Method for elastic uniaxial stress of single crystals: description and applications

N Ya Minina¹, N B Brandt¹, A M Savin² and E V Bogdanov¹
¹Physics Department, Moscow State University, 119992 Moscow, Russia
²Low Temperature Laboratory, Helsinki University of Technology, FIN-02015 TKK, Finland
E-mail: min@mig.phys.msu.ru

Abstract. The original method for a strong elastic uniaxial compression of single crystals up to 5 \( \div \) 6 kbar, calibration procedure and its applications for semimetal and semiconductor samples are described in details for the first time. This method simplifies sample installation and adjustment; it prevents the frequent destruction of samples, which often takes place in conventional methods of uniaxial compression between two anvils.

1. Introduction
The well-known phrase “uniaxial stress breaks crystal symmetry and samples” combines both the advantage and disadvantage of this kind of strong external influence. The latter one causes the relatively rare use of the uniaxial stress technique in physical experiments. For the sake of avoiding a destructive shear stress, a sample between two anvils should have faces that are perfectly flat and parallel in addition to being placed strictly along the stress direction. In this paper the original method for a strong elastic uniaxial compression of single crystals up to 5-6 kbar and the calibration procedure are described in detail for the first time. This method permits the simplification of sample installation and adjustment. In addition, it prevents the frequent destruction of samples that takes place in conventional methods of uniaxial compression between two anvils. The method was developed in Moscow State University and has been successfully used in scientific research in Copenhagen University, Moscow State University, and Humboldt University in Berlin. Along with the method description, some results are discussed for the illustration of the method possibilities.

2. Method description
In the method under consideration (see figure 1a), a sample in the form of a parallelepiped is firmly fastened with epoxy in an elastic ring along the “Y” direction, and afterward a tensile stress is to be applied to the ring in the “X” direction. The ring transforms a tensile stress into a compressive one, applied to the both ends of the sample in the plane of the ring. Axial distribution of the stress in the ring and a rigid fastening in the ring prevent the sample of a premature destruction. Moreover, in case when plastic deformation of a single crystal develops by a sliding process, the fixation of the main cleavage planes of the crystal inside the ring moves the plastic flow limit to higher values, until it starts developing along secondary cleavage planes. This takes place, for example, in Bi, Sb, and Bi,Sb\(_{1-x}\) alloys in which the elastic limit at low temperatures is determined by the plastic flow along (111) planes. Securing the easiest (111) cleavage planes in the plane of the ring with Araldit epoxy makes it possible to increase the elastic loading by more than 10 times in comparison with the
previous uniaxial stress experiments [1]. The strain tensor components in the elastic region, determined by X-Ray diffraction, reach $\varepsilon_{xx} = 0.13\%$, $\varepsilon_{yy} = -0.3\%$, $\varepsilon_{zz} = 0.1\%$ in Bi samples under tensile stress $F = 300$ N. The maximum pressure achieved by this technique is limited not by the sample destruction, but by the breaking of the ring in points of stress concentration.

Figure 1. (a) Sample (1), ring (2), “holder” for the fixation in the stretching device (3). (b) Ring fixation in the stretching device.

With the help of a special stretching device, the lower part of which is depicted in figure 1b, the stress can be applied to the ring just at the liquid helium temperature, and the value of the stress can be determined by the elongation of the calibrated spring (this part is not depicted). This method is very convenient for magnetotransport and optical measurements and is applicable for single crystals with quite different elastic properties: (Bi [1], Sb [2], Hg$_{1-x}$Cd$_x$Te [3], GaAs [4]). Concerning optical measurements, the device on figure 1b is open for illumination, and experiments under uniaxial stress are much easier than with devices for hydrostatic (or quasi hydrostatic) compression.

Calibration of the ring with Bi, Bi$_{0.95}$Sb$_{0.05}$ or GaAs samples inside was performed at liquid nitrogen temperature from the shift of X-Ray diffraction angles from different crystallographic planes in dependence on the stress $F$ applied to the ring. In figures 2a and 2b, we represent the dependence of diffraction maxima and diffraction angles on the applied stress $F$. Reversible change of both characteristics under loading and unloading demonstrates the elastic nature of the strain. From calculations made on the base of X-Ray measurements, the useful empirical correlation (figure 3) was determined. It is used to find a compressive force $F_c$ applied directly to the sample from the tensile force $F = F_{ring}$, which is applied to the ring (figure 1a). The calibration curve depicted in figure 3 permits evaluation of the direct compressive force for the samples of different cross sections $A$ and Young Modulus $E_s$, if only the ring dimensions and elastic properties are preserved.

Figure 2. (a) X-ray diffraction maximum (Bi$_{0.95}$Sb$_{0.05}$ at $T = 77K$) corresponding to (555) plane at different force $F$ applied to the ring: 1 – 0, 2 – 90 N, 3 – 180 N, 4 – 270 N, 5 – 0 after the pressure is removed . (b) Change of diffraction angle for planes 1 – (633) and 2 – (555) in Bi$_{0.95}$Sb$_{0.05}$ under loading (open circles) and unloading (filled circles) of the sample.
Figure 3. Ratio between compressive force $F_\text{S}$ applied to the sample and tensile force $F = F_\text{Ring}$ applied to the ring for samples with different cross sections $A$ (squares) and samples with different elastic constants $E_\text{S}$ (circles). Calculations are made on the base of X-ray measurements.

