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A Cell for the Uniaxial Compression of Samples in μ SR Experiments

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Abstract—A cell for the uniaxial compression of crystals at pressures of up to 5 kbar and used in μ SR experiments to investigate aluminum acceptor impurities in silicon is described. The dimensions of the samples under study were $\sim 9.5 \times 9.5 \times 20$ mm. The pressure was monitored with calibrated resistor strain gauges.

Solutions to the problems that frequently arise in condensed matter physics require that the properties of substances exposed to pressure be studied. For example, the possibility of constructing a model quantum computer based on a uniaxially stressed silicon crystal doped with an acceptor or donor impurity is currently being discussed in the literature [1]. This correspondingly requires that the behavior of these impurities be studied in a silicon crystal exposed to different stresses.

This work describes an autonomous cell for applying uniaxial compression to crystals at pressures of up to 5 kbar, thereby allowing samples to be studied in polarized muon beams (the μ SR method [2]). The cell was successfully used in experiments to study the interaction of an Al acceptor impurity in silicon [3] using a beam of polarized negative muons. These (μ SR) experiments were performed in an external magnetic field and over a wide range of changes in the sample temperature (4.2–390 K). The diameter of the muon beams produced in intermediate-energy accelerators is ~ 1.5 cm. The optimum sample volume for MSR experiments is therefore 2–3 cm³. This volume is considerable, compared to that of the samples of several cubic millimeters that suffice, e.g., for EPR experiments [4]. When developing a cell that holds a sample in a stressed state under conditions of a limited space in a helium cryostat, a compromise solution is needed that provides the following: a sample volume that ensures a sufficient number of muon stops; sufficiently high structural strength of the cell withstanding the operating pressure; and a minimal amount of foreign matter on the path of the muon beam. The last is decisive in reducing the ratio of the number of background events (from the cell's walls) to the number of valid events (from the sample). In addition, the cell must be made of a nonmagnetic material.

A high-pressure μ SR spectrometer has already been used with beams of positive muons (e.g., in [5, 6], in

which a uniform pressure of up to 1.4 GPa was created using helium in a cell mounted inside a closed cycle refrigerator). It should be noted that experiments in beams of negative muons are more labor-intensive than those with positive muons, for the following reasons: (1) as a rule, the intensities of μ^- beams are substantially lower than the intensities of μ^+ beams; (2) the competing processes of nuclear capture of negative muons appreciably reduce the number of detected $\mu^- \rightarrow e^-$ decay events; and (3) a considerable loss in the muon polarization is observed (up to 1/6 of the initial polarization level, in the best case). Under such conditions, the requirement that the background-to-effect ratio be minimized is even more important.

Figure 1 shows a schematic of the cell. All of its elements were manufactured of Bp2 beryllium bronze, heat treated to a hardness HRC 34. The samples studied were silicon prisms 18–22 mm high having a base cross section of 9.5×9.5 mm² and crystallographic axes oriented differently to the prism bases. During the manufacturing of the cell, special attention was given to the parallelism of the planes of the cell elements, which are all perpendicular to the direction of the applied axial pressure. For example, the deviation in the parallelism of the prismatic silicon samples' base planes was ~ 5 μ m.

The required pressure was produced by a press at room temperature. For this purpose, cell body 3 was fixed in a slab rigidly linked to the press using an outer thread. Rod 1 of the press applied the required pressure through a hole in fixing screw 2 to pressure bearing 4 and then, through plunger 5 and sample bearing 6, the pressure was transferred to the sample. The final position of pressure bearing 4 was fixed by screw 2. The cell was attached to a cooled rod in the cryostat using the inner thread of fixing screw 2. Window 8 with dimensions of 14×25 mm to allow the passage of the muon beam ensured quite a large gap between the sample and

Fig. 1. Cell for uniaxial compression of samples: (1) forcing rod of the press; (2) fixing screw; (3) cell body; (4) pressure bearing; (5) plunger; (6, 9) sample bearings; (7) sample; (8) window for a muon beam; and (10) screw support.

the cell's bearing wall to minimize the number of background events.

