# МАТЕРІАЛИ ДЛЯ СЕНСОРІВ

# SENSOR MATERIALS

### РАСЅ 71.30.+Н, 73.20.FZ УДК 681.586, 537.32

# SILICON MICROCRYSTALS WITH HIGH PIEZORESISTANCE AT CRYOGENIC TEMPERATURES FOR SENSORS APPLICATION

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#### The summary

The studies of piezoresistance of boron doped p-type silicon whiskers with [111] crystallographic orientation mounted on the spring elements, fabricated of the invar alloy, were carried out in the wide ranges of strains  $\varepsilon = \pm 1.26 \times 10^{-3}$  rel. units and temperatures 4.2–300 K. The measurements were carried out in a helium cryostat. There were investigated silicon whiskers with different types of boron doping: 1) heavily doped crystals with metallic conductivity; 2) in the vicinity of metal-insulator transition (MIT) from the metallic side of MIT and 3) in the vicinity of MIT from the insulating side of MIT. Resistance vs. strain (tensile and compressive) dependences at fixed temperatures: 4.2 K, 77 K and 300 K for Si were measured fo whiskers, mounted on in the tempereture range 4.2-300 K for the heavily doped silicon whiskers in the whole temperature range. The classic piezoresistance was observed. Non-classic piezoresistance at helium temperatures was revealed In Si whiskers with definite boron concentration in the vicinity of MIT. Gauge factor of Si whiskers with boron concentration presence from the insulating side of MIT achieves at 4.2 K the magnitude  $GF_{42K} \approx -10000$  at compressive strain and  $GF_{4.2K} \ge 8000$  at the tensile strain. Obtained characteristics of p-type Si whiskers mounted on the spring elements allowed to forecast the performance of piezoresistive mechanical sensors characteristics on their basis. The possibility of construction of the mechanical parameters sensors (strain gages, pressure sensors etc.) of two types was shown: sensors based on heavily doped p-type Si whiskers for the wide temperature range 4.2-300 K and high-sensitive sensors based on Si crystals with boron concentration in the vicinity of MIT for control and signaling systems at cryogenic temperatures.

Key words: piezoresistance, silicon, whiskers, cryogenic temperatures, mechanical sensors.

### Аннотация

# МИКРОКРИСТАЛЛЫ КРЕМНИЯ С ВЫСОКИМ ПЬЕЗОСОПРОТИВЛЕНИЕМ ПРИ КРИОГЕННЫХ ТЕМПЕРАТУРАХ ДЛЯ ПРИМЕНЕНИЯ В СЕНСОРАХ

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Проведены исследования пьезосопротивления нитевидных кристаллов (НК) кремния р-типа с кристаллографической ориентацией [111], легированных бором, на упругих элементах из инварного сплава в широком диапазоне деформаций є=±1,26×10<sup>-3</sup> отн. ед. и температур 4,2-300 К. Измерения проводились в гелиевом криостате. Исследовались НК кремния с различной концентрацией бора: 1) сильно легированные кристаллы с металлической проводимостью; 2) вблизи перехода металл-изолятор (ПМИ) с металлической стороны; 3) вблизи ПМИ с изолирующей стороны. Определялись зависимости относительного изменения сопротивления НК p-Si, закрепленных на балках из инвара, от деформации растяжения и сжатия при фиксированных температурах: 4,2 К, 77 К и 300 К, а также температурные зависимости коэффициента тензочувствительности этих кристаллов в диапазоне температур 4,2-300 К. В сильно легированных НК p-Si во всём диапазоне температур наблюдался классический пьезорезистивный эффект. В НК Si с концентрацией бора вблизи перехода металл-изолятор при гелиевых температурах обнаружено неклассическое пьезосопротивление. Величина коэффициента тензочувствительности в НК Si с концентрацией бора, соответствующей изолирующей стороне ПМИ, при 4,2 К достигала значений  $K_{_{4,2K}} \approx -10000$  при деформации сжатия и  $K_{_{4,2K}} \ge 8000$  при деформации растяжения. Полученные характеристики НК кремния р-типа, закреплённых на упругих элементах, позволяют прогнозировать характеристики пьезорезистивных сенсоров механических величин на их основе. Показана возможность создания на основе этих кристаллов сенсоров механических величин (деформации, давления и др.) двух типов: для широкого диапазона температур 4,2-300 К на основе сильно легированных НК Si p-типа и высокочувствительных сенсоров на основе кристаллов кремния с концентрацией бора вблизи ПМИ для систем контроля и сигнализации при криогенных температурах.

