Evidence of Magnetically Driven Structural Phase Transition in Parent Compounds $RFeAsO$ ($R = \text{La, Sm, Gd, Tb}$): study of low-temperature X-ray diffraction

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We report measurements of structural phase transition of four parent compounds $RFeAsO$ ($R = \text{La, Sm, Gd, and Tb}$) by means of low-temperature X-ray diffraction (XRD). Magnetic transition temperatures associated with Fe ions ($T_{N1}$) are also determined from the temperature dependence of resistivity. As $R$ is changed from La, through Sm and Gd, to Tb, both the $c$-axis and $a$-axis lattice constants decrease significantly. Meanwhile both the structural phase transition temperature ($T_S$) and $T_{N1}$ decrease monotonously. It is also found that the temperature gap between $T_S$ and $T_{N1}$ becomes smaller when the distance between FeAs layer becomes shorter. This result is consistent with magnetically driven structural phase transition and suggests that the dimensionality have an important effect on the AFM ordering.

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I. INTRODUCTION

The recent discovery of superconductivity in layered pnictide-oxide quaternary compounds $ROTMnPn$ ($R = \text{lanthanides, } Tm = \text{Fe, Ni, } Pn = \text{P, As}$) has sparked enormous interest in this class of materials.\textsuperscript{1,2,4,15,6-7,9} Besides this 1111-type layered compounds, superconductivity was subsequently discovered in other iron-based layered compounds with similar FeAs(Se) layers, i.e., 122 systems, 111 systems, and 11 systems.\textsuperscript{11} In all the FeAs-based parent compounds, there is a structural phase transition in the temperature range 100-200 K, and a stripe-type antiferromagnetic (AFM) ordering associated with Fe ions accompanying the structural transition.\textsuperscript{12,13,14} Various chemical doping approaches can suppress the structural transition and AFM order, and high-$T_c$ superconductivity consequently appears. Meanwhile, low-$T_c$ superconductivity has been observed in undoped FeP-based\textsuperscript{4} and NiAs(P)-based\textsuperscript{15} compounds with similar layered structure, but there is neither structural transition nor AFM ordering associated with Fe(Ni) ions in these compounds. This result implies that there is a relationship between structural transition/AFM ordering and high-$T_c$ superconductivity. Theoretically, the origin of the AFM order is still controversial. There are two different physical pictures. One suggests that the AFM order stems from the short range magnetic exchange coupling between local moments.\textsuperscript{13,16} The other suggests that the AFM order stems from band structural calculations.\textsuperscript{12,14} In particular, a theory based on a Heisenberg-type local moment exchange model suggests that the structural transition can be driven by a nematic Ising magnetic order due to the presence of intrinsic magnetic frustration.\textsuperscript{19} The theory\textsuperscript{19} predicts that the difference between the structural and magnetic transition temperatures is controlled by the magnetic coupling between layers: the difference becomes larger when the coupling is weakened. Recent neutron experiment results in 4% Ni-doped BaFe$_2$As$_2$ have supported this prediction.\textsuperscript{20}

In this paper, we report the investigation of structural phase transition detected by means of low-temperature X-ray diffraction (XRD) in the parent compounds LaFeAsO, SmFeAsO, GdFeAsO, and TbFeAsO. The AFM order temperatures associated with Fe ions ($T_{N1}$) and the AFM ordering temperatures ($T_{N2}$) associated with the magnetic rare earths Sm, Gd and Tb are also obtained by measuring magnetic susceptibility and trans-
port properties. A systematic comparison of the structural transition temperature \( T_S \) with \( T_{N1} \) is made. As \( R \) is changed from La, through Sm and Gd, to Tb, both the \( c \)-axis and \( a \)-axis lattice constants decrease significantly. Meanwhile both \( T_R \) and \( T_{N1} \) decrease monotonously. It is also found that the temperature gap \( (T_S - T_{N1}) \) becomes smaller when the distance between FeAs layers becomes shorter. Therefore, our experimental results provide concrete evidence supporting the theory proposed in Ref.12 and suggest the dimensionality may have important effect on the AFM ordering and on the superconductivity mechanism as well.

