



INSTITUTE OF NUCLEAR PHYSICS

Republic of Kazakhstan

Comparative Studies of the Irradiation Effects of ZrWN Coating with Helium and Krypton Ions at Low and High Temperatures

*Sergey Kislitsin¹, Alexandr Potekaev², Anatolii Klopotov²,
Tamara Aldabergenova¹*

¹ Institute of Nuclear Physics, Almaty, Kazakhstan

² National Research Tomsk State University, Tomsk, Russia

³ Tomsk State Architecture and Building University, Tomsk, Russia

SCOPE

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1. MOTIVATION

The results of our previous studies of the ion irradiation effect on the properties of triple nitride coatings ZrTiN, MoTiN showed a rather high stability of the structure and properties of these coatings.

Uglov V.V., et al. "Stability of Ti-Zr-N coatings under Xe-ion irradiation" *Surface&Coatings Technologies*, Vol. 204, pp.2095 – 2098, 2010.

Kislitsin S. B. , et al. Steel surface, TiCrN, TiMoN coatings structural phase state change features after low-energy alpha particles irradiation. *IOP Conf. Series: Materials Science and Engineering*. V.289, pp. 012010 (1-6). 2018.

Aldabergenova T.M., et al. Thermal annealing effect on structure and properties of tungsten surface irradiated with high fluence and low energies alpha-particles . *Russian Physics Journal*. V.61, No 8, P.1499 – 1505, 2018.

In present work the effect of low-energy He and Kr ions on the structure and properties of ZrWN coatings has been studied. Replacing of Ti with tungsten caused by the fact that, according to our idea, tungsten will give high strength and thermal resistance to the coating.

2. SAMPLES PREPARATION

The ZrWN coatings were synthesized on substrate from Cr18Ni10Ti stainless steel by the method of magnetron sputtering of targets from two magnetrons. The sputtered targets Zr (99.96 wt% purity) and W (99.97 wt% purity) were used for this purpose. Gas mixture with nitrogen content of ~ 25% was used as a plasma-forming gas. Thickness of the applied coatings was controlled by the coating application time. The required coating thickness should be ~ 600 nm, since the samples with ZrWN coatings are designed for irradiation with krypton ions of 280 keV energy and alpha-particles of 45 keV energy. The projective range of the ions should be completely located in the coating in order to exclude the influence of the steel substrate. The projective range of the ions should be completely located in the coating in order to exclude the influence of the steel substrate. Calculations of the projective range of the low-energy krypton ions (280 keV) and alpha-particles (45 keV) in ZrWN coatings by SRIM software showed that the projective range does not exceed 100 nm, therefore, the coatings ~ 600 nm thick satisfy the specified task.

3. INVESTIGATION METHODS AND IRRADIATION PROCEDURE

- *X-ray diffractometry (XRD)*
Diffractometer **BRUCKER D8 Advance**



- *Scanning electron microscopy (SEM)*
Jeol **JSM-7500 FA**



- *Atomic force microscopy (AFM)*
AIST-NT



- **Hardness measurements (H)**
Scanning nanohardness meter
«Nanoscan-Compakt»



- **Corrosion resistance (CR)**
Corrosion cell «Gamry Instruments»



- **Rutherford backscattering (RBS)**
Accelerator UKP – 2-1



IRRADIATION OF ZrWN COATINGS WITH LOW-ENERGY He, Kr, IONS



HEAVY ION ACCELERATOR DC-60 *INP BRANCH in Astana*

Low-energy channel DC-60
(channel of ECR source):
E - 20÷25 keV/ charge

Irradiation conditions with low-energy
helium and krypton ions

Marking of samples	Ion type	Energy KeV	Radiation T, °C	Fluence cm ⁻²	Beam current μA
ZrWN-2	⁴ He ²⁺	45	room	10 ¹⁶	25
ZrWN-3	⁴ He ²⁺	45	600	10 ¹⁷	25
ZrWN-4	⁸⁴ Kr ¹⁴⁺	280	room	10 ¹⁶	10
ZrWN-5	⁸⁴ Kr ¹⁴⁺	280	600	10 ¹⁷	10

