Tensile Properties and Microstructure of SiC Fibre Reinforced Multi Metal Matrix Composites

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Abstract

The matrix coated fibre production route is established for titanium matrix composites of highest quality. However, the commonly used method for consolidation (hot isostatic pressing) leads to shrinkage, distortion or even fibre fracture. Some fibre arrangements require consolidation without pressure. The process proposed in this paper uses low melting alloys to consolidate the composite material by infiltrating the matrix coated fibres. Tensile tests at room temperature have shown that these metal matrix composites may have the potential to compete with conventionally processed titanium matrix composites. No significant decrease in strength is expected up to 400°C. One important goal of the paper is to emphasize important points that have to be considered for the development of this new type of composite material.

1 Introduction

Titanium matrix composites (TMCs) are considered to be attractive candidate materials for structural components. TMCs show superior mechanical properties (such as tensile, creep and fatigue) compared to unreinforced titanium alloys [1]. However, the serial application of TMCs is still very limited due to cost restrictions and processing difficulties. Potential applications may be found in aircraft engines where high specific strength and stiffness are needed in combination with heat resistance. Both, the very fine microstructure of the matrix alloy as well as the fairly homogeneous fibre distribution are responsible for these exceptionally good properties [2]. Current processes are using unidirectional or hot isostatical pressing for the consolidation of the composite material [2], which is currently the best available technology. However, the pressing of the material leads to shrinkage of 10-15%, distortion of the geometry and, in the worst case, fibre breakage. To diminish these difficulties, a pressureless process has been taken into account for the production of SiC fibre reinforced metal matrix composites for high temperature applications. Especially for ring applications where the fibres are orientated in circumferential direction, a process without shrinkage and distortion offers many advantages. In some cases it is even difficult or impossible to limit the shrinkage to the radial direction of the fibres when conventional pressing techniques are used. For this reason, new consolidation methods without significant shrinkage would be purposeful, which can be realized by infiltration techniques. Metal matrix composites consolidated by infiltration are named multi metal matrix composite (MMMC or 3MC). The primary matrix (PM) corresponds to the titanium coatings on the fibres and cares for well-defined distances between the fibres. The secondary matrix (SM) develops during the infiltration process and fills the gaps between the coated fibres. One import framework requirement is, however, that the good material properties of TMCs can be maintained.

A major object of research is the selection of alloys for the infiltration processes. The first approach is to use filler metals commonly applied for brazing titanium alloys. There are different types of brazing solders described in literature [3]. One can distinguish silver based, aluminium based, titanium based and zirconium based brazing solders with considerably different material properties. Interdiffusion and intermetallic phase formation is also strongly influenced by the composition of the primary matrix. Therefore, pure titanium and Ti-6242 are used for comparison (see Table 2). A further important aspect that will be addressed in this paper is the influence of the fibre volume fraction (Figure 3).

2 Experimental

The tensile test specimens can be manufactured either by machining fibre reinforced rods (e.g. 12x80 mm with 3.5mm reinforced part) to tensile test specimens or by infiltrating samples already exhibiting the final geometry. In the latter case only the funnel has to be cut after infiltration. Both techniques lead to comparable tensile test specimens. The base fibre material was the SiC fibre SCS-6 from Specialty Materials Inc. with a diameter of 142 µm including an outer carbon coating of approx. 3µm. The coating materials used in the studies were either CP titanium grade 1 (Ti) or the near-α titanium alloy Ti-6Al-2Sn-4Zr-2Mo (Ti-6242). Before infiltration the coated fibres were put into the drilled holes of the specimens. The lower ends of the holes were closed by plugs, and the funnels at the upper ends of the vertically fixed specimens were charged with one of the low melting alloys specified in Table 1. A schematic representation of the manufacturing procedure is depicted in Figure 1.