II. EXPERIMENTAL

The polycrystalline \( R \)FeAsO \((R = \text{La}, \text{Sm}, \text{Gd}, \text{Tb})\) samples were synthesized by solid state reaction in vacuum using powders of \( R \)As, \( R_2O_3 \) (for \( 	ext{TbFeAsO}, \text{Tb}_3O_7 \) was used instead), FeAs and Fe\(_2\)As. \( R \)As was presynthesized by reacting stoichiometric \( R \) pieces and As powders in evacuated quartz tubes at 1223 K for 24 hours. FeAs and Fe\(_2\)As were prepared by reacting stoichiometric Fe powders and As powders at 1023 K for 20 hours. The powders of these intermediate materials were weighed according to the stoichiometric ratio of \( R \)FeAsO respectively, and then thoroughly mixed in an agate mortar. The mixtures were pressed into pellets under a pressure of 4000 kg/cm\(^2\). All the processes were operated in a glove box filled with high-purity argon. The pellets were sealed in evacuated quartz tubes and heated uniformly at 1433-1453 K for 40 hours.

The sample purity was first checked by measurements of powder X-ray diffraction (XRD) at room temperature using a D/Max-rA diffractometer with Cu \( K_\alpha \) radiation and a graphite monochromator. Low temperature X-ray diffraction (LTXRD) measurements for powder specimens were performed using the RINT 2500 system, Rigaku Co. An X-ray beam was generated by a rotating Cu anode. The specimens were cooled by a \( ^{3} \)He gas circulating cryo-cooler and can be cooled down to about 10 K. The temperature stability is better than 0.1% during the LTXRD measurements. At several temperatures entire profiles of reflection peaks were measured with a step size of 0.01° and a step-counting time of 6 s and refined by the Rietveld method using the reported crystal structure. For some reflection planes X-ray diffraction measurements with a step size of 0.005° and a step-counting time of 60 s were performed to accumulate more counts at certain temperatures. From the observed profile the \( d \) value of (220) peak, the integrated intensity \((I.I.)\) and also the full-width-at-half-maximum \((FWHM)\) were obtained. In these analysis the profile was fitted to a Pseudo-Voigt function.

The electrical resistivity was measured by a standard four-terminal method. The samples for transport property measurements were cut into a thin bar. The temperature dependence of d.c. magnetic susceptibility was measured on a Quantum Design magnetic property measurement system (MPMS-5) under magnetic field of 1000 Oe.

III. RESULTS AND DISCUSSION

Fig. 1(a) shows the room-temperature XRD patterns of \( R \)FeAsO \((R = \text{La}, \text{Sm}, \text{Gd}, \text{Tb})\) samples and Fig.1(b) shows the variations of lattice constants \( a \) and \( c \) with the radius of \( R \) ions. For all the four parent compounds, the XRD peaks can be well indexed based on a tetragonal cell with the space group of \( P4/nmm \) (No. 129), which indicates that the samples are in a uniform single phase without obvious trace of impurity phases. As \( R \) is changed from La to Sm, Gd, and then Tb, all the peaks shift to larger \( 2\theta \)'s significantly, implying a remarkable shrinkage of lattice in both \( a \)-axis and \( c \)-axis directions. This result is consistent with the fact that the radius of \( R \) ions decreases gradually as \( R \) goes from the light to heavy rare earth elements. It can also be found from Fig.1(b) that the \( c \)-axis shrinks slightly more quickly than the \( a \)-axis does.

Fig. 2 shows the temperature dependence of resistiv-
ceptibility of genetic rare earth elements other than La, the magnetic susceptibility determined from the measurements of Mössbauer spectroscopy and neutron diffraction. The AFM order temperature determined in our measurements, is $133 \pm 1$ K, consistent with previous reports. For LaFeAsO, GdFeAsO and TbFeAsO, this kind of AFM order associated with Fe ions is measured around $134 \pm 1$ K within the experimental error. For GdFeAsO and TbFeAsO, the peak temperature in the $d\rho/dT$ curve corresponds to the AFM ordering of Fe ions moments as the peak position in the curves of $d\rho/dT$ versus $T$. As shown in the inset, $T_{N1}$ decreases significantly as $R$ is changed from La to Sm, Gd, and Tb. Neutron studies have confirmed that the resistivity anomaly is caused by the structural phase transition and the following formation of antiferromagnetic SDW state. Previous reports have proposed that the peak position in $d\rho/dT$ is associated with the AFM ordering of Fe ions moments rather than the structural phase transition. Indeed, the studies of neutron diffraction reported that the AFM ordering temperature of LaFeAsO is about 135 K, about 20 K lower than the structural phase transition temperature $T_S$ of $158 \pm 1$ K. The $T_{N1}$ value of LaOFeAs determined from the resistivity is 132 K, consistent with the AFM order temperature reported by the neutron diffraction. The AFM order temperature determined from the measurements of Mössbauer spectroscopy and $\mu$SR relaxation is also in agreement with $T_{N1}$, the peak temperature in the $d\rho/dT$ versus $T$ curves. Thus we can regard $T_{N1}$ as the characteristic temperature at which the magnetic moments of Fe ions become AFM ordered. For $R = $ Sm, Gd, and Tb, $T_{N1}$ is 133 K, 128 K, and 122 K, respectively.

