

Effect of pressure on the iron arsenide superconductor Li_xFeAs ($x=0.8, 1.0, 1.1$)S. J. Zhang,¹ X. C. Wang,¹ R. Sammynaiken,² J. S. Tse,³ L. X. Yang,¹ Z. Li,¹ Q. Q. Liu,¹ S. Desgreniers,⁴ Y. Yao,⁵ H. Z. Liu,⁶ and C. Q. Jin^{1,*}¹*Institute of Physics, Chinese Academy of Sciences, Beijing 100190, China*²*Saskatchewan Structural Science Center, University of Saskatchewan, 116 Science Place, Saskatoon, Canada S7N 5E2*³*Department of Physics and Engineering Physics, University of Saskatchewan, 116 Science Place, Saskatoon, Canada S7N 5E2*⁴*Laboratoire de physique des solides denses, Department of Physics, University of Ottawa, Ottawa, Ontario, Canada K1N 6N5*⁵*Steacie Institute for Molecular Sciences, National Research Council of Canada, Ottawa, Ontario, Canada K1A 0R6*⁶*Natural Science Research Center, Harbin Institute of Technology, Harbin 150080, China*

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Resistance measurements of a “111” type superconductor Li_xFeAs ($x=0.8, 1.0, 1.1$) were performed at high pressure. The superconductivity transition temperature (T_c) is found to decrease almost linearly with increasing pressure and the pressure derivative, dT_c/dP , becomes smaller with increasing Li content. Electron spin resonance experiment at ambient pressure on a stoichiometric sample shows gradual decrease in the spin moment and the loss of coherence of spin coupling as the temperature is lowered. A very weak Fe local moment is observed when the temperature is lowered to T_c . *In situ* synchrotron radiation powder x-ray diffraction at high pressure shows changes in the resistivity are not related to a structural transition.

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I. INTRODUCTION

The recent discovery of $\text{LaO}_{1-x}\text{F}_x\text{FeAs}$ (Ref. 1) (“1111” type) with a superconducting transition temperature (T_c) of 26 K has stimulated extensive interest in the search for other iron arsenide superconductor systems. Through chemical substitution, “1111” type compounds with T_c as high as 50 K (Refs. 2–6) have been synthesized. A closely related group of compounds of chemical formula AFe_2As_2 ($\text{A}=\text{Ba}, \text{Sr},$ and Ca , “122” type) with a tetragonal ThCr_2Si_2 structure were later found to be also superconductive by chemical doping. For example, partial substitution of Ba by K in BaFe_2As_2 has led to a superconducting T_c of 38 K.⁷ Similar to the high T_c -layered cuprate superconductors, the iron arsenide systems cannot be explained by the conventional BCS theory. It is now generally believed that the superconductivity is related to the spin in the system. Very recently, a superconducting LiFeAs with T_c of 18 K (Ref. 8) was found by one of the authors. This compound crystallizes in a tetragonal Cu_2Sb -type structure⁹ and is composed of Fe_2As_2 layers linked by edge-sharing FeAs_4 tetrahedra (Fig. 1). This superconductor system is named “111” type following the convention of the “1111” or “122” types mentioned above. Almost simultaneously, several groups have also investigated and reported the electronic structure and superconductivity of LiFeAs .^{10–12}

Chemical substitution has been used to tune T_c by introducing charge carriers or to alter the electronic structure in ReFeAsO ($\text{Re}=\text{rare earth metals}$) and AFe_2As_2 . Besides chemical substitution, pressure is another effective way to modify T_c by changing the carrier concentration and exchange interaction through the compressed lattice. Soon after the discovery of superconductivity in LaFeAsO ,¹ enhancement of T_c from 26 to 43 K was achieved in LaOFeAs at 4 GPa.¹³ In the past, high-pressure studies have provided unequivocal evidence showing that the superconductivity in cuprate superconductors is primarily related to electron pairing

in the Cu-O plane. If this was the case in iron arsenide systems, it is expected that the superconducting transition temperature will also be more sensitive to Fe-Fe distances in the FeAs layers than between the layers. Therefore measurement of the electrical properties under pressure should offer insight to understand the superconducting mechanism of the iron arsenide systems. Pressure-tuned superconductivity of

