Article

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Study of Mn_4Si_7 Silicide Alloys Produced Under Different Conditions Using an X-ray Diffractometer

Mn_4Si_7 silicide crystals obtained by hot isostatic pressing (HIP) and diffusion methods were studied. As a result of the research, 11 peaks were identified in the Mn_4Si_7 crystal obtained by the HIP method, and 14 peaks in the Mn_4Si_7 crystal obtained by the diffusion method. The crystal size of Mn_4Si_7 silicide (D_{HIP}) was established from $8.8\cdot10^{-9}$ m to $3.6\cdot10^{-8}$ m, (D_{Diff}) from $6.2\cdot10^{-10}$ m to $9.1\cdot10^{-8}$ m. It has been established that the lattice tension between the atoms of the Mn_4Si_7 silicide crystal (ε_{HIP}) varies from 0.01 to 0.41, (ε_{Diff}) from 0.31 to 3.71. The dislocation density on the crystal surface (δ_{HIP}) turned out to be from $3.5\cdot10^{10}$ to $3.2\cdot10^{12}$, (δ_{Diff}) from $1\cdot10^{11}$ to $3.2\cdot10^{14}$. The degree of crystallization of Mn_4Si_7 silicide obtained by the (HIP) method is 7.02 %, the degree of amorphy is 92.98 %. It has been established that the Mn_4Si_7 silicide obtained by the diffusion method has a degree of crystallization of 9.3 % and a degree of amorphism of 90.7 %. (COD-1530134) (d). It has been established that the degree of crystallization of high-manganese silicide Mn_4Si_7 is low, and the degree of amorphy is high due to the fact that Mn and Si are bound in a non-stoichiometric state.

Keywords: diffusion, crystallization, nonstoichiometric, dislocation density, lattice tension, amorphous, agglomeration

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Introduction

Currently, the demand for electricity is growing every day, so a number of scientists are conducting research on the production of silicide materials with thermoelectric properties by various methods. As a result of the introduction of Mn atoms in the vapor phase into silicon atoms by the diffusion method, a liquid solution is formed, and after solidification, a high-manganese silicide is formed [1, 2]. In addition to the diffusion method, there are other methods, in which it is possible to form not only high manganese silicide, but also other semiconductor structures in the state of a thin film [3–12].The diffusion coefficient *D(T)* on the manganese-silicon surface is determined by (1) below.

$$D(T) = D_0 \exp(-E_m/kT), \quad (1)$$

where $D(T)$ is the solubility of manganese in silicon. $D(T) = 5 \cdot 10^{22} \cdot \exp[(6.94 - 2.78)/kT]$ cm^{-3} and diffusion $D_0 = (6.9 \pm 2.2) \cdot 10^{-4}$ cm$^2 \cdot$s^{-1}, activation energy $E_m = (0.63 \pm 0.03)$ eV, $D(T)$ diffusion coefficient from 10^{-6} to $3 \cdot 10^{-5}$ cm^2/s [3]. Mn_4Si_7 – Si film growth mechanism varies depending on the crystallization temperature [11]. A coating of manganese silicide Mn_4Si_7 was obtained in an ampoule at high temperature [13]. It was found that the distribution of Mn diffusion in Si sharply reduces the concentration of manganese at a depth of 15–20 microns [14]. Using high-energy photoelectron spectroscopy and synchrotron radiation, it was discovered that the growth of a manganese film on the Si(111)7×7 surface after the deposition of ~6 Å Mn leads to the formation of a manganese silicide film when a thin coating is applied, annealed at temperatures up to 600 °C [15]. An increase in the germanium concentration to 1 % in Mn_4Si_7 leads to the destruction of layered deposits and significant changes in thermoelectric properties [16]. The composition of $Si_{(1-x)}Mn_x$ coatings grown using a pulsed laser must be chemically homogeneous [17]. In ($MnSi_{1.71-1.75}$) nickel diffusion is reduced, the use of chromium for diffusion is effective [18]. The quality factor of Mn_xSi_{1-x}/Si in the temperature range T = 300–600 K is ZT = 0.59±0.06 [19–23]. Our work examines the preparation of high-manganese silicides Mn_4Si_7 by the diffusion method and the study of the resulting samples using an *X*-ray diffractometer (XRD-6100) SHIMADZU.

