

Damage evaluation of W-coated SiC by thermal conductivity measurement

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Abstract

To evaluate the damage of W-coated SiC/SiC composites by thermal conductivity, evaluation of thermal conductivity measurement was performed for W-coated SiC, which was damaged by electron beam-induced thermal impact. After thermal impact of about 6 MW/m², melted phases around W/SiC interphase and reduction of thermal conductivity were observed. It was suggested that the temperature of the W/SiC interphase was raised to the melting point of a reaction phase, by the thermal impact of about 6 MW/m², and therefore a melted phase flowed out from the W/SiC interphase and cracks, reducing the thermal conductivity. Numerical calculations showed that the temperature of the W/SiC interphase could be controlled by the thickness of the W coating. The relation between melted phases and reduction of thermal conductivity will be investigated.

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1. Introduction

The benefits of silicon carbide (SiC), which is often considered in a fiber-reinforced ceramic matrix composite form because of its brittle nature, include high performance through high temperature operation, low activation energy and low after-heat. Therefore, SiC has been researched as a candidate for the structural material of advanced energy systems, for example, advanced gas turbines, high temperature gas-cooled reactor (HTGR) and fusion reactor [1–3].

Although SiC/SiC composites have excellent characteristics, there are some properties that require improvement. One of them is erosion. When SiC/SiC composites are considered as the plasma facing material of fusion reactor, their relatively high erosion rate is a problem to be solved. Coating high Z materials on the

surface of SiC/SiC composites is a promising solution for the erosion problem.

When the major concern of fusion research was demonstration of a high temperature plasma, even if the plasma was short-lived, high Z materials were not considered to be proper materials for fusion, because of high radiation losses. However, their high melting point and very low sputtering yield made them a candidate for plasma facing materials [4,5]. Especially W and its alloys were researched as armour materials of copper [6,7], carbon fiber-reinforced carbon composites (CFC) [8–11] and SiC/SiC composites. Among these coated materials, W-coated SiC/SiC composites are expected to show better performance, due to excellent characteristics of SiC/SiC composites.

To evaluate the characteristics of W-coated SiC/SiC composites, many mechanical testing methods are being used. In this work, evaluation of W-coated SiC/SiC composites by thermal conductivity was explored. Fig. 1 shows the concept of the use of thermal conductivity as an evaluation tool.

Evaluation by thermal conductivity is thought to be a meaningful method, because it can show, or give, a permissible operation range for plasma facing material

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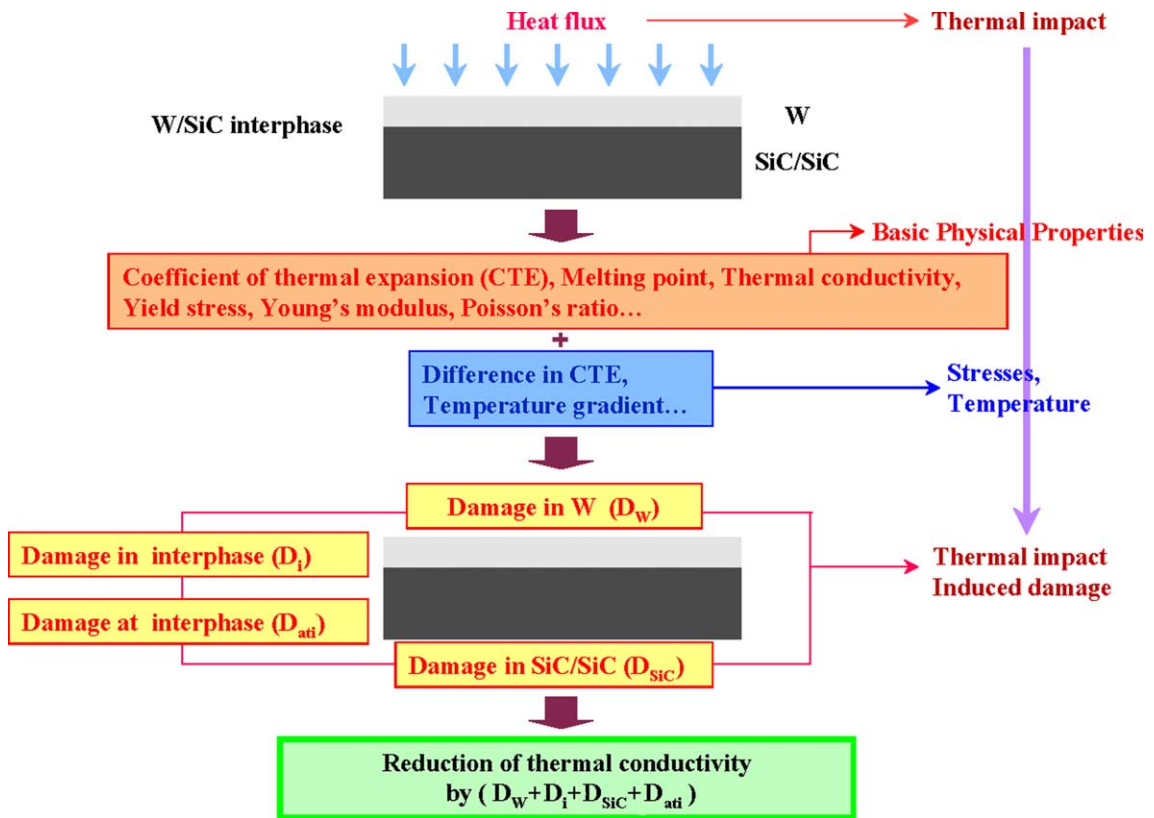


