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Enhanced resistance of plasma-sprayed TiC coatings to thermal shocks

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In previous tests the maximum observed TiC coating thickness which showed good resistance to 0.7 s thermal shocks was 400 μm. For this thickness, only microcracks perpendicular to the surface (segmentation) were observed, whereas for thicker ones, spalling inside the coatings occurred. With a combination of substrate surface modification (macroroughening or spraying a bondcoat) and preheating to 375°C before the thermal shocks, it has been possible to completely avoid the delamination during heating and to promote the resistance to delamination on cooling. This allows a doubling of the thickness of thermal shock resistant coatings up to 1mm. Hemispherical coated limiters were tested in TdeV (Tokamak de Varennes) with plasma currents of 210 kA and have absorbed 20 kW during 1.2 s associated with a power deposition factor of about 10 MW m^{-2} $s^{0.5}$.

1. Introduction

Design studies for ITER plasma facing components are summarized in a report by McGrath et al. [1]. The divertor must be able to withstand heat fluxes in the range of 10 MW m^{-2} and must not seriously contaminate the plasma with erosion products. Materials of different thermal expansions must be joined and must perform under high temperatures and high thermal stresses. They should be easily repairable. In this framework, plasma-sprayed TiC coatings have been developed, characterized and tested [2] for relatively long heat pulses (1 s). Coatings in the range of 200–500 μm have been evaluated on a test limiter in TdeV, and the results have been reported at the SOFT 90 conference [3]. Another group [4,5] has used the same spraying parameters to make TiC coatings in order to test simulated disruptions (using 10 ms laser pulses). Both studies were performed with similar $(P/A)t^{0.5}$ factors [6], where P/A is the power deposited per unit surface and t is the time, leading to similar surface temperatures and stresses. Due to a higher stress gradient in the disruption simulations, the stresses induced at the interface are lower for relatively thick coatings. Long and short pulse studies are hence complementary.

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The aim of the work since the SOFT 90 conference was to test different approaches to increase the thickness of shock resistant TiC coatings and better understand their failure mechanisms. A study [7] has been made of the influence of different surface preparations of the metallic substrate (grit-blasting, macroroughening [8] or spraying a bondcoat [9]) combined with modified thermal shock parameters (shock duration and preheating of the material). These tests were performed on coupons with an electron beam gun. With a combination of surface modification and preheating, it has been possible to double the thickness of thermal shock resistant coatings to 900 μm. In the case of the macroroughened substrates, a detailed study [10] has also been made. The enhanced resistance of coatings applied on macroroughened substrates is likely due to the lamella foldings and the particular thermal stress distribution.

In this paper we report on the evaluation, with the test limiter facility of TdeV, of coatings made according to the different surface preparations pretested with the coupons.

2. Experimental procedure

2.1. Coating preparation

Titanium carbide and the metallic bondcoat were deposited on Inconel 625. The substrates were hemi-

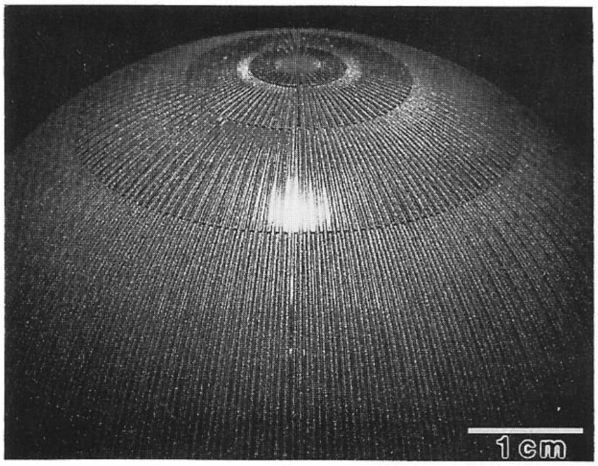

Fig. 1. Hemispherical Inconel limiter before being coated with TiC. The macroroughening was done by electric discharge machining.

spherical limiters (diameter 70 mm). The coatings (thickness: 900 μm–1 mm) were plasma-sprayed according to the conclusions of a previous study on coating fabrication [11]. A Cerac 99.5% pure TiC powder was sieved to +20–35 μm, and sprayed in an inert gas enclosure. The porosity was estimated at 15%. The C/Ti ratio was about 0.71 after spraying, in comparison with 0.96 for the as-received powders [3]. No further variations were detected after numerous thermal shocks with surface temperatures up to 2500°C. With this C/Ti ratio the melting temperature remains around 3000°C [12]. NiCrAlY bondcoats (Amperit 413.0) were sprayed in air.

