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Irradiation of SiC_f/SiC composites, fibres and matrices in HFR Petten

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Abstract

This paper reports on the pre-irradiation tests of SiC_f/SiC materials as a contribution to the EFDA Technology Programme on advanced materials. A high temperature irradiation of SiC materials in the High flux Reactor in Petten, denoted as 'SICCROWD' has recently started. The SiC_f/SiC materials included are supplied by the EU partners. Further materials are included in the frame of an IEA collaborative effort, by Japanese and USA partners. The irradiation matrix comprises a variety of fibres and matrix materials, as well as composites with 1-D, 2-D or 3-D architectures. CVD monolithic SiC serves as a reference material and should enable comparison with other, e.g. complementary, irradiation data. The specimen types are bars, for four-point bend tests, and discs for flash diffusivity measurements. Both types will be used to measure dimensional changes. Some specimens have been mounted to allow for in-situ determination of thermal conductivity degradation by neutron irradiation. The irradiation rig, materials and test matrix will be presented as well as the results of the pre-irradiation characterisations.

Keywords: Advanced materials; Structural materials; Composites; Siliconcarbide

1. Introduction

As a contribution to the European Fusion Technology programme under EFDA, NRG has a task on irradiation testing of SiC_f/SiC composites. SiC_f/SiC composites are envisaged as candidate structural materials for future fusion reactors because of the low activation of SiC, their good corrosion resistance and excellent high temperature performance. However, the impact of neutron irradiation on the mechanical behaviour, swelling and thermal conductivity of the SiC_f/SiC composites at a high temperature has still to be assessed, and the application window has to be determined [1]. The irradiation matrix comprises a large variety of fibres and matrix materials, as well as composites with 1-D, 2-D or 3-D architectures. The materials are currently being irradiated in the High Flux Reactor in Petten at high temperatures up to a dose of about 4–5 dpa using a dedicated irradiation rig denoted 'SICCROWD' [2–4]. Pre-irradiation testing on each sample is required to

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Table 1
Materials and type of specimens included in the SICCROWD irradiation

ID	Material	Supplier	Weave	Fibre	Matrix	600–650 °C		900–950 °C	
						Bar	Disk	Bar	Disk
A	CERASEP N3-1	SNECMA	3-D	Nicalon	CVI SiC	7	3	6	4
B	CERASEP N4-1	SNECMA	3-D	Hi-Nicalon	CVI SiC	7	3	6	4
C	UBE1	UBE ind.	div.	SA-Tyrannohex	CVI SiC	8	4	8	4
D	ENEA1	ENEA	3-D	Hi Nicalon	PIP(HPCS)-SiC+CVD coating	8	3	8	4
E	ENEA2	ENEA	3-D	Hi Nicalon	PIP(PCS+HPCS)-SiC		3	4	4
F	ENEA3	ENEA	2-D	Hi Nicalon	PIP(PCS+HPCS)-SiC		3	4	4
G	ENEA4	ENEA	3-D	Nicalon	SVI/SiC infiltration+coating		2	2	
H	MORTON	PNL	CVD	Monolithic SiC		2	2	2	2
H	MORTON	PNL	CVD	Monolithic SiC			5		3
I	PNL2	PNL	2-D	Tyrannohex	CVD SiC		2		2
J	PNL3	PNL	3-D	Nicalon S	PIP SiC		2		2
K	PNL4	PNL	1-D	Various	Various		9		11
L	DuPont	DuPont	2-D	Hi Nicalon/150 PyC	ICVI SiC matrix	6		3	
L2	SN4B	MER		Nicalon S	PIP–CVR	3			
M	ORNL CVI-1234	ORNL	2-D	Hi Nicalon S	SVI SiC	8		6	

deconvolute irradiation effects from statistical effects due of the large scatter in physical and mechanical properties of the SiC_f/SiC composites.

2. The SICCROWD irradiation in HFR

A dedicated rig was designed for the two-level high temperature irradiation. The achieved fast neutron dose is in the order of 3.35×10^{25} n/m^2 ($E>0.1$ MeV) and the total neutron dose is approximately 7.30×10^{25} n/m^2, which corresponds to 4–5 dpa in SiC. The SiC_f/SiC composites are irradiated at two temperature levels of 600–650 and 900–950 °C in the same rig.

3. Materials include in the irradiation

The SICCROWD irradiation includes various types of SiC_f/SiC Composites from European, Japanese and US partners. Table 1 lists the different composites, their composition and the available specimen types.

