INTRODUCTION

Fiber reinforced ceramic matrix composites (CMCs) are a class of materials which are quasi-ductile and have a high temperature mechanical stability.1 In such CMCs, the matrix is a brittle oxide or nonoxide ceramic and it is the fiber which is the main load bearing component which imparts strength and ductility to the material.1 However, CMCs with a pseudo-ductile matrix have not been explored till date. Of particular interest are hence MAX phases, being a class of materials which are damage tolerant and demonstrate pseudo-ductility both at high temperature.2 Hence, Ti3SiC2 which is one of the most studied MAX phases is used in the current work as the matrix material along with chopped silicon carbide fiber (SiCf) as the reinforcement.

The approach of Ti3SiC2-based composites to obtain a combination of properties at high temperature which pure Ti3SiC2 lacks, has been explored briefly in the past. The oxidation resistance of Ti3SiC2 is known to be poor above 1100°C, hence Zhou et al used Al to form a solid solution of Ti3SixAl1-xC2 which had poor mechanical properties at high temperature. This problem was addressed by the addition of SiC particles to fabricate a composite material which resulted in improved oxidation resistance and flexural strength at high temperature.3 The damage resistance along the grain boundary is also believed to be improved, implied by the enhanced contact damage resistance of Ti3SiC2/SiC composites.4 Wan et al improved the elastic modulus and fracture toughness by adding 30 vol.% of SiC particles to Ti3SiC2, however, the...
flexural strength was compromised by nearly 125 MPa as compared to the pure material at room temperature.\textsuperscript{5}

Typically structural materials that are to be used at high temperature under mechanical loads are evaluated with respect to their strength and their creep resistance.\textsuperscript{6} However, the creep resistance of MAX phases based composites is only studied to a limited extent. Radovic et al.\textsuperscript{7,8} investigated the tensile creep of fine and coarse grained \( \text{Ti}_3\text{SiC}_2 \) and concluded that the creep response is mainly governed by the build-up and relaxation of internal stresses. Zhen et al.\textsuperscript{9} assessed the compressive creep of \( \text{Ti}_3\text{SiC}_2 \) at 1100-1300°C. The creep response was mainly attributed to dislocation movement and the fine grained \( \text{Ti}_3\text{SiC}_2 \) had a higher creep resistance than its coarse grained counterparts. Lagos et al.\textsuperscript{10} reinforced \( \text{Ti}_3\text{SiC}_2 \) with carbon fibers but the resulting composite had a poor bending strength due to pores acting as flaws being a result of the high fiber content. Dash et al. reinforced \( \text{Ti}_3\text{SiC}_2 \) with SiC whiskers and thereby reduced the creep rates by two orders in magnitude.\textsuperscript{11} The present work presents an investigation of creep response of chopped SiC\( _f \) fibers reinforced \( \text{Ti}_3\text{SiC}_2 \) MAX phase composites for potential use in high temperature load bearing applications. The reinforcement of SiC\( _f \) is in a macroscale as compared to the SiC whisker reinforced \( \text{Ti}_3\text{SiC}_2 \) which is in microscale.

### EXPERIMENTAL

Short SiC fiber (diameter 10 \( \mu \text{m} \), length 1 mm, Hi-Nicalon, NGS Advanced Fibers) containing \( \text{Ti}_3\text{SiC}_2 \) based composites were fabricated by wet mixing of synthesized \( \text{Ti}_3\text{SiC}_2 \) MAX phase and de-sized chopped SiC\( _f \). The synthesis process of \( \text{Ti}_3\text{SiC}_2 \) was molten salt based with Ti/Si/Al/C in the ratio of 3/1/0.2/2 and is described in detail elsewhere.\textsuperscript{12,13} Al was used to enhance the purity of \( \text{Ti}_3\text{SiC}_2 \) phase during the molten salt shielded synthesis (MS\textsuperscript{3}). Since the sizing of SiC\( _f \) was polyvinyl alcohol, the de-sizing was performed by continuous stirring of chopped fibers in hot water (80°C) for 1 hour followed by filtration. The mixing of \( \text{Ti}_3\text{SiC}_2 \) and de-sized SiC\( _f \) was done by simultaneous ultra-sonication and high shear mixing for 30 minutes in ethanol. 10 vol.% of SiC\( _f \) was added to the \( \text{Ti}_3\text{SiC}_2 \) matrix. The slurry was then dried in a rotary evaporator at 60°C with continuous agitation to avoid segregation of SiC\( _f \) and \( \text{Ti}_3\text{SiC}_2 \) powder. The dried powder was loaded into a graphite die (inner diameter 20 mm) and a cylinder (\( \phi \) 20 mm, 10 mm height) was sintered at 1250°C with a holding time of 35 minutes and a uniaxial pressure of 80 MPa. The heating rate was 100 K/min and the load was applied once the peak temperature of 1250°C was reached. The sintered materials were annealed in argon atmosphere at 1500°C for 1 hour to induce grain growth of the matrix phase and further subjected to creep and microstructural investigation. The composites with 0 and 10 vol.% SiC\( _f \) and fine grained \( \text{Ti}_3\text{SiC}_2 \) matrix is referred to as TSCF and TSCF-10SiC\( _f \), respectively. The annealed (1500°C) counterparts are named as TSCD and TSCD-10SiC\( _f \).