Experimental

As a result of the interdiffusion of Mn atoms with Si atoms at high temperature, a thin coating of high-manganese silicide Mn_4Si_7 was formed. The P_{Mn} calculation of the mass of manganese used to form Mn_4Si_7 during the diffusion process [1] is found from (2) below.

$$P_{Mn} = G \cdot t \cdot S. \quad (2)$$

Here G — evaporation rate (mg/(cm$^2 \cdot$s), S — evaporation surface (cm^2), t — evaporation time (minutes). Taking this ratio into account, the mass of manganese consumed for evaporation was calculated ($P_{Mn} = mg$). Growth temperature of fine Mn_4Si_7 coating was chosen to be 1100 °C. The growth rate of a thin Mn_4Si_7 layer is determined by Mn and Si diffusion (Fig. 1*A*). As a result of experiments, it was established that on a Si surface with a size of 1 cm^2 and a thickness of 0.5 cm, a thin layer of Mn_4Si_7 is formed, the thickness of which depends on temperature. HIP (hot isostatic pressing) was carried out under isostatic pressure at a temperature of 1100 °C in an argon atmosphere [2] (Fig. 1*B*).

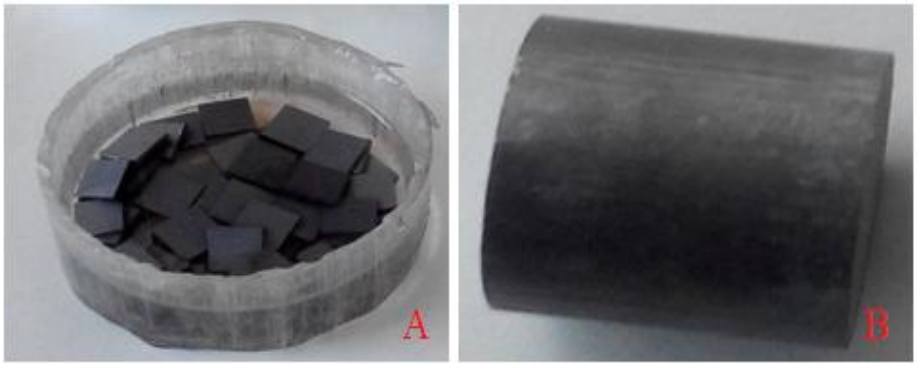
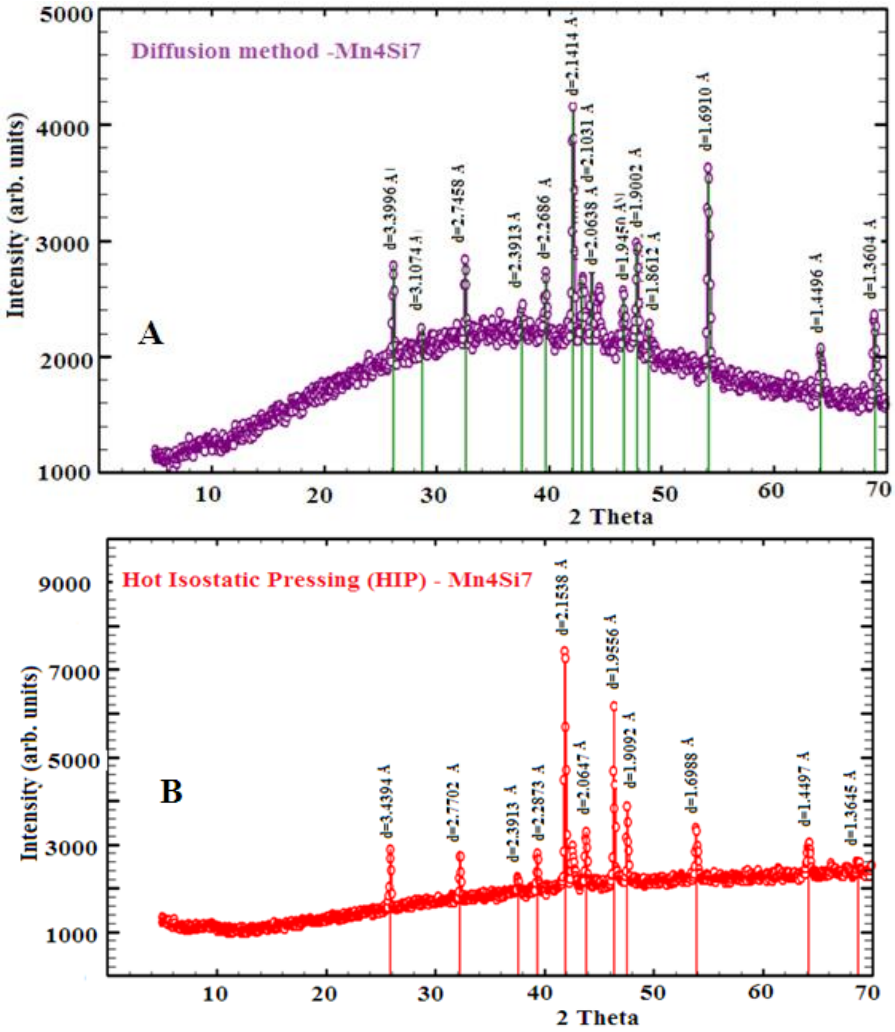


