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Relationships between the accumulative damage and dielectric properties of woven BN-coated Hi-Nicalon™ SiC fibre-reinforced SiC matrix composites

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Abstract

The accumulative damage behaviour of BN-coated Hi-Nicalon™ SiC fibre-reinforced SiC matrix composite was examined under tensile cyclic loading at room and elevated temperatures. The accumulative damage occurring during the cyclic loading was quantitatively characterised using the damage parameter obtained by the hysteresis loop curves. The damage parameter increased with increasing applied stress beyond the matrix cracking stress, and it subsequently retained a nearly constant value until just before fracture. Moreover, the dielectric constant, dielectric loss and loss tangent of the composite were measured before and after the fracture in the frequency range 1–1000 MHz. The dielectric properties had similar frequency dependency before and after the fracture. However, the dielectric constant, dielectric loss and loss tangent were lower in the post-fractured specimens than in the pristine ones. The reduction of the dielectric properties was associated with the accumulative damage stored in the specimens. In addition, the relationships between the dielectric properties and the damage parameter were described in detail.
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Keywords: B. Composites; C. Dielectric properties; Damage parameter; Accumulative damage

1. Introduction

Continuous ceramic fibre-reinforced ceramic matrix composites have been considered as light-weight structural applications because they have unique damage tolerance properties which never achieved by monolithic ceramic materials [1–3]. Unlike the catastrophic fracture behaviour of the monolithic ceramics, the composites exhibit noncatastrophic fracture behaviour that is characterised by accumulative microfracture. Therefore, quantitative characterisation as well as the evaluation of the accumulated damage occurring in the composites is an important subject for the inspection of the composites. Theoretically, the damage parameter could be used to characterize the damage evolution occurring in the composites, which is associated with the change in the materials stiffness [1,4,5].

On the other hand, there are three types of nondestructive evaluation (NDE) techniques for evaluating the extent of damage or life-limiting characteristics such as thermal fatigue, oxidation and damage from ash bridging, including localised cracking, damage from local burning and elongation at elevated temperatures [6–14]. For examples, ultrasonic technique coupled with advanced digital signal processing, has been used to detect the damage in composites [6–10]. In addition, thermal imaging techniques have been developed to detect variable porosity and discrete flaws in composites [6,7,11,12]. Furthermore, X-ray imaging has been used to inspect the flaws and defects such as matrix cracks and pores [6,7,13–15]. Although these techniques have their own advantages, it is difficult to use them for the in situ quantitative evaluation of the damage behaviour of composites; moreover, these methods are insensitive to chemical reaction damage such as oxidation damage. Therefore, it is required to establish a new, effective evaluation process of the damage accumulated

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in the composites, which includes the chemical and physical damages. Recently, a submillimeter range electromagnetic wave has been successfully used to detect the accumulative damage stored in continuous SiC fibre-SiC ceramic matrix composite [16]. However, the frequency dependence of the dielectric properties and the relationships between the dielectric properties and the damage parameter are not well understood.

In this study, tensile cyclic loading–unloading test was performed at room and elevated temperatures using BN-coated Hi-Nicalon™ SiC fibre-reinforced SiC matrix composite. The accumulative damage occurring during the cyclic loading was characterised by the damage parameter. In addition, the dielectric constant, dielectric loss, and loss tangent of the composites were measured for the pristine and post-fractured specimens in the frequency range 1–1000 MHz. Moreover, the relationships between the damage parameters and the dielectric properties were discussed.

2. Experimental procedure

2.1. Composite material

The composite used in this study has been obtained by polymer infiltration-pyrolysis (PIP) from planar woven fabric preform consisting of Hi-Nicalon™ SiC fibres (Nippon Carbon Co., Ltd., Tokyo, Japan). The fibre was continuous β-SiC fibre coated with a BN layer of ~0.4 μm in thickness obtained by chemical vapour deposition (CVD). To reduce pores in the matrix, the PIP process was repeated up to 10 times, which effectively reduces the content of pores in the matrix [17,18]. In addition, a fine β-SiC powder (~4 μm in diameter) was randomly dispersed in the matrix during the fabrication procedure of the composite, and this addition further reduced the pores in the matrix [19,20]. The details of the fabrication are described elsewhere [17]. Hereafter, the fabricated composite was denoted as SiC/BN/SiC. The fibre volume fraction and porosity volume fraction of the composite were approximately 28% and 6.4%, respectively.

