INFLUENCE OF PROTON IRRADIATION ON STRUCTURE AND MECHANICAL PROPERTIES OF NANOSCALE MULTILAYER METALLIC ZR/NB COATING

D.G. Krotkevich

Scientific Supervisor: E.B. Kashkarov

National Research Tomsk Polytechnic University, Tomsk, Russia, Lenin Avenue 43, 634050

E-mail: dimka.kro@gmail.com

ВЛИЯНИЕ ОБЛУЧЕНИЯ ПРОТОНАМИ НА СТРУКТУРУ И МЕХАНИЧЕСКИЕ СВОЙСТВА НАНОРАЗМЕРНОГО МНОГОСЛОЙНОГО МЕТАЛЛИЧЕСКОГО ПОКРЫТИЯ Zr/Nb

Д.Г. Кроткевич

Научный руководитель: Е.Б. Кашкаров

Национальный исследовательский Томский политехнический университет, Россия,

г. Томск, пр. Ленина, 43, 634050

E-mail: dimka.kro@gmail.com

Аннотация. *Образцы наноразмерного многослойного Zr/Nb покрытия, полученные путем магнетронного распыления, были подвергнуты облучению пучком протонов с током 2 мкА и 4 мкА на линейном ускорителе ЭСГ-2,5. Анализ распределения протонов и дефектов по глубине был проведен в программном пакете SRIM. Методом рентгеноструктурного анализа определены размер кристаллитов и микронапряжения после облучения. Оценка изменения дефектной структуры покрытий также была проведена по результатам измерения электросопротивления. Механические характеристики, такие как твердость и модуль Юнга были рассчитаны из данных по наноиндентированию.*

Introduction. Nanoscale multilayer metallic (NMM) coatings represent a perspective class of heterogeneous materials, they are widely studied to determine the correlation between materials properties (interfaces, grain size, etc.) with mechanical properties [1, 2] and radiation damage tolerance [3-5].

One system deserves a special attention: Zr/Nb multilayer coating. The Zr-Nb alloys are widely known as construction material for nuclear industry due to high mechanical properties, radiation damage tolerance, and low capture cross-section for thermal neutrons. Thereby, the use of Zr/Nb multilayer coatings should show improved features of Zr-Nb alloys as a result of their layered structure. Also it should lead to high immiscibility due to incoherent (hcp-Zr, bcc-Nb) type of interface [6].

In this paper, the technique of creating irradiation-induced defects using linear ion accelerator with Van de Graaf generator is described. Also evolution of microstructure and mechanical properties due to irradiation is shown.

Materials and methods. The Zr/ Nb multilayer coating was deposited on single crystal Si substrate via DC magnetron sputtering. The total thickness of the coating was 1350 nm; the thickness of individual layer was 57 nm for Zr and 39 nm for Nb. The penetration depth and distribution of defects were determined with SRIM-2013 simulation code. For irradiation, specimens were covered with Al foil with thickness of 11 μm (in order to reduce the energy of proton beam) before they were mounted in the chamber of linear ion accelerator with Van de Graaf generator. They were exposed to 900 keV proton beams with dose range from 4.5×10^{16} to 1.35×10^{17} protons/cm^2,the irradiation time ranged from 1 to 3 hours, and ion current was 2 and 4 μA. X–ray diffraction

(XRD) analyses before and after irradiation were performed on Shimadzu XRD-700S diffractometer with Cu Kα radiation (λ = 0,154 nm) to observe changes in microstructure. The hardness and the Young's modulus of the initial and irradiated coatings were evaluated by a nanoindentation method using the Berkovich indenter. Also electrical resistance was measured.

Results and discussion. The depth profile and vacancy distribution per layer of 900 keV proton beam were carried out using SRIM-2013 simulation code. The most probable stopping range is 850-950 nm.

The XRD pattern for irradiated specimens (Fig. 1) revealed-that no additional reflexes were found but variations in the reflex intensities and FWHM were clear.

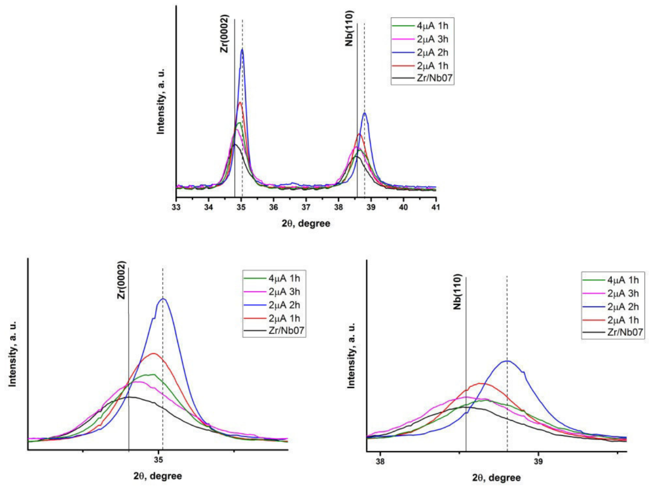

Fig.1. XRD pattern of irradiated samples with detailed information of Zr(0002) plane and Nb(001) plane

As demonstrated in Figure 1, proton beam irradiation resulted in peak shifting toward higher 2Θ angle, it probably means that structure was distorted due to stress caused by irradiation. In all cases intensities were increased during the irradiation.

