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Pressure calibration in solid pressure transmitting medium in large volume press

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The pressure limit in the large-volume-press (LVP) is increasing, but the *in situ* pressure calibration in LVP is still not a well resolved problem. The variation of the electrical resistance of the manganin with pressure in a hydrostatic condition is well known and is widely used in the pressure calibration in LVP. However, the hydrostatic pressure condition is hard to be maintained for the unavoidable solidification of the pressure transmitting medium (PTM) with pressure increasing. Moreover, our understanding about the relationship between pressure and manganin's resistance in a solid transmitting medium is still limited. Therefore, it is difficult to calibrate higher pressure using manganin. We measured the electrical resistance of manganin under pressure in pyrophyllite, MgO, and NaCl, respectively. The results show a linear relationship between the resistance and pressure in the same PTM with good reproducibility. In addition, the resistance-pressure relationships of manganin in different PTM are obviously different. So the resistance of manganin in a given solid PTM can be satisfactorily used as a pressure gauge only in the same PTM but cannot be used in other pressure media. Our results make it possible to calibrate higher pressure in a solid pressure transmitting medium in LVP. *Published by AIP Publishing.* [http://dx.doi.org/10.1063/1.4973448]

INTRODUCTION

In the study of high pressure, large volume press (LVP) is irreplaceable because of its unique properties, e.g., large sample chamber volume and high temperature control precision. LVP is also the best choice of high pressure device in the industrial production of super-hard materials.[1–4] With the development of technology,[5,6] the achieved pressure range in LVP is extending,[7] while the related pressure calibration problem remains to be solved. The pressure in LVP can be calibrated by the well-known fixed phase transition points of some materials (such as Bi, Tl, and Ba).[8–10] The pressure points sandwiched between the fixed points or beyond them are interpolated or extrapolated and it cannot apply to *in situ* pressure calibration. Besides, the accurate pressure is dependent on the precise calibration of the material's phase transition points. And this method is used infrequently in higher pressure range. In addition, based on the synchrotron X-ray diffraction technology, the equation of state of a specific material can be used to calibrate the pressure in LVP.[5–7] However, considering the cost of the synchrotron radiation devices, it is difficult to calibrate the pressure in all the current LVP by means of the equation of state.

The electrical resistance of manganin demonstrates an almost linear dependence on the hydrostatic pressure, and temperature has no significant effect on this linear relationship. The electrical resistance of manganin was used as a pressure gauge in the early high pressure experiments[11] because of its unique advantages such as convenient usage, high precision, and low cost. But the hydrostatic pressure environment in the sample chamber is heavily dependent on the pressure medium. Hydrostatic pressure can be attained in a liquid pressure medium, but most of the liquid pressure medium will solidify at higher pressure (usually higher than 15 GPa).[12] The solidification of the pressure transmitting medium (PTM) is a limit for using manganin as a pressure gauge.

The shortcomings of this method would be avoided if manganin could be directly used to calibrate the pressure in the solid pressure medium. Some attempts have been made. Samara and Giardini, for instance, reported that the pressure coefficient of manganin's resistance showed poor instability and reproducibility because the manganin is very sensitive to the shear stress existed in the solid pressure transmitting medium. Therefore, manganin is not suitable for pressure calibration in a solid PTM.[12] Instead, Fujioka *et al.* performed related high pressure experiments in an octahedral sample chamber using pyrophyllite as a PTM. They found that the relation between the pressure and resistance of manganin shows good linearity and reproducibility, which is in close agreement with the value obtained in the hydrostatic condition.[13] That is strange because the pressure coefficient of manganin's resistance should be unavoidably altered by the shear stress in a solid PTM, and it should not be the same with that under hydrostatic pressure.

Considering very few experimental researches have been carried out in this field, and some fundamental conclusions were still controversial, it is difficult to give an opinion of

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the feasibility of using manganin as a pressure gauge in the solid PTM. In the present work, we measured the relationship between the electrical resistance of manganin and pressure in pyrophyllite, MgO, and NaCl, three different solid PTM, to further verify the feasibility of manganin as a pressure calibration material in the solid PTM. The results show a linear relationship between the resistance and pressure in the same solid PTM with good reproducibility. But the relationships in different PTM are obviously different. So, the resistance-pressure relationship in manganin calibrated in a given solid PTM can be satisfactorily used only in the same PTM.

