

Evidence from High-Pressure Experiments that $\text{PrBa}_2\text{Cu}_3\text{O}_x$ is a Normal $\text{YBa}_2\text{Cu}_3\text{O}_x$ -like Oxide Superconductor

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Abstract

It has recently been shown that crystals of $\text{PrBa}_2\text{Cu}_3\text{O}_x$ grown by the traveling-solvent floating-zone (TSFZ) technique exhibit superconductivity at temperatures as high as 85 K, whereas flux-grown samples have traditionally been insulating. We discuss recent experiments which reveal an evident parallelism in the pressure dependence of the transition temperature $T_c(P)$ for superconducting $\text{PrBa}_2\text{Cu}_3\text{O}_x$ and $\text{YBa}_2\text{Cu}_3\text{O}_x$, thus giving evidence that TSFZ-grown $\text{PrBa}_2\text{Cu}_3\text{O}_x$ may be an ordinary member of the (rare-earth)- $\text{Ba}_2\text{Cu}_3\text{O}_x$ superconducting family.

1 Introduction

Shortly after the discovery of the high- T_c oxide superconductors it was noted that $\text{PrBa}_2\text{Cu}_3\text{O}_x$ appears to occupy a unique place among the rare-earth oxides [1, 2]. While most $\text{RBa}_2\text{Cu}_3\text{O}_x$ (R-123, R = rare earth) compounds exhibit superconductivity with maximum transition temperatures near $T_c \approx 92$ K, the isostructural compound Pr-123 was reported to be an insulator. A number of models invoking hole filling by $\text{Pr}^{(3+a)+}$ ions, magnetic pair breaking by localized Pr 4f-moments, and carrier-localization by the hybridization of Pr 4f- and O 2p-orbitals were proposed to explain this puzzling behavior [3, 4, 5, 6, 7, 8], but no single model has yet been able to give a consistent explanation of all experimental observations.

Very recently, Pr-123 single crystals prepared by the traveling-solvent floating-zone (TSFZ) technique [9, 10] and epitaxial thin films [11, 12] have been found to show bulk or granular superconductivity at temperatures as high as 92 K; this suggests that Pr-123 may be a normal member of the R-123 family of superconductors, since a maximum transition temperature near 92 K is characteristic for the two-layer superconductors in this family. A more critical test would be to measure T_c as a function of the oxygen content x for Pr-123 and determine whether $T_c(x)$ follows the highly nonlinear dependence characteristic for Y-123 which arises from oxygen ordering superstructures on the chain sites in the basal plane [13, 14]. Unfortunately, it has not yet been possible to synthesize high quality superconducting Pr-123 samples over a wide range of oxygen doping level x .

An equally meaningful way to test the possible equivalence of the superconducting state in Pr-123 and Y-123 is to compare the dependence of T_c on pressure over as wide a pressure range as possible. Previous high-pressure resistivity experiments [15] to 10 GPa on two underdoped TSFZ-grown Pr-123 single crystals with $T_c(1 \text{ bar}) \simeq 60$ K and 85 K revealed that T_c increases unusually rapidly with pressure, surpassing 100 K at 10 GPa in both cases, as seen in Fig. 1.

In this paper we compare these results on Pr-123 to those of very recent experiments on Y-123 where $T_c(P)$ was determined to 17 GPa over a wide range of oxygen content [16]. We find an evident parallelism in the pressure dependence $T_c(P)$ for Y-123 and Pr-123, giving evidence that the superconducting state in these two systems may have the same origin, i.e. Pr-123 may be just another R-123 superconductor.

2 Experiment

Pr-123 single crystals were grown by the TSFZ method, followed by an annealing treatment in an oxygen atmosphere. Predried Pr_6O_{11} , BaCO_3 , and CuO (99.99%) were used as starting materials. The crystals were grown in an infrared radiation furnace with four 300 kV halogen lamps as radiation source and under an atmosphere of argon with 0.1% oxygen gas flow. The details of the crystal growth are reported elsewhere [9, 17]. The superconducting orthorhombic Pr-123 crystals were prepared

by heating as-grown crystals in flowing pure oxygen gas at 850°C for 24 hours and then at 550°C for 77 hours. The sample used for the present high-pressure study is a small piece of a TSFZ-grown crystal with dimensions approximately $0.20 \times 0.20 \times 0.04$ mm³.