In order to obtain more information about the magnetism associated with Fe ions and $R^{3+}$ ions as $R$ is magnetic rare earth elements other than La, the magnetic susceptibility of $R$FeAsO ($R = $ Sm, Gd, Tb) was measured under the magnetic field of 1000 Oe, as shown in Fig.3. From the temperature dependence of magnetic susceptibility shown in Fig. 3(a) and its inset, it can be seen that the GdFeAsO and TbFeAsO have much larger magnetic susceptibility compared to SmFeAsO because Gd$^{3+}$ and Tb$^{3+}$ ions have much larger magnetic moments. At low temperatures, clear phase transitions caused by the AFM ordering of the magnetic moments of $R$ ions can be found at $T_{N2}$. The AFM order temperature of $R$ ions, i.e., $T_{N2}$ determined in our measurements, is $5.56 \pm 0.05$, $4.11 \pm 0.05$, and $2.54 \pm 0.05$ K for SmFeAsO, GdFeAsO, and TbFeAsO, respectively, consistent with previous reports. For SmFeAsO, as shown in the inset of Fig.3(b), a kink associated with the AFM ordering of Fe ions can be observed around $134 \pm 1$ K, consistent with the $T_{N1}$ value of $133 \pm 1$ K within the experimental error. For GdFeAsO and TbFeAsO, this kind of AFM order associated with Fe ions is buried in the large magnetic signals from the $R^{3+}$ ions. The magnetic contributions from the $R^{3+}$ ions obey the Curie-Weiss law very well. As shown in the Fig.3(b), the
inverse of $\chi$ increases strictly linearly with $T$ as $T > T_{N2}$. For the SmFeAsO sample, the magnetic susceptibility does not exhibit the Curie-Weiss behavior because the contribution from the Fe ions which is linearly dependent on temperature is comparable to the contribution from the Sm$^{3+}$ ions. By fitting the Curie-Weiss law, we obtained that the effective magnetic moments $\mu_{eff}$ are 8.0, and 9.7 $\mu_B$/f.u. for GdFeAsO and TbFeAsO respectively, which are consistent with the theoretical values of magnetic moments of free Gd$^{3+}$ and Tb$^{3+}$ ions. If we subtract the Curie-Weiss term which should originate from the contributions of $R^{3+}$ ions, we can also find a slight drop in the subtracted term ($\chi - \chi_{CW}$) around $T$ of 120-140 K for GdFeAsO and TbFeAsO (not shown here), where $\chi_{CW}$ is the Curie-Weiss fitting function. But it is very hard to distinguish whether such a drop in $\chi - \chi_{CW}$ occurs at the AFM order temperature of Fe ions or the structural phase transition temperature. Therefore, we will take $T_{N1}$ as the transition temperature associated with the AFM order of Fe ions.

As mentioned above, the neutron studies have revealed that a structural phase transition occurs just before the AFM ordering in LaFeAsO. It is generally believed that the structure of the parent compounds RFeAsO transforms from tetragonal to orthorhombic when the temperature is lower than the structural transition temperature $T_S$. Such a structure phase transition can be detected by the splitting of (220) peak in the low temperature x-ray diffraction. Fig. 4 shows the temperature dependence of the (220) peak $d$ value for RFeAsO samples. The inset shows the intensity of (220) peak before and after the structural phase transition. It can be seen that the (220) peak splits into two peaks when the temperature is lower than the structural phase transition temperature. From the temperature dependence of (220) peak $d$-value, the structural phase transition temperature $T_S$ can be easily determined as the splitting point of this peak. As $R$ changes from La to Tb, the (220) peak $d$ value decreases significantly, and $T_S$ decreases as well.