2.2. Surface preparation

As stated in section 1 three techniques were compared: grit blasting, macroroughening and use of a metallic bondcoat. The macroroughening shown on fig. 1 was made by electric discharge machining (EDM). The surface is covered by a series of grooves about 400 μm wide, 200 μm deep and 100–300 μm apart from each other. The thicknesses of the coatings are 1 mm except for the coating deposited on a grit blasted surface which is 900 μm thick.

2.3. Test limiter

TdeV operates with ohmic heating, has a major radius of 0.866 m, a toroidal magnetic field of 1.5 T and a typical minor radius of 0.27 m as defined by a double null divertor. For the shots in question, the discharges were 1.2 s long, with a plasma current of 210 kA and a line average electron density of 2.50×10^{19} m^{-3}. The three hemispherical limiter heads were successively tested inside TdeV via the test limiter facility which allows these limiters to be inserted vertically via an airlock. Their position can be changed between shots. They were preheated to about 375°C. Their heat losses were sufficient to cool to this temperature before the next shot. They are equipped with three thermocouples – the center one being 15 mm behind the surface, while the other two are situated 17 mm horizontally on the electron and ion sides, 5 mm from the surface. The test limiter can be observed with filtered video cameras allowing us to detect defects induced during the discharges. The power deposited on the heads is a function of the relative position of the test limiter with respect to the plasma edge.

The power density is evaluated by two methods. The first one uses the thermocouples and is described below; the other is based on simulation and is described elsewhere [13]. An example of simulation is given in section 3.4. It is important to note that the power deposition on the ion side is different than that on the electron side. When the head is entirely outside the last closed flux surface (LCFS), there is more power deposited on the ion side. As the head is gradually put inside the plasma the power deposition on the electron side increases more rapidly and eventually more power is deposited on this side. Values of the maximum temperatures attained by both surface thermocouples as a function of the position of the limiter are used to calculate the power e-folding lengths. These values, along with the temperature rise, are used to calculate the maximum surface power densities (taking into account the spherical shape of the limiter).

3. Experimental results and discussion

3.1. Onset of delamination

As discussed previously [3] two types of damage are observed: segmentation and delamination. Segmentation (network of cracks perpendicular to the interface) is likely to occur during cooling. It is beneficial since it gives a certain elasticity to the coating. Delamination, which is always detrimental, is of two kinds: pulloff delamination and edge delamination. Pulloff delamination occurs suddenly during the heating phase leading to a complete exfoliation of the heated region. Electron beam tests on coupons have shown that it can be avoided by preheating [7]. As reported below this is confirmed with the limiter. The edge delamination occurs during cooling and progresses gradually shock after shock from the side to the center of the coupons. In the case of a hemispherical limiter where the edges are not affected by the heat flux, a similar edge delamination may be initiated at the segmentation cracks under repeated high power loads.

Segmentation was observed after each tokamak shot with a portable telescope. The delamination is ob-

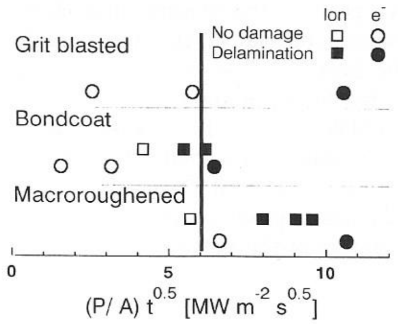

Fig. 2. Diagram showing the onset of delamination as a function of $(P/A)t^{0.5}$. The vertical line is the onset of damage established by the electron beam tests.

served on the video pictures recorded during the shots. Upon cooling ($t > 1.2$ s), the delaminated regions are visible since they cool down much more slowly than the intact regions. Video pictures taken during cooling were used to evaluate the power at the onset of delamination.

