

# Enhancement of superconductivity in thin films of Sn under high pressure

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We investigated the pressure effects of superconductivity on thin films of Sn. The elemental superconductor Sn with a body-centered-tetragonal structure,  $\beta$ -Sn, exhibits superconductivity below the superconducting transition temperature ( $T_c = 3.72$  K) at ambient pressure.  $T_c$  of Sn increases with lowering dimension such as in thin-film and nanowire growth, or by high-pressure application. For thin films,  $T_c$  exhibits a slight increase up to approximately 4 K compared to the bulk value, attributable to the crystalline size and lattice disorder. By applying pressure on a bulk Sn,  $T_c$  initially decreases from 3.72 K as the pressure increases. Further increasing pressure up to 10 GPa,  $T_c$  increases to 5.3 K with the structural transformation. However, the combination of these effects on thin films of Sn, namely, thin-film growth and pressure effects, remains underexplored. In this paper, we combined film-growth and pressure-application techniques to further increase  $T_c$  using a diamond anvil cell with boron-doped diamond electrodes. The drop of electrical resistance suggesting the onset of  $T_c$  on the thin film reached above 6 K in the  $\gamma$ -Sn phase. Further, the upper critical magnetic field was drastically enhanced. Atomic force microscopy suggests that the refinement of the grain size of the thin film under nonhydrostatic pressure conditions contributes to stabilizing the higher  $T_c$  of  $\gamma$ -Sn.

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## I. INTRODUCTION

Applying high pressure to directly compress a material is a useful approach to investigate intriguing physical properties and search for new materials [1,2]. For instance, oxygen—a gas at ambient conditions—exhibits metallic behaviors and superconductivity at high pressures [3,4]. Recently, high-temperature superconductors such as hydrogen-rich materials and nickelates under high pressure have attracted considerable attention [5–18]. In addition, recent discoveries of high-temperature superconducting states in elemental solid Ti and Sc at extreme pressures imply the potential of high-temperature superconductivity in the high-pressure phases of other elements [19–21].

Some elemental superconductors with thin-film dimensions show an increase in superconducting transition temperature ( $T_c$ ) compared to the bulk value, attributable to the crystalline size and lattice disorder [22–25]. In the case of Sn,  $T_c$  for thin films and nanowires varies slightly depending on the size and surface morphology [26–29]. The mechanism of  $T_c$  enhancement is not thoroughly clear although it has been proposed to arise from changes in the phonon density of states, the electron density of states, and the electron-phonon coupling [30–32].

Sn exhibits various crystal structures at high pressures [33–35]. Half a century ago, Wittig investigated the electrical

transport properties of bulk Sn at high pressures of up to 16 GPa and revealed the highest  $T_c$  is 5.3 K at 11.3 GPa on the pressure-induced phase,  $\gamma$ -Sn, where  $\beta$ -Sn shows superconductivity below 3.72 K at ambient pressure [36]. However, research on the combination of pressure application and thin-film growth on elemental superconductors is inadequate. Here, we hypothesize that changing the crystalline size of a thin film via pressure application could stabilize the higher  $T_c$ .

In this paper, we combined thin-film growth and pressure-application techniques to increase  $T_c$  using a diamond anvil cell (DAC) with boron-doped diamond (BDD) electrodes [37–39]. We investigated the pressure effects on the superconductivity of thin films of Sn compared to the bulk sample. We observed a higher  $T_c$  for the thin film compared to previous studies on the high-pressure phase. Further, we observed a drastically enhanced critical magnetic field on the thin film under high pressure.

## II. EXPERIMENTAL PROCEDURE

Thin films of Sn were deposited on a diamond anvil by resistance heating evaporation. The target metal was high-purity Sn, purchased from Kojundo Chemical Laboratory Company, Ltd. Comparing with the high-pressure measurements, we also prepared Sn thin films on a diamond substrate for electrical transport measurements at ambient pressure. The film deposition on the two diamonds was performed simultaneously. The optical images of the thin films on the diamond anvil and the diamond substrate are shown in Fig. 1(a) and the

