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Investigation of fusion reactor candidate materials erosion in plasma disruption simulation experiments

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Abstract

The results of the investigations on new kinds of perspective materials for the high power units of fusion reactors in conditions of action simulating the expected plasma disruptions have been presented. Erosion, changes of the surface topography, the element and phase composition of the intermetallic alloys of a Ti–Al–V system and the type GR reactor graphite impregnated with an 50wt%Ti/50wt%Cu alloy under the action of hydrogen pulsed plasma fluxes with a maximum ion energy of 2 keV have been studied. The flux specific energy Q was equal to 100 J/cm^2, the pulse duration changed in the 15–20 μs range and the number of pulses N was from 3 to 30. It has been established that the heat-treated intermetallic alloy containing 15 at.% V has a high erosion stability depending on the number of irradiation pulses and has a minimum erosion coefficient of 0.2–0.3 μg/(J cm^2) in comparison with investigated alloys. The impregnated graphites are subjected to less erosion compared to that of the initial reactor graphite at a sufficiently small number of irradiation pulses ($N \leqslant 15$). © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

At present the choice of materials for plasma facing components (first wall, divertor, limiters and baffles) of developed large-scale thermonuclear installations and future fusion reactors (FR) with a magnetic confinement of plasma is one of the principal problems of their creation, in particular the creation of an international thermonuclear reactor (ITER) [1–3].

In the ITER the working conditions of the plasma facing materials (PFM) are characterized by extremely stringent requirements [2,3], which at a normal operating regime of the reactor are caused, first of all, by the influence of intensive thermal and corpuscular flows as well as by powerful short-time thermal impacts induced by plasma disruptions. All these effects result in erosion, changes of the structural and phase state, and the physical and mechanical properties of these materials [4–8].

Severe working conditions and lack of data on the properties of materials and the features of their behavior in a real construction explain that many different types of perspective materials are being considered at present. At the current stage of developing the ITER, consideration is being given to three main kinds of candidate armour materials: beryllium, carbon-base materials, tungsten and its alloys [2,3]. The currently available data on the behavior of these materials in conditions simulating the expected plasma disruptions are still lacking.

Taking into consideration that at the moment the final choice of PFM has not been made yet, the investigation of new perspective kinds of materials is of great importance.

The alloys of a Ti–Al system are considered as a perspective basis for manufacturing the first wall, with working temperatures of up to 700–1000°C. The γ-TiAl alloys have a low density ($\rho = 3.9$ g/cm^3), a relatively low atomic number Z, high melting temperatures (~1500°C) and high resistance to oxidation at temperatures of up to 900–1000°C [9].

The purpose of the present work is to discover the main erosion features of perspective armour materials for FR high power units: intermetallic alloys of Ti–Al–V system, tungsten, carbon materials, impregnated reactor graphite, in conditions simulating the expected plasma disruptions.

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2. Materials and experimental procedure

The following materials have been studied.

1. The intermetallic alloys of Ti–Al and Ti–Al–V systems with a vanadium content of 2, 9 and 15 at.% in the charge (TiAl and TiAlV1, TiAlV2 and TiAlV3 alloys, respectively). In this the binary alloy 52at.%Ti/48at.%Al was chosen as a base alloy (TiAl). The alloys under investigation were made by a multiple arc melting in the atmosphere of argon. A number of these samples were thermally heated in vacuum ($P{\sim}10^{-5}$ torr) at 1473 K for 3 h (regime TO_1), then they were annealed at 873 K for 10 h (regime TO_2),
2. The type GR reactor graphite impregnated with a melt of the following composition 50wt%Ti/50wt%Cu. Samples 9 mm in diameter and 3 mm in height were made of the initial and impregnated graphite.

To compare a degree and a character of erosion of the given new materials with the similar characteristics of the ITER candidate materials [3,5], the silicicated graphites of SG-T (50–70% SiC) and PG-MS (50–55% SiC + 10–11% Cu) types and non-annealed sheet tungsten and sintered tungsten of technical purity (99.95%) were investigated.

The samples were irradiated by high temperature pulsed hydrogen plasma fluxes (HTPPF) with a maximum ion energy of about 2 keV at the incident flux specific energy $Q \approx 100$ J/cm^2 and a pulse duration of 15–20 μs. The number of pulses N changed from 3 to 30.

The samples were studied by metallography, scanning electron microscopy (SEM), X-ray spectral microanalysis (XRSMA) and qualitative X-ray phase analysis (QXRPA).

