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Neutron irradiation effects on properties of insulator coatings for ITER in-vessel components

B.S. Rodchenkov [a,*], V.M. Ivanov [b], G.M. Kalinin [a], A.V. Kozlov [c], Yu.S. Strebkov [b], E.N. Scherbakov [c]

[a] *Engineering Centre of Nuclear Equipment Strength, Reliability and Lifetime (ENES), P.O. Box 788, Moscow 101000, Russia*
[b] *Research and Development Institute of Power Engineering (RDIPE), P.O. Box 788, Moscow 101000, Russian Federation*
[c] *Sverdlovsk Branch, Research and Development Institute of Power Engineering, Zarechnyi, 624, Russian Federation*

Abstract

Insulator coatings are used in ITER to reduce currents flowing from the first wall modules to the vacuum vessel and between the limiter plates. Al_2O_3 and Al_2MgO_4 are proposed as an insulating materials. Coatings should be sprayed by plasma on the surfaces of first wall keys, flexible cartridge and on the lateral sides of the ITER port-limiter plates. This paper deals with the investigation of electrical and mechanical properties of insulator coatings including irradiation effect. Specimens were manufactured from austenitic stainless steel 18Cr–10Ni–Ti and titanium alloy *П*T-3B grade. Insulator coatings made from Al_2O_3, Al_2MgO_4 and $Al_2O_3 + 13\%TiO_2$ have been tested. The NiCr intermediate layer has been used to provide better adhesion of insulator coatings with the substrate. The specimens were irradiated in IVV-2M reactor at the temperature 240 °C. The irradiation dose was ~0.16 dpa. The electrical conductivity and break-down voltage of coatings were measured before and after irradiation. Appearance of cracks was also studied after compression loading up to 200 MPa.
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1. Introduction

Insulator coatings are used in ITER to reduce currents flowing from blanket modules and in-vessel components to the vacuum vessel. The applied voltage is estimated below 50 V. Coatings for the flexible cartridges of the blanket modules, keys and central pin of the module alignment will operate at the temperatures 150–300 °C, compression loading up to 200 MPa, thermal–mechanical cycles and irradiation. The maximum estimated damage dose of irradiation is about 0.3 dpa. Alumina, aluminium–magnesium oxide (spinel structure) and these oxides mixed with titanium oxide were selected for the insulating coatings. Plasma spraying was selected as a reference method for depositing the ceramic coating.

The plasma sprayed Al_2O_3 and $MgAl_2O_4$ have been successfully used as an electrical insulating materials for the core application of fission reactors [1]. These materials were also used for tokamak JT-60 [2]. Influence of irradiation on the properties of these insulating materials was investigated mainly for the materials manufactured by hot pressing or by another sintering methods [3–5]. Relatively limited information is available for plasma sprayed materials [1,6].

Electrical and mechanical properties that are critical for the ITER application were investigated and results are presented in this paper.

2. Experimentals

Austenitic stainless steel of 18Cr–10Ni–Ti type and titanium alloy *П*T-3B grade (Ti + (3.5–5)%Al +

* Corresponding author. Tel.: +7-095 263 7444; fax: +7-095 264 7934.
E-mail address: rodchen@entek.ru (B.S. Rodchenkov).

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(1.5–2)%V) were used as the substrate materials. The austenitic 18Cr–10Ni–Ti steel (Russian designation 08X18H10T) is similar to 316L(N)-IG steel proposed to manufacture the ITER blanket components. Titanium alloy is one candidate material to manufacture the flexible cartridge for the blanket modules [6]. NiCr alloy was used as an intermediate layer to improve adhesion of coating.

The samples were made in the form of disks with 15 mm diameter and 4–5 mm thickness height. Firstly, the NiCr intermediate layer was sprayed on one side of disks (sand-blasted before spraying), then the insulating coatings were applied. The characteristics of investigated coatings are presented in the Table 1. An additional layer containing $Al_2O_3 + 13\%TiO_2$ with thickness of about 0.05–0.1 mm was deposited to improve resistance to wear.

The samples were irradiated in IVV-2M reactor at temperature 240 ± 10 °C by fast neutron flux 1.6×10^{14} 1/cm^2 s ($E > 0.1$ MeV) that approximately corresponds to a damage dose ~0.16 dpa. For irradiation the samples were placed into a capsule filled with He under pressure ~0.8 MPa.

The electrical resistivity, break-down voltage and capacity to withstand the compression load up to 200 MPa were determined before and after irradiation.

The electrical resistance was measured at 20 °C in accordance with requirements of GOST 6433.2 [7]. Guard ring has been used to prevent the surface current effect on the results of electrical resistance measurement of irradiated specimens. The specimens were heated to 55–60 °C and exposed for 1 h before measurement of electrical resistance to avoid any effect of humidity. The measurement accuracy of break-down voltage was no worse than ±0.2 kV. The axial load compression tests were carried out on test machine equipped with the test assembly located in the hot cells. Test temperature was 20 °C. The view of the test assembly is shown schematically in Fig. 1. The loading shaft was 6 mm in diameter.

