Construction of ultrasonic measurement and strain measurement system under high pressure and research on spin/orbit order

Keisuke Matsuura, Kohei Matsuura
Department of Advanced Materials Science,The University of Tokyo

Authors

Keisuke Matsuura : Specialized field is measurement of physical properties of strongly correlated electronic materials. In particular, he has targeted magnetic insulators with orbital degeneracy. In this study, he was mainly working for the part related to ultrasonic measurement.

Kohei Matsuura : Specialized field is measurement of properties of superconductivity. In particular, he has been working for the measurement of physical properties in superconductivity under high pressure. In this study, he was trying to optimize the cubic anvil high pressure apparatus for ultrasonic measurement and strain measurement.

abstract

In the research field of strongly correlated electron systems which show various physical properties, the electron phases such as superconductivity and magnetism have been studied. The measurements of physical properties under external control have been thought to be important to clarify the relationship between such electronic phases. The measurement of various physical properties under pressure in various material systems has been done because pressure is thought to be one of the effective control parameters. To apply high pressure to the target materials, various pressure cells have been developed. In the physical property measurements under high pressure, there are pressure medium and a pressure cell around the sample. Since the sample space is very small, not only is it difficult to measure the temperature and pressure in the sample space, but also the measurable physical properties are limited compared with the ambient pressure condition. Recently, in the study of strongly correlated electron systems, the information on the crystal structure has become important more and more. However, there are not so many reports which show the measurement of such properties under high pressure by the low-cost method. For this reason, in this study, we investigated the possibility of ultrasonic measurement and measurement of physical properties by using piezoelectric film to develop the measurement method under high pressure.

1 Background

1.1 Cubic Anvil Apparatus

In the high-pressure study, the following two points are required from the technical point of view. First, to investigate the changes in physical properties by the pressurization, it is necessary to get highly accurate results which is comparable to the measurement result under ambient pressure. Secondly, as a variable for

controlling physical properties, it is necessary to extend the upper limit of the pressure to control the electron phase with a high energy scale. From the above first requirement, piston cylinder cells have been often used in low temperature experiments. The feature of the piston cylinder cell is that the volume of the sample space is larger than that of other pressure cells. The sample size can be up to several mm, and various physical properties have been measured so far. Ultrasonic measurements have also been attempted. However, the upper limit of the generated pressure is about 3 GPa. From the second requirement, diamond anvil cells have been used in the region of ultrahigh pressure. The diamond anvil cell is a pressure device making use of the hardness of diamond. This device is compact enough to be placed on the palm of the hand and can generate the highest pressure. Moreover, the light and X rays passes through the diamond used for the anvil. Therefore, in the material science it is often used for optical experiments and non-contact measurements. However, since the sample size is about several ten μm, high techniques are required to perform contact measurements. As mentioned above, in the measurements of physical properties under high pressure, we use different pressure cells depending on the required pressure value and measurement techniques. In measuring the physical properties under pressure and determining the electronic phase diagram, it is necessary to be able to measure multiple physical properties with one pressure device. Therefore, the expansion of measurement techniques of every pressure cells is important.

In this study, a constant load cubic anvil high pressure apparatus was used[1, 2]. It is a device which can generate hydrostatic high pressure by isotropically pressurizing six surfaces of a cubic gasket. As shown in fig.1(a) and (b), a sample is sealed in a Teflon cell together with a pressure medium and the cell is attached to a cubic gasket made of pyro ferrite that is pressurazied by six anvils. The pressurization by the plunger pump from the vertical direction is distributed and the gasket is pressurized from six directions(1(c)). The anvils in the four directions except upper and lower anvils contact with the four terminals from the sample, and the anvil itself plays the role of the electrodes. In addition, Teflon sheet is bonded with the anvil to avoids the electrical contact between the four anvils and the stage and to reduce friction between them. The most significant feature is that this device have big sample space compaled with other pressure cells that realizes this pressure region. We can stably measure electric conductivity , AC magnetic susceptibility and specific heat with this device. Therefore, it is possible to measure physical bulk properties under high pressure same as under ambient pressure.

1.2 Measurement of elastic constant in solid by using ultrasound

Ultrasonic measurement can detect small change in lattice state caused by fluctuation of electron orbital and spin. So far, ultrasonic measurements have been applied to a variety of materials such as the quantum spin system, superconducting materials, and skyrmion system. In particular, in the orbital degenerate system, the induced strain in crystal by ultrasound couples with the electric quadruple (electronic orbital). We can measure the orbital susceptibility corresponding to the magnetic susceptibility in magnetic materials. Since high-frequency signals are used, ultrasonic measurements are less affected by noise signal compared to magnetization measurements in pulsed magnetic fields. Ultrasound measurement is one of the powerful tools under extreme environments. In the following, the sound velocity measurement by the pulse echo method will be described.

Figure 2 shows the schematic illustration of the pulse-echo method. In pulse-echo method, electric pulse signal excites the elastic wave via transducer and this elastic wave propagates and reflects in sample. The elastic wave arriving at opposite-side transducer is again converted into the electric signal. Then, the output electric signal is detected as the pulse signal with regular interval. The series of the pulse signal are called 0th, 1st, 2nd, 3rd pulse, etc., depending on the arrival time.

In the following, we explain how to measure the elastic constant. Here, we adopted the phase comparison

