

A..D. POGREBNJAK* and M.V. ILYASHENKO**

*W-ions implantation and deposition of carbon
film on the Fe₅₄-Cr₂₀-Ni₁₆-Mn₁₀ (100) single
crystals surfaces*

ABSTRACT

After W ion implantation with $5 \cdot 10^{16} \text{ cm}^{-2}$ dose the surface alloy of the Fe₅₄-Cr₂₀-Ni₁₆-Mn₁₀ single crystal was studied using Rutherford Backscattering Spectrometry (RBS), (elastic resonance reactions ⁴He(O¹⁶, O16), Auger electron spectroscopy (AES), Atomic Force Microscope (AFM), Raman spectroscopy and Transmission Electron Microscopy (TEM). The surface alloy layer is composed of a number of structures: amorphous carbon film with diamond-like regions, layer with a small number of defects, FCC structure with large angle disorientation of subgrains and dislocation density up to $\sim 7\text{--}8 \cdot 10^9 \text{ cm}^{-2}$, layer with net subgrains structure and dislocation density up to $5 \cdot 10^{10} \text{ cm}^{-2}$. The maximum of W concentration is observed at the depth of 32 nm, and penetration depth reaches 130 nm. Carbon film on the surface protects against ion sputtering.

* Sumy Institute for Surface Modification, Shevchenko Wul. 17, 40011 Sumy, Ukraine, apogrebnjak@SIMP.sumy.ua

** Sumy State University, Rymskii-Korsakov Str. 2, 40007 Sumy, Ukraine, kpe@ssu.sumy.ua

1. INTRODUCTION

High dose ion implantation is an effective method of surface modification and improving technical characteristics of metals and alloys. The surface properties such as wear resistance, microhardness, friction coefficient, fatigue and corrosion resistance can be significantly enhanced as a result of high dose ion implantation.

In this paper high dose W^+ implantation has been used for modification of $Fe_{54}-Cr_{20}-Ni_{16}-Mn_{10}$ (100) single crystal. Single crystal material was chosen in order to minimize the influence of different crystal orientations and of the grain borders. Tungsten was chosen as a doping element because of its high atomic mass. Therefore it can be easily detected with the help of RBS. Heavy tungsten ions create also a large number of defects when implanted up to relatively low concentration (~ 1 at%)[1-3].

Carbon, which is usually present in residual atmosphere, deposits on the target surface. The changes in carbon layer caused by ion beam lead to its amorphisation (the formation of carbon layer occurs due to re-composition of hydrocarbons caused by ion beam). Sometimes formation of a diamond-like film on the surface is observed. Due to their interesting properties as high hardness, wear resistance and good tribological characteristic such films even increase the results of ion implantation.

In case of heavy-ions implantation radiation defects are formed in the near surface layers of metals. Such changes lead to the improving of hardness and wear resistance of surface [4].

2. EXPERIMENTAL CONDITIONS

The sample size was 10·10·2 mm. The surface of the samples was mechanically and electrochemically polished before annealing. The typical sample composition was: Fe-54 at %, Cr-20 at %, Ni-16 at %, Mn-10 at %.

W ion implantation was performed with the help of vacuum-arc ion source with the following parameters: voltage – 40 kV, pulse duration – 200 μ s, pulse frequency – 50 Hz, vacuum – 10^{-3} Pa [5].

For TEM analysis we prepared the foils with thickness between 100 to 200 nm. After irradiation the samples were jet-polished to the necessary thickness.

After W ion implantation up to $5 \cdot 10^{16}$ cm⁻² dose the surface alloy of the Fe₅₄-Cr₂₀-Ni₁₆-Mn₁₀ samples were studied by using RBS, elastic resonance reactions ⁴He(O¹⁶, O16) with He⁺ energy 1754 keV, AES, AFM, Raman spectroscopy and TEM. AES also gave us additional information about element distribution (especially for carbon because RBS is less sensitive for carbon due to small atomic mass) [6-8].

3. RESULTS AND DISCUSSION

Ion implantation leads to the formation of multilayer structure within the samples. It is shown that discontinuous amorphous carbon film of complicated relief with 20–40 nm thick diamond-like carbon areas is formed on the sample surface. The formation of carbon layer is due to the deposition of carbon from residual vacuum contamination.

RBS energy spectrum obtained using ⁴He⁺ of 3052 keV energy is presented in Figure 1. Analysis of the spectra shows that there is W-profile displacement in the depth of the specimen as result of diminishing of ions scattering because of this layer. W-ions concentration on the 30 nm depth is 0,7 at % with the entire profile length nearly 100 nm.

There is a high oxygen concentration on the film/single crystal boundary. There is a small re-distribution of metal alloy elements on the 100–300 nm depth (out of the zone of implanted ions direct influence).

