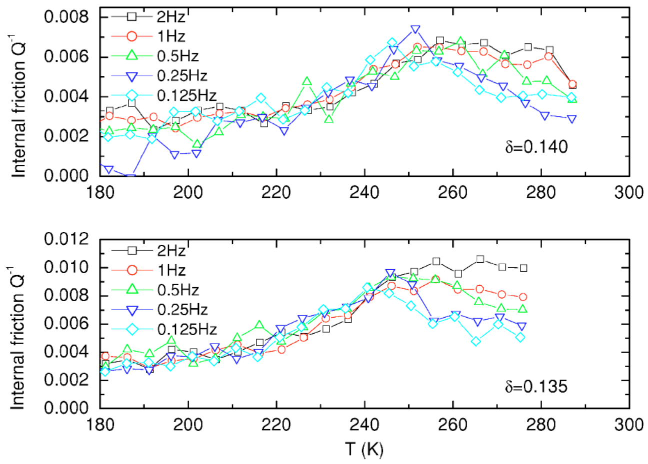

FIG. 4. (Color online) Temperature dependence of the internal friction of $La_2NiO_{4.135}$ and $La_2NiO_{4.140}$ with various frequencies in a cooling process from room temperature.

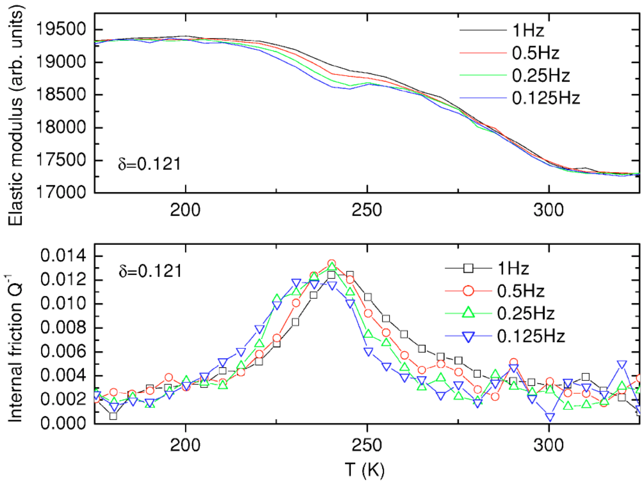

FIG. 5. (Color online) Temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.121}$ with various frequencies at a heating rate of 2 K/min.

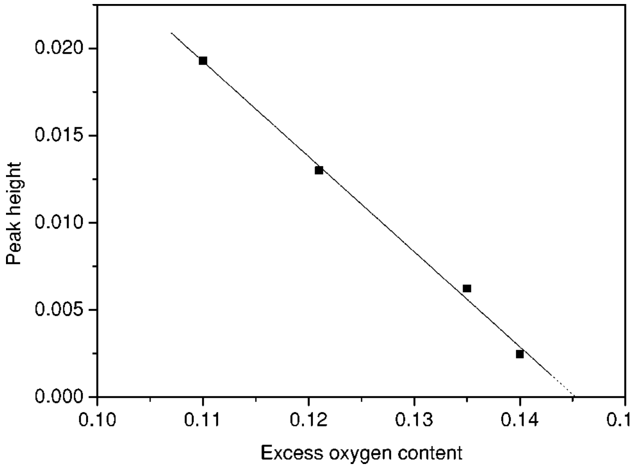

FIG. 7. Excess oxygen content dependence of the peak height of the internal friction peak with $f = 1$ Hz measured in the heating process with a rate of 2 K/min.

dering process starts at a higher temperature compared to the relaxation region. To confirm this further, we measured the internal friction with a very slow heating rate and did not find any change of the peak height. This can be understood easily because in the relaxation region, even if the disordering process occurs, it should be very slow because of the low mobility of excess oxygen. So the information obtained from the elastic spectrum is related to the state of interstitial oxygen in the low-temperature phase. Then the coexistence of the relaxation peak and the frequency-independent softening of shear modulus shows the coexistence of 1D ordering and 3D ordering arrangement of interstitial oxygen in the low-temperature phase. Similar phenomena were also observed for $La_2NiO_{4.135}$ and $La_2NiO_{4.140}$.

