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Anisotropic damage behavior of SiC/SiC composite tubes: Multiaxial testing and damage characterization

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ABSTRACT

The results of a large experimental campaign concerning the mechanical behavior of SiC/SiC composites tubes under uniaxial and biaxial loadings (both tension–torsion and tension-internal pressure) are presented. The anisotropy of the elastic moduli, damage onset and failure properties has been characterized. The orientation of matrix cracking was analyzed, based on surface observations, and its connection to the macroscopic stress–strain response provides important insight into the underlying deformation mechanisms. However, the macroscopic behavior still exhibits unexplained features, and mechanisms specific to the textile architecture are proposed.

1. Introduction

Ceramic-matrix composites are currently studied for a potential use as structural materials for in-core components of advanced nuclear reactor concepts [1]. SiC/SiC composites, composed of 3rd generation SiC fibers (Hi-Nicalon type S – Nippon Carbon or Tyranno SA-3 – Ube Industries) in a SiC matrix, separated by a carefully optimized weak interphase (often pyrocarbon) [2] are promising materials because of their thermal and under irradiation stability and their reproducible and "tolerant" mechanical behavior [3] (compared to the fragile behavior of monolithic SiC). Actually, they can reach significant strains to failure due to the creation of matrix cracks and their deflection at the fiber/matrix interphase.

The design of SiC/SiC structures requires a large amount of material properties to be identified on an extensive experimental database. Although for normal operating conditions only the elastic properties and damage criteria are required, for accidental conditions non-linear damage models and failure criteria must be built. For structural applications, textile architectures of fiber reinforcements are considered, and the mechanical behavior can consequently be very anisotropic, complex and architecture dependant. Building, identifying and validating such models therefore requires a characterization of the material for various loading cases, including multiaxial tests. Biaxial tests have been conducted in the past on composites tubes with 1st generation fibers [4,5] (Nicalon, Nippon Carbon) and a textile architecture made of woven plies wrapped on a cylinder with yarns parallel and transverse to the tube axis. Large evolutions in material elaboration mainly due to SiC fibers improvements but also to the braided architecture limit the current use of these data with respect to the nuclear fuel cladding tube application. More recently, the uniaxial behavior of 2D woven 3rd generation composites at several orientations has been studied [6,7]. However, available data on the anisotropic behavior of these materials remain scarce. The macroscopic behavior underlying mechanisms have been thoroughly characterized and modeled in the case of uniaxial minicomposites [8,9]. However, except for uniaxial cases [10–12], the damage mechanisms are poorly known for 2D or 3D SiC/SiC composites because of the complexity of the microstructure and the variety of loadings that can be applied. Quantitative data about the cracking properties, such as orientation or density evolution is a valuable input for the development of micromechanics based constitutive laws.

In the following, 3rd generation SiC/SiC composite tubes similar to nuclear fuel cladding concepts are characterized at ambient temperature under biaxial loadings: tension-internal pressure (as expected loadings in the application) and tension–torsion, to explore the effect of the stress eigenbasis orientation on the damage mechanisms [13]. All three in-plane components of the average strain tensor at the tube surface are measured using Digital Image Correlation (DIC). Acoustic emission provides qualitative global information

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about the development of the damage processes. Various anisotropic properties required for structural simulations, such as elastic moduli, damage thresholds, and failure criteria are built and identified using the gathered data. In addition, all these stress–strains curves define a rich experimental database of a major interest to build, identify and validate a macroscopic behavior law required for non-linear simulations. From the damage mechanisms point of view, the orientation of matrix cracking is analyzed, using both *in-situ* optical images and post-mortem SEM observations, and provides an insight into the macroscopic behavior underlying mechanisms. The uniaxial behavior however exhibits specific non-linearities that cannot be explained by the visible damage at the surface, and mechanisms associated to the textile architecture are proposed.

2. Experimental methods

2.1. Studied material

The considered material is a SiC/SiC composite based on 3rd generation SiC fibers (Hi-Nicalon™ type S, Nippon Carbon) of 12 μm average diameter. The fibers are coated with a 100 nm pyrocarbon interphase and the fiber preform is densified by the SiC matrix, using for both the Chemical Vapor Infiltration (CVI) process. Optical observations of the composite show no obvious damage due to the elaboration, which is expected from the agreement between the thermal expansion coefficients of the 3rd generation fibers and the CVI matrix [14]. The fiber architecture is composed of three layers with a ±45° orientation relatively to the tube axis (Fig. 1A). The internal layer consists of two filament wound sublayers (that ensures a smoother surface in current fuel cladding concepts) and the intermediate and external layers are 2×2 2D braids (see Fig. 1A). The resulting tubes, after densification, have a regular internal diameter of 7.85 mm. The external diameter is around 9.6 mm, with fluctuations within ±0.1 mm due to the surface irregularity resulting from the braiding process. An average value of the external diameter is measured for each sample by laser profilometry. The average porosity was characterized on a limited number of tubes (3) using X-ray tomography and was between 10.4% and 11.1% for all tubes. The sample total lengths were approximately 98 mm except for compression tests where it was around 65 mm to avoid buckling. After bonding the sample as described below, the corresponding gage lengths were respectively 68 mm and 35 mm.