**Ключевые слова**: пьезосопротивление, кремний, нитевидные кристаллы, криогенные температуры, сенсоры механических величин.

#### Анотація

## МІКРОКРИСТАЛИ КРЕМНІЮ З ВИСОКИМ П'ЄЗООПОРОМ ПРИ КРІОГЕННИХ ТЕМПЕРАТУРАХ ДЛЯ ЗАСТОСУВАННЯ В СЕНСОРАХ

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Проведено дослідження п'єзоопору ниткоподібних кристалів (НК) кремнію р-типу з кристалографічною орієнтацією [111], легованих бором, на пружних елементах з інварного сплаву в широкому діапазоні деформацій  $\varepsilon = \pm 1,26 \times 10^{-3}$  відн. од. і температур 4,2–300 К. Вимірювання проводились в гелієвому кріостаті. Досліджувались НК кремнію з різною концентрацією бору: 1) сильно леговані кристали з металевою провідністю; 2) поблизу переходу метал-діелектрик (ПМД) з металевого боку; 3) поблизу ПМД з діелектричного боку. Визначалися залежності відносної зміни опору НК р-Si, закріплених на балках з інвару, від деформації розтягу і стиску при фіксованих температурах: 4,2 К, 77 К і 300 К, а

також температурні залежності коефіцієнта тензочутливості цих кристалів у діапазоні температур 4,2–300 К. В сильно легованих НК p-Si у всьому діапазоні температур спостерігався класичний п'єзорезистивний ефект. В НК Si з концентрацією бору поблизу переходу метал-ізолятор при гелієвих температурах був виявлений некласичний п'єзоопір. Величина коефіцієнта тензочутливості в НК Si з концентрацією бору, що відповідає діелектричному боку ПМД, при 4,2 К досягала значень  $K_{4.2K} \approx -10000$  при деформації стиску і  $K_{4.2K} \ge 8000$ при деформації розтягу. Отримані характеристики НК кремнію р-типу, закріплених на пружних елементах, дозволяють прогнозувати характеристики п'єзорезистивних сенсорів механічних величин на їх основі. Показано можливість створення на основі цих кристалів сенсорів механічних величин (деформації, тиску та ін.) двох типів: для широкого діапазону температур 4,2–300 К на основі сильно легованих НК Si р-типу і високочутливих сенсорів на основі кристалів кремнію з концентрацією бору поблизу ПМД для систем контролю і сигналізації при кріогенних температурах.

**Ключові слова**: п'єзоопір, кремній, ниткоподібні кристали, кріогенні температури, сенсори механічних величин.

#### Introduction

The problem of creating high-sensitive mechanical sensors for cryogenic temperatures is very actual for different branches of science and technique, particularly, for aerospace instrumentation and cryoenergetics. Since the semiconductor mechanical sensors operation usually based on the piezoresistance effect [1] it seems to be interesting to study the silicon piezoresistance behaviour at liquid helium, since it is the principal material for sensors application.

The giant piezoresistance present in p-type silicon from the insulating side of metal-insulator transition (MIT) when the hopping conduction on localized states exists was predicted theoretically by Shklovsky and Efros [2]. The uniaxial stress in [100] direction influence on the resistance of p-type silicon crystals at cryogenic temperatures are presented in [3]. The study of hopping conduction of strained boron doped silicon in the vicinity of MIT, in the low temperature range 0.05–0.75 K was recently carried out [4]. But in all these papers there is no information about the magnitude of piezoresistance in silicon at cryogenic temperatures and there were no attempts to use this effect to constract high-sensitive mechanical sensors.