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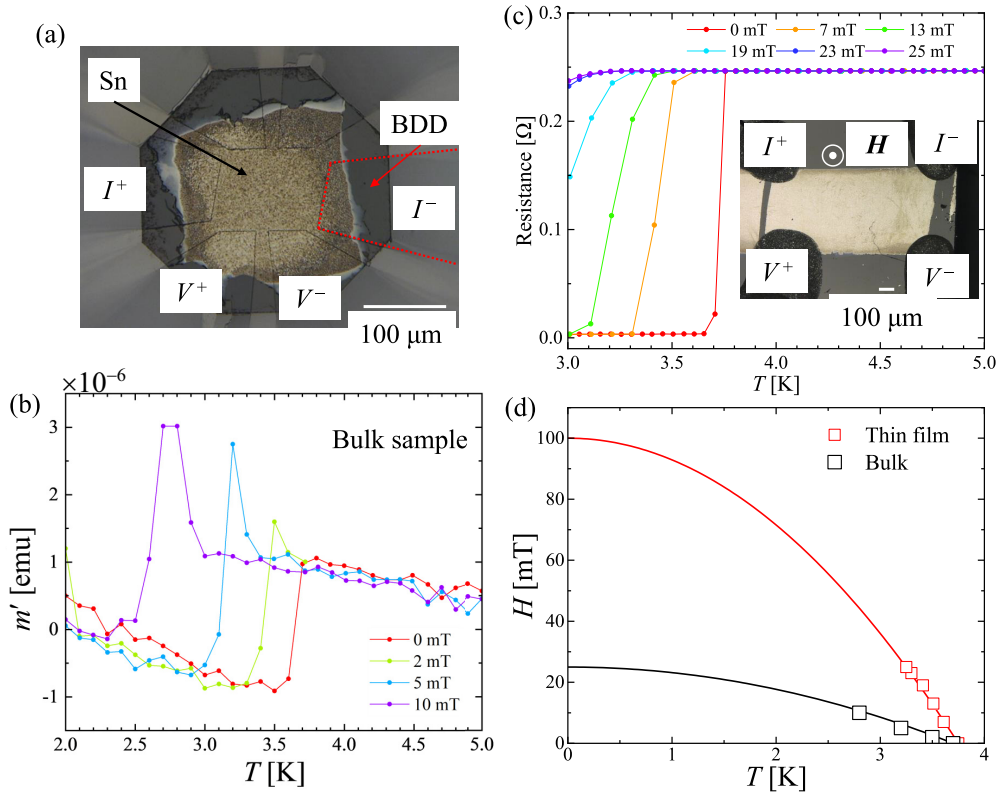


FIG. 1. (a) Optical image of a thin film of Sn on a diamond anvil with BDD electrodes. The dotted area indicates one of the BDD electrodes. (b)  $T$  dependence of the in-phase component of  $m'$  on the bulk Sn. (c)  $T$  dependence of  $R$  on a thin film of Sn on a diamond substrate. The magnetic field was applied perpendicularly to the film. The inset shows the optical image of a thin film of Sn on a diamond substrate. The thickness of the thin film was approximately 100 nm. (d)  $T$  dependence of the critical magnetic field on the thin film and bulk Sn.

inset of Fig. 1(c), respectively. The film thickness and surface morphology of the films were evaluated via atomic force microscopy (AFM; Nanoscope, SII NanoTechnology, Inc.) at room temperature. For the magnetic measurements of the bulk Sn, a wire of high-purity Sn (Nilaco Corporation) was used.

High-pressure generation was performed using DAC. The pressure value at room temperature was evaluated using ruby fluorescence and the Raman shift of diamond [40,41]. In the electrical transport measurements, a diamond anvil with BDD electrodes with a culet size of 300  $\mu\text{m}$  was used [37–39]. The BDD electrodes were deposited homoepitaxially on the diamond anvil by microwave plasma chemical vapor deposition [42]. This electrode exhibits high durability and can be reused until the diamond anvil itself fractures. Further, thin films could be deposited directly on the diamond anvil with BDD electrodes, eliminating the need for an electrode fabrication process after thin-film deposition [37,43,44]. The pressure-transmitting media in the solid, liquid, and gaseous states are compatible with this system. A gasket of stainless steel was preindented and a 200- $\mu\text{m}$ -diameter hole was drilled. The insulating layer was prepared using a MgO-epoxy mixture. We termed this setup as nonhydrostatic. We also performed high-pressure generation with a better hydrostatic pressure condition than the nonhydrostatic measurement using glycerol as the liquid pressure-transmitting medium. A 150- $\mu\text{m}$ -diameter hole was drilled in the insulating layer of the MgO-epoxy mixture to prepare the sample space, which was filled with glycerol. We termed this setup as quasihydrostatic.

In quasihydrostatic pressure measurements, the pressure value was evaluated using the Raman shift of diamond [41]. The electrical transports under a magnetic field ( $H$ ) perpendicular to the surface were measured by a four-terminal method by a physical properties measurement system (Quantum Design).

For magnetic measurements, we used a miniature DAC in combination with a superconducting quantum interference device magnetometer (MPMS, Quantum Design) [45–50]. A nanopolycrystalline diamond with culet size of 600  $\mu\text{m}$  and a preindented tungsten gasket with a hole size of 200  $\mu\text{m}$  were used [51]. Bulk Sn pieces and ruby powders were loaded into the sample space without a pressure-transmitting medium. The in-phase component of the ac magnetic response ( $m'$ ) was measured. The frequency and amplitude of the ac field were 3 Hz and 0.2 mT, respectively.