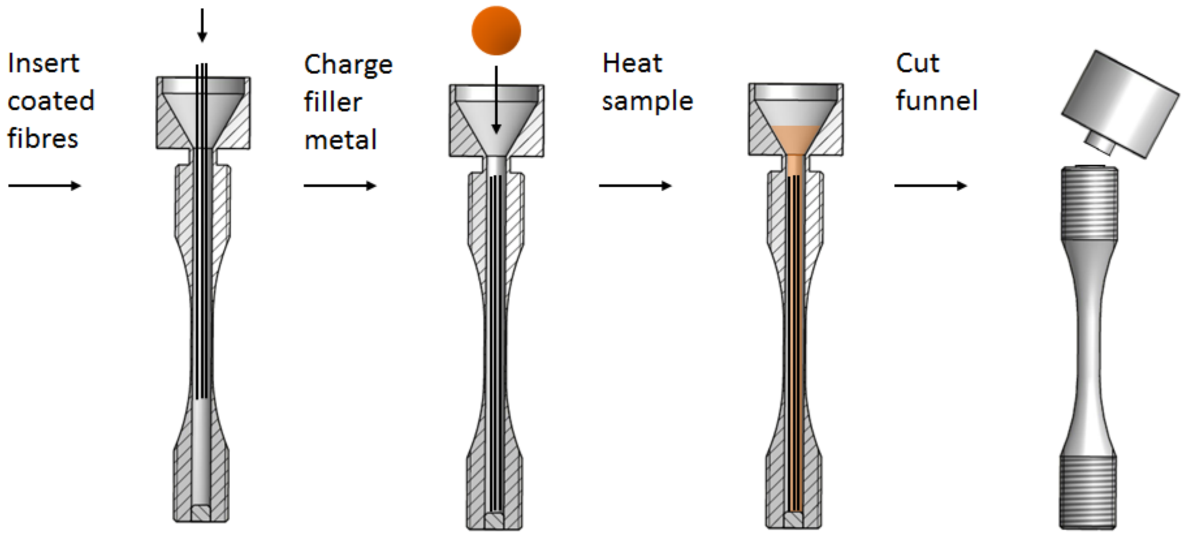

Figure 1. Schematic representation of infiltration process.

Table 1. Composition, melting range and working temperatures of filler metals used in the studies described in this paper.

Alloy	Composition [wt.-%]	Melting range [°C]	Working temperature [°C]	Remarks
AgCuSn	Ag60Cu30Sn10	600-730	740-780	Table 2
AgCu	Ag72Cu28	780[1]	800-820	Table 2
ZrTiNiCuAl	Zr63.6Ti3.3Ni8Cu21.7Al3.3	~860[2]	860-950	Table 2,3

[1]Eutectic point

[2]Liquidus temperature determined by own DSC measurement deviating from calculated values by Cao et al. [4].

The samples were placed in a high vacuum furnace and heated up to working temperature at pressures below 10^{-5} mbar. Depending on the alloy used holding times between 15 and 30 minutes proved to be suitable to infiltrate the fibres supported by gravity and capillary effect.
The specimens described above were tested by a servo-hydraulic tensile testing machine. Quasistatic tensile tests were carried out with cross head speeds of v_{CH} = 1 mm/min using clip gauges to measure elongation. Fractography and microanalytical investigations were carried out using SEM (Ultra 55, Zeiss) equipped with EDS. By this means the extend of diffusion and intermetallic phase formation could be recognized and different fracture types could be related to the microstructural constituents.

3 Results

3.1 Mechanical testing

The results of a material screening can be found in Table 2. The volume fractions of the components are distinguished to facilitate the comparison with further studies (fibre volume fraction: V_F, volume fraction of primary matrix: V_{PM}, volume fraction of secondary matrix: V_{SM}, volume fraction of containment: V_C).

Table 2. Screening test: Data of specimen (Study 1), process parameters and results of tensile tests.