Included are four-point bending bars and discs for the measurement of thermal diffusivity (TD) of the CERASEP N3-1 and CERASEP N4-1 materials from SNECMA. These composites have a 3-D weave of Nicalon and Hi-Nicalon fibres, respectively, in a CVI SiC matrix. From ENEA a few special CERASEP N3-1 pieces were received which were Si-infiltrated or coated with Si–Al–Mg, for morphological analysis before and after irradiation. ENEA also supplied several Hi-Nicalon/PIP SiC matrix composites in 2-D and 3-D weaves.

From UBE Industries in Japan, bend bars of Tyranno SA/CVI SiC were received as well as TD discs of a 1-D composite with 98% fibre content Tyrannohex SA, with the fibres in in-plane and out-of-plane direction. PNNL supplied a variety of TD discs with a dense 1-D fibre structure embedded in an amorphous matrix, for measuring the parallel and perpendicular fibre thermal conductivity. Some advanced CMC's based on Tyranno SA and Hi-Nicalon Type S were supplied as well.

In situ monitoring of the thermal conductivity of the SiC is performed similar to a design of Snead et al. [5,6]. In this design, a thin silicon carbide bar is connected to a heat source (γ-heating of TZM) at one side in the centre of the rig. The other end of the bar is connected to a heat sink, i.e. the containment of the irradiation rig. The thermal conductivity can be determined by measuring the absolute temperatures at both ends

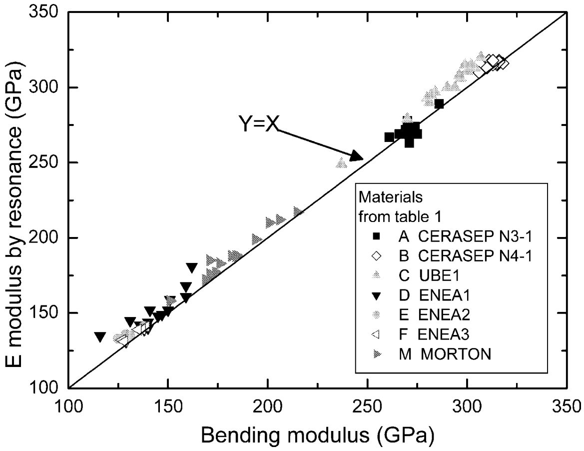

Fig. 1. The bending modulus vs. the dynamic Young's modulus for the different SiC_f/SiC composites showing the scatter between the specimens.

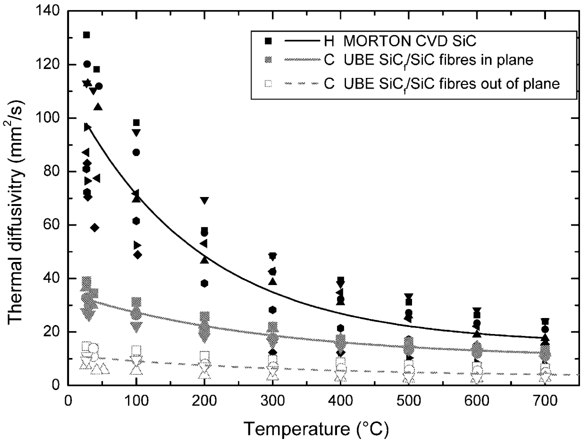

Fig. 2. The thermal diffusivity of the Morton CVD SiC (all black dots) and the UBE SiC_f/SiC composite with fibres in plane (grey dots) and out of plane (white dots) vs. temperature. Different symbol shape means a different individual specimen.

of the thin bar, note the temperature gradient is related to the thermal conductivity. The in-situ measurement of the silicon carbide is monitored at both temperature levels of 600–650 and 900–950 °C.

4. Pre-irradiation test results

Pre-tests were done on all of the specimens before irradiation in order to enhance comparison of the mechanical and physical properties before and after irradiation. The scatter in the properties such as the bending modulus and thermal diffusivity for some of the SiC_f/SiC composites can be large from specimen to specimen due to the large pores and inhomogeneities within the composite.

The bending modulus has been measured by a four point bending setup with a span ratio of 40/20 on an Instron 4501 testing machine. The strain was measured directly on the bar according to the ASTM 855 standard. First, a few specimens were bend to fracture. The onset of movement and fracture of single fibres in the SiC matrix can be determined from the derivative of the loading curve. This stress value was used to fix the limits for the measurement of the bending modulus (ca. 55 MPa). The average value was taken of at least five tests on the same specimen on both sides. It should be noted that in the as-received state the specimens show a 'settling' effect, which presumably is due to the tip of the displacement transducer settling in the hollow parts of the surface. Because of the large surface texture there are hollows on the surface up to about 0.3 mm. The texture makes the determination of the thickness of the sample difficult, causing systematic errors in the bending modulus. The specimens are, therefore, polished to reduce this settling effect and enhance the results that will be obtained after irradiation. This will also improve the relative comparison between the pre-irradiation tests and the post irradiation examination results and will make the irradiation effects more clear.

