ELSEVIER

Journal of Nuclear Materials 283–287 (2000) 1077–1080

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

The behavior of coatings and SiC_f/SiC composites under thermal shock

Jinnan Yu [a,*], Zhongwen Yao [a], Gang Yu [a], Fengmin Chu [a], Xiuzhang Tang [a], Yi Zeng [a], Tetsuji Noda [b]

[a] *China Institute of Atomic Energy, P.O. Box 275-51, Beijing 102413, People's Republic of China*
[b] *National Research Institute for Metals, Tsukuba Laboratories, 1-2-1 Sengen, Tsukuba-shi, Ibaraki 305, Japan*

Abstract

The behavior of various coatings and SiC_f/SiC composites under thermal shock is described in this paper. The erosion of the SiC_f/SiC composites and TiC + TiN coatings under thermal shock is less than that of WC, TiN, TiC and B_4C coatings. The surface of multi-arc ion coating (MAIC) TiC, TiN and TiC + TiN coatings under thermal shock has cracks due to the interface between coating and substrate. The ion beam enhanced deposition (IBED) TiC coatings and plasma spraying coating (PSC) B_4C and WC coatings under thermal shock do not have cracks. It seems that these coatings approximate to three-dimensional heat transfer. The surface of 2D Hi-Nicalon SiC_f/SiC composites under thermal shock indicates sublimation and trace melting. The melting region is only presented in the matrix, and the fiber retained its integrity. This means that the matrix has poor thermal diffusivity, especially for interface damage between fiber and matrix.

1. Introduction

In order to prevent the first wall and plasma-facing component (PFC) from sputtering and erosion, various coatings are investigated as a protective layer, such as TiC, TiN, TiC + TiN, B_4C and WC. These coatings have good resistance to sputtering by the escaping plasma ions. But the erosion induced by the disruption of the plasma is important to be explored for these coatings. The disruption of the plasma will lead to plasma energy discharges at small part to form thermal shock. There are three methods to simulate the plasma disruption [1], which are plasma guns, lasers, and electron beams [2–4]. This paper describes the results of the thermal shock experiments with the laser method on various coatings on 316L stainless steel (316L) and two kinds of SiC_f/SiC composites.

The protective layer under thermal shock suffers thermal stresses that arise from the different thermal expansion coefficients between the substrate and the coating layer [5]. Therefore, it is easier to examine the effects of thermal stresses with the coating on 316L because of the high thermal expansion coefficient of 316L. It shows that the thermal stresses are harmful to the thin layer of TiC, TiN, TiC + TiN produced with ion coating, but harmless to the B_4C and WC thick layer produced with the plasma spray method, and to the TiC thin layer produced with ion beam enhanced deposition (IBED).

SiC_f/SiC composite is considered as one of the candidates for first wall and plasma-facing materials of fusion reactor because of its high temperature strength, low induced radioactivity, non-catastrophic failure mode and good irradiation resistance. The behavior of erosion under thermal shock was investigated with two kinds of SiC_f/SiC composite specimens. One is a two-dimensional weave (2D) SiC_f/SiC composite specimen. The other specimen is a one-dimensional weave (1D) SiC_f/SiC composite specimen which had interface damage between the fiber and matrix produced by the cyclic electron irradiation [6]. The experiment results show different behaviors for these specimens under thermal shock.

* Corresponding author. Tel.: +86-10 6935 7232; fax: +86-10 6935 7008.

E-mail addresses: yujn@iris.ciae.ac.cn, yujn@sun.ihep.ac.cn (J. Yu).

0022-3115/00/$ - see front matter
PII: S 0 0 2 2 - 3 1 1 5 (0 0) 0 0 2 8 4 - 1

2. Experimental

There are six kinds of coating on the 316L stainless steel (316LSS). The size of the 316L substrate was ∅19 mm × 1 mm. The surface of the substrate was degreased using methyl alcohol and milled smooth, as well as polishing with Al_2O_3 polishing paste. Then, the substrate was cleaned and dried prior to coating. Three kinds of coating, including TiC, TiN and TiC + TiN, were coated on the clean surface of 316LSS substrate with the multi-arc ion coating (MAIC) method. The thickness of the coating by this method is about 2 μm. The coating of TiC + TiN was a multi-layer coating made by controlling the inlet of methane and nitrogen. The inner sub-layer is TiN with more nitrogen inlet, with thickness of about 0.1 μm. The second sub-layer is Ti(CN) with suitable inlet ratio of CH_4 to nitrogen, with thickness of about 1.8 μm. The outside sub-layer is TiC with inlet of methane, with thickness of about 0.1 μm.