2.2. Testing setup

2.2.1. Tension-internal pressure and tension–torsion tests

Tension-internal pressure tests are conducted using a custom servohydraulic testing machine which is "force-controlled" insuring a constant biaxiality ratio value $\alpha = \sigma_{zz}/\sigma_{\theta\theta}$ and a given stress rate $\dot{\sigma}_{eq} = \sqrt{\sum_{ij} \dot{\sigma}_{ij}} = 1$ MPa s^{-1}. In order to minimize spurious bending loads (due to misalignments and tightening procedures), tubes are adhesively bonded "in place" to stainless steel grips (see Fig. 1B) using a high shear strength epoxy resin (reference 9323 B/A, 3M) [12]. The upper grip is fixed relatively to the test frame and equipped with an inlet for applying internal pressure (with silicone oil). The lower grip is fixed to the cylinder applying axial displacement. Because of their open porosity, the studied CVI composites cannot ensure leaktighness, even at small stresses and without damage. An intermediate neoprene bladder is therefore used, whose opening ends inside the adhesive joint, ensuring appropriate leaktightness up to tested pressures above 1200 bar. Uniaxial tension and compression tests are conducted on the same machine, without pressure related equipment.

Tension–torsion tests are conducted using a hydraulic tension–torsion testing machine, similarly controlled at the same stress rate and a constant biaxiality ratio value $\beta = \sigma_{zz}/\sigma_{\theta z}$. The tubes are also "in place" adhesively bonded to metallic grips.

2.2.2. Imaging and acoustic emission

Images for *in-situ* observations and DIC strain measurement are acquired using a low noise camera (Orca Flash 2.8, Hamamatsu Photonics) equipped with a ×0.385 telecentric lens (TC 1216, Opto Engineering), giving a magnification independent of the lens-object distance. Motions parallel to the optical axis therefore induce no bias on the measured in-plane displacements. The observed area is 18.1 mm high and covers the full diameter of the tube. The acquisition rate is set between 5 and 11 images per second. To allow a proper observation of cracking and because the natural texture of the material provides enough contrast for DIC measurements, no artificial speckle pattern is applied on the tube.

Acoustic Emission (AE) is often used for a qualitative assessment of damage during mechanical testing of composites, since damage processes release energy in the form of ultrasonic waves. All presented tests are instrumented with an acoustic emission acquisition system (AMSY-6 system, +34 dB pre-amplifier and VS700-D sensor, Vallen Systeme). This system allows recording descriptors of the acoustic events (such as event maximum amplitude, duration, or energy). In the following a focus will be made on the cumulated energy of the acoustic events (which has been proposed as a "damage variable" [15]) and the cumulated number of hits, sometimes found to agree with the surface crack density [12].

2.3. Surface strain measurement

Digital Image Correlation is a widely used technique, allowing measuring the components of the displacement field parallel to

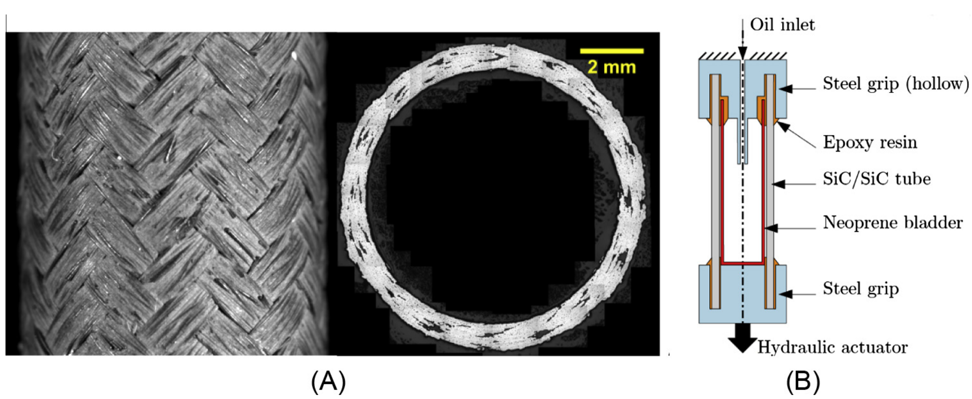

Fig. 1. (A) View of the outer surface and cross section of a SiC/SiC composite tube tested in this study. (B) Principle schematic of the tension-internal pressure setup for the testing of porous SiC/SiC tubes (not to scale). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)