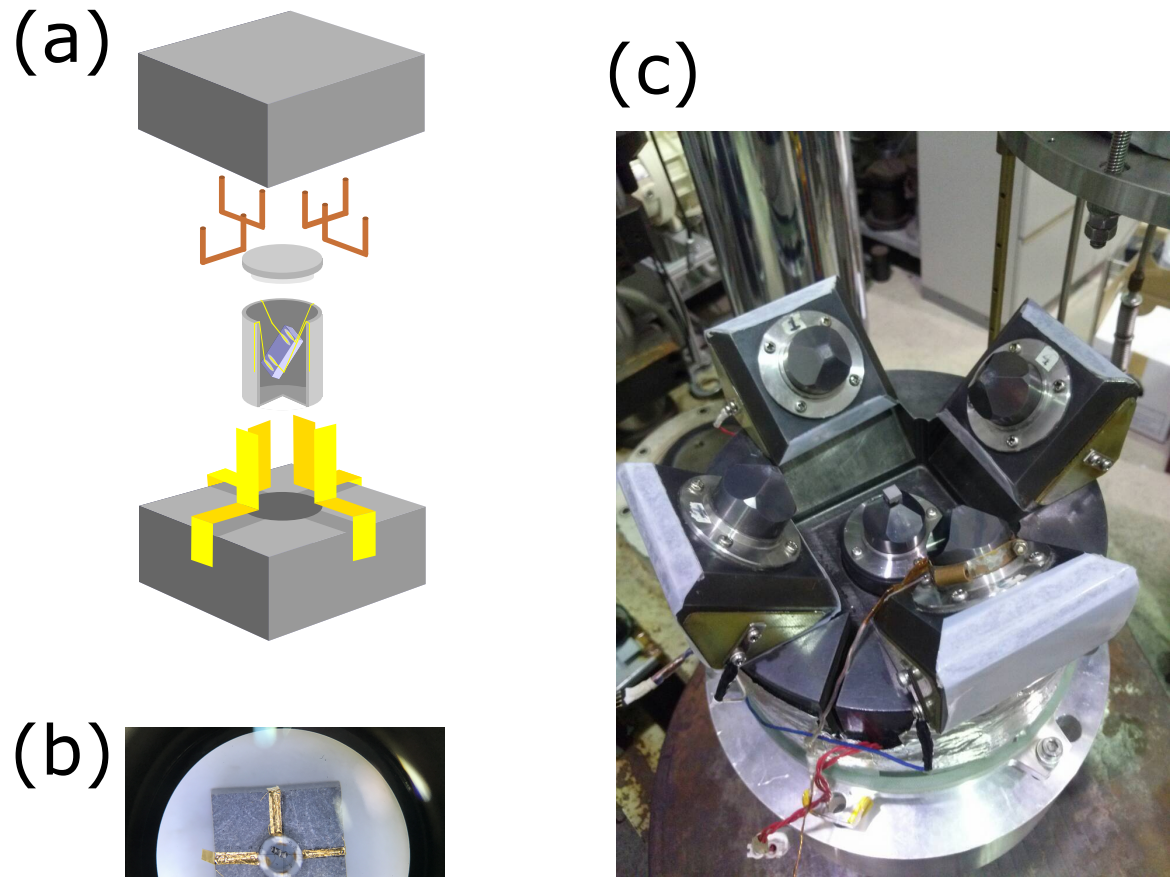

Figure 1　cubic anvil high pressure apparatus.(a)The set-up of the pressurazied space. (b)The photo of the pressurazied space.(c)anvils which pressurize the sample and play the electrodes

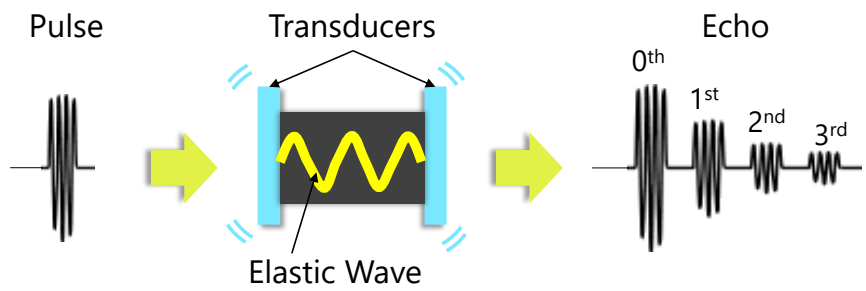

Figure 2　Schematic illustration of the pulse-echo method.

method for measuring the elastic constant [3].

In the phase comparison method, the phase of n-th echo is shifted from the reference signal by ϕ_n. ϕ_n is given by

$$\phi_n = 2\pi(2n+1)fT_0 = 2\pi(2n+1)f\frac{L}{v}, \tag{1}$$

where L, f, and v are the sample length, the frequency of reference signal, the velocity. In eq. (1), the total differential of the phase $d\phi_n$ divided by ϕ_n leads to the following equation.

$$\frac{d\phi_n}{\phi_n} = \frac{df}{f} + \frac{dL}{L} - \frac{dv}{v}. \tag{2}$$

In usual case, we can ignore the change of the sample length ($\Delta L = 0$). If the frequency is fixed ($df = 0$), the relative change of the velocity $\Delta v/v$ is given by

$$\frac{dv}{v} = -\frac{d\phi_n}{\phi_n} \quad (\because df = 0). \tag{3}$$

Therefore, the relative change of the velocity is detected as that of the phase.

Figure 3 is the blockdiagram of the ultrasound measurement system. High-frequency continuous signal (10-200 MHz) from a signal generator is divided into two signals for sample and for reference signal by a signal divider. The signal for sample can pass through when the pulse signal from the pulse generator enters the diode switch (R&K Co.、 Ltd.、 SW series). Therefore, the signal for sample can be chopped at regular intervals by using pulse generator and diode switch.

The chopped high-frequency signal excites the ultrasound transducer, which generated an ultrasound wave into the sample by inverse piezoelectric effect.

Ultrasound wave propagates through the sample and continues to reflect in sample. When the ultrasound waves reached the opposite end of the sample, they were again converted into the electric signals by piezoelectric effect.

On the other hand, quadrature hybrid (R&K Co.、 Ltd.、 QH series) converted the reference signal into two signals shifted by $0°(V_{\rm ref}^{\cos} = |V_{\rm ref}|\cos(\omega t))$ and by $90°(V_{\rm ref}^{\sin} = |V_{\rm ref}|\sin(\omega t))$.