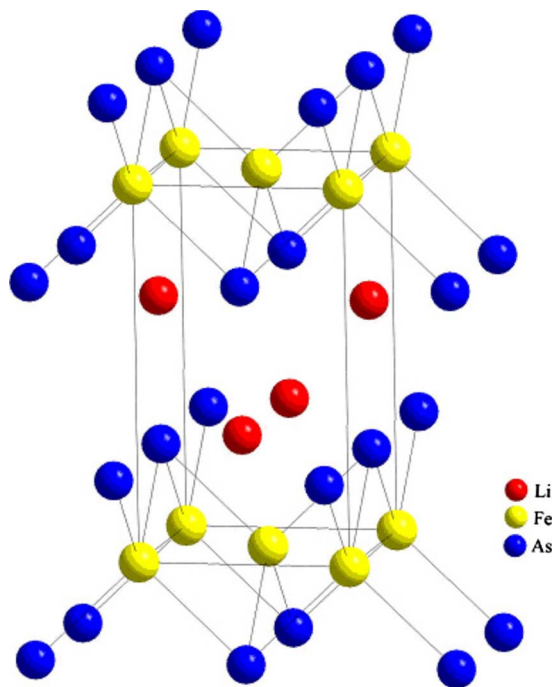


FIG. 1. (Color online) The crystal structure of LiFeAs with space group $P4/nmm$ that is featured by alternative (FeAs) layer being interlaced with Li similar to that of Cu_2Sb . The lattice parameters are $a=3.77 \text{ \AA}$ and $c=6.36 \text{ \AA}$. In this figure, the red (gray) spheres are Li atoms, the blue (dark gray) spheres are As atoms, and the yellow (light gray) spheres are Fe atoms.

several “1111” type and “122” type compounds have already been reported in the literature.^{6,13,15–22}

LiFeAs has some notable features different from “1111” type or “122” type compounds. While all undoped “1111” and “122” type compounds exhibit spin-density wave (SDW) transition,^{14,23–26} it is absent in LiFeAs.^{8,10–12} Although the superconductivity transition temperature in LiFeAs is comparatively low, the structural simplicity of this compound makes it a convenient model to study the superconducting mechanism both by theory and experiment. The absence of SDW state in LiFeAs suggested that the superconducting behavior may be associated with a marginally ordered magnetic state. The objectives of this investigation are (i) to study the effect of pressure on the electrical conductivity and T_c ; (ii) to explore whether pressure can alternate the magnetic and crystal structure, and (iii) to characterize the spin (magnetic) state. For these purposes, *in situ* resistance measurements and synchrotron radiation x-ray powder diffraction were performed using the diamond anvil cell (DAC) technique. In addition, variable temperature electron spin resonance measurements at ambient pressure were performed. Resistance measurements were performed on three samples Li_xFeAs with $x=0.8, 1.0, 1.1$. Stoichiometric LiFeAs ($x=1.0$) samples were used in the ESR and diffraction experiments. The ESR spectra of potential impurities FeAs and Fe_2As_2 were also measured. The effect of pressure on the superconductivity transition temperature and possible changes in the electronic structure driven by pressure will be discussed.

II. EXPERIMENTAL DETAILS

“111” type LiFeAs was prepared from mixing appropriate ratio of starting materials Li (99.9%) and FeAs following the procedure reported in Ref. 8. FeAs was prepared in advance. Li_xFeAs samples with nominal $x=0.8, 1.0, 1.1$ were obtained.

Variable temperature electron paramagnetic resonance (EPR) experiments were conducted on stoichiometric LiFeAs, Fe_2As_2 , and FeAs. The measurements were performed with a Bruker EMX X-band spectrometer fitted with an Oxford liquid-helium cryostat. The operating conditions were optimized at microwave frequency of 9.35 GHz, power of 2 mW, modulation of 2 G, and liquid-helium flow rate of 1 l/h. The samples were kept under argon and sealed in an EPR tube with a lock tight valve.