Figure 1. Mn_4Si_7 crystals obtained by diffusion and HIP methods

Results and Discussion

As a result of studying Mn_4Si_7 silicide crystals obtained by diffusion and (HIP) hot isostatic pressing on an *X*-ray diffractometer (XRD-6100) SHIMADZU, peaks corresponding to Mn_4Si_7 silicide crystals (COD-1530134) were found in the database [21]. 14 peaks from the Mn_4Si_7 silicide crystal obtained by the diffusion method (Fig. 2*A*) and 11 peaks from the Mn_4Si_7 silicide obtained by the hot isostatic pressing (HIP) method (Fig. 2*B*) were defined. The results obtained using an *X*-ray diffractometer may be due to the effect of mutual agglomeration Mn and Si atoms at high temperature.



A — obtained by the diffusion method; *B* — obtained by the hot isostatic pressing (HIP)

Figure 2. *X*-ray diffraction analysis

The difference in interatomic distance (Δd) of Mn_4Si_7 obtained by diffusion method is small from 0.01 Å to 0.14 Å compared to the interatomic distance (d) reported in the database (COD-1530134). The difference in interatomic distances (Δd) of Mn_4Si_7 obtained by hot isostatic pressing was found to be in the range from 0.01 Å to 0.05 Å (Table). This is apparently due to the influence of agglomeration and non-stoichiometric bonds at high temperatures, which can lead to an expansion or reduction of the distance between the atoms of the silicide Mn_4Si_7 (d) [20].

T a b l e

The difference in interatomic distances of Mn_4Si_7 obtained by hot isostatic pressing and obtained by the diffusion method

COD-1530134 (d) Å	Diff (d) Å	Diff (Δd) Å	HIP (d) Å	HIP (Δd) Å
3.42	3.39	–0.03	3.43	+0.01
3.24	3.10	–0.14		
2.76	2.74	–0.02	2.77	+0.01
2.44	2.39	–0.05	2.39	+0.05
2.27	2.26	–0.01	2.28	+0.01
2.15	2.14	–0.01	2.15	
1.94	1.94		1.95	+0.01
1.85	1.86	+0.01		

Based on *X*-ray diffraction analysis of Mn_4Si_7 silicide samples, the size of Mn_4Si_7 crystals was determined using the Scherrer equation [10] (3).

$$D = \frac{K\lambda}{\beta \cos\theta}. \quad (3)$$

Here *K*-0.9 is a constant depending on the shape of the crystallites, the angle at the center of the Θ-peak, λ — 0.15406 (nm) is the *X*-ray wavelength. In the β-half, the peak width of the diffraction profile is determined by calculating the maximum height of the D-size of the crystallites, which is influenced by their small size. FWHM or β_{hkl} (full width at half maximum) respectively is a mathematical way of defining a peak. This method is used to generate "peaks" which can be used to calculate the resolution of the mass spectrometer determining the spectrum being analyzed. $\beta_{hkl} = \beta_t + \beta_a$ β_t — linewidth obtained from the external dimension of the crystal (4), β_a is the line broadening due to interatomic lattice tension (5) [11].

