



# The thermal shock resistance of a joining material of C/C composite and copper

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## Abstract

Plasma facing materials for the next fusion reactor devices will have severe problems such as thermal shock fracture, surface erosion and injection of sputtering particles to plasma caused by charged particle fluxes and very high heat shocks. A joining material, in which a carbon fiber reinforced carbon composite was joined to oxygen-free copper by inserting a molybdenum plate and some metallic films in the joining layers, was developed for a solution of those problems. The thermal shock resistance and the thermal shock fracture toughness were evaluated by an eccentric local heating method of arc discharge. The joining material did not fracture during severe thermal shock tests such as plasma disruption, however, thermal and delamination cracks were observed at the joining parts by scanning electron microscope (SEM). These results can be useful in contributing to the development and the safety design of plasma facing components for fusion reactor devices. © 1998 Elsevier Science B.V. All rights reserved.

## 1. Introduction

Plasma facing materials for the next fusion reactor devices (Large Helical Device, International Thermonuclear Experimental Reactor, Fusion Experimental Reactor, etc.) will have severe problems such as thermal shock fracture, surface erosion due to sublimation, and injection of sputtering particles to plasma caused by charged particle fluxes along magnetic force lines and very high heat shock. Joining materials of carbons and metals are being considered for plasma facing components because they might provide a solution to those problems [1–7].

The purpose of this study is contribute to the development and the safety design of plasma facing components for fusion reactor devices. In this study, thermal shock resistance and thermal shock fracture toughness of a joining material (CX-2002U/Cu) in which a carbon fiber reinforced carbon composite (C/C composite) was

joined to oxygen-free copper were evaluated by an eccentric local heating method of arc discharge. The microstructures were also examined by a scanning electron microscope (SEM).

## 2. Experimental

### 2.1. Materials tested

The CX-2002U/Cu joining material manufactured by Toyo Tanso corporation was made by joining a C/C composite (CX-2002U), which was reinforced by a felt of rayon carbon fiber and was impregnated with pyrolytic graphite by the chemical vapor infiltration method (CVI), to oxygen-free copper at 690–815 by a silver brazing (BAg-3). A molybdenum plate of about 1 mm in thickness and some metallic films such as copper, steel and silver were inserted in the joining layers to accommodate thermal expansions [1]. The homogeneous heat transfer across the joining material was monitored by electron beam heating to check the manufacturing process [1].

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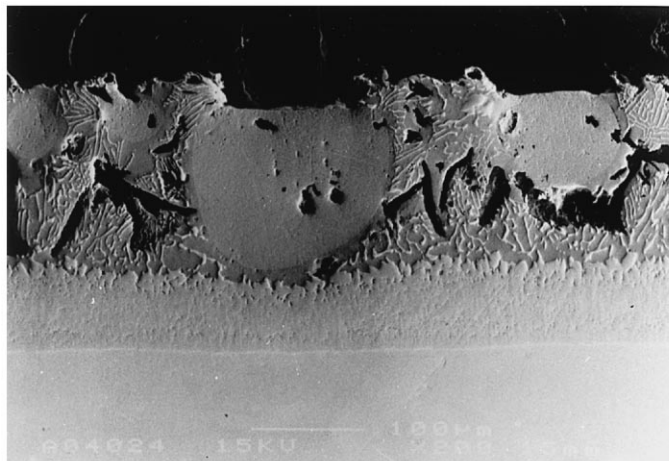
Specimens of the CX-2002U/Cu joining material and the CX-2002U composite were disks of 20 mm in diameter, 3.5 and 2.0 mm in thickness, respectively. The CX-2002U/Cu joining material had the carbon fibers perpendicular to copper to help high thermal conductivity. The CX-2002U composite was cut to measure properties in the perpendicular direction.