EXPERIMENTS

High pressure experiments were carried out in a 6 $\times$ 800 MN cubic press. The pressure control precision of the press is about 2 MPa. The PTM was an assembled cube ($32.5 \times 32.5 \times 32.5$ mm^3) consisting of two equal sized pieces ($32.5 \times 32.5 \times 16.25$ mm^3). A manganin wire 4 mm in length and 0.1 mm in diameter was used in this work and was located in a PTM in the center of the cubic. The chemical composition of manganin is Cu: 84 wt. %–Mn: 12 wt. %–Ni: 4 wt. %. We used the four-probe method to avoid the contact effects and lead-wire resistances. A constant-current source supplies the current (I), and the voltage signal (U) was exported to a multichannel data recorder. The change of electrical resistance of manganin can be obtained with simple mathematics (R = U/I). A specific experimental assembly is shown in Fig. 1.

RESULTS AND DISCUSSION

Pressure gradient widely exists in the solid PTM in high pressure experiments.[14] As a result, the measured electrical resistance of manganin wire is an integrate one. Thus the error would be generated from the existence of pressure gradient

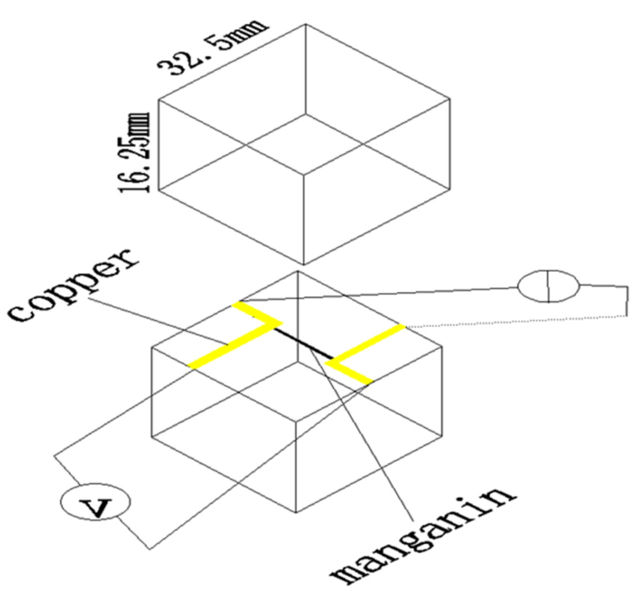

FIG. 1. Sketch of the experimental configuration.

in the solid PTM. In our experiments, the pressure gradient along the axis of symmetry of the pyrophyllite cube is about 50 MPa mm^{-1} when the pressure reaches 5.5 GPa.[14] The manganin wire used in this work was 4 mm in length. The biggest difference of the force loaded on the manganin wire is between the center and edge. In our measurements, it is 100 MPa (50 MPa/mm $\times$ 2 mm) when the pressure reaches 5.5 GPa. Therefore, the biggest error resulting from a pressure gradient in our experiments could be 100 MPa/5.5 GPa = 1.8%. Besides, the error of the multi-channel data recorder is about 1/6000. So the biggest error resulting in our experiments could be estimated about 1.8%.

The previous researchers generally used the manganin wire to calibrate the pressure according to the formula

$$P = K_0 \Delta R / R_0, \tag{1}$$

where K_0 is the resistance coefficient of pressure, which is a constant for manganin, and ΔR denotes $R_P - R_0$. R_0 is the resistance of the manganin wire under ambient pressure and R_p is the resistance of the manganin wire at pressure p. In large volume press, it is difficult to exactly measure the electrical resistance of the manganin wire in the low pressure region including R_0, so the above method is no longer applicable.[13]

However, if we compile Equation (1) to its equivalence form,

$$P = K R_p / R_{pA} - K_0, \tag{2}$$

where R_{PA} is the resistance at a fixed pressure point (in this experiment R_{PA} is at the phase transition pressure of Bi under 2.55 GPa). K denotes $K_0 R_{PA}/R_0$, which is also a constant for the manganin wire. Therefore, we can obtain the pressure in the cell using the measured R_{PA} and R_P through Equation (2) instead of using an inaccurately measured R_0. We measured the relationship between the electrical resistance and pressure of manganin in three different solid pressure transmitting media (pyrophyllite, MgO, and NaCl) through this method (Fig. 2). The pressure-resistance data with two different colors represent two experimental results, respectively. R_{PA} is the resistance of the manganin wire under 2.55 GPa.

As seen in Fig. 2, the linear relation between the resistance of the manganin wire and pressure demonstrated good reproducibility. But a slight deviation from the linearity was observed below 1.5 GPa. Because the contact resistance and the plastic flow of the pyrophillite remain significant in the low pressure region up to 1.5 GPa. Fig. 3 displayed the relationship between the electrical resistance and pressure in pyrophyllite, MgO, and NaCl, respectively. It is obvious that they are different. The pressure coefficient of the resistance change is the minimal in pyrophyllite and maximal in MgO. So the resistance-pressure relation of manganin measured in a given solid PTM can be satisfactorily used as a pressure gauge only in the same PTM, but it cannot be used in other PTM.

