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Original article

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INFLUENCE OF RAPID THERMAL TREATMENT ON THE MECHANICAL PROPERTIES OF SUBMICROSTRUCTURES BASED ON NICKEL AND CHROME FILMS

Abstract. The results of a study of the phase composition, surface morphology, grain size and mechanical properties of submicrostructures based on chromium and nickel before and after rapid thermal treatment (RTT) at temperatures from 200 to 550 °C are presented. Surface morphology and grain size were determined using atomic force microscopy. Mechanical properties were determined by nanoindentation. Rapid thermal treatment of nickel and chromium films significantly affects the change in phase composition, surface morphology, grain size and properties. The formation of silicides (according to the diffusion mechanism) and new phases occurs in the films: the $CrSi_2$ phase is formed at temperatures of 350 °C and above, the Ni_2Si phase at 300 °C, and the NiSi phase at 350 °C and above. When the phase composition changes, the grain size increases. In the RTT ranges from 200 to 300 °C and from 450 to 550 °C for chromium-based submicrostructures, the correlation between microhardness and grain size is carried out according to the Hall–Petch law – microhardness increases with decreasing grain size. For nickel-based submicrostructures, the Hall–Petch law is satisfied in the temperature range from 200 to 300 °C and from 500 to 550 °C. In the temperature range of 300–450 °C for chromium-based submicrostructures and 300–500 °C for nickel-based submicrostructures, microhardness decreases with decreasing grain size and vice versa, i.e. a "negative Hall–Petch effect" occurs. This effect is associated with the phase transitions $Cr \rightarrow CrSi_2$ and $Ni \rightarrow Ni_2Si \rightarrow NiSi$, restructuring of submicrostructures due to the diffusion mechanism, morphological rearrangement of vacancy defects and annealing of point defects inside grains, as well as the corresponding reconstruction of grain boundaries. The considered submicrostructures based on chromium and nickel can be used in microelectronics for Schottky diodes, ohmic contacts and gates.

Keywords: thin films, nickel and chrome silicides, silicon substrate, rapid thermal treatment, grain size, mechanical properties, atomic force microscopy, nanoindentation

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ВЛИЯНИЕ БЫСТРОГО ТЕРМИЧЕСКОГО ОТЖИГА НА МЕХАНИЧЕСКИЕ СВОЙСТВА СУБМИКРОСТРУКТУР НА ОСНОВЕ ПЛЕНОК НИКЕЛЯ И ХРОМА

Аннотация. Представлены результаты исследования фазового состава, морфологии поверхности, размера зерна и механических свойств субмикроструктур на основе хрома и никеля до и после быстрой термической обработки при температуре от 200 до 550 °C. Морфологию поверхности и размер зерна определяли с помощью атомно-силовой микроскопии, механические свойства – методом наноиндентирования. Быстрая термическая обработка субмикроструктур на основе никеля и хрома существенно влияет на изменение фазового состава, морфологии поверхности, размера зерна и свойств. Происходит формирование силицидов (по диффузионному механизму) и новых фаз: фаза $CrSi_2$ формируется при температуре 350 °C и выше, фаза Ni_2Si – при 300 °C, а фаза $NiSi$ – при 350 °C и выше. При изменении фазового состава происходит рост размера зерна. В диапазонах БТО от 200 до 300 °C и от 450 до 550 °C для субмикроструктур на основе хрома корреляция микротвердости и размера зерна выполняется согласно закону Холла–Петча – микротвердость растет с уменьшением размера зерна. Для субмикроструктур на основе никеля закон Холла–Петча выполняется в диапазоне температур от 200 до 300 °C и от 500 до 550 °C. В диапазоне температур 300–450 °C для субмикроструктур на основе хрома и 300–500 °C для субмикроструктур на основе никеля микротвердость снижается с уменьшением размера зерна и наоборот, то есть происходит «отрицательный эффект Холла–Петча». Такой эффект связан с фазовыми переходами Cr → $CrSi_2$ и Ni → Ni_2Si → NiSi, реструктуризацией субмикроструктур из-за диффузионного механизма, морфологической перестройки вакансионных дефектов и отжигом точечных дефектов внутри зерен, а также соответствующей реконструкцией межзеренных границ. Рассмотренные субмикроструктуры на основе хрома и никеля можно применять в микроэлектронике для диодов Шоттки, омических контактов и затворов.

