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INFLUENCE OF THE CREEP ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF SiC/SiC COMPOSITES

SiC/SiC composites were subjected to creep testing at the temperature of 980 and 1100°C. The specimens were tested at maximum stress levels of 100, 150 and 200 MPa. Their microstructure after creep testing were analyzed using a Scanning Electron Microscope (SEM), and the chemical composition was determined by Energy Dispersive Spectroscopy (EDS) and Electron Spectroscopy methods. To evaluate mechanical degradation, the modulus of elasticity (Young's modulus) was determined from the stress-strain data during loading and unloading. All tested specimens were not ruptured after 200 hours of creep testing at 100 MPa, regardless of the temperature. However, at 980°C for the stress of 200 MPa creep specimens were ruptured between 33 and 106 hours, while at 1100°C, they were ruptured in less than 1 hour. It was concluded that the stress level of 200 MPa exceeded the fracture strength of the composite matrix. Analysis of the microstructure of the specimens subjected to creep testing at 1100°C revealed a large number of cracks in the matrix material, which facilitated oxygen penetration and ultimately led to the destruction of the matrix material.

Keywords: Ceramic matrix composites (CMCs); SiC/SiC composites; aerospace materials; creep resistance; microstructure; scanning electron microscopy (SEM)

1. Introduction

Ceramic Matrix Composites (CMCs) reinforced with continuous ceramic fibers are considered promising structural materials for high-temperature applications. Suitable selection of the matrix material and reinforcing phase, as well as the development of innovative microstructure, has significantly increased the resistance to catastrophic cracking of such composites compared to monolithic ceramics. In the case of SiC/SiC composites, the innovative microstructure refers to engineered fiber architectures, tailored interphases, and controlled porosity, which together enhance crack deflection, energy dissipation, and damage tolerance.

Leading aviation companies are interested in introducing CMCs into their aircraft engines. General Electric (GE) has been advancing CMC technology for over 30 years, investing more than \$1.5 billion in the past decade. GE Aviation has focused heavily on developing CMC materials, refining manufacturing processes, and scaling up production. This investment led to the commercial debut of CMC high-pressure turbine shrouds in the LEAP engine, certified by the Federal Aviation Author-

ity and the European Aviation Safety Agency (EASA) in May 2016. These shrouds have accumulated over four million flight hours in LEAP engines used by Airbus, Boeing, and COMAC. GE Aviation is also developing the GE9X, the world's largest aircraft engine, featuring five CMC components in its hot section. These include combustor liners (inner and outer), stage 1 shrouds and nozzles, and stage 2 nozzles. The GE9X, introduced in 2020, powers Boeing 777X aircraft. Additionally, CMCs are being integrated into advanced military engines to enhance performance, offering higher thrust and improved fuel efficiency for future systems [1].

Snecma, Safran Group is considered together with GE as a leader in the field of CMC materials, particularly in SiC/SiC and C/SiC. The manufacturing route used for CMCs is Chemical Vapor Infiltration (CVI). Snecma has extensive experience in rocket propulsion systems and military applications with the C/SiC system developed by Herakles. For example, the M88 engine includes flame-holders, exhaust cones and engine flaps made of C/SiC composites [2]. Rolls-Royce, after acquiring Hyper-Therm High Temperature Composites, Inc. (now Rolls-Royce High Temperature Composites), moved its CMC activities to the USA.

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According to information from the FAA Clean Program, Rolls-Royce has developed static CMC components made of SiC/SiC (HPT blade tracks and shrouds) and is in the phase of engine testing. There is also involvement in joint investigations with Boeing and ATK-COI as well as with NASA, in the development of an OCMC (Oxide-oxide Ceramic Matrix Composite) exhaust cone/mixer. Rolls-Royce is very reserved in publishing information about its activities in composite material research, although presentation titles and a few accessible materials suggest increased activity in this field, including in SiC/SiC [3,4]. Compared to other leading aerospace companies, publicly available information on Pratt & Whitney's (RTX – Raytheon's Advanced Technology business) advancements in CMC technology is relatively limited. There have been publications on the investigation of SiC/SiC systems, including multilayer EBC (Environmental Barrier Coatings) systems, for around 15 years. Experience with long-term testing of SiC/SiC liners in gas turbines with durations of over 10,000 hours is also available [5,6]. Limited publicly available information exists regarding the testing and operational experience of CMCs in military aircraft engines. Nevertheless, indirect references to such applications can be found in various sources, albeit lacking in technical specificity. Historical development programs conducted in collaboration with NASA have been documented. While GE has adopted a phased implementation strategy beginning with static components, Pratt & Whitney has expressed a contrasting viewpoint. The company identifies greater potential for CMCs in rotating components exposed to extreme temperatures (up to 2700°F), rather than in static parts. This perspective is primarily attributed to the relatively low thermal conductivity of CMCs compared to surrounding materials, which may limit their effectiveness in static applications. Pratt & Whitney is a minority owner of CMC manufacturer COI-Ceramics (a joint venture between Orbital ATK, P&W, and Dow Corning). There is a Pratt & Whitney Center of Excellence in Composites at the University of California in Santa Barbara (UCSB) [7]. There are few publications available on IHI Corporation's activities in CMCs. A relatively detailed description of their developments can be found in the IHI Engineering Review series, which showcases components such as vanes and blades, as well as the developed process routes for manufacturing CMC parts, including material selection, forming, and thermal treatment. Currently, no specific information is available about possible or planned applications. Overall, gaining a comprehensive understanding of the Japanese CMC landscape remains challenging, with the exception of the two well-established SiC fiber manufacturers (UBE Corporation, NGS Advanced Fibers Co., Ltd.) [8].

