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Journal of the European Ceramic Society 31 (2011) 1141–1144

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Original Article

Crack propagation analysis of SiC_f/SiC composites by gas permeability measurement

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Received 1 April 2010; received in revised form 12 December 2010; accepted 21 December 2010
Available online 3 February 2011

Abstract

Permeability of helium gas through the NITE-SiC_f/SiC composites after applying tensile stress was measured experimentally in a vacuum apparatus. Tensile stress equal to 1.1–1.2 times the proportional limit stress (PLS) was applied parallel to the direction of the reinforced fiber. Results of the permeability experiments revealed that the permeability rapidly increased when threshold stress was applied on the specimens. The permeability of helium gas was governed by the narrowest diameter of the permeation pathway. In the case of NITE composites, the diameter of the pathway was calculated to be below 0.65 μm. The NITE composites exhibited superior performance even when the applied stress was greater than 1.2 times the PLS. Fiber bundles considerably magnified the permeability of helium gas because of the relatively large pore size of the intra-fiber bundles. Transverse cracks propagated with increasing stress and they connected fiber bundles when the applied stress was 1.10–1.15 times the PLS.

Keywords: B. Composites; B. Fibers; B. Non-destructive evaluation; C. Fracture; D. SiC

1. Introduction

Continuous SiC fiber reinforced SiC matrix (SiC_f/SiC) composites are regarded as prime structural candidates for application in advanced nuclear energy systems, such as fusion reactors, because of their superior mechanical properties at high temperature, good irradiation resistance, and chemical stability.[1] The nano-infiltrated transient eutectoid (NITE) process has enabled the production of a nearly full dense SiC matrix while protecting polycrystalline SiC fibers during the sintering procedure.[2] They have excellent gas permeability, high resistance of crack propagation depending on low matrix porosity, and excellent high temperature mechanical properties as compared to composites obtained by other conventional processing techniques.[3] Therefore, NITE-SiC_f/SiC composites are one of the candidates for structural materials for blanket and the plasma facing materials of fusion reactors.[4]

Environmental effects are extremely important while considering the application of the abovementioned composites to aerospace and nuclear reactors. The presence of cracks easily leads to internal oxidation and degradation of permeability or thermal conductivity.[5–7] Therefore, a better understanding of crack propagation behavior can increase the possibility of future applications of the composites. However in the case of NITE-SiC_f/SiC composites, it is difficult to detect micro cracks because of the high resistance of crack propagation, which depends on the low porosity of the matrix. In the present study, the gas permeability of several SiC_f/SiC composites was measured,[8] and It was found that permeability is depends on the micro structure of pores and cracks in the fiber bundle and the matrix.[9] Therefore, helium gas permeability was measured in this study to evaluate the crack propagation behavior. Helium gas was suitable for this high accuracy measurement because of the small diameter of helium atoms.

The aim of this study was to evaluate the effect of crack propagation on SiC_f/SiC composites with the applied stress being greater than the proportional limit stress (PLS). PLS is a more important parameter than fracture strength while designing the structural component.[10] To investigate crack propagation behav-

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doi:10.1016/j.jeurceramsoc.2010.12.028

ior, measurement of gas permeability, which was affected by micro pores and micro cracks, was performed.

2. Experimental

The SiC_f/SiC composites were prepared by the NITE process. These composites comprised layers of a unidirectional SiC fiber bundle with a SiC matrix. Tyranno SA fibers coated with pyrolytic carbon were used as the reinforcement element of the NITE-SiC_f/SiC composites. The diameter and the volume fraction of the SiC fibers were approximately 10 μm and 45%, respectively. The density of the NITE-SiC_f/SiC composites was approximately 3.0 g/cm^3. The specimens prepared by the NITE process for use in a tensile test and subsequent gas permeability measurement were flat plate shaped with a size of 40 mm × 10 mm × 1.5 mm. The gauge length was 20 mm for tensile test.

The tensile test was performed prior to the gas permeability measurement, to introduce micro cracks in each specimen by applying stress greater than the PLS. The tensile test was performed on a digital machine (5581, Instron Corporation, Japan) in air at room temperature. The crosshead speed was 0.5 mm/min. The strain was measured using strain gauges (FLK-6-11-1L, Tokyo Sokki Kenkyujo Co., Ltd., Japan), the gauges being 6 mm long and 3 mm wide located at the center of the both side of the specimens. The average value of strain calculated using both the strain gauges was adopted as the strain of the specimen. On the bases of the stress–strain curve, after the onset of nonlinearity, loading stress was removed completely and then, the specimen was removed from the fixture carefully in order to prevent it from being damaged.

