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Study of Niobium Thin Films under Pressure

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Niobium is widely used in many important superconducting applications. At ambient pressure, bulk Nb has the highest critical temperature, $T_c \approx 9.25$ K among the superconducting elements. Thin films of Nb show several differences in behavior in comparison with bulk Nb, e.g. substantial increase in the upper critical field (H_{C2}). Critical temperature of superconducting transition is usually lower for thin films than in bulk sample and depends on thickness of the film, size of grains etc. We prepared 100 nm thick niobium thin films in the high vacuum DC magnetron sputtering system, with $T_c = 8.95$ K at ambient pressure. In this study, we performed measurements of superconducting transition temperature by electrical resistivity measurements of Nb thin film under hydrostatic pressure of up to 30 kbar. We observed an increase of T_c with increasing value of pressure ($\mathrm{d}T_c/\mathrm{d}p = 7.3$ mK/kbar). On the other side in the case of bulk sample of Nb we observed a decrease of T_c value ($\mathrm{d}T_c/\mathrm{d}p = -2.5$ mK/kbar) with increasing applied pressure. Difference in superconducting properties between niobium bulk and thin film under pressure is discussed.

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1. Introduction

In past decades the substantial progress was achieved in preparation of well defined thin films and also in high pressure techniques. The superconductivity of bulk samples under pressure has been already studied in most of elements and many of compounds. However the influence of pressure on superconducting properties of thin films is still not a very well explored field of solid state physics. At ambient pressure Nb has the highest critical temperature among the elemental superconductors about 9.25 K [1]. Niobium belongs to *d*-band superconductors with *bcc* structure. Measurements of superconducting T_c of bulk Nb samples have been already preformed up to 132 GPa using highly sensitive magnetic susceptibility technique with diamond anvil cell [2]. The measured pressure dependence of T_c provided evidence for electronic topological transitions in bulk Nb at 5 GPa and around 60 - 70 GPa. Above 70 GPa authors observed steep decrease of T_c, which was explained by the Fermi level moving to the low density of states region [2]. Thin films of Nb have been very intensively studied by S. Bose and coworkers in several papers [3–5]. They studied e.g. the influence on superconducting properties of the thickness of thin film, size of grains and other.

2. Experimental details

Thin films of Nb have been prepared by DC magnetron sputtering system (AJA International, USA) in the high vacuum. As a substrate for deposition we used the glass microscope cover slides, which were sonicated in acetone and isopropylalcohol prior to film deposition. We have chosen optimal sputtering conditions to consistently produce films with the highest T_c and similar grain size for the 100 nm film thickness. Typical size of grains was few nms width and ~ 60 nm long [6]. Thickness of thin film and size of grains were chosen in order to have properties as similar as possible to bulk sample. Grains with size smaller than 8 nm are not superconducting due to quantum size effect (QSE) [7]. The residual resistivity ratio (RRR) of the thin film was around 3, which is in good agreement with reports from other research groups [4]. In case of bulk polycrystalline sample the RRR value was about 15. The electrical contacts on thin film have been realized by four golden wires bonded by ultrasonic contact bonder. Sensitive ac-resistance measurements under hydrostatic pressure up to 30 kbar were carried out in a piston-cylinder pressure cell. As a manometer we used Pb with $T_c = 7.19$ K. The temperature and magnetic field dependences of resistance at various pressures were performed between 1.6 and 300 K and in magnetic fields up to 4 T. Magnetic field was oriented perpendicular to thin film surface.

3. Results and discussion

Figure 1 shows pressure dependence of superconducting transition for bulk sample of Nb. The width of transition is around 200 mK. The T_c was estimated as temperature at which the resistance dropped to 50% of its normal state value (the solid line in Fig. 1). In previous work [8], the measurements of critical temperature T_c under pressure were performed using samples with very high RRR (from 500 to 11000) and with very narrow transition (from 3 to 38 mK). In our measurements the pressure dependence of superconducting transition temperature T_c was found to be $\mathrm{d}T_c/\mathrm{d}p = -2.5$ mK/kbar for bulk sample, which is in good agreement with result of T.F. Smith [8] ($dT_c/dp = -2.5$ mK/kbar). In case of thin film of Nb we observed substantially different behavior of T_c under pressure. Figure 2 shows pressure dependence of superconducting T_c for thin film of Nb. The

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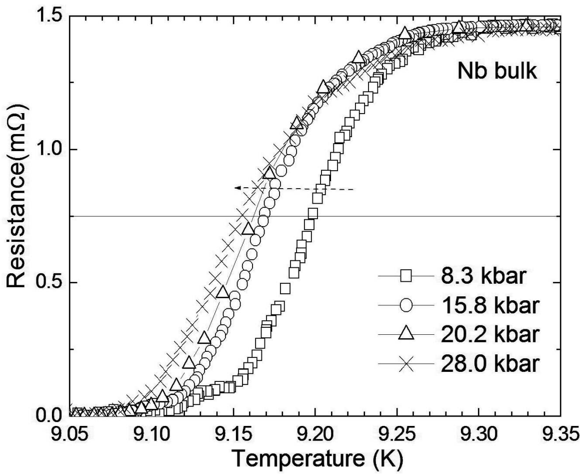

Fig. 1. Temperature dependence of resistance at various pressures for bulk Nb sample.