The SiC/SiC ceramic matrix composites investigated in this study demonstrated high strength properties at elevated temperatures. Compared to currently used superalloys, they exhibited significantly greater thermal durability and lower density (typically <3 g/cm³). Among the low-density materials for long-term operation at extremely high temperatures, ceramic composites with a silicon carbide matrix reinforced with continuous, polycrystalline SiC fibers with a small diameter (~10-15 µm) seem to be a potential candidate for application in the aviation industry.

The maximum operating temperature of SiC/SiC composites already introduced into the production of structural components for military and commercial aircraft engines is currently 1250°C. An essential aspect of SiC/SiC composites is their microstructure under the influence of synergistically acting factors in operating conditions, such as temperature and load. In work [9], the SiC/SiC composite – S200H (certified aerospace material) – is characterized by its creep strength. An analysis of creep test results and microscopic examinations were conducted to assess the influence of mechanical and thermal loading on the microstructure of the SiC/SiC composite [9].

Zhu and Kagawa [10] investigates the creep behaviour of different types of SiC/SiC under high-temperature conditions in both inter and oxidizing environments. Standard SiC/SiC contains Nicalon™ fibers with pure SiC matrix, enhanced SiC/SiC contains Nicalon™ fibers with a SiC matrix modified with boron-based, glass-forming particulates and Hi-Nicalon™/SiC reinforced with Hi-Nicalon™ fibers, were tested under 1300°C and constant tensile loads to measure time-dependent deformation (creep strain) and the time to rupture. In this study was found that Hi-Nicalon™/SiC is the most creep-resistant material due to its improved fiber properties and oxidation-resistant matrix. What is more for this material the lowest creep strain rate and the longest time to rupture were observed, making it highly suitable for high-temperature applications. The study highlights the importance of matrix modifications and advanced fiber reinforcements for improving the high-temperature performance and oxidation resistance of SiC/SiC composites. Kalluri and Bhatt [11] evaluated the uncoated CMC under steady thermal gradients with superimposed mechanical loads such as tensile creep and sustained-peak low-cycle fatigue to understand the thermal gradient sustaining capability of uncoated, SiC/SiC composites with different fiber architectures and matrices. SiC/SiC composites with 2D and 3D fiber architectures, 2D balanced architecture CMCs and 3D fiber weave CMCs were tested under 69 MPa peak stress up to 80 h. It was found that no CMC specimen failures were observed after 80 h of creep test. There is a lack of studies in the literature on the impact of creep on the microstructure and mechanical properties of CMC composites at temperatures of 980°C and 1100°C under various stresses. Therefore, this article investigates these aspects.

2. Materials and method of examination

The composite material studied in this research effort was manufactured by COI Ceramic S by polymer infiltration and pyrolysis process (PIP) – known commercially as S200H (TABLE 1). The composite consisted of eight plies of High-

TABLE 1

Components of investigated CMC [12]

Material	Fibers	Fibers coating	Matrix
S200H	Hi-Nicalon	BN	SiNC

NicalonTM [0°/90°] fabric woven in an 8-harness satin weave (8HSW). The fibers are made of SiC, and the matrix is made of SiNC. The fibers are coated with boron nitride (BN) and silica nitride Si₃N₄.

Creep tests were performed on 16 samples: 4 samples at a temperature of 980°C and maximum stress of 100 MPa, 4 at a temperature of 980°C and a maximum stress of 200 MPa, 4 at a temperature of 1100°C and maximum stress of 100 MPa, 2 at a temperature of 1100°C and maximum stress of 200 MPa, and 2 at maximum stress of 150 MPa. The creep tests were carried out in an air atmosphere according to the ASTM C1337-17 standard. All tests were performed by Element Materials Technology (Lancaster, UK), and were carried out in load control with a loading rate of 0,1 kN/s. The strain was monitored using an Epsilon side-contacting Axial Furnace Extensometer with a 25,4 mm gauge length. For tests conducted at elevated temperature, a short profile, 2-zone “zip” furnace was used. The specimen was cold-gripped outside of the furnace.

Tests were discontinued upon reaching one of the following criteria:

- 200 hours at load,
- 2% total strain.

All testing was conducted under the laboratory's UKAS Flexible Scope in accordance with internal procedure – CMC Strain Monitored, Load Controlled Axial Creep Tests which meets the desired requirements.