The erosion coefficients S [μg/(J cm^2)] of the irradiated samples were determined by mass loss. The error of measuring S basically depended on the accuracy of definition of the incident flux specific energy Q and was equal to about 30%.

3. Experimental results and discussion

The metallographic investigation of the Ti–Al–V system complex intermetallic alloys has shown that the microstructure of the TiAl alloy in its initial cast state is characterized by the presence of columnar dendritic crystals with a distance between the secondary axes of about 30–40 μm. The interaxial zones are marked by the formation of light precipitates, which according to [9] are a non-equilibrium γ-phase (TiAl-base solid solution). A dispersed plate-like (lamellar) structure forms in the volume of dendrites. The thickness of these plates does not exceed 1 μm. The structure of the TiAl alloy after its heat treatment (TO_1) is characterized by complete elimination of the dendritic segregation and by formation of practically equiaxed grains of about 100 μm in size.

Alloying by 2 at.% V slightly decreases the dendritic segregation; it was maximum for the alloy containing 15 at.% V among all the studied ones; the TiAlV2 alloy practically did not have it. In this, the TiAlV1 alloy has rather large grains of the plate-like structure; and as to the alloy TiAlV2, it is characterized by the formation of the “β-transformed” structure. The heat treatment of alloys containing 2–15 at.% V significantly, but not completely as opposed to the alloy TiAl, eliminates the dendritic segregation.

The QXRPA showed (Table 1) that the samples of the TiAl alloy in its initial cast state had a two-phase structure and consisted of γ-phase and α_2-phase, the last one being an intermetallic phase Ti_3Al. The α_2-phase content is less than that of γ-phase. Homogenizing annealing on regime TO_1 decreases the content of α_2-phase in the alloy. This agrees with the data of phase diagram [10].

The formation of $\gamma + \alpha_2$- and $\beta + \alpha_2$-phases, with preferential content of γ- and β-phases, respectively, was observed in the alloys with addition of 2 and 9 at.% of vanadium. The TiAlV3 alloy consists of $\gamma + \alpha_2 + \beta$ phases. The heat treated TiAlV1 alloy has two phases $\gamma + \alpha_2$ as before, TiAlV2 alloy has three phases $\gamma + \alpha_2 + \beta$ in its structure as a result of partial decomposition of β-phase, and annealed alloy TiAlV3 is two-phase ($\gamma + \beta$).

The investigations on erosion of intermetallic alloys showed (Table 2) that erosion coefficients of the studied alloys depended on their composition, the regime of their preliminary heat treatment and the number of irradiation pulses. In particular, at the starting stage of irradiation ($N = 5$) the initial two-phase alloy TiAl has

Table 1
Phase composition of the investigated alloys of system Ti–Al–V in various states

Material	State (preliminary treatment)	Main phases	
		Before irradiation	After irradiation
TiAl	Initial	$\gamma + \alpha_2$	α_2
TiAl	TO_1	$\gamma + \alpha_2$	–
TiAlV1	Initial	$\gamma + \alpha_2$	α_2
TiAlV1	TO_1	$\gamma + \alpha_2$	–
TiAlV1	TO_2	$\gamma + \alpha_2$	–
TiAlV2	Initial	$\beta + \alpha_2$	β
TiAlV2	TO_1	$\alpha_2 + \beta + \gamma$	–
TiAlV2	TO_2	$\gamma + \alpha_2 + \beta$	–
TiAlV3	Initial	$\gamma + \alpha_2 + \beta$	β
TiAlV3	TO_1	$\gamma + \beta$	–
TiAlV3	TO_2	$\gamma + \beta$	$\beta + \gamma$

Table 2
Erosion coefficients of the intermetallic alloys of Ti–Al–V system in various initial states after pulsed plasma fluxes irradiation