In our experiments the rings with inner diameter $D_{\text{in}} = 3$ mm, outer diameter $D_{\text{out}} = 4.5$ mm and thickness $d = 0.75$ mm were cut by the electro-erosion method from nonmagnetic alloy. The sample dimensions are determined by geometry of the ring. The data in figure 3 reflect X-Ray measurements on Bi, Bi$_x$Sb$_{1-x}$ and GaAs samples. Decrease of the sample cross sections $A$ leads to increase of the attainable compressive pressure $P = F_\text{S}/A$ (figure 3), however $A$ should not be too small and the sample should not bend under loading.

Advantages and possibilities of the technique are illustrated below by some experimental results obtained on Bi, Bi$_x$Sb$_{1-x}$ alloys and on GaAs/Al$_x$Ga$_{1-x}$As heterostructures. These materials are quite different in their elastic properties. Rather soft Bi with Young Modulus $E_\text{S} = 380$ kbar and hard GaAs with $E_\text{S} = 1200$ kbar are specially chosen for illustration among other samples investigated under uniaxial compression in the ring.

3. Method application

3.1 Electronic topological transitions in Bi and Bi$_x$Sb$_{1-x}$ alloys

Since this method gives the possibility of continuous applications of elastic uniaxial strain at low temperature, it permitted to obtain the most convincing picture of electronic topological transitions (ETT) development in Bi and Bi$_x$Sb$_{1-x}$ alloys [1]. ETTs of $2\frac{1}{2}$ type [5] take place at certain conditions (for example under compression) if one or more parts of a Fermi surface (FS) arise or vanish. It was shown that, in accordance with the theory [5], these transitions are accompanied by anomalies in thermoelectric power and resistivity [1], and the peculiarities of these anomalies are determined by the character of the FS change.

Usually, very high hydrostatic pressure is necessary for ETTs observation in metals, semimetals, and semiconductors. The different situation arises under uniaxial stress. In Bi and Bi$_x$Sb$_{1-x}$ alloys, uniaxial stress along a binary $C_2$ or bisector $C_1$ axes breaks a crystal symmetry and leads to a non-equivalent shift of electron extrema $L_i$ in respect to the Fermi level (FL) and the hole extremum $T$ (see, for example, the sketch on figure 4a). As a result, more than 10 electronic topological transitions of different type were observed in this system with the help of the uniaxial stress technique described above [1]. One of these transitions, as an example, is represented below.

Transition $1h \rightarrow 1h + 2e$ (Bi + $10^{-2}$ at.% Sn) is illustrated in figure 4. The letters "h" and "e" denote hole and electron ellipsoids of the FS. Quantum oscillations of magnetoresistance $dR/dH(H)$ – so called Shubnikov-de Haas (SdH) oscillations – in figure 4b are determined by FS cross sections. In Bi doped with $10^{-2}$ at.% Sn electron extrema $L_i$ are above the FL (see figure 4a), and only the hole FS exists at zero stress (high oscillation frequency in figure 4b). Under uniaxial compression along binary axis two equivalent $L_i$ extrema go down and reach the FL. Correspondingly, at higher stress two electron ellipsoids (low frequency, figure 4b) appear on the background of the hole FS. The emergence of the electron FS cross section $S$ and its increase under strain is depicted in figure 4a ($S_{\text{max}}$ is the maximal value of $S$ obtained under compression). At the same compressive strain $\varepsilon_{yy} \approx \varepsilon_{xx}$
-0.8x10^{-3}$, where ETT occurs, the anomalous behavior of thermoelectric power $V$ and resistance $R/R_0$ ($R_0$ – resistance at zero pressure) in dependence on strain takes place (figure 4c).

**Figure 4.** (a) – Configuration of electron $L_1$ and hole $T$ extrema near the FL at zero stress and in ETT point in Bi + $10^{-2}$ at.% Sn; emergence and increase of the electron FS cross section in dependence on strain. (b) – Magnetoresistance oscillations in Bi + $10^{-2}$ at.% Sn at different strain $\varepsilon_{yy}$: 1 – 0; 2 – $-1.5x10^{-3}$; 3 – $-2.4x10^{-3}$. (c) – Thermoelectric power (left side) and resistance (right side) in dependence on strain.

It has been shown that uniaxial stress permits to shift band extrema across the Fermi level and directly observe the FS transformation of a different type. This effect is accompanied by singularities of the energy spectrum: in this way, the saddle point in the electron and hole energy spectrum of Bi$_x$Sb$_{1-x}$ alloys was determined by disruption (appearance) of the dumbbell-shaped FS under compression [6], and elimination of inter-valley scattering channel in Bi$_x$Sb$_{1-x}$ in the course of transformation from a three valley to a single valley conduction band under compression was detected [7].