3.2. Effect of the preheating

Tests with the electron beam on coupons [7] have shown that preheating the substrate before the thermal shock always leads to a pronounced improvement in the coating resistance to sudden (pulloff) delamination. Indeed, for a coating thickness larger than 500 μm, preheating is necessary for shocks of 10 MW m^{-2} $s^{0.5}$. In the present work, the hemispherical limiter coated with 900 μm on a grit blasted substrate and preheated to 375°C before testing, resists a value of about 6–10 MW m^{-2} $s^{0.5}$ on the electron side (it was not possible to evaluate the power on the ion side). It showed no evidence of pulloff delamination. This result is in accord with the electron beam test.

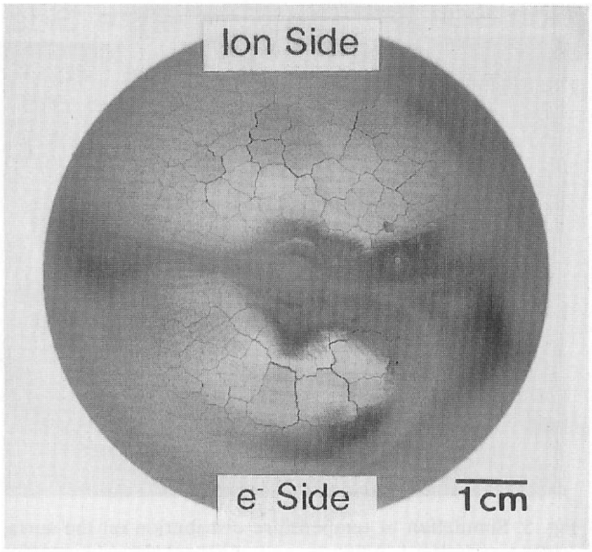

Fig. 3. Photography of the limiter coated with TiC on macroroughened Inconel (see fig. 1). The regions heated by the plasma are segmented.

3.3. Effect of the surface preparation

Two heads which had special surface preparations were coated with 1 mm of TiC. The one which has a bondcoat resisted 5 MW m^{-2} $s^{0.5}$ on both sides. These results are plotted on fig. 2 which summarizes the different behavior as a function of power. The vertical line is the onset of damage as established with the electron beam tests [7]. The bondcoat used was severely cracked. A more ductile bondcoat could have given better results.

The head having the grooves (fig. 3) showed a slight delamination at 7.9 MW m^{-2} $s^{0.5}$ and a more evident one at 9.0 MW m^{-2} $s^{0.5}$ (10.3 on the electron side). These values are higher than those obtained on the

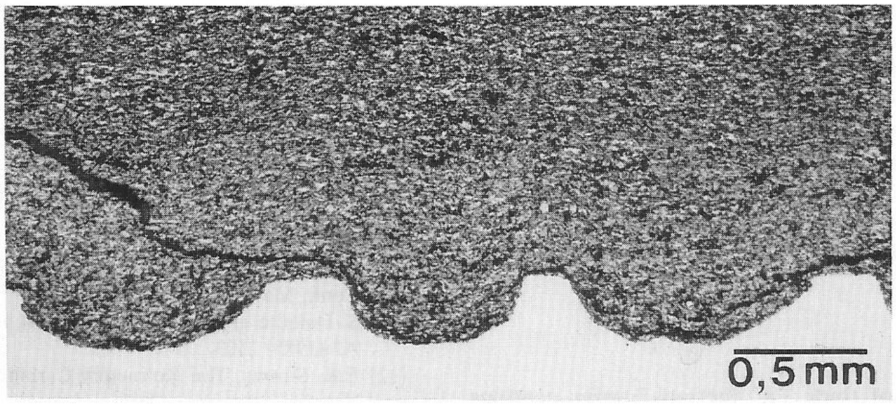

Fig. 4. Cross-section optical micrography of the limiter shown on fig. 3. The section was taken on the ion side. Some perpendicular cracks are trapped in the grooves.

