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## FORMATION OF $Mn_4Si_7$ FILMS BY MAGNETRON SPUTTERING AND A WIDE RANGE OF THEIR THERMOELECTRIC PROPERTIES

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**Abstract.** In the paper, films of higher manganese silicide  $Mn_4Si_7$  have been made and a lot of their properties have been investigated. The composition and structure of the films formed by ion-plasma magnetron sputtering were examined by scanning electron microscopy and X-ray analysis. The temperature dependences of film resistivity (by the four-probe method), of their Seebeck coefficient (by the two-probe method), as well as their Hall constant and optical reflectivity spectra (at room temperature). Their thermoelectric figure of merit, the energy-gap width (0.66 eV), charged-particle density and mobility, etc., were calculated. The properties of the films in the amorphous and polycrystalline phases were compared. The thermopower of the  $Mn_4Si_7$  film was established to increase by about 6 times during the transition from the amorphous phase to the polycrystalline one. The results obtained indicate that it is possible to use this film in heat wave detectors.

**Keywords:** magnetron sputtering, cleaning of silicon wafer, resistivity, silicon, thermoelectric properties

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Научная статья

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## ПОЛУЧЕНИЕ ПЛЕНОК $Mn_4Si_7$ МЕТОДОМ МАГНЕТРОННОГО РАСПЫЛЕНИЯ И ШИРОКИЙ СПЕКТР ИХ ТЕРМОЭЛЕКТРИЧЕСКИХ СВОЙСТВ

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**Аннотация.** В работе были изготовлены пленки высшего силицида марганца  $Mn_4Si_7$  и изучен широкий спектр их свойств. Состав и структура пленок, полученных методом ионно-плазменного магнетронного распыления, исследовались методами сканирующей электронной микроскопии и рентгенофазового анализа. Были измерены температурные зависимости удельного сопротивления пленок (четырёхзондовый метод), их коэффициента Зеебека (двухзондовый метод), а также постоянная Холла и спектры оптического отражения (при комнатной температуре). Рассчитаны термоэлектрическая добротность, ширина запрещенной зоны (0,66 эВ), концентрация и подвижность заряженных частиц и др. параметры. Проведено сравнение свойств пленок, находящихся в аморфной и поликристаллической фазах. Установлено, что термоэдс пленки при переходе из аморфного состояния в поликристаллическое увеличивается примерно в 6 раз. Полученные результаты доказывают, что пленку можно применять в детекторах тепловолнового излучения.

**Ключевые слова:** магнетронное распыление, высший силицид марганца, удельное сопротивление, коэффициент Зеебека, термоэлектрические свойства

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## Introduction

Many research centers around the world are currently conducting studies on the creation of environmentally friendly and low-cost energy sources. In this regard, great results have been achieved in the conversion of wind, light and heat energy into electrical one, which has led to an increase in the efficiency of the generated photovoltaic and thermoelectric elements [1 – 8]. In addition, scientific research on the creation of new types of photo- and thermoelectric films has been constantly developing [9 – 14]. Among these materials, the most promising one is a film based on a higher manganese silicide (HMS), whose thermoelectric figure of merit can reach 0.4 in the temperature range of 20 – 700 °C [15 – 21]. A  $Mn_4Si_7$  thin film can be used for fabrication high-quality thermal elements and show the possibility of creating nanostructures with high thermal properties based on fundamental research on various physical properties, quantum effects, and size factors. Sensitivity cells based on HMS structures are also promising when using highly sensitive receivers of electromagnetic waves in visible and IR fields.

The goal of the present paper is to form the  $Mn_4Si_7$  film by the ion-plasma magnetron sputtering method and to study its thermoelectric properties.