The other kind of TiC coating on the clean surface of 316L substrate was about 700 nm in thickness made by the IBED method. The fifth and sixth kinds of coating were B_4C and WC, coated on the 316 stainless steel using the plasma spraying method (PSC) with particle sizes 10–45 μm in the argon protection atmosphere. In the PSC method, the surface of the substrate was degreased using methyl alcohol and Al_2O_3 grit blasted to improve the adhesion between the 316SS and the coating. The thickness of the B_4C and WC coatings was 0.25 and 1 mm, respectively.

The SiC_f/SiC specimens include the 1D and 2D Hi-Nicalon SiC_f/SiC composites prepared by chemical vapor infiltration (CVI) using thermal decomposition of trichloro-ethyl-silane (ETS). The specimen size of the SiC_f/SiC composites was ∅10 mm × 1.8 mm, and were mounted in a 316SS ring with internal diameter ∅10 mm and outside diameter ∅19 mm. The specimen of 1D Hi-Nicalon SiC_f/SiC composite had been irradiated by the cyclic electron irradiation, and was mounted on the irradiation target chamber with cooling systems in vacuum. The specimen was initially at 18°C. Then the temperature was increased to 650 ± 40°C by 14 MeV electron beam heating and maintained for about 11.4 min, which was called one irradiation cycle. The rate of increase in the irradiation temperature was about 750°C/min. The total number of cycles was 31 and the damage doses reached 0.17 dpa. The thermal diffusivity of the irradiated specimen decreased to 0.018 cm^2/s from an unirradiated value of 0.023 cm^2/s at 293 K, which indicated interface damage between the fiber and matrix by the cyclic electron irradiation [6].

The thermal shock experiments were done using a Laser Device Lextra 200, Lambda Physik Company. Experimental parameters are as follows: HV const at 21 kV, KrF wave length 248 nm, pulse width 28 ns, frequency 20 Hz. The laser beam was focused by a convex lens and the spot was uniform with an area of 0.84 × 0.4 mm. The injection energy on the specimens was about 245 mJ per pulse. In order to compare the behavior of the various coatings and SiC_f/SiC composites under thermal shock, a pulse switch was designed to keep three pulses passing through the gate for each laser irradiation. The injection energy on the specimen was about 0.72 J per each laser irradiation in 0.1 s. The heat load on the specimen was 2.14 MJ/m^2 for each thermal shock.

The specimens were kept dry and the mass of the specimens was measured before and after laser irradiation to detect mass loss. The surface of the specimens was observed by scanning electron microscope (SEM) to investigate the behavior of the coating and SiC_f/SiC composites under thermal shock.

3. Results and discussion

3.1. Erosion

The loss of mass in each specimen was measured after laser irradiation. The mass losses of the SiC_f/SiC composites and the TiC + TiN coating under thermal shock are 2.5, 3.4 g/m^2 pulse, respectively, which are the lowest; the mass losses of the WC and TiN coatings under thermal shock are 16.5, 42.6 g/m^2 pulse, respectively, which are the second lowest. The values of the mass loss for the TiC and B_4C under thermal shock are 66.1, 90.9 g/m^2 pulse, respectively. As B_4C is a low Z material, this mass loss value of B_4C coating under thermal shock is not serious to influence the plasma operation. These coatings are all able to be coated on carbon fiber-reinforced carbon composites (CFC) as a protective layer against erosion. As the enthalpy of SiC sublimation is high, it needs much more energy to sublimate SiC, which brings the thermal energy out to induce quick decreasing of the surface temperature. Therefore, the erosion of SiC_f/SiC composites under thermal shock is the lowest.