### III. EXPERIMENTAL RESULTS

#### A. Ambient pressure

Figure 1(b) shows the temperature ( $T$ ) dependence of  $m'$  for the bulk Sn under  $H$ , where no background signal from DAC is subtracted. Below 3.7 K, a diamagnetic signal suggesting a superconducting state was observed at  $H = 0$ . The  $T_c$  onset was decreased by applying  $H$ . Figure 1(c) shows the  $T$  dependence of the electrical resistance ( $R$ ) under  $H$  perpendicular to the film surface. The residual resistance ratio (RRR) was estimated to be 11. At  $H = 0$ , an  $R$  drop suggesting the

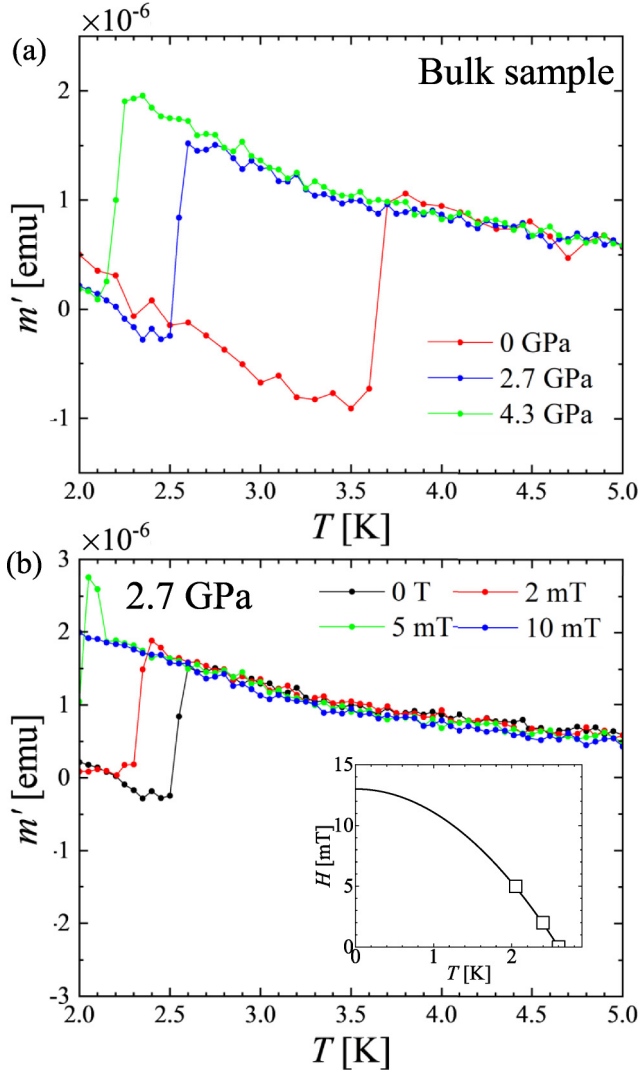


FIG. 2. (a)  $T$  dependence of  $m'$  on bulk Sn under high pressures. (b)  $T$  dependence of  $m'$  at 2.7 GPa under varying magnetic fields. The inset shows the  $T$  dependence of the critical magnetic field.

onset of a superconducting transition was observed at 3.75 K.  $T_c$  slightly increased compared to the bulk value and was similar to values from previous studies on thin films [27,28]. By applying  $H$ , the onset of  $T_c$  decreased with increasing  $H$ . However, the critical magnetic field was three times higher than that of the bulk Sn [Fig. 1(d)]. An  $H_c$  of 100 mT estimated using  $H_c(T) = H_c[1 - (T/T_c)^2]$  was similar to that of a previous study [28]. Considering  $H_c$ , the thin film transformed into a type-II superconductor, as previously reported [28,52,53].

### B. Nonhydrostatic pressure on bulk Sn

Figure 2(a) shows the  $T$  dependence of  $m'$  on the bulk Sn under high pressures. On applying pressure,  $T_c$  decreased, as previously reported. Figure 2(b) shows the  $T$  dependence of  $m'$  at 2.7 GPa under varying  $H$ . A decrease of  $T_c$  was observed by applying  $H = 2$  mT. At  $H = 10$  mT,  $T_c$  was below 2 K.  $H_c$  was evaluated to be 13 mT, as shown in the inset of Fig. 2(b).