Superconducting transitions were investigated under high pressure. The resistance was measured by four-probe electrical conductivity methods in a diamond anvil cell made of CuBe alloy. Pressure was generated by a pair of diamonds with 600- μm -diameter culet. A sample hole of 300 μm diameter was drilled in the gasket after its thickness was reduced from 250 to ~ 30 μm by preindentation. Then it was covered with a thin cubic boron nitride (BN) layer for electrical insulation between the gasket and the electrodes. Gold wire of 18 μm diameter was used as electrode leads. The sizes of all the three samples were about $200 \times 100 \times 20$ μm^3 . The pressure was determined by a standard ruby-fluorescence method at room temperature before and after each cooling down. For the $x=1.0$ sample, pressure was ap-

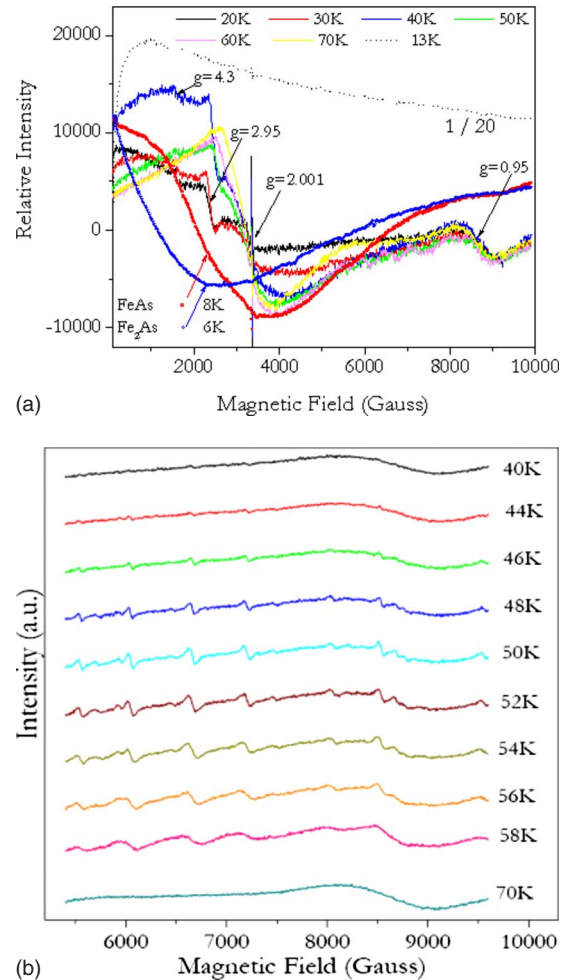


FIG. 2. (Color online) ESR measurements of LiFeAs (a) Full. (b) The high-field region as the temperature is lowered from 70 to 42 K.

plied up to 18 GPa. The results of the other two samples ($x=0.8, 1.1$) were almost identical to that of $x=1.0$. For the $x=1.1$ sample, pressure was applied above 30 GPa while for the $x=0.8$ sample, pressure was applied up to 38 GPa.

To study the effect of pressure on the structure of LiFeAs, angle dispersive x-ray diffraction (XRD) were performed at the HXMA beamline of the Canadian Light Source.²⁷ In the XRD experiments, a stoichiometric Li_xFeAs ($x=1.0$) sample was loaded in gasketed diamond anvil cells without pressure-transmitting medium since LiFeAs itself is relatively “soft” as compared with copper oxide superconductors and the compound is very hygroscopic sensitive precluding the use of silicon oil. The wavelength used in the experiment was 0.5091 Å.

III. RESULTS AND DISCUSSIONS

Figure 2(a) shows a comparison of the full range EPR spectra of LiFeAs from 70 to 13 K. The broad features of a paramagnetic Fe system at $g=2.95, 2.001,$ and 0.95 become prominent at 70 K. As the temperature is lowered, the relaxation time lengthens and the main bands become more