PM	SM	T [°C]	t [min]	UTS [MPa]	ε_{max} [%]	E [GPa]	V_F [%]	V_{PM} [%]	V_{SM} [%]	V_C [%]
Ti	AgCuSn	740	15	1598	1,12	163	32	21	22	25
Ti-6242	AgCuSn	740	15	1728	1,20	168	31	22	24	23
Ti	AgCu	820	15	1654	1,11	171	33	21	22	24
Ti	AgCu	820	15	1590	1,07	175	32	21	26	21
Ti-6242	AgCu	820	15	1721	1,18	159	34	24	22	22
Ti-6242	AgCu	820	15	1778	1,22	164	32	23	25	20
Ti-6242	ZrTiCuNiAl	860	15	1696	1,05	196	33	24	19	24

The influence of the primary matrix on the ultimate tensile strength (UTS) is significant within this study. The UTS of samples with Ti-6242 is about 100-150 MPa higher than the UTS of samples with pure Ti primary matrix. But the strength of samples with pure titanium coating is remarkably high if we keep in mind that CP-Ti has a three to four times lower yield strength than Ti-6242. The effect of the secondary matrix cannot be reliably recognized because of the low number of samples. A strong statistical spread is not expected because the deviations of the results of equal samples infiltrated with AgCu are quite low.
A further point that can be derived from Table 2 is that comparable results can be achieved with Zr-based filler materials. Due to the fact that Zr-based brazing solders have been successfully used to produce high strength joints [5] the potential of this type of filler material for infiltration processes seems also promising. Therefore, the influence of the process parameters on the mechanical properties was investigated in a further study (Table 3).

Table 3. Data of specimen, process parameters and results of tensile tests (average of 3 specimens).

PM	SM	T [°C]	t [min]	UTS [MPa]	ε_{max} [%]	E GPa	V_F [%]	V_{PM} [%]	V_{SM} [%]	Batch
Ti-6242	ZrTiCuNiAl	880	30	1366	1,0	165	25	18	34	1
Ti-6242	ZrTiCuNiAl	910	15	1450[1]	-	-	30	22	25	1
Ti-6242	ZrTiCuNiAl	950	30	1022	0,9	143	25	18	34	1
Ti-6242	ZrTiCuNiAl	880	30	1575	1,1	158	28	20	29	2[2]

[1]Single value. Sample broken out of gauge length.
[2]Batch 2 contained Co impurities

Tensile strength and stiffness are lower in comparison to the results summarized in Table 2. This can at least partially be explained by lower fibre volume fractions. Due to the fact that the volume fraction of the primary matrix decreases together with V_F the amount of secondary matrix and its influence on the mechanical properties increases. This becomes more and more critical if there are fewer and fewer contact areas between the matrix coatings (Figure 4, right). Furthermore, there is a degradation of the fibre distribution. Higher infiltration temperatures (950 °C) were chosen to realize better homogenization by interdiffusion of PM and SM. The desired effect could not be observed; in fact the results were not satisfactory and less reproducible (see Figure 2). The UTS of specimens infiltrated at 880 °C for 30 min were almost as high and reproducible as the results of the TMC reference.

