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# DEFORMATION-INDUCED EFFECTS IN INDIUM ANTIMONIDE MICROSTRUCTURES AT CRYOGENIC TEMPERATURES FOR SENSOR APPLICATIONS

The authors investigate deformation-induced changes in the electrophysical parameters of the indium antimonide microcrystals at cryogenic temperatures in strong magnetic fields up to 10 T. It is determined that for strongly doped InSb microcrystals, the gauge factor at liquid-helium temperature is  $GF_{4.2K} \approx 72$  for the charge carrier concentration of  $2 \cdot 10^{17}$  cm<sup>-3</sup>, while being  $GF_{4.2K} \approx 47$  for the concentration of  $6 \cdot 10^{17}$  cm<sup>-3</sup>, at  $\varepsilon = -3 \cdot 10^{-4}$  rel. un. For the development of magnetic field sensors based on the magnetoresistive principle, the effect of a giant magnetic resistivity reaching 720% at a temperature of 4.2 K is used.

Ключевые слова: InSb whiskers, gauge factor, magnetoresistance, sensor.

Despite the rapid progress and significant advances in microelectronics, there remain a lot of problems that require further detailed study of the physical properties and possible applications of whisker crystals. Modern cryogenic electronics requires highly sensitive, high-speed devices and components of integrated circuits, capable of operating at various temperature intervals, including the cryogenic temperature range. Principles of cryoelectronics are used to build a number of devices (cryotrons, quantum and parametric amplifiers, resonators, filters, sensors, delay lines, etc.) based on silicon technologies [1]. On the other hand, apart from using traditional silicon whisker crystals in modern microelectronics, scientists carry on intensive studies of other materials and structures. For instance, there is an ongoing work on creating solid state electronics based on siliconon-insulator structures [2]. Using polycrystalline silicon in manufacturing of microelectronic devices makes it possible to create multilayer structures. One of the advantages of such structures is that the resistivity of the created layers varies within a very wide range (several orders of magnitude).

However, the need for deep cooling and related technological difficulties considerably restrict the use of such materials. Moreover, one of the most important areas of modern magnetoelectronics is the study of the magnetoresistive effect (**MR**), and in recent years the emphasis has been placed on the phenomenon of giant magnetoresistance (**GMR**). In the developed devices [1], in which there are new effects due to the interaction of «magnetic electrons» with artificially created nanosized structures, a combination of magnetism and electronics is used, so they claim the birth of a new area of magnetism and technology — magnetoelectronics. In this case, InSb whisker crystals, due to their morphology, high values of charge carrier mobility, structural perfection and high mechanical strength, are a good model for studying the influence of external factors, in particular magnetic field, for the concentrations corresponding to the metal-semiconductor transition [3].

It is known that film materials using the magnetoresistive effect, are sensitive to the electric current. Therefore, by changing the value of the current one can change the amplitude of the magnetoresistance. Thus, the authors of [4] established the dependence of the magnetoresistance of multi-layer materials based on Co, Ni or Pt on the measuring current value. The obtained results are explained [5] by the different effect of the torque moment transfer by spin-polarized charge carriers at different values of current. This effect was anticipated by the authors of [6, 7] and experimentally studied in [8-10].

On the other hand, the authors of [11, 12] studied the manifestation of the magnetoresistive effect in semiconductor-dispersed magnets. Here, however, the magnetoresistance did not reach high values (as opposed to InSb), which directly affects the sensitivity of the devices developed on their basis.

Previous studies conducted for InSb microcrystals at cryogenic temperatures in strong magnetic fields up to 14 T allowed detecting a number of effects and to assess a number of electrophysical parameters such as Shubnikov – de Haas (SdH) oscillations (and their period) [13] and Dingle

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temperature [3], to detect GMR and negative magnetoresistance, associated with significant spin-orbital exchange interaction.