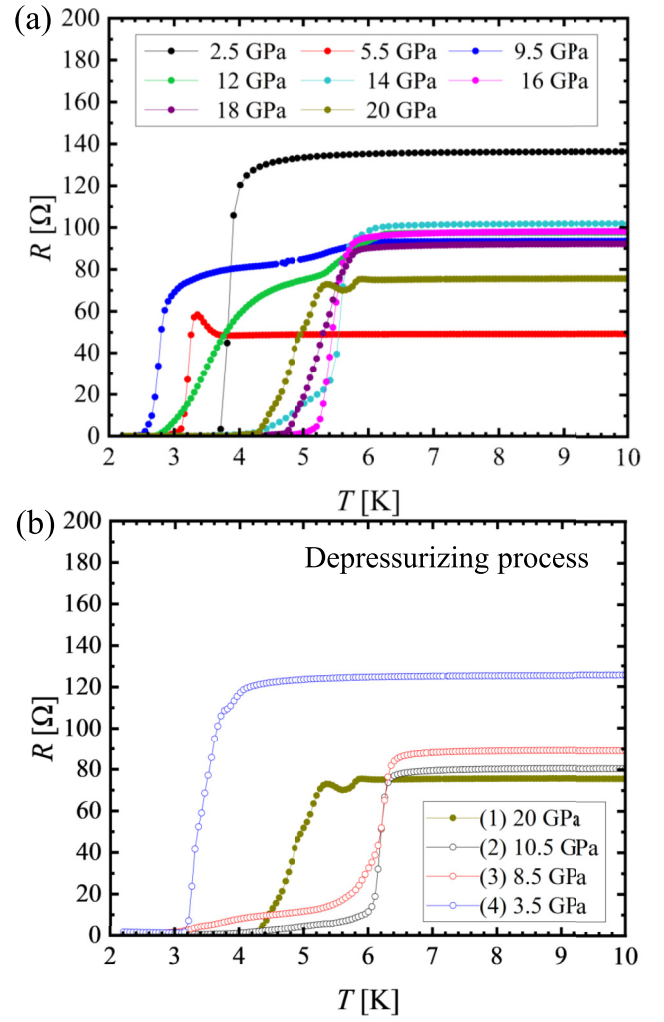


FIG. 3.  $T$  dependence of electrical  $R$  under nonhydrostatic pressure with a (a) pressurizing process and (b) depressurizing process. The number in (b) indicates the order of measurements.

### C. Nonhydrostatic pressure on thin film

Figure 3(a) shows the  $T$  dependence of  $R$  on a thin film of Sn under high pressures on pressurization. As shown in the inset of Fig. 4(a), RRR was estimated to be 1.4 at 2.5 GPa, which was much lower than that under ambient pressure. We speculate that the crystallographic defect was introduced because of the nonhydrostatic pressure condition. A  $R$  drop suggesting the superconducting transition was observed around 4 K, whereas  $T_c$  was 3.75 K under ambient pressure.  $T_c$  decreased on further increasing the pressure, as previously reported.  $R$  showed peak behavior just above  $T_c$  at 5.5 GPa, possibly due to the granularity or disorder on the thin films. Above 9.5 GPa,  $R$  tended to slightly decrease around 6 K, suggesting the superconducting transition, where the  $\gamma$ -Sn phase could emerge. With further pressure application, the  $R$  drop became significant, and  $T_c$  slightly decreased. After applying 20 GPa, the pressure was decreased to 10.5 GPa. Figure 3(b) shows the  $T$  dependence of  $R$  for a thin film of Sn under high pressures with depressurization. The onset of  $T_c$  increased to 6.3 K at 10.5 GPa, and  $\gamma$ -Sn remained at 8.5 GPa.  $\gamma$ -Sn vanished when the pressure was decreased to 3.5 GPa.