$$\beta_t = \frac{k\lambda}{L_c \cos(\theta)}; \quad (4)$$

$$\beta_a = 4\varepsilon \operatorname{tg}(\theta). \quad (5)$$

This line broadening can be used to measure crystal size and lattice voltage. The size (*D*) of crystals of high-manganese silicide Mn_4Si_7 obtained by the diffusion method ranges from $6.2\cdot10^{-10}$ m to $9.1\cdot10^{-8}$ m. (HIP) The size (*D*) of Mn_4Si_7 silicide crystals obtained by hot isostatic pressing has been established to be from $8.8\cdot10^{-9}$m to $3.6\cdot10^{-8}$m.

It has been established that the size of Mn_4Si_7 crystals obtained by the (HIP) method is approximately 3 times smaller than that of Mn_4Si_7 silicide crystals obtained by the diffusion method. Lattice deformation or strain between Mn and Si atoms during crystal formation occurs due to high temperature and agglomeration (6).

$$\varepsilon = \frac{\beta_{hkl}}{4 * \operatorname{tg}\theta}. \quad (6)$$

Here (ε) lattice strain [3] between the atoms of Mn_4Si_7 silicide crystals obtained by the diffusion method varies from 0.31 to 3.71. It was found that the lattice strain (ε) between the atoms of Mn_4Si_7 silicide crystals obtained by hot isostatic pressing (HIP) varies from 0.01 to 0.41 (Fig. 3).

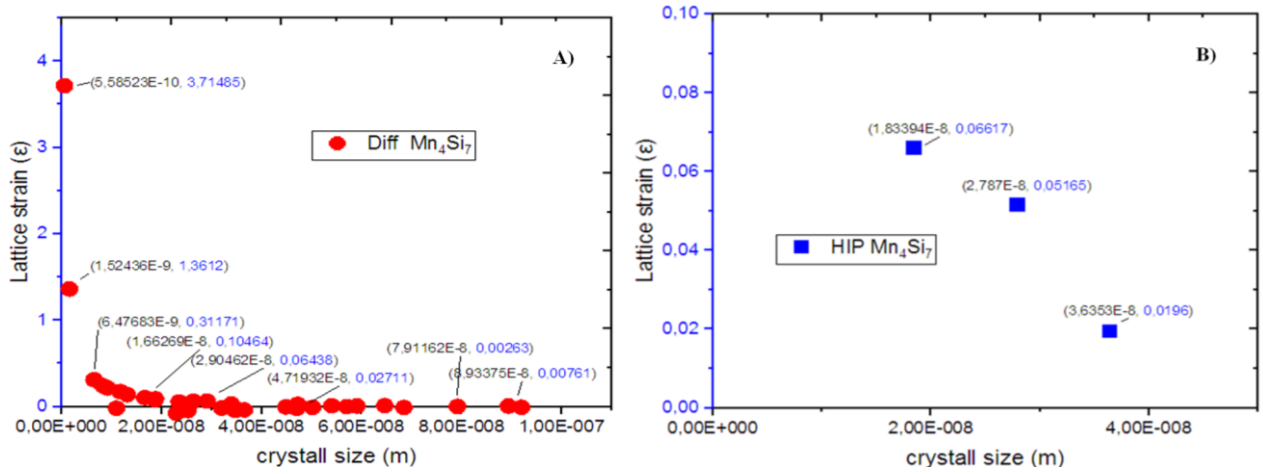


Figure 3. Dependence of Mn_4Si_7 crystal size on lattice strain obtained by the diffusion (*A*) and obtained by hot isostatic pressing method (*B*)

By comparing the results obtained in cases where high temperature alone was not sufficient to minimize lattice deformation, it was found that lattice deformation was greatest for silicides prepared by the diffusion method. It has been established that for silicides produced together with high temperature and high pressure, lattice deformation is 15–20 times less. Lattice dislocations arise as a result of the formation of crystals of high-manganese silicide Mn_4Si_7, which have different sizes during formation and deformation in the crystal

lattice. Types of dislocations include edge and screw dislocations. The density of dislocations is determined (7) [4].

$$\delta = \frac{1}{D^2}. \quad (7)$$

The dislocation can be moved by the sliding method and the diffusion method; Dislocations perpendicular to the displacement vector move by diffusion, causing growth or compression of the plane as a result of expulsion by diffusion. The dislocation density (δ) on the surface of high-manganese silicide Mn_4Si_7 obtained by the diffusion method ranges from $1 \cdot 10^{11}$ to $3.2 \cdot 10^{14}$. It has been established that the dislocation density of Mn_4Si_7 silicide (δ), obtained by the (HIP) method, ranges from $3.5 \cdot 10^{10}$ to $3.2 \cdot 10^{12}$ (Fig. 4).

