ACTION OF AGE-HARDENING ON THE COPPER SINGLE CRYSTALS AFTER ION IMPLANTATION

O.P. Kul'ment'eva[1], A.I. Kul'ment'ev[2]
[1] *Sumy State University, Sumy 2 Rymskij-Korsakov St , PO Box 277, 40030 Sumy, Ukraine, тел.+3(054-2) 63-90-18, E-mail: opkulm@mail.ru*
[2] *Institute of Applied Physics, NAS of the Ukraine, Petropavlovskaya St. 58, 40030 Sumy, Ukraine*

High-dose implantation (up to (1 - 5)·10^{17} cm^{-2}) of tantalum ions into a copper single crystal of (100), (110) and (111) orientation has been investigated. Modified properties just after ion implantation and subsequent age-hardening during ten years were studied. It was shown that ion implantation and subsequent masstransfer process results in sufficient long-term stable changes of the microhardness.

Introduction

High dose ion implantation (requiring doses of the order of 10^{17} cm^{-2}) is an effective technique of surface modification and improving of its properties (wear resistance, corrosion, hardness and so on) for metal materials [1]. This method is now intensively developing due to its advantages compared to the traditional methods of surface treatment [2]. The processes, which take place In near surface region of metals during conventional ion implantation [2] are well understood. Situation with high-dose implantation of a single crystal by multiple-charged heavy ions is much more complicated. Partly this is due to experimental difficulties of preparing high quality surfaces of metal single crystals, and partly due to relative scarcity of high intensity heavy ion sources.

As a result there is an urgent problem in investigation of main process leading to formation of ion distribution in such experiments. In most cases it is taken for granted that specimen with modified surface would be used for a long time in some device. But up to now investigation of age-hardening of modified properties is practically absent.

Therefore in this paper we tried to analyses the changes in the surface properties of Cu (100), (110) and (111) single crystal after high dose (10^{17} cm^{-2}) Ta^+ ion implantation. Special attention was paid to the study of age-hardening of these properties after approximately ten years period in natural condition.

For these purposes we used three different types of experiments, in which the influence of ion implantation has been investigated: 1) computer simulation of ion and point defect distributions after Ta^+ implantation; 2) microhardness measurements of samples surface and its dependence on single crystal orientation and under subsequent age-hardening.

Irradiation conditions and experimental methods

In experiment, ion implantation is produced using implanter "Diane-2". Beam of tantalum have been used to produce modification in the surface properties of material. Next operation parameters were used: ion type Ta^+, ions energy 40 keV, frequency of pulses 50 Hz, pulse duration 200 μs, ion current 10 mA, ion beam diameter 20 mm, implantation dose 10^{17}... 5·10^{17} cm^{-2}, residual pressure 10^{-4} Torr. Prepared targets had the form of rectangular samples with 10×10×3 mm dimensions. We investigated Cu single crystals, which was cut parallel to the crystallographic planes (100), (110) and (111). During implantation the samples were cooled by water and their temperature didn't exceed 473 K.

TRIM [3] Monte Carlo simulations were performed In order to calculate the defect densities produced In the copper after tantalum ion implantation.

The surface morphologies of the samples were studied by scanning electron microscope SEM-103 with accelerating voltage 25 keV. Microhardness of the samples was measured with a PMT-3 facility, and the four-faced diamond pyramid was used. The applied load on the pyramid was 0.5, 0.6, 0.7 and 1.0 N. Microhardness was measured for implanted and non-implanted surfaces of single crystal that allow to determine the relative changes of surface hardness as a result of ion implantation. The second measurements of microhardness were performed on the same samples by the same facility after approximately ten years period in natural condition.

Results and discussion

With help of TRIM program computer simulation of tantalum ions implantation were carried out. It should be stressed, that parameters of simulation: initial energy, angle of incidence and charge distribution in the ion beam were the same as in above described experiments. The parameters of resulting ion ranges are shown in Fig.1a. For example the mean range of Ta^+ is relatively small and equals to 89 Å (Fig. 1).

In the same simulations we also studied distribution of primarily displaced matrix atom (Fig.1b). It should be noted that this distribution is nonzero in the nearsurface region. This feature can lead to sufficient target sputtering. To check this proposition we measured sputtering coefficient in computer experiment witch was equal to 8.95 atom per ion. Therefore matrix sputtering can alter large changes in final distribution of implanted atoms. Furthermore computer simulations showed that there is great radiation damage of the target lattice because for normal incidence each Ta ion produces on average 468 vacancies in Cu lattice.

SEM investigation shows that heavy ion implantation into single crystals leads to the various changes in their surface morphology. On the initially plane surface of Cu (111) there can be seen numerous metal droplets. They are typical for high-dose ion implantation. On the Cu (100) surface besides these droplets radiation damage in the form of rectangular holes is clearly visible.