Moreover, the influence of external factors, such as deformation, allowed to determine the occurrence of the Berry phase [14] in the longitudinal magnetoresistance, which was previously observed by the authors of [15-17] in bismuth. It is obvious that the deformation leads to a change in the scattering mechanisms, and may also affect electrophysical parameters in the transverse magnetoresistance, in particular the GMR value.

The authors of this work aimed at studying the deformation-induced changes in the electrophysical parameters of the indium antimonide microcrystals at the cryogenic temperatures in strong magnetic fields up to 10 T, particularly in the transverse magnetoresistance, in order to investigate the possibilities of using such crystals in the magnetoresistive sensors of deformation and magnetic field, which could be used under difficult operating conditions.

## **Experiment details**

In order for the InSb whisker crystals to grow, i.e., for the material to be transferred to the crystallization zone, it was necessary to create a concentration gradient. This was achieved by creating a temperature gradient between the dissolution zone and the crystallization zone.

The whisker crystallization temperature was 720 K, while the evaporation zone temperature was 850 K. While growing, the crystals were doped with tin admixture, and the concentration of charge carriers, determined by using Hall effect, was from  $6 \cdot 10^{16}$  to  $6 \cdot 10^{17}$  cm<sup>-3</sup>. The chosen InSb whiskers were 2-3 mm in length and had lateral dimensions of about 30-40 µm. Gold microwires (10 microns in diameter) were pulse-welded to the InSb micro-crystal to create eutectic contacts.

InSb whisker conductivity was studied in the temperature range from 4.2 to 300 K. For these studies, crystals were cooled down to the temperature of 4.2 K in a helium cryostat. The temperature was measured by using a Cu–CuFe thermocouple, calibrated with a CERNOX sensor.

The magnetic field effects of the whiskers were studied using a Bitter magnet with the induction of up to 14 T and the time scanning of field of 1.75 T/min in the temperature range of 4.2-77 K. Stabilized electric current along the whisker was created by the current source Keithley 224 in the range of 1-10 mA, depending on the resistance of the crystal. CERNOX sensor was used to measure magnetic parameters. Being weakly sensitive to magnetic field induction *B*, the device has a variation of the output signal of about 1% at B = 15 T.

In order to evaluate the possibility of using InSb microcrystals in mechanical sensors, the authors used a technique of deforming the samples by producing a difference between the linear expansion coefficients of the crystal itself and the substrate on which it was fixed [18]. The uniaxial deformation of microcrystals was carried out by affixing the crystals on the substrates with HL-931 glue with a polymerization temperature of 180°C. According to this method, when the crystal is fixed on the substrate, the thermal stress in the former can be estimated by the ratio

$$\sigma_{t}(T) = \frac{1}{t_{c}} \int_{T_{0}}^{T} \frac{\alpha_{s}(T) - \alpha_{c}(T)}{\frac{1 - v_{s}(T)}{E_{s}(T)t_{s}} + \frac{1 - v_{c}(T)}{E_{c}(T)t_{c}}} dt, \qquad (1)$$

- were  $\alpha_c$  and  $\alpha_s$  are temperature coefficients of linear expansion of the crystal and substrate, respectively;
- $E_c$ ,  $E_s$  and  $v_c$ ,  $v_s$  are Young's moduli and Poisson coefficients of crystal and substrate materials, respectively;
  - $t_c$  and  $t_s$  are the thickness of the crystal and the substrate.

Parameter  $T_0$  in this formula corresponds to the technological temperature at which a rigid connection is formed between the crystal and the substrate, e.g., this may be the temperature of adhesive polymerization.

To calculate the thermal deformation of the whiskers, the authors used the temperature dependences of the thermal expansion coefficients and of Young's moduli for InSb and Cu from [19-21].

Assuming that the elastic coefficients depend on temperature slightly in the temperature range of 4.2-50 K, we can bring the formula (1) to the following form:

$$\varepsilon_{t}\left(T\right) = \gamma \int_{T_{0}}^{T} \left[\alpha_{s}\left(T\right) - \alpha_{c}\left(T\right)\right] dt, \qquad (2)$$

were  $\gamma$  is the coefficient that characterizes the efficiency of the transmission of deformation from the substrate to the crystal, its value depending on the geometry of the samples and the methods of their fixation. In our case, the value of  $\gamma$  is calculated according to [18] and is equal to 0.7.