Ключевые слова: тонкие пленки, силициды никеля и хрома, кремниевая подложка, быстрая термическая обработка, размер зерна, механические свойства, атомно-силовая микроскопия, наноиндентирование

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Introduction. Transition metal silicides are good electrical conductors with resistivity comparable to that of metals and metal alloys. They also have good thermoelectric properties [1]. These silicides find application as: the Schottky barriers and ohmic contacts [2], gate and interconnect metals, epitaxial conductors in heterostructures [3] since they have lower electrical resistance than polycrystalline silicon and are compatible with the silicon substrate.

Some of the widely available metals for the formation of silicides are Cr and Ni. A common method for obtaining silicides based on these metals is rapid heat treatment of the metal layer on the surface of the silicon wafer. The reaction of Cr and Si results in the formation of the $CrSi_2$ phase at temperatures of 350 °C and above [4, 5]. During heat treatment of Ni on Si, Ni_2Si silicides are formed (at temperatures of 200–300 °C) with subsequent phase transition to NiSi and $NiSi_2$ at temperatures above 350 °C [6]. Silicides based on Cr and Ni are used to create barriers for Schottky diodes, interconnections and contacts in integrated circuits [6–10].

Studies of the properties of Cr and Ni silicides are mainly focused on their phase composition and electrophysical characteristics. Mechanical properties (which are not unimportant) have been studied to a small extent or for a very long time [11, 12]. When studying the mechanical properties of nickel silicides, difficulties arose due to the brittleness of the materials obtained [13], and chromium silicide study was carried out mainly for bulk materials [14]. The elastic modulus of $CrSi_2$ was determined by the ultrasonic method and calculated using the first principles method [15]. The use of a modern nanoindentation method and small loads (from µN to mN) allows working with small thicknesses and volumes of material [16], which opens up opportunities for determining the mechanical properties of thin nanometer films.

The *aim of the work* is to determine the mechanical properties of submicrostructures based on nickel and chromium films before and after rapid thermal treatment at temperatures from 200 to 550 °C.

Materials and research methods. Chromium (Cr) and nickel (Ni) films were deposited on silicon substrates by magnetron sputtering of 99.5 % pure chromium and nickel targets in 99.993 % pure argon using an SNT Sigma installation (StratNanoTek Invest, Belarus) [17, 18]. For chromium films, the discharge pressure and power during deposition were 0.5 Pa and 5.1 kW (the power density was about 5.85 W/cm^2 at a discharge voltage of 680 V), respectively, and for nickel films – 0.35 Pa and 7.1 kW (the power density was about 8.15 W/cm^2 at a discharge voltage of 480 V), respectively. The silicon substrates were epitaxial layers of phosphorus-doped silicon with a resistivity of 0.58–0.63 Ohm · cm and a thickness of 5.3–5.8 µm, formed on substrates of *p*-type single-crystal silicon with a resistivity of 10 Ohm · cm and orientation (111). Before deposition of Ni and Cr films, the substrates were treated first in an ammonium peroxide solution and then in an aqueous HF solution.

Then the substrates with Cr and Ni films were subjected to rapid thermal treatment (RTT) in the heat balance mode by irradiating the back side of the substrates with an incoherent light flux of constant power quartz halogen lamps in a nitrogen environment for 7 s until the temperature reached 200 to 550 °C using a JetFirst 100 setup (Jipelec Qualiflow, France). The temperature of the working side of the substrate was controlled by a thermocouple with an accuracy of ± 0.5 °C.

The phase composition was studied by X-ray diffraction (XRD) analysis using an ULTIMA IV diffractometer (Rigaku, Tokyo, Japan).

Film thickness was determined by scanning electron microscopy (SEM) on an S-4800 device (Hitachi, Tokyo, Japan). To determine the film thickness, a vertical cleavage of a silicon wafer with a film was made and its SEM image was obtained.

The surface structure of Cr- and Ni-based submicrostructures before and after RTA was studied on a Dimension FastScan atomic force microscope (AFM) (Bruker, USA) in the PeakForce QNM mode using a standard CSG10_SS silicon cantilever (TipsNano, Russia) with a tip radius of 5 nm and a cantilever stiffness of 0.3 N/m.

The physical and mechanical properties (elastic modulus E and microhardness H) were determined on a Hysitron 750 Ubi nanoindenter (Bruker, USA). Nine indentations were performed with a load of 50 µN (for Cr-based submicrostructures) and 100 µN (for Ni-based submicrostructures). The indentation depth did not exceed 1/10 of the film thickness in order to exclude the influence of the substrate on the E and H values.

