Distribution of Molybdenum Atoms over Depth of the Surface Layer of a Niobium Single Crystal Produced by Ion Bombardment

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One of the fundamental directions in the use of ion implantation of metals is the creation of surface alloys of any composition. And their quantitative characteristic is the distribution of atoms of the impurity element in depth of the surface layer of the metal substrate [1-5]. In the study of the contribution of each component in changing the electronic structure, emission-adsorption and catalytic properties of multicomponent metal alloys, the creation of particularly pure metal solid solutions, as well as the determination of the actual concentration of alloying elements and their distribution in the surface layer of crystals are of great value. To study the distribution profiles of the concentration of ion-implanted atoms of refractory metals in alloys based on refractory metals, the method of layer-by-layer analysis using Auger electron spectroscopy in conjunction with an argon ion cannon is used. In this work, on the example of implantation of low-energy molybdenum ions into a single crystal of niobium facet (100), the features of creating surface alloys based on refractory metals with a low concentration of the alloying element and the distribution of the ion-implanted metal in the near-surface region of the crystal are studied [5-9].

1. INTRODUCTION

Today, silicon films play an important role in the development of electronics and microelectronics. The construction of multifunctional ultra-high vacuum installation with a solid-state ion source is described in [2]. When using refractory metals as an ion emitter, special attention should be paid to the presence of easily evaporable impurities in them. Even in well-purified single crystals by zone melting, ions of impurity elements can be observed in large quantities in the initial stage of the source operation.

2. DESCRIPTION OF OBJECTS AND INVESTIGATION METHODS

To separate emitter ions from impurity ions, we designed and manufactured an electromagnetic ion separator. The diameter of inlet and outlet slots of the separator was ~ 8 mm. The operation of the solid-state ion cannon that we used is based on the electron impact ionization of the working substance atoms. This source makes it possible to obtain a stream of accelerated ions of any refractory metal. One of the limitations of obtaining ions of refractory metals with a high density is the creation of a sufficiently high vapor pressure of these metals in the source ionization chamber. In this construction of the ion cannon, we obtained an integral ion current on the target of ~ $5 \cdot 10^{-8}$ A. In this case, the vacuum in the device chamber was not worse than ~ 10^{-7} Torr. The operating vacuum in the installation chamber was $(3-5) \cdot 10^{-10}$ Torr. The ion current was measured with a Faraday cylinder installed in the manipulator of the experimental setup. When using molybdenum as an implanter, the integral ion current was ~$2 \cdot 10^{-8}$ A. The flux of molybdenum ions in this mode was ~ 10^{-11} ion·cm^{-2}·s^{-1} and the ion energy was $E_p = 3$ keV.

We installed cathode units with niobium single crystal facet (100) in the manipulator of the experimental setup. Obtaining an atomically pure surface of niobium single crystals by high-temperature heating is described in [3]. Changes in the elemental composition of the crystal surface were controlled by taking Auger spectra, measuring the thermal emission current and determining the operation of the crystal output. When studying the distribution of molybdenum atoms in the near-surface region of ion-doped niobium, we used a molybdenum single crystal as a test object. It is due to the possibility of comparing the Auger peaks of pure molybdenum and the target doped with molybdenum ions from a single crystal of niobium (100).

The study of the distribution profiles of the concentration of doped molybdenum ions over the depth of niobium was carried out as follows. After reaching a vacuum in the device chamber of ~ 10^{-9} Torr, the ion-doped crystal was placed against the ion-argon cannon and using the SNA-1 puffing system argon was puffed from a special canister to the required pressure. Fig. 1 shows the mass spectrum of the residual gas in the device chamber after argon puffing at a pressure of ~$2 \cdot 10^{-9}$ Torr.