Afterwards, the sample signal and the reference signal are multiplied in the double balanced mixer (R&K Co.、Ltd.、MX series、Frequency band: 0.1-1000 MHz). In the case of nth echo signal ($V_{echo}^{(n)} = |V_{echo}^{(n)}|\cos(\omega t + \phi_n)$), the sin- and cos- parts of these multiplied signals are given by

$$\begin{aligned} V_{\rm ref}^{(\cos)} \cdot V_{\rm echo}^{(n)} &= |V_{\rm ref}|\cos(\omega t) \cdot \left[|V_{\rm echo}^{(n)}|\cos(\omega t)\exp\left(-(2n+1)\alpha L\right) \cdot \cos(\omega t + \phi_n)\right] \\ &= |V_{\rm ref}| \cdot |V_{\rm echo}^{(n)}|\exp\left(-(2n+1)\alpha L\right)\frac{1}{2}\left(\cos(2\omega t + \phi_n) + \cos(\phi_n)\right), \end{aligned} \tag{4}$$

and

$$\begin{aligned} V_{\rm ref}^{(\sin)} \cdot V_{\rm echo}^{(n)} &= |V_{\rm ref}|\sin(\omega t) \cdot \left[|V_{\rm echo}^{(n)}|\cos(\omega t)\exp\left(-(2n+1)\alpha L\right) \cdot \cos(\omega t + \phi_n)\right] \\ &= |V_{\rm ref}| \cdot |V_{\rm echo}^{(n)}|\exp\left(-(2n+1)\alpha L\right)\frac{1}{2}\left(\sin(2\omega t + \phi_n) + \sin(\phi_n)\right), \end{aligned} \tag{5}$$

respectively. High-frequency components ($\geq 2\omega$) in multiplied signals are removed in the low-pass filter (R&K Co.、 Ltd.、 LP series、 DC-1MHz or 10MHz) and only DC signals ($\cos\phi_n$ and $\sin\phi_n$) passed through. These DC signals at n-th echo are measured by the oscilloscope.

The phase of n-th echo ϕ_n and amplitude A_n are calculated as

$$\hat{\phi_n} = \arctan\left(\frac{V_{\sin}^{(n)}}{V_{\cos}^{(n)}}\right), \hat{A_n} = \sqrt{\left(V_{\cos}^{(n)}\right)^2 + \left(V_{\sin}^{(n)}\right)^2}. \tag{6}$$

Using these values, the relative change of the ultrasound velocity is

$$\frac{dv}{v} = -\frac{d\phi_n}{\phi_n} = -\frac{\hat{\phi_n}(x) - \hat{\phi_n}(x_0)}{\phi_n}, \tag{7}$$

and the ultrasound attenuation is

$$\frac{d\alpha}{\alpha} = \frac{-20\log_{10}\left(\dfrac{\hat{A}_n(x)}{\hat{A}_n(x_0)}\right)}{(2n+1)L}. \tag{8}$$

Thus, the voltage signals of cos and sin components give the phase ϕ_n and amplitude A_n.

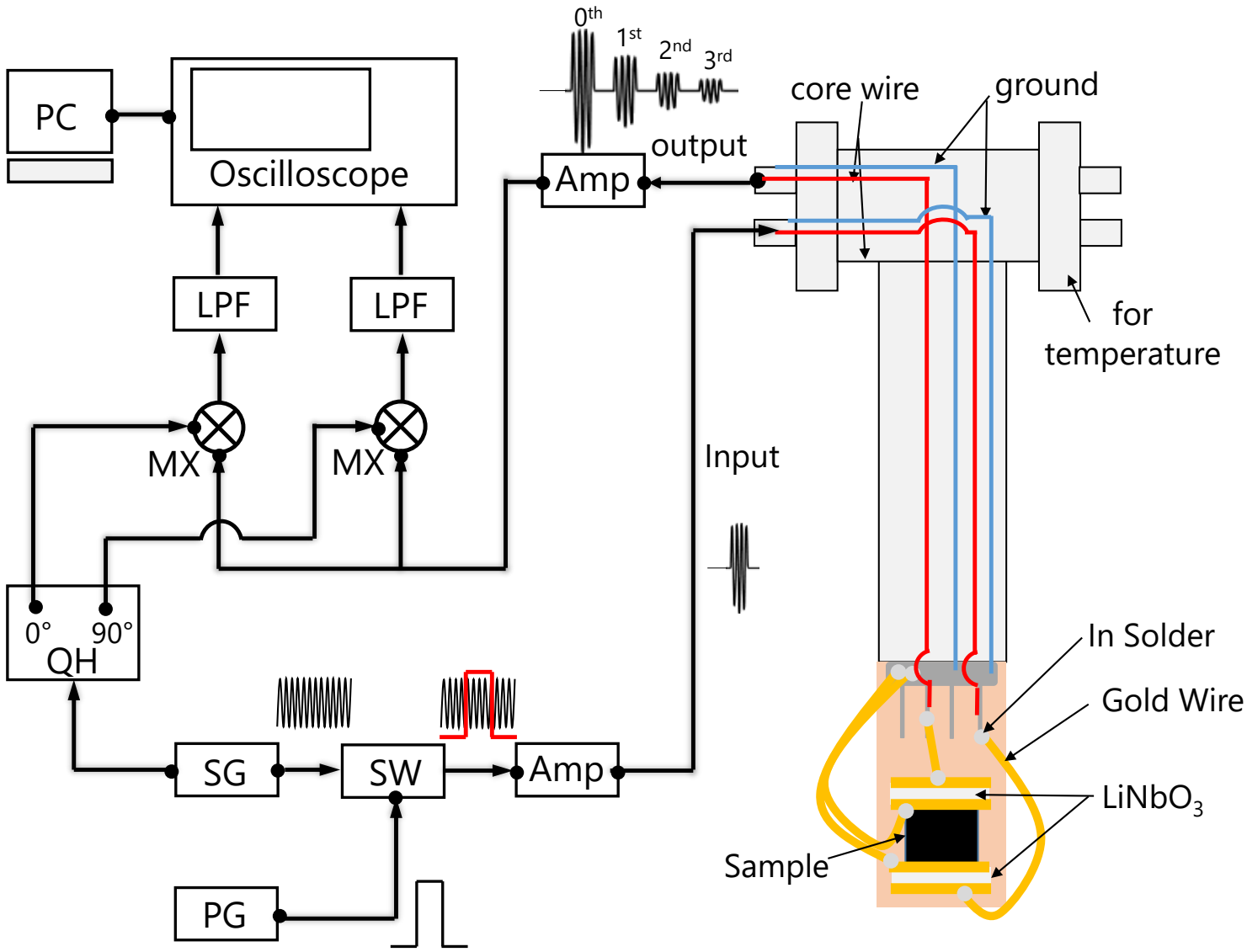

Figure 3 Blockdiagram of the ultrasound measurement system. Abbreviations are as follows: PG (pulse generator), SG (signal generator), Amp (signal amplifier), QH (quadrature hybrid), Mixer (double balanced mixer), SW (diode switch), LPF (low-pass filter), and Oscilloscope.

