Carbon fiber reinforced SiC matrix composites (C/SiC) is one of attractive candidates in the ultrahigh temperature components such as turbine rotor and internal combustion part. The objective of this paper is to investigate the characterization of C/SiC composite containing nano scale SiC powder. Especially, the effects of thermal characteristics and thermal shock damage on the C/SiC composite using nondestructive technique were evaluated in detail because the composite is expected to be used in the conditions of high temperature. The C/SiC composite was fabricated by a liquid phase sintering (LPS) process with commercial SiC powder of about 30 nm. The matrix region for C/SiC composites prior to the sintering were prepared by a slurry infiltration technique. The composite material was consolidated at the temperature of 1820°C, pressure of 20 MPa and 2 hours holding time in hot press, respectively. The fractured surfaces of C/SiC composites were also observed to examine the fracture profile of fiber bundle region and the adhesion between carbon fabric and matrix. The parameters such as velocity and attenuation ratio of ultrasonic wave were used to evaluate nondestructively the degree of damage of the composite underwent cyclic thermal shock.
1. Introduction

Recently attention for the green energy and the environment is increasing and many countries are trying to establish their own technique systems for the green energy equipments having high performance and efficiency. It is necessary to develop advanced materials for obtaining the core original technique and enhancing energy industry because the existing techniques have lots of limitations in the developing of advanced green energy and high efficiency of energy. Some researchers are studying on the ceramic composite to complement the ceramics disadvantage of brittleness. These advanced ceramic composite is expected as candidate of ultrahigh temperature materials in the aerospace, nuclear power plant, transportation equipment and defense industry. A C/C composite has a lot of advantages as high temperature structural materials due to its good mechanical properties and durability of abrasion, but it shows a fatal defect of oxidation at 400–500 °C. Therefore, a C/SiC ultrahigh temperature composite that has high heat-resisting property, high strength and anti-corrosion at high temperature, is expected as a material for solving the defect of the C/C composite. However, even basic study on material property to develop the C/SiC composite has not conducted still today, and it is urgently needed to establish the fabrication processing for reliable C/SiC composite. Many researchers are usually used the CVI(chemical vapor infiltration), CVD(chemical vapor deposition) and RS(reaction sintering) methods to fabricate ceramic composite. CVI and CVD methods require too much times for fabrication of the composite and hard to densify completely the composite. RS method decreases the mechanical property of composite at high temperature due to the extra silicon in the SiC materials. In this study, LPS(liquid phase sintering) method was used to fabricate the C/SiC composite because it is possible to do the densification of SiC matrix at lower temperature than that of eutectoid sintering by optimization of additive materials.[1],[2] Therefore, it is important to control the ratio of additive materials for sintering of the SiC matrix by LPS method, and arrangement and laminated structure of fibers are also important to obtain the high quality of the fibers weaving body. A new densification technique of SiC complex slurry needs to impregnate the slurry into the carbon fibers bundle for the C/SiC composite. The developed C/SiC composite should be tested about the effects of temperature because it will be used at extreme conditions of high temperature and pressures. Study of the cracks on the composite caused thermal stress by the rapid temperature change between the inside and the outside of material should be conducted because these cracks might remarkably decrease the mechanical properties of the composite. In this study, a new impregnation method of SiC complex slurry into fibers bundle was suggested, and the effects on thermal shock damage of C/SiC composite using nondestructive technique[3] were studied.

2. Materials and Fabrication process

The SiC power size used in this study is average 30nm and diameter of carbon fiber is about 5μm. Two kinds of sintering additives of Al2O3 and Y2O3 particles were mixed in the designed ball milling device with SiC complex slurry at the condition of 160rpm blending speed and for 12 hours. The compositional ratio of Al2O3 and Y2O3 was fixed at 1.5 wt%. The 1.5wt% was an optimal composition ratio at previous study of monolithic SiC composite. The plane woven fabrics of carbon fiber were prepared for the C/SiC ultrahigh temperature composites. In order to impregnate the mixture of SiC slurry and additive materials into the plane woven fabrics the carbon fibers were desized in the electric furnace. Desizing temperatures have changed from 500℃ to 600℃ to find out the optimal mild condition of carbon fibers. The carbon fibers were damaged at the temperature over 600℃, and the fibers were desized at 550℃ in this study. Fig. 1 shows the photo of carbon fibers according to the desizing temperatures. As shown in figure, the carbon fibers did not get damaged and were the mildest at 550℃ and 600℃. Fig. 2 shows the schematic
diagram of C/SiC composite. Fig. 2(a) represent the instrument for impregnation of the mixture of SiC complex slurry including the additives into the prepared plane woven fabrics of carbon fiber experienced desizing treatment. The plane woven fabrics were set on the filter and the SiC complex slurry impregnates into the plane woven fabrics by the vacuum device under the SiC slurry. Fig. 2(b) shows the process of LPS method in the hot press under an argon atmosphere for the fabrication of C/SiC composite. C/SiC composites with laminated prepregs of plane woven fabrics including the SiC complex slurry were fabricated under the temperatures of 1820°C and the pressure of 20 MPa for 2 hours. The phase diagram of Al2O3-Y2O3 system was used to determine the sintering temperature for the C/SiC composite.