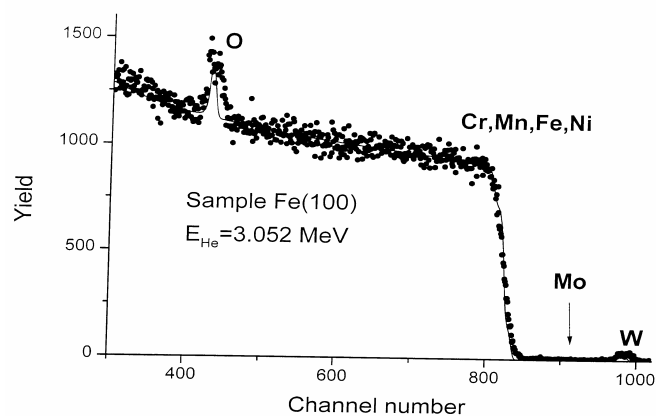


Fig. 1. RBS energy spectrum obtained using He^{+4} of 3052 keV energy for $\text{Fe}_{54}\text{-Cr}_{20}\text{-Ni}_{16}\text{-Mn}_{10}$ after implantation of W ions (40 keV) ions with a dose $5 \cdot 10^{16} \text{ cm}^{-2}$

The Figure 2 demonstrates picture of untreated sample surface obtained with the help of Atomic force microscope (AFM). Picture of sample surface implanted with W ions is present in Figure 3. There is a wave-like relief of the carbon film on the surface. The average height of the “hills” is between 20 to 40 nm.

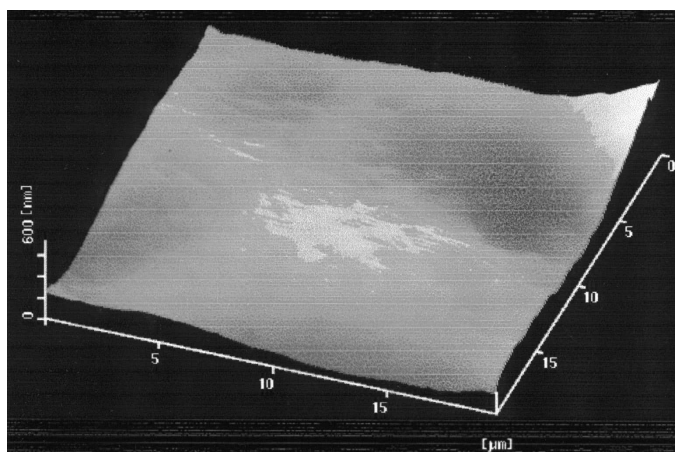


Fig. 2 AFM patterns for $\text{Fe}_{54}\text{-Cr}_{20}\text{-Ni}_{16}\text{-Mn}_{10}$ untreated samples

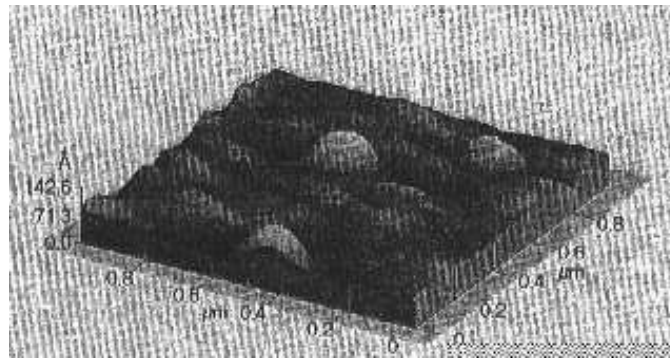


Fig. 3. AFM patterns for $Fe_{54}-Cr_{20}-Ni_{16}-Mn_{10}$ samples implanted with W ions (the dose was $5 \cdot 10^{16} \text{ cm}^{-2}$)

Results of the analysis a film by means of Raman spectroscopy on the reflection are shown in Figure 4. Peaks in the region of 1530 cm^{-1} demonstrate the formation of amorphous phase and in the region of 1313 cm^{-1} – diamond-like carbon [9].

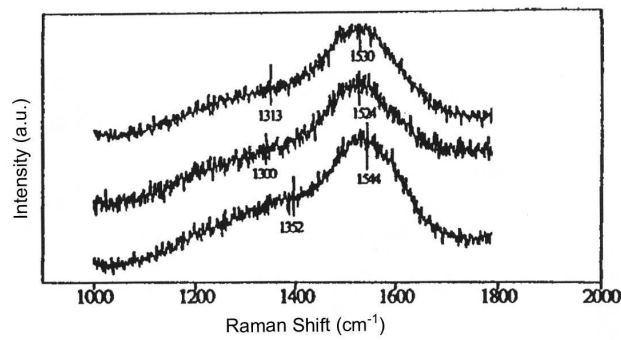


Fig. 4. Results of the analysis the film by means of Raman spectroscopy on the reflection for three different regions.

Surface structure of single crystal alloy $Fe_{54}-Cr_{20}-Ni_{16}-Mn_{10}$ after implantations with ions W is presented in Figure 5.

The layer under amorphous carbon film has almost no defects.