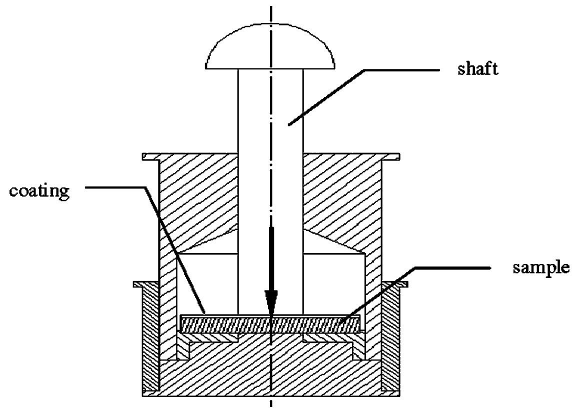

Fig. 1. Schematical view of test assembly for the ceramic coatings examination under compression load.

3. Results and discussion

The results of electrical resistivity measurements on the insulating coatings before and after irradiation are summarized in Table 2. The various types of coatings in the unirradiated state have small difference in electrical resistivity. Minimum value of electrical resistivity $(6.7 \pm 0.1) \times 10^{12}$ Ω cm was measured for the double-layer coating ($Al_2O_3 + Al_2O_3 \cdot 13\%TiO_2$) sprayed on the austenitic steel substrate. Maximum value was ~$(18 \pm 1) \times 10^{12}$ Ω cm for the Al_2O_3 and $MgAl_2O_4$ coatings sprayed on the Ti substrate.

Irradiation resulted in a decrease of electric resistivity for all investigated insulating coatings. The reduction coefficient of electrical resistivity due to irradiation, K_{irr} was calculated. K_{irr} equals to the ratio of electrical resistivity of irradiated specimen to the unirradiated value. The maximum reduction of electrical resistivity after irradiation was observed for the coatings ($Al_2O_3 + Al_2O_3 \cdot 13\%TiO_2$) and ($MgAl_2O_4 + Al_2O_3 \cdot 13\%TiO_2$) sprayed on the austenitic steel. The minimum effect was found for one layer Al_2O_3 coatings sprayed on Ti-alloy ($K_{irr} = 1.3$) and on steel ($K_{irr} = 3.8$).

Table 1
Characteristics of investigated samples

Substrate (material)	Intermediate coating		Coating	
	Material	Thickness (mm)	Material	Thickness (mm)
18Cr–10Ni–Ti	NiCr	0.08	Al_2O_3	$0.5_{-0.05}$
18Cr–10Ni–Ti	NiCr	0.06	$MgAl_2O_4$	$0.5^{\pm 0.02}$
18Cr–10Ni–Ti	NiCr	0.05	$Al_2O_3 + Al_2O_3 \cdot 13\%TiO_2$	$0.5^{\pm 0.02} + 0.05^{\pm 0.02}$
18Cr–10Ni–Ti	NiCr	0.05	$MgAl_2O_4 + Al_2O_3 \cdot 13\%TiO_2$	$0.46^{\pm 0.04} + 0.05^{\pm 0.02}$
*П*T-3B	NiCr	0.05	Al_2O_3	$0.48^{\pm 0.02}$
*П*T-3B	NiCr	0.05	$MgAl_2O_4$	$0.5^{\pm 0.05}$

Table 2
Electrical resistivity of coatings before and after irradiation

Coating	Substrate material	Electrical resistivity ρ, 10^{12} Ωcm		
		Before irradiation	After irradiation	Reduction coefficient (κ_{irr})
Al_2O_3	Steel	13.9–14.5	4.0–3.4	3.8
$MgAl_2O_4$	Steel	15.8–15.0	2.1–1.3	9.0
$Al_2O_3 + Al_2O_3 \cdot 13\%TiO_2$	Steel	13.9–13.1	0.2–0.08	99
$MgAl_2O_4 + Al_2O_3 \cdot 13\%TiO_2$	Steel	6.9–6.5	0.02–0.01	670
Al_2O_3	Ti-alloy	17.7–16.9	13.7–12.7	1.3
$MgAl_2O_4$	Ti-alloy	18.8–17.8	4.9–3.9	4.1

The results of electrical strength (break-down voltage) measurement of insulating coatings before and after irradiation are presented in Fig. 2. The coatings had break-down voltages in the range 2–5 kV for initial condition. The break-down voltage of one layer coatings Al_2O_3 was 3.5 ± 0.2 kV. An additional layer of Al_2O_3 with 13%TiO_2 decreased the electric strength of this type coating to 2.5 ± 0.2 kV. One layer coating of spinel $MgAl_2O_4$ had an intermediate value of electric strength $\sim 3.0 \pm 0.2$ kV. Both Al_2O_3 and $MgAl_2O_4$ coatings sprayed on the Ti-alloy had almost the same values of the break-down voltage, 4.5 ± 0.2 and 4.0 ± 0.2, respectively. The irradiation reduced the electric strength of insulating coatings by 10–30%. The reduction coefficient was approximately the same for one-layer coatings applied both to steel and Ti-alloy, 1.12–1.16. The most significant decrease of electrical strength was observed for the multi-layer coating Al_2O_3-Al_2O_3 + 13%TiO_2 with $K_{irr} = 1.43$.

