Effect of local reinforcement particle distribution on mechanical properties of in-situ TiB/TiC\textsubscript{1-x} particle reinforced Ti-5Al-5Mo-5V-3Cr – comparison between model and experiment

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Abstract. In-situ TiB and TiC\textsubscript{1-x} particle-reinforced titanium matrix composites (TMCs) based on a near-\textbeta Ti-5Al-5Mo-5V-3Cr alloy (Ti-5553) reacting chemically with 4 vol.-% B\textsubscript{4}C were processed by spark plasma sintering (SPS). Different local distributions of the reinforcement particles formed within the composite were adjusted by varying the conditions during powder preparation. The measured Young’s modulus, hardness and compressive yield strength of the composites increased, as the reinforcement particles were distributed more homogeneously within the matrix. In addition, Young’s modulus and hardness were modelled considering the hybrid TiB-whisker and TiC-particle reinforcement applying the rule of hybrid mixtures (RoHM) and the Fu-method, respectively. The compressive yield strength was modelled in accordance with the summation of the particular effective strengthening mechanisms and the Clyne-method. Measured values of the Young’s modulus and the hardness were overestimated by the modelling as the clustering of the reinforcement particles increased. The compressive yield strength of the composites is modelled appropriately using the Clyne-method. However, pronounced agglomeration of the reinforcing particles within the matrix resulted in an overestimation of the measured values.

1. Introduction

Owing to the low density of titanium, ceramic particle reinforced titanium matrix composites (TMCs) provide a combination of outstanding specific properties, such as high specific strength, specific stiffness, and hardness with heat- and/or corrosion resistance [1–4]. However, the strength of these composites is limited by the comparatively low strength of the pure titanium matrix. A considerable increase in strength of the composite would be possible, if the pure titanium matrix was replaced by a high strength titanium alloy. In this context, the high strength near-\textbeta alloy Ti-5Al-5Mo-5V-Cr (Ti-5553) appears to be a suitable matrix material. In Ti-5553, the high-temperature modification (\textbeta-phase) is stable up to ambient temperature. The high strength results from the solution strengthening effect of the alloying elements in addition with a specific heat treatment including solution treatment and aging (STA) leading to a \textbeta-microstructure with small dispersed \alpha-precipitations [5–10].

According to the literature it is well established that most effective strengthening in particle reinforced TMCs is achieved by titanium carbide particles TiC\textsubscript{p} and whisker-shaped titanium boride TiB\textsubscript{w}.
[11–18]. By means of powder metallurgy processes it is possible to create such ceramic reinforcement particles in-situ within the matrix during the TMC-synthesis. The advantage of this in-situ method is that the reinforcement’s surfaces are free of contaminations like oxygen or nitrogen. Consequently, a stronger bonding of the particle/matrix interfaces is facilitated [11]. Titanium exhibits high chemical reactivities with boron and carbon. Hence, B₄C-powder can be used as reactive powder for the in-situ formation process of the TiC- and TiB-reinforcing particles together with titanium [16]. The solid-state reaction behavior is based on a variety of complex diffusion-controlled processes between the titanium and the B₄C-particles occurring at elevated temperatures up to 700–1200 °C (sometimes even higher). Detailed information concerning the thermodynamics and the kinetics of the solid-state reaction between pure titanium and B₄C are given in references [19–24]. Among others, the spark plasma sintering is a common fabrication technique for such TMCs. Unlike described in the literature, own studies have demonstrated the necessity of considering the real stoichiometrically composition of the TiC₁₋ₓ particles formed during the in-situ reaction with B₄C, see reaction equation (1) below [25].

\[
(x + 6)Ti + B_4C \rightarrow xTi + 4TiB + 2TiC_{0.5}
\]  

(1)

Furthermore, in the case of Ti-5553 the solid-state reaction behavior is strongly influenced by the matrix alloying elements, resulting in a more sluggish reaction kinetics compared to the reaction with pure titanium [26]. In addition, it could be shown that the largest diameter of completely transformable B₄C clusters was limited to ~ 10 µm, if the sintering process was conducted at 1200 °C with a dwell time of 15 minutes. Consequently, the powder preparation conditions strongly influence the final degree of solid-state reaction between titanium/Ti-5553 and B₄C by controlling the formation or prevention of undesirably large B₄C clusters in the initial powder mixtures [25].

Beside the degree of B₄C conversion, the powder preparation conditions significantly affect the local distribution of the in-situ formed reinforcement particles within the TMCs as well. Low energy powder milling in conjunction with the sluggish reaction kinetics cause the in-situ formation of reinforcement particles predominantly in the vicinity of the former matrix particle surfaces. This particle clustering corresponds to an inhomogeneous local distribution of the reinforcement particles [25, 26].