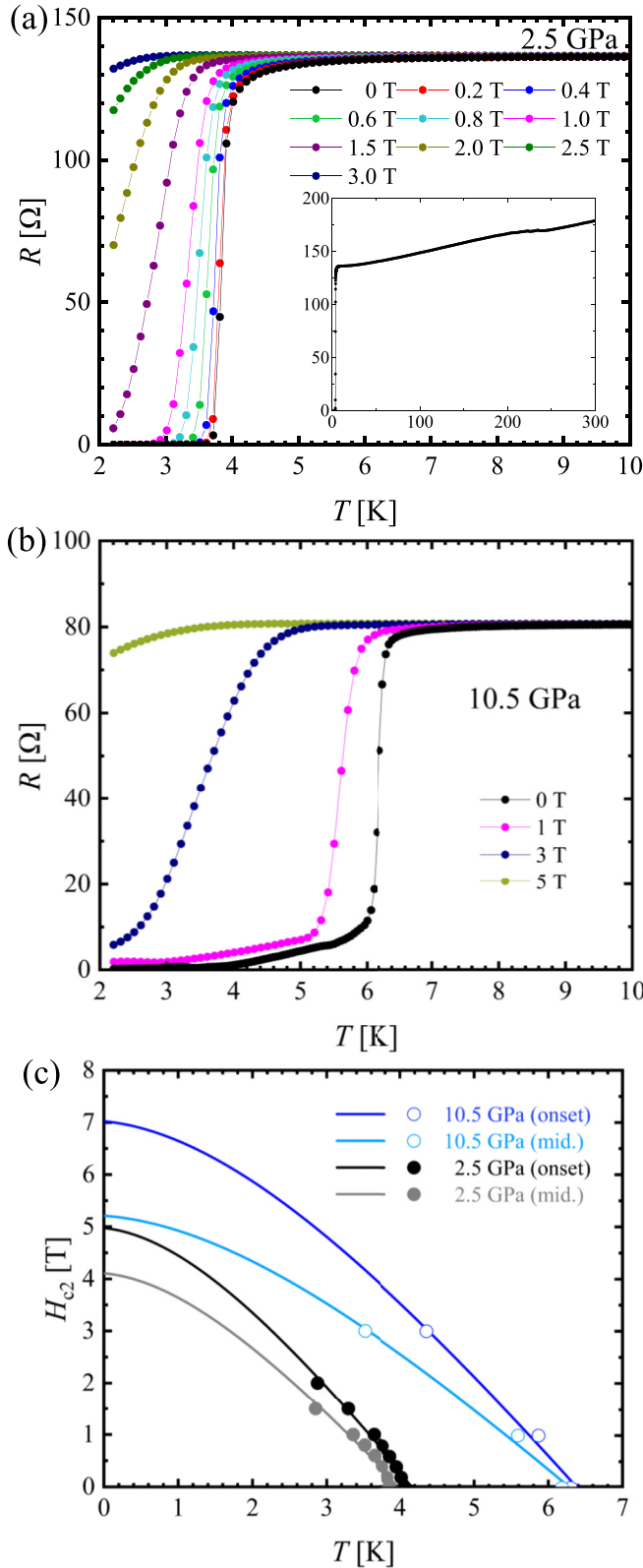


FIG. 4. (a) and (b)  $T$  dependence of  $R$  under a magnetic field at (a) 2.5 and (b) 10.5 GPa. The inset of (a) shows  $T$  dependence of  $R$  between 2 and 300 K at 2.5 GPa without an external magnetic field. (c)  $T$  dependence of the upper critical magnetic field at 2.5 and 10.5 GPa. The solid lines represent the fitting curves estimated by the WHH model.

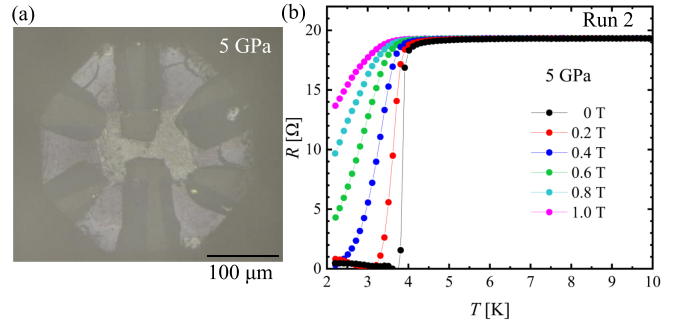


FIG. 5. (a) Optical image of a thin film of Sn at 5 GPa and (b)  $T$  dependence of  $R$  at 5 GPa.

Figures 4(a) and 4(b) show the  $T$  dependence of  $R$  for a thin film of Sn under various  $H$  at 2.5 and 10.5 GPa under the nonhydrostatic pressure condition.  $T_c$  was observed at 1 T, whereas the upper critical magnetic field ( $H_{c2}$ ) was approximately 0.1 T under ambient pressure. A drastic  $H_{c2}$  enhancement was also observed in the  $\gamma$  phase at 10.5 GPa. As shown in Fig. 4(c),  $H_{c2}$  estimated using the Werthamer-Helfand-Hohenberg (WHH) model reached several teslas at high pressures [54–56]. Notably, the  $H_{c2}$  enhancement of  $\beta$ -Sn under the nonhydrostatic pressure condition was observed reproducibly. Figure 5(b) shows the  $T$  dependence of  $R$  for a thin film of Sn with the other setup at 5 GPa. The optical image of a thin film of Sn is shown in Fig. 5(a). A decrease in  $R$  suggesting the superconducting transition was observed even at  $H = 1.0$  T.

There are two possible scenarios for the  $H_{c2}$  enhancement. The first is the enhancement of the flux-pinning force. The crystallographic defects introduced by nonhydrostatic pressure could serve as pinning centers, potentially leading to an increase in  $H_{c2}$ . The other is the shortening of the mean free path of the electrons and the resulting shortening of the coherence length. Considering that the thickness of the thin film was approximately 100 nm, the electrical resistivity of the normal state near  $T_c$  was estimated to be approximately  $2.5 \mu\Omega \text{ cm}$  for the ambient pressure measurement. This value is consistent with those reported in previous studies [28,53]. In contrast, the electrical resistivities of the thin films for 2.5 GPa of run 1 and 5 GPa of run 2 were approximately  $1.7 \times 10^3$  and  $81 \mu\Omega \text{ cm}$ , respectively. The increase in the electrical resistivity near  $T_c$  suggests that the mean free path of the electrons is shortened by the application of pressure.

#### D. Quasihydrostatic pressure on thin film

We performed high-pressure generation with a better hydrostatic pressure condition using glycerol as the liquid pressure transmitted medium. Figure 6(a) shows the  $R$ - $T$  of the thin film at 9 GPa under quasihydrostatic pressure conditions. The optical image of the thin film inside the DAC is shown in the inset of Fig. 6(a). The RRR was within 2–3 in quasihydrostatic pressure measurements, which was slightly higher than those for the nonhydrostatic pressure condition. An  $R$  drop suggesting a superconducting transition was observed around 5 K. Figure 6(b) shows  $R$ - $T$  under a