Results and discussion. SEM images of cross sections of samples with films showed an increase in thickness with increasing RTT temperature (Fig. 1, 2). The initial film thickness was: for Cr – 27.8 nm, for Ni – 59.5 nm. A significant increase in thickness occurs after RTT at 350–400 °C for Cr films (up to 66.1 nm) and after RTT at 300–350 °C for Ni films (up to 139 nm). RTT of nickel and chromium films also significantly affects the change in phase composition, surface morphology, grain size and electrophysical properties. These changes are described in more detail in [17, 18]. Let us note the main points:

1. During RTT, silicides and new phases are formed. Thus, in Cr-based films, the $CrSi_2$ phase is formed at temperatures of 350 °C and above (Fig. 1 *c*, *d*) [17]. In Ni-based films, the Ni_2Si phase is formed first at 300 °C, and then the NiSi phase at 350 °C and above (Fig. 2, *b*–*d*) [18]. Phase transitions are the main reason for the formation of new submicrostructures, changes in surface morphology, grain size, and electrophysical properties.

2. In the RTT temperature range from 400 to 550 °C, the surface of the Cr/Si structure acquires a wavy texture (see Figure 1 *c*, *d*), caused by the phase transition of the Cr film into the $CrSi_2$ layer with a large lattice parameter and formed due to the diffusion of silicon atoms from the substrate into the

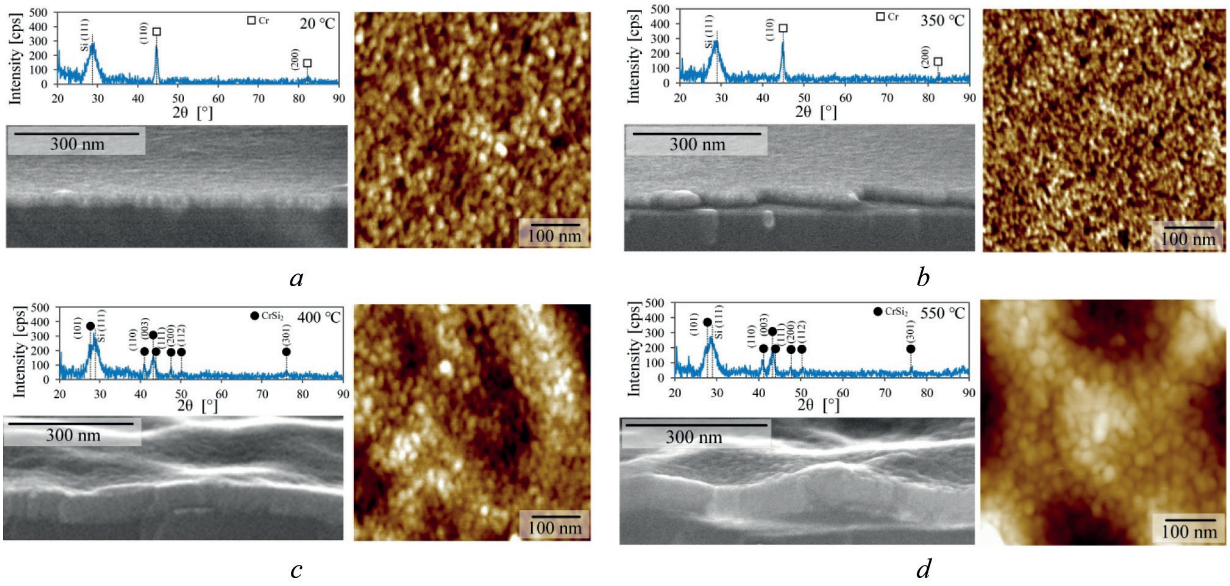


Fig. 1. X-ray phase analysis, SEM images of a cross section and AFM images of the surface of heterostructures based on chrome films after RTT: *a* – initial film; *b* – at 350 °C; *c* – at 400 °C; *d* – 550 °C