Our investigations [5–8] of boron-doped p-type silicon microcrystals in the vicinity of MIT in the temperature range 4.2–300 K revealed the existence of giant piezoresistance in these crystals at cryo-genic temperatures, particularly, at liquid helium temperature. The magnitude of gauge factor of p-Si microcrystals with elevated boron concentration in the vicinity of MIT from the insulating side equals to 4.2 K  $GF_{4.2K} \approx -5.7 \times 10^5$  for the compressive strain and  $GF_{4.2K} \approx 3 \times 10^5$  for the tensile strain.

The main purpose of this work was to study the piezoresistive characteristics of boron doped silicon microcrystals mounted on the spring elements in the temperature range 4.2–300 K to evaluate the possibility to create on their basis the piezoresistive mechanical sensors operating at cryogenic temperatures.

#### The object of investigation and method

P-type silicon whiskers of [111] crystallographic orientation grown from the vapour phase by chemical transport reactions [9] were chozen as the object of investigation. Silicon whiskers were grown as regular hexagonal prisms elongated 3–5 mm long in [111] direction and with 20–30  $\mu$ m back face width. These microcrystals due to their structural perfection and excellent mechanical properties are a good model material to study the piezoresistance. Besides this their growth direction corresponds to the maximal longitudinal piezoresistance in p-type silicon. At the same time, Si whiskers are very promising in creating to create on their basis of piezoresistive mechanical sensors, operating in severe conditions [10-12].

Silicon microcrystals with boron concentration in the vicinity of MIT near the metallic side different from that near the insulating side were selected for investigations. Microcrystals were selected with metallic conduction with  $N_B = 1 \times 10^{19}$  cm<sup>-3</sup> (Si:B1 set) and crystals with  $N_B = 5,5 \times 10^{18}$  cm<sup>-3</sup> (Si:B2 set) from metallic side of MIT and crystals with  $N_B = 3 \times 10^{18}$  cm<sup>-3</sup> (Si:B3 set) from the insulating side of MIT taking into account that critical impurity concentration corresponds to that of vicinity of MIT in p-type silicon, equals to  $N_c = 5 \times 10^{18}$  cm<sup>-3</sup> [3], .

Silicon whiskers characteristics were measured in the temperature range 4.2–300 K by specially developed arrangement with spring element in the form of the cantilever beam. Microcrystals were mounted on the cantilever beam in such a way that one of crystals was under tensile strain, and the other — under compressive strain. Spring element was fabricated of invar alloy 36N, because the thermal expansion coefficient (TEC) magnitude of invar alloy at low temperatures was approximately equal to the magnitude of TEC for silicon [13]. Spring element in the form of the cantilever beam was strained by the special mechanism, which gives the possibility to achieve different strain levels of spring element and crystals, mounted on this spring element, in the range of strains  $\varepsilon = 0 - \pm 1.26 \times 10^{-3}$ relative units, that corresponds to the strain range of spring elements in piezoresistive mechanical sensors, particularly in pressure sensors. The measurements were carried out at different strain levels of the beam:  $\varepsilon_1 = 2,42 \times 10^{-4}$  rel. un.,  $\varepsilon_2 = 5,95 \times 10^{-4}$  rel. un.,  $\varepsilon_3 = 8,79 \times 10^{-4}$  rel. un.,  $\varepsilon_4 = 1,08 \times 10^{-3}$  rel. un.,  $\varepsilon_5 = 1,26 \times 10^{-3}$  rel. un., which corresponds to the uniaxial tensile or compressive strain of Si whiskers according to the place where they were mounted on the beam. The arrangement with spring element and microcrystals was placed in helium cryostat. During the experiment Si whiskers were dc supplied and resistance of crystals was measured by digital device with computer automatic data registration; simultaneously the temperature sensors data was registrated.