### Material and methods

Magnetron sputtering synthesis of HMS films is carried out using  $\text{SiO}_2/\text{Si}$  substrate. Before the formation of the HMS film, the  $\text{SiO}_2/\text{Si}$  substrate was cleaned in two stages:

1. The cleaning of the silicon wafers surface  $\text{SiO}_2/\text{Si}$  ( $d = 60$  mm) was carried out using an ammonia-peroxide mixture at a temperature of  $60 - 70$  °C, washing in deionized water, drying in a centrifuge;

2. The vacuum treatment (cleaning) of the silicon wafer surface was carried out using an argon plasma flow on EPOS-PVD-DESK-PRO magnetron sputtering machine. The plasma flow was created by a source of ions with a cold cathode at a voltage of  $2 - 3$  kV and a current of up to 100 mA during  $3 - 5$  min. A group of plates ( $2 - 4$  pieces) was located on a rotating tool during the treatment.

HMS films were formed using an EPOS-PVD-DESK-PRO magnetron sputtering machine at a pressure of  $10^{-4}$  Pa and room temperature. The purity of  $\text{Mn}_4\text{Si}_7$  target was 99,5 %. The diameter and the thickness of the target were 76 mm and 6 mm, respectively [22, 23].

The composition and structure of the target were studied by Quanta 200 3D scanning electron microscope (SEM) from the Dutch Company FEI before placing the target into the magnetron machine (Fig. 1).

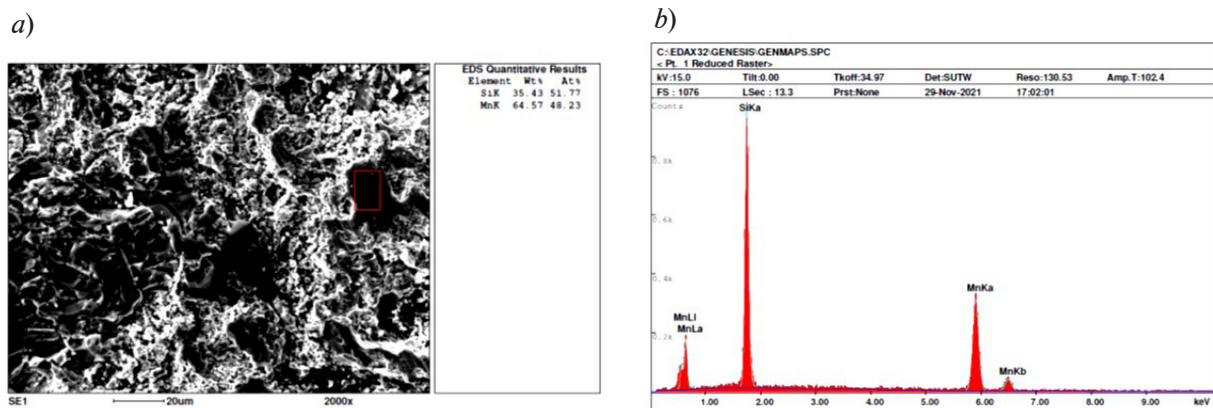


Fig. 1. The results of studying the  $\text{Mn}_4\text{Si}_7$  target with a scanning electron microscope (SEM): the surface image (a) and energy dispersive X-ray spectrum (b)

The thermoelectric properties (the resistivity and the Seebeck coefficient) of the manufactured  $\text{Mn}_4\text{Si}_7$  film were determined by placing it in a vacuum of 10 Pa, using the four-probe method and two-probe one, respectively [24].

It is known that the thermoelectric figure of merit  $ZT$  of thermoelectric materials is a dimensionless quantity determining by the following formula:

$$ZT = \alpha^2 \sigma T / \kappa, \quad (1)$$

where  $\alpha$ ,  $\mu\text{V}/\text{K}$ , is the Seebeck coefficient;  $\sigma$ ,  $\text{S}/\text{cm}$ , is the electrical conductivity;  $\kappa$ ,  $\text{W}/(\text{m}\cdot\text{K})$ , is the thermal conductivity;  $T$ ,  $\text{K}$ , is the temperature [25 – 27].

