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Superconductivity in titanium probed by AC magnetic susceptibility to 120 GPa

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Superconductivity in titanium probed by AC magnetic susceptibility to 120 GPa

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We used a highly sensitive AC magnetic susceptibility technique to probe superconductivity in elemental titanium (Ti) under extreme pressures to 120 GPa in a diamond anvil cell (DAC). The measurements reveal that the critical temperature (T_c) of Ti rises monotonically with increasing pressure, reaching 6.1 K at 120 GPa. Our results confirm the bulk nature of the superconductivity in Ti, as evidenced by a robust diamagnetic response in the AC magnetic susceptibility. Our work provides a routine technique to probe Meissner effect of elemental superconductors at megabar pressures.

Keywords: superconductivity, high pressure, susceptibility

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1. Introduction

Elemental superconductors, despite their simplicity, are crucial for both fundamental research and technological applications.[1–4] They provide foundational knowledge for understanding superconductivity, serve as benchmarks for more complex systems, and continue to offer insights through the study of their behavior under extreme conditions. To date, more than 50 elemental solids in the periodic table have been reported to be superconductors at ambient pressure or high pressure.[5,6] The transition elements are by far the most successful and exhibit high critical superconducting temperature (T_c), e.g., scandium (Sc) ($T_c > 30$ K at 250 GPa),[5,7–9] titanium (Ti) ($T_c \sim 26$ K at 240 GPa),[10,11] vanadium (V) ($T_c \sim 17$ K at 120 GPa),[12] and yttrium (Y) ($T_c \sim 20$ K at 100 GPa),[13] etc. This is reasonable, as the presence of d-electron character in the conduction band significantly enhances the electronic density of states at the Fermi energy, $N(E_F)$, a parameter that drives an exponential increase in the critical temperature, according to the BCS theory. Under pressure, the increase in fractional ion core volume promotes s–d electron transfer and raises the d-electron count, which generally leads to an expected enhancement of the critical temperature T_c.[14,15]

Applying extreme pressures using diamond anvil cells (DACs) significantly limits sample size and homogeneity, posing considerable challenges for *in-situ* characterization. This limitation is especially pronounced when probing the magnetic signatures of superconductivity. Despite the growing interest in elemental superconductors above megabar pressures ($\geq$ 100 GPa), the Meissner effect, a definitive hallmark of superconductivity, has been successfully measured in only a limited number of these materials, i.e., Sc and Lu,[5] Yb,[6] Y,[13] and chalcogens.[16]

Superconductivity in high-pressure phases of titanium (Ti) was initially observed with a maximum critical temperature of 3.5 K at 56 GPa.[17] Subsequent studies demonstrated significant pressure-enhanced superconductivity in Ti over a broad range, with maximum T_c values exceeding 26 K.[10,11] However, the Meissner effect has never been reported in the elemental superconductor Ti. In this paper, we present the results of temperature-dependent AC magnetic susceptibility measurements using a balanced coil system conducted on Ti at pressures up to 120 GPa. Our findings confirm the bulk nature of superconductivity in Ti, as evidenced by a robust diamagnetic response in the AC magnetic susceptibility.

2. Experimental methods

The diamond anvil cell, made of BeCu alloy, contains two opposing type IIa diamond anvils with 0.2 mm diameter culets. A CuBe gasket 2.75 mm in diameter by 300 μm thick was preindented to 35 μm thickness and then a 140 μm diameter hole was laser drilled through the center of the preindentation area. The prepared gasket was placed back on the diamond inside the main coil, as shown in Fig. 1(a). A small chip of Ti was cut from a high-purity foil obtained from the Alfa Aesar Chemicals and then packed as densely as possible into the gasket hole, as shown in Fig. 1(b). No pressure medium was used in the present experiments, for the purpose of maximizing the sample volume under extreme pressure conditions, and thus

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the magnitude of the diamagnetic signal at the superconducting transition. Pressure was determined by diamond Raman shift[18] at low temperature (∼ 4 K).