Figure 6 shows the temperature dependence of the shear modulus of $La_2NiO_{4+\delta}$ samples with various frequencies in the heating process. The fraction of relaxation softening shows a decreasing tendency for samples with a larger excess oxygen content, indicating that the fraction of 1D ordering of interstitial oxygen decreases. Because the shear modulus is affected by many factors (such as density, size, etc.), it is more suitable to use the internal friction to do the analysis quantitatively. Assuming that the elastic dipole does not change in samples with a different excess oxygen content, according to Nowick's theory,[24] the peak height is proportional to the concentration of the defects, what here is the content of single interstitial oxygen in the 1D ordering state. Although most of the interstitial oxygen in 1D ordering forms clusters which do not contribute to the internal friction peak mentioned already, it is reasonable to assume that the amount of the single interstitials is proportional to the amount of the 1D ordering phase. So the peak height is proportional to the amount of the 1D ordering phase. Figure 7 shows the excess oxygen content dependence of the height of the internal friction peak with $f = 1$ Hz measured in the heat-

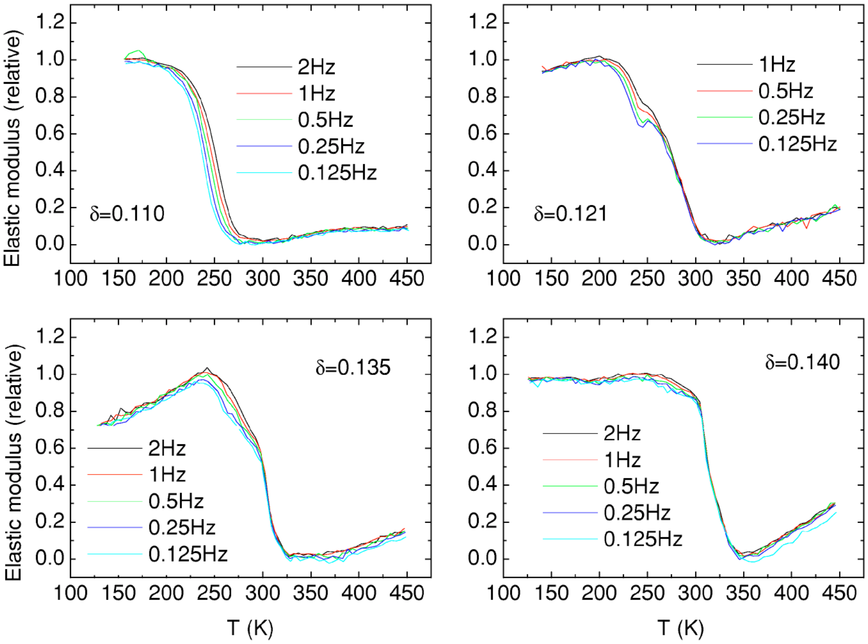

FIG. 6. (Color online) Temperature dependence of the shear modulus with relative units of $La_2NiO_{4+\delta}$ samples with various frequencies in the heating process with a rate of 2 K/min.

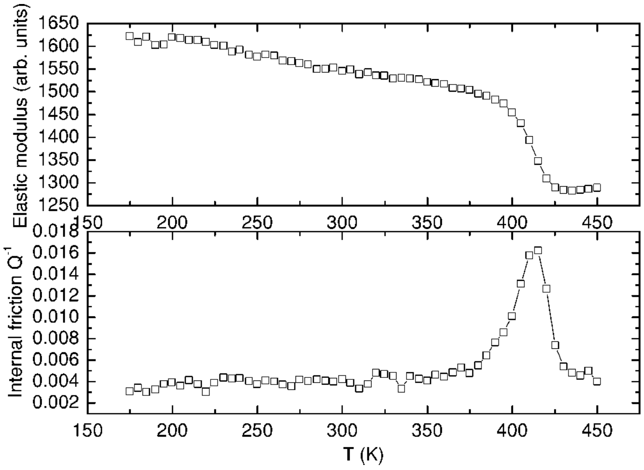

FIG. 8. Temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.147}$ with $f=0.125$ Hz at a heating rate of 2 K/min.

