Dependence of the superconducting transition temperature of MgB$_2$ on pressure to 20 GPa

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Abstract
The dependence of $T_c$ on nearly hydrostatic pressure has been measured for an isotopically pure (11B) MgB$_2$ sample in a helium-loaded diamond-anvil-cell to nearly 20 GPa. $T_c$ decreases monotonically with pressure from 39.1 K at ambient pressure to 20.9 K at 19.2 GPa. The initial dependence is the same as that obtained earlier ($dT_c/dP \approx -1.11(2)$ K/GPa) on the same sample in a He-gas apparatus to 0.7 GPa. The observed pressure dependence $T_c(P)$ to 20 GPa can be readily described in terms of simple lattice stiffening within standard phonon-mediated BCS superconductivity.

1 INTRODUCTION
The recent discovery [1] of superconductivity at high temperatures (40 K) in the $s,p$ metal compound MgB$_2$ was quite unexpected. To aid in the search for even higher values of $T_c$ in this class of superconductor, one would like to pinpoint both the pairing mechanism and the electronic/phononic features which lend this simple binary compound its extraordinarily high value of $T_c$. Of particular value in this regard are studies of the variation of the superconducting and normal-state properties under hydrostatic and uniaxial pressure since they can be carried out on a single sample. Whereas in transition-metal compounds the pressure-induced changes in $T_c$ can be quite complicated, in simple metal superconductors like Al, In, Sn, or Pb, $T_c$ invariably decreases with increasing hydrostatic pressure [2] due to lattice stiffening, electronic properties playing a subordinate role.

In all high-pressure experiments on MgB$_2$ known to us [3, 4, 5, 6, 7, 8, 9], $T_c$ is found to monotonically decrease under pressure, but the magnitude of $dT_c/dP$ varies considerably (see the Table below). Several studies have been carried out in the pressure range to 2 GPa: in ac susceptibility measurements in a piston-cylinder cell with liquid Fluorinert pressure medium, Lorenz et al. [3] obtain $dT_c/dP \approx -1.6$ K/GPa, whereas in electrical resistivity studies with similar pressure techniques,
Saito et al. [5] and Choi et al. [8] find -2.0 K/GPa and -1.36 K/GPa, respectively. Recent ac susceptibility [6] and neutron-diffraction [10] measurements in a He-gas apparatus on a high quality, isotopically pure (\textsuperscript{11}B) sample yield \(dT_c/dP \simeq -1.11(2)\) K/GPa and the bulk modulus \(B \simeq 147.2(7)\) GPa, respectively, giving the volume dependence \(d \ln T_c / d \ln V = B d \ln T_c / d P = +4.16(8)\); these authors suggest that the lower magnitude of \(dT_c/dP\) may result from the complete lack of shear-stress effects in the helium pressure medium. Good agreement with this result was obtained in subsequent He-gas studies by Lorenz et al. [7] using an improved sample, where \(dT_c/dP \simeq -1.07\) K/GPa; a remeasurement in He-gas of the sample studied earlier in Fluorinert yielded the comparable value \(dT_c/dP \simeq -1.45\) K/GPa, implying that differences among samples may affect \(dT_c/dP\) as much or more than shear stresses.

The first \(T_c(P)\) studies in the pressure range above 2 GPa were carried out by Monteverde et al. [4] to 25 GPa in electrical resistivity measurements using the solid pressure medium steatite in an opposed anvil cell. These authors report widely differing pressure dependences \(T_c(P)\) for three of the four samples prepared, the initial dependence \(dT_c/dP\) varying from -0.35 to -0.8 K/GPa which they attribute to lattice defects such as Mg nonstoichiometry. Very recently Tissen et al. [9] carried out ac susceptibility measurements in a diamond-anvil-cell (DAC) to 28 GPa with methanol-ethanol pressure medium and obtained the initial dependence \(dT_c/dP\) \(\simeq -2\) K/GPa, finding that \(T_c\) decreases to 11 K at 20 GPa and 6 K at 28 GPa, almost twice the decrease observed by Monteverde et al. [4]. Tissen et al. [9] also report a broad bump in the pressure dependence \(T_c(P)\) near 9 GPa which they speculate arises from an electronic Lifshitz transition. The results of all known \(T_c(P)\) measurements on MgB\(_2\) are summarized in the Table.