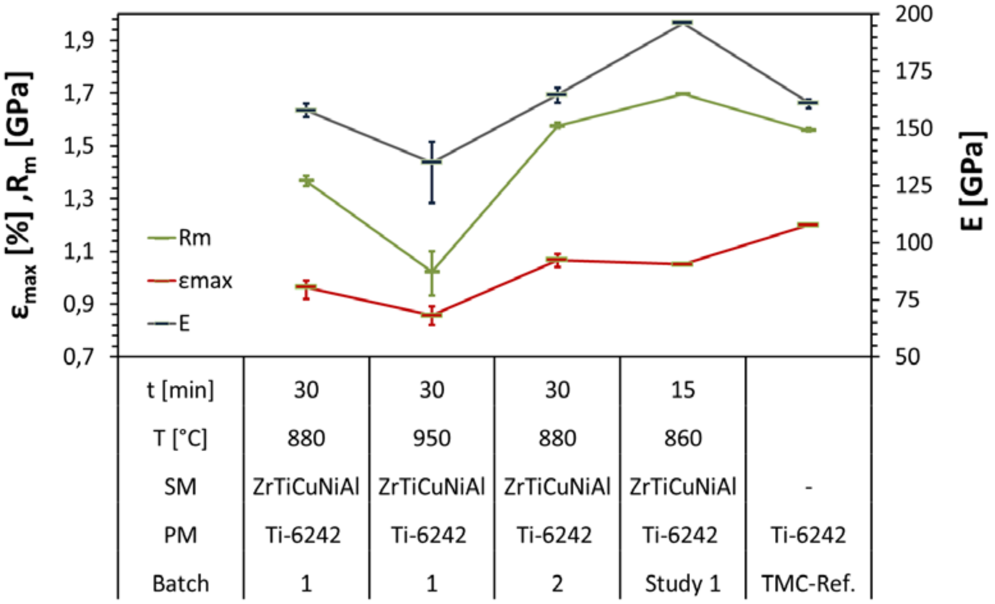

Figure 2. Graphical representation of results of tensile tests (cp. Table 3 and Table 2: one sample from Study 1).

The reason for the low performance of the samples infiltrated at 950 °C will be discussed together with the results of microstructural investigation (Section 3.2). A crucial factor that determines the material properties of TMC is the fibre volume fraction. Figure 3 demonstrates that the fact that the UTS can be increased with the V_F is also valid for 3MC.

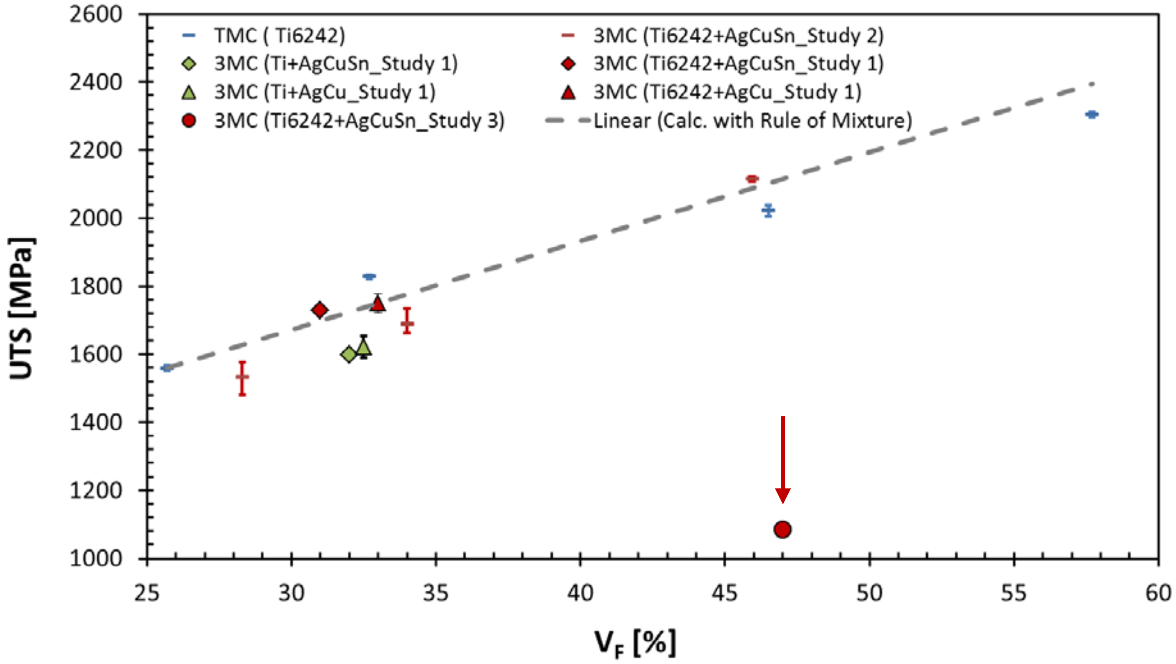

Figure 3. Fibre volume fraction calculated with UTS (fibre): 3500 MPa and yield stress (Ti-6242): 890 MPa compared with experimental results of TMC and 3MC.