To determine the modulus of elasticity (Young's modulus E), data recorded during the loading of samples to the desired stress levels and data from the unloading process (for samples that reached 200 hours of testing) were analyzed. The raw data were not subjected to any reduction or filtering prior to further calculations. The linear regression function available in Microsoft Excel was used for data analysis. Linear regression was performed over the stress interval from 5 to 50 MPa for the loading data. For the unloading data, the stress interval from 50 to 5 MPa was analyzed. In cases where unloading was stopped before the stress reached 5 MPa, the stress interval used for calculations ranged from 50 MPa to the minimum recorded value during unloading.

Examinations using scanning electron microscopy (SEM) were performed with a Tescan Vega 3 microscope, applying an accelerating voltage of 20 kV and a using detector for backscattered electrons (BSE). To eliminate carbon contamination during the analysis, conductive carbon tapes from AGAR Scientific were used. For chemical composition analysis in micro-areas, an energy-dispersive X-ray spectroscopy detector (EDS – Energy Dispersive Spectroscopy) was employed. Afterwards, samples were coated with gold on the Quorum Q150R ES vacuum sputter coater.

3. Results and discussion

Based on the analysis of the creep test results of the SiC/SiC composite, it was found that samples tested at a temperature

of 980°C and a maximum stress of 100 MPa reached a life of 200 hours without failure, while the samples at the same temperature but with a higher maximum stress of 200 MPa failed in the range of 33-106 hours (Fig. 1). The samples that were tested at a higher temperature – 1100°C, with a maximum stress of 100 MPa and 150 MPa, also reached a life of 200 hours without failure (Fig. 2). However, increasing the stress to 200 MPa resulted in the samples failing in less than one hour (Fig. 3).

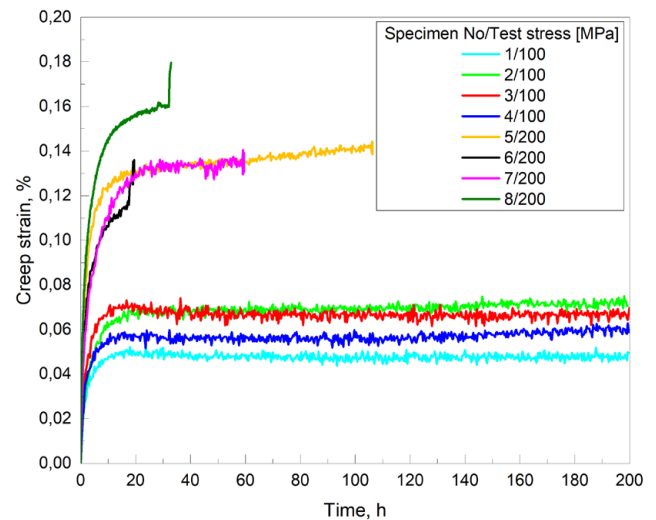


Fig. 1. Strain/time creep curves of S200H tested at 980°C

All samples tested at 980°C at 100 MPa ran out at 200 hours with a very slight increase in strain (Fig. 1). The strain rate at 200 hours was close to zero for each sample. Samples crept at 1100°C at 100 MPa and also ran out at 200 hours (Fig. 2). During creep, samples reached ~0.2% creep strain (~0.3% total strain). The criterion of 200 hours was also reached by samples tested at 150 MPa. These samples crept up to ~0.45% creep strain (~0.6% total strain). The specimens tested at 1100°C at 200 MPa ruptured after a very short time of testing, below 1 hour (Fig. 3). The tested stress value was higher than the matrix cracking

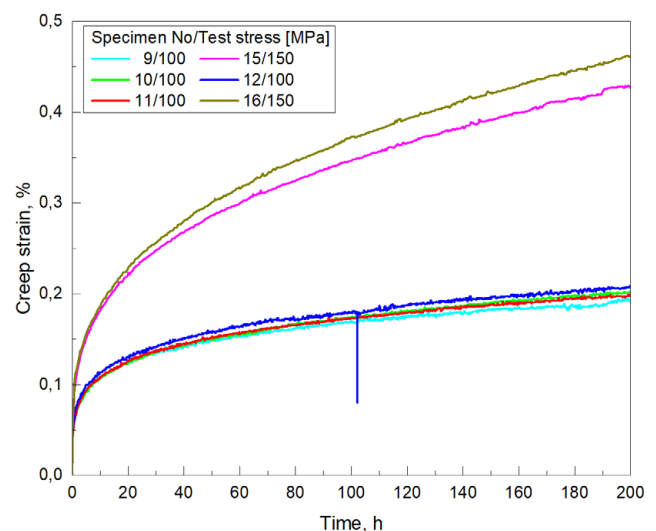


Fig. 2. Strain/time creep curves of S200H tested at 1100°C

strength of the S200H [13]. The formation of cracks in the matrix under continuous load (200 MPa) and temperature (1100°C) acting synergistically caused the material oxidize rapidly [14].