### Results and discussion

The  $\text{Mn}_4\text{Si}_7$  film formed by magnetron sputtering is in an amorphous phase before thermal annealing; this was identified by electron microscope. Fig. 2 presents the SEM-images of the  $\text{Mn}_4\text{Si}_7$  film before and after annealing. Silicon and manganese atoms deposited on silicon oxide almost completely cover the substrate. The annealing of the  $\text{Mn}_4\text{Si}_7$  film was carried out at 620 K for 1 h. at a pressure of  $10^{-3}$  Pa using an equipment. The annealed film was cooled in vacuum to room temperature.

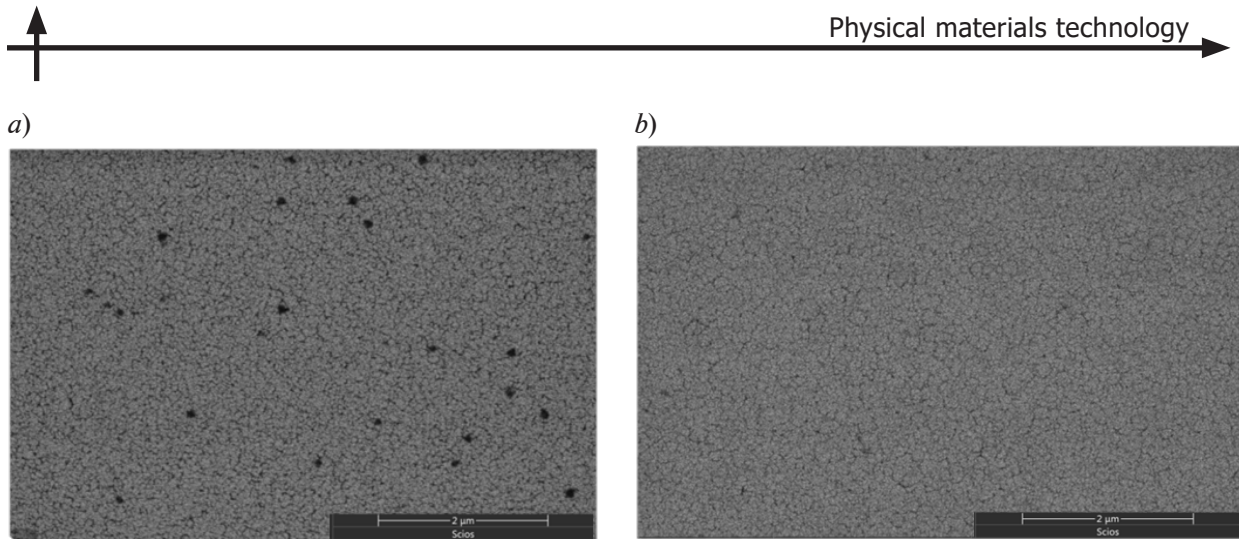


Fig. 2. SEM-images of the  $\text{Mn}_4\text{Si}_7$  film surface before (a) and after (b) annealing at 620 K and  $10^{-3}$  Pa

The resistivity of the formed  $\text{Mn}_4\text{Si}_7$  film was  $2 \cdot 10^{-5} \Omega \cdot \text{cm}$  at room temperature; when heated to a temperature of 750 K, its resistivity rose to  $5 \cdot 10^{-5} \Omega \cdot \text{cm}$  (see Fig. 3, a). The electrical conductivity of this film was  $5.0 \cdot 10^4 \text{ S/cm}$  at room temperature. As evident from Fig. 3, b, its electrical conductivity dropped to  $1.2 \cdot 10^4 \text{ S/cm}$  when heated to 750 K.