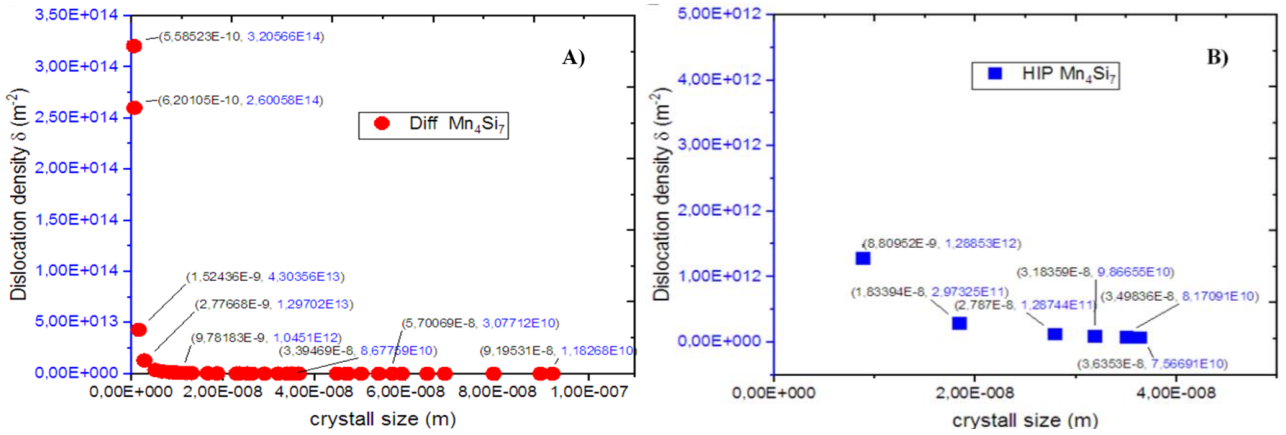


Figure 4. Established that the dislocation density of Mn_4Si_7 silicide, obtained by the diffusion method (*A*) and obtained by the (HIP) method (*B*)

The degree of crystallinity of the Mn_4Si_7 was calculated using the program (*Match-3!*). Calculations showed that the degree of crystallization of Mn_4Si_7 obtained by the diffusion method is 9.3 %, the degree of amorphism is 90.7 %, the degree of crystallization of Mn_4Si_7 obtained by the (HIP) method is 7.02 %, degree of amorphy 92.98 %. Mn_4Si_7 has five positions for Mn atoms and four for Si atoms as indicated by (mp-680339), which is the reason for its high degree of amorphism. 1) Mn^{3+} is bonded to ten Si+1.71 atoms. Mn–Si bond distances range from 2.27–2.71 Å.

Mn^{3+} is bonded to eight Si+1.71- atoms in an 8-coordinate geometry. Mn–Si bond distances range from 2.27–2.56 Å. Mn^{3+} is bonded to eight Si+1.71- atoms in an 8-coordinate geometry. Mn–Si bond distances range from 2.28–2.52 Å. Mn^{3+} is bonded to eight Si+1.71- atoms in an 8-coordinate geometry. There are four shorter (2.36 Å) and four longer (2.38 Å) Mn–Si bonds. 5) Mn^{3+} is bonded to eight Si+1.71- atoms in an 8-coordinate geometry. There is a spread of Mn–Si bond distances within the range of 2.32–2.44 Å. For Si there are four equivalent states Si+1.71.