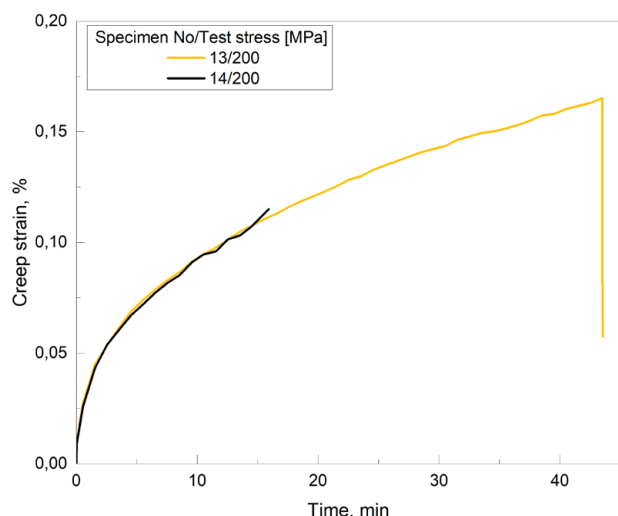


Fig. 3. Strain/time creep curves of S200H tested at 1100°C with a stress of 200 MPa

The modulus of elasticity (E) calculated for all samples based on loading data (with stress interval 5-50 MPa) were similar for both temperatures: at 980°C AV (SD) = 119 (10) GPa and at 1100°C AV (SD) = 112 (9) GPa (Fig. 4). No trend was found for 980°C and 1100°C experiments. The resulting values are lower than those determined from the tensile tests with the use of the same stress interval for E calculations. Measured E at 980°C was 143 GPa and at 1100°C 157 GPa. This could be caused by different loading rate during creep and tensile test as explained in [10], where the higher loading rate gave higher strength, failure strain and Young's modulus, which was interpreted by the controlling factor: creep of fibers. For samples with run out the E was calculated for unloading data. For both temperatures a tendency towards a decrease in stiffness was observed. This is most likely due to an oxidation of material during 200 hours of testing and increase of microcracks in the matrix after a tests.

Fractographic analysis of the rupture surfaces after tensile testing at room temperature and creep testing showed the pres-

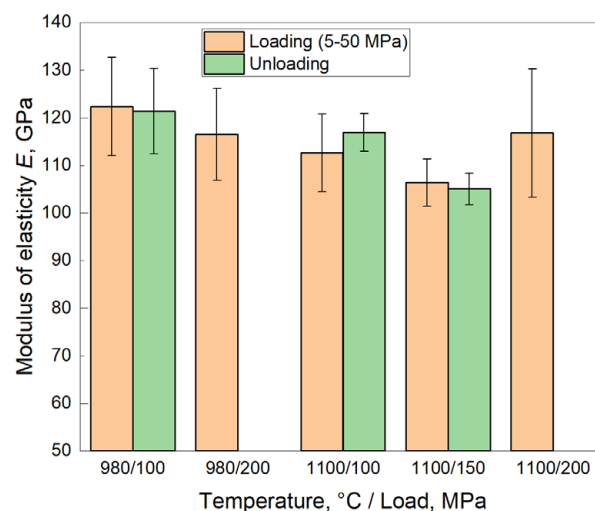


Fig. 4. Values of the elastic modulus E of the tested composite under different loading conditions

ence of a fiber pull-out mechanism (Fig. 5). Fiber pull-out was shown to occur more intensively in the tensile process at room temperature (Fig. 5a), compared to the creep tests (Fig. 5b,c), where loading is accompanied by oxidation. As the creep testing temperature increases, the fiber pull-out mechanism becomes less prominent and visible.

SEM analysis revealed the fibers in the material which are located longitudinal and transverse due to 8HS woven type. The matrix of S200H material has porosity below 5% (based on manufacturer information) and visible cracks produced during PIP process (Figs. 6 and 7). The matrix during PIP process is infiltrated several times. During this manufacturing process, cracks are formed and later are healed due to next steps of infiltration. Porosity can be observed inside the tows and in the matrix. Pores are spherical and elliptical in shape, elongated in one direction, longitudinal and perpendicular to the material plies. Another important features which can be observed are matrix pockets which are the matrix rich location in the material. They can occur as interlaminar matrix layers between plies or as interlaminar matrix pockets within a single layer (Figs. 6b and 7b). SEM analysis revealed the formation of cracks in the matrix material of the composite. These cracks are open cracks that provide an easy pathway for oxygen infiltration (Figs. 6c,d