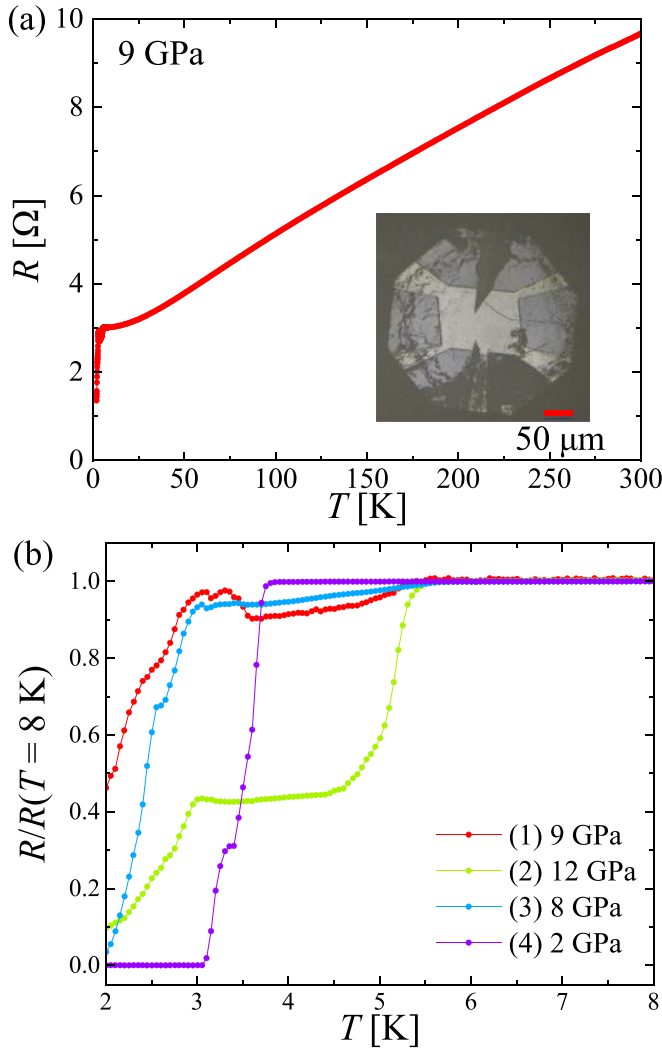


FIG. 6. (a)  $T$  dependence of  $R$  at 9 GPa. The inset shows the optical image of the thin film. (b)  $T$  dependence of  $R$  under high pressures between 2 and 8 K.

quasihydrostatic pressure condition between 2 and 8 K with the warming process. The  $R$  value was normalized using the value at 8 K. At 9 GPa,  $R$  slightly decreased with decreasing  $T$  around 5.3 K, suggesting the superconducting transition on  $\gamma$ -Sn. An  $R$  drop was also observed around 3 K. The  $\gamma$ -Sn phase became significant on further pressure application. The onset of  $T_c$  slightly increased at 12 GPa. We also decreased the pressure from 12 to 8 GPa. A decrease of  $R$  suggesting the superconducting transition of  $\gamma$ -Sn was slightly observed, whereas  $\gamma$ -Sn was clearly observed at 8.5 GPa under the nonhydrostatic pressure condition. With a further pressure decrease,  $\gamma$ -Sn vanished at 2 GPa.

#### IV. DISCUSSION

##### A. Pressure dependence of $T_c$

Figure 7 shows the pressure dependence of  $T_c$  for the thin film and bulk samples compared with results from a previous study [36]. For the bulk Sn, the behavior of  $T_c$  with respect to pressure was in good agreement with the previous study. A

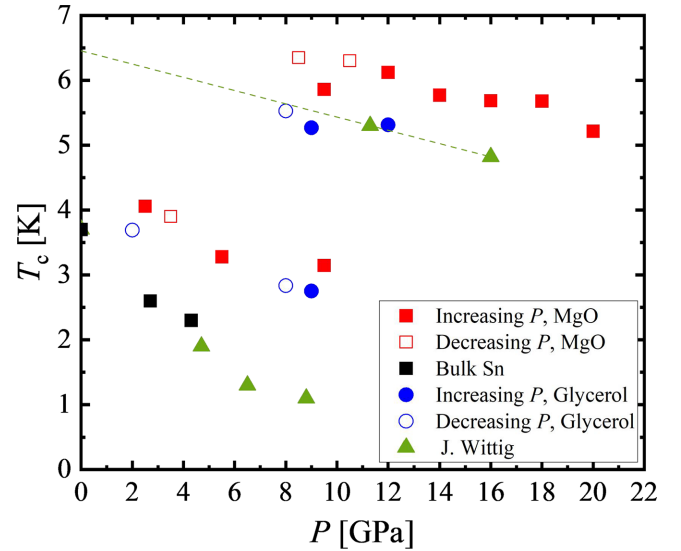


FIG. 7. Pressure dependence of the superconducting transition temperature. The green triangles indicate the results by Wittig [36]. The dotted line indicates a guide for the eyes.