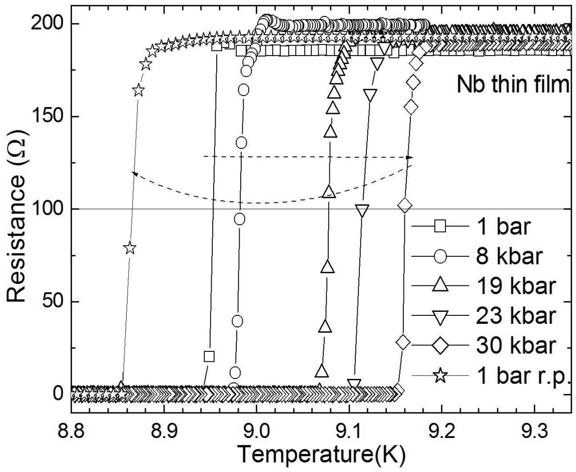

Fig. 2. Temperature dependence of resistance at various pressures for thin film of Nb. Measurement of R vs. T at ambient pressure after releasing of pressure is shown by stars (1 bar r. p.).

criterion for estimation of T_c was the same as in previous case. The width of transition is very narrow (about 30 mK), which points to high purity of sample and also to high homogeneity of the applied pressure. The value of $T_c = 8.95$ K at ambient pressure is lower than in case of bulk sample ($T_c = 9.25$ K) [1, 8]. With increasing pressure, the superconducting T_c increases with the rate of $dT_c/dp = 7.3$ mK/kbar. At maximum pressure of 30 kbar the value of the achieved superconducting transition temperature was 9.15 K, which is close to value of bulk sample at ambient pressure and even higher than for bulk sample at the same pressure.

We suppose, that by application of pressure, the strains induced in thin film during sputtering are released and the properties of thin film get closer to those of bulk sample. After releasing the pressure we measured the superconducting transition again at ambient pressure (see Fig. 2) and surprisingly the T_c was lower than for "as-prepared" thin film. It will be important to compare microstructure of thin film before and after application of pressure.

Additional measurements under higher pressure are needed to see whether the increase of T_c in case of thin film will continue or whether the behavior of T_c will follow the behavior of the bulk sample.

4. Conclusions

We performed measurements of superconducting transition temperature by electrical resistivity measurements of Nb thin film as well as the bulk sample under hydrostatic pressure up to 30 kbar. We prepared 100 nm thick niobium thin films, in the high vacuum DC magnetron sputtering system, with $T_c = 8.95$ K at ambient pressure. We observed an increase of T_c with increasing value of pressure ($\mathrm{d}T_c/\mathrm{d}p = 7.3$ mK/kbar). On the other side in the case of bulk sample of Nb we observed a decrease of $T_c = 9.25$ K value ($\mathrm{d}T_c/\mathrm{d}p = -2.5$ mK/kbar) with the increasing applied pressure. The difference in behavior of thin film and bulk sample comes from the changes of microstructure of the thin film. Measurements under higher pressures are needed.

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References

[1] D.K. Finnemore, T.F. Stromberg, C.A. Swenson, *Phys. Rev.* **149**, 231 (1966).

[2] V.V. Struzhkin, Y.A. Timofeev, R.J. Hemley, H. Mao, *Phys. Rev. Lett.* **79**, 4262 (1997).

[3] S. Bose, P. Raychaudhuri, R. Banerjee, P. Vasa, P. Ayyub, *Phys. Rev. Lett.* **95**, 147003 (2005).

[4] S. Bose, R. Banerjee, A. Genc, P. Raychaudhuri, H.L. Fraser, P. Ayyub, *J. Phys.: Condens. Matter* **18**, 4553 (2006).

[5] S. Bose, P. Raychaudhuri, R. Banerjee, P. Ayyub, *Phys. Rev. B* **74**, 224502 (2006).

[6] G. Pristáš, S. Gabáni, E. Gažo, V. Komanický, M. Orendáč, H. You, *Thin Solid Films* **556**, 470 (2014).

[7] S. Bose, Ch. Galande, S.P. Chockalingam, R. Banerjee, P. Raychaudhuri, P. Ayyub, *J. Phys.: Condens. Matter* **21**, 205702 (2009).

[8] T.F. Smith, *Phys. Lett. A* **33**, 465 (1970).