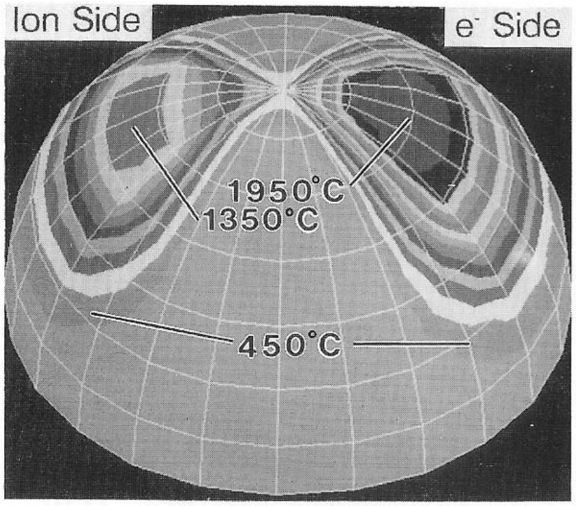

Fig. 5. Simulation of temperature distribution on the surface of a test limiter head at the end of a TdeV shot (# 12558).

bondcoated head, but cannot be directly compared to those of the grit blasted one due to the thermocouple failure on the ion side. However the extent of delamination progressed much more slowly in the macroroughened head even after many more shocks. Fig. 4 illustrates the effect of the folding of the TiC lamellae on the crack propagation. Cracks initiated at the coating surface propagate towards the interface during thermal cycling. The possible crack propagation in a direction parallel to the coating-substrate interface has been avoided by the presence of the relief. This behavior is in accordance with the folding of the lamination of the coating and with the calculated stresses [10]. Thus, perpendicular cracks may be trapped in the grooves, increasing the resistance to delamination.

3.4. Simulation of temperature distribution

Figs. 5 gives an example of the surface temperature distribution at 1.2 s; the shape of the heated zone is similar to the corresponding video map and the pattern of surface damage (fig. 3). Power values are adjusted in order to reproduce the maximum temperature rise of the thermocouples after ~ 6 s. These power values are in agreement with those calculated by the analytical method (section 2.3). It is important to emphasize that to obtain reasonable agreement, we must take into account the reduced thermal conductivity ($\approx 1/8$) [7] of the plasma-sprayed TiC compared to bulk value.

4. Conclusion

The failure of thick TiC thermal barrier coatings exposed to high heat fluxes during 1.2 s discharges in TdeV was examined for three surface preparation techniques prior to the plasma spray deposition. Preheating before thermal shock eliminated delamination during the heating phase of the shock. With a bondcoat, a 1 mm thick coating can resist delamination up to about 5 MW m^{-2} $s^{0.5}$. In the case of a 1 mm coating sprayed on a macroroughened substrate, the resistance was increased to 9 MW m^{-2} $s^{0.5}$. Moreover, the macroroughening has a positive effect on the resistance to the propagation of the delamination.

Acknowledgements

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THERMAL SHOCK EVALUATION OF PLASMA - SPRAYED TiC THICK COATINGS BY MEANS OF THE TEST LIMITER FACILITY OF THE TOKAMAK DE VARENNES*

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The Tokamak de Varennes has an auxiliary limiter facility which allows various materials to be exposed to plasma discharges. Thick, plasma-sprayed TiC coatings (200-500 μm) have been evaluated. The factor which limits the thickness is presently the thermal shock resistance. Samples whose stoichiometry and micro-structure were analysed have been pre-tested using a 15 kW electron beam system. The results were used to optimize the deposition of coatings on hemispherical heads which were tested in the tokamak. With plasma currents of 200 kA, the heads have absorbed 30 kW during 0.7 sec leading to a power deposition of about 15 MW/m^2. The coatings tested so far indicated an optimum thickness of about 400 μm which is still too thin for long-term operation. For this thickness, only microcracks (segmentation of the surface) are observed. For thinner coatings, the Inconel substrate was melted, whereas for thicker ones spalling inside the coatings was observed. No contamination of the core plasma by Ti has been observed, although the production of Ti from the surface has been observed, and Ti has been detected on collector probes placed on the vessel wall. AES evaluation of the exposed surface of a TiC flake has shown no significant difference in Ti and C content when compared to the reverse (unexposed) side.

1. INTRODUCTION

During the last several years, carbon materials have been used for plasma facing surfaces in the large Tokamaks. Unfortunately, carbon is susceptible to chemical sputtering which affects plasma confinement. Titanium carbide is of interest because of its low physical and chemical sputtering yield (1). Thin TiC has been used in JT-60 and Asdex (2) and discarded mainly because of its very small thickness (5 μm) which is eroded by sputtering and cannot sufficiently protect the metal substrate. JET and TEXTOR have coated their first wall with beryllium (3) and boron respectively. However, these coatings are thin and are not compatible with long-pulse operation. The present trend is to look for new materials such as bulk boronized graphite (4).