Fig. 1 shows the microstructures of the CX-2002U/Cu joining material at (a) the joint of the C/C composite and molybdenum plate and (b) the joint of the molybdenum plate and copper. In Fig. 1(a), an iron-rich layer made of a steel film containing flow-type particles of molybdenum and large elliptic or spherical particles of copper was found by a detailed X-ray analysis [8] and the layer permeates into the C/C composite. Pores and cracks occurred by thermal expansion differences in the

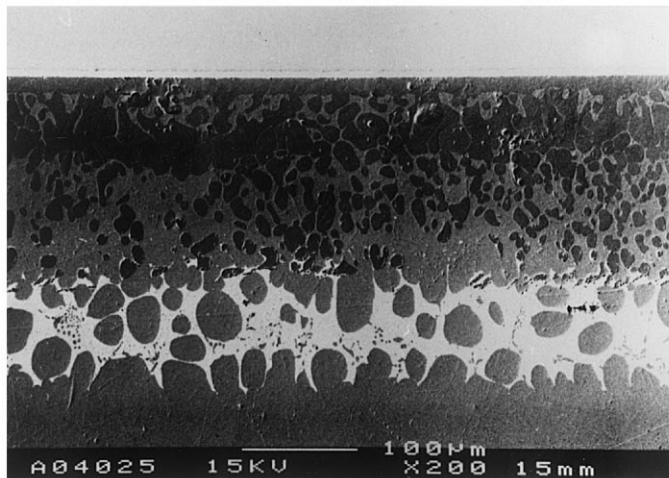
joining process are observed in the layer. In Fig. 1(b), a heat affected zone of about 5  $\mu\text{m}$  in width is found in the molybdenum plate. A copper-rich layer of about 90  $\mu\text{m}$  in width containing small iron particles that are less than 25  $\mu\text{m}$  in diameter and an iron-rich layer of about 80  $\mu\text{m}$  in width containing copper particles were detected by a detailed X-ray analysis [8]. Spherical and isolated copper particles that are less than 45  $\mu\text{m}$  in diameter are mixed in a silver-rich layer. These figures prove the good joining of the CX-2002U/Cu material.

## 2.2. Thermal shock resistance and thermal shock fracture toughness tests

Thermal shock resistance [9],  $\Delta$ , and thermal shock fracture toughness [10],  $\sqrt{K}$ , were evaluated by an eccentric



(a) joining part of C/C composite and molybdenum



(b) joining part of molybdenum and copper

Fig. 1. Microstructures of CX-2002U/Cu.

local heating method [11] shown in Fig. 2 and were calculated as the critical values fractured by transient heating of electric power,  $P$ , as follows,

$$\Delta = \sigma_t k / E \alpha = S^{\star} \beta P / \pi h (a/R)^2, \quad (1)$$

$$\nabla = K_{IC} k / E \alpha = F_{Ic} (\pi c)^{1/2} \beta P / \pi h (a/R)^2, \quad (2)$$

where  $\sigma_t$ ,  $k$ ,  $E$ ,  $\alpha$  and  $K_{IC}$  are the tensile strength, the thermal conductivity, the Young's modulus, the coefficient of thermal expansion and the mode I fracture toughness of the material tested, respectively.  $S^{\star}$  is a specific dimensionless thermal stress and is  $13.71 \times 10^{-3}$  in this study [11].  $F_{Ic}$  is a dimensionless stress intensity factor at the top of the side slit and is  $17.9 \times 10^{-3}$  in this study [11].  $\beta$  is a heat efficiency factor for the arc discharge heating and is about 0.48 for the CX-2002U/Cu joining material and about 0.49 for the CX-2002U composite.  $R$ ,  $h$ ,  $a$  and  $c$  are the radius and the thickness of the specimen, the radius of the heating area and the length of the side slit, respectively. The arc discharge heating time in the thermal shock tests is 0.7 s.

The evaluation method of thermal shock parameters by the eccentric local heating in this study can simulate very severe heat shocks and charged particles fluxes along magnetic force lines and can contribute conveniently to the selection of plasma facing materials.