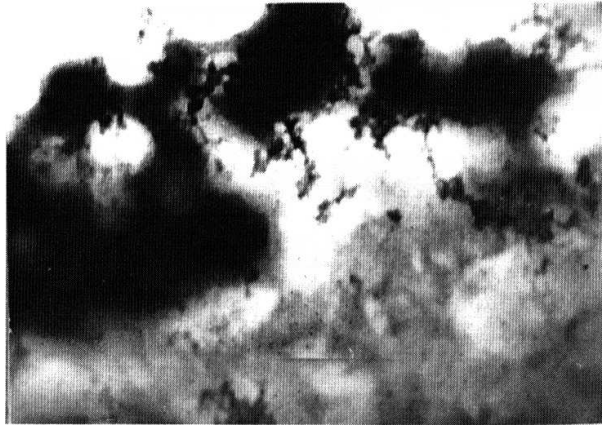


Fig. 5. Analysis of structural and phase changes in the $\text{Fe}_{54}\text{-Cr}_{20}\text{-Ni}_{16}\text{-Mn}_{10}$ subsurface layer implanted with W ions (the dose was $5 \cdot 10^{16} \text{ cm}^{-2}$)

Figure 6 demonstrates sublayer formation in single crystal surface. This layer is of FCC structure. There are subgrains in this layer with large angle disorientation. The density of dislocations is up to $\sim 7\text{-}8 \cdot 10^9 \text{ cm}^{-2}$.

Dislocations scalar density sharply increases up to $2.1\text{-}5 \cdot 10^{10} \text{ cm}^{-2}$ on the $0.3\text{-}0.5 \text{ }\mu\text{m}$ depth. Dislocations are distributed chaotically or they may form net substructure. In the modified layer the isolation of subgrains and secondary phase particles are noticed.

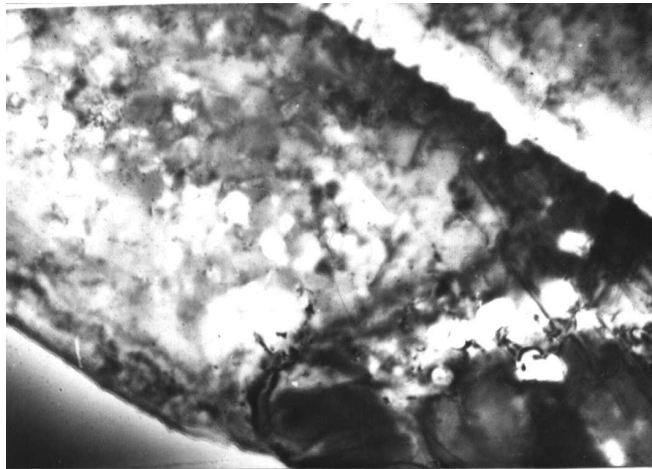


Fig. 6. Sublayer formation in $\text{Fe}_{54}\text{-Cr}_{20}\text{-Ni}_{16}\text{-Mn}_{10}$ single crystal as a result of W ions implantation at the $5 \cdot 10^{16} \text{ cm}^{-2}$ dose

The AES studies, which are shown in Figure 7, help us to precise the results of previous investigations.

The thickness of carbon film measured by AES is close to one obtained with the help of AFM. Surface carbon concentration is not 100%. It says about non-uniformity of surface relief.

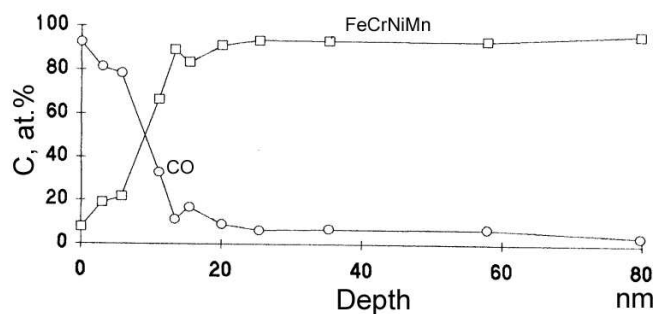


Fig. 7. AES results for $Fe_{54}Cr_{20}Ni_{16}Mn_{10}$ single crystal implanted with W ions at the $5 \cdot 10^{16} \text{ cm}^{-2}$ dose

4. CONCLUSIONS

During ion implantation of W-ions the amorphous film with diamond-like regions was formed. It has complicate island-like relief. The conditions of its formation are thermodynamically non-equilibrium. The ion beam transmits the supplementary energy to the carbon atoms. This energy is significantly higher then their thermal energy (the sample temperature was about 250°C). Also re-composition of hydrocarbons takes place and also contributes to amorphous layer formation.

It has sense to assume that this film significantly affects to the surface properties of the sample. Besides, carbon film protects surface from ion sputtering and therefore increase the concentration of implanted impurity.

W⁺ implantation leads to the penetration of W atom to depth up to 130 nm. Maximum concentration of W is observed at the depth of ~30 nm. Due to intensive sputtering processes the W-concentration is insignificant (~0.7 at.%).

Also during ion implantation intensive processes of re-crystallization and defect structure formation take place. Insignificant redistribution of elements is observed as a result of radiation enhanced diffusion. The maximum of defect concentration is at the depth of 0.3-0.5 μm. This depth also corresponds to the formation of multi-layer structure and subgrains structure with large angle disorientation. The layer, which lies under carbon amorphous film, has even less defects than non-implanted sample.

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