An excellent summary of high-pressure \(T_c(P)\) data on MgB\(_2\) and their interpretation is contained in a recent paper by Chen et al. [11]. In the present paper we determine \(T_c(P)\) in a DAC with dense helium pressure medium and find \(T_c\) to decrease monotonically with pressure from 39.1 K to 20.9 K at 19.2 GPa. The initial pressure dependence is in excellent agreement with that, \(dT_c/dP \simeq -1.11(2)\) K/GPa, determined in earlier He-gas measurements to 0.7 GPa on the same MgB\(_2\) sample.

\section{EXPERIMENT}

The powder sample of MgB\(_2\) for these studies is the same as used in previous \(T_c(P)\) [6] and neutron-diffraction [10] studies to 0.7 He-gas pressure and is made using isotopically-enriched \textsuperscript{11}B (Eagle Picher, 98.46 atomic % enrichment). A mixture of \textsuperscript{11}B powder (less than 200 mesh particle size) and chunks of Mg metal were reacted for 1.5 hours in a capped BN crucible at 800°C under an argon atmosphere of 50 bar. The resulting sample displays sharp superconducting transitions in the ac susceptibility with full shielding [6]; at ambient pressure the temperature of the superconducting onset lies at 39.25 K and the midpoint at 39.10 K. In this paper we define \(T_c\) at a given pressure from the superconducting midpoint.
$T_c(P)$ can be determined to very high pressures using a helium-loaded diamond-anvil-cell made of hardened Cu-Be alloy with binary Cu-Be inserts fitted with 1/6-carat 16-sided type Ia diamond anvils (Drukker) and 0.5 mm culet diameter. Gaskets of Ta$_{80}$W$_{20}$ alloy with diameter 2.7 mm and thickness 290 µm are preindented to about 70 µm. The MgB$_2$ sample ($80 \times 80 \times 25 \, \mu m^3$) together with several small ruby spheres (5-10 µm dia.) [12] are placed in a 240 µm dia. hole drilled through the center of the gasket. The pressure clamp is placed in a continuous flow cryostat and superfluid $^4$He is loaded into the gasket hole at 2 K to serve as pressure medium. The pressure in the gasket hole can be increased at any temperature at or below room temperature (RT) by loading a double-membrane [13] with a few bars of He gas to force the diamond-anvils together. Temperature is measured by calibrated Pt and Ge thermometers thermally anchored to the top diamond.

The pressure in the cell is determined to within 0.2 GPa at any temperature below room temperature (RT) by measuring the pressure-induced shift in the ruby R1 fluorescence line. A second ruby chip at ambient pressure located just outside the pressure cell allows the correction for the temperature shift of the ruby line. In the temperature range of this experiment (2 - 300 K) the pressure-dependent shift in the wavelength of the ruby R1 fluorescence line, $d\lambda/dP = 3.642 \, \AA/GPa$, is independent of temperature [14]. Upon cooling from room temperature, there is negligible change in pressure below 50 K; the pressure is normally measured at temperatures close to the temperature of MgB$_2$’s superconducting transition.

In the DAC the superconducting transition of the MgB$_2$ sample is determined inductively to ± 0.1 K using two balanced primary/secondary coil systems connected to a Stanford Research SR830 digital lock-in amplifier by slowly varying the temperature (∼ 0.5 K/min.) through the transition. The ac susceptibility studies were carried out using a 3 G (r.m.s.) magnetic field at 1000 Hz. Over the transition the signal changed by ∼ 5 nV with a background noise level of ∼ 0.2 nV, as seen in Fig. 1. Further details of the He-gas and DAC high-pressure techniques are given elsewhere [15].

3 RESULTS OF EXPERIMENT AND DISCUSSION

In Fig. 1 we display the temperature dependence of the ac susceptibility at three different pressures; the sharp superconducting transition is seen to shift to lower temperatures with pressure. $T_c$ versus pressure is plotted in Fig. 2 to 20 GPa, thus extending the pressure range of our previous He-gas studies on the same MgB$_2$ sample nearly thirtyfold. Within experimental error, $T_c$ decreases linearly with pressure to ∼ 10 GPa, consistent with the rate -1.1 K/GPa (dashed line) from the earlier He-gas studies [3]; the $T_c(P)$ data exhibit the upward (positive) curvature expected from increasing lattice stiffening at higher pressures. The decrease of $T_c$ with pressure is
much less than that obtained by Tissen et al. [9], but somewhat greater than the largest dependence obtained by Monteverde et al. [4]; in addition, we do not observe the bump in $T_c(P)$ near 9 GPa reported by Tissen et al. [9].