prominent. In the intermediate region between 50 and 70 K, distinct features due to spin coupling are evident. However these patterns become very weak as the temperature is lowered below 50 K. At 30 K an additional high-spin band at $g=4.3$ is observed. The overall intensity of Fe spin decreases as the temperature is lowered accompanying with an overall decrease in the paramagnetism. A large zero-field absorption is observed as the LiFeAs becomes superconducting when the temperature is below 18 K. Figure 2(b) shows the highly resolved features of a transitional state in the high-field region as the temperature is lowered from 70 to 42 K. As the temperature is increased from 42 to 70 K the bands reappear with increasing intensity to approximately 52 K and then broaden into the large high-temperature background at 60 K. The disappearance of these high-field bands as the temperature drops below 42 K is unexpected since bands will normally sharpen as the temperature is decreased. This is the result of decrease in spin-spin and spin-orbit interactions and an overall decrease in the paramagnetism. When the temperature is decreased, spin-orbit interactions are observed resulting in bands sharpening. Approaching the superconductivity temperature resulted in a reduction in unpaired spin density at the Fe sites and a reduction in the overall paramagnetism. In Fig. 2(a), the EPR spectra of Fe_2As and FeAs, at the lowest achievable temperature, at 8 and 6 K, respectively, are overlaid. These spectra represent the signal obtained over the temperature range and clearly show that a large zero-field absorption at temperatures below 13 K is not present. This indicates that the presence of Fe_2As and FeAs as contaminants could only contribute to the overall background of the EPR spectra and did not contribute to the absorption structures observed in LiFeAs. A very small magnetic moment in LiFeAs is consistent with the observation made in other iron arsenide superconductors.

Figure 3(a) shows the measured resistance of the $x=1.0$ sample as a function of temperature at different pressures. Different from the “1111” type $R\text{FeAsO}$ or “122” type AFe_2As_2 superconductors,^{23–26} no clear evidence of a SDW phase transition characterized by a rapid decrease in resistance was found in LiFeAs. LiFeAs becomes superconductive at ambient pressure at 18 K. Figure 3(b) shows the superconducting transition is suppressed by increasing pressure. As shown in Fig. 4, for the $x=1.0$ sample, the superconductivity critical temperature (T_c) is found to decrease linearly with increasing pressure with $dT_c/dP = -1.38$ K/GPa. The Li_xFeAs samples with $x=0.8$ and 1.1 exhibit the same trend with $dT_c/dP = -1.48$ K/GPa and -1.22 K/GPa, respectively. For the $x=0.8$ sample, the superconductivity transition reappears at low pressure once unloaded from 11, 20, and 38 GPa.

The lithium content seems to have little effect on the superconductivity transition temperature in Li_xFeAs at ambient pressure. This observation supports a first-principles calculation showing there is little change in density of states (DOS) at Fermi surface with the Li content.²⁸ Surprisingly the T_c seems to depend on the Li content under pressure. From the experimental data, it is found that dT_c/dP becomes smaller with increasing Li content, changing from -1.48 K/GPa in $\text{Li}_{0.8}\text{FeAs}$ to -1.38 K/GPa in $\text{Li}_{1.0}\text{FeAs}$ and then -1.22 K/GPa in $\text{Li}_{1.1}\text{FeAs}$. The high Li mobility of Li in the