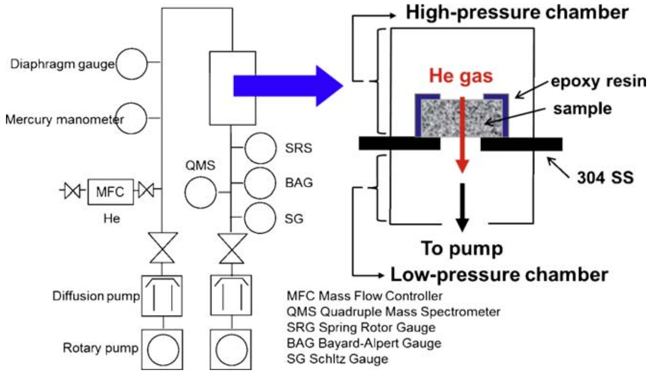

Fig. 1. Vacuum device for measurement of helium gas permeability.

The vacuum system used to measure the permeability of helium gas is shown in Fig. 1. The device consists of low and high-pressure chambers. The specimen was fixed between the two chambers using epoxy resin. The permeation was tested at room temperature. The direction of helium flow was kept perpendicular to the length of the SiC bundle layers. The pressure of helium gas in the high-pressure chamber was adjusted using a mass flow controller and mercury manometer. The pressure ranged from 10^2 to 5×10^5 Pa. After the adjustment of the pressure, the increase in the pressure in the low-pressure chamber was measured using several vacuum gauges. A spinning rotor gauge (SRG), BA ionization gauge (BA), and quadruple mass spectrometer (QMS) were used for the pressure ranges 10^{-5}–1 Pa, 10^{-6}–10^{-5} Pa, and lower than 10^{-6} Pa, respectively. The signal intensities of the BA and QMS were calibrated, and the absolute pressure was measured using the SRG. Diffusion

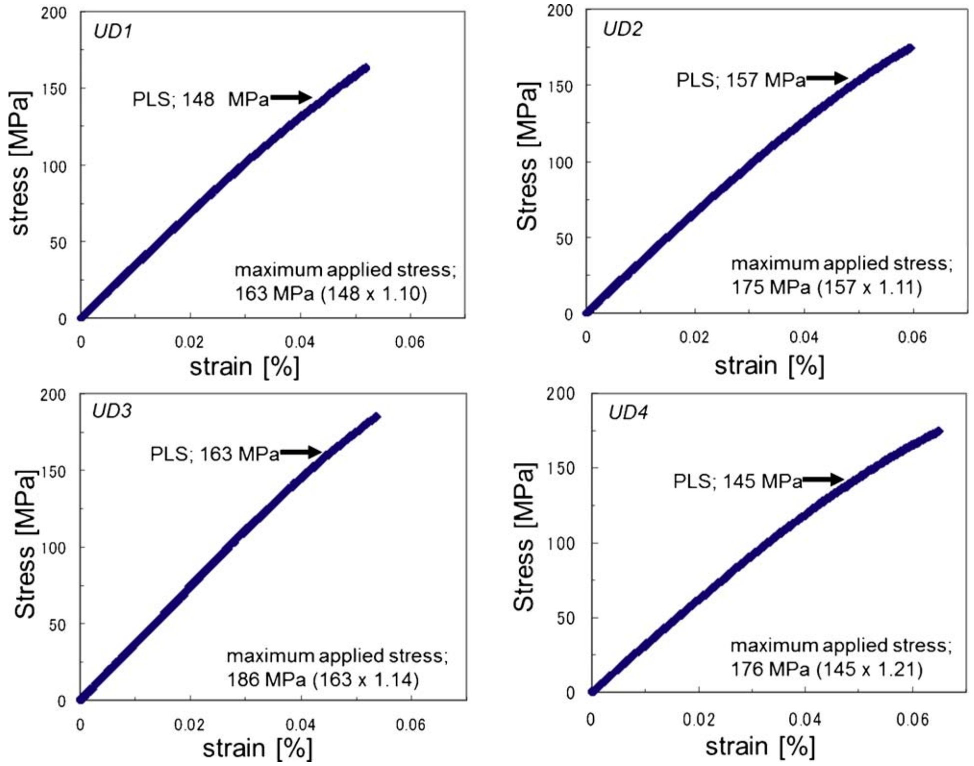

Fig. 2. Stress–strain curves of NITE-SiC/SiC composites corresponding to monotonic tensile tests.