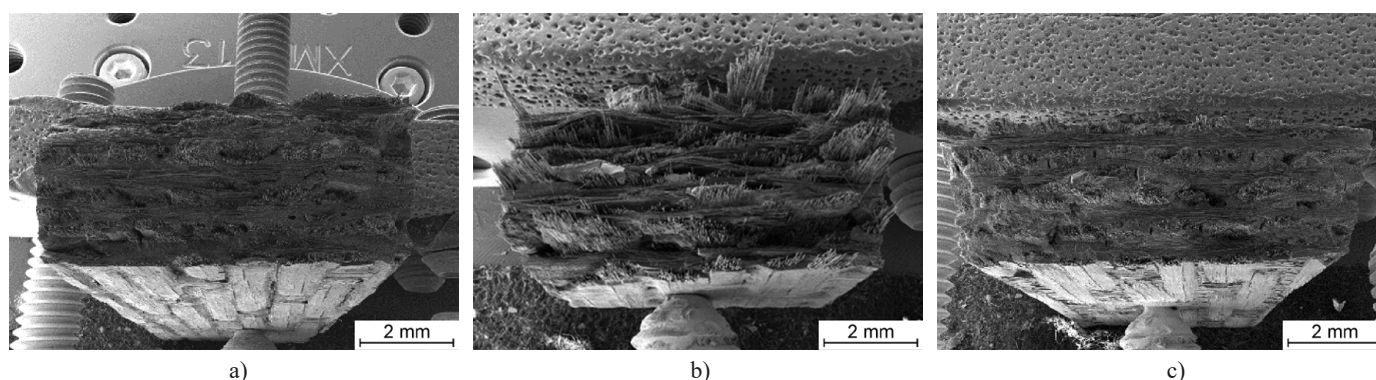


Fig. 5. Fracture surfaces of SiC/SiC composite subjected to a) tensile test at room temperature, b) creep test at 980°C and c) creep test at 1100°C

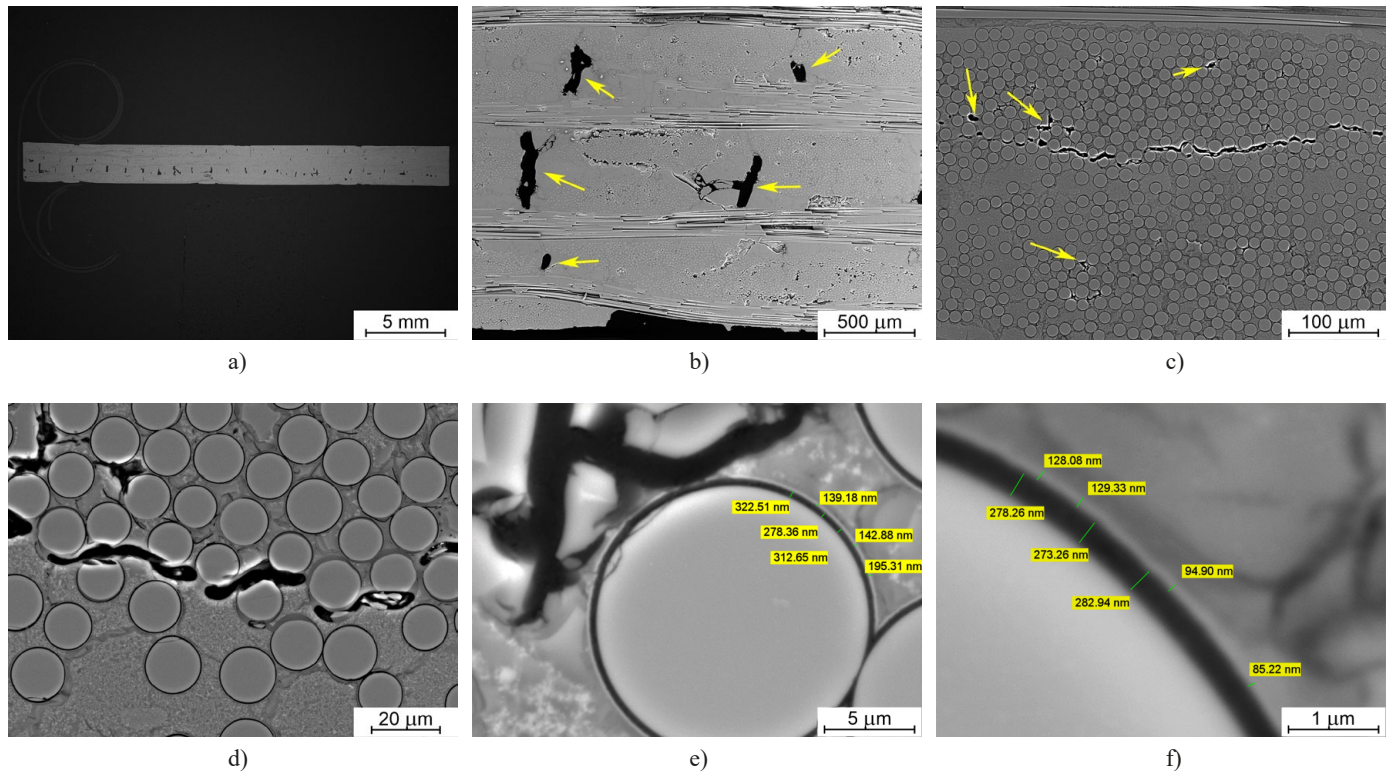


Fig. 6. SEM pictures of S200H after creep test in 980°C a) full cross-section, b) inter-laminar matrix pockets, c,d) open cracks, e,f) Si₃N₄ and BN coating

and 7c,d). The cracks predominantly propagate along the fiber direction, following the architecture of the woven 8HS composite. This orientation suggests that the matrix is particularly

susceptible to thermomechanical mismatch stresses along the fiber axis, possibly exacerbated by residual stresses from the PIP process and repeated thermal cycling.