3.2. SEM observations in TiC, TiN, and TiC + TiN coatings

The six kinds of coatings under thermal shock are all melted at the center part of the laser spot, and are shown in Figs. 1–4. The fraction of the melting region is about 0.0262, 0.0250, 0.0210, 0.1801, 0.0570, 0.0350 and 0.0137 of laser spot for TiC, TiN, TiCN, TiC(IBED), B_4C, WC coatings and SiC_f/SiC composites, respectively. As the melting region absorbs much more thermal energy than the surrounding coating and the substrate, the evaporative atoms remove much more thermal energy out of the melting region. Radiating heat loss, heat also plays an important role in removing the thermal energy. The

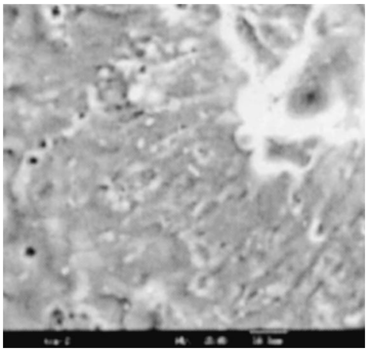

Fig. 1. The surface topography of TiC coating at the left part of laser spot center, after three pulses laser irradiation in 0.1 s, injection energy 2.14 MJ/m^2 (magnification 1000×).

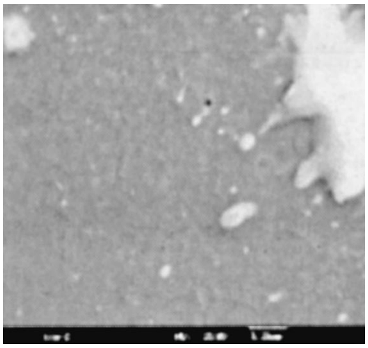

Fig. 2. The surface topography of TiN coating at the left part of laser spot center, after three pulses laser irradiation in 0.1 s, injection energy 2.14 MJ/m^2 (magnification 2000×).

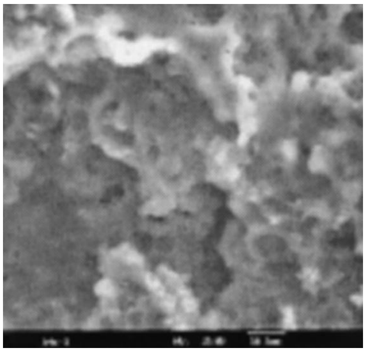

Fig. 3. The surface topography of B_4C coating at the center part of laser spot, after three pulses laser irradiation in 0.1 s, injection energy 2.14 MJ/m^2 (magnification 1000×).

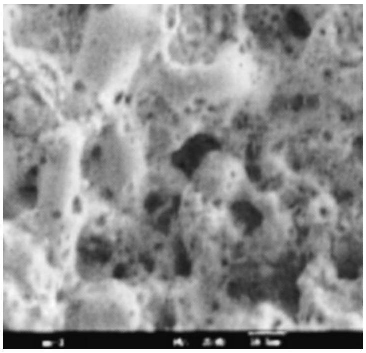

Fig. 4. The surface topography of WC coating at the center part of laser spot, after three pulses laser irradiation in 0.1 s, injection energy 2.14 MJ/m^2 (magnification 1000×).

surface temperature decreases very rapidly and the melting region is quickly quenched and solidified.

The TiC and TiN coatings in the beam spot region surrounding the melting area have a moiré-cracking pattern, which is shown in Figs. 1 and 2. The width of the cracks in the TiC coating is little larger than that in the TiN coating. This is because TiC and TiN coatings made with the MAIC method have an interface between the coating and the substrate. There are three thermal cycles in each laser irradiation. The temperature cycle of the coating on the beam spot is different from that part of the substrate. The coating in the beam spot area suffers thermal stresses that arise from the different temperature cycles and the different thermal expansion coefficients between the coated layer and the substrate. The stress is large enough to induce the cracks. There are several cracks beside the melting area for the TiC + TiN coating and no cracks in other regions. This is because the TiC + TiN coating is a multi-layer construction. The stresses can be relaxed at the surfaces between the TiN, Ti(CN) and TiC. But the stresses cannot be relaxed between the melting region and the multi-layer and the cracks are formed to relax the stresses.

The TiC coating with the IBED method has no boundary between the TiC layer and the substrate. As the IBED method produces atomic mixing at the surface between the TiC layer and substrate, the thermal stresses are distributed over the whole region. Thus, the TiC coating made by the IBED has no cracking beside the melting region under thermal shock.

The surface of the B_4C and WC coatings under thermal shock has some trace of melting, as shown in Figs. 3 and 4. But there is no cracking. This is because the coating is thick enough not to induce large thermal stresses between the coating and the substrate.