On the other hand, "thick" coatings as applied by plasma spraying can also be used for specific applications such as the protection of metallic components. Such spraying can even be envisaged for in-situ remote repairs (3). With plasma spraying it is possible to deposit coatings of TiC several mm thick. The thickness is presently limited by the thermal shock resistance. These coatings are brittle and tend to develop high stresses at thermal gradients. We report in this paper the evaluation of coatings of three different thicknesses which have been tested in the Tokamak de Varennes (TdeV) with a test limiter facility. Their behavior is compared to that of samples tested with an electron beam.

2. EXPERIMENTAL PROCEDURE

2.1. TiC spraying and characterization

The coatings were sprayed according to the conclusions of a previous study on coating fabrication criteria (5). A commercial Cerac TiC powder 99.5% pure was sieved (20 - 35 μm). Plasma spraying was carried out into an inert gas enclosure on Inconel samples and limiters. The porosity was 17%. The C/Ti ratio was about 0.71. It was measured by Auger Electron Spectroscopy (AES) and X-ray diffraction. In this last case, the lattice parameter

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(4.3262 ± .0004 Å) was correlated to the C/Ti ratio with a well established relation by Storms (6). This small loss of C increases the melting point to its maximum value of 3067°C (6).

2.2. Electron beam thermal shocks

Thermal shock pretesting was performed with a modified electron beam evaporation device as described previously (7). The surface was monitored with a black and white video camera. The surface temperature was measured with a two-colour pyrometer. The experiment was computer driven and the substrate was allowed to cool to 300°C before each pulse. The thermal shock series was stopped when damage was observed or when the surface temperature reached a temperature value very different from the previous one (caused for example by delamination).

2.3. Test limiter facility

The TdeV operates with ohmic heating, has a major radius of 0.866 m, a toroidal magnetic field of 1.42 Tesla and a typical minor radius of 0.24 m as defined by one of four poloidal graphite limiters. For the shots in question, the discharges were 0.7 sec long, with a plasma current of 200 kA and a line-average electron density of 3.0E19 m^{-3}. Three hemispherical limiters (diameter = 70 mm) have been coated with 250, 330 and 500 μm of TiC according to the results of the electron beam thermal shock measurements described above. They were successively tested inside the TdeV via the test limiter facility which allows these limiters to be inserted vertically via an airlock. Their vertical position can be changed between shots. They are equipped with three thermocouples - the center one being 15 mm behind the surface, while the other two are situated 17 mm horizontally on the electron and ion sides, 2 mm from the surface. The test limiter can be observed with video cameras and the emission from sputtered atomic Ti can be measured along six different chords in front of the test limiter head using detectors equipped with interference filters. The power deposited on the heads is a function of the relative position of the test limiter with respect to the plasma and the main graphite limiters which are also adjustable. This power density is evaluated by two methods. The first one uses the bulk temperature elevation and an estimate of the size of the area imprinted on the TiC. The second is described in section 3.4 along with simulation calculations.

3. EXPERIMENTAL RESULTS AND DISCUSSION

3.1 Thermal shock resistance

The different damages observed on the limiter heads are illustrated in Fig. 1 together with the cross-section micrographs of the corresponding coatings. When the coating is thin (250 μm) there is insufficient protection of the Inconel which melts. One can also observe a segmentation of the TiC surface. On the other hand, when the coating is thick (500 μm) there is again a segmentation of the surface but the coating fails by delamination. Although the TiC adheres well to the Inconel, there are fatal cracks parallel to the substrate surface within the coating near the interface. An intermediate thickness (330 μm) protects the Inconel and does not delaminate. Fig. 2 shows the aspect of the segmented surface which seems to ensure a beneficial elasticity. Similar results have been obtained with the electron beam tests (5).

