

MICROSTRUCTURE OF MOLYBDENUM-SILICON SURFACE LAYERS FORMED BY COMPRESSION PLASMA FLOWS

V.M. Astashynski¹, V.V. Uglov², N.T.Kvasov³, Yu.A. Petukhou³,
A.A. Azhoichyk², R.S. Kudaktsin², A.M. Kuzmitski¹, A.A. Mishchuk¹

¹B.I. Stepanov Institute of Physics, NAS B,

220072 Nezavisimosti ave. 70, Minsk, Belarus, ast@imaph.bas-net.by

²Belarusian State University, 220030 Nezavisimosti ave. 4, uglov@bsu.by

³Belarusian State University of Informatics and Radioelectronics,

220013 Brouki str. 6, spadar_pett@tut.by

Introduction

Interaction of compression plasma flows (CPF) with metals and semiconductors is intensively studied for the last decade as an effective tool for synthesis of novel materials and their modification. Peculiarities of CPF-action are connected with high thermal power density transmitted to the target (up to 4-5 GW/m²) and relatively long duration of stable plasma flow (up to 200 μs). CPF treatment of binary and multicomponent materials results in the formation of supersaturated solid solutions, intermetallides, deep alloying, improvement of hardness and thermal stability of mechanical properties /1-3/. Thereupon one of promising research direction is connected with CPF action on binary “metal-silicon” systems and their use for the formation of metal silicides. The use of concentrated energy flows is effective for synthesis of refractory compounds, for example, molybdenum silicides that have broad range of applications due to their high melting point, low electrical resistance, high oxidation resistance and low density /4,5/.

Experimental

Molybdenum coating was formed on single-crystal silicon plates (100) and (111) by vacuum arc deposition (cathode arc current was 150 A, substrate bias – 120 V, deposition time 1 min). Thickness of molybdenum film was 0,13 μm that provides high adhesion strength.

Compression plasma treatment was carried out in the gas discharge magnetoplasma compressor of compact geometry (MPC) and mini-MPC device. Experiments were performed in a residual gas mode such that the pre-evacuated MPC chamber was filled with orifice gas (nitrogen) up to pressure of 400 Pa. Power density absorbed by the target W was varied in the range from 0,2 to 1,5 GW/m².

Surface morphology and microstructure of cross-section were studied by scanning electron microscopy (SEM) using a LEO1455VP microscope. Electron survey was carried out in phase contrast mode (registration of backscattered electrons only). Microrelief of the surface was studied by atomic force microscopy with a SOLVER P47 PRO scanning microscope.

Results and discussion

The action of CPF with low power density ($0,2-0,5 \text{ GW/m}^2$) mainly affects surface of the molybdenum film. AFM studies showed that initial film is continuous and consists of crystallites with dispersion of sizes from 20 nm to 1,3 μm (Fig. 1a). CPF action results in the formation of separated asperities at the surface (Fig. 1b). Their height is up to 30 nm, average distance between tops is 200 nm, lateral size is $\sim 100 \text{ nm}$.