Projective range (SRIM):
He (40 keV) ~ 120 nm
Kr (280 keV) ~ 80 nm

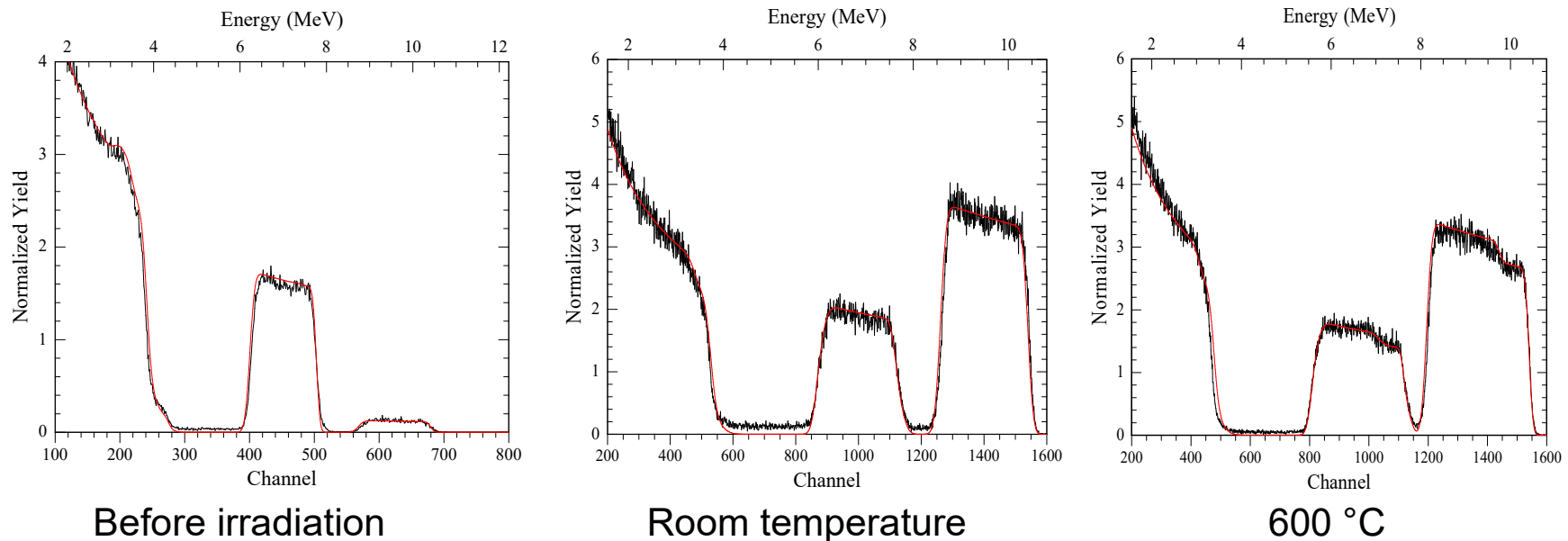


4. RESULTS

Sputtering

Since low-energy ion irradiation can lead to a noticeable sputtering of surface, the thickness and changes in the elemental composition were controlled experimentally as before then after ion irradiation. The thickness of the coatings was controlled by the Rutherford backscattering method (RBS) on the proton beam. For He ions with 45 keV energy sputtering yield for Zr, W and N atoms is ~ 0.01 at/ion (SRIM), therefore thickness of the sputtered layer ~ 0.9 nm at the fluence of 10^{17} cm^{-2} and can not be detected by RBS.

Spectra's of backscattered protons for the samples with ZrWN coatings irradiated with Kr ions of 280 keV energy

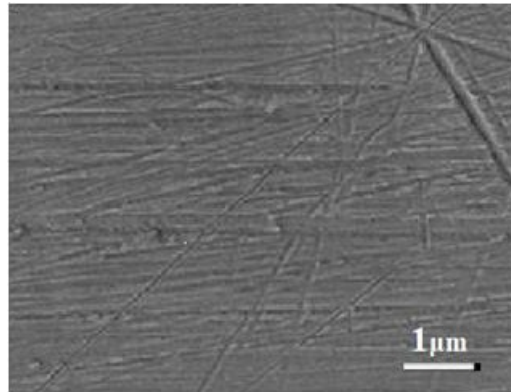


For Kr ions with 280 keV energy sputtering yield for Zr, W and N atoms is ~ 2.0 at/ion.
Thickness of the sputtered layer at the fluence of 10^{16} cm^{-2} at room temperature – 20 nm (measured) [12 nm calc]
Thickness of the sputtered layer at the fluence of 10^{17} cm^{-2} at 600 °C – 100 nm (measured) [120 nm calc]