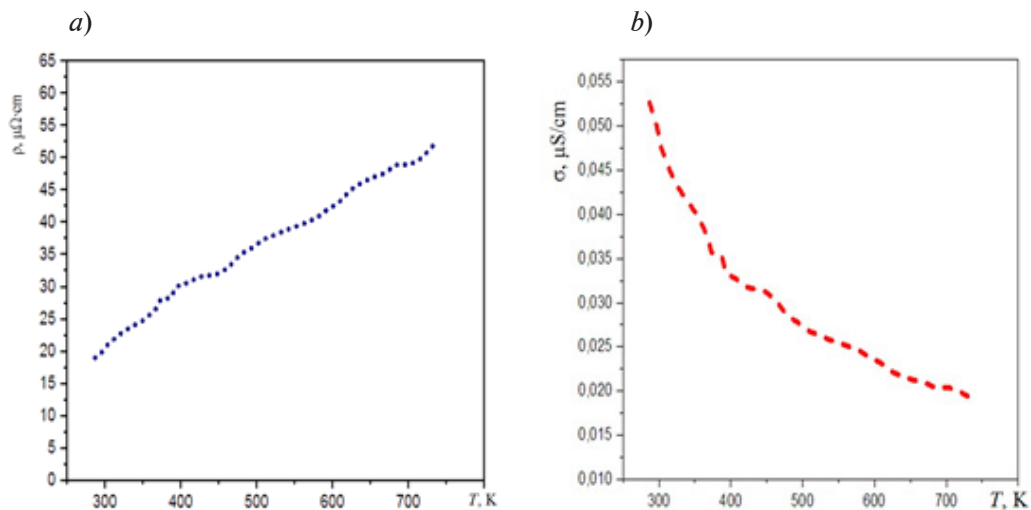


Fig. 3. Temperature dependences of the resistivity (a) and electrical conductivity (b) for the  $\text{Mn}_4\text{Si}_7$  film in amorphous phase

The graphs of the resistivity and electrical conductivity versus temperature for the  $\text{Mn}_4\text{Si}_7$  annealed film are presented in Fig. 4. It was  $7.86 \cdot 10^{-6} \Omega \cdot \text{cm}$  at room temperature, and when heated to 700 K, its resistivity dropped to  $3.90 \cdot 10^{-6} \Omega \cdot \text{cm}$  (see Fig. 4, a). The electrical conductivity of this film was  $1.2 \cdot 10^5 \text{ S/cm}$  at room temperature. When heated to 700 K, it rose to  $2.7 \cdot 10^5 \text{ S/cm}$  (see Fig. 4, b).

As can be seen from these graphs, a decrease in resistivity with increasing temperature (see Fig. 4, a) and an increase in electrical conductivity (see Fig. 4, b) confirm that the film has a polycrystalline structure.

Fig. 5 presents temperature dependences of the Seebeck coefficient for the  $\text{Mn}_4\text{Si}_7$  film in amorphous and polycrystalline phases. As evident from these graphs, the Seebeck coefficient in the case of the polycrystalline phase turned out to be approximately 6 times higher than that in the case of the amorphous one.

The thermoelectric figure of merit of the  $\text{Mn}_4\text{Si}_7$  film was calculated by Eq. (1). Thermal conductivity value of the  $\text{Mn}_4\text{Si}_7$  film was taken from Ref. [28].

The purification process concurrently enhances the  $\sigma$  and  $S$  values, and decreases the  $\kappa$  value for the  $\text{Mn}_4\text{Si}_7$  samples, leading to an extraordinarily high thermoelectric figure of merit  $ZT$ . The