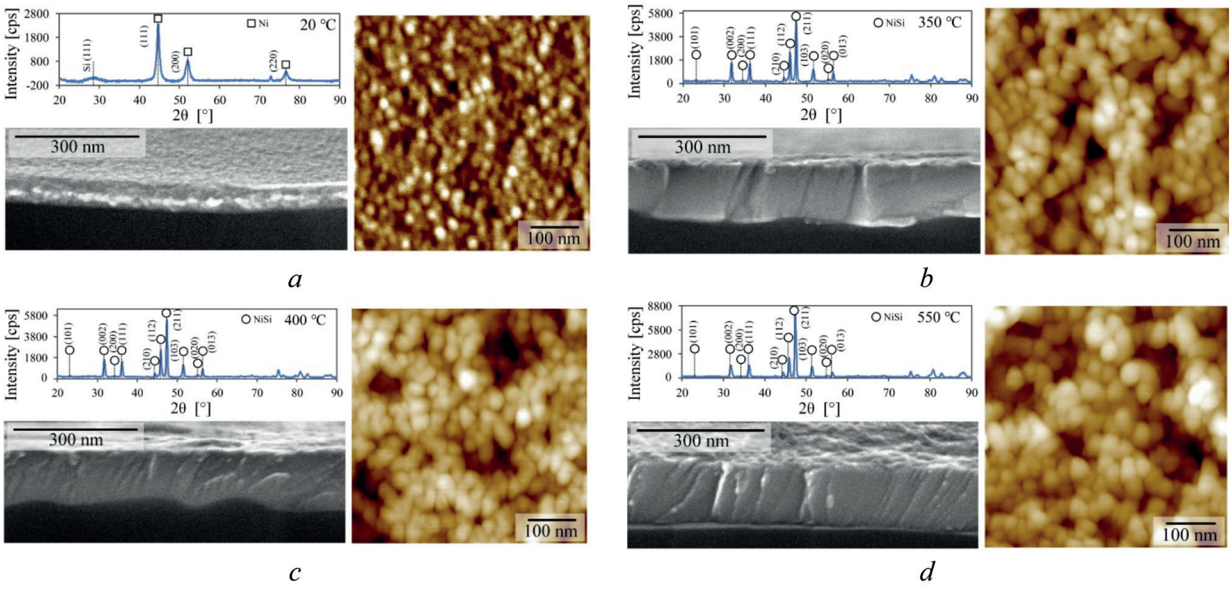


Fig. 2. X-ray phase analysis, SEM images of a cross section and AFM images of the surface of heterostructures based on nickel films after RTT: *a* – initial film; *b* – at 350 °C; *c* – at 400 °C; *d* – 550 °C

growing layer. These phase changes are accompanied by an increase in the size of the crystalline grains and cause an increase in the surface roughness parameters, specific surface energy and specific resistance of the $CrSi_2$ layers [17]. RTT at temperatures of 200–250 °C leads to coarsening of the nickel film grains due to the formation of the Ni_2Si phase, and then the NiSi phase (see Figure 2, *b*–*d*). In this case, there is an increase in roughness and average grain size, and the specific electrical resistance decreases.

3. The formation of Cr and Ni silicides occurs by a diffusion mechanism – diffusion of Ni atoms into the substrate and diffusion of silicon atoms from the substrate into the Cr film [8, 10, 17–22].

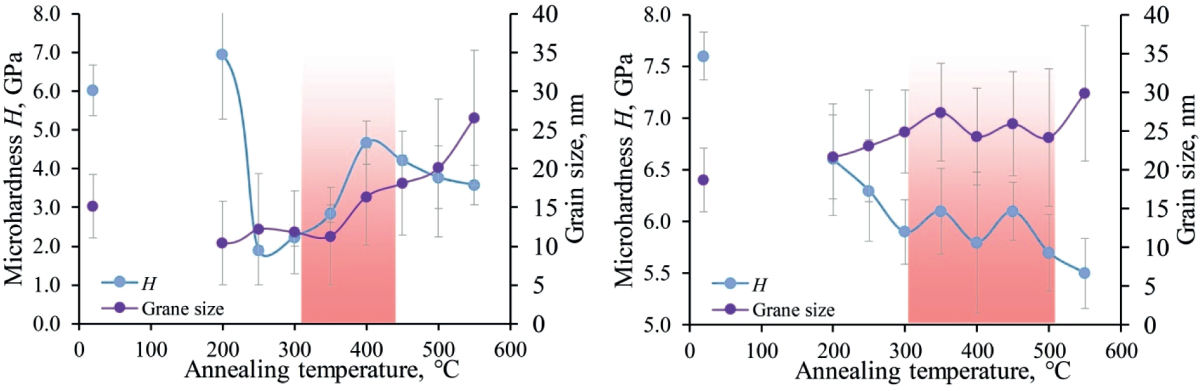
The mechanical properties (elastic modulus *E* and microhardness *H*) of submicrostructures based on nickel and chromium films before and after RTT are shown in Table and Figure 3. On Cr-based submicrostructures, there is an almost direct dependence of the elastic modulus on the grain size (see Table), while on Ni-based submicrostructures there is no correlation (see Table). The elastic modulus