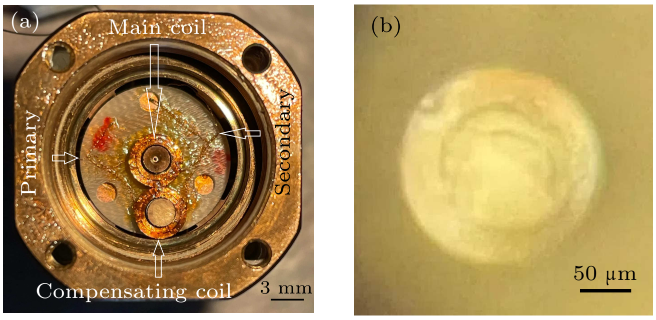

Fig. 1. Pictures of the coil system and the sample chamber. (a) A close view of the balanced coil system. The drilled pre-indented BeCu gasket was placed on top of a diamond inside the main coil to serve as a sample chamber. A dummy BeCu gasket was placed inside the compensating coil for compensating the background of the gasket in the main coil. The reference sample NbTi was placed below the dummy gasket. The primary leads connect to the AC current source (Keithley 6221), and the secondary leads connect to the lock-in amplifier (SR830) via a pre-amplifier (SR554). (b) A close view of the sample chamber with Ti sample sealed in the hole.

As illustrated in Fig. 1(a), the superconducting transition is detected inductively using a balanced primary and/or secondary coil system connected to a Stanford Research SR830 digital lock-in amplifier via an SR554 transformer preamplifier. A relatively low noise level is achieved by using this transformer preamplifier (SR554) to ensure good impedance matching. Each coil (main or compensating coil) has an inner diameter of ∼ 3.0 mm and contains a primary part (8 layers × 33 turns/layer) and a secondary part (8 layers × 33 turns/layer). The primary parts of the main coil and the compensating coil were connected in series with the same polarity, while the secondary parts of the main coil and the compensating coil were connected in series with the opposite polarity. The primary leads connect to the AC current source (Keithley 6221), and the secondary leads connect to the lock-in amplifier (SR830) via a pre-amplifier (SR554). The supplied AC current is ∼ 10 mA and 1023 Hz. In Fig. 1(a), a dummy BeCu gasket was placed inside the compensating coil for compensating the background of the gasket in the main coil. A tiny piece of NbTi superconductor was placed inside the compensating coil as a reference sample. The superconducting transition of the Ti sample is expected to occur with an opposite polarity than that of the reference sample. The DAC was placed inside a Montana closed-cycle cryostat for a cryogenic environment 4–300 K. Further experimental details of the high pressure and AC magnetic susceptibility techniques are published elsewhere.[5,6,13]

3. Experimental results and discussion

After initial pressurization to 10 GPa, the DAC was cooled to low temperatures to search for a superconducting transition, but none was detected above 4 K in the AC susceptibility at pressures of 38 GPa, 69 GPa, 85 GPa, or 90 GPa. In Fig. 2, data at 90 GPa is included for comparison, where we observed only the superconducting transitions of NbTi and Pb. A small piece of NbTi was placed inside the compensating coil as a reference for both transition size and polarity. The superconducting transition of Pb was unexpectedly detected from the solder, which could have been avoided by using a lead-free solder. Starting at 101 GPa, a superconducting transition of Ti emerges, with T_c rising monotonically with pressure, reaching 6.1 K at 120 GPa. The real part of the AC magnetic susceptibility decreases by approximately 250 nV upon cooling through the superconducting transition, consistent with bulk shielding. The much stronger superconducting signal observed compared to scandium (Sc) in Ref. [5] is attributed to the smaller coil diameter, higher number of coil turns, and larger sample size in this experiment. Note that we define T_c as the temperature at the onset of the diamagnetic transition.

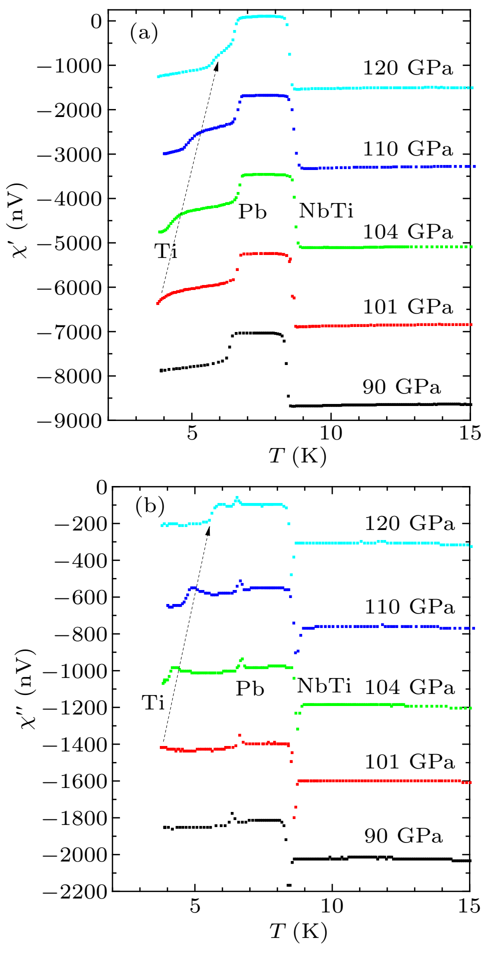

Fig. 2. Real part (a) and imaginary part (b) of the AC magnetic susceptibility signal versus temperature for Ti at different pressures ranging from 90 GPa to 120 GPa. Curves are shifted vertically for clarity. The superconducting transition temperature T_c, which is defined by the transition onset point, is seen to increase monotonically with pressure. Both real and imaginary parts give a consistent $T_c(P)$.