### 3. Results and discussion

#### 3.1. Thermal shock resistance and thermal shock fracture toughness

Fig. 3 shows the heating electric power or the heat flux in the thermal shock tests. In the figure, white and black symbols indicate data of "did not fracture" and "fractured", respectively. The CX-2002U composite fractured by heating powers of about 5 kW (177 MW/m<sup>2</sup>) in the thermal shock resistance test and about 2

kW (71 MW/m<sup>2</sup>) in the thermal shock fracture toughness test, respectively. The CX-2002U/Cu joining material did not fracture even by very severe heating powers of 12.7 kW (449 MW/m<sup>2</sup>) or 10.9 kW (386 MW/m<sup>2</sup>) near the maximum power of the thermal shock test apparatus. Fig. 4 shows values of (a) the thermal shock resistance,  $\Delta$ , and (b) the thermal shock fracture toughness,  $\nabla$ , calculated by the range between the maximum data of "did not fracture" and the minimum data of "fractured". The  $\Delta$  and  $\nabla$  values of the CX-2002U composite on perpendicular to felt surfaces are  $60.5 \pm 0.9$  W/mm and  $94.8 \pm 7.7$  W/mm<sup>1/2</sup>, respectively. When these values are compared with graphites for fusion reactor devices (ETP-10, IG-430U [12]), the  $\Delta$  and  $\nabla$  values are about 70% and about two times those of the graphites, respectively. The  $\nabla$  value of the CX-2002U composite was improved by the reinforcement of carbon fibers. The  $\Delta$  and  $\nabla$  values of the CX-2002U/Cu joining material are over 83.0 W/mm and over 327.3 W/mm<sup>1/2</sup>, respectively. These values are over 1.4 times and over 3.5 times those of the CX-2002U composite, respectively. The high thermal shock parameters of the CX-2002U/Cu joining material are considered to be reinforced by metals which have very high thermal shock properties.

Fig. 5 shows the appearances of the CX-2002U/Cu joining material after (a) the thermal shock resistance and (b) the thermal shock fracture toughness tests. These specimens received the eccentric local heatings of (a) 449 and (b) 386 MW/m<sup>2</sup> by arc discharge, respectively. These heating powers were about 40 times that of plasma facing materials for a fusion reactor in the next generation (10 MW/m<sup>2</sup> on a divertor plate [2]) and the specimens did not have forced cooling on the copper side. Therefore, the thermal shock tests in this study are considered to simulate very severe accidents such as cooling pipe breakages and plasma disruptions. After the thermal shock tests, there were signs of erosion and redeposition of carbon on the CX-2002U side and marks of melting on the copper side. While delamination

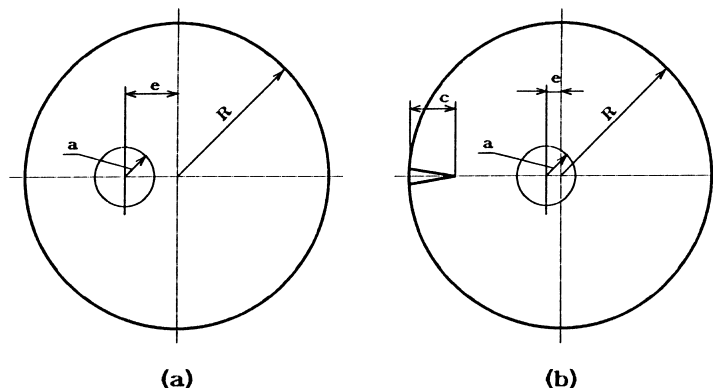


Fig. 2. Test methods of (a) thermal shock resistance and (b) thermal shock fracture toughness.

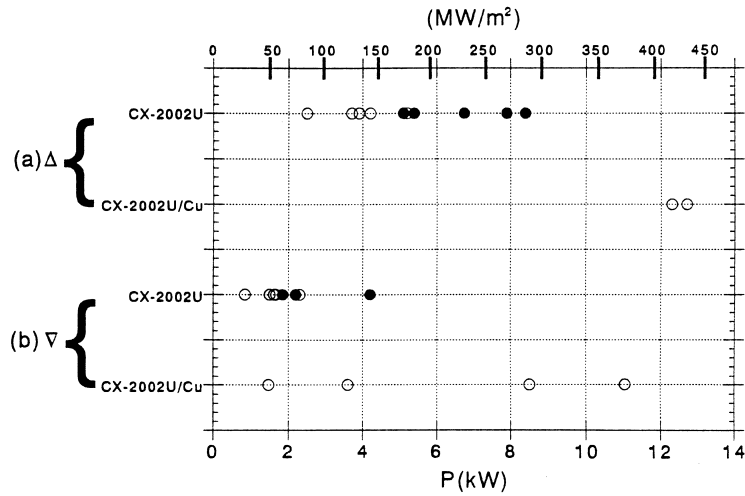


Fig. 3. Heating electric power or heat flux in thermal shock tests.