3. Results and Discussions

In order to observe the microstructure for C/SiC composite a scanning electron microscope (SEM) was used and the surface of C/SiC composite fabricated by LPS method was polished by diamond powders. A X-ray diffraction (XRD) was also used to analyze the phases transformations of additive materials in the
morphology of C/SiC composites. Fig. 3 shows the representative microstructure of the C/SiC composite. As shown in figure SiC complex slurry infiltrated densely into the plane woven fabrics even though some micro-pores were found in the plane woven fabrics. The sintered density of the C/SiC composite was 2.88Mg/m³ and it was similar to the theoretical density. The sintering density of the C/SiC composite was measured by the Archimedes’ method. Therefore, the impregnation method designed in this study was a little effective to infiltrate SiC slurry by changing the vacuum levels. The mechanical properties of C/SiC composite were investigated by three point bending test at room temperature and the specimen for the test was processed by dimensions of $6 \times 40 \times 2(t)$ mm³. The test was performed at 32mm of span length and 0.5mm/min of crosshead speed. The flexural strength of the C/SiC composite was about 305 MPa and lower extremely than that of monolithic SiC materials at previous study.[4],[5] This is a little lower than theoretical strength, and it is regarded that a lot of micro-cracks in the plane woven fabrics as shown in Fig. 3 are caused of the gap. Fig. 4 represents the fracture surface of the C/SiC composite. As shown in figure the fracture surface showed a typical brittle on the matrix of SiC material, but the pull-out phenomena in the plane woven fabrics were observed at the interface by detachment between the carbon fibers and SiC matrix. The fracture surface was also not clear at the plane woven fabrics and arranged in tiers. Some part of the plane woven fabrics was apart from the fabric bundles by weak connection of fibers and SiC materials. We are supposed to do something like coating of carbon fibers to improve the bonding of fibers and matrixes in the future study.

Fig. 3 Microstructure of C/SiC composite using SEM

Fig. 4 Facture surface of the C/SiC composite
In this study an ultrasonic wave[6], was used to evaluate the mechanical properties of the C/SiC composite according to the thermal shock[7] damage nondestructively. A device was designed for the repetitive thermal shock damage of the C/SiC composite. The specimen was heated in the furnace temperature of 320°C for 20 minutes, and dropped into the water tank located under the furnace and cooled in it(20°C) for 2 minutes. The cooled specimen goes up automatically inside the furnace and heated again. These cycles are repeated. The both parameters of velocity and attenuation coefficient of ultrasonic wave in the C/SiC composite were calculated by measuring the amplitude and the propagating time of the waves and the thickness of the specimen. The velocity and the attenuation coefficient are measured, and the cracks generated at the surface of the specimen were also observed using microscope at each thermal shock. Fig. 5 shows the velocity and attenuation coefficient of ultrasonic wave in the C/SiC composite according to the thermal shock cycles. The velocity of ultrasonic wave did not show a big gap as thermal shock damages increased. The velocities according to the thermal shock cycles were about 8000m/sec within measuring errors in spite of the production of lots of micro-cracks on the specimen. This says that micro-cracks have not influenced the velocity of ultrasonic wave. Meanwhile, the attenuation coefficients were linearly increased with thermal shock cycles. This is because the generated micro-cracks by thermal shock interrupt the propagation of ultrasonic wave and the waves are scattered by them. The micro-cracks are linked-up each other and developed large crack as the thermal shock cycles increased steadily. The large cracks reduce remarkably the energy of propagating ultrasonic wave through the C/SiC composite. The attenuation coefficients are gradually increased by the scattering of waves caused micro and macro cracks. An equation for the attenuation coefficient was derived by using the exponential growth method and represented in the Fig.5(b). Therefore, the possibility to evaluate the degree of thermal shock damage of the C/SiC composite nondestructively was suggested by measuring the attenuation coefficient. This method can apply to evaluate the mechanical properties of flexural strength and density of the C/SiC composite.

![Graph](image-url)  
(a) Velocity of ultrasonic wave  
(b) Attenuation coefficient of ultrasonic wave  
Fig. 5 Velocity and attenuation coefficient of ultrasonic wave in the C/SiC composite according to the thermal shock cycles
4. Conclusions

From the study the following conclusions were obtained;

(1) In order to impregnate the mixture of SiC slurry and additive materials into the plane woven fabrics the carbon fibers were desized in the electric furnace. Desizing temperatures have changed from 500°C to 600°C to find out the optimal mild condition of carbon fibers.

(2) The sintered density of the C/SiC composite fabricated by new impregnation method was 2.88Mg/m³ and the flexural strength was about 305MPa.

(3) An ultrasonic wave was used to evaluate the mechanical properties of the C/SiC composite according to the thermal shock damage nondestructively, and a device was designed for the repetitive thermal shock damage test.

(4) A possibility to evaluate the degree of thermal shock damage of the C/SiC composite nondestructively was suggested by measuring the attenuation coefficient.

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References