Number of pulses, N	Erosion coefficients S, μg/(J cm^2)										
	TiAl		TiAlV1			TiAlV2			TiAlV3		
	Initial	TO$_1$	Initial	TO$_1$	TO$_2$	Initial	TO$_1$	TO$_2$	Initial	TO$_1$	TO$_2$
5	1.2	1.5	0.4	0.6	1.6	0.4	0.4	0.8	0.3	0.3	0.3
10	1.9	0.3	2.3	0.4	0.2	1.1	0.7	0.7	1.6	0.3	0.1
15	2.0	–	0.7	–	–	2.5	–	–	1.2	–	–
⟨S⟩	1.7	0.9	1.1	0.5	0.9	1.3	0.55	0.75	1.0	0.3	0.2

the most erosion coefficient among the studied alloys. In increasing the number of irradiation pulses, the changes of the structural and phase state and topography taking place in the surface layers result in a noticeable increase of S (up to $N = 10$) for all investigated alloys. As the number of pulses N is enhanced, the averaged erosion coefficients (⟨S⟩) equalize for different alloys and after 15 pulses of irradiation they depend to a lesser extent on the chemical composition of the initial material. Within the limits of experimental error, the averaged erosion coefficients of the cast alloys for the Ti–Al–V system are practically the same and change in the 1.0–1.3 μg/(J cm^2) range; as to the alloy TiAl, ⟨S⟩ = 1.7 μg/(J cm^2).

The preliminary heat treatment of the alloys has a different effect upon the changes of their erosion coefficients. In particular, at the starting stage of irradiation the treatment according to TO$_1$ regime causes S to increase to some extent for TiAl and TiAlV1 alloys. The erosion coefficients of the alloys containing 2 and 9 at.% V undergo a further increase as a result of the TO$_2$ treatment. Under these conditions, S of the alloy with 15 at.% V does not depend, to within the experimental error, on the previous heat treatment.

The erosion coefficients for most of thermally treated samples decrease with increasing the number of pulses, and after 10 pulses of HTPPF action the averaged values of S ranged from 0.2 to 0.9 μg/(J cm^2), i.e. generally they are significantly less than those of the cast alloys. The last appears to be due to homogenization of these alloys as a result of their annealing and to their approximation to the more stable equilibrium state.

An analysis of the results obtained has shown that the alloy with 15 at.% V is more stable to HTPPF action than the others of the Ti–Al–V system. In this, the erosion coefficients of the preliminarily heat treated samples are in the 0.2–0.3 μg/(J cm^2) range and practically do not change from the number of pulses.

HTPPF action brings about changes of the phase state (Table 1) and a significant decrease of the number of registered spectrum lines. As shown by the results on flow turning and according to [10], the formation of single phase (α_2-phase) state in TiAl alloy results from the cooling with high rates. Consequently, fixation of α_2- as well as β-phase in the irradiated samples testifies that plasma action results in the quenching of the surface layers with a high cooling rate (~10^7 K/c).

It is necessary to note that the results of metallographic and qualitative X-ray phase analysis have not allowed to reveal a one-to-one correspondence between the erosion coefficient S and the structural and phase state of the investigated samples. However, it can be assumed that the observed significant decrease of the average values of S (at $N = 15$) for thermally treated alloys indicates an preferential influence of the processes of homogenization in these alloys on their erosion.

The studied intermetallic alloys have a high stability to preservation of their chemical composition and to selective evaporation of the elements from the surface layers (Table 3). In this, the irradiated intermetallic alloy TiAlV3 (TO$_2$) has a tendency to a slight enrichment of its surface with vanadium and to slight selective evaporation of aluminium.

Table 3
Results of the X-ray spectral microanalysis for TiAl and TiAlV3 alloys before and after HTPPF action

Alloy	State	Element composition, [a] at.%						
		Al	Ti	V	Fe	Cr	Ni	Si
TiAl	Initial	46.94	52.62	0.00	0.23	0.06	0.00	0.15
TiAl	Irradiated	46.00	50.36	0.32	1.79	0.64	0.13	0.75
TiAlV3	TO$_2$	46.89	38.21	13.93	0.53	0.00	0.00	0.43
TiAlV3	Irradiated	42.79	38.85	15.53	1.71	0.07	0.04	1.01

[a] The presence of V in the irradiated alloy TiAl and increasing the content of Fe, Cr, Ni and Si in irradiated alloys is due to redeposition of V evaporated from a nearly located TiAlV3 sample and to redeposition of Fe, Cr, Ni and Si evaporated from the sample holder made of steel 0.12%C/18%Cr/10%Ni/Ti, respectively.

Thus, the investigations performed have shown that the preliminarily heat treated intermetallic alloy TiAlV3 has a relatively low erosion coefficient, i.e. it has good characteristics from the point of view of possible penetration of impurities into plasma. However, as the SEM investigation has shown, the influence of HTPPF results in cracking of the surface of all studied alloys of the Ti–Al–V system, including TiAlV3, with the formation of cracks 0.08–0.96 mm in length. This is a significant disadvantage of these alloys for their using in FR and suggests that it is necessary to change the technology of their manufacturing.