In the systematic Study 2 the UTS of 3MC with fibre volume fractions between 25-35 % is about 100 MPa lower than that of conventional TMC. At higher volume fractions (V_F = 46 %) the results of TMC (~2000 MPa) and 3MC (~2100 MPa) are both close to the value calculated with the rule of mixture. However, it is important to emphasize the fact that a critical effect can be observed if the coating thicknesses of the matrix coated fibres are too thin. This is demonstrated in Figure 3 containing the results of samples with 5µm-thick titanium coatings (Study 3). Apparently, the critical value for high V_F is much lower for 3MC than for TMC.

3.2 Microanalytical investigation

The SEM pictures of samples infiltrated with AgCu (left) and ZrTiCuNiAl (right) are displayed in Figure 4. In both cases the microstructure of the Ti-6242 coating has changed because of the diffusion of elements from the filler metal into the primary matrix exceeding the solubility at room temperature. During cooling fine intermetallic precipitates form in the outer part of the titanium coating. Furthermore, in the case of AgCu, intermetallic layers of titanium and copper (e.g. TiCu, Ti_2Cu) form between secondary and primary matrix. The complexity increases if the filler contains further elements (e.g. the phase Ti_5Sn_3Cu in the experiments with AgCuSn). In the case of the Zr-based filler material large brittle intermetallic crystals adjoin the primary matrix. The rest of the secondary matrix generally consists of finer intermetallic components. A further detail that can be recognized from the right picture in Figure 4 is the fact that the titanium coatings of adjacent fibres grow together during the infiltration process.

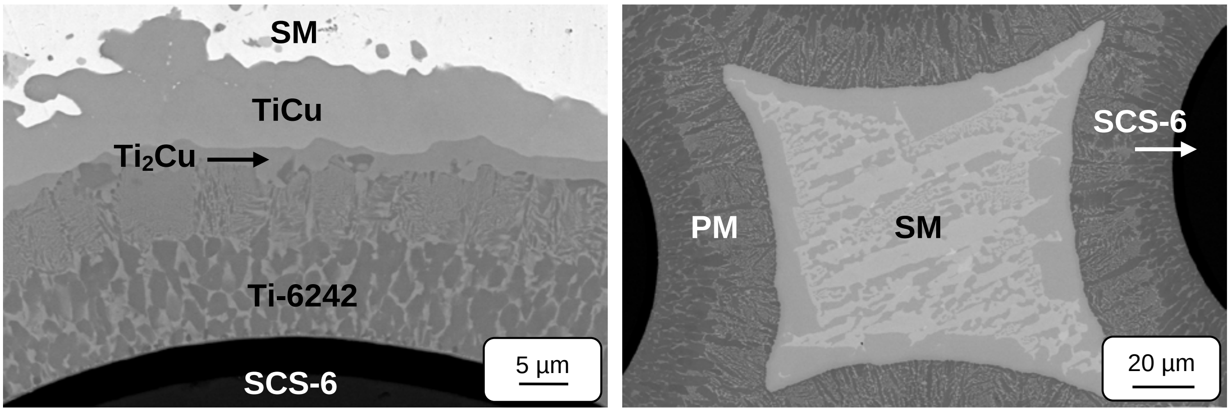

Figure 4. Microanalytical investigation. SEM micrograph of sample with PM: Ti-6242 and SM: AgCu (left). SEM micrograph of sample with PM: Ti-6242 and SM: ZrTiCuNiAl.

This phenomenon is even more pronounced in processes with ZrTiCuNiAl. As mentioned before, this coalescing of adjacent fibre coatings is considered to be important for the performance of 3MC.

3.3 Fractography

After the tensile tests the broken samples were investigated by SEM. Typical fracture surfaces can be found in Figure 5. A Similar behaviour was observed at samples infiltrated with AgCu or AgCuSn filler metal. The primary matrix consisting of Ti-6242 as well as the secondary matrix (AgCu) show ductile fractures (Figure 5, left). However, there are pronounced secondary cracks between PM and SM (red arrow). These cracks occur especially between the intermetallic phases TiCu and Ti_2Cu (see above Figure 4, left). In the case of samples that were infiltrated with ZrTiCuNiAl we can distinguish ductile fracture in the primary matrix and brittle fracture in the secondary matrix (Figure 5, right). Due to the fact that the secondary matrix consists of several intermetallic phases the fracture surface is not homogeneous. Differing from the samples infiltrated with AgCu there are no fractures in the transition zone of primary and secondary matrix.