## **Experimental results**

The use of semiconductor sensors based on the piezoresistive effect remains the most common means for converting mechanical quantities into an electrical signal due to the high sensitivity and reliability of the design [18]. Therefore, the focus of the experimental studies of InSb semiconductor crystals at temperatures of 4.2-300 K in strong magnetic fields (up to 14 T)



Fig. 1. Temperature dependence of the resistance of deformed InSb samples with different concentration of charge carriers in the vicinity of the metal dielectric transition (in  $cm^{-3}$ ):

$$1 - 6.10^{16}$$
;  $2 - 2.10^{17}$ ;  $3 - 6.10^{17}$ 

was on determining exactly how the deformation affected the samples. Thus, **Fig. 1** shows the temperature dependence of the resistance of microcrystals fixed on copper substrates with an average deformation level  $\varepsilon = -3 \cdot 10^{-4}$  rel. un. in the temperature range from 4.2 to 300 K.

Preliminary studies [3, 13] for free (not fixed) InSb whiskers allowed estimating the gauge factor (GF) at low temperatures, which can be determined by the ratio

$$GF = \frac{R - R_0}{R_0 \varepsilon},\tag{3}$$

were  $R_0$  is the resistance of the undeformed (free) crystal;

R is the resistance of the deformed crystal;

 $\boldsymbol{\epsilon}$  is the uniaxial strain acting on the crystal.

Fig. 2 presents the temperature dependences of the gauge factor for these crystals calculated from the experimental data. Throughout the entire studied temperature range, strongly doped crystals (Fig. 1) demonstrate the temperature dependence of resistance that is typical for metal. Piezoresistance manifests itself typically in these crystals [22]: when subjected to compression deformation, the resistance of the crystals decreases.

At the temperature of liquid helium,  $GF_{4.2K} \approx 72$  for InSb microcrystals with a concentration of  $2 \cdot 10^{17}$  cm<sup>-3</sup> and  $GF_{4.2K} \approx 47$  for the crystals with a concentration of  $6 \cdot 10^{16}$  cm<sup>-3</sup>, at  $\varepsilon = -3 \cdot 10^{-4}$  rel. un. However, for InSb microcrystals with a concentration of  $6 \cdot 10^{16}$  cm<sup>-3</sup>, gauge factor exhibits non-typical properties: above the temperature of the liquid nitrogen, it changes its sign from positive to negative. The absolute value of the gauge factor both at the temperatures reaches  $GF \approx 350$ , which can be explained by the fact that the charge carrier concentration approaches the values of the dielectric state of the metal — dielectric phase transition. Apparently, this is caused by the fact that the ensemble of charge carriers at low temperatures becomes reduced, because the carriers are being freezed out, and their transport is believed to be caused by strong spin-orbit interaction in the range of jump conductivity by twice localized impurities [3, 13, 23].

Investigation of the behavior of the magnetic resistivity at low temperatures can also help understand the processes occurring in crystals at low temperatures. Thus, the authors of [3] noticed that magnetoresistance changes its sign from positive to negative in the longitudinal direction, which indicates a characteristic feature of the studied samples. Another such feature was described in [24]: magnetoresistance deviated from the quadratic dependence in the range of relatively weak magnetic fields.

The authors of [24] explained the emergence of a negative magnetic resistivity by the formation of «pairs», i.e., two states with paired spins, that are relatively close to each other and distant from others, which exist near the Fermi level.

In addition, the authors of both [3] and [25] received high values of Lande *g*-factor ( $g \approx 60$ ) at a temperature of 4.2 K, indicating a strong spin-orbit interaction.