Fig. 1. Schematic flow diagram of damage evaluation by thermal conductivity.

(considering accident cases such as disruption and failure of cooling), a tool for non-destructive evaluation of damage (using the laser flash method) and a possibility for maintenance and/or monitoring of plasma facing components (including in-situ maintenance/monitoring).

The objective of the present work is to evaluate thermal impact-induced damage of W-coated SiC, especially the W/SiC interphase. The detailed objective is to investigate the characteristics of the damage, the mechanism of the damage, and the effect of W-coating thickness on the temperature distribution. A further objective of this work is to evaluate damage in W and SiC of W-coated SiC. The final goal is to evaluate W-coated SiC/SiC composites compared to W-coated SiC.

2. Experiment

2.1. Specimen details

W was coated on SiC by hot pressing [12]. The purity of the W powder was 99.9% and the grain size was about 0.6 μm . The grain size of the SiC powder was smaller than 30 nm and the purity was 99.5%. The hot pressing

temperature, pressure and time for W coating were 1400 $^{\circ}\text{C}$, 20 MPa and 1 h, respectively. The coating configuration was W powder on a SiC plate, which was sintered before coating. The sintering temperature, pressure and time of the SiC plate were 1780 $^{\circ}\text{C}$, 20 MPa and 1 h, respectively.

The coated specimen was disk-shaped for the measurement of thermal conductivity, with a diameter of 9.92 mm and a thickness of 2.67 mm. The specimen was ground with the Mitsui diamond grinding wheel of grain size 170 and concentration 100, before the experiment.

2.2. Cross-section of W/SiC interphase

The cross-section of the W/SiC interphase was studied using Field Emission Scanning Electron Microscope (SEM, JSM-6700F, JEOL). Interphase thickness was 20–30 μm and a reaction phase consisting of WC and WSi_2 , based on XRD analysis [12], was observed. Thicknesses of W coating and SiC were 1.04 and 1.60 mm, respectively.

The thermal conductivity of the W/SiC interphase was about 3.6 W/mK, calculated using simple parallel-slab model [13] with the measured thicknesses and

thermal conductivities of W (108 W/mK) and SiC (15 W/mK) at room temperature, which lowered the thermal conductivity of W-coated SiC (21 W/mK) considerably.

2.3. Thermal impact by electron beam

Thermal impact by electron beam from an electron beam gun (JEBG-303UA, JEOL) was used to heat the W surface of specimen. The acceleration voltage of the electron beam gun was 7 kV and the beam diameter was about 12 mm. The heating time was 7 s. The heat input condition is shown in Fig. 2. It was based on a numerical

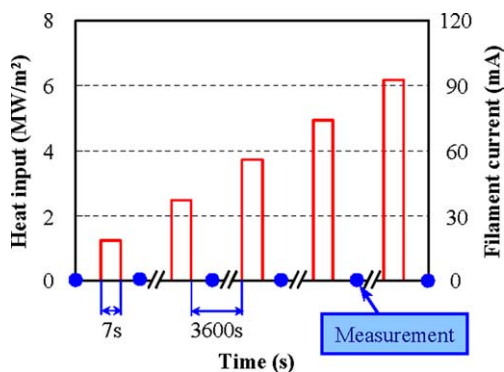


Fig. 2. Heat input and measurement hysteresis.