ing process at a rate of 2 K/min for the slowly cooled $La_2NiO_{4+\delta}$ samples in the range of $0.110<\delta<0.145$. The peak height decreases linearly when more excess oxygen atoms are incorporated. This shows that for $0.110<\delta<0.145$ the amount of the 1D ordering phase decreases linearly, and the fraction of the 3D ordering phase increases linearly as a function of the content of excess oxygen. In $La_2NiO_{4.110}$ all excess oxygen is in the 1D ordering state,[4] which is also confirmed by the absence of frequency-independent softening of the shear modulus in the heating process. Then it can be concluded that the change of the ordering of excess oxygen from 1D to 3D is not abrupt but smooth. We extended the fitted line in Fig. 7, which meets with the x axis at the point $\delta=0.145$ where the peak height is zero, which indicates that all excess oxygen is three-dimensionally ordered. Figure 8 shows the temperature dependence of the shear modulus and internal friction of $La_2NiO_{4.147}$ with $f=0.125$ Hz at a heating rate of 2 K/min. The absence of the relaxation peak and the regular modulus below 300 K indicate that excess oxygen is totally three-dimensionally ordered. For $La_2NiO_{4.147}$, there is an internal friction peak near 410 K. In our previous work it is studied in detail.[25] There exists thermal hysteresis of the shear modulus and internal friction in the heating and cooling processes; the decrease of the shear modulus and the peak position are independent of the frequency; and the peak height is proportional to the rate of the heating and reciprocal to the work frequency. All these are the characters of the first-order phase transition of the martensitic type,[26] for which the internal friction is closely related to the transformed volume fraction. All these figures show that a reversible first-order phase transition takes place here.[11,12,25] Considering the results of Tamura *et al.*,[11,12] it is concluded that the phase transition is from the orthorhombic phase (*Fmmm*) to the tetragonal phase ($I4/mmm$) and the $La_2NiO_{4.147}$ sample has orthorhombic *Fmmm* symmetry at low temperature. Our results show that the *Fmmm* phase is correlated to the 3D ordering of excess oxygen. Because in the high-temperature tetragonal phase all excess oxygen is disordered, it seems reasonable to conclude that the phase transition near 410 K from *Fmmm* to $I4/mmm$ is related to the order-to-disorder change of the excess oxygen. The phase transition for $La_2NiO_{4.147}$ is very different compared to the one in range of $0.110<\delta<0.145$, which takes place through the diffusion process.[4] The space group for $0.110<\delta<0.145$ and for $\delta>0.145$ is $I4/mmm$ and *Fmmm*,[11] separately, which also shows that there is some difference between the 3D ordering clusters for $0.110<\delta<0.145$ and the 3D ordering phase for $\delta>0.145$. The 3D ordering of interstitial oxygen for $\delta>0.145$ causes the symmetry change from $I4/mmm$ to *Fmmm*, which shows it is coupled to the lattice, while for $0.110<\delta<0.145$ we suggest that 3D ordering clusters form because of the interaction of the interstitial oxygen atoms through a pattern of tilts of the NiO_6 octahedra.

Comparing the results on the relation of the charge ordering and interstitials mentioned in the introduction[4–9] with our results, it is clear that charge ordering is related to the 3D ordering clusters for $0.110<\delta<0.145$. Tranquada *et al.* proposed a model of a completely ordered arrangement of excess oxygen atoms corresponding to a stoichiometric value of $\delta=2/15$,[10] but it cannot explain our results. From the results reported, in the range of the 1D ordering, $\delta<0.110$, when an arbitrary oxygen content does not correspond to a unique 1D staging, phase separation occurs to two pure 1D staging phase nearby; and the density of interstitial oxygen in the occupied interstitial layers in 1D ordering is about a fixed value.[4] This shows that the state of occupied interstitial layers in 1D staging is a stable low-energy state. Considering that $\delta=0.110$ corresponds to the pure stage-2 ordering with occupied and empty interstitial layers alternated along the c dimensional direction,[4] and in addition the fact that for $0.110<\delta<0.145$ the amount of the 1D ordering phase decreases linearly, we suggest that for $\delta>0.110$, the added interstitial oxygen goes into the empty interstitial layers. Our data also show in the 3D ordering clusters the density of the interstitial inserted to the empty layers is near $1/3\{[(0.145-0.110)/0.110]=0.32\}$ compared to the occupied layers in the 1D staging. The interstitial oxygen inserted to the empty layers pin the interstitial oxygen in the occupied layers through a pattern of tilts of the NiO_6 octahedra and form 3D ordering clusters. This model is very similar to the 1D staging for the different density of interstitial oxygen in different layers, so the forming process of the 3D ordering clusters by the diffusion the process should be very similar to 1D staging. This is confirmed by Tranquada *et al.*[10] Considering of the modulation wave vectors: and observed by Tranquada *et al.*,[10] a model structure is shown in Fig. 9. Note the additional interstitial oxygen has a period of $2c$ in the c direction, but the vector should also be observed because along the c direction. The pattern of the tilts of the NiO_6 is mainly determined by the occupied interstitial layers in our model. The ideal interstitial concentration inserted to the empty layers in our model is 0.033, which is consistent with our data. The interstitial oxygen in the occupied plane is disordered along a direction similarly as in 1D ordering. The ideal interstitial concentration in the occupied layers in our model is 0.100; it should have an additional capacity of 0.010 because of the disordering similarly as stage-2 staging.[4] In this case the interstitial oxygen inserted in the empty layers will produce a fluctuation of the modulation of the lattice potential with a