Fig. 3 summarizes the results of thermal shock evaluation for both the electron beam tests and tokamak tests. For a comprehensive representation of the results, the heat flux parameter F

$$F = P/A \cdot t^{0.5}$$

where P/A: power density in kW/cm^2

t: duration of the heat flux in s

was chosen, as used elsewhere (8). This parameter allows one to combine the factors of power density and duration of the heat load into one parameter which is in accord with the process of one-dimensional surface heating. The corresponding damage is indicated together with the number of discharges. This gives a tentative diagram of five regions. Obviously at sufficiently low power there is no damage. For a given thickness if the power is too large the Inconel will melt. However for very thick coatings the delamination will prevent the TiC from being cooled by conduction and it will melt. The conditions for proper behavior correspond to the segmentation region.

The actual optimum thickness (400 μm) is not sufficient to cope with sputtering after long-term operation. In order to increase it, a substrate topography which would avoid delamination for thicker coatings is presently under investigation. Encouraging results have been obtained.

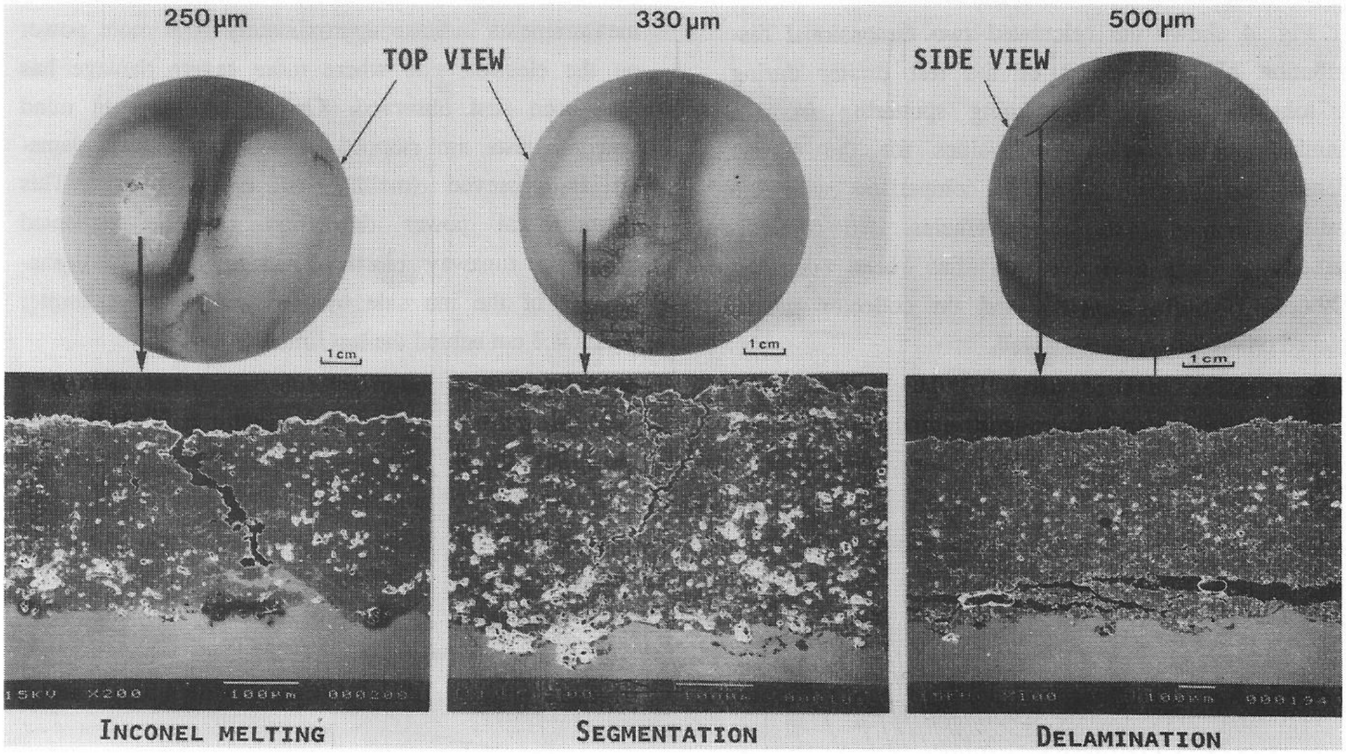

FIGURE 1
Photographs of the Tokamak test limiter heads after contact with the plasma and cross-section SEM micrographs of the TiC coatings on Inconel. The left side of the heads face the electron side of the plasma. Similar microstructures are observed on the ion side although the defects (melting and delamination) are less pronounced.