The moment after the external pressure is removed, the pressing force of screw 2 can be reduced. When the cell is cooled, the pressure applied to the sample can change because of unequal changes in the linear dimensions of the cell and sample materials. Since the entire cell was manufactured of the same material and the linear thermal expansion coefficient for bronze is higher than that for silicon by one order of magnitude, no decrease in the sample compression was expected upon its cooling to liquid helium temperatures. To avoid any uncertainty between the applied initial load and the pressure developed in the sample after the load was removed and during sample cooling, the pressure in the sample was monitored with an EB001 П1-5-350Б resistor strain gage (Research Institute for Studies in Physics, Penza). The gage was glued on one side of the silicon sample with БФ-2 adhesive, which was then subjected to high-temperature polymerization. The strain gage was calibrated at room temperature as the sample was exposed to changing pressure. Figure 2 presents such a calibration curve for a resistor strain gage ($R_0 = 349.14 \, \Omega$ and $K = 2.07$, where R_0 is the resistance

Fig. 2. Pressure dependence of the strain gage resistance at room temperature (dotted line shows the mean regression).

of the strain gage with no deformation and K is the tensosensitivity coefficient). The main error in calibration came from measuring the pressure produced by the press and was $<3\%$.

During the subsequent cooling of the sample, the readings of the resistor strain gage could only change due to temperature-induced changes in the linear dimensions of the cell's structural elements, the sample itself, or the strain gage material and its ohmic resistance. In order to compensate for these changes, we recorded the readings of the strain gage stuck to the sample as a function of the temperature (in the operating range 10–60 K) with and without pressure. Figure 3 shows such dependences for the same sample as in Fig. 2 obtained without pressure ($P = 0$) and with pressure ($P = 1.7$ kbar) applied to it. As can be seen, there is a constant difference of $1 \, \Omega$ for the temperature range of 10–60 K (this difference is $0.84 \, \Omega$ at room temperature) between the readings of the resistor strain gage for the sample with and without pressure. Assuming that the contribution of temperature effects is independent of the pressure, we can state that the initial compression of the sample achieved at room temperature was not only maintained during its cooling, but additional compression was also observed. Assuming that the coeffi-

Fig. 3. Temperature dependences of the strain gage resistance at two pressure values $P = 0$ and 1.7 kbar (lines show the mean regressions).

cient of pressure-induced changes in the strain gage resistance [7] is $\Delta R/(R_0 \Delta P) = 0.84/(350 \times 1.7) = 1.4 \times 10^{-3} \text{ kbar}^{-1}$, we find that the sample can be additionally compressed at low temperatures by $\sim 0.3 \text{ kbar}$.

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REFERENCES

1. Belita, K., Xuedong, Hu, and Das Sarma, S., *Phys. Rev. B*, 2002, vol. 66, no. 11, p. 115201.
2. Schenck, A., *Muon Spin Rotation Spectroscopy*, Bristol: Adam Higler Ltd, 1985.
3. Mamedov, T.N., Andreika, D., Andrianov, D.G., *et al.*, *Pis'ma Zh. Eksp. Teor. Fiz.*, 2004, vol. 80, no. 5, p. 385 [*J. Exp. Theor. Phys. (Engl. Transl.)*, 2004, vol. 80, no. 5, p. 339].
4. Van Duijn-Arnold, A., Mol, J., Verberk, R., *et al.*, *Phys.Rev. B*, 1999, vol. 60, no. 23, p. 15829.
5. Butz, T., Kalvius, G.M., Lindgren, B., *et al.*, *Hyperfine Interact.*, 1986, vol. 32, p. 881.
6. Kratzer, A., Mutzbauer, K., Henneberger, S., *et al.*, *Hyperfine Interact.*, 1994, vol. 87, p. 1055.
7. Tsiklis, D.S., *Tekhnika fiziko-khimicheskikh issledovaniy pri vysokikh i sverkhvysokikh davleniyakh* Moscow: Khimiya, 1976. Translated under the title *Handbook of Techniques of High-Pressure Research and Engineering*, New York: Plenum Press, 1968.