2 Objective of this study

Electrical resistance measurements and AC magnetic susceptibility measurements have been mainly performed for low temperature cubic-anvil-type high pressure equipment. Combination of the cubic-anvil-type high pressure equipment with ultrasonic measurements, however, have not yet been established. The accomplishment of this research will pave a way to the development of physical property measurement under multiple extreme environments such as low temperature, high pressure and strong magnetic field.

In this research, first, we verified a method to directly detect the structural phase transition on the electronic phase diagram by measuring the strain accompanied by the structural phase transition under high pressure condition in the cubic anvil high pressure apparatus. Furthermore, we aimed to realize ultrasonic measurement and establish a method to precisely measure the temperature dependence of structural physical quantity. In this study, we constructed an ultrasonic measurement system and verified whether ultrasonic echo can be observed under normal pressure, with the sample actually sealed with the pressure medium in the pressure cell.

3 Result and Discussion

3.1 Detection of strain by structural phase transition under high pressure condition

The strain gauge method is one of the laboratory-level methods for detection of the strain accompanying the structure transition of materials. As shown in fig.4(a), in this method, the sample is bonded with commercial strain gauge and the distortion of the sample is detected as the change of the resistance of strain gauge. Although there is the report that shows the strain gauge method with the piston cylinder cells[4], this method with the cubic anvil high pressure apparatus is difficult from the limitation of the sample size. Therefore, to detect the distortion in the cubic anvil high pressure apparatus by the laboratory level measurement, we thought that it is necessary to establish a new method with an alternative sensor for the strain gauge or to enlarge the size of the sample space. In this study, we considered to apply piezoelectric film made from PVDF (polyvinylidene fluoride) which is an ultrasonic transducer as an alternative sensor for the strain gauge. We also investigated whether the size of the sample space can be changed without changing the generated pressure.

3.1.1 Detection of distortion by using piezoelectric films

A piezoelectric film itself converts mechanical strain into electric charge. As shown in Fig. 4 (b), we considered that the distortion of the sample can be measured by simply measuring the electrical signal generated by this charge. Actually, there are a lot of attempts to use piezoelectric film as strain gauge in various research fields[5, 6, 7]. In particular, unlike the strain gauge, we can easily cut the film into required shapes. we thought that this characteristic is very important in the high pressure studies. Therefore, if we could detect strain by this method, we thought that it could be used even with the conventional sample space of the cubic anvil apparatus.

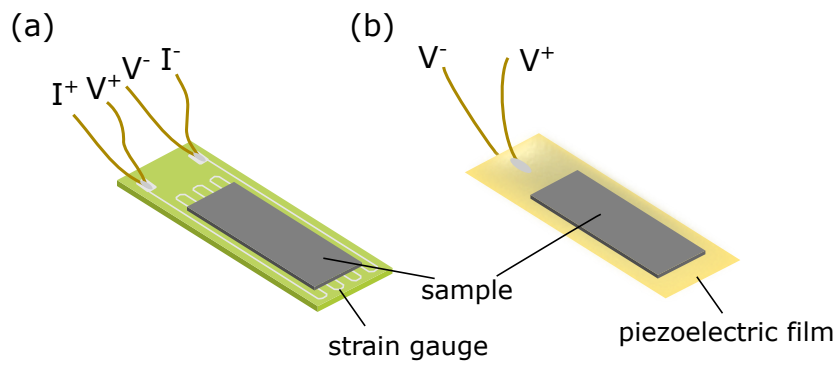

Figure 4 (a)The concept of the detection of strain accompanied with the structural phase transition by strain gauge method. (b)The concept of the detection of strain by using the piezoelectric film.

In order to verify the possibility of this method, as shown in Fig. 5, we attached a strain gauge and a piezoelectric film to a piezo actuator and examined their response when driving a piezo actuator at room temperature. Piezoelectric film is cut into a size of about 1 mm which is about the size of the sample used for the measurement under pressure. The result is shown in Fig. 6. The vertical axis represents the voltage of the piezoelectric film and the horizontal axis represents the change in the resistance of the strain gauge measured by constructing a bridge circuit used by the strain gauge method. The voltage of the piezoelectric film was measured by connecting it to a multimeter. As shown in the figure, it is understood that there is a correlation in the response to distortion, but the response of the piezoelectric film varies particularly when the distortion

is small. To investigate this result, we have studied the papers on the detection of static strain by piezoelectric film. It has been reported that the charge induced by strain in the piezoelectric film is suppressed when it is connected to the measurement systems. It is known that a piezoelectric film is a differential sensor and is sensitive to dynamic strains. The attempts have already been made in the previous researches to apply it to the detection of the static strain because the degree of freedom on the shape of the piezoelectric film. One of that is to measure the charge induced by strain using non-contact method. Another is to measure the change of the resonance frequency of the bimorf structure which is constructed with two piezoelectric films. However, these researches have been done at room temperature. Further verifications are necessary to judge whether it is similarly useful in low temperature and high pressure conditions.

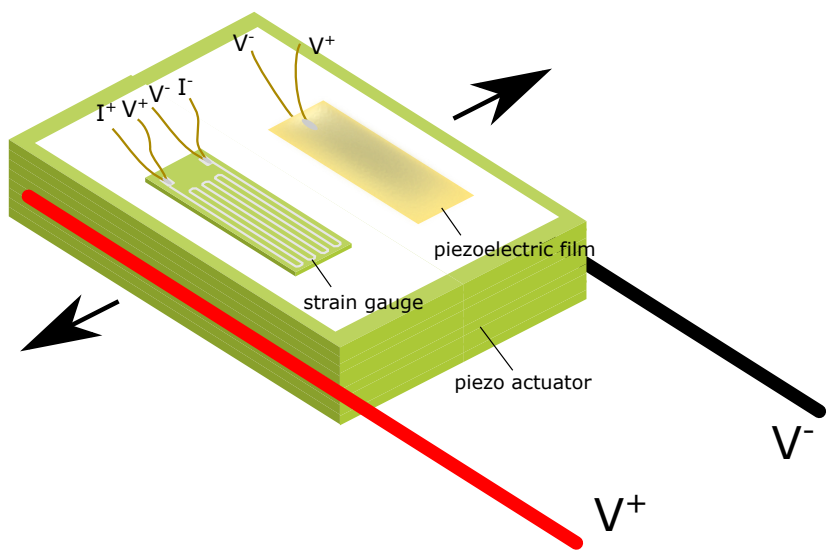

Figure 5 Concept of the condition to verify whether distortion can be detected by a piezoelectric film. Response of the strain gauge and the piezoelectric film when driving the actuator was investigated.