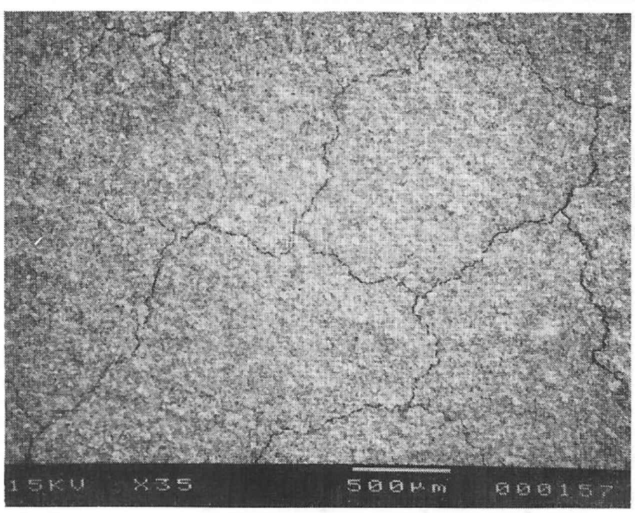

FIGURE 2
SEM micrograph of the segmented surface of the limiter head coated with 330 μm of TiC.

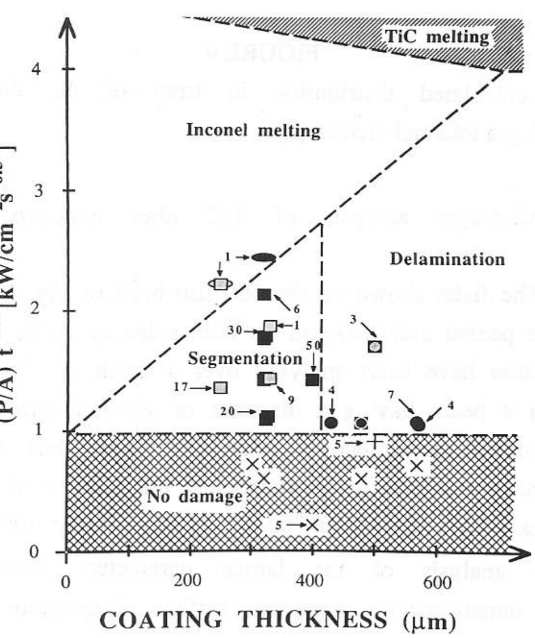

FIGURE 3
Experimental observations: ×(+) no damage, ■(▣) segmentation, ●(◉) delamination, ⬬(◎) Inconel melting, respectively for electron gun and Tokamak tests. The number of thermal shocks is given when different from ten.

3.2 Ti spectroscopy near the test limiter

Fig. 4 shows the calculated two dimensional distribution of Ti in front of the test limiter during a tokamak discharge, assuming sputtering from a hemispherical surface. The values for the atomic density are obtained from a comparison with the calibrated spectroscopic measurements. No contamination of the core plasma has been detected although Ti has been detected on collector probes placed on the vacuum vessel wall.

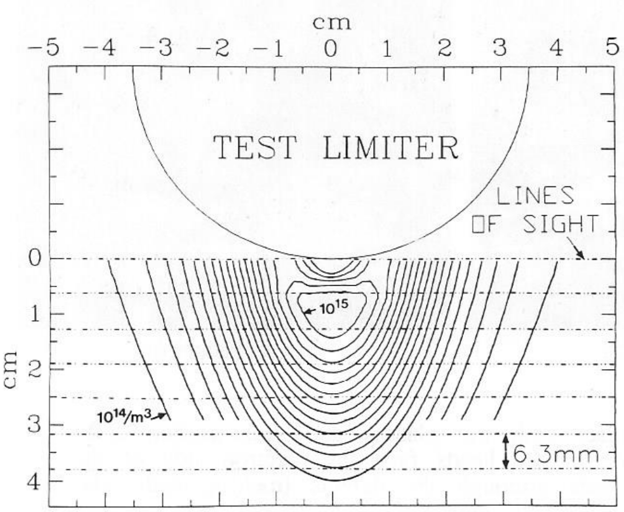

FIGURE 4

Ti calculated distribution in front of the limiter during a tokamak discharge.