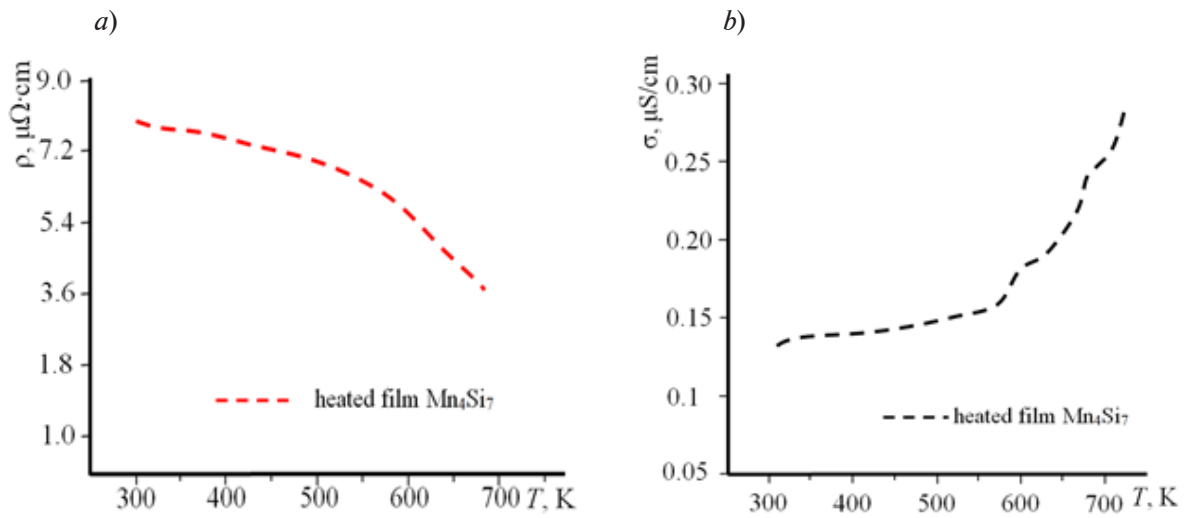


Fig. 4. Temperature dependences of the resistivity (a) and electrical conductivity (b) of the  $Mn_4Si_7$  annealed film in polycrystalline phase

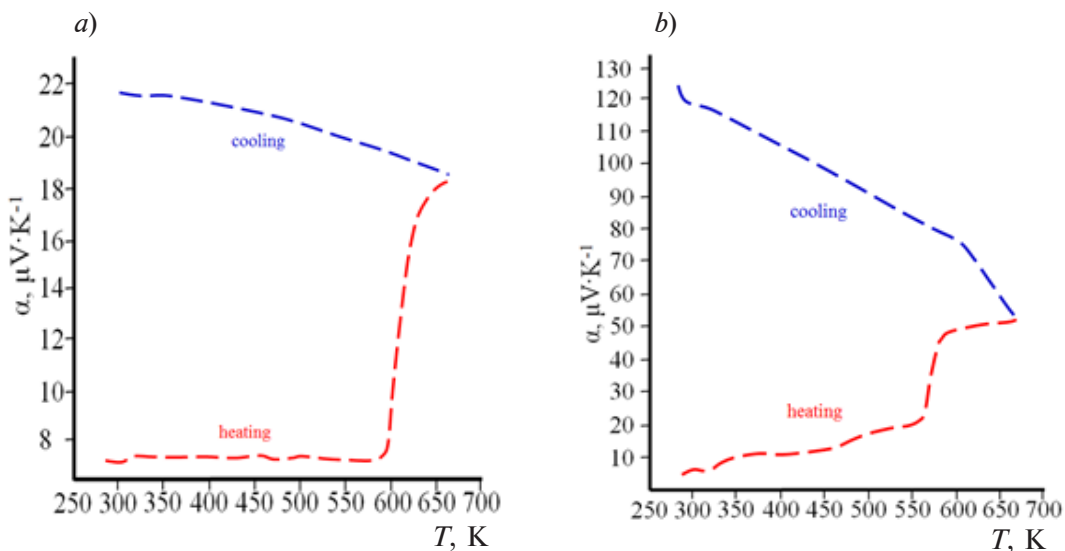


Fig. 5. Temperature dependences of the Seebeck coefficient for the  $Mn_4Si_7$  film in amorphous (a) and polycrystalline (b) phases