It can be seen from Fig. 1 that argon is the major fraction in the mass spectrum. The spraying rate of the material was calculated by the formula [1]:

$$\overset{0}{S} = 0.006 \cdot Y \cdot J + \frac{A}{\delta} \tag{1}$$

Under the conditions of our experiment for niobium: atomic mass $A = 41$ g/mol, substance density $\delta = 8.4$ g/cm^3, argon ion energy $E = 1$ keV, and irradiation of the target surface was produced close to normal. Under these conditions, the sputtering coefficient of the substance $Y = 1$ [4]. By increasing the argon

pressure in the chamber, one can significantly increase the current density of Ar⁺ ions and reduce the etching time of each monolayer. At current densities of Ar⁺ ions of $\sim 10^{-6}$-10^{-7} A/cm², the removal time of one atomic layer varied from 1 to 7 min. We produced spraying of the surface layer of the target in the sparing mode. After removing each layer, the Auger spectra were taken and processed quantitatively, the curve of dependence of the concentration of ion-doped Mo atoms on the depth of niobium (100) was constructed by the points.
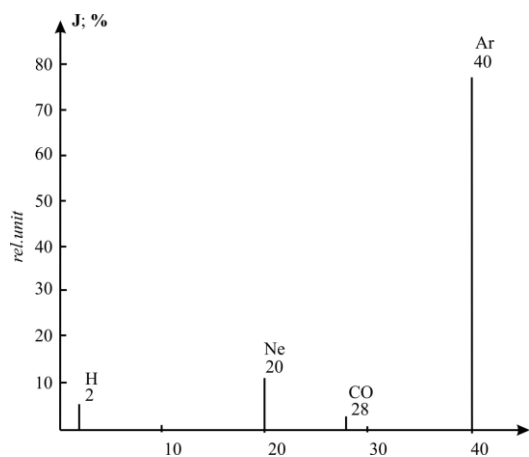

Fig. 1 – Mass spectra of residual gas after argon ($P_{Ar} \sim 2 \cdot 10^{-7}$ Torr)

3. RESULTS AND DISCUSSION

The experiments were carried out at a dose of molybdenum ions $D = 3 \cdot 10^{14}$ ion·cm⁻². The energy of molybdenum ions was equal to $E_p = 3$ keV. In this case, a low molybdenum Auger peak at 190 eV (transition $M_5N_4N_3$) appeared in the Auger-spectrum simultaneously with niobium Auger-peaks (Fig. 2). Quantitative analysis by elemental sensitivity factor method of the Auger spectra taken from the surface of niobium (100) doped with molybdenum ions at a dose of $3 \cdot 10^{14}$ ion·cm⁻² showed that the concentration of molybdenum atoms in the surface layer of ion-implanted niobium was ~ 1 at. %.
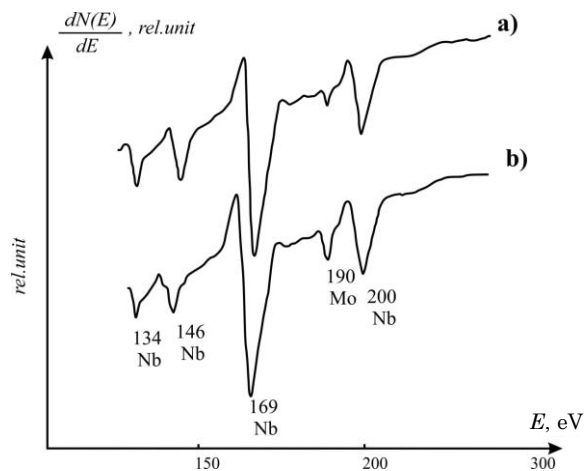

Fig. 2 – a) Auger spectra of niobium surface (100) implanted with molybdenum ions at $D = 3 \cdot 10^{14}$ ion·cm⁻²; b) after removal of four atomic layers

The distribution of Mo atoms over the depth of niobium (100) was studied. An ion-doped Nb (100) sample was installed against the flange with an argon ion cannon, and layer-by-layer etching of the crystal surface with argon ions was carried out. In this case, the current density of argon ions was $2 \cdot 10^{-7}$ A·cm⁻². At such a current density and ion energy $E_r = 1$ keV, one layer of Nb and Mo atoms was removed in ~ 7 min. Then every 7 min, i.e., after removing one layer from the surface of the ion-implanted niobium by turning the manipulator, the crystal was installed in the optical center of the Auger analyzer and the Auger spectra were taken. Fig. 2b shows the Auger spectrum taken during etching of the surface layer of niobium when the maximum concentration of molybdenum atoms was reached, i.e., after removing four atomic layers from the surface of niobium doped with molybdenum ions. After removing ~ 10 atomic layers from the crystal surface, the concentration of Mo atoms decreased to ~ 1 at. %. Auger spectra were processed quantitatively and the concentration profile of the Mo distribution in Nb was constructed. Fig. 3 shows the distribution of Mo atoms over the depth of the near-surface region of the niobium single crystal facet (100). It can be seen from Fig. 3 that in the surface layer of ion-doped niobium, the concentration of Mo atoms at a depth of 4 atomic layers was ~ 3 at. %.