similar tendency was observed for the thin film in the  $\beta$ -Sn phase, however, its  $T_c$  was higher compared to bulk Sn. One possible reason is the geometry of the thin film. As shown in Fig. 1(a), the thin-film area occupies approximately 70% of the culet of the diamond anvil, which produces the pressure distribution. In the  $\gamma$ -Sn phase,  $T_c$  with quasihydrostatic pressure measurements showed a trend similar to previous results [36]. Meanwhile,  $T_c$  with nonhydrostatic pressure showed a higher value. We observed the highest  $T_c$  of 6.3 K at 10.5 GPa, which was approximately 10% higher than that reported in a previous study [36]. The highest  $T_c$  is not fully explained by the pressure gradient. Assuming that the  $T_c$  of bulk  $\gamma$ -Sn continues to change at a rate of  $-0.11$  K/GPa, it is necessary to decrease the pressure to 4 GPa from the  $\gamma$  phase above 10 GPa. However, the  $\gamma$  phase cannot exist metastably under this pressure as was observed during the  $\gamma$  to  $\beta$  phase transition on the decompression process [Fig. 3(b)]. We emphasize that we observed the midpoint of  $T_c$  to be greater than 6.0 K [Fig. 4(b)].

##### B. Atomic force microscopy

To investigate the morphology on the thin films, we performed AFM measurements before and after applying pressure under the nonhydrostatic pressure condition. Figures 8(a) and 8(b) show the optical and AFM images of the thin film before pressurization. The thin film of Sn was deposited on a diamond anvil without BDD electrodes. The average film thickness was evaluated to be 70 nm. A magnified AFM image is shown in Fig. 8(c). The average grain size was estimated to be approximately 300 nm. Figure 8(d) shows the optical image of the thin film after applying pressure up to 4.2 GPa. The pressure was evaluated by the diamond Raman shift. Most of the thin film was peeled off and transferred to the MgO-epoxy mixture. We measured the small remaining thin-film area. Figure 8(e) shows the AFM image of the thin film after pressurization.

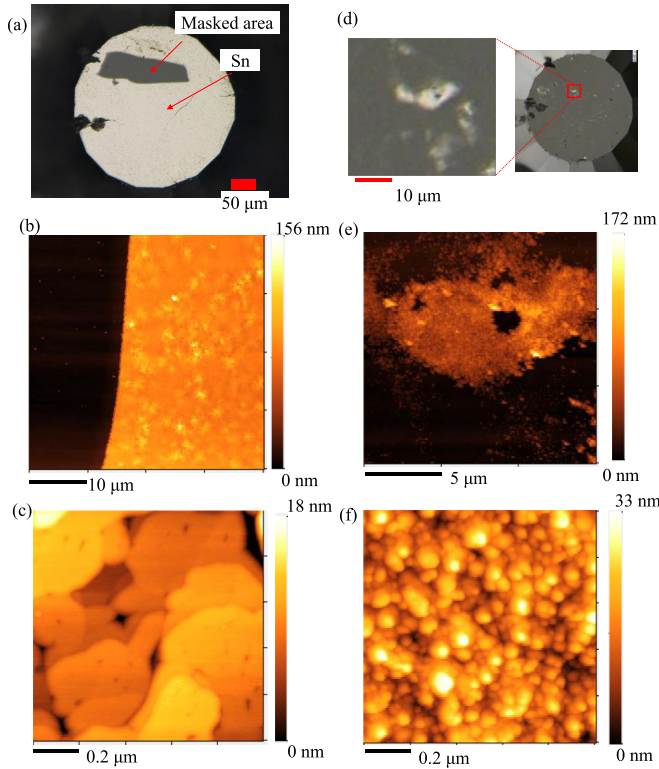


FIG. 8. (a) Optical image of the thin film of Sn on a diamond anvil before pressurization. (b) and (c) AFM image on the thin film before pressurization. (d) Optical images of the thin film of Sn on the diamond anvil after pressurization. (e) and (f) AFM image of the thin film after pressurization.

The average film thickness was evaluated to be 60 nm. A magnified view is shown in Fig. 8(f). The grain size was reduced to several tens of nanometers on pressure application.

Next, we performed AFM measurements under a quasi-hydrostatic pressure condition for comparison with the nonhydrostatic pressure measurements. The thin film was the same as that used in the  $R$ - $T$  measurement. Figures 9(a) and 9(b) show the AFM images of the thin film before applying pressure. The average thickness and average grain size were evaluated to be 82 and 600 nm, respectively. Figures 9(c) and 9(d) show the AFM images on the thin film after applying pressure up to 12 GPa. The pressure-transmitting medium was removed using ethanol and nitrogen gas. The average thickness was evaluated to be 91 nm. Unlike the nonhydrostatic pressure measurements, grain refinement was not observed.