It was found that all investigated insulating coatings withstand the axial compression load up to 5 ton without visual mechanical damages (cracks, breaks, etc.) in the unirradiated state. The possibility of compression loading in hot cell was limited by load to ~600 MPa. All irradiated coatings survived with no visual damage on the surface after axial compression tests.

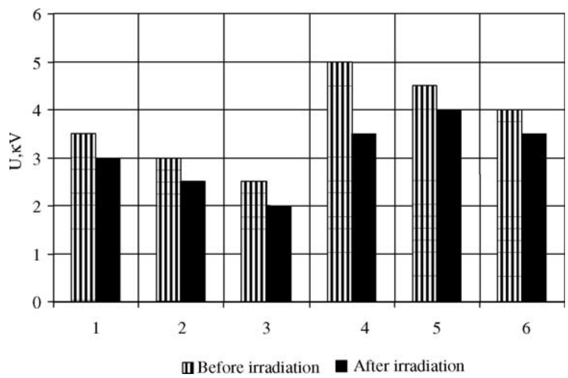

Fig. 2. Break-down voltage of insulating coatings. 1 – SS–NiCr–Al_2O_3, 2 – SS–NiCr–$MgAl_2O_4$, 3 – SS–NiCr–Al_2O_3–(Al_2O_3 + 13%TiO_2), 4 – SS–NiCr–$MgAl_2O_4$–(Al_2O_3 + 13%TiO_2), 5 – Ti–NiCr–Al_2O_3, 6 – Ti–NiCr–$MgAl_2O_4$.

Summarizing results, it should be noted that the irradiation to a dose ~0.16 dpa at 240 °C resulted in a decrease in the electrical resistivity of all investigated coatings. Taking into account that the thickness of all coatings was practically the same, the observed difference in coatings behaviour under irradiation are resulted from two factors: influence of the material type and quality of sprayed coatings. The least effect of irradiation was found for Al_2O_3 coatings sprayed on austenitic steel and Ti-alloy. Moreover, the better irradiation resistance was exhibited by monolayer ceramic coatings. The effect of irradiation on multi-layer coating on the base of Al_2O_3 with addition of 13%TiO_2 was more significant than for single layer coatings. The properties degradation due to irradiation for $MgAl_2O_4$ (spinel) coating was higher than for Al_2O_3 coating.

The results are correlated with earlier data obtained in [3,4,8] for insulating ceramics and plasma sprayed coatings during in pile measurement and post irradiation investigation. The value of conductivity increased by a few orders of magnitude due to irradiation in comparison with unirradiated condition, and depends on irradiation conditions, type of coating material, and regimes of deposition that affect the structure and porosity. The results show that the low electrical conductivity both in unirradiated and irradiated condition was exhibited by the high purity ceramics. Additions of titanium oxide resulted in increase of conductivity. The electrical resistivity, break-down voltage and compression strength of coatings satisfy the requirements for electrical insulation of ITER in-vessel components [6].

4. Conclusion

- Irradiation at the temperature 240 °C up to damage dose of 0.16 dpa results in decrease of the electrical resistivity and break-down voltages of plasma sprayed insulating coatings of Al_2O_3 and $MgAl_2O_4$.
- The monolayer Al_2O_3 coatings exhibited the least irradiation effect compared with multi-layer coatings of $MgAl_2O_4$ and Al_2O_3 with $Al_2O_3 \cdot 13\%TiO_2$.
- All investigated insulating coatings irradiated at 240 °C to 0.16 dpa kept the electrical characteristics

and compression mechanical properties required for ITER in-vessel components.
- The preferred coating is Al_2O_3 for which the change of service properties (electrical resistivity, breakdown voltages and compression strength) after irradiation was a minimum.

References

[1] V.M. Ivanov, V.V. Kudinov, Plasma spraying of high temperature coatings. M., Mashinostroenie, 1981 (in Russian).
[2] M. Onozuka et al., Fusion Technol. 29 (1996) 73.
[3] C. Kinoshita, S. Zinkle, J. Nucl. Mater. 233–237 (1996) 100.
[4] T. Shikama, S. Zinkle, J. Nucl. Mater., 258–263 (1998) 1861.
[5] L. Snead, D. White, S. Zinkle, J. Nucl. Mater., 226 (1995) 58.
[6] Materials Assessment Report, ITER Doc. G A1 DDD 198-05-28 W 0.3, 1998.
[7] GOST 6433.2-71, Electro-insulator materials. Methods of electrical conductivity measurement under constant applied voltage (in Russian).
[8] K. Nada et al., J. Nucl. Mater. 233–237 (1996) 1289.