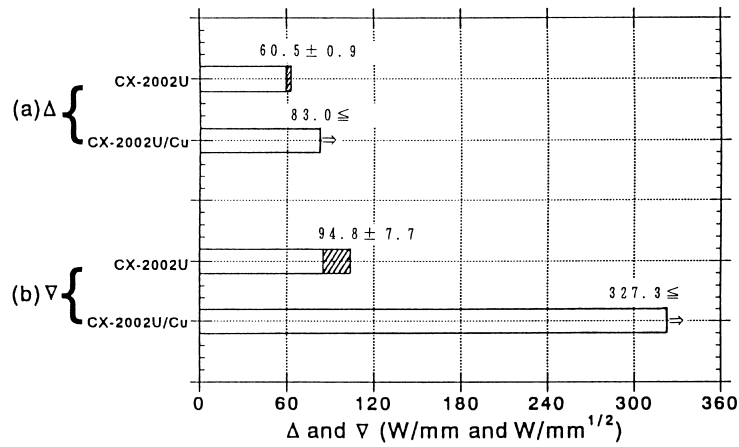


Fig. 4. Values of (a) thermal shock resistance and (b) thermal shock fracture toughness.

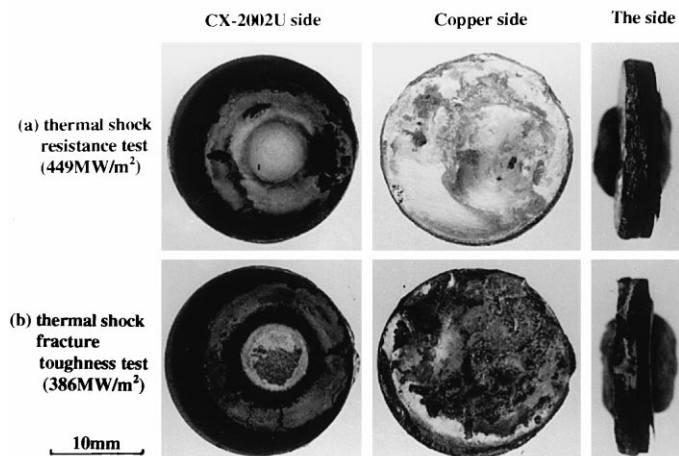


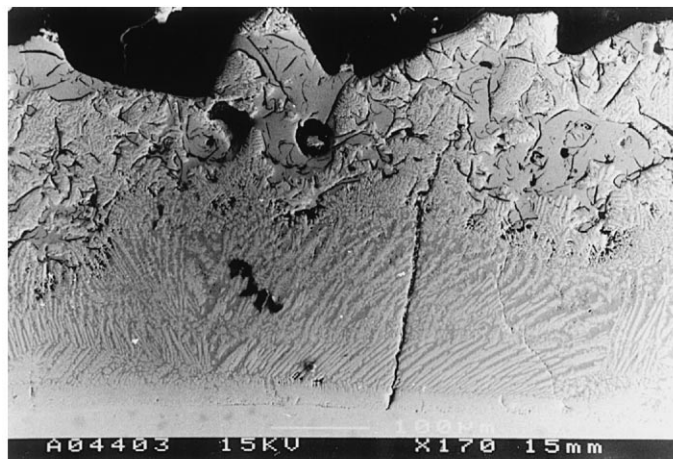
Fig. 5. Appearances of CX-2002U/Cu after thermal shock tests.

cracks were also observed at the boundary of the C/C composite and molybdenum plate on the side surfaces. However, thermal cracks did not propagate from the circumference or the top of the side slit to the center of the heating area on the heating surface.