In its turn, the deformation leads to the redistribution of energy zones and to the manifestation of the negative magnetic resistivity effect, associated with a change in the density of states near the Fermi level in the magnetic field. If the «pair» is ionized once, its level is shifted upward when the magnetic field increases. Some of the levels shift downward, and others shift upwards, which leads not only to the displacement of the Fermi level, but also to the change in the density of states in its vicinity.

The authors of [13, 17] demonstrated that if the chemical potential is equal to or lies slightly below the second subband, then the increase of the magnetic field B and the movement of the charge carriers in the boundary zones cause the chemical potential to shift, and due to this fact, the resistance first drops sharply and then monotonously grows. This is connected to the competing influence of the magnetic field on the magnitude of the multiplier and the index in the expression of the carrier dispersion probability at the Fermi level.

Fig. 3, *a* shows the experimental results on the transverse magnetoresistance for deformed crystals with a charge carrier concentration of  $6 \cdot 10^{16}$  cm<sup>-3</sup> (which corresponds to the dielectric state of the metal – dielectric phase transition). The figure demonstrates that the deformation causes changes in the conduction mechanisms inside the crystals, which is reflected in the anomalous behavior of the gauge factor. Thus, at low temperatures, Shubnikov de Haas oscillations begin to manifest themselves in InSb crystals for the transverse magnetoresistance, same as they do for the longitudinal magnetoresistance.

For the deformed InSb microstructures with a charge carrier concentration of  $6 \cdot 10^{17}$  cm<sup>-3</sup> (which corresponds to the metal state of the metal-dielectric phase transition), the magnetoresistance significantly increases, reaching 250% (Fig. 3, *b*).

The probable reason for the growth of the magnetoresistance in such crystals is, obviously, the release of «freezed-out» excess charge carriers due to an increase in their mean concentration in the crystal, and, consequently, an increase in average mobility. This leads to the linearization of the characteristics and a decrease of the temperature coefficient of resistance of crystals in the 4.2-70 K range.

On the other hand, in the deformed InSb microcrystals with the charge carrier concentration of  $2 \cdot 10^{17}$  cm<sup>-3</sup> (which corresponds to the metaldielectric phase transition), a giant magnetic resistance was also observed, as well as its significant increase in value (Fig. 3, *c*), reaching 720%. In this case, however, the values of the temperature magnetoresistivity coefficient were lower for the temperature range of 4.2-70 K, which increases the sensitivity to the magnetic field.



Fig. 3. Transversal magnetoresistance of InSb whiskers with different tin concentration (in cm<sup>-3</sup>) for strained samples at temperature range 4.2–70 K:  $a - 6\cdot10^{16}$ ;  $b - 6\cdot10^{17}$ ;  $c - 2\cdot10^{17}$ 

### Application

The present stage of the development of new branches of science and technology (space and aviation technology, cryogenic technology, cryoenergy, etc.) highlights the problem of creating miniature highly sensitive mechanical, thermal, and magnetic sensors with a special capacity to operate at low temperatures [26-31].

The studies on the influence of deformation and magnetic field on indium antimonide microcrystals with a charge carrier concentration from  $6 \cdot 10^{16}$  to  $6 \cdot 10^{17}$  cm<sup>-3</sup> allowed identifying a number of effects that make such materials suitable for use as basis for the piezoresistive sensors and magnetic field magnetoresistive sensors.

A photo of a standard physical quantity sensor, developed during this study, is shown in **Fig. 4**.

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Fig. 4. Typical view of sensors of physical quantities

The temperature coefficient of resistance (**TCR**) for such microcrystals was found to be  $TCR \approx 0.004 \ \Omega/K$  (Fig. 1, curves 2, 3) at room temperature (Fig. 2, *a*). Such samples can be used to create mechanical sensors for two temperature ranges: from 4.2 to 50 K and from 50 to 300 K, since there is a significant increase in  $GF_{300K} \approx 720$  at room temperature (Fig. 2, *a*).

