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Fusion Engineering and Design 75–79 (2005) 789–793

Fusion Engineering and Design

www.elsevier.com/locate/fusengdes

Mechanical and thermal properties of SiC_f/SiC composites irradiated with neutrons at high temperatures

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Available online 29 September 2005

Abstract

A high fluence irradiation of SiC_f/SiC composites has been performed to study the effect of neutron irradiation on the mechanical and physical behaviour of those composites. The fibre reinforced silicon carbide composites have been irradiated at two temperature levels of 600 °C and 900 °C up to a fluence of 3.5×10^{25} n/m^2 ($E > 0.1$ MeV). The stiffness of the bending bars after irradiation changed with factors between 0.75 and 1.04 for the different composites while the bending strength after irradiation was reduced with a factor of 2 in some cases. The laser flash thermal diffusivity ratio's (α_{irr}/α_o) of the composites measured at 600 °C are from 0.1 to 0.5 and from 0.25 to 0.75 for an irradiation temperature of 600 °C and 900 °C, respectively. The dimensional changes observed are small.

Keywords: Neutron; Silicon carbide; Conductivity

1. Introduction

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 most of the SiC_f/SiC composites at a high temperature has still to be assessed. In the SICCROWD irradiation programme, a scoping study of various fibre reinforced SiC_f/SiC composites with different fibres, matrices and infiltration processes was performed. The results of the SICCROWD are used for the selection of better fibre, interface and matrix materials in order to design improved composites for fusion applications [1].

In this paper, the results of the post irradiation examination are reported. Bending properties, dimensional changes and thermal diffusivity have been measured after neutron irradiation in the high flux reactor in Petten.

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0920-3796/$ – see front matter
doi:10.1016/j.fusengdes.2005.06.307

2. Irradiation in the high flux reactor in Petten

The SiC_f/SiC composites have been irradiated in one dedicated rig at two high temperature levels of 600 °C and 840–900 °C. The average temperature of the latter slightly decreased during the irradiation in approximately 125 full power days. The achieved fast neutron dose in silicon carbide is from 2.2×10^{25} n/m^2 to 3.5×10^{25} n/m^2 ($E > 0.1$ MeV), where the range is due to the flux buckling. The fluence correspond to a displacement damage of 1.1–2.0 dpa in silicon carbide using the damage cross-section presented in ref. [2], equivalent to 3.5 dpa with the traditional cross-section of 1×10^{25} n/m^2 ($E > 0.1$ MeV) ∼1 dpa in SiC.

3. SiC_f/SiC materials included in the irradiation

The SICCROWD test matrix comprises various types of SiC_f/SiC composites supplied by European, Japanese and US partners [3]. Four-point bending bars for strength analysis and thermal diffusivity discs are included. Two composites from SNECMA, CERASEP N3-1 and CERASEP N4-1 were irradiated. These composites have a 3D weave of Nicalon and Hi-Nicalon fibres, respectively, in a CVI SiC matrix. In addition, ENEA supplied a few special CERASEP N3-1 pieces 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 2D and 3D weaves.

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

4. Results of the post-irradiation examination

Dimensional changes, $(d_{irr}/d_0) - 1$, of all specimens have been measured using LVDT's with an accuracy of