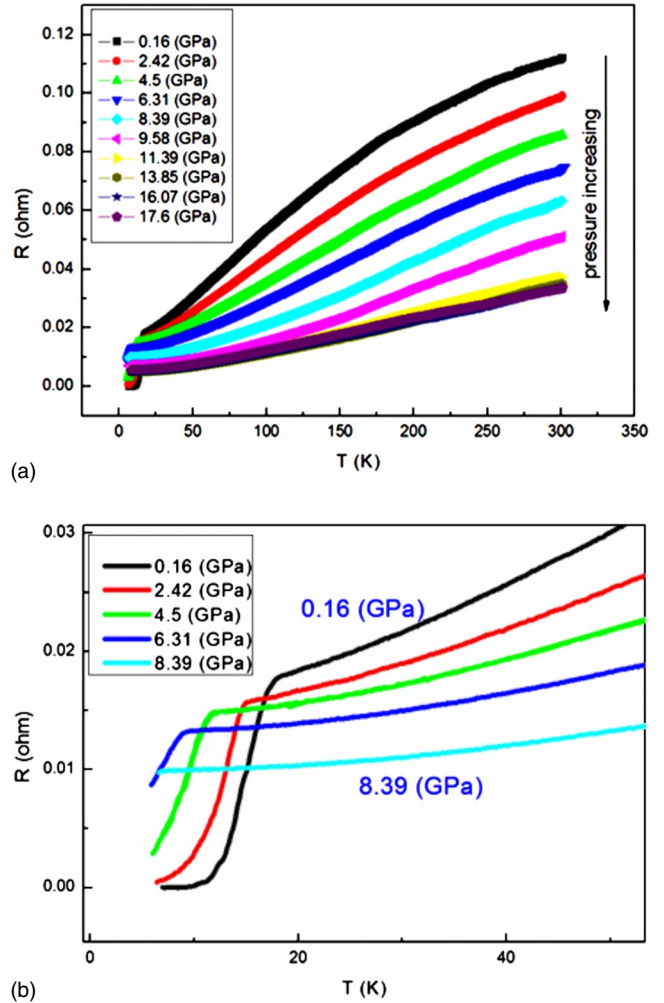


FIG. 3. (Color online) (a) The temperature dependence of resistance for Li_xFeAs ($x=1.0$) compound at variant pressures, (b) the selective region showing the superconductivity evolution with pressure from near ambient to 8 GPa.

solid may facilitate the formation of vacancies at the Li sites. Increasing nominal Li content reduces the Li vacancies in Li_xFeAs . Therefore the reduced dT_c/dP with increasing Li content shown in Fig. 4 indicates that the pressure response to the Li vacancies is related to T_c change. The pressure effect on T_c for $x=1.0$ sample is compared with other iron arsenide superconductors in Fig. 5. Two general behaviors of pressure on the T_c were observed in these iron arsenide superconductors. First, the T_c is enhanced by initial compression. At higher pressures the T_c will then decrease. This behavior is observed in $\text{LaFeAsO}_{1-x}\text{F}_x$ (Refs. 13 and 16) and AFe_2As_2 ($A=\text{Ca}, \text{Sr}, \text{Ba}$).¹⁷ The second is that the T_c is suppressed by pressure. This is observed in LiFeAs and in $R\text{FeAsO}$, where R is a rare-earth element with smaller ion radius.^{6,15}

Both the intralayer and interlayer Fe-Fe distances are found to affect the superconducting transition temperature in the iron arsenide superconductors. Cooper pairings only exist within a thin shell at the Fermi level. In the iron arsenide compounds the density of states at Fermi surface is dominated by the Fe 3d orbitals. Therefore it is reasonable to

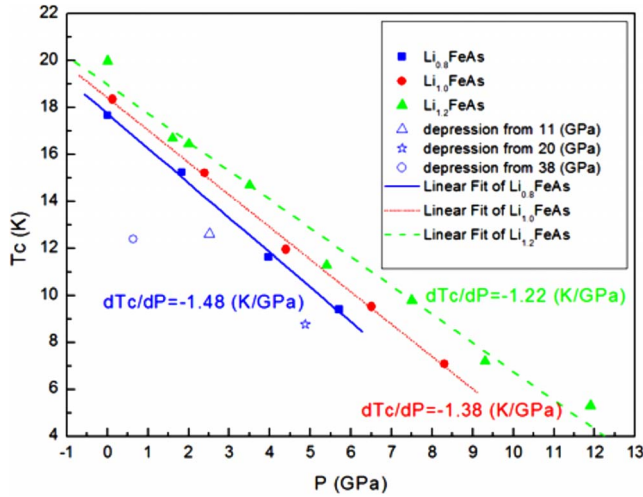


FIG. 4. (Color online) The T_c - P phase diagram of Li_xFeAs obtained from resistance measurements with slope $dT_c/dP = -1.48$ K/GPa, -1.38 K/GPa, -1.22 K/GPa for $x=0.8, 1.0, 1.1$, respectively. Points are experimental data while the lines are linear fit to the experimental data. The symbol Δ , \star , and \circ represent experimental data during decompression from 11, 20, and 38 GPa to ambient pressure, respectively, for $x=0.8$ sample.

expect superconductivity in these compounds is sensitive to the lattice geometry. Following the cuprate superconductors, it is reasonable to assume the in-plane Fe-Fe distance is more important in affecting the T_c in iron arsenide superconductors. From experimental observations, it is apparent that there may be an optimal in-plane Fe-Fe distance, d_{opt} , where T_c is maximized. For superconductors when the zero pressure in-plane Fe-Fe distance is larger than d_{opt} , T_c will be enhanced under pressure. This effect was manifested in LaFeAsO where a large La ion radius results in larger ambient lattice parameters and T_c was found to increase on compression. In contrast, in RFeAsO , where R is a rare-earth element with smaller ion radius than La, the Fe-Fe distance is smaller than