As seen in Fig. 2, the width of the superconducting transition generally increases from $\sim 0.3$ K for $P \leq 10$ GPa to 0.9 K at 17.8 GPa, increasing somewhat further for the data with decreasing pressure. This increase in width $\Delta T_c$ is usually, but not always, accompanied by a slight broadening of the ruby R1 fluorescence line; both broadening effects point to a pressure gradient of approximately $\pm 0.3$ GPa at the highest pressures. These broadening effects in $T_c$ are symptomatic of a small ($\pm 1.5\%$) pressure gradient in the cell arising from shear stresses in the solid helium pressure medium. Since at $T_c \approx 40$ K helium is fluid only for pressures $P \leq 0.5$ GPa, all $T_c(P)$ data in Fig. 2 were taken with the sample surrounded by solid helium. After the sample is cooled through the melting curve of helium [16], the differential thermal contraction of solid helium, sample, and pressure cell lead to small shear stresses and pressure gradients at temperatures near $T_c$. In the diamond-anvil-cell it is not possible to cool slowly through the melting curve of helium with a well-defined temperature gradient, like in the He-gas experiments [3]. In addition, since helium freezes at RT for $P \approx 12$ GPa, to increase the pressure above 12 GPa the diamonds must compress solid helium, a process which adds further shear stresses. The increase of the transition width with decreasing pressure (open circles in Fig. 2) is curious and may indicate that the sample and ruby spheres have come into direct contact with the gasket or diamond anvils.

At a given pressure, solid helium is the softest of all solids. Other pressure media, such as frozen Fluorinert, methanol-ethanol or solid steatite, will support much larger shear stresses and pressure gradients, with a potential broadening and shifting of the superconducting transition, particularly in elastically anisotropic samples such as MgB$_2$ where the compressibility along the $c$ axis is 64% larger than along the $a$ axis [10]. Shear stresses are known to influence the pressure dependence of $T_c$ in elastically anisotropic materials such as the high-$T_c$ oxides [17] or organic superconductors [18].

Since helium freezes for temperatures near $T_c \approx 40$ K at 0.5 GPa, the strictly linear pressure dependence of $T_c$ observed in the He-gas measurements to 0.7 GPa [3] or 0.8 GPa [4] shows that the very weak shear stresses in solid He have negligible influence on the value of $T_c$ in this material. The approximant agreement reported by Lorenz et al. [4] for the pressure dependences of the same sample in He-gas (-1.45 K/GPa) or frozen Fluorinert FC77 (-1.6 K/GPa) [3] speaks against large shear stress effects in the latter pressure medium, although the evidence here is not as compelling due to the absence of an error estimate for $dT_c/dP$ and lack of comparative data on a series of samples.

Substantially larger shear stresses are generated in the frozen pressure medium methanol-ethanol at very high pressures. At 20 GPa the width of the superconducting transition in the ac susceptibility data of Tissen et al. [3] has increased by $\sim 3$ K which corresponds to a pressure gradient at that pressure of $\Delta P \approx 3.5$ GPa or 18%, an order
of magnitude higher than in the present helium-loaded DAC measurements; a similar analysis of Tissen’s data near 9 GPa yields a pressure gradient of $\Delta P \approx 1$ GPa. It is conceivable that the shear stresses leading to these pressure gradients may be responsible for the bump in $T_c(P)$ near 9 GPa and perhaps also for the unusually large decrease in $T_c$ to 28 GPa found by these authors. Further experiments are required to establish whether or not the anomalous $T_c(P)$ dependence is reproducible. The same comment applies to the widely differing $T_c(P)$ results obtained by Monteverde et al. in their quasi-hydrostatic pressure measurements using the solid pressure medium steatite. In such a pressure cell the shear stresses are often very large and sufficient to crush dense samples or compact loosely sintered samples.

From a study of existing data, Tissen et al. have suggested that larger values of $|dT_c/dP|$ are obtained for samples exhibiting lower ambient-pressure values of $T_c$. The data in the Table below lend some support to this suggestion. However, $T_c$ values determined in ac susceptibility and electrical resistivity measurements are difficult to compare directly, the latter usually lying higher. Further experimentation under carefully controlled conditions is clearly necessary to investigate this possible correlation.