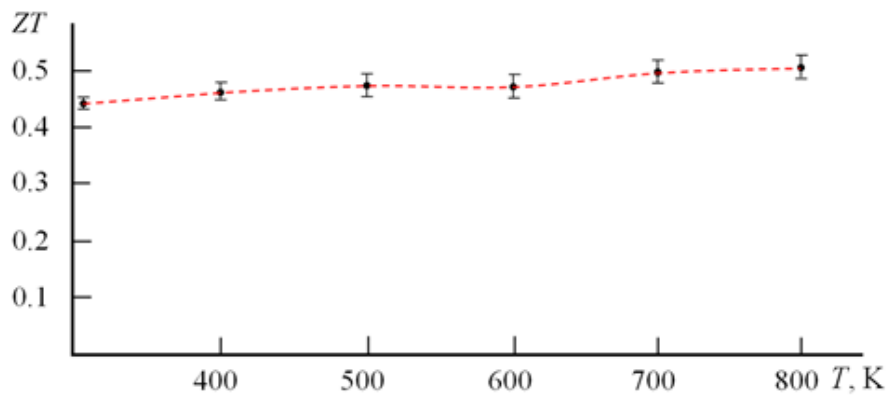


Fig. 6. Temperature dependence of  $ZT$  for the  $Mn_4Si_7$  film in polycrystalline phase





corresponding curve for the  $\text{Mn}_4\text{Si}_7$  film exhibits the maximum  $ZT$  ( $(ZT)_{\max}$ ), namely, about 0.5 at 800 K, and this is the highest value currently known and reported for thermoelectric systems (Fig. 6).

As a result of the experiments conducted with the HMS film, it was found that the thermoelectric properties of the film are better in the crystalline phase than in the amorphous one. This is due to the fact that the bond between manganese and silicon atoms is very weak and there are surface defects on the areas that are not completely covered of material. Surface defects of the film are disappeared as a result of structural relaxation and forming new bonds between the manganese and silicon atoms during the annealing, and the total structure acquires semiconductor properties.

Optical properties of the  $\text{Mn}_4\text{Si}_7/\text{SiO}_2$  film were measured using an HR-4000 high-precision spectrometer according to the law of light reflection (Fig. 7). The graphs showed that the  $\text{Mn}_4\text{Si}_7$  film had a high sensitivity in the visible and IR regions for the corresponding wavelengths. In addition, it is possible to determine the band gap of the  $\text{Mn}_4\text{Si}_7$  silicide film from these data, using the Kubelka – Munk equation. The band gap of the film is 0.66 eV. Here  $\alpha$  is the absorption coefficient,  $h\nu$  (eV) is the photon energy,  $R$  (%) is the reflection coefficient,  $\lambda$  (nm) is the light wavelength [29, 30].