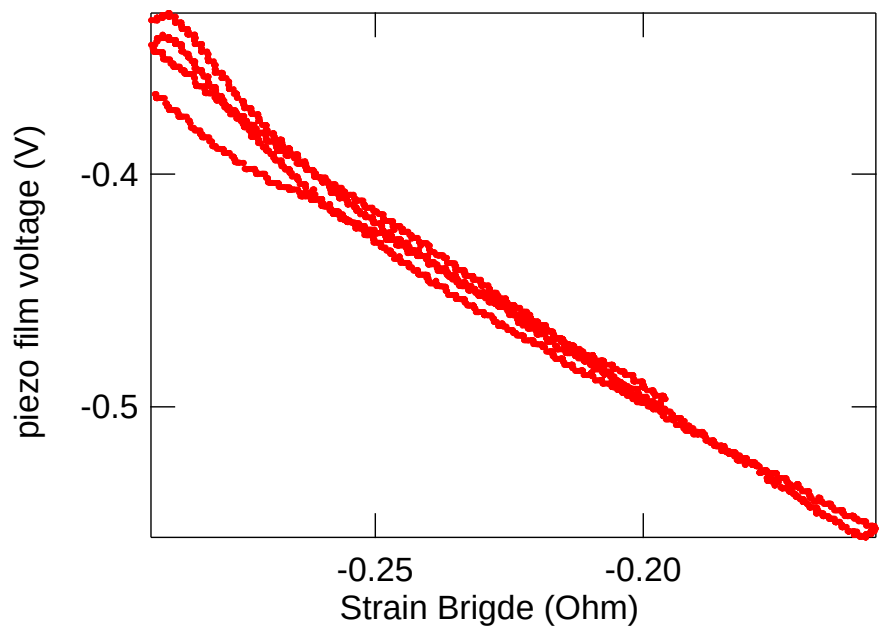

Figure 6 The response of the piezoelectric film and the strain gauge when driving the piezo actuator.

3.1.2 Examination of the sample space

In this research, our goal is to realize the detection of distortion accompanying the structural phase transition under high pressure. Moreover we aim to construct the ultra-sound measurement system.

In order to perform the measurement by the strain gauge method same as the conventional method, it is necessary to enlarge the volume of the sample space. Conventionally, the dimension of Teflon cell is following. The outer diameter is ϕ 2 mm and the height is 3 mm. In the above condition, the maximum of the size of the sample for the high pressure measurement is about $0.8 \times 0.3 \times 0.3$ mm. In the strain gauge from Japanese manufacturers, the smallest one is the high-temperature strain gauge from the Tokyo Instruments Laboratory. The gauge length is 0.2 mm and the gauge width is 0.8 mm. Even using this strain gauge, it is impossible to put it into the sample space without cutting off the parts except the gauge part. It is extremely difficult to make the electronic contact on the strain gauge after attached to the sample in this condition.

When measuring the sound velocity using the pulse-echo method, it is necessary to separate the 0th, 1st, 2nd, 3rd, ... th echoes appearing at regular intervals in time. The echo interval depends on the sample length, and a sample of about 1 mm - 3 mm is typically desirable. Furthermore, in addition to the thickness of the measurement sample, we need to consider the thickness of the transducer (about 100 mu m) to be attached to both sides of the sample. It exceeds the size of the sample that can be used by electrical resistance measurement.

From the above limitation, it is important to verify whether it is possible to enlarge the sample space. The upper limit of the pressure in the cubic anvil high pressure apparatus is known to depend on the size of the head of the six anvils[1, 2]. Not to change the upper limit of pressure from 8 GPa, we attempted to change the size of the Teflon cell without changing the size of the gasket. The dimension of the conventional Teflon cell is the outer diameter of ϕ 2 mm and the height of 3 mm. In this study, we investigated whether three structural phase transition points of Bi used for pressure calibration can be observed by using the Teflon cell with the diameter of ϕ 3 mm and a height of 3 mm. Every structural phase transition occurs at 2.55 GPa(I - II), 2.7 GPa(II - III) and at 7.7 GPa(III' - V) [1, 2].

Since the hole of the gasket for the cell set-up was slightly small, this time we scratched the hole by using a file and attached the cell into the gasket. The result is shown in Fig. 7. For the comparison, the results of pressure calibration of a Teflon cell with a conventional ϕ 2 mm and a height of 3 mm are also shown in the graph. The horizontal axis shows the magnitude of the load applied by the plunger pump and the vertical axis shows the value of the electrical resistance. Although the two structural phase transition points on the low pressure could be observed same as the conventional condition, the structural phase transition point of the highest pressure could not be observed. Fig.8 is a plot of the observed structural phase transition point into the calibration curve of the pressure in the conventional cell. As the two points on the low pressure were observed under nearly the same load as the conventional one, we think that the pressure can be similarly generated. It can be seen that the electric resistance is not stable under low pressure. We think that when the gasket starts to be compressed, the terminals are greatly affected than the conventional situation since the volume of the gasket decreases. Therefore, it is thought that it is necessary to suppress the pressurizing speed more than in the conventional condition. Since electric resistance increased at high pressure, we checked the gasket condition after depressurization. we found that the side of the cell was torn. In the future it is necessary to verify whether this is due to slight scratching of the gasket. Based on the above result, we expect that it is possible to make the size of the cell larger than that of the conventional one. we would like to determine the maximum of the cell size that can stably generate the pressure from ϕ 2 mm to ϕ 3 mm.