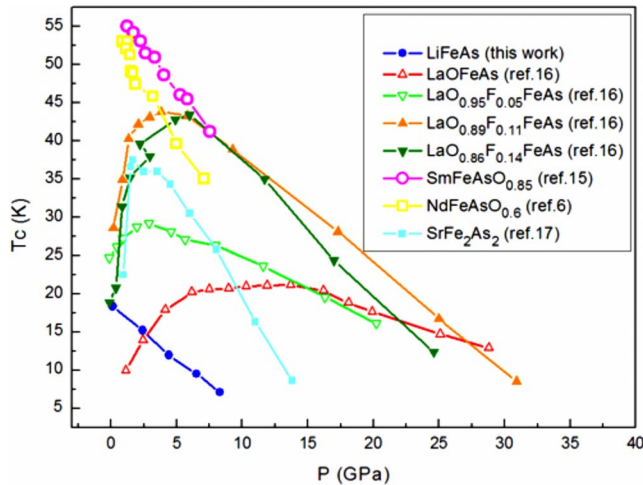


FIG. 5. (Color online) The comparison of T_c - P phase diagram among iron arsenide superconductors. All points are experimental data with reference indicated.

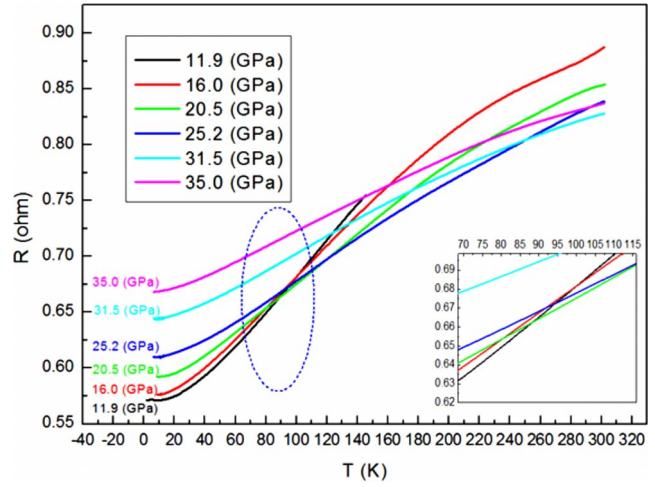


FIG. 6. (Color online) The temperature dependence of resistance for Li_xFeAs ($x=1.1$) compound from 12 to 35 GPa. Crossover of R - T curves is highlighted in the inset.

the d_{opt} and resulted in the suppression of the T_c on initial compression. Owing to the very small lithium ionic radius, the in-plane Fe-Fe distance in LiFeAs is the shortest among the iron arsenide compounds. Therefore, comparing to other iron arsenide compounds, LiFeAs has already been “precompressed” by chemical pressure. This may be the reason that pressure-induced increase in resistance at higher pressure for “111” LiFeAs was not observed in “1111” or “122”.

There are two unusual features in the R - T relationship for the Li_xFeAs sample with different Li concentration. Usually, the resistivity should decrease with increasing pressure. Furthermore, the slope dR/dT of LiFeAs with increasing pressure is expected to be parallel to each other. This is not the case in Li_xFeAs . As shown in Fig. 6, the R vs T curves are very sensitive to the pressures. First of all, at low temperature (<120 K), the rate of change in the resistance vs temperature $(dR/dT)_P$ at low pressure (e.g., 11 GPa) is much larger than at high pressure (e.g., 35 GPa). A second unusual feature is that the resistance of the sample at higher temperature increases at higher pressure. For example, the sample measured at 31.5 GPa has a lower resistance than that measured at 35 GPa at room temperature. A similar behavior is also observed in the stoichiometric in $\text{Li}_{1.0}\text{FeAs}$ sample. As shown in Fig. 7, the resistance decreases much faster with increasing pressure at high temperature (290 K) than at low temperature (30 K) and the resistance does not continue to decrease when the pressure is above 12 GPa for both curves.