The AFM results revealed that the grain refinement was observed only under the nonhydrostatic pressure condition. In the  $R$ - $T$  results, the maximum  $T_c$  value under the nonhydrostatic condition was 10% higher than that under the quasi-hydrostatic condition, suggesting that grain refinement plays a pivotal role in stabilizing the higher  $T_c$ . Smaller grain sizes tend to have higher  $T_c$ , because of phonon softening under ambient pressure [32]. Houben *et al.* performed nuclear resonant inelastic x-ray scattering on nanostructured films and bulk Sn to investigate the phonon density of states and observed a decrease in the high-energy phonon modes and a slight increase in the low-energy phonon modes in

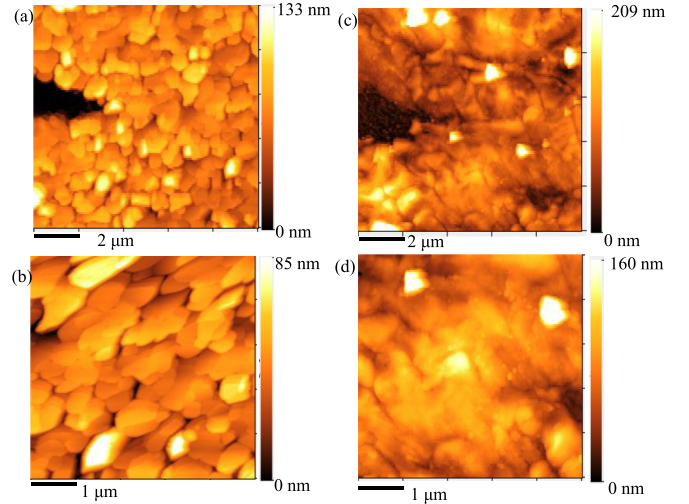


FIG. 9. AFM under quasi-hydrostatic pressure condition. (a) and (b) AFM image of the thin film before pressurization. (c) and (d) AFM image of the thin film after pressurization.

nanostructured films [31,32]. Using the obtained phonon spectra, calculations based on the Allen-Dynes-McMillan formalism yielded  $T_c$  values in good agreement with the experimental data. In nanostructured films, the electron-phonon coupling increased by up to 10%, suggesting that phonon softening and the associated change in electron-phonon coupling play a major role in the  $T_c$  increase. We consider that the  $T_c$  increase under the nonhydrostatic pressure condition is related to the grain size and that grain refinement could induce changes in the electron-phonon coupling.

### C. Possible higher $T_c$ on Sn

Recently, anomalies in magnetization, resistance, and heat capacity suggesting the superconducting transition were observed around 5.5 K in nanowires of Sn [57]. Further, based

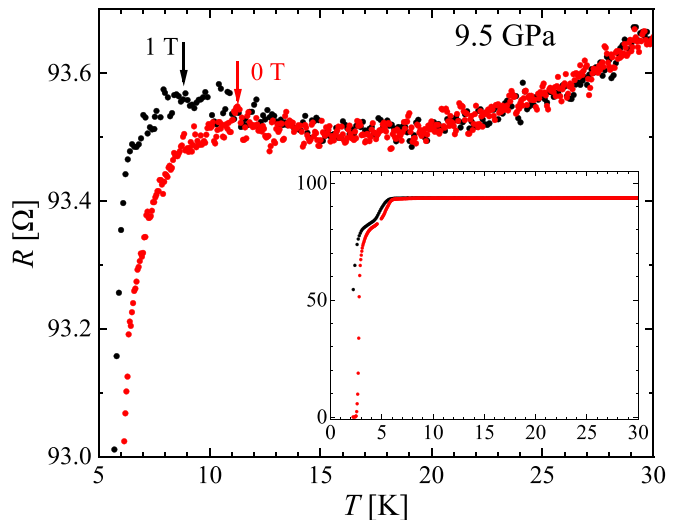


FIG. 10. Temperature dependence of the electrical resistance at 9.5 GPa. The downward arrows indicate the onset of the anomalies in the resistance.

on scanning tunneling microscopy, a thin film of Sn deposited on the  $\text{SrTiO}_3$  substrate exhibited superconductivity around 8 K [58]. Indeed, in our thin film, a decrease of  $R$  was observed at 11 K under 9.5 GPa, suggesting the signature of the superconducting transition, and the anomaly shifted to lower  $T$  by applying the magnetic field (Fig. 10). On the other hand, some granular or amorphous thin films exhibit a decrease in resistance at higher  $T$  than  $T_c$  due to the effects of fluctuations [59–62]. Further investigations such as magnetic measurements [63,64], heat capacity measurements [65], and scanning tunneling microscopy [66] under high pressure may offer insights for the possible stabilization of higher  $T_c$ .

## V. CONCLUSION

In conclusion, we demonstrated the pressure effect on the superconductivity of thin films of Sn. We observed superconductivity below 6.3 K in the  $\gamma$  phase of Sn, which was

approximately 10% higher than previous bulk results. Further, the  $H_{c2}$  drastically increased under the nonhydrostatic high-pressure condition. We also observed the signature of the superconducting transition at higher  $T$  than  $T_c$ . AFM results suggest that the grain refinement under nonhydrostatic pressure contributes to the stabilization of the higher  $T_c$  of  $\gamma$ -Sn.

## ACKNOWLEDGMENTS

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## DATA AVAILABILITY

The data that support the findings of this article are openly available [67].

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