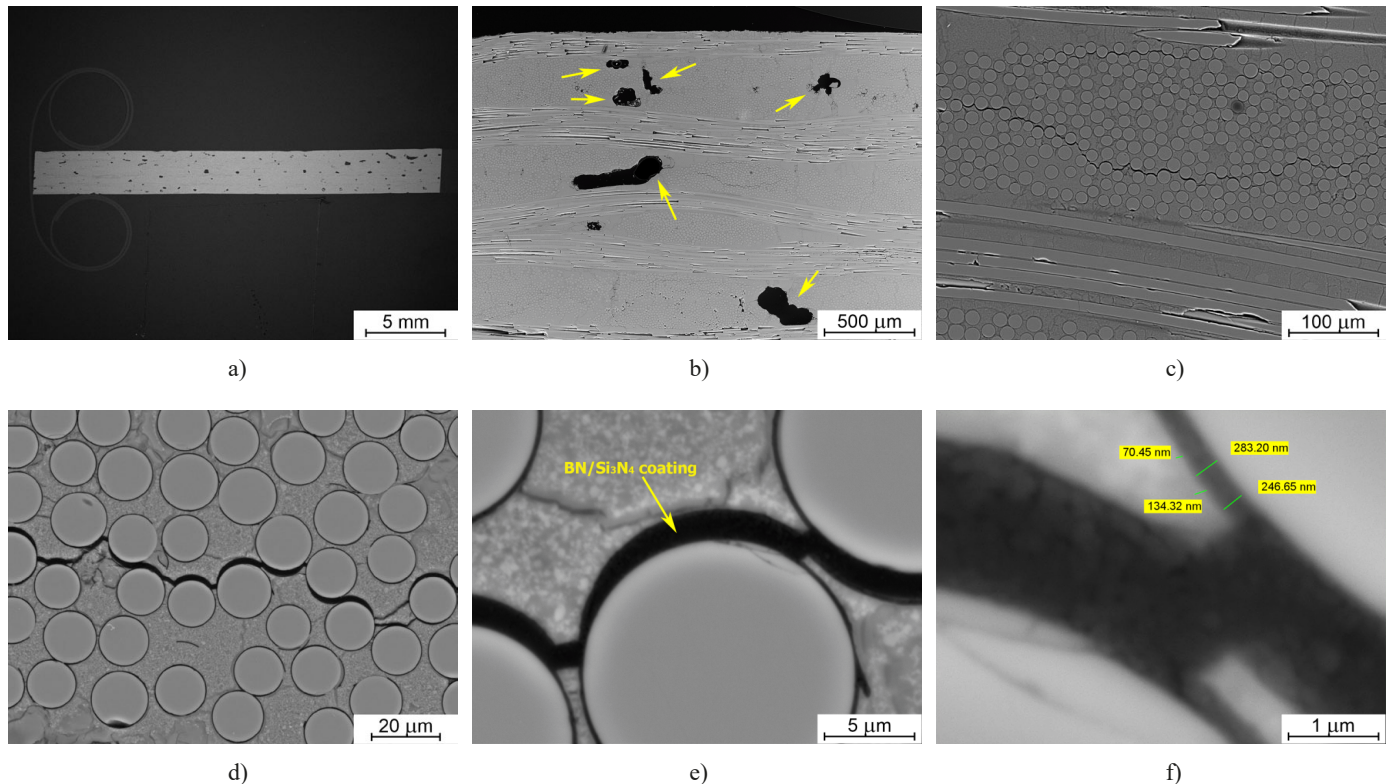


Fig. 7. SEM pictures of S200H after creep test in 1100°C a) full cross-section, b) inter-laminar matrix pockets, c,d) open cracks, e,f) Si₃N₄ and BN coating

In the specimens tested at 980°C, the matrix cracks were relatively narrow and did not appear to compromise the integrity of the fiber coatings. The BN/Si₃N₄ interphase remained intact, and the cracks propagated adjacent to, but not through, the fiber coatings (Fig. 6e,f). Boron nitride is still visibly present and measurable, indicating that the fibers retain their properties (Fig. 6e,f). The coatings on the Si₃N₄ and BN fibers had thicknesses of approximately 300 nm and 100 nm, respectively (Figs. 6e,f and 7f)). This behaviour indicates that the interphase retained its protective function, effectively decoupling the fibers from the matrix and limiting the extent of oxidative damage.