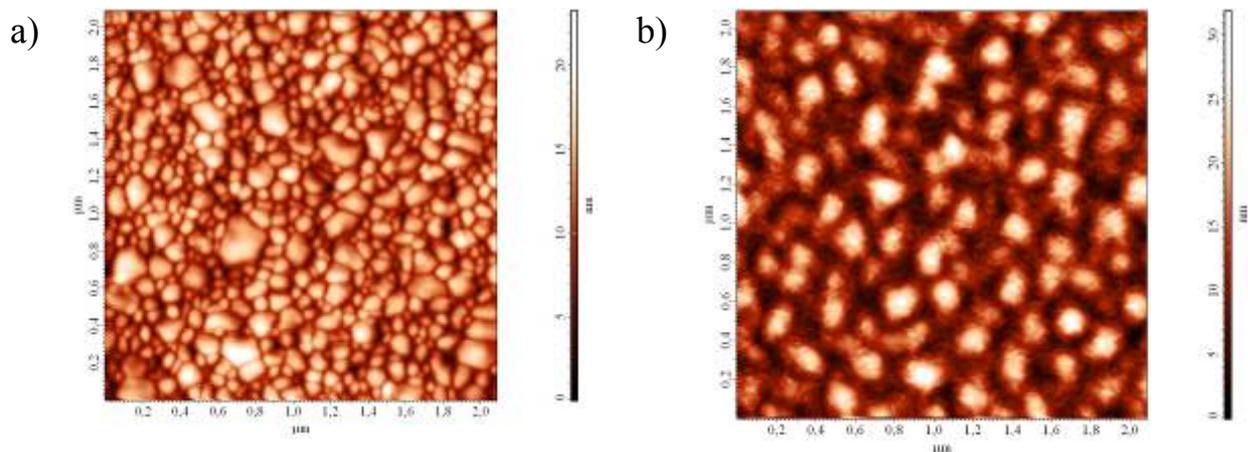


Fig. 1 AFM-images of initial (a) and CPF-treated (b) Mo film ($W=0,4 \text{ GW/m}^2$)

The asperities detected by AFM is most likely correspond to molybdenum clusters formed by partial evaporation of the film, nucleation in the shock-compressed plasma layer followed by re-deposition on the surface and migration due to thermal-activated diffusion [6].

The action of CPF with power density $W>0,5 \text{ GW/m}^2$ results in the formation of uniform molybdenum-alloyed silicon layer 3-5 μm thickness (Fig. 2-3) with average Mo concentration 2-3 at.%. SEM studies showed the formation of silicon cells with lateral size $\sim 1 \mu\text{m}$, while molybdenum is localized mainly at the edges. For silicon substrate Si(100) cells axes are directed normally to the surface, for Si(111) they are directed both normally and obliquely (under $\sim 30^\circ$ angle).

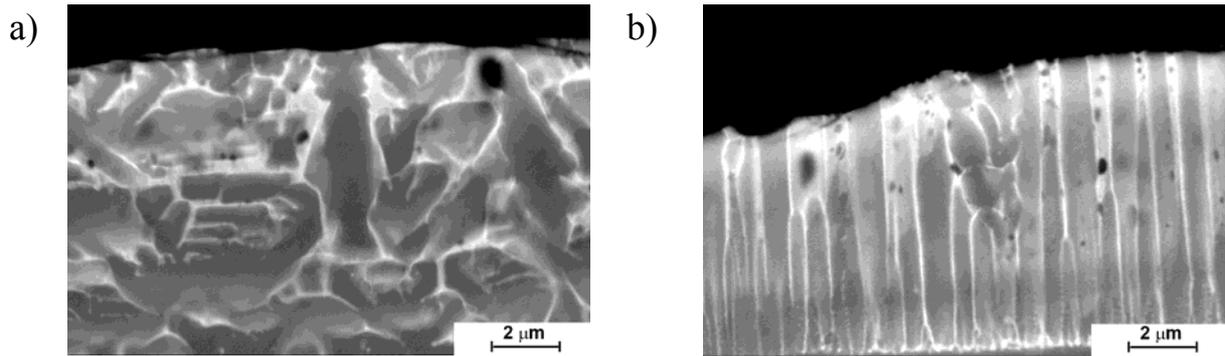


Fig. 2 Cross-section SEM-images of CPF-treated Mo/Si(111) and Mo/Si(100) systems ($W=1,5 \text{ GW/m}^2$)

Cells formation is connected with molybdenum edging out the solidification front due to its low solid solubility in silicon /7/. According to phase diagram /8/ liquid containing up to 15 at.% of molybdenum solidifies at temperatures lower than silicon melting point. This results in liquid overcooling near the phase interface due constitutional overcooling /3/. In these conditions any asperity at the crystallization front gets to the overcooled region of the melt and solidifies providing additional molybdenum edging. Further crystallization of this asperity results in the formation of the cell. For Si(100) substrate the direction of preferred cell growth (along the face of silicon single crystal cell) coincides with the direction of heat transfer that results in the growth of normally-oriented cells; for Si(111) substrate these direction are different that leads to the disarrangement of cells' axes.