The phase composition of the sintered composites was identified by X-ray diffraction (XRD, D4 Bruker). The microstructures of the polished composites were characterized using a scanning electron microscope (SEM, TM3000, Hitachi).

Cylindrical specimens with \( \phi \) 5 mm and a height of 10 mm were cut out by electro-discharge machining for the compressive creep testing. Creep tests were performed in air in the temperature range of 1100-1300°C with constant loads of 20-120 MPa. The samples were heated at a rate of 5 K/min to the testing temperature by a resistance heated furnace and the load was applied by an electromechanical machine (INSTRON 1362). The deformation was measured by an LVDT sensor (Solartron Metrology). The tested samples were polished for microstructural observation. The steady-state creep rate was determined according to the following equation:\textsuperscript{6}

\[
\dot{\varepsilon} = \dot{\varepsilon}_0 A \left( \frac{\sigma}{\sigma_o} \right)^n \exp \left( -\frac{Q}{RT} \right)
\]

where \( A, \sigma, n, \) and \( Q \) are, a dimensionless constant, applied stress, stress exponent and activation energy for creep, respectively, and \( \dot{\varepsilon}_0 = 1 \text{ s}^{-1}, \sigma_o = 1 \text{ MPa} \) were considered for defining the units.

Samples for post creep analysis using transmission electron microscopy (TEM) were prepared by Focused Ion Beam (FIB, Zeiss Crossbeam 540) equipped with Scanning Transmission Electron Microscopy detector (4-Channel aSTEM Detector).

### RESULTS AND DISCUSSION

The morphology of starting \( \text{Ti}_3\text{SiC}_2 \) powder and chopped SiC\( _f \) is shown in Figure 1A,B. The XRD (Figure 1C) shows a basal texture on the top surface is similar to that of monolithic material.\textsuperscript{11} The degree of basal plane alignment may have been hindered by the presence of chopped SiC\( _f \). The crystallinity of SiC\( _f \) fiber was not high enough to be detected by X-ray diffraction. The alignment of SiC fibers could not be concluded from XRD data. The successful fabrication of a composite depends on the interaction of the matrix and reinforcement and in the present case the XRD suggests the absence of any reaction between \( \text{Ti}_3\text{SiC}_2 \) and SiC\( _f \) due to the absence of any third phase apart from TiC which forms due to decomposition of \( \text{Ti}_3\text{SiC}_2 \). The peaks identified majorly correspond to Ti3SiC2 and only the high intensity peaks are labeled. The absence of any reaction between SiC\( _f \) and \( \text{Ti}_3\text{SiC}_2 \) is further supported by Spencer et al.\textsuperscript{14} The XRD of TSC-20SiC\( _f \) was like TSC-10SiC\( _f \) and
Figure 2A,B illustrate polished cross-sections the perpendicular and parallel surface of sintered TSC-10SiCf, respectively. The SiCf are aligned in the x-y plane if z-axis is considered as the normal direction or the direction along which the uniaxial pressure is applied. The SiCf appear dark because of material contrast and there is no identifiable reaction layer between SiCf and Ti3SiC2. This is in accordance with the work of Spencer et al.14 The fibers are randomly oriented in the x-y plane with the axis of the fiber aligned along the plane. The length of the fibers is below 1 mm which suggests the possibility of fiber breakage during sintering. However, the degree of orientation of the fibers in the x-y plane is not high and this maybe the reason for the smaller size of fiber observed in Figure 2A. Figure 2B shows a cross-section with the fibers confirming the orientation of chopped SiCf. The density of the SiCf reinforced Ti3SiC2 CMCs were above 99% with no pores visible in the SEM of TSC-10SiCf. The sintered material is textured and has a very small grain size (1.2 ± 0.6 µm) because of fine starting powder and the type of sintering method employed for densification. The pressure applied during the sintering may have been responsible for aligning the platelets of the starting powder resulting in a strong basal texture of the perpendicular surface (Figure 2C) whereas the parallel surface (Figure 2D) has a mix of prismatic and pyramidal orientation. The microstructure (not shown here) of TSCD-10SiCf is duplex with elongated Ti3SiC2 grains of length 500 µm and thickness 50 µm in a matrix of fine grains similar to that of TSC-10SiCf.