The impulse resonance method (ASTM C885) was employed to measure the dynamic Young's modulus of the SiC_f/SiC bars using a Poisson's ratio of $\nu = 0.167$. In Fig. 1, the results of both types of measurements are plotted. At ambient temmperature, the static Young's modulus compares well to the dynamic Young's modulus as shown in the graph. However, it can also be observed that for some of the composites, like UBE1, ENEA1 and ORN, the scatter is large between the specimens.

The thermal diffusivity has been measured by the Laser flash method (ASTM E 1461) on discs with a radius varying from 6.3 to 9.8 mm. As for

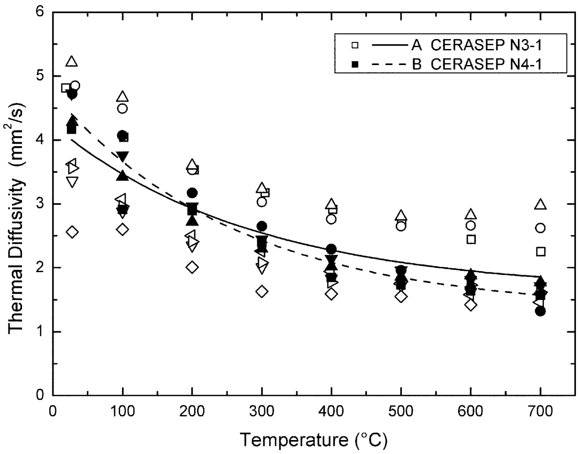

Fig. 3. Thermal diffusivity of various CERASEP SiC_f/SiC composites vs. temperature. Note the large difference in thermal diffusivity compared with Fig. 2. The black dots correspond to individual specimens of CERASEP N4-1, where the white dots correspond to specimens of CERASEP N3-1

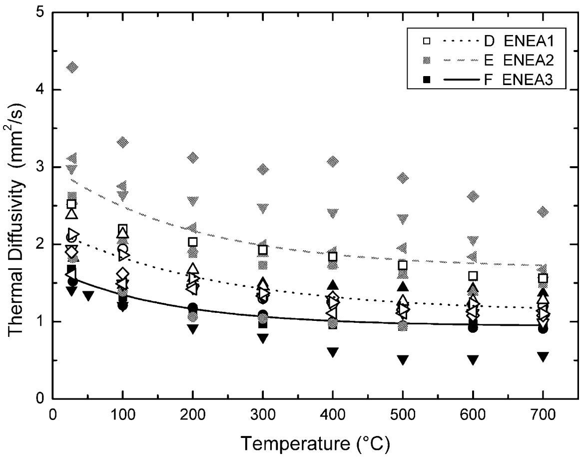

Fig. 4. Thermal diffusivity of three kinds of SiC_f/SiC supplied by ENEA. Different symbol shape means a different individual specimen, where white dots correspond to ENEA1, grey dots to ENEA2 and black dots to ENEA3.

most materials, the thermal diffusivity of SiC_f/SiC composites decreases with increasing temperature as shown in Figs. 2–4. The structure, weave and matrix are denominating factors determining the thermal diffusivity of the SiC_f/SiC Composites, i.e. the composites with the larger pores show a lower diffusivity. The thermal diffusivity of the composites is reduced compared with bulk silicon carbide since the pores act as thermal barriers [7]. In Fig. 2, the influence of fibre direction is shown (UBE material), where a clear difference in thermal diffusivity can be noticed between the in plane fibres and out of plane fibres.

5. Conclusions

A large variety of SiC composites are being irradiated in the HFR, Petten at two temperatures of 600–650 and 900–950 °C up to a dose of 4–5 dpa. The SiC composites have different fibres, matrixes and architectures and have been produced by different infiltration techniques like CVI and PIP. A large scatter was observed in the thermal diffusivity and the Young's modulus of the SiC_f/SiC materials due to the pores and inhomogeneities in their structures, even comparing specimens from the same batch of material. The large test-matrix of the SICCROWD irradiation and the post irradiation results expected by the second half of 2003 will enhance the selection of the fibre reinforced SiC for future fusion power plants.

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