Discussing the results of implantation of molybdenum ions in a single crystal of niobium facet (100) and layer-by-layer analysis, we note the following. When the surface of the substrate metal is bombarded with alloying metal ions, not only the atoms of the surface layer of the matrix metal are sprayed, but also the atoms of the implanted impurity are eliminated. These processes impose a limit on the number of embedded atoms in the surface layer of the target. This phenomenon is due to the low concentration of molybdenum atoms (~ 1 at. %) during implantation with a dose of $\sim (3\text{-}5) \cdot 10^{14}$ ion·cm⁻². When implanting small doses ($D < 10^{16}$ ion·cm⁻²) of the alloying element, dilute ion-implanted alloys with an impurity element concentration of ~ 1 at. % are formed [10-15]. The depth of the maximum content of the alloying element in the surface layer of the crystal is mainly determined by the ion energy of the impurity element [17, 18].
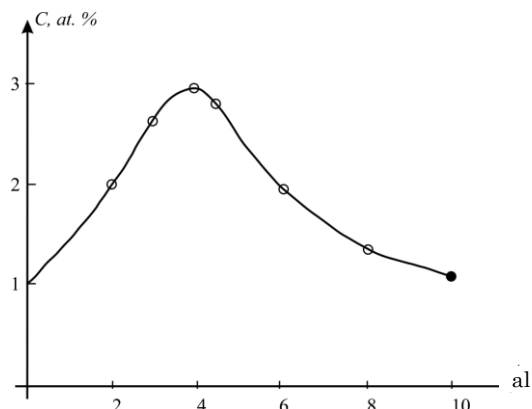

Fig. 3 – Distribution of molybdenum atoms over the depth of the near-surface region of the ion-doped niobium single crystal facet (100)

4. CONCLUSIONS

Thus, it can be noted that a surface alloy of niobium with molybdenum with a thickness of ~ 3-5 atomic layers with a concentration of molybdenum atoms of ~ (2-5) at. % was obtained by ion implantation.

Based on the results obtained, the following conclusions can be made.

1. The implantation of Mo ions into the surface layers of Nb(100) was studied in the dose range $(3\text{-}4) \cdot 10^{14}$ ion·cm^{-2} and with energy $E_p = 3$ keV.

2. The distribution of Mo atoms over the depth of the near-surface region of Nb was determined. It was shown that the concentration of Mo in the surface layer is ~ (1-2) at. %, and at a depth of ~ 4 atomic layers, the concentration of Mo reaches ~ 3 at. %.

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Розподіл атомів молібдену в глибині поверхневого шару монокристалу ніобію, отриманого іонним бомбардуванням

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Одним із фундаментальних напрямів використання іонної імплантації металів є формування поверхневих сплавів будь-якого складу, а їх кількісна характеристика – розподіл атомів домішкового елемента в глибині поверхневого шару металевої підкладки [1-5]. При вивченні внеску кожного компонента в зміну електронної структури, емісійно-адсорбційних і каталітичних властивостей багатокомпонентних металевих сплавів, створення особливо чистих металевих твердих розчинів, а також визначення фактичної концентрації легуючих елементів і їх розподілу в поверхневому шарі кристалів, мають велике значення. Для дослідження профілів розподілу концентрації іонно-імплантованих атомів тугоплавких металів у сплавах на основі тугоплавких металів використано метод пошарового аналізу за допомогою оже-електронної спектроскопії в поєднанні з аргоновою іонною гарматою. У роботі на прикладі імплантації низькоенергетичних іонів молібдену в монокристал ніобію огранювання (100) розглянуто особливості створення поверхневих сплавів на основі тугоплавких металів з низькою концентрацією легуючого елемента та розподілом іонно-імплантованого металу в приповерхневій області кристала [5-9].

Ключові слова: Поверхня, Електронна спектроскопія, Надвисокий вакуум, Монокристал, Іонна імплантація.