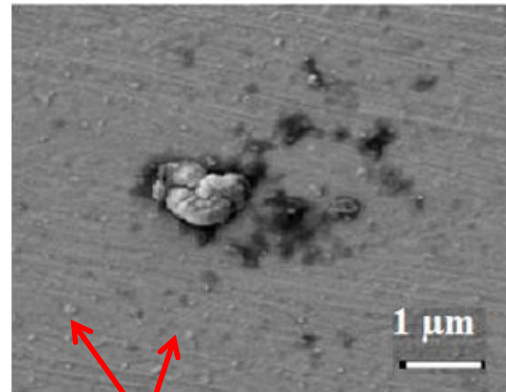
Changes in the structure of the ZrWN coating surface under irradiation with low-energy helium and krypton ions.

Alpha- particles $E = 45 \text{ keV}$, $R_p = 100 \text{ nm}$

Before irradiation

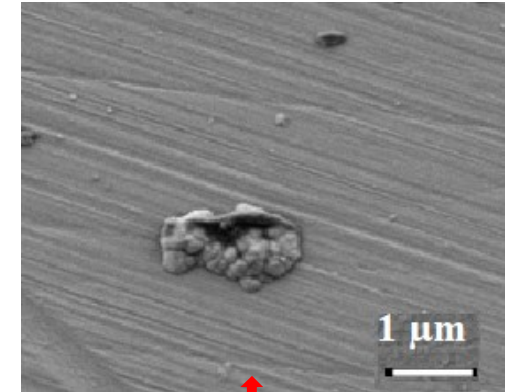


$F = 10^{16} \text{ cm}^{-2}$, room temperature



Appearance of small ($\sim 20\text{-}50 \text{ nm}$) rounded formations on the surface during room temperature irradiation, which can be treated as bubbles filled with helium.

$F = 10^{17} \text{ cm}^{-2}$, $600 \text{ }^\circ\text{C}$

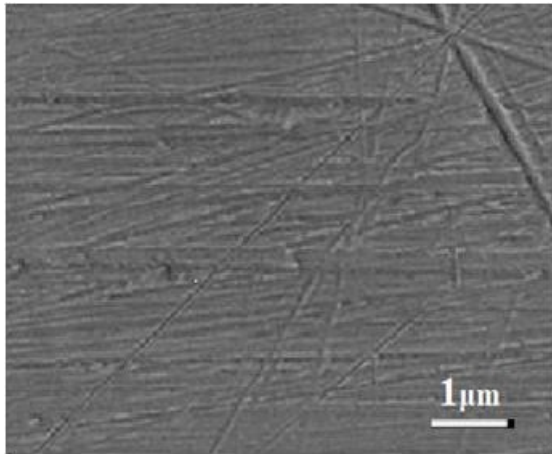


Surface smoothing

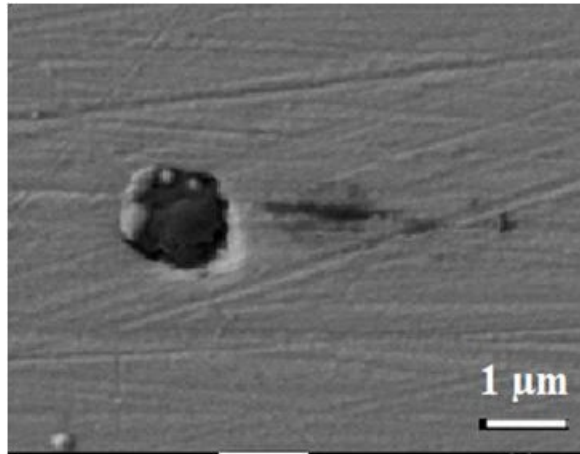
Irradiation with low energy He ions up to fluence 10^{17} cm^{-2} not leads to changes in elemental composition of the surface layers