calculation (Finite Element Method, FEM) using the measured thermal conductivity of W and SiC at room temperature. The emissivity of 0.4 (W) and 0.6 (SiC) were used for the thermal boundary conditions of the finite element calculation. Fig. 3 shows the through-thickness temperature distribution of the center of the W-coated SiC (the case of $T_w = t_w/t_{w/SiC} = 1.04 \text{ mm}/2.67 \text{ mm} = 0.4$), calculated by FEM, after the thermal impact of 6 MW/m^2 . From the FEM results of other

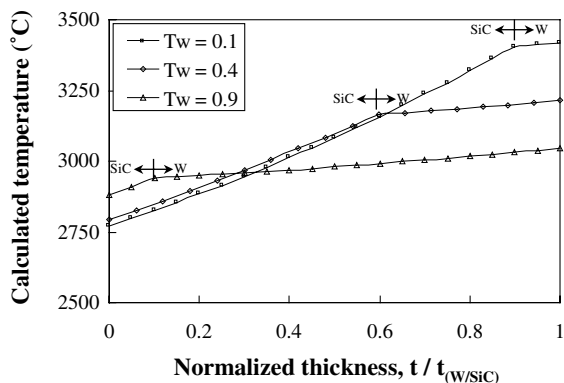


Fig. 3. Through-thickness temperature distribution in the W/SiC specimen after the thermal impact of 6 MW/m^2 , calculated by FEM.

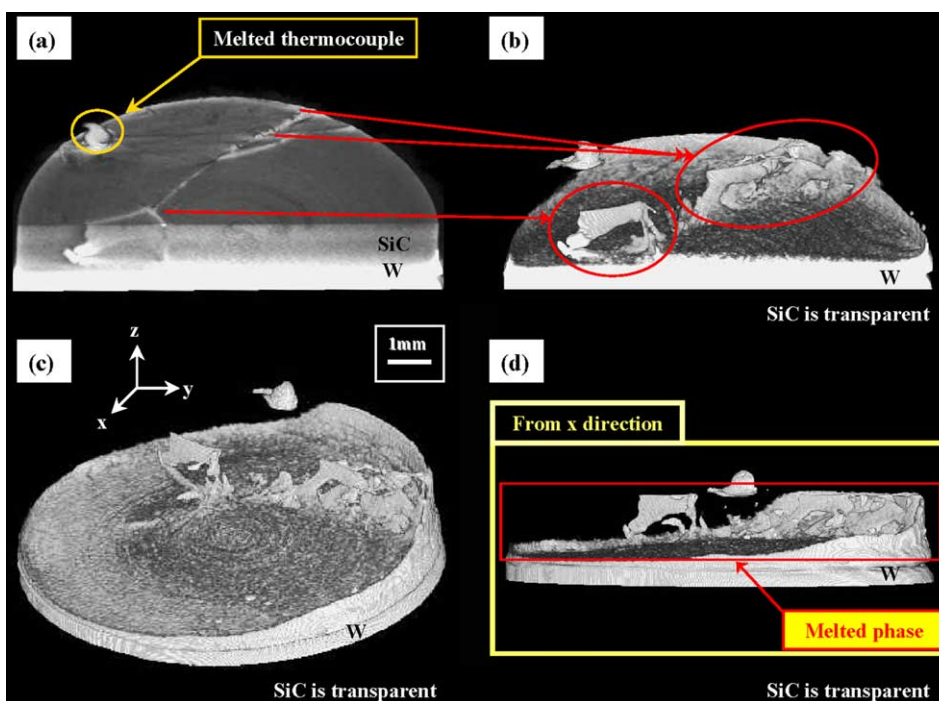


Fig. 4. X-ray CT images of the damaged specimen. (a) CT image with W and SiC. (b) CT image with SiC transparent at the same angle of (a). (c) 3-Dimensional CT image with SiC transparent. (d) CT image from the x direction of (c).

positions of the specimen, the temperature of the W/SiC interphase after the thermal impact of 6 MW/m^2 was expected to be more than $2900 \text{ }^\circ\text{C}$, which is close to the melting points of WC ($2870 \text{ }^\circ\text{C}$) [14] and WSi_2 ($2900 \text{ }^\circ\text{C}$) [15].

2.4. Thermal conductivity measurement and X-ray CT observation

Thermal conductivity was measured by the laser flash method (TC7000, ULVAC-RIKO, Inc.) at room temperature. No surface polishing for thermal conductivity measurement was performed after each thermal impact.

