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# Measurement of interfacial residual stress in SiC fiber reinforced Ni-Cr-Al alloy composites by Raman spectroscopy

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ABSTRACT

Raman spectroscopy was used to measure Raman spectra of the inner SiC fibers and surface C-rich layers of SiC fibers, composite precursors and $SiC_f$/Ni-Cr-Al composites. The residual stresses of the inner SiC fibers and surface C-rich layers were calculated, and the effect of the ($Al + Al_2O_3$) diffusion barrier layer on the interfacial residual stress in the composites was analyzed in combination with the interface microstructure and energy disperse spectroscopy (EDS) elements lining maps. The results show that the existence of ($Al + Al_2O_3$) diffusion barrier improves the compatibility of the $SiC_f$/Ni-Cr-Al interface, inhibits the adverse interfacial reaction, and relieves the residual stress inside SiC fibers and at the interface of composite material. Heat treatment can reduce the residual stress at the interface. As the heat treatment time increases, the residual stress at the interface decreases.



## 1. Introduction

The new generation of advanced aero-engines requires continuous research and development of new materials, especially composite materials, to reduce the weight of parts and increase the aero-engine thrust-weight ratio [1]. Ni-based superalloys are currently main materials in the preparation of thermal structures used above 700 °C in aero-engines. However, high specific density limits their further application. SiC fiber reinforced Ni-based superalloy composites have higher specific strength, specific rigidity, high temperature resistance and structural stability, which makes it an effective way to reduce the weight of high temperature parts and improve mechanical properties [2].

The fabrication temperatures of SiC fiber reinforced Ni alloy composites are higher than 950 °C. The coefficients of thermal expansion of SiC fibers and Ni alloys are quite different, which will cause great residual stress at the interface during fabrication of the composites. At the same time, SiC fibers and Ni alloys will react seriously and form new phase near the interface, resulting in the change of interface volume and the formation of residual stress. Excessive interface residual stress will form stress concentration near the interface, causing the initiation and propagation of interfacial cracks and degrading the strength and decreasing the mechanical properties of the composites [3]. The previous study [4] shows that the addition of an ($Al + Al_2O_3$) diffusion barrier coating at the $SiC_f$/Ni interface, while alleviating the thermal expansion coefficient mismatch between the fiber and the matrix alloy and inhibiting excessive interface reaction, can also protect the fiber C-rich coating to ensure the bonding between the fiber and the matrix alloy and reduce the interfacial residual stress.

There are many methods to determine the residual stress of metal matrix composites. X-ray diffraction [5] is common method of the residual stress measurement at present. However, the X-ray penetration ability is limited, so this method is only suitable to measure and estimate the average residual stress of the surface layer (5–7 μm) of composites. Neutron diffraction [6] has strong penetrability, but it is costly and difficult to obtain. Laser Raman method [7,8] can calculate the residual stress in composites by measuring the displacement of the Raman peak under the effect of residual stress. This method has the advantages of high spatial resolution, no contact and damage samples, wide spectrum range and so on, and the residual stress distribution can be obtained, which can better reflect the microscopic residual stress distribution in the interface of the composite material.

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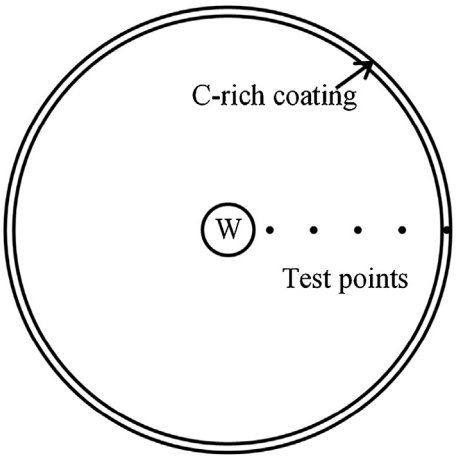


**Fig. 1.** Schematic diagram of test points on cross-section of the SiC fiber.

So far, there are few researches on SiC fiber reinforced Ni alloy composites. A few reports mainly focus on the fabrication of composites and the study of interfacial reactions, and there is no report on the interface residual stress of this kind of composites. In this paper, SiC fiber reinforced Ni-Cr-Al alloy composites with ($Al + Al_2O_3$) diffusion barriers were prepared, and the residual stress intensity and distribution of inner SiC and surface C-rich coating in SiC fibers, precursor wires and composites were measured by laser Raman spectroscopy. The interface morphology and element lining maps of the composites were obtained by scanning electron microscopy, and the influence of the ($Al + Al_2O_3$) diffusion barrier on the interfacial residual stress of the composites were analyzed. The composite samples were treated by vacuum heat treatment. The influence of heat treatment process on the interfacial residual stress was studied, and the cause for the change of residual stress was analyzed.