The structure of the sample was confirmed using an X-ray precession camera. Figures 2 (a) and (b) show the precession photographs of the $(hk0)$ and $(h0l)$ reflections of the sample. The orthorhombicity of the crystal can be recognized from the splitting of the $(hk0)$ reflections. The lattice parameters obtained are $a = 3.875(1)$ Å, $b = 3.938(1)$ Å, and $c = 11.808(3)$ Å. Very weak ring-like diffuse scattering is visible in the strong reflections of $(h0l)$, indicating the existence of stacking faults along the c -axis introduced during the single-crystal growth. This is consistent with the fact that the sample has a relatively large superconducting transition width (~ 20 K) in the ac susceptibility.

The chemical composition of the sample was determined from scanning electron microscope X-ray energy dispersion spectrum (SEM-EDS) analysis. The atomic ratio of Pr:Ba:Cu is found to be close to the 1:2:3 stoichiometry. The oxygen content of the sample is unknown, since the sample size is too small to allow an appropriate chemical analysis. The nominal oxygen content x for PrBa₂Cu₃O _{x} is estimated from the known $T_c(x)$ dependence for Y-123 at ambient pressure [13].

The He-gas pressure system (Harwood) used in this study is capable of generating hydrostatic pressures to 1.4 GPa. The CuBe pressure cell (Unipress) is inserted into a two-stage closed-cycle refrigerator (Leybold) which operates in the temperature range 2 - 320 K. The pressure in the cell is measured by a calibrated manganin gauge and can be changed at any temperature above the melting curve of the He pressure medium. The superconducting transition of the Pr-123 sample in the high pressure environment is measured by the ac susceptibility technique using a miniature primary/secondary coil system located inside the 7 mm I.D. bore of the pressure cell. In the present studies a magnetic field of 1.1 Oe (rms) at 507 Hz is applied along the short dimension of the sample ($H \parallel c$ -axis). For the present Pr-123 crystal the superconducting onset is located at $T_c \simeq 61.0$ K at ambient pressure. Further details of the experimental setup are given elsewhere [18].

3 Results and Discussion

We would now like to compare the pressure dependence of T_c for Pr-123 shown in Fig. 1 to that for Y-123 at similar doping levels. Fortunately, Y-123 is the most studied of all high- T_c oxides and its pressure dependence $T_c(P)$ has been determined over a wide range of pressure and oxygen content x [19, 20]. The most extensive high-pressure studies to date on Y-123 were carried out by Fietz et al. [21] to 1 GPa using a He-gas apparatus. These experiments reveal that dT_c/dP changes dramatically as a function not only of the oxygen content x , but also of the *temperature* at which the pressure is changed! In strongly underdoped Y-123 the (positive) pressure derivative

dT_c/dP is enhanced by more than an order of magnitude if the pressure is applied at room temperature (RT), as in the vast majority of high-pressure experiments to date [19, 20], rather than at low temperatures (≤ 90 K). This strong enhancement in $(dT_c/dP)_{RT}$ is believed to originate from the ordering of oxygen defects in the chain sites following the application of pressure at room temperature, an effect which is frozen out at sufficiently low temperatures for lack of oxygen mobility [21]. Such pressure-induced relaxation effects in T_c are not restricted to Y-123; for many high- T_c oxides the pressure dependence of T_c has been found to depend sensitively on the detailed pressure/temperature history of the sample [22].