Although, the strain vs time graphs presented in the following obviously contain both primary and secondary creep contributions; the discussion will only be based on the analysis of secondary creep. Where it should be noted that the obtained creep behavior is a result of behavior of fibers, matrix, and interfaces as well as possible anisotropies.

Figure 3A,B show the strain vs time graph for creep tests carried out at 1100°C. The addition of 10 vol.% of SiCf did not result in any significant increase in the creep resistance of TSCF at 1100°C, 40 MPa (Figure 3A). The red curves in Figure 3A are almost superimposable. Due to the high uncertainty at low creep rates conclusions on contributing mechanisms are limited, although the apparent small difference under this condition appear to suggest a similar mechanism under this particular temperature/stress combination (i.e., dislocation creep is only contributing factor). This is not to misunderstand that there is a transition in creep mechanisms beyond this point. It is only that other deformation mechanisms apart from dislocation-based deformation are activated stronger with the increase in temperature and applied mechanical stress, but final conclusions would require further experimental evidence based on post-creep microstructural analysis.

The addition of similar content of SiCf in TSCD resulted in a significant reduction of the deformation during creep at
1100°C, 40 MPa. Even at the end of the testing time the secondary creep stage just started leading to a potential overestimation and higher uncertainty in the obtained secondary creep rate results. The coarse grains might have facilitated efficient load transfer to the SiCf due to the ability to delaminate and being prone to bending. On the other side it should be noted that in a case like the one discussed above, where the creep rate of the matrix is low anyway any addition of a second phase with a low creep rate will lead to a less obvious effect than in a case like the TSCD where the matrix has a high creep rate.

However, at a higher stress 1100°C, 80 MPa, the effect of addition of SiCf to TSCF is more pronounced (Figure 3B), perhaps partly related to the effect stated above, ie that the addition of a material with low creep rate has a stronger effect onto a matrix material with a higher creep rate, that is, the creep rate of the matrix increased significantly at this higher applied stress. The presence of non-creeping SiCf may be responsible for ceasing of mechanism by which coarse grains of Ti₃SiC₂ are known to deform. However, the addition of 10 vol.% SiCf restricts the delamination in coarse grained Ti₃SiC₂ in the present case.

Figure 3C,D show the creep deformation curves of the composites obtained at 1200°C. The trends regarding deformation of monolithic fine and coarse grained Ti₃SiC₂ and the respective effect of the SiCf reinforcement are the same as those at 1100°C. At 1200°C the primary creep regime is shorter than at 1100°C. The effect of the SiCf addition for TSCF can already be clearly seen for this temperature at 40 MPa (Figure 3C). The addition of SiCf leads to a creep response of TSCD-10SiCf that almost agrees with that of the TSCF without fibers, hence increasing the grain size and addition of 10 vol.% SiCf lead to effects of similar magnitude that eliminate each other, although probably coincidental since in case of different grain sizes or different fiber contents the result will be different. This is due to the fact that finer grain size is more creep resistant in the case of MAX phases. It was shown by Zhen et al that coarse grained Ti₃SiC₂ are more prone to delamination and consequent creep. However, the addition of 10 vol.% SiCf restricts the delamination in coarse grained Ti₃SiC₂ in the present case.

Figure 4A shows that for most applied stresses and temperatures the creep rates of TSCF-10SiCf are nearly one order of magnitude lower than that of TSCF at 20 MPa, although the data obtained at 40 and 80 MPa indicated that both TSCF and TSCF-10SiCf seems to converge for the lower temperature range.