3.3 Auger analysis of TiC after exposure to plasma

The flake shown on the 500 μm head of Fig. 1 has been peeled and analysed on both sides by AES. The surfaces have been analysed over a depth of 100 nm with a beam having a diameter of about 1 mm. No significant difference of the C/Ti ratio has been detected between the exposed and the unexposed surfaces. This has been confirmed by X-ray diffraction analysis of the lattice parameter. This is not unreasonnable since the surface temperature has been measured on the electron beam test experiment to reach only 1800°C for this power level. This temperature is very low when compared to those reached in the plasma torch (~ 3500°C) where dissociation is produced.

3.4 Temperature distribution

Fig. 5 shows the typical measured temperatures inside a head during and after a discharge. These measurements indicate approximately 10% more power on the electron side where more severe damage has also been first observed. One should bear in mind that the tests are stopped as soon as a fatal damage is observed (melting or delamination). This asymmetrical power deposition can be attributed either to runaway electrons or to the partial shadowing of the ion side by the main graphite limiter which is 3 cm behind the test limiter.

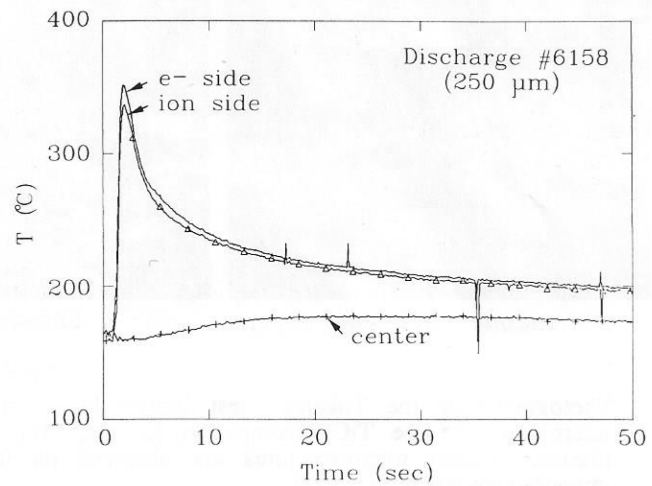

FIGURE 5

Temperature evolution inside the head during and after a plasma discharge.

The temperature rise of the head as a function of position from the plasma edge shows that the power deposition varies exponentially in the scrape-off layer. Using this, along with the bulk temperature rise, we calculate the power density on the limiter surface. More sophisticated calculation of the temperature distribution at the surface and inside the head has been developed using a 3-D finite element program. Fig. 6 gives an example of the surface temperature distribution which resembles very closely the power deposition profiles calculated analytically. In addition, the temporal variations of the temperature distribution reproduces very well the measurements shown in Fig. 5. A further use of the code will be to calculate internal stresses and help to develop thick, resistant coatings.

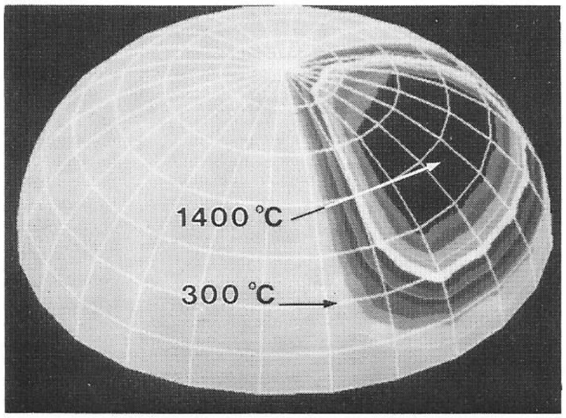

FIGURE 6
Simulation of temperature distribution on the surface of a test limiter head (1 sec after the onset of discharge, duration of discharge 0.8 sec and power 1.5 kW/cm²).

4. CONCLUSION

TdeV has a test limiter facility which can be used to evaluate the behaviour of materials in contact with the plasma and to assess their influence on the plasma.

Plasma-sprayed TiC coatings having a thickness of about 400 μm have been found to resist thermal loads of 2 kW/cm².

The sputtered Ti atoms are confined to the exterior plasma. The C/Ti ratio of the exposed surface is not significantly modified after 8 discharges.

A program has been developed to simulate the temperature evolution in the test heads.

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