The main experimental results of the investigation on the erosion of different carbon materials exposed to HTPPF action are illustrated in Fig. 1.

Fig. 1 shows that increasing the number of pulses *N* results in a sharp decrease of the erosion coefficients for the type GR reactor graphite from ~10 to 0.5 μg/(J cm^2). The results of SEM have shown that HTPPF action causes the surface relief to become smooth at the expense of healing fine pores and microcracks, and smoothing sharp edges with respect to big pores. In this, additional opening of the big pores and formation of cracks about 0.1 mm in length are found.

The silicicated graphites of PG-MS and SG-T types have a more stable erosion coefficient depending on the number of pulses in comparison with that of the reactor graphite, although a decrease of their *S* with an increase of *N* was also observed. In particular, the coefficient *S* of the type PG-MS graphite drops from 0.7 to 0.4 μg/(J cm^2) in increasing *N* from 5 to 30, and the average value of *S* for SG-T graphite at $N = 10$ is equal to 0.9 μg/(J cm^2). In this, the SEM investigation did not allow to observe any noticeable cracking of the sample surface.

It should be pointed out that after 30 pulses of irradiation the coefficient *S* of the reactor graphite practically corresponds to that of the type PG-MS graphite at similar conditions of irradiation.

To investigate the possibilities of decreasing the type GR reactor graphite erosion, it was impregnated with a metal melt of the following composition: 50wt%Ti/50wt%Cu. The results of XRSMA have shown that the surface chemical composition of the impregnated materials is extremely non-uniform both in initial, and in irradiated states. This is due to high porosity of the initial graphite material and, as a result of this, to preferential movement of the melt material along the pores and cracks of the carbon matrix. In this, the surface of the impregnated samples is covered with uniformly distributed metal precipitates (Fig. 2(a)).

An analysis of the element composition obtained from the integral XRSMA spectra from an area of about 1 mm^2 has shown (Table 4) that in the initial state the

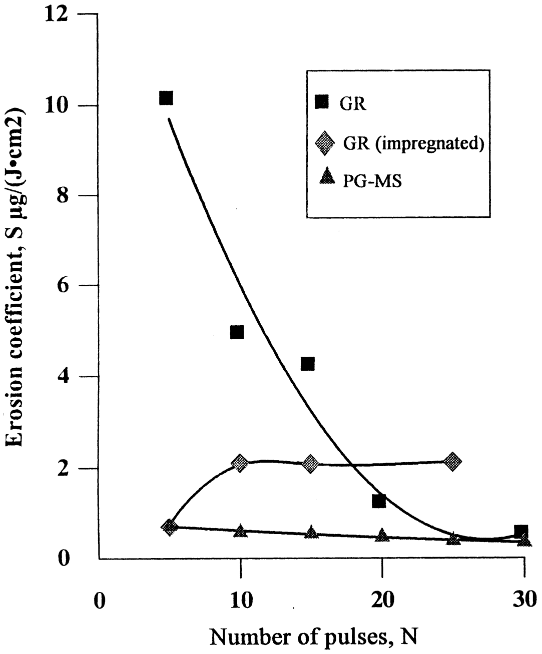

Fig. 1. Erosion coefficients of various carbon materials in relation to the number of HTPPF pulses.

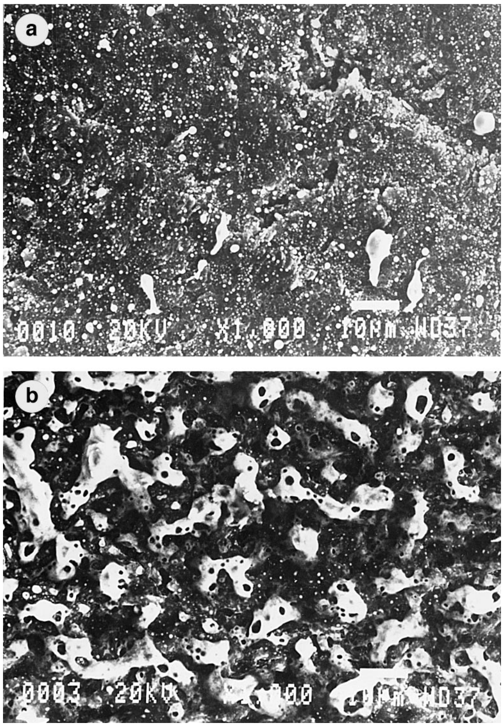

Fig. 2. Topography of impregnated reactor graphite GR before (a) and after (b) HTPPF action ($N = 10$).