Table I listed the K and K_0 parameters in these three different PTM by fitting the resistance-pressure data above 1.5 GPa to Equation (2). It can be seen that the obtained K_0 parameters in the solid PTM are greater than the values derived in the hydrostatic condition, indicating that the resistance of the manganin wire is more sensitive to pressure in a liquid PTM. When the solid PTM is compressed, the shear stress

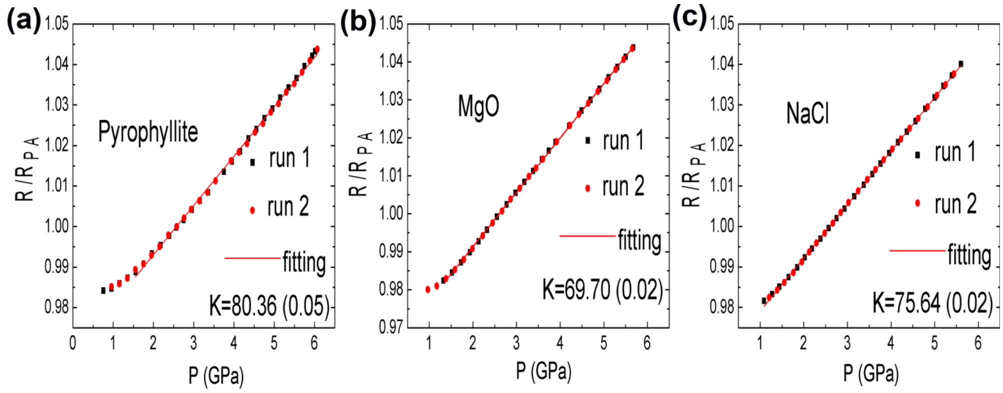

FIG. 2. The relative resistance of manganin as a function of the pressure in three different solid media.

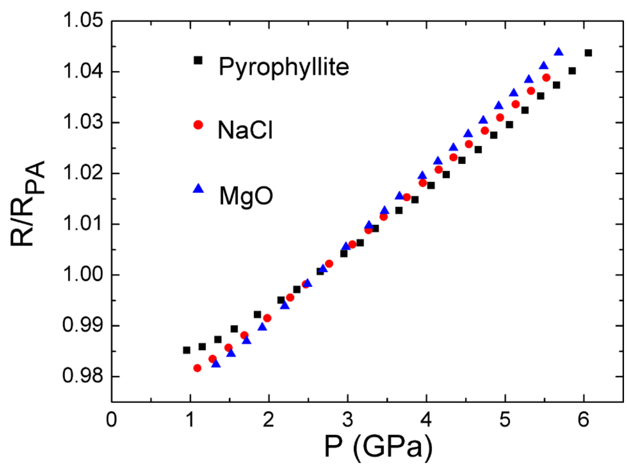

FIG. 3. Different responses of the manganin's resistance to the pressure in various PTM.

TABLE I. K and K_0 in different pressure transmitting media.

	Phyrophyllite[a]	NaCl[a]	MgO[a]	Normal and isopentane[b]
K	80.36 (0.05)	75.64 (0.02)	69.70 (0.02)	...
K_0	77.76 (0.05)	73.06 (0.02)	67.10 (0.02)	41.977 (0.282)

[a]This work.
[b]Data from Ref. 12.

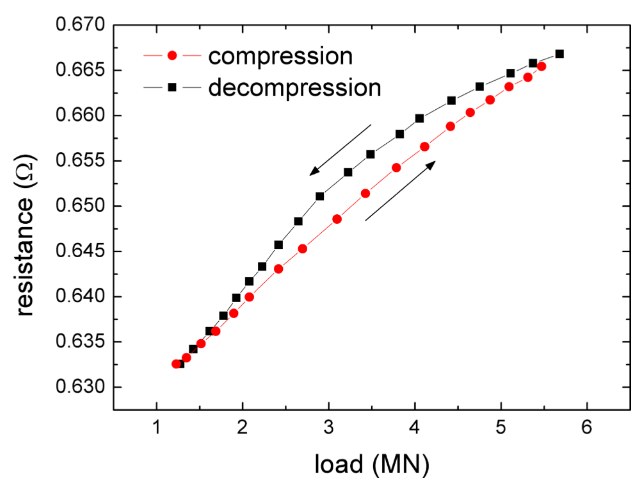

FIG. 4. Resistance of manganin in the compression and decompression processes.