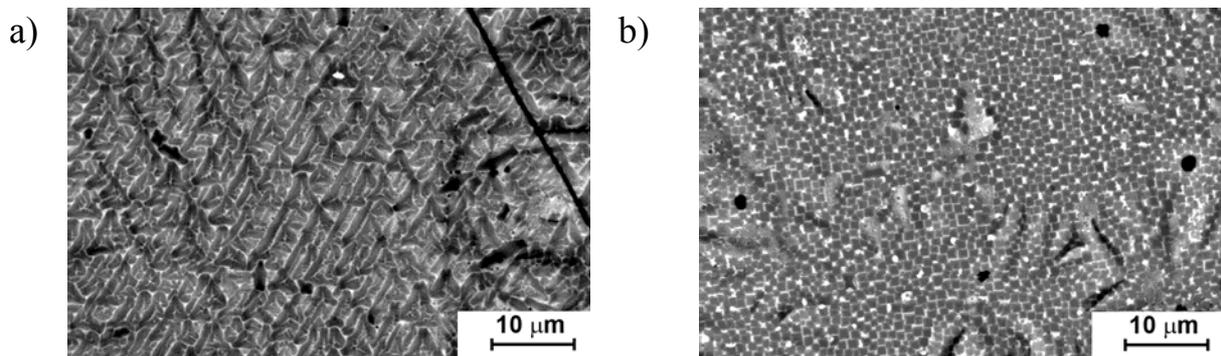


Fig. 3 Surface SEM-images of CPF-treated Mo/Si(111) and Mo/Si(100) systems ($W=1,5 \text{ GW/m}^2$)

In contrast with microstructure of other CPF-treated metal-silicon systems (Cr/Si, Ti/Si, Fe/Si, Zr/Si) /9/ silicon cells have no prolonged dendritic branches. This is connected with the decrease of constitutional overcooling due to low difference between temperatures of eutectic solidification (1673 K) and silicon crystallization (1687 K).

Conclusions

The action of compression plasma flows with power density 0,2-0,4 GW/m² on “molybdenum layer-silicon substrate” systems results in the formation of asperities at the surface (up to 30 nm height, average distance between tops is 200 nm, lateral size is ~100 nm) due to partial evaporation of the molybdenum film, nucleation in the shock-compressed plasma layer followed by re-deposition on the surface and migration due to thermal-activated diffusion. Increase of power density up to 1,5 GW/m² leads to the formation of uniform molybdenum-alloyed silicon layer 3-5 μm thickness, silicon cells with lateral size ~1 μm with molybdenum is localized at the edges. Cells formation is connected with molybdenum edging out the solidification front due to its low solid solubility in silicon and their growth direction is determined by crystal-lattice orientation of single crystal substrate and direction of heat transfer.

References

1. **V.M. Anishchik, V.V. Uglov, V.V. Astashynski, V.M. Astashynski, S.I. Ananin, E.A. Kostyukevich, A.M. Kuzmitski, N.T. Kvasov, A.L. Danilyuk, I.N. Rumianceva.** *Vacuum* **70** (2003) 269–274
2. **V.V. Uglov, V.M. Anishchik, V.M. Astashynski, N.N. Cherenda, I.G. Gimro and A.V. Kovyazo.** *Surface and Coatings Technology* **200** (2005) 245-249.
3. **V.V. Uglov, R.S. Kudaktsin, Yu.A. Petukhou, N.T. Kvasov, A.V. Punko, V.M. Astashynski, A.M. Kuzmitski.** *Applied Surface Science* **258** (2012) 7377-7383.
4. **T. Yamada, H. Yamane.** *Intermetallics* **19** (2011) 908-912
5. **E. Bruneton, S. Martoia, S. Schelz.** *Thin Solid Films* **519-2** (2010) 605-613
6. **V.M.Astashynski, S.I.Ananin, V.V.Askerko, E.A.Kostyukevich, A.M.Kuzmitski, V.V.Uglov, N.N.Cherenda, V.M.Anishchik, Yu.V.Sveshnikov, V.V.Astashynski, N.T.Kvasov, A.L.Danilyuk, A.V.Punko.** *Vacuum* **78/2-4** (2005) 157-160
7. *Crystal Growth of Si for Solar Cells.* Ed. **K. Nakajima, N.Usami** (2009) 219-251
8. *Smithells Metal Reference Book.* Ed. **E.A. Brandes, G.B. Brook** (1999) 11-220.
9. **Yu.A. Petukhov, V.V. Uglov, N.T. Kvasov, V.M. Astashynski, A.M. Kuzmitski.** *Proceedings of 10th International Conference on Modification of Materials with Particle Beams and Plasma Flows, Tomsk* (2010) 136-139