In the Si+1.71- state, Si+1.71- is bonded to five Mn^{3+} atoms in a 4-coordinate geometry. In the Si+1.71- state, Si+1.71- is bonded to four Mn^{3+} atoms in a 4-coordinate geometry. In the Si+1.71- state, Si+1.71- is bonded to five Mn^{3+} atoms in a 5-coordinate geometry. In the Si+1.71- state, Si+1.71- is bonded to five Mn^{3+} atoms in a 5-coordinate geometry. According to the data obtained, Mn_4Si_7 silicides are formed using non-stoichiometric Mn and Si bonds. Based on this, the Mn_4Si_7, obtained by the diffusion and (HIP) method, has a high degree of amorphism and a low degree of crystallization, therefore the Mn_4Si_7 silicide alloy has a generally polycrystalline structure. Consequently, Mn_4Si_7 polycrystals are isotropic due to the random orientation of individual crystals and have the characteristics of an amorphous material [8]. This is shown by *X*-ray diffraction analysis (XRD-6100) (Fig. 5).

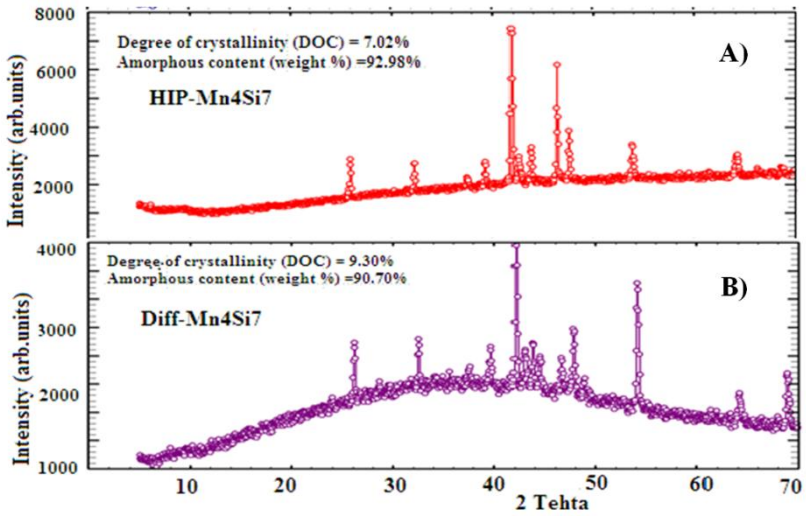


Figure 5. Degree of crystallization of silicide Mn_4Si_7 obtained by the (HIP) method (*A*) and obtained by the diffusion method (*B*)

Crystallization requires the interaction of Mn and Si particles and the formation of crystalline bridges between the particles as a result of agglomeration at high temperature. After this process, a stable particle or agglomerate is formed. Due to the formation of Mn_4Si_7 silicides using non-stoichiometric bonds, they exhibit electrophysical properties characteristic of semiconductors.

Conclusions

A study of Mn_4Si_7 silicide crystals obtained by (HIP) and diffusion methods showed that there are 11 peaks of Mn_4Si_7 obtained by the (HIP) method and 14 peaks of Mn_4Si_7 obtained by the diffusion method. It has been established that the size of Mn_4Si_7 silicide crystals (D_{HIP}) is from $8.8\cdot10^{-9}$ m to $3.6\cdot10^{-8}$ m, (D_{Diff}) from $6.2\cdot10^{-10}$ m to $9.1\cdot10^{-8}$ m. It has been determined that lattice tension between the atoms of the Mn_4Si_7 silicide crystal (ε_{HIP}) varies from 0.01 to 0.41, (ε_{Diff}) from 0.31 to 3.71. The dislocation density on the crystal surface (δ_{HIP}) turned out to be in the range from $3.5\cdot10^{10}$ to $3.2\cdot10^{12}$, (δ_{Diff}) from $1\cdot10^{11}$ to $3.2\cdot10^{14}$. The degree of crystallization of Mn_4Si_7 silicide obtained by the (HIP) method is 7.02 %, degree of amorphy — 92.98 %. (COD-1530134) Compared with the interatomic distance (d) in Mn_4Si_7 silicide obtained by diffusion method, the interatomic distance difference (Δd) is shorter from 0.01 Å to 0.14 Å. (HIP) Interatomic distance difference (Δd) of Mn_4Si_7, obtained by hot isostatic pressing was found to be in the range from 0.01 Å to 0.05 Å. It has been established that the degree of crystallization of high-manganese silicide Mn_4Si_7 is low, and the degree of amorphy is high due to the fact that Mn and Si are bound in a non-stoichiometric state.

