

Mass Transfer of an Implanted Dopant in Materials under High-Power Pulsed Ion Beam Impact¹

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Abstract – The results of experimental studies of the mass transfer features in Ti-Si and Ti-Fe systems during repeated alternate processes of Ti ion implantation and irradiation with a high-power ion beam are presented for ion beam energy densities of $\leq 2 \text{ J/cm}^2$. The analysis of the final state of samples surface after the combined treatment is carried out.

1. Introduction

One of prospective directions of materials modification is connected with a formation of deeply doped layers of given physical and mechanical properties. For these purposes, different kinds of combined treatment are investigated. The method in which a surface of material is treated consecutively by means of ion implantation and pulsed irradiation with high-power ion beam (HPIB) in various combinations has a big potential. In this case, the HPIB intensifies the mass transfer processes leading to increasing penetration depth of a previously implanted dopant and creation of new structure-phase states inaccessible with conventional methods of ion-beam and ion-plasma modification of materials. The physical basis for the said processes is ultrafast heating of sub-surface layers under HPIB impact up to the melt phase followed by cooling due to heat dissipation deep into material with estimated rates of $10^8\text{--}10^{11} \text{ K/s}$ [1, 2] and arisen high temperature gradients ($10^6\text{--}10^7 \text{ K/cm}$) and pressure gradients.

Advantages of ion implantation, such as dose controllability, embedding of a wide range of chemical elements, and absence of thermodynamical limitations in doping, are well known. An important progress related to metallic materials is in new possibilities for high-speed and high-dose doping, which appear with the development of high-productivity vacuum arc-based implanters [3–5] and short-pulse HPIB-based implanters [6, 7] along with methods for reduction of surface sputtering effects [8, 9] limiting the implanted dose. Using the HPIB treatment in addition to ion implantation keeps the advantages mentioned and, at the same time, allows for extended control of elemental and phase composition of surface layers and for

significant changes of many operational properties of materials and units.

Mass transfer in film-substrate binary structures and elemental re-distribution in surface layers of metals with complicated composition (accounting also for saturation with residual gases) under HPIB treatment were considered in many earlier works (see, for instance, the review [10]). The case of HPIB-induced mass transfer of the implanted dopant attracted less attention. The possibility of increasing the depth of doping for the implanted impurity at a few (1 to 3) HPIB pulses was demonstrated in [11–13]. In this work, the mass transfer features are studied at multiple cycles comprising ion implantation and HPIB irradiation. The HPIB energy density ($\leq 2.0 \text{ J/cm}^2$) was restricted to a value corresponding to evaporation of a surface layer that is considerable by comparison with the implantation depth, which is $\leq 0.1 \mu\text{m}$. Also, the analysis of the final state of the surface of samples after the combined treatment is performed.

2. Experimental Conditions and Research Methods

The samples irradiation was carried out on the facility for combined materials treatment [9] consisting of the “Raduga” implanter and HPIB source (75% H^+ , C^+ , 500 keV, 100 ns). Investigations of the mass transfer of a previously implanted dopant under HPIB irradiation were fulfilled for the case of Ti implantation into Si and Fe samples. The experimental protocol was as follows. After the repetitively-pulsed implantation of Ti ions (charge state of 1–3, 50 keV, 400 μs , 50 pps rep-rate) with the dose of $\sim 1 \cdot 10^{17} \text{ ions/cm}^2$, the samples were irradiated with two HPIB pulses of the given energy density. Then, this cycle was repeated. The number of these cycles being done in one technological operation was up to eight.

For studying the surface morphology of the samples, the optical microscopes FOM-2-16 and MIM-11 were employed. 3-D images and profilograms of surfaces were obtained using the device Micro Measure 3-D Non Contact Profilometry by STIL Company with sensitivity of 1 nm. The surface state was analyzed over the area of $0.3 \times 0.3 \text{ mm}$.

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For determination of the elemental composition of surface layers and element concentration profiles, the method of Rutherford backscattering of helium ions (1.5 MeV energy) was used.

In the course of experiments, the operating pressure provided by the cryogenic pumping system was of $\approx 5 \cdot 10^{-6}$ torr.