It seems that the coating made with the MAIC method under thermal shock approximates to two-dimensional heat transfer because of the interface between the coating and the substrate. The coating showed cracks under thermal shock. But the coatings made with the IBED and PSC methods under thermal shock ap-

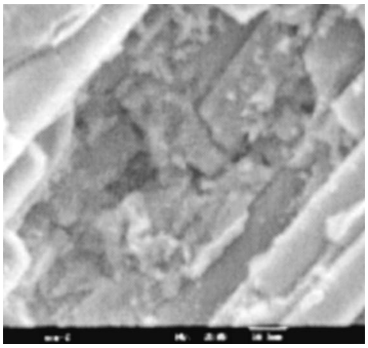

Fig. 5. The surface topography of 2D SiC_f/SiC composites at the center part of laser spot, after three pulses laser irradiation in 0.1 s, injection energy 2.14 MJ/m^2 (magnification 1000×).

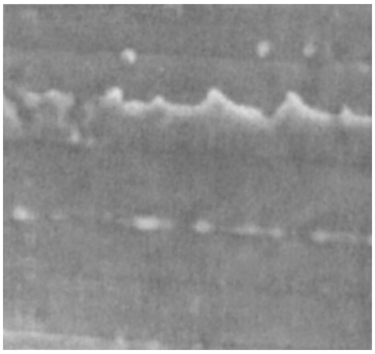

Fig. 6. The surface topography of 1D SiC_f/SiC composites at the center part of laser spot, with injection energy 10.18 MJ/m^2 in 0.44 s (magnification 1000×).

proximate to three-dimensional heat transfer and there are no cracks in the surface of these coatings.

3.3. SEM observation in SiC_f/SiC composites

The surface of 2D Hi-Nicalon SiC_f/SiC composites under the thermal shock indicates sublimation and trace melting, as shown in Fig. 5. The melting was only present in the matrix, and not in the fiber. The more obvious picture is shown in Fig. 6, which is the surface of 1D Hi-Nicalon SiC_f/SiC composites under thermal shock. This specimen had been irradiated by the cyclic electron irradiation and suffered interface damage between the fiber and the matrix. Fig. 6 shows that the matrix is melted in the melted region, but the fibers were inspected and showed no damage. This is because the density of the matrix is lower than that of the SiC fiber. When heat reached the fiber, the heat was transferred quickly along the fiber. But as the surface of the matrix is rough, it is easier to be melted at the tip of infiltration. This means that the matrix has much more thermal resistance than that of fiber, especially for interface damage between the fiber and the matrix.

4. Summary

1. The erosion of SiC_f/SiC composites and TiC + TiN coating under thermal shock is the lowest; the erosion of WC and TiN coating under thermal shock is the second lowest. The mass loss of B_4C under thermal shock is not serious to influence the plasma operation.
2. The surface of TiC, TiN and TiC + TiN coatings made with the MAIC method under thermal shock has a crack pattern. It seems that these coatings approximate to two-dimensional heat transfer because of the interface between the coating and the substrate. The surface of the TiC coatings made with the IBED method, and B_4C and WC coatings made with the PSC method, under thermal shock does not crack. It seems that these coatings approximate to three-dimensional heat transfer.
3. The surface of 2D Hi-Nicalon SiC_f/SiC composites under thermal shock indicates sublimation and trace melting. The melting is only present in the matrix, and the fiber retains integrity. This is because the density of the matrix is lower than that of the SiC fiber. When heat reaches the fiber, the heat is transferred quickly along the fiber. However, the surface of the matrix is rough, and it is easier to be melted at the tip of infiltration. This means that the matrix has much more thermal resistance than the fiber, especially for interface damage between the fiber and the matrix.

References

[1] J.F. Crawford, J.M. Gahl, J.M. McDonald, J. Nucl. Mater. 203 (1993) 280.
[2] H. Madarame, T. Sulegawa, Fus. Eng. Des. 19 (1992) 339.
[3] T. Tanabe, M. Fujine et al., J. Nucl. Mater. 200 (1993) 120.
[4] S. Akiyama, S. Amada, A new method to evaluate the thermal shock resistance of ceramics by laser pulse irradiation, Mater. Eng.
[5] K. Fujii, R. Yamada, J. Nucl. Mater. 258–263 (1998) 1953.
[6] J. Yu, Y. Chen, Y. Wang et al., J. Nucl. Mater. 233–237 (1996) 771.