To allow a meaningful comparison between different systems, it is essential to experimentally determine the *intrinsic* dependence of the superconducting transition temperature on pressure, $T_c^{intr}(P)$, by eliminating such relaxation effects. This can be accomplished by carrying out the entire high-pressure experiment at temperatures sufficiently low that the relaxation processes are frozen out; for Y-123 this means maintaining the sample at temperatures below 200 K throughout the entire experiment [21, 23]. In their extensive high-pressure studies on Y-123 at temperatures below 90 K, Fietz et al. [21] showed that the intrinsic value of the pressure derivative $(dT_c/dP)_{intr}$ changes markedly with oxygen content, increasing from +2 K/GPa for $x \simeq 6.4$ ($T_c \approx 20$ K) to +3 K/GPa for $x \simeq 6.5$ ($T_c \approx 40$ K) to +7 K/GPa for $x \simeq 6.7$ ($T_c \approx 60$ K) before falling to +3.5 K/GPa for $x \simeq 6.8$ ($T_c \approx 80$ K) and then to a value near zero for optimally doped $x \simeq 6.92$ ($T_c \simeq 92$ K). These values of $(dT_c/dP)_{intr}$ for $x \simeq 6.7$ and 6.8 agree reasonably well with those from Fig. 1 for $\text{PrBa}_2\text{Cu}_3\text{O}_{6.6}$ (+7.4 K/GPa) and $\text{PrBa}_2\text{Cu}_3\text{O}_{6.8}$ (+3.8 K/GPa) [24]. Before reaching any conclusions, however, we need to establish the importance of oxygen ordering effects in $T_c(P)$ for Pr-123. We return to this question below; first we consider studies on Y-123 to pressures well above 1 GPa.

In Fig. 3 we show the results of very recent experiments by Sadewasser et al. [16, 25], using both He-loaded diamond-anvil-cell and He-gas systems, which determine the intrinsic dependences $T_c^{intr}(P)$ to 17 GPa for a series of Y-123 crystals across the entire range of oxygen content from strongly underdoped to optimally doped, extending our earlier study [26]. One observes in this figure the same progression in the magnitude of the initial slope with increasing oxygen content x as discussed in the previous paragraph: $(dT_c/dP)_{intr}$ increases progressively for samples A (+2.0 K/GPa, $x \simeq 6.41$) \rightarrow B (+2.1 K/GPa, $x \simeq 6.41$) \rightarrow C (+2.3 K/GPa, $x \simeq 6.45$), becoming very large for sample D (+7.1 K/GPa, $x \simeq 6.66$), but decreases again for the optimally doped sample E (+0.24 K/GPa, $x \simeq 6.92$) [16, 25]. These values of the intrinsic initial slopes $(dT_c/dP)_{intr}$ for Y-123 as a function of the oxygen content x are in good agreement with those from the earlier studies of Fietz et al. [21] listed in the preceding paragraph.

The present data in Fig. 3, however, contain a great deal more information than the above studies to 1 GPa since they were carried out to much higher pressures. The progression in $T_c^{intr}(P)$ with oxygen content is quite interesting. The higher the initial

slope $(dT_c/dP)_{intr}$, the larger the transition temperature before it passes through a maximum and the higher the value of the pressure $P(T_c^{\max})$ at this maximum. Note that for the optimally doped sample E both the initial slope $(dT_c/dP)_{intr}$ and the pressure $P(T_c^{\max})$ are nearly zero. The progression of $T_c^{intr}(P, x)$ for Y-123 with varying oxygen content x is quite complex and thus a good fingerprint to help identify superconductors with similar electronic and structural elements. Non R-123 oxide superconductors generally display a simpler behavior [19, 20]. The complexity in $T_c^{intr}(P, x)$ for Y-123 is likely a result of the presence of both chain and planar elements in its structure.

The $T_c(P)$ data for Pr-123 in Fig. 1 were obtained by applying the pressure at ambient temperature. For comparison with the intrinsic data $T_c^{intr}(P)$ on Y-123 in Fig. 3, we must determine the pressure dependence of T_c in Pr-123 free from relaxation effects. The most satisfactory way to do this would be to carry out the experiments entirely at low temperatures, as was done for Y-123. Only a single Pr-123 sample with $T_c(1 \text{ bar}) \simeq 61 \text{ K}$ (ac susceptibility onset) was available for the present study; this value of $T_c(1 \text{ bar})$ is close to that observed for $\text{PrBa}_2\text{Cu}_3\text{O}_{6.6}$ in Fig. 1. Experiments on this sample to pressures of 10 GPa and beyond are not possible in our diamond-anvil cell due to its limited accuracy and the relatively large width of the superconducting transition ($\sim 20 \text{ K}$). However, we can accurately determine the relative importance of relaxation effects in $T_c(P)$ for this sample to 1 GPa by measuring the ac susceptibility to very high accuracy in our He-gas high-pressure apparatus.