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Рентгендік дифрактометр арқылы түрлі жағдайларда өндірілген Mn_4Si_7 силициддің қорытпаларын зерттеу

Ыстық изостатикалық престеу (ЫИП) және диффузиялық әдістермен алынған Mn_4Si_7 силицид кристалдары зерттелді. Зерттеу нәтижесінде ЫИП әдісімен алынған Mn_4Si_7 кристалында 11 шың, диффузиялық әдіспен алынған Mn_4Si_7 кристалында 14 шың анықталды. Mn_4Si_7 силицидінің (D_{HIP}) кристалдық өлшемі $8{,}8\cdot10^{-9}$ м-ден $3{,}6\cdot10^{-8}$ м-ге дейін, (D_{Diff}) $6{,}2\cdot10^{-10}$ м-ден $9{,}1\cdot10^{-8}$ м-ге дейін белгіленді. Mn_4Si_7 силицид кристалының (ε_{HIP}) атомдары арасындағы тордың кернеуі 0,01-ден 0,41-ге дейін, (ε_{Diff}) 0,31-ден 3,71-ге дейін өзгеретіні айқындалды. Кристалл бетіндегі дислокация тығыздығы (δ_{HIP}) $3{,}5\cdot10^{10}$-нан $3{,}2\cdot10^{12}$-ге дейін, (δ_{Diff}) $1\cdot10^{11}$-ден $3{,}2\cdot10^{14}$-ке дейін болды. (ЫИП) әдісімен алынған Mn_4Si_7 силицидінің кристалдану дәрежесі 7,02 %, аморфия дәрежесі 92,98 %. Диффузия әдісімен алынған Mn_4Si_7 силицидінің кристалдану дәрежесі 9,3 % және аморфизм дәрежесі 90,7 % болатыны анықталды. (КОД-1530134) (ж.). Mn_4Si_7 жоғары марганец силицидінің кристалдану дәрежесі төмен және Mn және Si стехиометриялық емес күйде байланысқандықтан аморфтылық дәрежесі жоғары екені айқындалды.

Кілт сөздер: диффузия, кристалдану, стехиометриялық емес, дислокация тығыздығы, тордың керілуі, аморфты, агломерация

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Исследование сплавов силицида Mn_4Si_7, полученных в различных условиях, с помощью рентгеновского дифрактометра

Исследованы кристаллы силицида Mn_4Si_7, полученные методами горячего изостатического прессования (ГИП) и диффузии. В результате исследования в кристалле Mn_4Si_7, полученном методом ГИП, обнаружено 11 пиков, а в кристалле Mn_4Si_7, полученном методом диффузии, — 14 пиков. Определен размер кристаллов силицида Mn_4Si_7 (D_{HIP}) от $8{,}8\cdot10^{-9}$ м до $3{,}6\cdot10^{-8}$ м, (D_{Diff}) от $6{,}2\cdot10^{-10}$ м до $9{,}1\cdot10^{-8}$ м. Установлено, что решеточное напряжение между атомами кристалла силицида Mn_4Si_7 (ε_{HIP}) изменяется от 0,01 до 0,41, (ε_{Diff}) — от 0,31 до 3,71. Плотность дислокаций на поверхности кристалла (δ_{HIP}) составляла от $3{,}5\cdot10^{10}$ до $3{,}2\cdot10^{12}$, а (δ_{Diff}) — от $1\cdot10^{11}$ до $3{,}2\cdot10^{14}$. Степень кристаллизации силицида Mn_4Si_7, полученного методом (ГИП), составляет 7,02 %, а степень аморфности — 92,98 %. Установлено, что степень кристаллизации силицида Mn_4Si_7, полученного диффузионным методом, составила 9,3 %, а степень аморфности — 90,7 %. (КОД-1530134) (г). Установлено, что степень кристаллизации высокомарганцевого силицида Mn_4Si_7 низкая, а степень аморфности высокая из-за того, что Mn и Si связаны в нестехиометрическом соотношении.

Ключевые слова: диффузия, кристаллизация, нестехиометрический, плотность дислокаций, деформация решетки, аморфный, агломерация

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