## 2. Experimental

The continuous SiC fibers (Institute of Metal Research, Chinese Academy of Sciences (IMR), China) fabricated by chemical vaper deposition(CVD) were used, which have an outer diameter of ~100 μm and the ~2.5 μm thickness of C-rich coating on surface. Firstly, precursor wires were prepared by depositing ($Al + Al_2O_3$) diffusion barrier layer and Ni20Cr5Al matrix layer (~32 μm in thickness) on the surface of the SiC fibers using the dual-target mid-frequency magnetron sputtering device. Then the precursor wires were bonded tightly to form precast slabs that were laminated in hexagonal arrangement in a vacuum hot press mold. SiC fiber-reinforced Ni alloy matrix composites were fabricated by vacuum hot press diffusion. The vacuum hot press parameter is 950 °C/60 MPa/1 h.

Raman spectroscopy was performed using a LabRam HR800 Raman spectrometer. The excitation light was He-Ne, the laser wavelength was 632.81 nm, the power was 2.5 W, the spatial resolution was 1–2 μm, and the scan range of all Raman spectra was 300 to 1800 $cm^{-1}$. The test points were selected from the edge of the W core, along the radial direction of the SiC fiber cross-section every 10 μm, until the C-rich coating on the fiber surface, as shown in Fig. 1. Peak separation of the obtained Raman spectra was processed by the analytical software Peakfit, and the wavenumber position corresponding to the respective peaks were determined. The interface morphologies of the composite material were observed by scanning electron microscopy (SEM) (FEG XL30), and the distribution information of the elements at the interface was inspected by an energy dispersive spectrometer (EDS).

## 3. Result and discussion

### 3.1. Residual stress inside SiC fiber

Study [9] shows that β-SiC is a face-centered cubic structure, and the Si and C atoms in the crystal cell form covalent bonds to form a non-centrosymmetric diamond-like structure. In the Raman spectrum, there are two distinct first-order phonon scattering peaks of the SiC crystal. They are the transverse optical phonon mode (TO) peak at 790 $cm^{-1}$ and the longitudinal optical phonon mode (LO) peak at 973 $cm^{-1}$.

The stress on SiC fiber will cause the shift of Raman peaks, and the displacement value of the peak shift has an approximately linear relationship with the stress. According to the displacement of the TO peak of the SiC crystal, the stress value of the SiC crystal can be determined [10]. When compressive stress is present on SiC crystal, the TO peak shift toward to higher wave number, and when the tensile stress is present, the TO peak shifts to the lower wave number. The displacement value $\triangle\omega$ of TO peak of the SiC crystal and the axial stress $\sigma$ of the crystal satisfy the following linear relationship [11]:

$$\Delta\omega_{TO}(cm^{-1}) = -3.27\sigma(GPa) \quad (1)$$

where $\Delta\omega_{TO}$=$\omega_{TO}$-$\omega_{0TO}$, $\omega_{0TO}$ is the TO peak value in the unstressed state, $\omega_{0TO}$ = 790 $cm^{-1}$. The Raman spectra is separated into peaks to obtain TO peak. According to the TO peak value, the displacement value $\Delta\omega$ of the TO peak can be calculated, and the stress values at different positions in the SiC fiber were obtained by Formula (1).

Fig. 2 shows Raman spectra of SiC fiber, precursor wire and composite obtained from different position along the radial of the SiC fibers. It can be seen that the Raman spectra at different positions inside the SiC fiber have obvious changes, with increasing distance from the W core. According to the displacement value $\Delta\omega$ of the TO peak in Fig. 2 and Formula (1), the value and distribution of residual stress along the radial of the SiC fiber are calculated, as shown in Fig. 3.

From Fig. 3, it can be seen that the SiC fibers are under residual compressive stress, and the internal stress of the fibers is parabolic. Along the radial of the fiber, the SiC fibers are composed of the first deposition layer with large SiC grains, good crystallinity, and few stacking faults, the second deposition layer with small and uniform grains size and a little bit of crystalline silicon, and C-rich deposition layer [12]. At the first test point (10 μm from the W core) position, the TO peak is strong and symmetrical, and the stress is greater. The reason is that this point is located in the first deposition layer where the large grains are pressed against each other and the stress in the radial direction of the fiber is less dispersed. At the next tested position (20 μm), the TO peak is weaker and broadened. This point is located in the transitional deposition layer, where the SiC grain size becomes smaller and the faults such as stacking faults increase. The radial stress of the fiber here is more dispersed, so the stress decreases. At the third tested position (30 μm), the TO peak intensity continues to weaken and the waveform is broadened. This point is located in the second deposition layer, where the SiC grain size is fine and homogeneous and the layer disorder increases [13], so the stress is smaller here. The TO peak at the fourth tested point (40 μm) is similar to that at the third tested position. However, the stress increase, because it is affected by compressive stress produced by C-rich coating. As shown in Fig. 3, after the process that the SiC fibers are fabricated to the precursor wires, the compressive stress inside the fibers is reduced, and the stress at the portion near the C-rich coating becomes tensile stress. This is due to the deposition of ($Al + Al_2O_3$) diffusion barrier layer and Ni-Cr-Al alloy layer on the surface of the SiC fibers. The higher thermal expansion coefficient of the deposited layers causes tensile stress on the fibers, which weakens the internal compressive stress of the SiC fiber. SiC fibers are