would shorten the manganin wire simultaneously and change its shape. According to the electrical resistance formula $R = \rho L/S$ (where ρ is the material's resistivity, L is the length of the material, and S is the material's cross-sectional area), the decrease in length will reduce the measured electrical resistance of the manganin wire and consequently decrease the pressure coefficient of resistance. This effect has also been well studied and is used to study the compressibility of solid materials at high pressure.[15–19] Accordingly, the more the volume contraction of the solid PTM under high pressure, the pressure coefficient of resistance will be smaller. NaCl is much easier to be compressed than MgO. As a result the resistance variation of manganin is slower, and the fitted K_0 is larger in NaCl than that in MgO (seen in Table I and Fig. 3). Pyrophyllite is a composite containing SiO_2, Al_2O_3, and H_2O; thus it is hard to compare its compressibility with the other two materials. The pressure coefficient in the liquid PTM is the largest because the shear stress in liquid is negligible

We use the calibrated resistance-pressure relationship of the manganin wire in the decompression process to further verify its reliability. The resistance variation of manganin exhibits hysteresis in the same pressure region when the pressure is released (Fig. 4). Previous researches reported that the pressure hysteresis existed in the decompression cycle (at the same loading force, the pressure is larger when the releasing of pressure occurs) because of the friction of the gasket and the deformation of the pressure transmitting medium. And, the hysteretic resistance of manganin in the decompression run (Fig. 4) is owing to the practical pressure loaded on the manganin wire is different from that in the compression process.[20]

Fig. 5 shows the pressure calibration curve in pyrophyllite fitted with Bi I–II and Tl II–III phase transition points as well as the highest pressure point in compression (the initial data point in decompression). For comparison, the load-pressure data calibrated with the resistance change of the manganin wire in decompression were also plotted (Fig. 5). These two series of data meet very well, so it was verified that the calibrated resistance-pressure relationship of the manganin wire can be well used for the pressure calibration in the release of pressure. The load-pressure data shown in Fig. 6 are two experimental results using pyrophyllite as a PTM in decompression. The

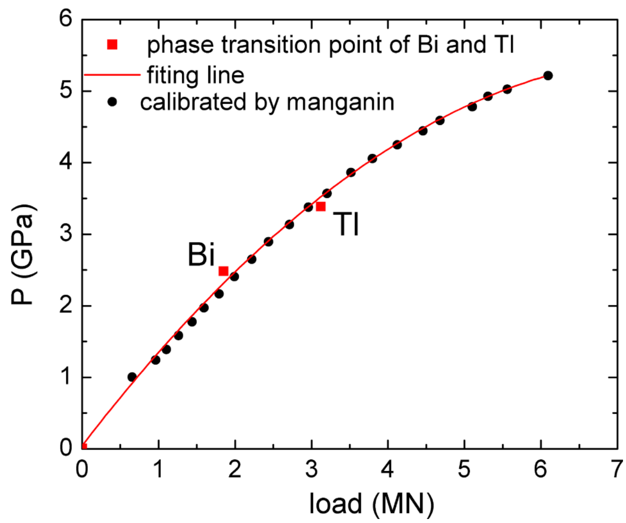

FIG. 5. The load-pressure curves calibrated by manganin and the phase transition points of Bi and Tl.

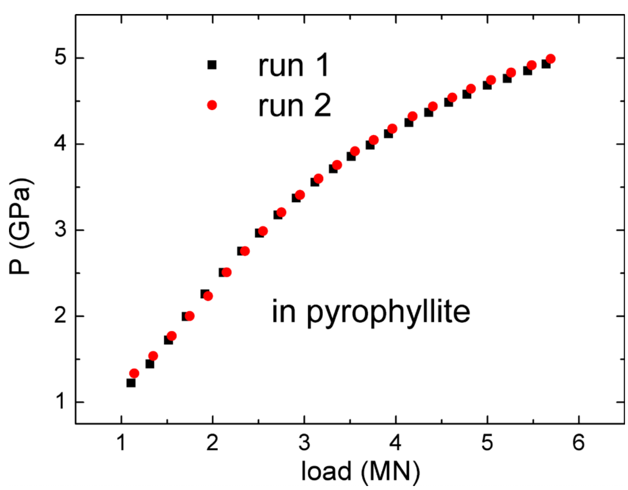

FIG. 6. The load-pressure curves during decompression in two different experiments using pyrophyllite as a transmitting medium.

results show good reproducibility of the pressure calibration curve. As discussed above, the calibrated resistance-pressure relationship of the manganin wire in a given solid PTM can be satisfactorily used as a pressure gauge in the same PTM.

CONCLUSION

In conclusion, the electrical resistance of the manganin wire was measured under pressure in three different solid PTM (pyrophyllite, MgO, and NaCl). The results show a linearity of the resistance-pressure relationship in the same PTM with good reproducibility. In addition, the resistance-pressure relationships of manganin in different PTM are obviously different. Thereby, the resistance of manganin in a given solid PTM can be satisfactorily used as a pressure gauge only in the same PTM. Our results show a possibility to calibrate the pressure in the solid pressure transmitting medium.

ACKNOWLEDGMENTS

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