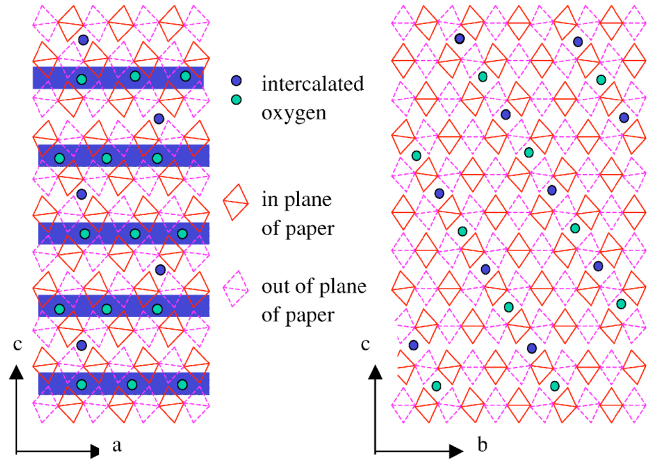

FIG. 9. (Color online) Schematic model of the interstitial oxygen ordering in $La_2NiO_{4+\delta}$. The shadow in the *ac* plane shows the occupied interstitial layers similar as in 1D ordering.

period of *a* along the *a* direction through the tilts of the NiO_6 octahedra, which is consistent with the period calculated from the charge ordering stripe with observed in $La_2NiO_{4.125}$ for $T > 110.5$ K.[8] This picture gives a possible way of the coupling of the stripe ordering and the interstitial ordering. No indication of spin order is observed until below 50 K for $\delta = 0.17$,[27] which is different compared to the phenomena for $0.110 < \delta < 0.145$. This also can be explained by considering the different states of the 3D ordering for them, because for $\delta > 0.145$ in the *Fmmm* phase the interstitial oxygen is totally three-dimensionally ordered with a homogeneous manner.

IV. SUMMARY

We present the results of the internal friction study of a series of $La_2NiO_{4+\delta}$ samples with different excess oxygen content. Different elastic spectra were found in the three ranges:

For $\delta < 0.110$, there is a relaxation internal friction peak due to the hopping of the single interstitials in the *ab* plane, and it is concluded that most of interstitial oxygen in 1D ordering forms clusters in the *ab* plane and does not contribute to the internal friction peak. Glass transition phenomena of the single interstitials were also observed.

For $0.110 < \delta < 0.145$, the existence of a relaxation peak and the frequency-independent softening of the shear modulus shows the coexistence of 1D and 3D ordering of interstitial oxygen for the low-temperature phase; the change of the peak height shows the fraction of the 1D ordering phase decreases linearly as a function of the excess oxygen content. It is concluded that the change of the ordering of the excess oxygen from 1D to 3D with increasing oxygen content is not abrupt but smooth. Glass dynamic phenomena were also observed in this range.

For $\delta > 0.145$, the absence of the relaxation peak shows that all excess oxygen is three-dimensionally ordered and the phase transition observed at 410 K is concluded to be related to the order-to-disorder change of the excess oxygen.

At last a model of the 3D ordering clusters in $0.110 < \delta < 0.145$ is given and the relation of the stripe order and interstitial order is discussed.

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