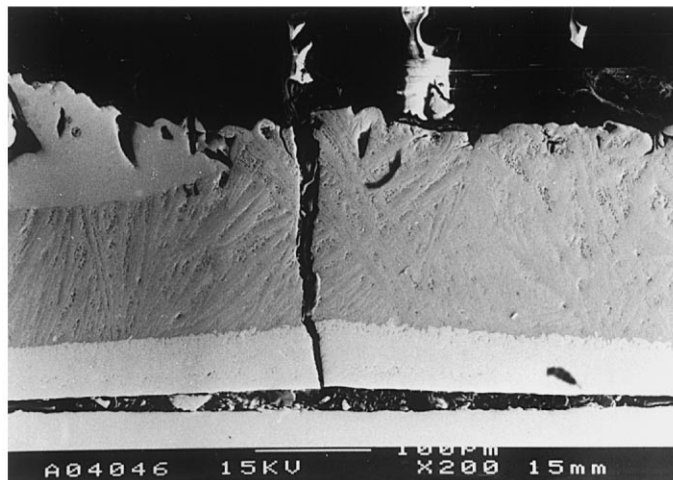
### 3.2. Microstructures after thermal shock tests

Fig. 6 shows microstructures of the joint between the C/C composite and the molybdenum plate after the thermal shock test at (a) the inside area of arc discharge heating ( $6759 \pm 355 \mu\text{m}$  from the edge) and (b) the near area of outside edge ( $1951 \pm 306 \mu\text{m}$  from the edge), respectively. Those microstructures change clearly at the distance from the edge. In Fig. 6(a), the copper layer of about  $100 \mu\text{m}$  in width does not contain particles and

the dendritic structures [13] of molybdenum are growing from the heat affected zone. Iron and molybdenum were found to distribute uniformly in the layer of about  $180 \mu\text{m}$  in width while the deposition of metallic compounds was not observed by detailed X-ray analysis [8]. Therefore, the layer is considered to be an alloy of iron and molybdenum. The dendritic structure supports the idea that the temperature of the area had risen at least over the melting point of iron ( $1539^\circ\text{C}$  [14]) up to near the melting point of molybdenum ( $2625^\circ\text{C}$  [14]) by the thermal shock test and was cooled rapidly under a severe temperature gradient. A thermal crack that was formed by tensile stress in the cooling process at the distance of  $6710 \mu\text{m}$  from the edge propagates perpendicular to the joining layer through the boundaries of the dendritic structures of molybdenum. In Fig. 6(b), a big elliptical



(a) inside area of arc discharge heating



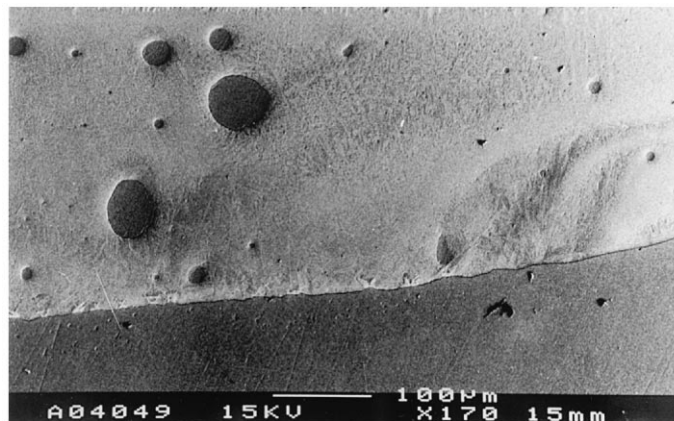
(b) near area of outside edge

Fig. 6. Microstructures of the joining part between C/C composite and molybdenum after thermal shock test.