Table 1
Average swelling of the various materials irradiated in the HFR SICCROWD

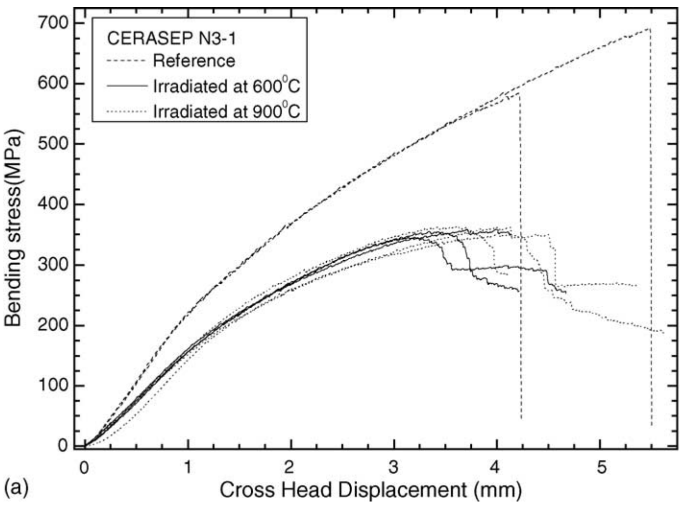
ID	Material	Fibre/matrix	Average swelling longitudinal (%)		Average swelling width (%)		Average swelling height (%)		Average volumetric swelling (%)	
			600 °C	900 °C	600 °C	900 °C	600 °C	900 °C	600 °C	900 °C
A	CERASEP N3-1	3D/Nicalon/CVI SiC	0.09	0.09	−0.1	−0.5	−0.2	−0.5	−0.2	−1.0
B	CERASEP N4-1	3D/Hi-Nicalon/CVI SiC	0.10	0.01	−0.5	−0.7	0.0	−0.2	−0.4	−0.9
C	UBE (in-plane)	SA-Tyrannohex/CVI SiC	0.3	0.2	0.1	0.1	0.6	−0.7	1.0	−0.4
	UBE (out-of-plane)		0.2	0.2	0.2	0.0	0.3	0.1	0.7	0.2
D	ENEA1	3D/Hi-Nicalon/PIP (HPCS)-SiC + CVD coating	−0.24	−0.17	−1.3	−1.9	−2.8	−6.1	−4.3	−8.0
E	ENEA2	3D/Hi-Nicalon/PIP (PCS + HPCS)-SiC		−0.87		−1.5		−1.4		−3.7
F	ENEA3	2D/Hi-Nicalon/PIP (PCS + HPCS)-SiC		−0.8		−1.2		−2.4		−4.3
H	Morton (PNL)	CVD/monolithic SiC	0.3	0.3	0.3	nd	0.4	nd	−0.2	
L	DuPont	2D/Hi-Nicalon/150 PyC/ICVI SiC matrix	0.3	0.1	nd	nd	nd	nd		
L2	SN4B (MER)	Nicalon S/PIP-CVR	0.2		0.2		0.0		0.5	
M	ORNL CVI-1234	2D/Hi-Nicalon S/SVI SiC	0.2	0.1	0.3	0.1	0.0	−1.5	0.5	−1.3

nd, not determined.

Table 2
Results of the bending tests up to fracture of SiC_f/SiC composites after irradiation at high temperature

	Pre-irradiation			Post-irradiation						
	E_{dyn} (GPa)	$E_{bending}$ (GPa)	σ_{max} (MPa)	E_{dyn} (GPa)	$E_{bending}$ (GPa)	ΔE_{dyn} (GPa)	$\Delta E_{bending}$ (GPa)	Rel. (%)	Rel. (%)	σ_{max} (MPa)
SEP N3-1										
c	275	269	645	196	196	−79	−74	−29	−27	334
h	269	263		205	199	−63	−64	−24	−24	349
SEP N4-1										
c	317	311	749	251	257	−67	−53	−21	−17	443
h	315	309		269	259	−46	−50	−15	−16	522
UBE										
c	307	292	273	299	295	0	10	0	3	280
h	304	290		309	295	10	11	3	4	271
ENEA1										
c	151	143	456	146	149	−5	5	−4	4	291
h	147	139		141	146	−6	3	−4	2	281
ENEA2										
h	136	129	583	106	102	−30	−27	−22	−21	569
ENEA3										
h	138	133	438	135	133	−3	1	−2	0	380
ORNL										
c	187	181		187	177	−1	−9	0	−5	325
h	189	179		186	182	−2	0	−1	1	315

c, irradiation temperature 600 °C; h, irradiation temperature 900 °C.



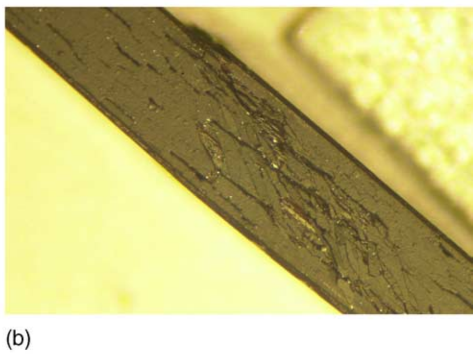


Fig. 1. (a) Four-point bending tests at RT of CERASEP N3-1 up to fracture and (b) photograph of the (shear) fracture plane of the CERASEP N3-1 bending bar.