Figure 4B shows that the creep rate of TSCD-10SiCf is around two orders of magnitude lower than that of TSCD. The slope of fitted straight lines is different for TSCD and TSCD-10SiCf and hence yielding different corresponding apparent activation energies. However, note, looking at the data it becomes clear that if the lowest temperature data for TSCD are not considered, then the remaining data points for both variants indicate a much closer activation energy for the remaining temperature range where data are obtained for both, hence the derived activation energies in these cases appear to be also biased by different temperature ranges that resulted from experimental boundary conditions.

Figure 4C,D shows the ln-ln plot creep rates vs the applied stress. It can be concluded from these data that within the limits of experimental uncertainty the Ti₃SiC₂ grades, irrespective of the grain size and the presence of SiCf, have similar stress exponents over the whole temperature and stress range.
As can be seen in Figure 4C, the creep rates for TSCF and TSCF-10SiCf are similar for the entire tested temperature range. The addition of SiCf resulted in a slight decrease in the creep rates. The decrease in creep rate is more pronounced at 1300°C than at 1100°C.

Figure 4D reveals that the creep rates of TSCD are clearly reduced by the addition of SiCf, even at a temperature of 1100°C. Based on literature reports it can be suggested that this might be related to the plastic deformation of coarse grained Ti3SiC2 by delamination and kinking, whereas fine grained Ti3SiC2 needs to be above the apparent brittle-to-plastic transition temperature to demonstrate substantial plasticity.9,16,17

Since Al was used for the synthesis of Ti3SiC2 powder, the presence of alumina is also believed to have an influence on the creep behavior of the composites. The matrix of all the different grades of composites has similar quantities of alumina since the starting powder was the same. Although the presence of alumina has an effect of the mechanical response of Ti3SiC2,18,19 the difference in creep response of the different grades of composites is solely due to either the grain size of the matrix or the presence of SiC fiber.

The activation energies and stress exponents for creep of SiCf reinforced Ti3SiC2 are compared to monolithic Ti3SiC2 in Table 1. The activation energy of TSCF-10SiCf is in the same range as that of TSCF. The rate controlling mechanism for creep can hence also be assumed to be similar for the temperature range. TSCD-10SiCf has a visible increase in activation energy due to the presence of SiCf. This can be an evidence of an effective suppression of creep deformation of TSCD as compared to TSCF with the addition of SiCf.

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which is also the range for the fabricated composites in the present work, hence being indistinguishable. Since discontinuous SiCf were added, the creep in fibers may have been induced by shear strains induced by the deformation of the matrix. Hence, the stiff, brittle SiCf can also undergo fracture to accommodate any deformation induced by the matrix, which might be induced by shear deformation or grain delamination. A post-test microstructural investigation after creep testing was performed to aid the understanding of the deformation mechanisms of the composites.

Figure 5A illustrates multiple fractures in SiCf perpendicular to the axis of the fiber which were not observed before creep (Figure 2A). The crack may have originated from the compressive and shear strains due to the mass flow by the virtue of the large deformation of the matrix material in the lateral direction.

Since the fibers are a local reinforcement on a microstructural scale, the mechanisms leading to the increased creep
resistance should also be in a microscale. An obvious effect on the microscale will be the percolation of fibers, although an effect that could not be verified by experimental evidence in the current work. The discussion of percolation effects should aid the understanding of observed behavior, although it should be noted that even above percolation limit the fiber contact will not increase their load carrying ability significantly since they are not bonded to each other. Considering a homogeneous composite, the critical percolation limit volume fraction ($\phi_{pcp}$) of SiC$_f$ can be calculated by the following relation:

$$\phi_{pcp} = \frac{0.7}{\text{aspect ratio}}$$

(2)

$\phi_{pcp}$ is the limit above which fibers start to form an interconnected network in terms of at least one line of fibers in contact through the specimen. Since the materials are sintered under uniaxial pressure, there will be a lack of three-dimensional connectivity (percolation) of SiC$_f$ rather a presence of two-dimensional arrays of SiC$_f$ perpendicular to the uniaxial stress during sintering might be assumed. The derived $\phi_{pcp}$ for such materials can hence be larger by a factor of two since Equation (2) is strictly valid only for randomly oriented fibers.$^{21}$

Since the length and diameter of the SiC$_f$ used are 1 mm and 10 μm, respectively, the aspect ratio can be considered 100. Hence, at a volume fraction of 1.4 vol.% the percolated limit is exceeded, and a network begins to form. Some point contacts of fibers can be seen in Figure 5B. Moreover some fibers also appear to crack due to other fibers contacting each other, probably as a result of bending.