Table 4
Element composition of GR type graphite samples impregnated with a 50wt%Ti/50wt%Cu melt, in initial state and after plasma irradiation at $N = 10$

State	Content of the main elements, wt%						
	Al	Ti	Si	Fe	Cr	Cu	Zn
Initial	0.21	37.88	3.34	3.91	0.04	54.62	0.00
Irradiated	0.87	23.34	23.69	26.41	0.84	21.02	1.72

impregnated graphite surface is enriched with copper in the ratio of wt%Cu/wt%Ti $\approx$ 1.4, suggesting that Cu is more movable during impregnation than Ti.

After HTPPF action the surface of the impregnated samples has clearly defined topography, with big pores filled with the melt (Fig. 2(b)), with marks of epicenters where an intensive melting takes place. Besides, extended cracks up to 0.12 mm in length, which resulted from the formation of thermal stresses, are observed on the surface of melt. In addition, the surface of some samples was covered with small cracks about 0.01 mm long both near the melt outflows, and outside them, as well as with isolated large initial pores (cracks) where the melt was fully evaporated.

The influence of HTPPF action on the impregnated graphite results in noticeable metallization of its surface at the expense of coming out the material of saturant onto its surface. This is most pronounced on the side surfaces of irradiated samples. According to the data of XRSMA (Table 4) for the irradiated samples exposed to preliminarily impregnation, the relative content of Ti and Cu on their surfaces decreases by a factor of 1.5–2.6, demonstrating that HTPPF action results in preferential erosion of saturant's elements.

As seen from Fig. 1, erosion of the impregnated reactor graphite increases with the number of pulses. This results, as mentioned above, from outflowing the melt onto the surface of the samples at the starting stages of irradiation and its evaporation. On further HTPPF action, there is a tendency of stabilizing the erosion coefficient ($S \approx 2.0$ µg/(J cm^2)).

To compare the erosion extent for developed new kinds of materials with that of traditionally considered candidate materials for the ITER, cold-rolled and sintered-annealed tungsten was studied. These investigations have shown that the cold-rolled tungsten is exposed to significant erosion ($S \approx 0.8$ µg/(J cm^2)). In this, the surface of samples cracked after 5 pulses of irradiation, and full stratification of the surface layers was observed after 10–15 pulses. As to the sintered tungsten, it had high resistance to erosion under the same conditions of irradiation, its averaged value of S after 30 pulses being equal to about 0.2 µg/(J cm^2). Its surface did not undergo a great deformation: cracking, stratification, formation of pits.

4. Conclusion

Thus, an analysis of the experimental results has shown that the erosion coefficients of the intermetallic alloys of system Ti–Al–V are determined by their initial composition and preliminary treatment. In this, the structural and phase transformations occuring in the alloys at the starting stage of HTPPF action change the extent of erosion on subsequent irradiation. In the limits of experimental error, this results in practically equal erosion coefficients of the cast alloys. The alloy TiAlV3 in thermally treated state has been found to have high stability to erosion depending on the number of pulses of HTPPF action. Among the investigated materials, its erosion coefficient is minimum and equals to 0.2–0.3 µg/(J cm^2). However, all the alloys studied are subjected to surface cracking.

It has been shown that the erosion coefficients of the considered graphite materials irradiated by HTPPF with a specific energy of about 100 J/cm^2 change in the 0.4–10 µg/(J cm^2) range, depending on the kind of materials and the number of pulses. In this case, the reactor graphite erosion decreases more than by an order of magnitude with an increase of the number of pulses, whereas the silicicated graphites are more stable and their erosion coefficients are small.

This work is the first to show that impregnation of the reactor graphite with a metal melt of the following composition 50wt%Ti/50wt%Cu significantly decreases erosion at the starting stages of irradiation. However, further HTPPF action at $N > 15$ causes this melt to intensively outflow onto the surface of a sample and increases erosion up to values exceeding that of the rector graphite under similar conditions of irradiation. In this case, the erosion of the impregnated graphite at the steady stage is due to primary evaporation of its metal components.

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