In Fig. 3, the dependence of T_c on pressure for Ti is shown from the present experiment to 120 GPa, indicating a monotonic increase with the slope of ~ 0.1 K/GPa. Compared with the previous high-pressure resistivity measurements on Ti,[10,11] the T_c values obtained from resistivity are higher than those from our AC susceptibility results, and the transition widths are also much broader. A small fraction of the sample becoming superconducting can cause a sharp drop in resistance, but bulk superconductivity typically occurs at lower temperatures. Generally, the onset of the superconducting transition in AC susceptibility aligns with the point where resistivity reaches zero.[6,19] Thus, the $T_c(P)$ data from our AC susceptibility measurements appear consistent with the $T_c^{\text{zero}}(P)$ data from resistivity measurements in previous studies,[10,11] as shown in Fig. 4.

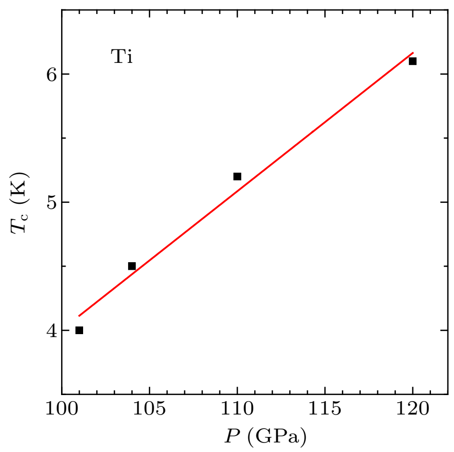

Fig. 3. Superconducting transition temperature T_c verse pressure to 120 GPa. Note that we define T_c as the temperature at the onset of the diamagnetic transition. The red line through data is a guide for the eye.

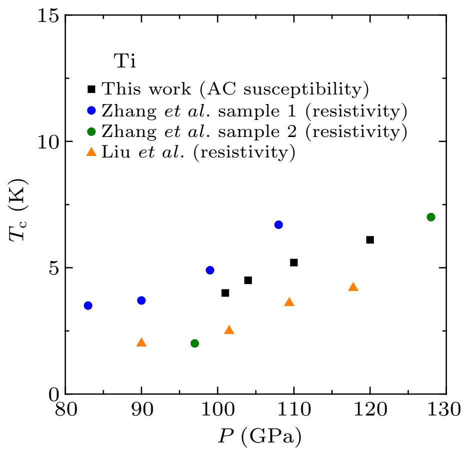

Fig. 4. Superconducting transition temperature T_c verse pressure in the range of 80–130 GPa. T_c determined by resistivity are extracted from the Refs. [10,11]. Note that T_c determined by AC susceptibility is at the onset of the diamagnetic transition, while T_c determined by resistivity is where resistivity reaches zero.

The superconductivity observed in the Ti-ω phase within the 100–120 GPa pressure range is considered as conventional phonon-mediated superconductivity, supported by electronic band structure and electron–phonon coupling calculations.[10,11] The steady increase with pressure in the Ti-ω phase below 120 GPa can be attributed to s–d electron transfer, which raises the d electron count and significantly enhances the electronic density of states at the Fermi energy.[20,21] Further AC susceptibility experiments will be conducted on Ti metal at pressures approaching 200 GPa to confirm bulk superconductivity in the Ti-γ and Ti-δ phases.[22,23] This will be particularly intriguing, as it has been suggested that electron–electron correlation effects, linked to the relatively narrow d bands in Ti, may be responsible for the substantial enhancement in the Ti-δ phase above 120 GPa.[10,11]

4. Conclusion

In summary, we performed high-pressure AC magnetic susceptibility measurements on elemental Ti up to 120 GPa in a diamond anvil cell and observed a robust diamagnetic signal, the Meissner effect, confirming the bulk nature of superconductivity in elemental Ti-ω phase.

Acknowledgements

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