Usually, the resistance and pressure are anticorrelated, that is, the higher the pressure is, the lower the resistivity will be. This is not the case here. The resistivity of the LiFeAs sample at 11 GPa below 120 K is lower than that of the sample measured at 16 GPa. This unusual observation may be related to local spin fluctuations. It has been suggested that the ordering state of strip-type antiferromagnetic phase is very sensitive to the in-plane Fe-Fe distance.²⁹ Reduction in the iron distance will improve the itinerant electronic characteristics that tend to suppress the long-range magnetic order. The in-plane Fe-Fe distance in LiFeAs is likely in the region competitive with the long-distance mag-

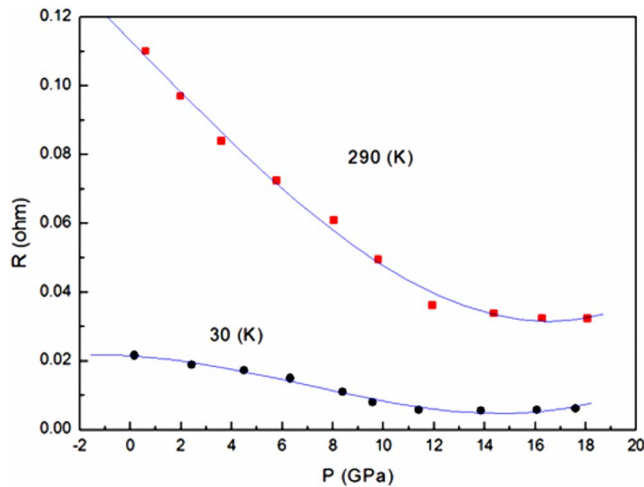


FIG. 7. (Color online) The R - P curve of Li_xFeAs ($x=1.0$) at variant temperatures. The solid lines are guide to the eyes.

netic order that can easily be suppressed by Li vacancies leading to a local magnetic order. Pressure increases the local magnetic order by compressing the in-plane Fe-Fe distance giving rise to a higher resistance. This result provides indirect evidence that magnetic interactions remain effect on LiFeAs .

To study the effect of pressure on the structure, synchrotron powder XRD experiments were performed on a sample with nominal stoichiometric LiFeAs . XRD patterns at selected pressures were plotted in Fig. 8. These diffraction patterns can be indexed to the known $P4/nmm$ structure as shown in 0.6 GPa case, except some minor diffraction peaks from an impurity. For example, the diffraction peak located between (110) and (111) of LiFeAs came from (111) of FeAs. The peak splitting at around 15° is due to shift of the (110) peak of the stainless-steel gasket surrounding the sample. No structural change is observed for pressure up to 20 GPa. So the diffraction results clearly show that the suppression of superconductivity and the unusual resistance change with pressure are not related to a structural phase transition.

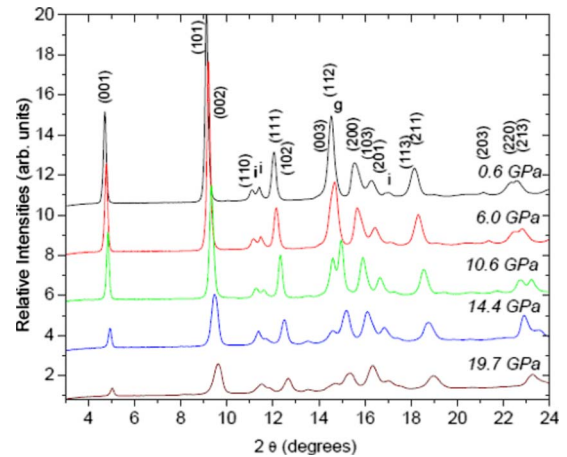


FIG. 8. (Color online) The selected XRD patterns of LiFeAs up to 19.7 GPa. Reflections labeled with “i” and “g,” respectively, correspond to an impurity phase (FeAs) and stainless steel (T301) used as gasket material. The wavelength of incident radiation is 0.5091 \AA .

In summary, ESR experiments suggest that LiFeAs exists weak local moment. Pressure is found to suppress superconductivity for Li_xFeAs superconductors, where higher Li content tends to release the effect. The turning of $R \sim P$ curve at higher pressure suggests the magnetic interactions remain effective in “111” type LiFeAs superconductor. Both the suppression of superconductivity and the abnormal resistance change are not related to the occurrence of a structural phase transition. It is noteworthy that there are two low-pressure measurements reported recently supporting the present observation on the suppression of the superconductivity transition temperature in LiFeAs .^{30,31}

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