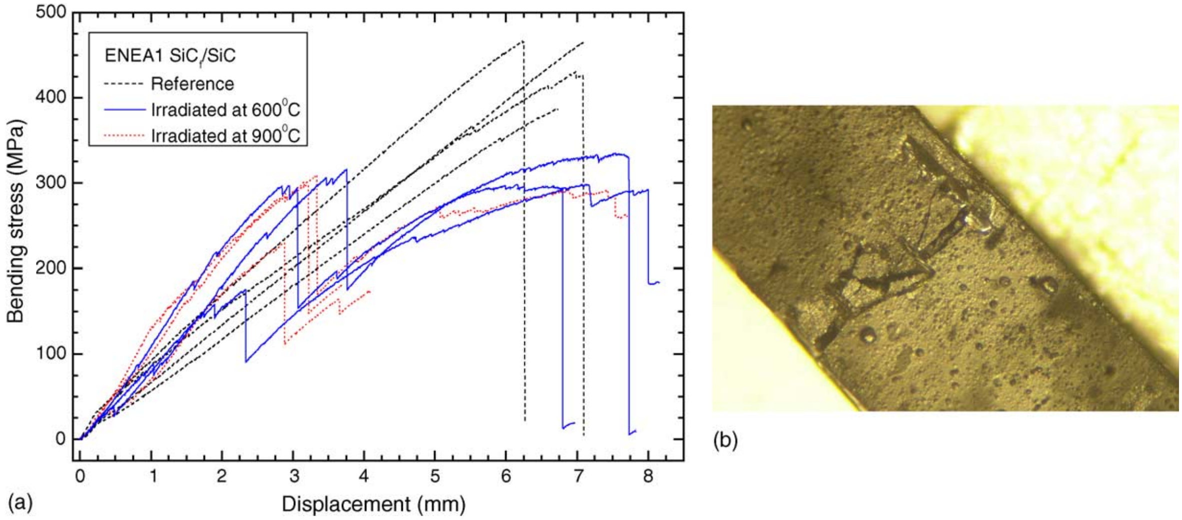


Fig. 2. (a) Four-point bending tests at RT of ENEA1 up to fracture and (b) photograph of the top surface of the irradiated ENEA1 bending bar after fracture.

3 μm. The measurements have been repeated a number of times to reduce the scatter due to the roughness of the composites. The results are summarised in Table 1. All the materials exhibit small dimensional changes of the order of <1%, similar as in ref. [4]. The volumetric swelling has been calculated from the linear swelling.

The bending modulus has been measured by a four-point bending setup with a span ratio of 40/20 mm on an Instron 4501 testing machine. The strain was measured directly on the bar according to the ASTM 855 standard. The bending modulus E_{bend} was calculated using the load and the displacement from the displacement transducers (average of five tests). However, bending to fracture resulted in exaggerated strain signals due to pullout of fibres or fracture of the bending bar. Therefore, displacement values from the crosshead displacement are reported corrected for machine stiffness, measured using a stiff thick bending bar. The impulse resonance method (ASTM C885) was employed to measure the dynamic Young's modulus of the SiC$_f$/SiC