We summarize the variations of the structural phase transition temperature ($T_S$) and AFM ordering temperature ($T_{N1}$) in Fig. 5. The structural and physical parameters for these four parent compounds are also listed in Table I. As $R$ is changed from La, through Sm and Gd, to Tb, not only $T_S$ and $T_{N1}$ decrease significantly, but the temperature gap between $T_S$ and $T_{N1}$ also becomes smaller, i.e., the AFM transition occurs at the temperature closer to the structural phase transition temperature. Actually the structural phase transition and AFM ordering happen simultaneously in the more three-dimensional 122 parent compounds like BaFe$_2$As$_2$.

The experimental results can be understood within the theory proposed in Ref. where an effective Heisenberg-type magnetic exchange model, the so called $J_1$-$J_2$-$J_z$ model with $J_1$, $J_2$ and $J_z$ being the in-plane nearest neighbor (NN), in-plane next nearest neighbor (NNN) and out-of-plane magnetic exchange couplings respectively, explains both the structural and magnetic transitions associated with the FeAs layers. In this model, a collinear AFM ground state is obtained when $J_1$ is less than $2J_2$, and a nematic Ising magnetic order transition that breaks lattice rotational symmetry takes place at a temperature equal to or higher than the collinear AFM transition temperature. By including the lattice and spin coupling, the nematic order naturally produces an orthorhombic lattice distortion. Therefore, the model captures both structural and magnetic transitions and suggests the structural transition be driven magnetically. A
quantitative prediction of this model is that the difference between the structural and AFM transition temperature is determined by the ratio between \( J_2 \) and \( J_1 \), i.e., \( J_2/J_1 \). The AFM transition temperature, \( T_{N1} \approx J_2/\log(J_2/J_1) \).

Using the calculated results in Ref. 19 and assuming that \( J_1 \approx J_2 \), we find that the ratio \( J_2/J_1 \) for the four typical parent compounds (La, Sm, Gd, Tb)FeAsO are \((4.10, 15.2, 25.1, 40.0)\) \times 10^{-4}\. The out-of-plane magnetic exchange coupling increases quickly as the lattice constant along c-axis decreases, and therefore the temperature difference between \( T_S \) and \( T_{N1} \) should decrease according to this model. The result is plotted in the inset of Fig.5. The experimental data of \((T_S - T_{N1})/T_{N1}\) are in agreement with the theoretical calculations. By measuring the spin wave gap around the wavevector \((0, \pi, 0)\) as shown in Ref. 20, the out-of-plane magnetic exchange coupling increases quickly with the decreasing c-axis lattice constant and therefore the temperature difference between \( T_S \) and \( T_{N1} \) is significantly influenced.

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| Sample     | \( r(R^{3+}) \) (Å) | \( a \) (Å) | \( c \) (Å) | \( T_S \) (K) | \( T_{N1} \) (K) | \( T_{N2} \) (K) |
|------------|---------------------|-------------|-------------|---------------|----------------|----------------|
| LaFeAsO    | 1.16                | 4.0349      | 8.7366      | 160           | 132            | -              |
| SmFeAsO    | 1.08                | 3.9385      | 8.4941      | 144           | 133            | 5.56           |
| GdFeAsO    | 1.05                | 3.9151      | 8.4660      | 135           | 128            | 4.11           |
| TbFeAsO    | 1.04                | 3.8994      | 8.4029      | 126           | 122            | 2.54           |

### IV. CONCLUSION

In summary, we have studied the structural phase transition of four parent compounds \( R \)FeAsO (\( R = \text{La, Sm, Gd, Tb} \)) by measuring low-temperature X-ray diffractions. As \( R \) is changed from La to Tb, \( T_S \) and \( T_{N1} \) as well as the c axis decrease significantly. Furthermore, the temperature difference between \( T_S \) and \( T_{N1} \) becomes smaller as the c axis becomes shorter. According to the theoretical calculations proposed in Ref. 20, the out-of-plane magnetic exchange coupling increases quickly with the decreasing c-axis lattice constant and therefore the temperature difference between \( T_S \) and \( T_{N1} \) is significantly influenced. The experimental data of \((T_S - T_{N1})/T_{N1}\) are in agreement with the theoretical calculations. This result supports the theoretical proposal that the structural phase transition is driven by a nematic Ising magnetic order due to the presence of intrinsic magnetic frustration, and indicates that the dimensionality could have important effect on the AFM ordering, and the magnetism may play an important role in high-\( T_c \) superconductivity of the iron pnictides.

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