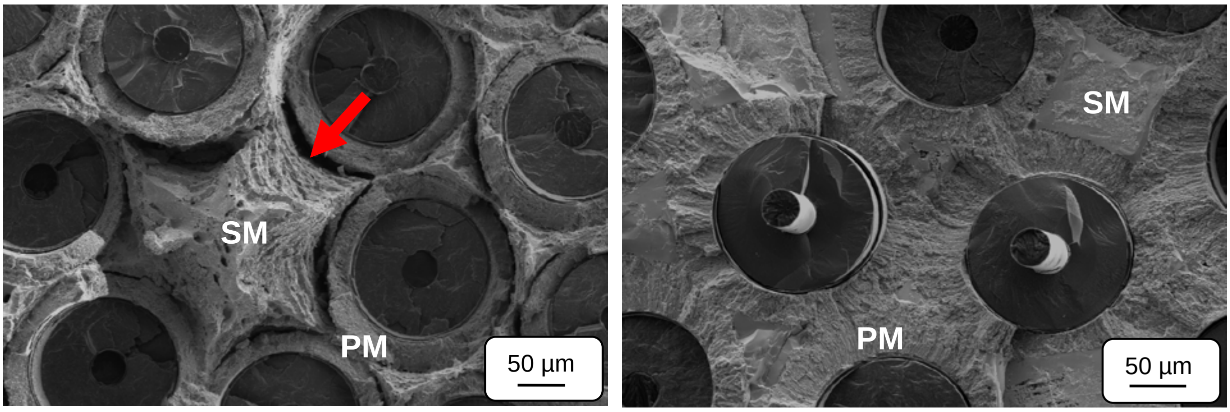

Figure 5. Fractographic investigation. Fracture surface of sample with PM: Ti-6242 and SM: AgCu (left). Facture of sample with PM: Ti-6242 and SM: ZrTiCuNiAl.

4 Conclusions

A new consolidation process for SiC fibre reinforced metal matrix composites has been evaluated. Multi metal matrix composites with various matrix systems have been investigated.

Tensile strength, stiffness and elongation at fracture are comparable with titanium matrix composites produced by hot isostatic pressing, which is remarkable because the strengths of the secondary matrices are significantly lower than that of titanium alloys like Ti-6-4 or Ti-6242. In most cases the strength of the 3MC produced by infiltration varies about 10% depending on the matrix system and the process parameters. The best results exhibited samples with a Ti-6242 primary matrix infiltrated with AgCu or AgCuSn (Table 2). This indicates that under the chosen conditions the effect of the secondary matrix is less significant. The statistical spread is quite low as can be seen in the diagrams in Figure 2 and Figure 3. However, strong degradations could be found if unsuitable conditions were chosen, namely too thin primary matrices (see Figure 3) and too high infiltration temperatures (Figure 3). The results seem promising, especially if we take into account that the material used in the studies described here was not perfect, having lower fibre volume fractions then theoretically possible. This leads to larger volume fractions of secondary matrix. Additionally, often pores were found.
Regarding the high temperature capabilities, not investigated yet, it must be mentioned that brazed joints made with the same filler material as the infiltration material AgCu showed no significant decrease in strength at tensile tests up to 400 °C. Therefore, good properties of the MMC concept presented here can be expected at least up to 400 °C. Considering literature [6, 7] and the own results we have to deal with interdiffusion between base metal and filler metal as well as phase formation. Brittle intermetallic phases can be an indication for poor fatigue properties. Further studies are required to focus on the fatigue behaviour and the possibilities to reduce or eliminate the formation of brittle intermetallics.

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