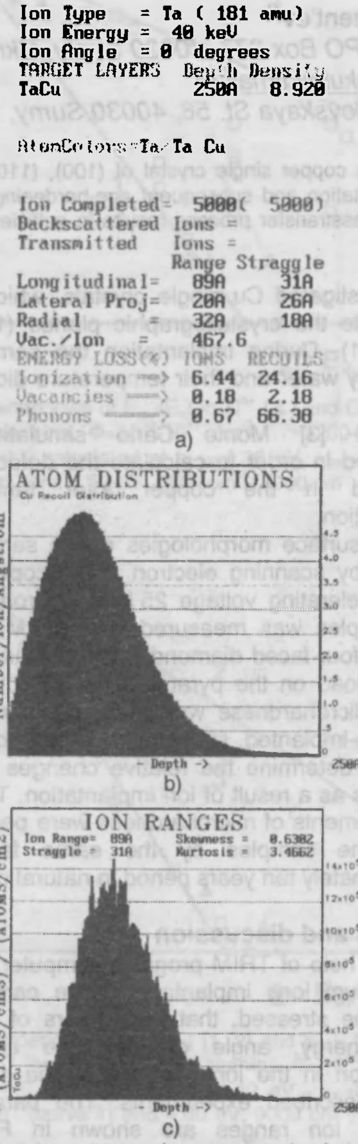

Fig. 1. Input parameters in computer simulation (a), distribution of target recoil (b) and implanted ions (c). The conditions in computer experiment was similar to the conditions in actual one.

We have measured changes of microhardness of single crystals with orientation (100), (110) and (111) caused heavy ion implantation. The results are shown in Table 1.

For samples with notable inhomogeneous experimentally determined microhardness may depend on the applied load. This well known fact results from the different depth of indenter embedding and from the sharp variation of hardening with depth in modified near surface region. Thus there exist a problem of proper choice of applied load.

In the work dependence of experimentally determined microhardness on the applied load was studied for samples with different orientation of modified copper single crystal. It was observed that decreasing of applied load leads to expected decreasing of diamond indenter embedding depth but at the same time to increasing of microhardness. Thus during implantation of Ta ions into Cu the processes of surface hardening take place. Among them distortion of crystal lattice and defects accumulation are most important ones. As a result after load removal the elastic recovery of indenter imprint takes place. In the range of applied load from 0.7 to 1.0 N experimentally determined values of microhardness tends to saturate, so henceforth in subsequent experiments the value of 0.7 N was used. Similar results were observed for other orientation of Cu single crystal.

Table 1. Microhardness Hμ and dept of indentor penetration h into copper single crystals before and after Ta+ implantation with different doses

Sample/dose	H_μ, MPa	*h*, μm
Cu (100), non-implanted	346* \ 332 ± 58	2,861 ± 0,272
Cu (100) D = 10^{17} cm^{-2}	458* \ 462 ± 48	2,402 ± 0,119
Cu (110) D = 10^{17} cm^{-2}	578 ± 185	2,222 ± 0,341
Cu (110) D = $5\cdot10^{17}$ cm^{2}	430 ± 40	2,489 ± 011
Cu (111) D = 10^{17} cm^{-2}	446*\ 418 ± 54	2,533 ± 0,164

*10 years early

From table 1 it is seen that after ion implantation with dose D = 10^{17} cm^{-2} the microhardness of all samples increases. For the same experimental conditions irradiation with the same total ion dose may produces different changes in the microhardness of Cu single crystals with different orientation. For as-implanted samples relative enhancement amounts to 32,4% and 28,9% for Cu (100) and Cu (111) single crystal orientation respectively.

From the other hand microhardness of Cu (110) non irradiated single crystal in the process of ten years natural ageing decreased by 4%. If we take this sample as reference point than after this process of ageing largest enhancement of Hμ was observed for (110) orientation and amounts to 74,1 % and the smallest (25,9 %) for (111) orientation.

Another operational parameter which can be easily varied and has important influence on the final results is ion dose. Increasing of ion dose may leads to decreasing of microhardness enhancement. For example in the case of (110) orientation the microhardness was equal to 578 MPa after $10^{17}cm^{-2}$ ion dose irradiation and to 430 MPa after $5\cdot10^{17}cm^{-2}$ dose but still remains sufficiently higher than in initial untreated samples. It should be stressed that despite the small decreasing of mechanical properties of modified Cu surface they maintain superior characteristics compared to untreated samples after ten years natural ageing.

Early [4] it was shown that tantalum implantation was accompanied by surface carbonization and oxidation. Carbon and oxygen atoms were presented in the residual atmosphere of vacuum chamber. The concentration of carbon and oxygen atoms on the surface reached 50 at. %. Presumably diffusion of these light gas atoms to the crystal surface and intensive sputtering of the latter is the driving forces for appearance of rectangular holes, which were observed by SEM techniques.

Conclusions

Tantalum ion implantation in a copper single crystal with (100), (110) and (111) orientation has been studied. Mechanical testing showed that ion implantation induced microhardness enhancement of the copper surface for all samples. It was shown that these enhancement of mechanical properties did not change sufficiently after ten years natural ageing. This is important evidence of reliability of ion implantation technique and their ability to produce stable long-term enhanced modification of various metal properties.

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