The values of FWHM along Zr(0002) and Nb(110) planes are given in Table 1. As seen from the table, irradiation with 2 μA current and time 1 and 2 hours led to considerable decrease in FWHM value along both (Zr and Nb) planes, however, increasing the time to 3 hours or current to 4 μA caused nearly no changes in FWHM value.

Table 1

Structural parameters of Zr (0002) and Nb (110) planes before and after proton irradiation

Plane	**Zr (0002)**			**Nb (110)**		
Irradiation parameters	FWHM, degrees	Crystalline size, nm	Microstrain	FWHM, degrees	Crystalline size, nm	Microstrain
native	0.69	12.51	0.0029	0.80	10.93	0.00331
2 μA, 1 h	0.47	18.71	0.00194	0.58	15.13	0.00239
2 μA, 2 h	0.35	25.04	0.00145	0.45	19.57	0.00185
2 μA, 3 h	0.67	12.93	0.0028	0.79	11.02	0.00329
4 μA, 1 h	0.55	15.84	0.00229	0.76	11.53	0.00314

As in case of the intensities of diffraction peak the most considerable change in crystalline size and microstrain were observed for current 2 μA and time 1 and 2 hours, thus in all cases irradiation led to increase in crystalline size and decrease of microstrain value. This phenomenon probably occurred due to ion-bombardment-induced grain growth (IBGG) in thin films [7].

Measurements of electric resistance showed the same picture, the most significant changes in values were observed for 2 μA current and time 1, 2 hours, proving that proton irradiation leads to a kind of ordering of the structure.

Formed nanoscale multilayer coatings showed high mechanical characteristics: hardness 4.9 GPa and Young's modulus of 128.9 GPa. Irradiation with a proton beam caused significant changes in the mechanical properties: hardness and Young's modulus (Fig. 2)

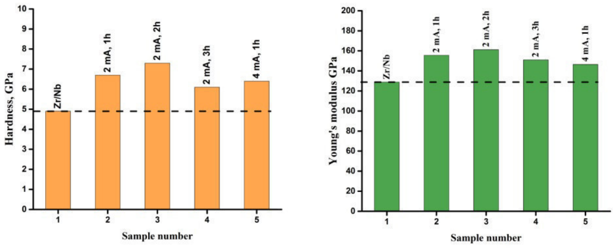

Fig. 2. Hardness and Young's modulus obtained from nanoindentation tests before and after proton irradiation

After irradiation, the hardness and Young's modulus of coatings increased: by 37% and 21% (2 μA, 1 h), 49% and 25% (2 μA, 2 h), 24% and 17% (2 μA, 3 h) , 48% and 13% (4 μA, 1 h), respectively.

Conclusion. Samples of the Zr/Nb NMM coating were irradiated with proton beam on the linear accelerator with Van de Graaf generator. The correlation between XRD results, FWHM narrowing, crystallite size, microstrain, changes in values of electrical resistance and mechanical properties were observed.

REFERENCES

1. Callisti M., Polcar T. Combined size and texture-dependent deformation and strengthening mechanisms in Zr/Nb nano-multilayers //Acta Materialia. – 2017. – Vol. 124. –pp. 247-260.
2. Zbib H. M. et al. Analysis of plastic deformation in nanoscale metallic multilayers with coherent and incoherent interfaces //International Journal of Plasticity. – 2011. – Vol. 27. – №. 10. – pp. 1618-1639.
3. Gao Y. et al. Radiation tolerance of Cu/W multilayered nanocomposites //Journal of Nuclear Materials. – 2011. – Vol. 413. – №. 1. –pp. 11-15.
4. Chen F. et al. Atomic simulations of Fe/Ni multilayer nanocomposites on the radiation damage resistance //Journal of Nuclear Materials. – 2016. – Vol. 468. – pp. 164-170.
5. Misra A. et al. The radiation damage tolerance of ultra-high strength nanolayered composites //JOM Journal of the Minerals, Metals and Materials Society. – 2007. – Vol. 59. – №. 9. –pp. 62-65.
6. Frutos E. et al. Length-scale-dependent mechanical behaviour of Zr/Nb multilayers as a function of individual layer thickness //Materials Science and Engineering: A. – 2015. – Vol. 632. – pp. 137-146.
7. Seita M. et al. Selective ion-induced grain growth: Thermal spike modeling and its experimental validation //Acta materialia. – 2013. – Vol. 61. – №. 16. – pp. 6171-6177.