In this He-gas experiment the superconducting onset at ambient pressure was first measured to be $T_c(1 \text{ bar}) \simeq 61 \text{ K}$ (pt. 1 in Fig. 4). A hydrostatic pressure of 0.8 GPa was then applied and held at 298 K for 2 hours before cooling to measure $T_c(0.72 \text{ GPa}) \simeq 65.0 \text{ K}$ (pt. 2); the reduction in the pressure from 0.8 to 0.72 GPa upon cooling arises from the contraction of the He pressure medium. The pressure was then fully released at low temperatures ($\leq 60 \text{ K}$) and T_c remeasured (pt. 3); T_c is seen to fully return to its initial value at ambient pressure. Since the pressure dependence $T_c(P)$ is the same whether the pressure is changed at ambient or low temperatures, we conclude that there are no measurable relaxation effects for this Pr-123 sample. The value of the intrinsic pressure derivative is large, $(dT_c/dP)_{intr} \simeq +5.5 \pm 0.6 \text{ K/GPa}$, although somewhat less than the $\sim +7 \text{ K/GPa}$ observed for the TSFZ-grown $\text{PrBa}_2\text{Cu}_3\text{O}_{6.6}$ sample or for the Y-123 sample D with comparable ambient pressure transition temperature $T_c(1 \text{ bar}) \approx 60 \text{ K}$. It is thus not unreasonable to assume that relaxation processes do not make an important contribution to the $T_c(P)$ -dependence to 10 GPa in Fig. 1 for the $\text{PrBa}_2\text{Cu}_3\text{O}_{6.6}$ sample; our previous experiments on Y-123 [16, 23] and other oxide superconductors [25, 27] have shown that relaxation effects, if present, diminish in importance at higher pressures due to an exponentially rapid increase in the relaxation time τ with pressure. Comparing now the data for $\text{PrBa}_2\text{Cu}_3\text{O}_{6.6}$ in Fig. 1 with those for Y-123 sample D in Fig. 3, we see that $T_c(P)$ shows a clear resemblance: T_c increases rapidly with pressure by more than +7

K/GPa for both samples, surpassing 100 K above 10 GPa, an increase of more than 35 K! The greater increase in T_c to 10 GPa for PrBa₂Cu₃O_{6.6} compared to Y-123 sample D (47 K versus 37 K) is consistent with the somewhat larger value of dT_c/dP for the former (+7.4 K/GPa versus +7.1 K/GPa).

The importance of relaxation effects for more strongly doped (larger oxygen content x) Pr-123 samples, like PrBa₂Cu₃O_{6.8} with $T_c(1 \text{ bar}) \approx 80 - 85$ K, has not yet been determined. However, Fietz et al. [21] have shown that relaxation effects in Y-123 tend to diminish with increasing oxygen content, particularly near optimal doping. Indeed, the optimally doped Y-123 sample E in Fig. 3 shows no relaxation effects in T_c whatsoever [16]. In view of the absence of relaxation effects for the Pr-123 sample in Fig. 4, it is reasonable to assume that they play no important role for the more highly doped PrBa₂Cu₃O_{6.8} sample. It was pointed out above that the initial slope dT_c/dP for PrBa₂Cu₃O_{6.8} fits in well with the detailed systematics for Y-123 with varying oxygen content established by Fietz et al. [21]. Although there is no Y-123 sample in Fig. 3 with a value of T_c near that (~ 80 K) for PrBa₂Cu₃O_{6.8}, it is evident that the latter exhibits a $T_c(P)$ dependence which is intermediate between that of the Y-123 samples D and E in Fig. 3. The $T_c(P)$ -dependences to very high pressures for the two Pr-123 samples in Fig. 1 are thus in good agreement with the systematics for comparably doped Y-123. To critically test these apparent systematics, the $T_c(P, x)$ -dependences for Pr-123 to pressures well beyond 10 GPa should be determined over a wide range of oxygen content x ; in addition, relaxation effects should be eliminated by carrying out the experiments entirely at low temperatures, as was done for the data on Y-123 in Fig. 3.