For InSb crystals with a charge carrier concentration corresponding to the dielectric state of the metal-dielectric phase transition, the gauge factor is  $GF_{4.2K} \approx 350$  and  $GF_{300K} \approx -350$ . The temperature coefficient of resistance of such samples is slightly lower ( $TCR \approx 0.001 \ \Omega/K$ ). The temperature-related change in the gauge factor is linear (Fig. 2, b). Such samples can be used to create piezoresistive sensors for a wide range of temperatures.

The research on the magnetoresistance of deformed InSb microcrystals with a charge carrier concentration from  $6.10^{16}$  to  $6.10^{17}$  cm<sup>-3</sup> (covering the metal – dielectric phase transition) at cryogenic temperatures showed the following. The samples with a charge carrier concentration of  $6 \cdot 10^{16} \text{ cm}^{-3}$ showed an instability of the temperature coefficient of the magnetoresistance in the range of 4.2-70 K, caused by oscillation phenomena occuring in the transverse magnetoresistance in magnetic fields up to 10 T. Such phenomena make it impossible to use deformed InSb microcrystals in magnetic field sensors at cryogenic temperatures. The deformed InSb crystals with a concentration of charge carriers of 6.10<sup>17</sup> cm<sup>-3</sup> demonstrated a significant increase in the magnetic resistance (up to 250%) at a sensitivity of 600 mV/T. The temperature resistance (magnetic resistivity) for the temperature range of 4.2-70 K in the fields up to 10 T was  $TCR \approx 0.57 \ \Omega/K$  (Fig. 3, b). As for the deformed InSb crystals with the charge carrier concentration of  $6.10^{17}$  cm<sup>-3</sup> (which corresponds to the metal-dielectric transition), there was detected the giant magnetoresistance (which increased up to 720%). The sensitivity to the magnetic field here was 1500 mV/T. The temperature coefficient of resistance for the temperature range of 4.2-70 K in the fields up to 10 T was  $TCR \approx 0.46 \ \Omega/K$  (Fig. 3, c).

#### Conclusions

The studies on the influence of deformation on the electrophysical parameters of indium antimonide microcrystals at cryogenic temperatures in strong magnetic fields (up to 10 T) allowed discovering a number of effects that make such materials suitable for use as basis for the magnetoresistive sensors of deformation and magnetic field, that could function under complex operating conditions.

It has been determined that the best option for piezoresistive sensors that could function in a wide temperature range (4.2-300 K) are the InSb microcrystals with carrier concentration of  $6 \cdot 10^{16} \text{ cm}^{-3}$  (which corresponds to the dielectric state of the metal-dielectric transition).

Magnetic field sensors based on magnetoresistive principle were developed using the giant magnetic resistivity effect reaching 720% at a temperature of 4.2 K. Such sensors contain deformed InSb microcrystals with a carrier concentration corresponding to the metal-dielectric transition  $(2 \cdot 10^{17} \text{ cm}^{-3})$ . The developed microelectronic sensor has ultra-high sensitivity to a magnetic field of 1500 mV/T, and the simplicity of its design provides low inertia and high performance at the same time.

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## ДЕФОРМАЦІЙНО-СТИМУЛЬОВАНІ ЕФЕКТИ В МІКРОСТРУКТУРАХ АНТИМОНІДУ ІНДІЮ ЗА КРІОГЕННИХ ТЕМПЕРАТУР ДЛЯ СЕНСОРНИХ ЗАСТОСУВАНЬ

У роботі досліджено деформаційно-стимульоване змінення електрофізичних параметрів ниткоподібних кристалів антимоніду індію за кріогенних температур у сильних магнітних полях (до 10 Тл). Ниткоподібні кристалі InSb вирощувалися методом хімічних газотранспортних реакцій. Температура зони кристалізації становила 720 К, зони випаровування — 850 К. Легування кристалів здійснювалося домішкою олова в процесі росту, а концентрація носіїв заряду, згідно з дослідженнями Холла, становила  $6 \cdot 10^{16} - 6 \cdot 10^{17}$  см<sup>-3</sup>. Для досліджень були вибрані ниткоподібні кристали InSb довжиною 2–3 мм з поперечними розмірами близько 30–40 мкм. Електричні контакти до ниткоподібних кристалів InSb були створені за допомогою мікродротів Au діаметром 10 мкм, які утворюють евтектику з мікрокристалом під час імпульсного зварювання.