$^{84}\text{Kr}^{14+}$, $E = 280 \text{ keV}$, $R_p \sim 100 \text{ nm}$

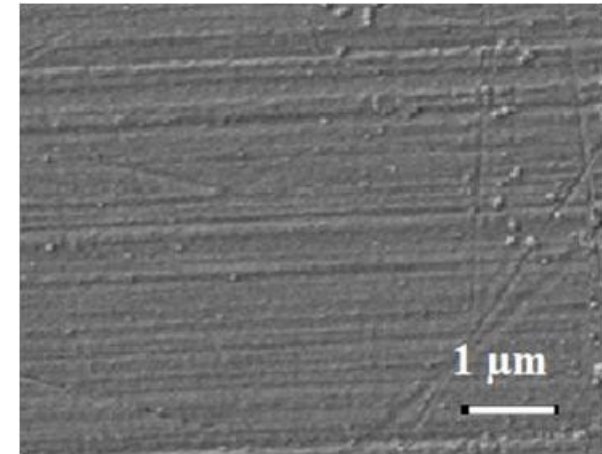
Before irradiation



$F = 10^{16} \text{ cm}^{-2}$, room temperature



$F = 10^{17} \text{ cm}^{-2}$, 600 °C



Surface smoothing

Smoothing of irradiated surface is observed. Craters from incident Kr ions, similar to those visible under irradiation of steel with Kr ions, are not recorded. Irradiation to the fluence of 10^{17} cm^{-2} at 600 °C stimulates more significant changes in the surface morphology.

Influence of irradiation with low-energy helium and krypton ions on physical-mechanical properties of ZrWN coating

Hardness

Hardness of the ZrWN coating before irradiation was 7.8 GPa, which significantly exceeds nanohardness (Hn) of steel (4.4 GPa). Irradiation with low-energy He ions of 45 keV energy to the fluence of 10^{16} cm⁻² at room temperature leads to a decrease in Hn to 3.8 GPa, and irradiation at the temperature of 600 °C to the fluence of 10^{17} m⁻² reduces the hardness to 0.9 GPa. Irradiation with low-energy krypton ions also results in hardness decrease of the ZrWN coating, but to a lesser extent than under irradiation with He ions. So, irradiation with Kr ions of 280 keV energy to the fluence of 10^{16} cm⁻² at room temperature stimulated a decrease in Hn to 4.8, and at the temperature of 600 °C to the fluence of 10^{17} cm⁻² – to 3.2 GPa.

Corrosion resistance

Corrosion resistance of irradiated ZrWN coatings degrades under irradiation with light (He) and heavy ions (Kr).

Coating	Ion type, energy	Fluence cm ⁻²	T irradiation, °C	Vcorr, mm/year
ZrWN	Non-irradiated	-	-	13.3×10^{-3}
ZrWN - 2	He, 45 keV	10^{16}	room	24×10^{-3}
ZrWN - 3	He, 45 keV	10^{17}	600	78×10^{-3}
ZrWN - 4	Kr, 280 keV	10^{16}	room	15×10^{-3}
ZrWN - 5	Kr, 280 keV	10^{17}	600	16×10^{-3}

5. CONCLUSION

The effect of irradiation with low-energy He and Kr ions at room temperature and 600 °C on the structure and properties of ZrWN coatings has been studied. Results of investigations allow to draw following conclusions:

- Irradiation with helium ions does not lead to changes in the structural-phase composition of the coating;
- Irradiation with krypton ions leads to formation new phase;
- Broadening of the X-ray reflections from structural defects generated by irradiation is observed;
- Irradiation with low energy He ions up to fluence 10^{16} cm⁻² does not cause significant changes in the surface structure;
- Irradiation with low-energy Kr ions leads to surface sputtering and a decrease in coating thickness. Sputtering is accompanied by a change in elemental composition of the surface layers due to differences in partial sputtering coefficients;
- Mechanical properties (hardness) and corrosion resistance of ZrWN coatings degrade under the influence of irradiation with low-energy He and Kr ions. Irradiation at high temperatures leads to a more considerable degradation of the properties of the ZrWN coating.
- The hardness and corrosion resistance of the irradiated coating exceed these characteristics of steel in a great extend.