In view of the importance of oxygen ordering phenomena in underdoped Y-123, their absence for the Pr-123 sample studied here (Fig. 4) is a surprising result. A parallel experiment in the He-gas system to 0.8 GPa on the Y-123 sample D revealed that relaxation effects increase the initial slope dT_c/dP by approximately 15% [16]. The absence of relaxation phenomena in the Pr-123 sample may be due to the reduced mobility of oxygen defects from enhanced disorder and/or strain in this system. Indeed, as evidenced by the broad (~ 20 K) superconducting transition, the inhomogeneity in the TSFZ-grown Pr-123 samples is quite large. The TSFZ-synthesis technique itself may also inhibit the relaxation phenomena in other ways. We note that enhanced disorder associated with extensive cation substitution in the 123 superconductor (La_{1-x}Ca_x)(Ba_{1.75-x}La_{0.25+x})Cu₃O_y may be the cause of the very weak relaxation effects observed in this system [28].

In summary, the pressure dependence $T_c(P)$ to 10 GPa for two TSFZ-grown Pr-123 samples is seen to change sensitively with the oxygen content in a manner which parallels that observed for the canonical R-123 system YBa₂Cu₃O_x over a wide range of oxygen content x . This supports the picture that the electronic and structural properties responsible for the superconductivity are very similar in both systems. The present experiments thus give evidence that TSFZ-grown Pr-123 may be just another member of the R-123 series of superconductors. Further experiments on

both TSFZ- and flux-grown Pr-123 samples over a wide range of oxygen content would be highly desirable to bring final clarity to this question.

Acknowledgments

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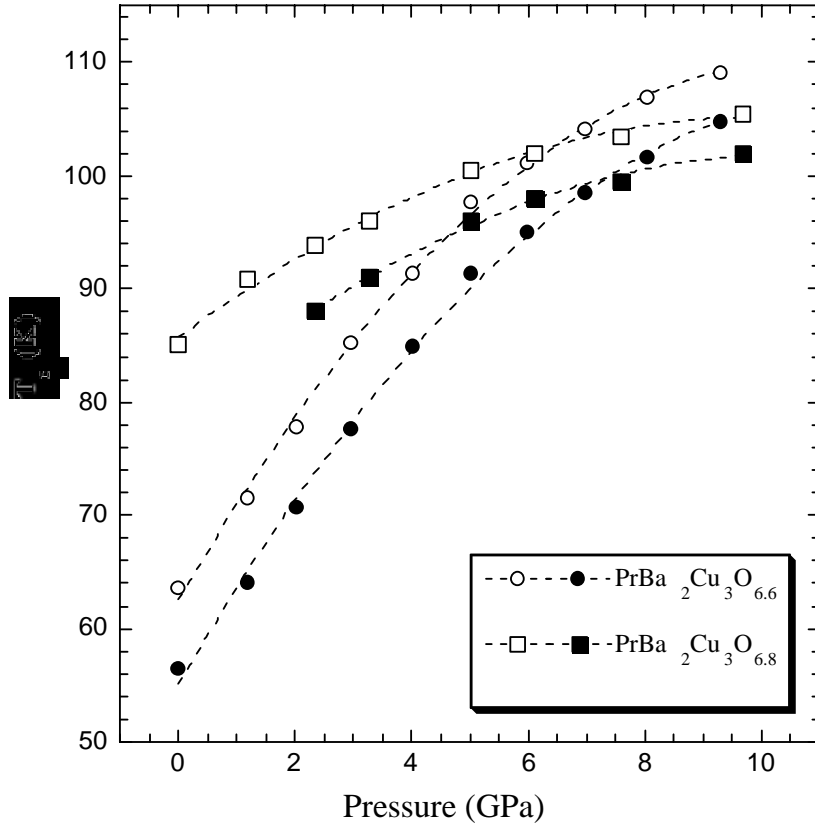