Grain size and elastic modulus of submicrostructures based on nickel and chromium films before and after RTT

RTT temperature, °C	Cr-based submicrostructures		Ni-based submicrostructures	
	Grain size, nm	Elastic modulus, *E*, GPa	Grain size, nm	Elastic modulus *E*, GPa
Initial	15.2 ± 1.4	254 ± 37	18.7 ± 4.1	170 ± 3
200	10.4 ± 0.9	203 ± 23	21.7 ± 5.4	95 ± 4
250	12.2 ± 1.1	74 ± 11	23.1 ± 7.2	149 ± 15
300	11.8 ± 1.1	118 ± 89	24.9 ± 5.3	129 ± 19
350	11.3 ± 1.0	152 ± 16	27.4 ± 6.3	179 ± 20
400	16.4 ± 1.5	156 ± 23	24.3 ± 6.3	152 ± 23
450	18.1 ± 1.6	131 ± 14	25.9 ± 6.7	141 ± 15
500	20.1 ± 1.8	94 ± 21	24.8 ± 8.9	161 ± 18
550	26.6 ± 2.4	116 ± 31	29.9 ± 8.7	127 ± 12

changes in the range of values: for Cr – from 74 to 254 GPa, for Ni – from 95 to 170 GPa. In this case, the maximum values were obtained on the original submicrostructures.

In the RTT ranges from 200 to 300 °C and from 450 to 550 °C for Cr-based submicrostructures, the correlation of microhardness and grain size is performed according to the Hall–Petch law [16, 23] (Figure 3, *a*) – microhardness increases with decreasing grain size. The same thing happens for Ni-based submicrostructures in the temperature range from 200 to 300 °C and from 500 to 550 °C (see Figure 2, *b*). The “negative Hall–Petch effect” [16, 24] (i.e. microhardness decreases with decreasing grain size and vice versa) occurs in the temperature range of 300–450 °C for Cr and 300–500 °C for Ni (see Figure 3, red areas on the graphs).



a *b*

Fig. 3. Dependence of microhardness and grain size on RTT temperature for heterostructures based on chromium (*a*) and nickel (*b*) films

The maximum microhardness of 5.1 GPa was obtained for the initial nickel-based submicrostructure without heat treatment. For chromium-based submicrostructures, the maximum microhardness was 6.9 GPa at 200 °C. The failure to observe the Hall–Petch law in the above temperature ranges (see Figure 3, red areas on the graphs) is probably associated with the phase transitions Cr → $CrSi_2$ and Ni → Ni_2Si → NiSi [17, 18], film restructuring due to the diffusion mechanism, morphological rearrangement of vacancy defects in the Ni film [25], and annealing of point defects inside the grains of polycrystalline Cr and the corresponding reconstruction of grain boundaries [26].

Conclusion. The study examined the effect of the rapid thermal treatment at temperatures from 200 to 550 °C on the microstructure and mechanical properties of the submicrostructures based on nickel and chromium films. Silicides (by the diffusion mechanism) and new phases are formed in the films: the $CrSi_2$ phase at temperatures of 350 °C and above, the Ni_2Si phase at 300 °C, and then the NiSi phase at 350 °C and above. When the phase composition changes, the grain size increases and the surface morphology changes. The elastic modulus changes in the following range: for Cr – from 74 to 254 GPa, for Ni – from 95 to 170 GPa. In the RTT ranges from 200 to 300 °C and from 450 to 550 °C for Cr-based submicrostructures, the correlation of microhardness and grain size is performed according to the Hall–

Petch law. For Ni-based submicrostructures, the Hall-Petch law is valid in the temperature range of 200 to 300 °C and 500 to 550 °C. The “negative Hall–Petch effect” occurs in the temperature range of 300–450 °C for chromium submicrostructures and 300-500 °C for the nickel submicrostructures (associated with the phase transitions, restructuring, rearrangement of vacancy defects, annealing of point defects within grains and reconstruction of grain boundaries).

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