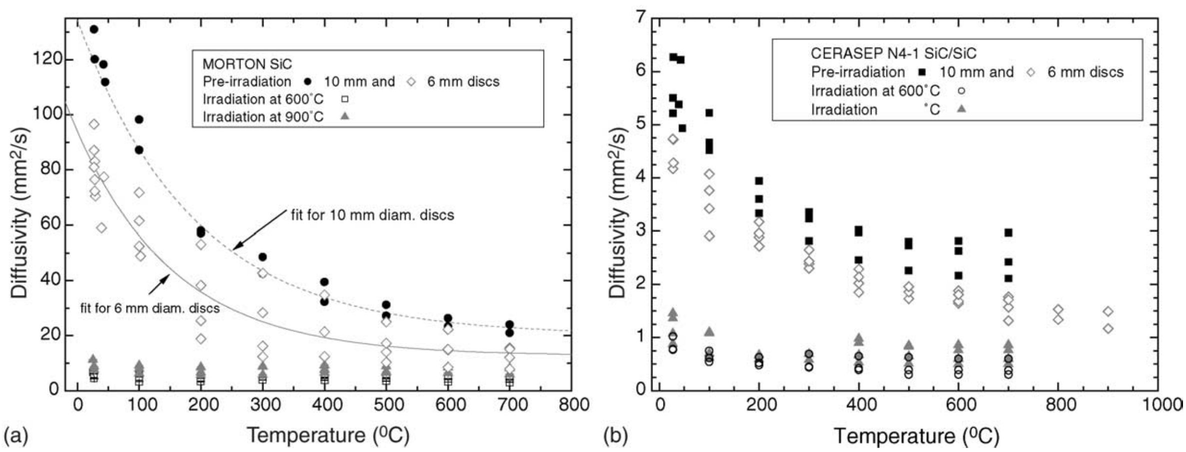


Fig. 3. (a) Thermal diffusivity of irradiated Morton CVD SiC including trend lines (exp) for un-irradiated discs of 10 mm and 6 mm and (b) thermal diffusivity of irradiated SEP N4-1.

bars using a Poisson's ratio of $\nu = 0.167$. The results of the bending and impulse excitation tests (E, σ_{max}) before and after irradiation are tabulated in Table 2. Typical bending curves are shown in Figs. 1 and 2. The Young's modulus of the CERASEP N3-1 and N4-1 as well as the ENEA2 composite decreased with more than 20%. The change of modulus of the other SiC_f/SiC composites due to neutron irradiation was small. The fracture strength of some of the composites reduced with factors up to 2, where the strength of UBE, ENEA2, ENEA3 and the ORNL does not seem to be affected to a large extend by the irradiation, see also ref. [5].

Thermal diffusivity has been measured by the laser flash method (ASTM E 1461) on discs with a radius varying from 6.3 mm to 9.8 mm, before and after irradiation on the same specimens to be able to deconvolute irradiation effects from the normal scatter in these types of materials. The diffusivity after irradiation is decreased strongly due to the irradiation-induced increase in point defects in the matrix of the composite [6]. In Fig. 3, the thermal diffusivity is plotted versus test-temperature of CVD SiC and of a SiC_f/SiC composite. The reduction of thermal diffusivity is larger for the better conducting composites, which is due to the higher density of those SiC_f/SiC's. The lower density material has more porosity and has, therefore, lower diffusivity due gas inside the pores which acts as heat barrier. The diffusivity of the pores is not affected by irradiation, while diffusivity of the matrix and fibres is affected. The reduction in thermal diffusivity is higher for the discs irradiated at 600 °C compared to the 900 °C irradiation.

5. Conclusions

The HFR SICCROWD irradiation of selected composites achieved a fluence from 2.2×10^{25} n/m^2 to 3.5×10^{25} n/m^2 ($E > 0.1$ MeV) at temperature levels of 600 °C and 840–960 °C (where the average of the high temperature region slightly decreased during irradiation). Other observations that were made from the post irradiation examination are:

- The dimensional changes are small: <1% on length at both irradiation temperatures.
- The static and resonance elastic modulus changes from +10% to −25% due to irradiation.
- The bending strength decreased for most of the composites because of irradiation, in some cases with a factor of 2.
- The thermal diffusivity ($T_{measurement} = 600$ °C) decreased as a result of irradiation:
  - with 50–90% for an irradiation temperature of 600 °C;
  - with 25–75% for an irradiation temperature of 850–900 °C.
- The reduction of thermal conductivity is larger for the better conducting material.

Acknowledgements

The work reported is carried out in the framework of the European Fusion Development Agreement with financial support from the European Commission and the Netherlands Ministry of Economic Affairs. The authors wish to thank the EU (B. Riccardi and R. Scholz), Japan (A. Kohyama and T. Ishikawa) and USA (L.L. Snead and G.E. Youngblood) partners for the collaboration.

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