Електропровідність ниткоподібних кристалів InSb досліджувалася в діапазоні температури від 4,2 до 300 К. Кристали охолоджували в гелієвому кріостаті. Температуру вимірювали за допомогою термопари Си–СиFe, каліброваної за допомогою сенсора CERNOX. Деформацію зразків ( $\varepsilon = -3 \cdot 10^{-4}$  відн. од. при 4,2 К) створювали за рахунок різниці в коефіцієнтах термічного розширення ниткоподібних кристалів та матеріалу підкладки, закріплюючи кристали на мідній підкладці та охолоджуючи до низьких температур.

На основі порівняння опору деформованих та недеформованих кристалів були визначені коефіцієнти тензочутливості. Значення коефіцієнта тензочутливості мікрокристалів InSb за температури рідкого гелію становить GF<sub>4.2K</sub>  $\approx$  72 за концентрації носіїв заряду 2·10<sup>17</sup> см<sup>-3</sup> та GF<sub>4.2K</sub>  $\approx$  47 за концентрації б ·10<sup>17</sup> см<sup>-3</sup>. Для зразків InSb з концентрацією 6·10<sup>16</sup> см<sup>-3</sup> коефіцієнт тензочутливості виявляє нетипові властивості: вище температури рідкого азоту він змінює свій знак з позитивного на негативний. Абсолютне значення коефіцієнта тензочутливості як за гелієвих температур, так і в області кімнатної досягає приблизно 350, що можна пояснити наближенням концентрації носіїв заряду до фазового переходу «метал — діелектрик».

Встановлено, що для застосування в п'єзорезистивних датчиках, працездатних в широкому температурному діапазоні (4,2–300 К), слід використовувати мікрокристали InSb з концентрацією носіїв заряду 6·10<sup>16</sup> см<sup>-3</sup>. Для розробки датчиків магнітного поля з магніторезистивним принципом дії використовується ефект гігантського магнетоопору, який досягає 720% за температури 4,2 К. Такий датчик містить деформовані мікрокристали InSb з концентрацією носіїв заряду, що відповідає металевому боку переходу «метал – діелектрик» і становить 2·10<sup>17</sup> см<sup>-3</sup>. Розроблений мікроелектронний датчик має надвисоку чутливість до магнітного поля (1500 мВ/Тл), а простота конструкції забезпечує одночасно низьку інерційність та високу продуктивність.

Ключові слова: ниткоподібні кристали, InSb, коефіцієнт тензочутливості, магнетоопір.

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## ДЕФОРМАЦИОННО-СТИМУЛИРОВАННЫЕ ЭФФЕКТЫ В МИКРОСТРУКТУРАХ АНТИМОНИДА ИНДИЯ ПРИ КРИОГЕННЫХ ТЕМПЕРАТУРАХ ДЛЯ СЕНСОРНЫХ ПРИМЕНЕНИЙ

Исследованы деформационно-стимулированные изменения электрофизических параметров микрокристаллов антимонида индия при криогенных температурах в сильных магнитных полях (до 10 Тл). Установлено, что значение коэффициента тензочувствительности микрокристаллов InSb при температуре жидкого гелия составляет  $GF_{4.2K} \approx 72$  при концентрации носителей заряда  $2 \cdot 10^{17}$  см<sup>-3</sup> и  $GF_{4.2K} \approx 47$  при концентрации  $6 \cdot 10^{17}$  см<sup>-3</sup> при деформации образцов  $\varepsilon = -3 \cdot 10^{-4}$  отн. ед. Для разработки датчиков магнитного поля с магниторезистивным принципом действия используется эффект гигантского магнетосопротивления, которое достигает 720% при температуре 4,2 К.

Ключевые слова: нитевидные кристаллы, InSb, коэффициент тензочувствительности, магнетосопротивление.

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