The silicon whiskers characteristics on spring elements in the temperature range 4.2–300 K were measured in the International Laboratory of High Magnetic Fields and Low Temperatures in Wroclaw, Poland.

#### Characteristics of silicon whiskers on spring elements

Relative changes of crystals resistance vs. strain  $\Delta R(\varepsilon)/R_0 = f(\varepsilon)$  dependencies at fixed temperatures 4.2 K, 77 K and 300 K for heavily boron doped p-type Si whiskers from Si:B1 set with resistivity  $\rho_{300K}$ =0.005 Ohm×cm, mounted on invar spring element, calculated from the experimental data are presented in Fig. 1. As it was predicted, in these crystals in all the vestigated temperature and strain ranges the classical piezoresistance was observed: crystals resistance increases under the tensile strain and decreases under the compressive strain. One can see from Fig. 1 that dependences of resistance vs. strain are monotonous, their non-linearity increases at cryogenic temperatures due to the piezoresistance nature in p-type silicon. Gauge factor for these crystals is determined as is

$$GF = \frac{\Delta R(\varepsilon)/R_0}{\varepsilon},$$
(1)

where  $R_0$  is the resistance of unstrained ("free") crystal,

 $\Delta R(\varepsilon)$  — the change of crystals resistance under strain,

 $\varepsilon$  — uniaxial strain,

positive for compressive and tensile strains in the temperature range 4.2–300 K. This is the evidence of classic piezoresistance in heavily doped p-type Si crystals at cryogenic temperatures. The gauge factor of Si whiskers with  $\rho_{300K}$ =0.005 Ohm×cm mounted on invar beam equals  $GF_{4.2K}$ =275 at tensile strain  $\epsilon$ =+1,02×10<sup>-3</sup> rel. un. and  $GF_{4.2K}$ =116 at compressive strain  $\epsilon$ =-1,02×10<sup>-3</sup> rel. un.



Fig. 1. Dependence of resistance vs. compressive (a) and tensile (b) strain for p-Si whiskers with  $\rho_{300K}$ =0,005 Ohm×cm on the invar spring element at different temperatures: 1 — 4,2 K; 2 — 77 K; 3 — 300 K.

Experimental results of studying the characteristics of p-Si whiskers from Si:B2 set ( $\rho_{300K}$ =0.010 Ohm×cm) with elevated boron concentration in the vicinity of MIT of the metallic side of transition, mounted on invar spring elements, are illustrated by curves of temperature dependences of crystal resistance at different levels of compressive (Fig. 2a) and tensile strain (Fig. 2b). In Fig. 2a it is clearly seen that the transition from classic piezoresistance to non-classic piezoresistance (crystal resistance increases under the ompressive strain) occurs at cryogenic temperatures. This transition occurs as soon as the strain level of crystal becomes greater at the lowest temperature. Hence the appearance of non-classic piezoresistance is due to the metal-insulator transition, just strain stimulates the transition of the crystal in the MIT region.



Fig. 2. Characteristics of p-type Si whiskers with  $\rho_{300K}$ =0,010 Ohm×cm on invar spring element at different levels of compressive (a) and tensile (b) strain.

More visual appearance of non-classic piezoresistance one could see in Fig. 3, which demonstrates crystal resistance vs. compressive strain at fixed temperatures: 4.2 K, 77 K dependences and 300 K.

For these crystals dependences  $\Delta R(\varepsilon)/R_0 = f(\varepsilon)$  at 77 K and 300 K are usual, but at liquid helium temperature this dependence demonstrates the great rise of crystal resistance under the compressive strain, which is typical for non-classic piezoresistance; in this case resistance very strong depends on the applied strain level.