Fig. 1. Pressure dependence of T_c for TSFZ-grown $\text{PrBa}_2\text{Cu}_3\text{O}_x$ single crystals. Open and solid symbols represent temperatures of the resistive onset and zero-resistance, respectively. Oxygen contents x are nominal (see text). The initial pressure derivative dT_c/dP from Ref. 15 is $+7.4$ K/GPa for $\text{PrBa}_2\text{Cu}_3\text{O}_{6.6}$ and $+3.8$ K/GPa for $\text{PrBa}_2\text{Cu}_3\text{O}_{6.8}$.

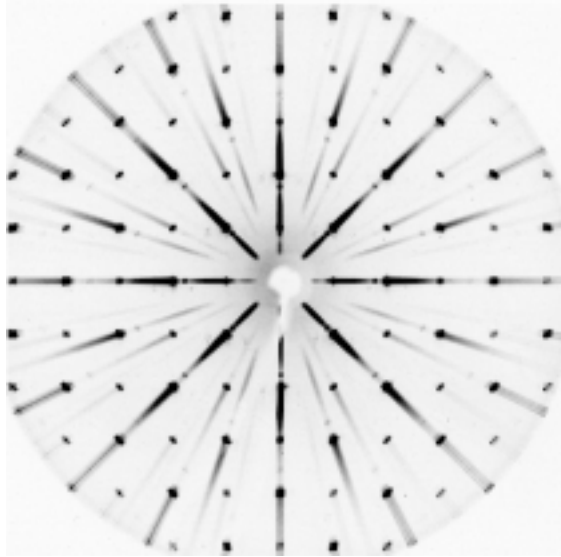


Fig. 2(a). Zero layer X-ray precession photographs of the $\text{PrBa}_2\text{Cu}_3\text{O}_x$ single crystal in $[001]$ zone axis.

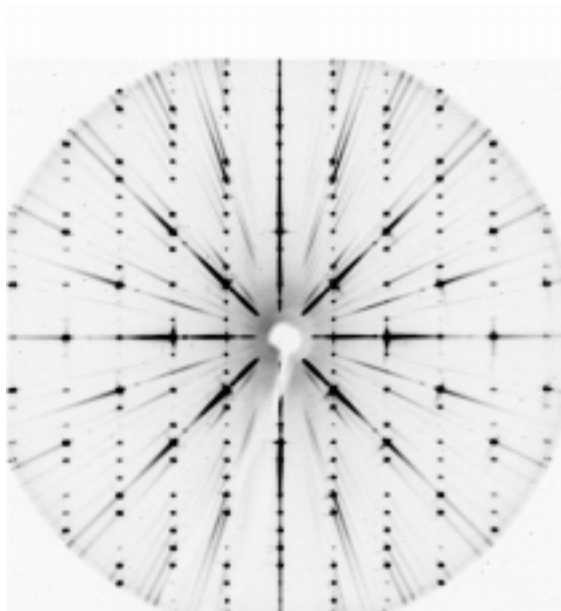


Fig. 2(b). Zero layer X-ray precession photographs of the $\text{PrBa}_2\text{Cu}_3\text{O}_x$ single crystal in $[010]$ zone axis.