To compare the present experimental results with theory, it is advantageous to use the Murnaghan equation-of-state to convert the $T_c$ versus pressure $P$ data in Fig. 2 to $T_c$ versus relative volume $V/V_0$ data

$$\frac{V}{V_0} = \left[1 + \frac{B'P}{B}\right]^{-1/B'},$$

where we use the value $B = 147.2$ GPa from Ref. and the canonical value $B' \equiv dB/dP = 4$ supported by a recent calculation. The resulting dependence of $T_c$ on relative sample volume is shown in Fig. 3. The maximum pressure applied in the present experiment (19.2 GPa) results in a volume decrease of $\sim 10\%$.

We now compare the $T_c$ versus $V/V_0$ dependence in Fig. 3 to that from our earlier He-gas data on the same sample. As discussed in the Introduction, in the latter study we obtained the initial volume dependence $d\ln T_c/d\ln V = Bd\ln T_c/dP \approx +4.16$. Since the volume changes from the 0.7 GPa He-gas pressure are very small ($\sim \frac{1}{2} \%$), we can write $d\ln T_c/d\ln V \approx [\Delta T_c/T_{c0}]/[\Delta V/V_0]$ from which follows the linear relation

$$T_c \approx T_{c0}\left(-3.16 + 4.16\frac{V}{V_0}\right),$$

which is plotted as the dashed line in Fig. 3. Note that the experimental $T_c$ data at higher pressures now lie below the dashed line, the more so the higher the pressure. The application of the equation of state has transformed the superlinear $T_c$ versus $P$ data to sublinear $T_c$ versus $V/V_0$ data. The dashed line intercepts the horizontal axis ($T_c = 0$ K) at $V/V_0 = 0.76$ which corresponds to a critical pressure of $P_c \approx 75$ GPa, an upper bound for the actual $P_c$. 5
Another method to extrapolate the initial volume dependence $d \ln T_c / d \ln V \simeq +4.16$ to higher pressures would be to assume that this differential relation holds at all pressures, allowing us to integrate this expression to obtain

$$T_c = T_{c0} \left( \frac{V}{V_0} \right)^{+4.16}, \quad (3)$$

which is plotted as the upper solid line in Fig. 3. This volume dependence must, of course, agree with the He-gas data at low pressures, where $V/V_0 \simeq 1$, but rises well above the present experimental data at higher pressures, yielding a much less satisfactory agreement with the experimental data than the above linear approximation. Note that according to Eq. (3) $T_c$ only reaches 0 K at infinite pressure where $V \to 0$.

It is not surprising that $T_c$ is not a linear function of $V/V_0$ to very high pressures. The reason is that $T_c$ depends exponentially on many fundamental parameters and it is the relatively small changes in these parameters which lead to the large change in $T_c$ under pressure seen in Figs. 2 and 3. Consider the McMillan equation \[20\]

$$T_c \simeq \frac{\langle \omega \rangle}{1.20} \exp \left\{ \frac{-1.04(1 + \lambda)}{\lambda - \mu^*(1 + 0.62\lambda)} \right\}, \quad (4)$$

valid for strong coupling ($\lambda \lesssim 1.5$), which connects the value of $T_c$ with the electron-phonon coupling parameter $\lambda$, an average phonon frequency $\langle \omega \rangle$, and the Coulomb repulsion $\mu^*$. The coupling parameter is defined by $\lambda = N(E_f) \langle I^2 \rangle / [M \langle \omega^2 \rangle]$, where $N(E_f)$ is the electronic density of states at the Fermi energy, $\langle I^2 \rangle$ the average squared electronic matrix element, $M$ the molecular mass, and $\langle \omega^2 \rangle$ the average squared phonon frequency.

We now proceed to estimate the dependence of $T_c$ on relative volume $V/V_0$ by inserting into the McMillan equation the relatively small volume (pressure) dependences of each parameter $\langle \omega \rangle$, $\lambda$, and $\mu^*$ obtained from our previous analysis of the high-pressure data on MgB$_2$ to 0.7 GPa \[9\]. In our previous paper, we showed that the volume dependences $\gamma \equiv -d \ln \langle \omega \rangle / d \ln V = +2.36$ and $d \ln \lambda / d \ln V = +3.72$ gave a good account of the experimental data to 0.7 GPa; $\mu^* = 0.1$ was assumed to be independent of pressure, a quite good assumption \[11\]. As suggested in the analysis of Chen et al. \[11\], we now integrate the volume dependences of these two parameters to obtain

$$\langle \omega \rangle = \langle \omega \rangle_0 (V/V_0)^{-2.36} \quad \text{and} \quad \lambda = \lambda_0 (V/V_0)^{3.72}. \quad (5)$$