3. Results and Discussion

In Fig. 1, the concentration profiles of Ti in the Ti-Si system obtained by the method of Rutherford backscattering are presented for different irradiation conditions. It is seen that after the 4 “implantation-irradiation” cycles, storage of the implanted dopant is accompanied by the increase of penetration depth up to ~ 270 nm (compared to the initial depth of ~ 90 nm) and formation of the flat profile of Ti concentration with the maximum near the surface. For the Ti-Fe system (Fig. 2), the depth of Ti diffusion after the 4 performed cycles is ~ 75 nm, and after the 8 such cycles, it achieves ~ 155 nm that exceeds the initial implantation depth by more than 5 times.

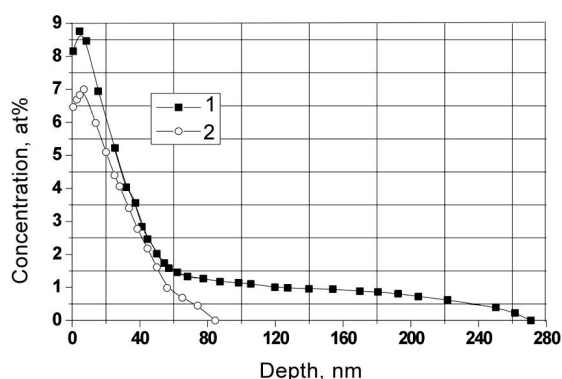


Fig. 1. Ti concentration profiles over the depth in Si sample: 1 – after the 4 cycles of Ti implantation (10^{17} ions/cm 2) and HPIB irradiation (~ 1.6 J/cm 2 , 2 shots); 2 – after Ti implantation, the dose is $\sim 10^{17}$ ions/cm 2

Typical feature of the mass transfer is almost linear growth of the depth of Ti embedding with increasing number of HPIB irradiation pulses for both Ti-Fe and Ti-Si systems. At the same time, the depth of dopant penetration remains less than the calculated thickness of melted layer obtained in [2] at the HPIB parameters and composition close to what we use. The same conclusion can be made from the comparison of temperature field calculations [12] with the experiments using a few HPIB shots for irradiation [11, 12]. Such character of dopant atoms migration is conditioned by decreasing liquid-phase lifetime and reducing dopant concentration gradient with depth below the surface that influence on the diffusion process. The resulting concentration profile with a sharp peak near the surface is formed probably due to dopant segregation

in the solidification front at high re-crystallization rate [2]. The presence of spikes in diffusant concentration profiles near the surface of various thin-film structures was noted in a number of experiments on their irradiation with the HPIB [1] as well as pulsed laser treatment [14]. The growth of implanted dopant concentration at the surface should be noted that is explained by increasing erosion resistance of the surface, i.e., reduction of its sputtering coefficient after HPIB treatment [9] for further implantation. Thus, varying ion beam fluence, number of shots, and combinations of implantation and irradiation processes, one can control the implanted dopant diffusion rate, depth, and storage.

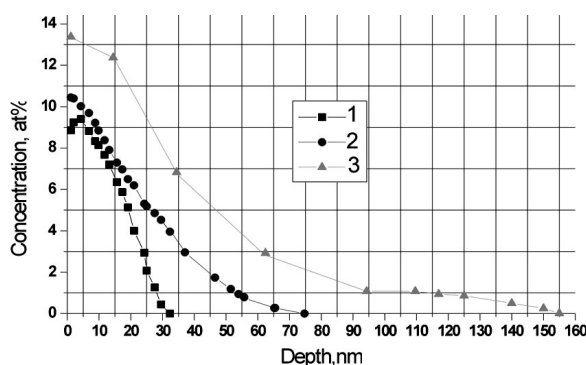


Fig. 2. Ti concentration profiles over the depth in Fe sample: 1 – after Ti implantation, the dose is $\sim 10^{17}$ ions/cm 2 ; 2 – after the 4 cycles of Ti implantation (10^{17} ions/cm 2) and HPIB irradiation (~ 1.8 J/cm 2 , 2 shots); 3 – after the 8 cycles

The character of changes in the morphology of Fe surface before and after the eight “implantation-irradiation” cycles is shown in Fig. 3. The roughness of the sample surface is $R_z \approx 0.4$ μ m for the former case (before treatment); then, after the first implantation it becomes a bit larger, $R_z \approx 0.48$ μ m, and after completing the course of the combined treatment it reaches the value $R_z \approx 1$ μ m. As is seen from Fig. 3, b, the topography of the final surface state is featured by appearance of the craters with the diameter and depth of up to 30 μ m and 1.2 μ m, respectively, that determine the roughness change. Their formation is connected, to a great extent, with the presence of impurities in the sample surface layer including those embedded at the initial treatment. We observed the reduction of the amount of the formed craters with increasing number of HPIB irradiation shots that was probably connected with the surface cleaning from impurities. The rate of this process, in its turn, depends on the initial amount of volatile elements in the surface layer.