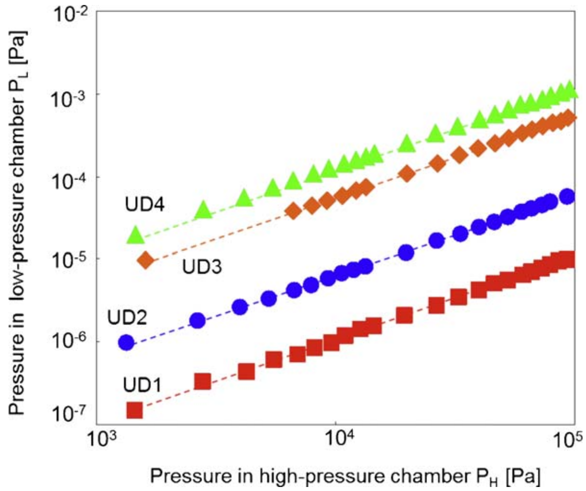

Fig. 3. Pressure in low-pressure chamber versus pressure in high-pressure chamber.

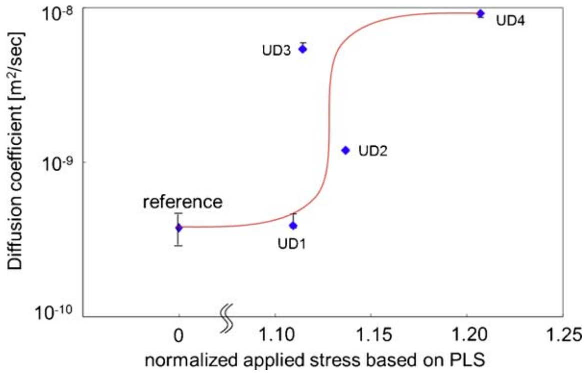

Fig. 4. Diffusion coefficient as a function of applied stress greater than the PLS.

coefficient K was obtained from the increase in pressure in the low-pressure chamber, using the relation [11]

$$K = \frac{P_L d S_{eff}}{P_H A} \quad (\mathrm{m^2/s}) \tag{1}$$

where P_L (Pa) is the low pressure in the low-pressure chamber; P_H (Pa), the pressure in the high-pressure chamber; S_{eff} (m^3/s), the effective pumping speed; d (m), the thickness of the specimen; and A (m^2), the geometrical area. Microstructures were observed using a scanning electron microscope (Model JSM-6700F, JEOL, Ltd., Japan).

3. Result and discussion

3.1. Tensile test

Fig. 2 shows typical stress–strain response curves and damage patterns identified in the experiment. This curve has a short linear range initially and is essentially nonlinear later. The onset stress of nonlinear fracture behavior is defined as the PLS. The various damage levels observed in Fig. 2 are elaborated on and discussed in the subsequent sections. The development of damage in the form of matrix cracks under tensile loading is generally raced by the direct of the carefully polished surfaces of the specimens. In this experiment, however, the level of damage is almost the same as that caused by PLS; thus it is difficult to detect micro cracks by scanning electron microscopy. The values of the onset stress obtained from the non-linear region of the stress–strain curve of composites UD1, UD2, UD3, and UD4 are 148, 157, 163, and 145 MPa, respectively. The values of the maximum stress applied to the composites are 163, 175, 186, and 176 MPa, respectively. Therefore, the degree of applied stress is approximately 1.10–1.20 times the PLS.

3.2. Permeability measurement

The measured P_H–P_L relationship for all the specimens is shown in Fig. 3. The pressure on the down-flow side of the SiC$_f$/SiC composite samples was found to linearly increase with increasing pressure on the upflow side. Thus, The flow of helium gas through the sample is believed to be molecular. The gas flow through the material is characterized by the ratio between the mean free path of helium, λ_{He} (m), and the narrowest path-way size D (m). The molecular gas flow can be described by the following relation:[12]

$$\frac{\lambda_{\mathrm{He}}}{D} > 0.3 \tag{2}$$

From this equation, the narrowest pathway size for helium gas is found to be below 0.65 μm, even in the case of UD4. Thus the crack size in the NITE-SiC$_f$/SiC composites is below 0.65 μm. This result indicates that the composites with low porosity have high resistance to crack propagation.