Figure 5C,D shows the post-creep microstructure of TSCD-10SiC$_f$. As compared to TSCF-10SiC$_f$, the cracks in SiC$_f$ are filled with matrix material in the case of TSCD-10SiC$_f$ (Figure 5C). This shows that due to creep the matrix material is displaced (Ti$_3$SiC$_2$) into the cracks of SiC$_f$, which is also a result of the stronger creep of the TSCD variant leading to a larger opening of cracks that are then easier to fill.

Both Figure 5C,D show higher amount of internal cracks as compared to post-creep microstructure of TSCF-10SiC$_f$ in Figure 5B. These interconnected cracks could be a result of grain boundary sliding (GBS) of the Ti$_3$SiC$_2$ matrix phase material followed by mostly elastic deformation of the SiC$_f$. The difference in deformation of matrix and fiber then either result in the fracture of SiC$_f$ or formation of larger cavities to accommodate the strain differences.$^{22}$ see also Figure 5C,D.

Circular cracks (red arrows) are visible around the SiC$_f$ in Figure 5D, it might be speculated that these maybe an indication of toughening by crack deviation and fiber de-bonding for TSCD-10SiC$_f$. Further in-depth studies are required to conclude on the effect of the presence of SiC$_f$ in coarse grained Ti$_3$SiC$_2$ on toughening of the composites.

Figure 6 shows the STEM image of the interface of SiC$_f$ and matrix material. The SiC$_f$ has an influence on the local alignment of the Ti$_3$SiC$_2$ which appears to orient in a radial manner around the SiC$_f$. The GBS along the SiC$_f$ and Ti$_3$SiC$_2$ grain boundary may have resulted in the formation of SiC needles shown in Figure 6B. This might result in an increased shear strength leading to the crushing of SiC$_f$ surface and Ti$_3$SiC$_2$ grains during relative GBS of Ti$_3$SiC$_2$ and SiC$_f$.$^{4}$ Whereas damage induced due to Ti$_3$SiC$_2$-Ti$_3$SiC$_2$ relative grain sliding might only be due to basal slip.
A material contrast is visible in the interface of the SiC\textsubscript{f} and Ti\textsubscript{3}SiC\textsubscript{2} in Figure 5B might indicate a reaction. As Ti\textsubscript{3}SiC\textsubscript{2} and SiC are known to be chemically stable with each other,\textsuperscript{14} the possibility of reaction can be ruled out. EDS investigation (Figure 7) performed on the interface shows that the interface is rich in Si as compared to the core of the SiC\textsubscript{f}. A slight oxygen concentration might suggest the oxidation of SiC\textsubscript{f} during high temperature creep. TSCD does not have any indication of reaction at the Ti\textsubscript{3}SiC\textsubscript{2}/SiC\textsubscript{f} interface because the time spent at high temperature was significantly less as it was more creeping.

4 | CONCLUSIONS

In summary, dense composites of SiC\textsubscript{f}/Ti\textsubscript{3}SiC\textsubscript{2} were fabricated with anisotropic fiber alignment. The creep resistance improved with the addition of SiC\textsubscript{f} although in general the creep response depends on the microstructure of the Ti\textsubscript{3}SiC\textsubscript{2} matrix. The activation energy for creep was increased by the addition of SiC\textsubscript{f} from 454 ± 29 to 576 ± 33 kJ/mol for coarse grained SiC\textsubscript{f}/Ti\textsubscript{3}SiC\textsubscript{2} composites, whereas it was unchanged for fine grained Ti\textsubscript{3}SiC\textsubscript{2} in the range of 650-700 kJ/mol. The fibers appeared to be well bonded so that the load was transferred from the matrix to the fiber during the creep as indicated by multiple fractures of the fibers. The overall creep mechanism can be attributed to dislocations and grain boundary sliding for fine grained SiC\textsubscript{f}/Ti\textsubscript{3}SiC\textsubscript{2} composites whereas added factors of delamination and grain bending can be considered for coarse grained counterparts. Fiber fracture was eminent in fine grained composites, while fiber fracture was accompanied with cavitation suggesting the higher plasticity of coarse grained Ti\textsubscript{3}SiC\textsubscript{2} matrix. The SiC\textsubscript{f} reinforcement
helps to maintain lower creep rates even at high temperature as it counteracts the creep rate with increasing grain size.

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