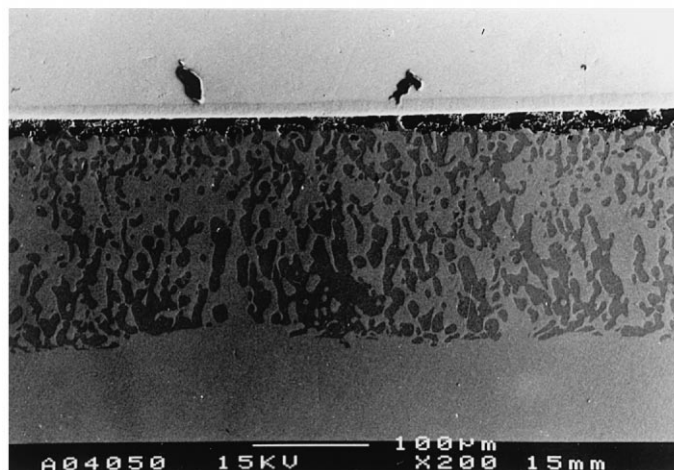
isolated copper particle and columnar structures are observed in the joining layer of about 190  $\mu\text{m}$  in width. The joining layer was found to be an alloy of iron and molybdenum by detailed X-ray analysis [8]. The columnar structure grown perpendicular to the joining layer as a result of cooling and temperature gradient effects supports the idea that the temperature of the area had risen at least over the melting point of iron. A big thermal crack of about 15  $\mu\text{m}$  in width at 1989  $\mu\text{m}$  from the edge propagates from pores of the C/C composite, divides the columnar structures through the heat affected zone of molybdenum and connects with a delamination crack at the boundary of the molybdenum plate. The delamination crack is about 1.94 mm in length along the molybdenum plate. These thermal and delamination cracks respectively are considered to have grown by the tensile and shear stresses induced by the

combination of cooling process and severe temperature gradient. However, they were not found by external observations.

Fig. 7 shows microstructures of the joint between the molybdenum plate and copper after the thermal shock test at (a) the inside area of arc discharge heating ( $6929 \pm 355 \mu\text{m}$  from the edge) and (b) the near area of outside edge ( $1945 \pm 306 \mu\text{m}$  from the edge), respectively. In Fig. 7(a), the joining layer includes spherical copper particles less than about 60  $\mu\text{m}$  in diameter and is considered to be an alloy of iron and molybdenum [8]. The temperature of this area had risen at least over the melting point of iron and the layer changed in width from 250  $\mu\text{m}$  to 340  $\mu\text{m}$  during the thermal shock test. In addition, a fine delamination crack is observed at the boundary of the layer. The joining layer of about 180  $\mu\text{m}$  in width shown in Fig. 7(b) suggests the rapid so-



(a) inside area of arc discharge heating



(b) near area of outside edge

Fig. 7. Microstructures of the joining part between molybdenum and copper after thermal shock test.

lidification of the separate fluid phases of iron and copper. A big delamination crack of about 17  $\mu\text{m}$  in width is observed at the boundary of the layer. However, layers or particles of silver were not observed in the areas of Fig. 7(a) and (b) by X-ray analysis [8]. A possible reason was that the temperatures of the joining material had risen at least over the melting point of silver (961.9°C [14]) and the melting silver diffused to the outside of the specimen through the delamination cracks that occurred at the boundary of the joining layers. The actual temperature of the specimen shown in Fig. 5(b) was measured to be 938°C including the measuring errors, near the melting point of silver, by the CA thermocouples set at the circumference.

#### 4. Conclusion

A joining material (CX-2002U/Cu) in which a C/C composite (CX-2002U) was joined to oxygen-free copper was developed for plasma facing components of fusion reactor devices. The joining material did not fracture even by very severe thermal shock tests over plasma disruptions. Therefore, the thermal shock resistance and the thermal shock fracture toughness of the CX-2002U/Cu joining material were over 83.0 W/mm and 327.3 W/mm<sup>1/2</sup>, and were over 1.4 times and 3.5 times those of the CX-2002U composite, respectively. The thermal shock parameters were improved by effects of reinforcements of metals which have very high performances in their thermal shock properties. External fractures were not observed but thermal and delamination cracks which occurred in the cooling process of the thermal shock tests were observed at the inside and the boundaries of the joining layer by SEM. Therefore,

thermal shock parameters need to be evaluated experimentally in addition to observations of the microstructures carried out in this study.

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