### 3.2 Anisotropy effects in 2D hole system at GaAs/Al$_x$Ga$_{1-x}$As heterointerface

For delicate in respect to stress materials, such as Bi and Bi$_x$Sb$_{1-x}$ alloys, the application of the method described above is a decisive factor for the extension of elastic limit and observation of quantum phenomena. However in semiconductor GaAs/Al$_x$Ga$_{1-x}$As heterostructures, it mainly secures samples and simplifies experiments. Below, we only briefly illustrate a strong change of 2D hole energy spectrum anisotropy observed in p-GaAs/Al$_x$Ga$_{1-x}$As under uniaxial compression up to 5 kbar.

The effect of uniaxial compression on valence band structure of single and double p-GaAs/Al$_{0.5}$Ga$_{0.5}$As heterostructures was investigated both theoretically and experimentally [4]. The form of the valence band potential strongly affects the energy spectrum of 2D holes in p-GaAs/Al$_{0.5}$Ga$_{0.5}$As heterostructures. In the case of a single heterojunction (triangular quantum well), the absence of inversion symmetry causes a spin splitting of the valence subbands, and so the two lists of the FS correspond to the ground heavy hole state (figure 5, P = 0 kbar). According to our theoretical calculations [4], external uniaxial compression in 2D hole plane causes a strong anisotropy of hole energy spectrum. Figure 5 illustrates the numerical calculations of 2D hole Fermi surface transformation in p-GaAs/Al$_x$Ga$_{1-x}$As under uniaxial compression. A strong stretching of the FS in the direction perpendicular to the direction of compression should be noted, while the contours of the hole FS’s in both subbands touch one another in the compression direction at pressure $P = 2.5$ kbar.

Predicted modifications of 2D hole FS were detected under uniaxial compression by measurements of quantum Hall effect, SdH oscillations (figure 6) and transport properties in p-GaAs/Al$_{0.5}$Ga$_{0.5}$As with carrier concentration $N_p = 7.6 \times 10^{11}$ cm$^{-2}$. A development of magnetic breakdown between two spin split subbands observed under uniaxial compression in [110] direction within the pressure range 1.5 – 3.0 kbar [8] reveals a strong experimental evidence of the FS
transformation depicted on figure 5. In this pressure range the distance between the two FS’s is minimal (figure 5), which allows tunneling of the holes between different orbits in the magnetic field. As a result, according to Fourier transform analysis, additional frequencies corresponding to new breakdown orbits on the FS appear in SdH oscillations. The possibility of transition from one quasi-classical orbit to the other one under magnetic breakdown condition affects the dynamic of charge carriers motion, and apart from the initial (at P = 0) orbits 1-4 and 2-3, the orbits 1-3 and 2-4, as well as their harmonics, arise under compression (figure 5, P = 2.5 kbar) [8].

**Figure 5.** The FS contours S₀ and S₁ in two spin-split subbands of 2D heavy holes at different pressures along the [110] direction. For P = 2.5 kbar, the magnetic breakdown regions are hatched, and the numerals 1, 2, 3 and 4 correspond to the different segments of the magnetic breakdown orbits.

Changes of 2D hole FS are illustrated by SdH oscillations on p-GaAs/Al₀.₅Ga₀.₅As sample with two perpendicular mesas (with directions of electrical current parallel and perpendicular to the direction of compression) represented in figure 6. In accordance with band structure calculations, magnetotransport measurements reveal that the differences between hole concentrations in the ground-state spin-split subbands and also between the corresponding effective masses decrease with increase of pressure [4].

**Figure 6.** SdH oscillations for GaAs/Al₀.₅Ga₀.₅As sample with hole concentration N₀ = 7.6 × 1₀¹¹ cm⁻² at different pressures applied along [110] direction. Dash lines correspond to the direction of current parallel to the direction of compression, solid lines correspond to the perpendicular direction of electrical current.
Strong modification of 2D hole FS anisotropy induces corresponding modification of p-GaAs/Al$_{0.5}$Ga$_{0.5}$As transport properties. Most evidently it is reflected in essential anisotropy of electrical resistance and mobility anisotropy under compression (figure 7) [9]. Data obtained on a two-mesa sample indicate strong increase of mobility (up to 3 times at $P = 5$ kbar) in direction of compression. It qualitatively correlates with evident decrease of the FS dimensions and 2D hole transport mass in this direction (figure 5). The reverse situation takes place in the direction perpendicular to the compression direction (figure 7). The small anisotropy at $P = 0$ is determined by the surface roughness scattering.

4. Conclusion
We have described an original method for strong elastic uniaxial compression of single crystals. This method is simple in realization, secures samples of a premature damage, permits application of uniaxial stress at liquid helium temperatures, and in some cases shifts the elastic limit to higher stress values. Experimental results obtained with the help of the method demonstrate the method possibilities. This work was supported by the Russian Foundation for Basic Research (project 07-02-00866) and Russian Federation President Grant Council (grant NS-5248.2006.2). We are grateful to our colleagues Dr. O.P. Hansen from Copenhagen University, Dr. W. Kraak from Humboldt University in Berlin for their cooperation, and student B. Dyatkin for the technical assistance with the paper preparation.

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