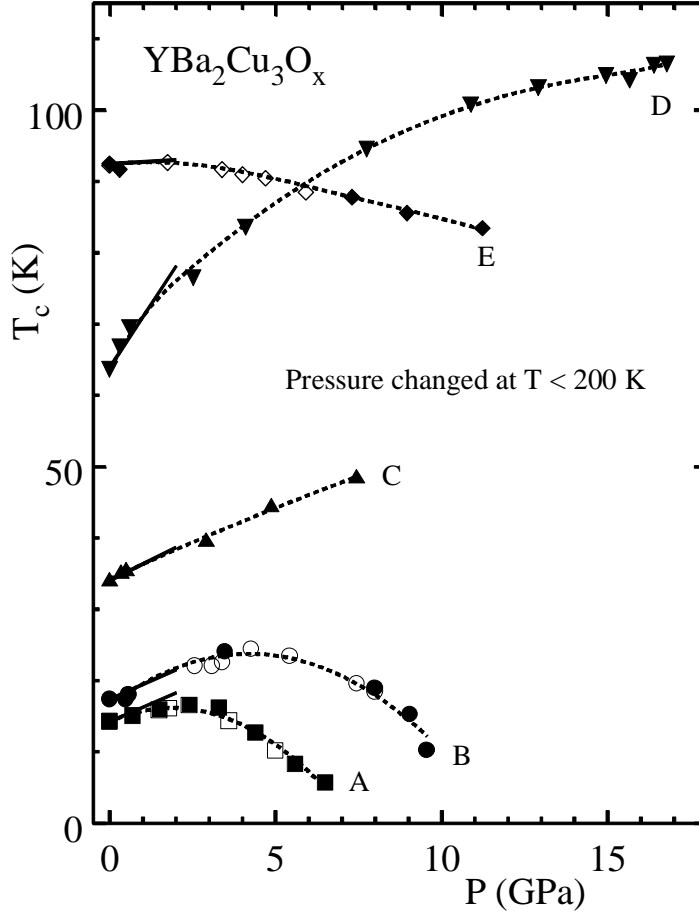


Fig. 3. Intrinsic pressure dependence $T_c^{intr}(P)$ from Ref. 16 for several $\text{YBa}_2\text{Cu}_3\text{O}_x$ crystals to 17 GPa. Solid (open) symbols indicate increasing (decreasing) pressure. The initial slopes from the He-gas data (see Ref. 18) to 1 GPa are given by the straight lines (see text for values). Dashed lines are guides to the eye.

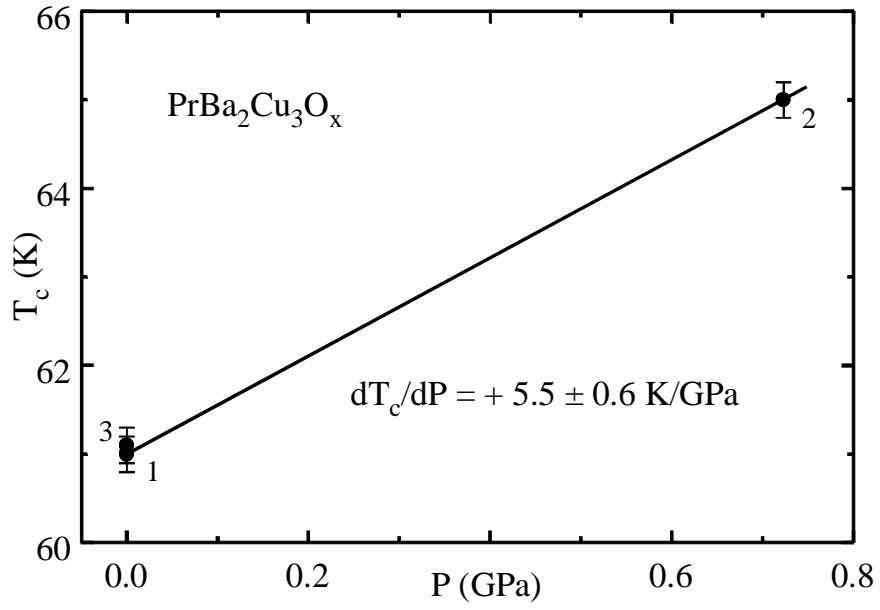


Fig. 4. Dependence of T_c on pressure for TSFZ-grown $\text{PrBa}_2\text{Cu}_3\text{O}_x$ single crystal. Values of pressure given were determined at temperatures near T_c . Numbers give order of measurement.