Fig. 3. Dependence of resistance vs. compressive strain for p-Si whiskers with  $\rho_{300K}$ =0,010 Ohm×cm on the invar spring element at different temperatures: 1 — 4,2 K; 2 — 77 K; 3 — 300 K.

Estimated temperature dependences of gauge factor for these crystals are presented in Fig. 4. At helium temperatures the gauge factor of such p-Si whiskers, mounted on invar spring elements, is strongly rised at tensile strain:  $GF_{4.2K} \ge 900$  at  $\varepsilon = +1.02 \times 10^{-3}$  rel. un. At compressive strain gauge factor changes his sign at cryogenic temperatures and it becomes negative one (Fig. 4, curve 1).



Fig. 4. Temperature dependences of gauge factor for p-Si whiskers with  $\rho_{300K}$ =0,010 Ohm×cm under compressive (1) and tensile (2) strain.

All these tendencies more are developed when we analyze the characteristics of p-Si whiskers from Si:B3 set with the resistivity  $\rho_{300K} \approx 0.013$  Ohm×cm with boron concentration in the vicinity of MIT near the insulating side of transition, mounted on invar spring elements (Figs. 5–7). At helium temperatures the crystals resistance increases by few orders of magnitude under the ompressive strain (Fig. 5, 6), i.e. in these crystals the giant non-classic piezoresistance is observed. At 4.2 K the magnitude of gauge factor achieve the value  $GF_{4.2K} \approx -10000$  at compressive strain, and  $GF_{4.2K} \geq 8000$  at tensile strain (Fig. 7). In this case dependence GF = f(T) is exponential in the region of cryogenic temperatures range.



Fig. 5. Characteristics of p-type Si whiskers with  $\rho_{300K}$ =0,013 Ohm×cm on invar spring element at different levels of compressive (a) and tensile (b) strain.



Fig. 6. Dependence of resistance vs. compressive strain for p-Si whiskers with  $\rho_{300K}$ =0,013 Ohm×cm on the invar spring element at different temperatures: 1 — 4,2 K; 2 — 77 K; 3 — 300 K.



Fig. 7. Temperature dependences of gauge factor for p-Si whiskers with  $\rho_{300K}$ =0,013 Ohm×cm under compressive (1) and tensile (2) strain.

### Conclusions

The fulfilled investigations gave the possibility to reveal at cryogenic temperatures non-classic piezoresistance with the magnitude approximately of 2 orders greater than the classic piezoresistance.for boron doped p-type silicon crystals in the vicinity of metal-insulator transition (MIT.

Characteristics of Si whiskers mounted on spring elements and measured in the wide temperature and strain ranges allowed to predict the possibility to construct ofpiezoresistive sensors based on these crystals. The analysis of these characteristics gives the possibility to create strain sensors (strain gages) of two types:

1) on the basis of heavily boron doped p-type Si whiskers with resistivity  $\rho_{300K} \approx 0.005$  Ohm×cm for the wide temperature range 4.2–300 K;

2) high-sensitive strain gages on the basis of Si whiskers with boron elevated concentration in the vicinity of MIT with resistivity  $\rho_{300K} \approx 0.010-0.013$  Ohm×cm, operating at the temperature of liquid helium. The gauge factor of such sensors is few orders of magnitude greater than for ordinary silicon strain gauges based on the classical piezoresistance.

According to that different types of piezoresistive mechanical sensors, particularly, pressure sensors and liquid level sensors for low temperatures may be constructed on the basis of boron doped p-type silicon whiskers. When heavily doped p-Si whiskers are used in pressure sensors, the operating temperature range of such sensors would be 4.2–300 K. When p-Si microcrystals with boron concentration in the vicinity of MIT with high non-classic piezoresistance are used, high-sensitive pressure sensors operating at liquid helium temperature could be developed. The output of such piezoresistive sensors could achieve at 4.2 K the magnitude of few hundreds of millivolts (without amplifying), which gives the possibility to use such sensors in different control and signaling systems.

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