For the case of implantation into Si sample, the surface distortion after HPIB irradiation are expressed weakly, and the surface roughness in all cases does not exceed values $R_z \approx 0.1-0.2$ μ m.

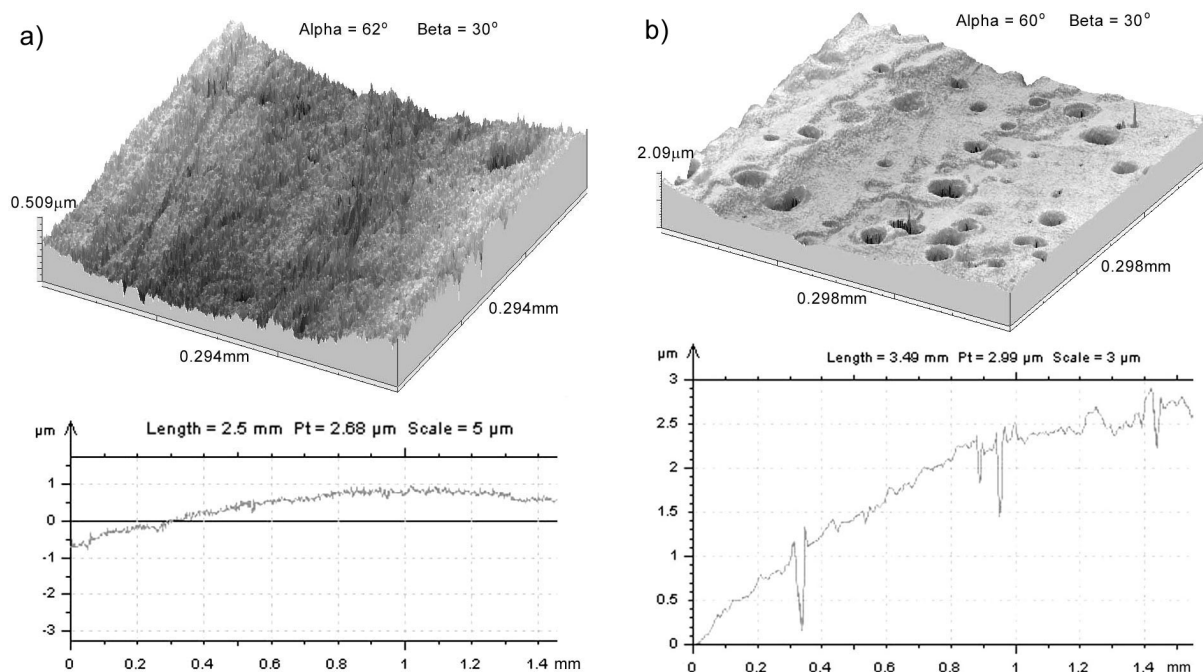


Fig. 3. Surface images and profilograms for Fe sample: *a* – before treatment; *b* – after the 8 cycles of Ti implantation (10^{17} ions/cm²) and HPIB irradiation (~ 1.8 J/cm², 2 shots).

4. Conclusions

Investigations of the method of deep doping based on multiple alternate treatment of material surface with ion implantation and pulsed HPIB of small energy density (≤ 2 J/cm²) have been performed. For Ti-Si and Ti-Fe systems, the possibility to control the dopant storage and depth of embedding has been shown. Character feature of the mass transfer is the almost linear growth of the depth of diffusant penetration with increasing total number of HPIB irradiation shots (up to 16 in the experiments). It has been demonstrated that the method used allows for increasing depth of Ti diffusion in Ti-Fe system by more than 5 times as compared to the initial depth (after the first implantation). At the same time, the dopant penetration depth remains less than the calculated melted layer thickness, which is ~ 1 μm for the parameters of the HPIB employed. This indicates the possibility of significant increasing the depth of doping at implantation with further increasing total number of HPIB irradiation pulses. In this case, the process of dopant embedding can be well combined with the short-pulse HPIB-based implantation accompanied with thermal impacts.

The feature of the final state of the surface of Fe samples after multiple HPIB impacts (≤ 16) is the presence of craters determining the roughness increase up to $R_z \approx 1$ μm . Whereas, for Si samples, the surface distortions and the roughness are small.

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