Furthermore, the ultra sound wave is high frequency. It in the MHz band. In the electrical resistance

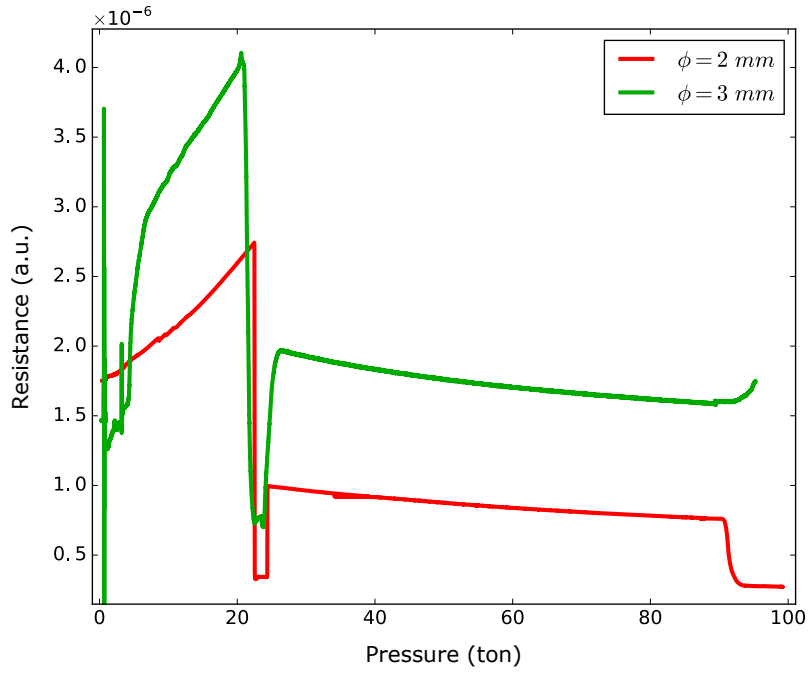

Figure 7 Pressure dependence of electrical resistance of Bi at room temperature

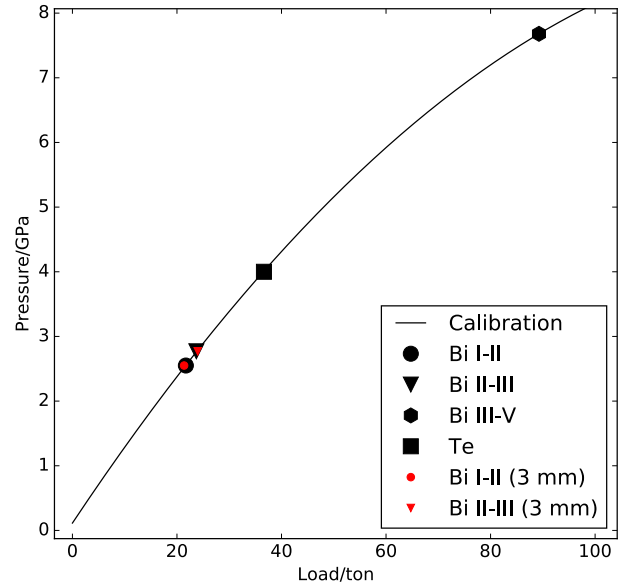

Figure 8 The plot of the structural phase transition points of Bi observed in the cell with the outer diameter ϕ 3 mm on the calibration curve in the cell with the outer diameter ϕ 2 mm

measurement and the AC magnetic susceptibility measurement, the measurement is perfomed with not so high frequency signal, and four of the six anvils except the upper and lower sides play as electrodes. In the case of a high frequency signal, there is the possibility that the ultra sound wave signal is suppressed in this situation. In fact, in a study that attempted ultrasonic measurement with a piston cylinder cell, coaxial cable was wired into inside of the piston cylinder, paying attention to suppression of the signal. Therefore, it is necessary to change the arrangement of the conventional electrode for the cubic anvil apparatus. In the conventional measurements, to measure the temperature of the sample space, a thermometer is attached to the anvil and the wiring is connected to the measurement system without making electrical contact with the anvil. From this arrangement, we thought that it would be possible to pass the coaxial cable from the side of the anvil to the part of the gasket. However, in the previous sample setup, the gold foil contact with the anvil. Therefore, it is

necessary to consider a method of extracting an electric signal from a sample without changing the size of the gasket. We attempted to verify whether it is possible to change the thickness of the upper and lower parts of the gasket. In this study, the size of the Teflon cell is ϕ2.5mm and verified whether the resistance can be measured with the anvil as an electrode as in the conventional case. As a result, the test was carried out three times, however, the electric signal could not be obtained immediately after the pressurization. From this result, when the height of the upper and lower parts of the gasket is different, the positions of the gold wire and the gold foil, which are the electrodes, are not in the center of pressure application. Therefore the effect of deformation by the compression of the gasket cause the gold wire or foil cut. In order to make the coaxial cable reach the pressurized part, it is necessary to consider another concept.

3.2 Ultrasound measurement in the high-pressure cell

In the present study, the MnV_2O_4 single crystal was cut into the cuboid shape with the dimension of 1.3 mm$\times$ 1.3 mm$\times$ 1 mm [Fig. 9(a)]. The surface was polished as mirror. Au was sputtered on the surface of $LiNbO_3$ which we used as transducers. After putting the sample in the teflon cell [Fig. 9(b)], it was connected with the probe, as shown in Fig. 9(c).