There were lines on the SiC surface after the thermal impact of about 6 MW/m^2 (Fig. 4(a)), which could not be identified using only SEM. To investigate the unknown lines on the SiC surface and the interior of the specimen, X-ray Computer Tomography (CT) observation (IX-300; photograph by I-Bit Co. Ltd.) of the damaged specimen was performed.

3. Results and discussion

3.1. Reduction of thermal conductivity after thermal impact

Fig. 5 shows the measured thermal conductivity of the specimen after each thermal impact. The variations of thermal conductivity before a thermal impact of about 6 MW/m^2 were in the order of measurement error. After the thermal impact of about 6 MW/m^2 , an abrupt reduction of thermal conductivity was observed together with the damage at the W/SiC interphase, which appeared to melt (Fig. 6). The type R thermocouple, which was attached at the SiC surface, was completely melted.

3.2. X-ray CT observation

To identify the metallic line on the SiC surface and investigate the interior of the SiC, X-ray CT observation

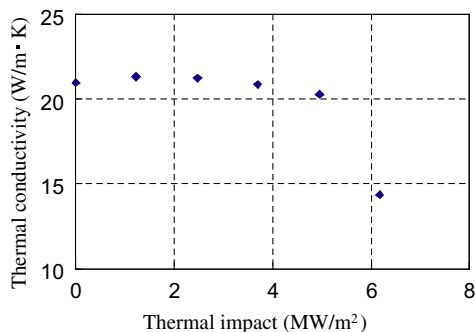


Fig. 5. Thermal impact and measured thermal conductivity.

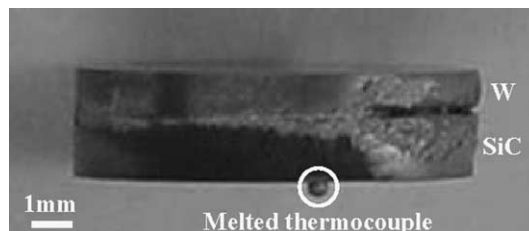


Fig. 6. W/SiC specimen after the thermal impact of 6 MW/m^2 .

was performed. The X-ray CT images are shown in Fig. 4. The line on the SiC, which is seen in Fig. 4(a), was confirmed to be a metallic phase in Fig. 4(b), (c) and (d) with the control of the strength of X-ray to transmit only the SiC. Fig. 4(b), (c) and (d) where the SiC is transparent, show the melted phase clearly from different angles. From the results of numerical calculations and the morphology of the metallic phase, it was thought that the metallic phase was melted W/SiC interphase material, because the temperature of W/SiC interphase was above the melting points for the reaction phases (WC and WSi_2) by the electron beam heating.

3.3. Effect of thickness of W coating on temperature distribution

Fig. 3 shows the through-thickness temperature distribution of the center of the W-coated SiC after the thermal impact of 6 MW/m^2 , for different W coating thicknesses. The ratio of the W coating thickness (1.04 mm) to the specimen thickness (2.67 mm) was nearly 0.4 ($T_w = 0.4$). Because the thermal conductivity of SiC was about 10% of that of W, the variation of temperature in SiC was larger than that of W in Fig. 3. In the case of $T_w = 0.1$, the thickness of W coating is 0.1 times of the thickness of W-coated SiC, the temperature of the W/SiC interphase was about $500 \text{ }^\circ\text{C}$ higher than that of the extreme case of $T_w = 0.9$. The results of numerical calculations showed the possibility of controlling the temperature of W/SiC interphase by varying the thickness of the W coating.

4. Summary

In order to establish a technique for evaluating W-coated SiC/SiC composites using thermal conductivity measurements, evaluation of thermal impact induced damage of W-coated SiC by thermal conductivity was performed. The summary of results is as follows.

- (a) After electron beam-induced thermal impact of about 6 MW/m^2 , melted phases around the W/SiC interphase and a concurrent reduction of thermal conductivity were observed.

- (b) In the case of a thermal impact of 6 MW/m², the temperature of the W/SiC interphase, calculated by FEM, was high enough for WC and WSi₂ to melt.
- (c) X-ray CT observation showed that the melted phase at the W/SiC interphase flowed out from the interphase and cracks of SiC.

From the above results, it appears that the temperature of W/SiC interphase was raised to the melting point of reaction phases, by electron beam heating of 6 MW/m², and that a melted phase flowed out from the W/SiC interphase and cracks to reduce the thermal conductivity.

Numerical calculations showed that the temperature of the W/SiC interphase could be controlled by varying the thickness of W coating. The relation between the melted phases and the measured reduction of thermal conductivity will be investigated.

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