Fig. 4 shows the diffusion coefficient obtained using Eq. (1) as a function of the applied stress, which is greater than the PLS. The diffusion coefficient for every specimen is constant for varying P$_H$. If the flow in the specimen becomes viscous, the permeability increases with P_H. The permeability in an undamaged composite is 2×10^{-10} m^2/s. The permeabilities in UD1, UD2, UD3, and UD4 are 2×10^{-10}, 1.2×10^{-9}, 6×10^{-9}, and 9×10^{-9} m^2/s, respectively. The permeability

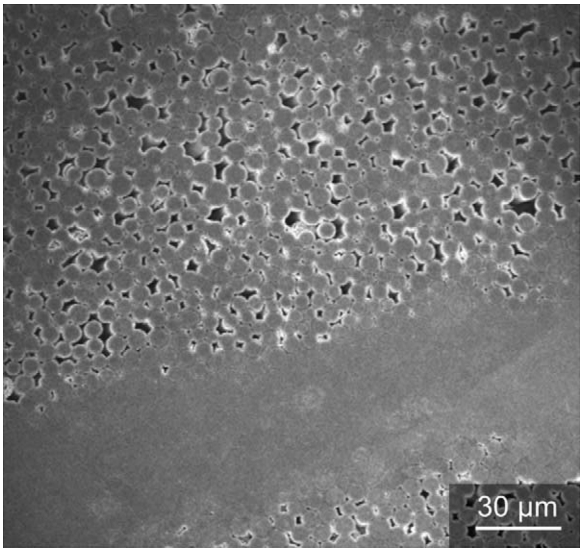

Fig. 5. Cross sectional image of the NITE-SiC/SiC composite observed by SEM.

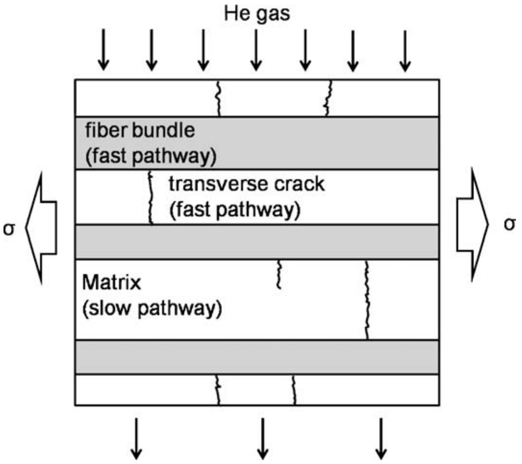

Fig. 6. Schematic image of helium gas pathway.

suddenly increases when the applied stress is 1.10–1.15 times the PLS. This sudden increment in the diffusion coefficient implies crack connection in the material.

3.3. Microstructure observation

Fig. 5 shows the microstructure of the NITE-SiC$_f$/SiC composite. The fiber bundle contains more pores than the matrix. Therefore, helium gas permeates preferentially through the pores in the intra fiber bundle (fast pathway). However, the permeation of the helium gas through the matrix (slow pathway) is poor because the porosity of the matrix is lower than that of the intra fiber bundle region. Cracks behave as fast permeation pathways for helium gas. Considering abovementioned facts and the results of the permeation tests (Fig. 4), we infer that the transverse cracks perpendicular to the stress axis connect fiber bundles when the applied stress is 1.10–1.15 times the PLS (Fig. 6).

4. Conclusions

The permeability of helium gas through the NITE-SiC$_f$/SiC composites was measured using a vacuum apparatus after applying stress greater than the PLS. Micro cracks were successfully detected by a helium gas permeability test. The permeabilities of helium gas through the SiC$_f$/SiC composites were from 10^{-8} to 10^{-10} m^2/s^{-1}, depending on the degree of damage. The narrowest diameter of the helium gas permeability pathway was below 0.65 μm, even when the applied stress was 1.21 times the PLS. The permeability increased when the applied stress was 1.10–1.15 times the PLS. Thus, the transverse cracks propagated with increasing stress, and they connected fiber bundles when the applied stress 1.10–1.15 times the PLS.

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