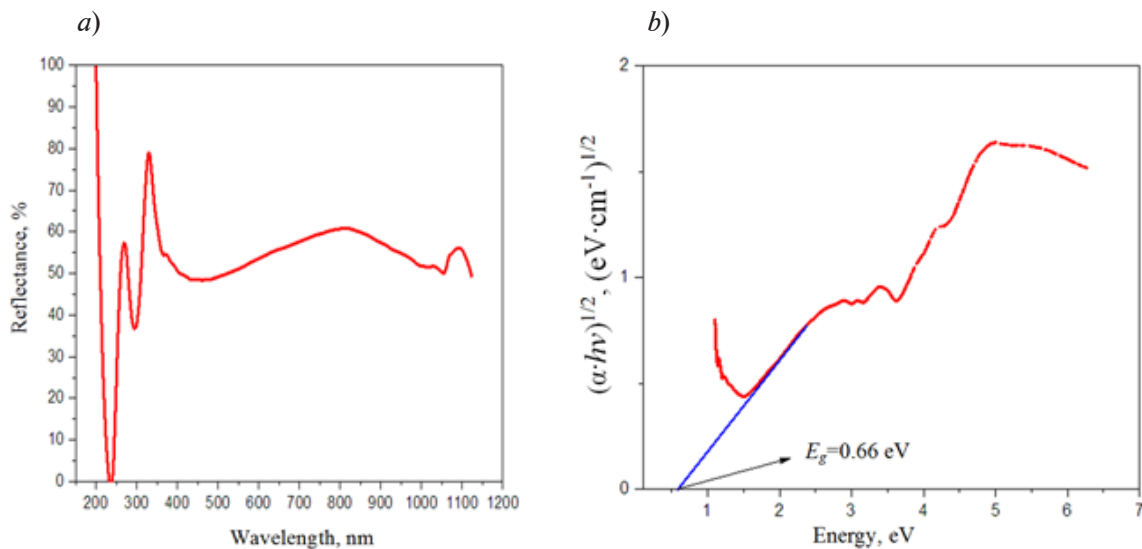


Fig. 7. The  $\text{Mn}_4\text{Si}_7$  film: the graphs of the light reflection coefficient versus the light wavelength (a) and of the absorbed photon energy versus the incident photon one (b)

The probability of absorbing a photon depends on the likelihood of having a photon and an electron interact in such a way as to move from one energy band to another. For photons which have an energy very close to that of the band gap, the absorption is relatively low since only those electrons directly at the valence band edge can interact with the photon to cause absorption. As the photon energy increases, not just the electrons already having energy close to that of the band gap can interact with the photon. Therefore, a larger number of electrons can interact with the photon and result in the photon being absorbed.

The absorption coefficient  $\alpha$  is related to the extinction coefficient  $k$  by the following formula:

$$\alpha = 4\pi k/\lambda, \quad (2)$$

Electrophysical properties of the  $\text{Mn}_4\text{Si}_7/\text{SiO}_2$  film were measured on HMS-3000 Hall Measurement System. Obtained electrophysical parameters of the  $\text{Mn}_4\text{Si}_7$  thin film are listed in Table.

Table

The obtained results on electrophysical properties of the  $Mn_4Si_7$  film

Parameter	Unit	Value
Volumetric concentration of charged particles	$cm^{-3}$	$4.5 \cdot 10^{21}$
Mobility of charged particles	$cm^2/(V \cdot s)$	1.65
Surface resistance	$\Omega$	313
Resistivity	$\Omega \cdot cm$	$7.83 \cdot 10^{-4}$
Hall coefficient	$cm^3/C$	$1.28 \cdot 10^{-3}$
Magnetoresistance	$\Omega$	$1.73 \cdot 10^{-1}$
Surface concentration of charged particles	$cm^{-2}$	$1.2 \cdot 10^{17}$
Electrical conductivity	S/cm	$1.28 \cdot 10^3$

Summary

The paper has been studied a formation of  $Mn_4Si_7$  (higher manganese silicide) films and their electrophysical properties. It was established that the thermoelectric power (the Seebeck coefficient) of the film increased during the transition from the amorphous phase to the nanocrystalline one. This effect is associated with the selective scattering of charge carriers at the boundaries of nanoclusters. The Seebeck coefficient of the film in the polycrystalline phase turned out to be approximately 6 times higher than that in the amorphous phase. The thermoelectric efficiency of  $Mn_4Si_7$  film exhibited the maximum value  $ZT_{max}$  of approximately 0.5 at 800 K.

Moreover, it was revealed that  $Mn_4Si_7$  films grown on a  $SiO_2/Si$  substrate had the highest conversion coefficient, and this is explained by the low thermal conductivity  $\kappa = 149$  W/mK of  $SiO_2/Si$ . The films of  $Mn_4Si_7$  on  $SiO_2/Si$  exhibited the high response speed, high sensitivity.

The results obtained indicate that the  $Mn_4Si_7$  films can be recommended for use in thermal wave radiation detectors in the visible and infrared electromagnetic ranges.

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