In contrast, the specimens subjected to creep at 1100°C exhibited more severe matrix degradation. The cracks were wider, more continuous (Fig. 7c,d), and in several cases, intersected the fiber coatings. SEM images revealed instances of coating delamination and partial removal, particularly in regions where the matrix crack intersected the fiber-matrix interface (Fig. 7e)). These observations suggest that at 1100°C, the oxidation kinetics are sufficient to degrade the Si₃N₄ and BN layers, especially in areas exposed to oxygen ingress through open cracks. The delaminated coatings are likely the result of a combination of mechanical separation due to crack propagation and chemical degradation due to oxidation. The formation of low-viscosity

borosilicate glass or volatilization of boron-containing species may further weaken the interfacial region, promoting coating detachment [17]. In some areas, remnants of the BN layer were observed displaced from the fiber surface, indicating that the interphase was no longer mechanically or chemically bonded to the fiber. The observed fracture behavior was primarily governed by the lower strength of the matrix, which led to crack initiation and propagation along the fiber direction. This is attributed to the fact that the matrix, unlike the continuous SiC fibers, typically contains more defects and has lower mechanical integrity, especially under thermal and oxidative stress.

EDS analysis showed oxidation of the SiC/SiC composite matrix after creep testing at both 980°C and 1100°C (Figs. 8 and 9). The fibers, however, did not undergo oxidation. The microstructure of the BN interphase did not change after the creep test and show a similar morphology to the one observed for the samples with that were not subjected to creep testing. The EDS analysis indicated some oxygen content in the BN interphase. However, similar levels oxygen are also found in the as-received SiC/SiC samples and thus they are not clear evidence of oxidation during the creep testing [16]. The elemental distribution maps shows oxidation of the matrix for S200H material. These observations are consistent with the retention of the mechanical properties after creep testing in 980°C and 1100°C.

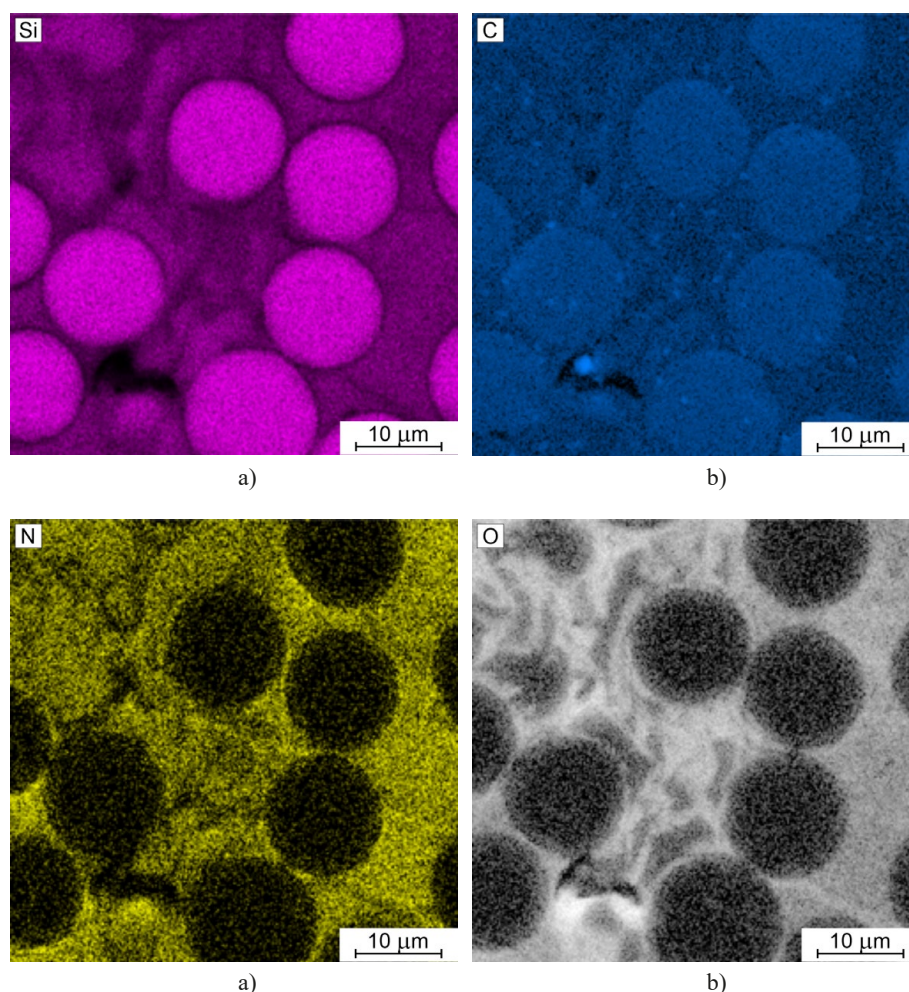


Fig. 8. EDS elemental mapping of S200H after creep test at 980°C a) Si, b) C, c) N, d) O