2.2. Tensile cyclic loading–unloading test

The composite was cut into a dog-bone type tensile test specimen with the long axis parallel to one of the fibre axis direction by a conventional cutting procedure. Overall specimen dimensions were $150 \times 20 \times 2\ mm^3$ and the central reduced section was $35 \times 9 \times 2\ mm^3$. The detailed shape and dimensions of the tensile specimen are reported elsewhere [21]. The surface of the specimen was unpolished to avoid cutting the fibre bundle. To achieve cooling at the gripped portion and avoid damage associated with gripping, copper plates (1 mm in thickness) were affixed to the gripped portions. Tensile cyclic loading–unloading tests were performed in ambient air with the crosshead displacement rate of 0.5 mm/min at room temperature (298 K) and 1400 K to obtain different accumulative damage, using a MTS 808 system (MTS System Co., Ltd., MI USA). A contact strain metre (MTS Model 632.59) was glued on an edge of each specimen in order to monitor the axial strain. Before loading, the specimens were heated to the set-point temperature at a rate of 50 K/min for a holding time of 10 min to achieve uniform temperature distribution in the specimen. After fracture, the fracture surfaces of the specimens were observed by scanning electron microscopy (SEM). In addition, the edge sides of the post-fractured specimens were polished and the polished surfaces were then observed by optical microscopy and SEM in order to examine matrix cracking and interface debonding occurring during the cyclic loading.

2.3. Measurement of dielectric properties

To measure the dielectric properties, each of the post-fractured specimens under cyclic loading at 298 and 1400 K was cut into rectangular samples perpendicular to the fibre lay-up with size of $8 \times 8\ mm^2$ from the gage-section of the specimen just behind the fracture surface. To examine the effect of the accumulated damage on the dielectric properties of the composite, the pristine composite was also cut into a sample with the same specifics with the post-fractured composites. Prior to measuring, two surfaces of the samples were polished and the parallel surfaces were coated by thin gold film using an ion coater (IB-3, Eiko Engineering Co., Ltd., Tokyo, Japan). The measurements of the dielectric constant, dielectric loss and loss tangent of the pristine and the post-fracture composites were performed at room temperature in the frequency range 1–1000 MHz by using a cavity reflection method (Concept 60, Novocontrol Co., Ltd., Germany).

3. Results

3.1. Mechanical damage behaviour

Fig. 1 shows typical monotonous tensile stress–strain curves of the composite obtained at 298 and 1400 K. The stress–strain curves consist of two appreciable different parts: (i) linear behaviour at the initial stage just after loading, and (ii) nonlinear behaviour after the stress reaches a characteristic value beyond which matrix cracking occurs [22,23]. This stress–strain behaviour was observed for all the samples, regardless of temperature. However, the matrix cracking stress is higher at 298 K than at

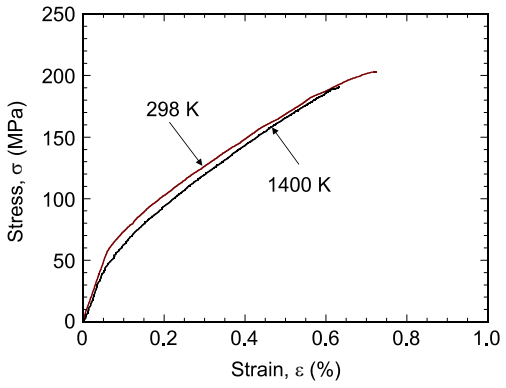

Fig. 1. Typical tensile stress–strain curves of SiC/BN/SiC at 298 and 1400 K.

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