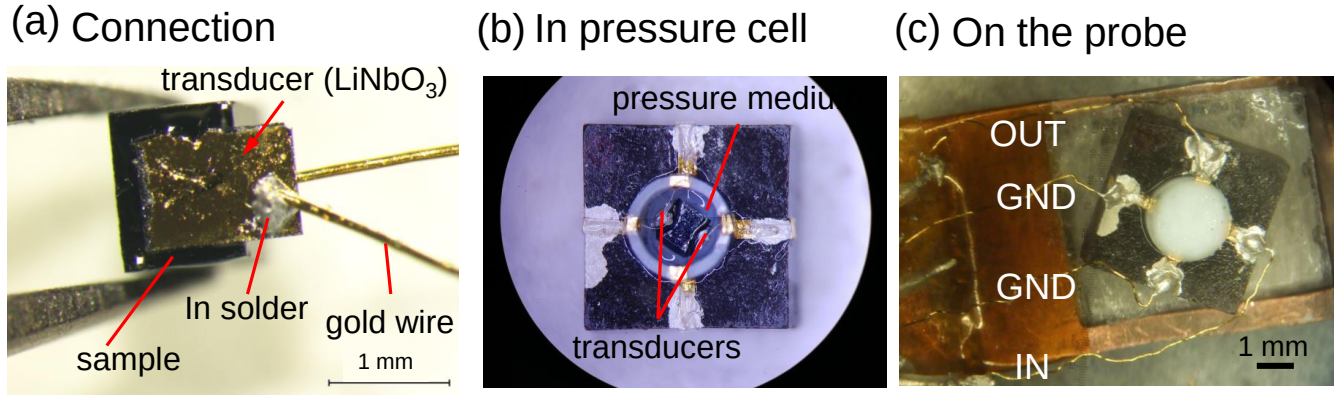

Figure 9 (a) The sample with $LiNbO_3$ transducers. (b) The sample in the pressure cell. (c) The sample connected with the probe.

Figure 10(a) shows the ultrasound echo signal. The vertical axis is the voltage of the electric signal. The horizontal axis is the time, triggered by the pulse signal from the pulse generator. At T =230 K, we observed the several echo signals with the time interval of about 400 ns which correspond to the echoes from 0 th to 4 th. The signal arriving before 0 th echo was considered as the electric noise. Solidification temperature of glycerin is 227 K, so we examined the influence of the solidification of glycerin on the echo signal. There was no distinct change between the echo signals at $T = 230$ K and $T = 225$ K.

Figure 10(b) is the waveform after the phase detection. Red and blue lines are the sin-component and the cos-component of the signal. The black line is the amplitude of the signal. The yellow line is the waveform of the gate signal of the boxcar-integrator. We measured the signal at this gate position. We confirmed that the echo signal was clearly detected in the amplitude signal. The temperature dependence of the amplitude signal revealed that the echo signals were not affected by the solidification of the glycerin [Fig. 10(c)].

Figure 11 shows the temperature dependence of the elastic constant C_{11} in MnV_2O_4. We observed the reduction of the elastic constant (elastic softening) toward the structural phase transition ($T_s = 56.5$ K), which was consistent with the previous study [8]. Thus, it was found that measurement can be carried out even under a sample enclosed in a Teflon cell.

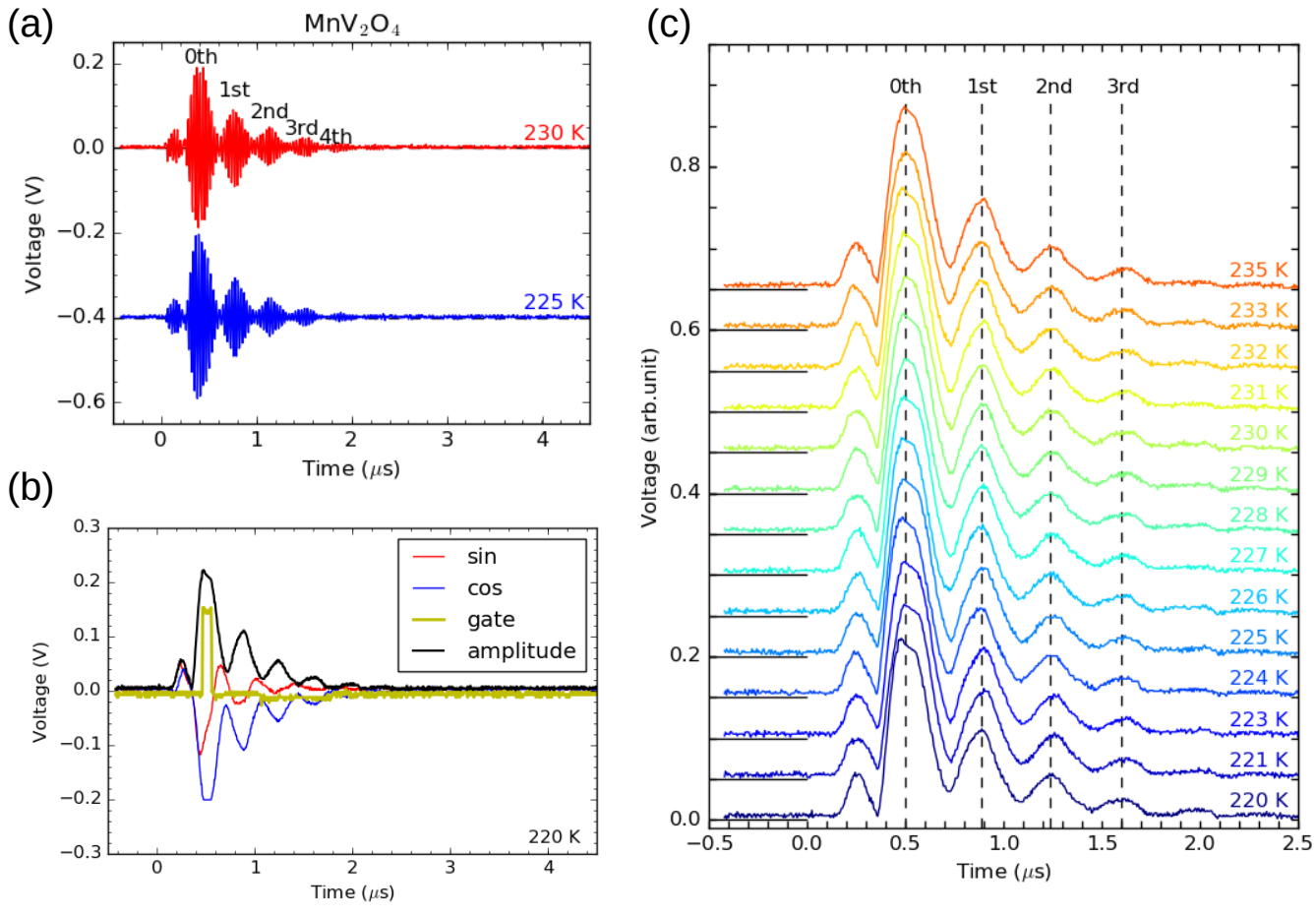

Figure 10 (a) Comparison of the echo waveforms around the solidification of the glycerin (T = 272 K). The waveform at T = 225 K was shown with the offeset of -0.4 V. (b) The signal after the phase detection. Red:sin-component of the signal after the phase detection, blue:cos-component of the signal after the phase detection, black: amplitude of the signal after the phase detection, yellow: the waveform of the gate signal by the boxcar-integrator. (c) The temperature dependence of the amplitude signal. 0th, 1st, 2nd, and 3rd denote the echo number. Each waveform is shown with the offset 0.05 V.