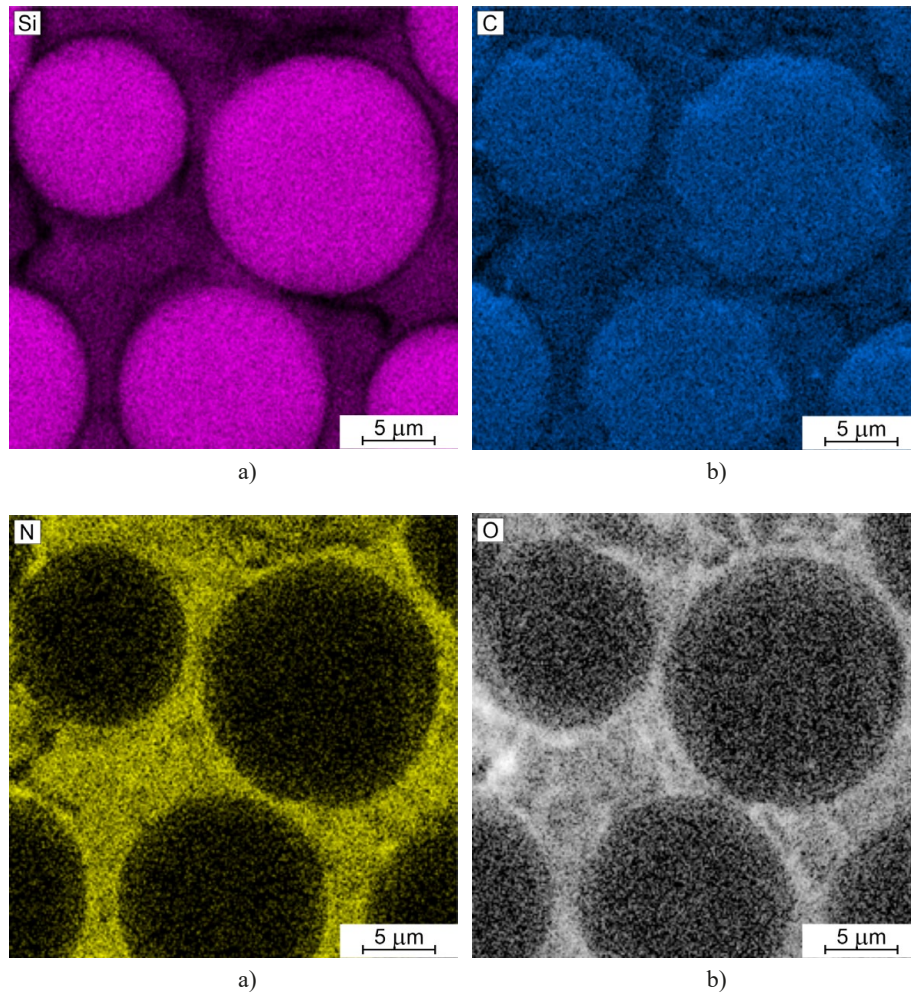


Fig. 9. EDS elemental mapping of S200H after creep test at 1100°C a) Si, b) C, c) N, d) O

4. Conclusions

In this study, the effect of high temperature creep on microstructure and durability of the S200H SiC/SiC composite is presented. Based on the conducted research, the following conclusions can be formulated:

- All samples tested at 100 MPa (4 at 980°C and 4 at 1100°C) ran out at 200 hours. At both temperatures the loading strain was similar, but samples tested at higher temperatures demonstrated three times higher creep strain during the creep testing. The behaviour of individual samples in each test group was consistent, particularly in terms of creep strain progression and stability under constant load.
- The modulus of elasticity determined from the loading data was lower than that obtained from tensile tests, which may be attributed to differences in loading rates. A higher loading rate in tensile tests resulted in higher Young's modulus, indicating that fiber creep plays a significant role in the mechanical behavior of the material.
- For specimens that reached 200 hours of testing, a general tendency toward stiffness reduction was observed, regardless of the temperature. This decrease is most likely due to material oxidation and the development of microcracks in the matrix, leading to degradation of the composite's mechanical properties.
- Specimens tested at 1100°C under 150 MPa run out at 200 hours. The values of loading, creep and total strain for both samples were similar. The total strain that the samples achieved at the end of the test was about 0.6%.
- For samples tested at 200 MPa (4 at 980°C and 2 at 1100°C), the loading strain was slightly higher at higher temperature. Failure of the specimens tested at 980°C occurred over a broad time range of 33 to 106 hours, indicating variability in individual sample performance. The two specimens tested at 1100°C failed in less than one hour. It is assumed that the load of 200 MPa exceeds the matrix cracking strength of S200H material.
- At 1100°C, a large number of cracks in the matrix material was an easy way for oxygen penetration and, as a result, failure of the material.
- At 1100°C, the oxidation kinetics are sufficient to degrade the Si₃N₄ and BN layers, especially in areas exposed to oxygen ingress through open cracks.

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