For the initial values of the parameters we use the logarithmically averaged phonon energy from inelastic neutron studies \[21\], $\langle \omega \rangle_0 = 670$ K and $\lambda_0 = 0.90$ from the McMillan equation. Inserting these two volume dependences in the McMillan equation, we obtain the dependence of $T_c$ on relative volume shown as the lower solid line in Fig. 3. The agreement with the experimental data is remarkable, much better than in the two previous approximations.
According to this estimate, approximately 50 GPa hydrostatic pressure would be required to drive $T_c$ to below 4 K. As in our earlier paper on the He-gas results to 0.7 GPa [6], the above analysis of the present $T_c(P)$ data to 20 GPa demonstrates that the monotonic decrease of $T_c$ with pressure arises predominantly from the decrease in $\lambda$ due to lattice stiffening ($d\ln \langle \omega^2 \rangle /d\ln V \simeq -2\gamma \simeq -4.72$), and not from electronic effects ($d\ln [N(E_f) \langle I^2 \rangle] /d\ln V \simeq -1$). A similar calculation was very recently carried out by Chen et al. [11] over a much wider pressure range; this paper also contains a detailed discussion of the pressures dependences of all parameters, including $\mu^*$.

The good agreement between the experimental data to 20 GPa and the predictions of the McMillan formula using the volume dependences determined from the He-gas data to 0.7 GPa lends additional evidence that superconductivity in MgB$_2$ originates from standard BCS phonon-mediated electron pairing.

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Summary of available high-pressure $T_c(P)$ data on MgB$_2$. $T_c$ values are at ambient pressure from superconducting midpoint in ac susceptibility $\chi_{ac}$ and electrical resistivity $\rho$ measurements. $dT_c/dP$ is initial pressure derivative. $P^{\text{max}}$(GPa) is the maximum pressure reached in experiment.

| $T_c$(K) | $dT_c/dP$ (K/GPa) | $P^{\text{max}}$(GPa) | measurement         | pressure medium | reference |
|---------|-------------------|------------------------|---------------------|-----------------|-----------|
| 39.1    | -1.1              | 19.2                   | $\chi_{ac}$, $^{11}$B isotope | helium          | present data |
| 39.1    | -1.11(2)          | 0.66                   | $\chi_{ac}$, $^{11}$B isotope | helium          | 1         |
| 39.1    | -1.09(4)          | 0.63                   | $\chi_{ac}$, $^{11}$B isotope | helium          | 22        |
| 39.2    | -1.11(3)          | 0.61                   | $\chi_{ac}$, $^{11}$B isotope | helium          | 22        |
| 40.5    | -1.12(3)          | 0.64                   | $\chi_{ac}$, $^{10}$B isotope | helium          | 22        |
| 39.2    | -1.07             | 0.84                   | $\chi_{ac}$         | helium          | 4         |
| 37.4    | -1.45             | 0.84                   | $\chi_{ac}$         | helium          | 4         |
| 37.4    | -1.6              | 1.84                   | $\chi_{ac}$         | Fluorinert FC77 | 3         |
| 37.3    | -2                | 27.8                   | $\chi_{ac}$         | 4:1 methanol-ethanol | 4         |
| 38.2    | -1.36             | 1.46                   | $\rho$              | 1:1 daphne-kerosene | 8         |
| 37.5    | -1.9              | 1.35                   | $\rho$              | Fluorinert FC70 | 3         |
| $\sim$35 | -0.35 to -0.8     | 25                     | $\rho$              | steatite, RT solid | 4         |
FIGURE CAPTIONS

Figure 1. Real part of the ac susceptibility versus temperature at three pressures.

Figure 2. Superconducting transition temperature (at midpoint) of MgB$_2$ versus pressure to 20 GPa from DAC measurements. Vertical error bars give width of transition; horizontal error bars give pressure gradient from broadening of ruby line. Data with filled circles (●) taken for monotonically increasing pressure, with open circles (○) for monotonically decreasing pressure. The straight dashed line has slope -1.11 K/GPa.

Figure 3. $T_c$ data from figure 2 plotted versus relative volume $V/V_0$. See text for explanation of solid and dashed lines.
Figure 1
Figure 2
Figure 3