4 Conclusion and Future works

In this study, it was found that to use the piezoelectric film for detecting the strain accompanied with the structural phase transition, it is necessary to suppress the attenuation of thr electric charge induced by the strain when it is connected to the measurement system. For this reason, the method of non-contact measurement of charge or construct a bimorph structure as reported in a previous study is required. In addition, it is necessary to verify the pressure and temperature dependence of the characteristics of the piezoelectric film even if the above methods can be realized. Furthermore, since the piezoelectric film also has a pyroelectric effect, it is necessary to investigate whether the induced charge is affected when the temperature changes.

This study suggests that there is a possibility that the Teflon cell of the cubic anvil can be larger than the conventional one. Therefore, before verifying the above method with piezoelectric films, we will attempt to verify whether we can detect the strain associated with the structural phase transition with the strain gauge. In this study, the two structural phase transition point of Bi used for pressure calibration was observed. Compared to the case of measurement with the conventional cells, it was thought to be that the same pressure was generated with the same load even when using a large cell. In this measurement, structural phase transition occurring at 7.7 GPa of Bi can not be detected due to cracking of the pressure cell. In the future, we would like to verify whether it is possible to stably generate the pressure up to 8 GPa by using the large cells for the measurements.

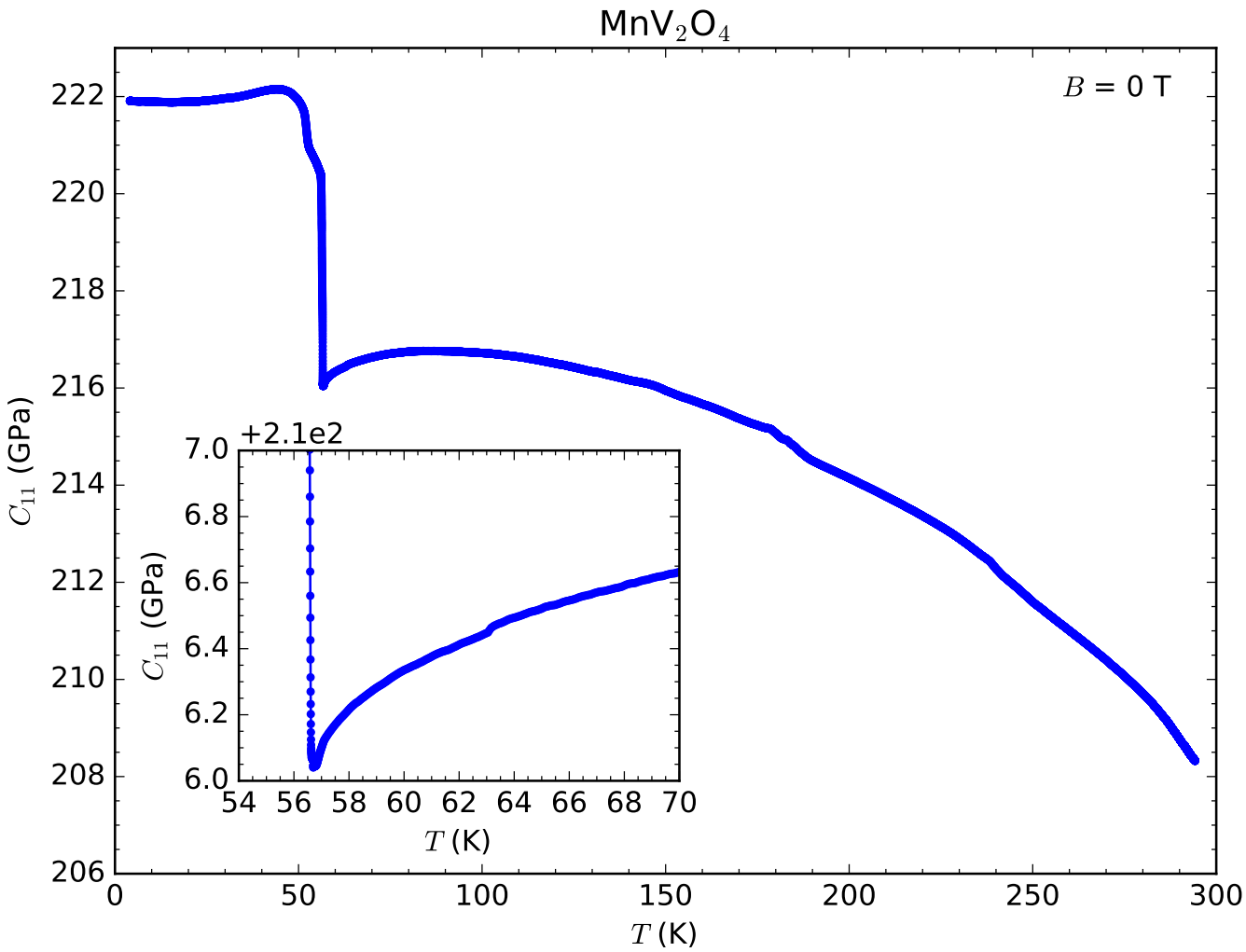

Figure 11 The temperature dependence of the elastic constant C_{11} in MnV_2O_4. The inset shows the enlarged graph around the structural phase transition ($T = 56$ K).

Furthermore, we would like to consider a new concept that can introduce the coaxial cable to the pressurized part even when changing the gasket.

Ultra sound echo could be observed at the ambient pressure even when the electric signal was taken out with the gold foil to the outside of the gasket in the setup of measurement under pressure. From this result, although there is a lot of challenging problem such as the arrangement of the coaxial cable, we expect the realization of the ultra sound wave measurement under high pressure in the future.

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