Generally, the simultaneous use of two different ceramic phases (TiCₓ and TiBₓ) within the metal matrix is designated as hybrid particle reinforcement. However, the mechanical properties of such composites, e.g. hybrid Young’s modulus, hybrid hardness and hybrid strength can hardly be predicted by material models typically including only one single reinforcement phase, its phase fraction and sometimes its morphology. Relevant models in this context concerning particle reinforced composites are the Hashin-Shtrikman-model [27, 28] or the Halpin-Tsai-model [11] (Young’s modulus), the Rice-model [29] or the Halpin-Tsai-model [30] (hardness) and the summation of the contributions of particular strengthening mechanism [31–35] and the Clyne-method [31, 36, 37] (strength). Fu et al. introduce two approaches, which enable the prediction of the Young’s modulus for hybrid particle/short-fiber reinforced polymer matrix composites: The Rule of Hybrid Mixtures (RoHM) and a modified laminate analogy approach – hereinafter referred to as the Fu-method [38]. The RoHM describes the hybrid Young’s modulus of the composite \( E_c \) resulting from the two separate subsystems particle/matrix and short-fiber/matrix, see Figure 1. \( E_c \) is calculated according to the RoHM in the following way

\[
E_c = E_{c1}V_{c1} + E_{c2}V_{c2}
\]  

(2)

\( E_{c1}, E_{c2} \) and \( V_{c1}, V_{c2} \) are the Young’s moduli and the relative hybrid volume fractions of the systems particle/matrix and short-fiber/matrix, respectively. \( E_{c1} \) and \( E_{c2} \) have to be calculated applying suitable models for the particular subsystems, each including only one single reinforcement phase. Note that the total reinforcement volume fraction \( V_r = V_p + V_f \) (\( V_p \) and \( V_f \) are volume fractions of the particles and short-fibers, respectively) should be used as reinforcement volume fraction for calculation of \( E_{c1} \) and
Finally, the relative hybrid volume fractions in equation (2) are defined as 

\[ E_{c2} = \frac{V_p}{V_t} \]

and 

\[ E_{c2} = \frac{V_f}{V_t}. \]

Figure 1. Application of the Rule of Hybrid Mixtures (RoHM) to a particle and short-fiber reinforced composite (schematically) according to Fu et al. [38].

The Fu-method is a two-step procedure. In a first step, the particle reinforced matrix is defined as a new phase – referred to as the effective matrix. Within the model, this effective matrix is reinforced by the short-fibers in a second step, see Figure 2. Both steps allow the application of material models for composites with a single reinforcement phase. In contrast to the RoHM, the calculation of the Young’s modulus of the effective matrix \( E_{m,eff} \) in the system particle/matrix is based on the relative volume fractions of the matrix \( V_{m,rel} \) and the reinforcing particles \( V_{p,rel} \). These are defined as 

\[ V_{m,rel} = \frac{V_m}{(V_m + V_p)} \]

and 

\[ V_{p,rel} = \frac{V_f}{(V_m + V_f)}, \]

where \( V_m \) and \( V_f \) represent the volume fractions of the matrix and the reinforcing particles, respectively.

Figure 2. Application of the Fu-method to a particle and short-fiber reinforced composite (schematically) according to Fu et al. [38].

The present study analyses the influence of the local reinforcement particle distribution within hybrid in-situ TiB/TiC particle-reinforced Ti-5Al-5Mo-5V-3Cr matrix composites on selected mechanical properties. Especially, with respect to improve the predictability of these properties, first investigations
focused on the comparison between the measured and the modelled values for the composite’s hybrid Young’s modulus, hardness and compressive yield strength. Suitable model approaches, which consider the hybrid particle reinforcement were applied to the TMC property calculations. Deviations between experiment and model were discussed including the role of the local distribution of the reinforcing particles within the TMCs.

2. Experimental and materials
In the present study, a Ti-5Al-5Mo-5V-3Cr powder (Ti-5553, TLS Technik GmbH & Co Spezialpulver KG, Germany) exhibiting a spherical particle morphology served as the matrix material. B₄C (abcr GmbH, Germany) with a significantly lower particle size was used as a reactive powder for the in-situ reinforcement of the matrix. The chemical compositions and particle size distributions of the initial powders are given in Table 1 and Table 2.

| Table 1. Measured chemical composition of the initial powders. |
|---------------------------------------------------------------|
| **Composition** | Ti | Al | Mo | V | Cr | Fe | O | N |
| **(wt.-%)** | bal. | 5.22 | 5.05 | 5.10 | 2.83 | 0.31 | 0.13 | < 0.01 |
| **B₄C** | bal. | 21.90 | 0.20 | 1.40 | 0.11 | 0.01 | < 0.01 |

| Table 2. Particle size distribution of the initial powders measured by laser diffraction particle size analysis. |
|---------------------------------------------------------------|
| **Values (µm)** | d₁₀ | d₅₀ | d₉₀ |
| **Ti-5553** | 22.59 | 33.36 | 47.83 |
| **B₄C** | 0.51 | 1.80 | 1.96 |

Powder mixtures of Ti-5553 and 4 vol.-% B₄C were prepared under high energy milling conditions to break up the large B₄C clusters existing in the initial B₄C-powder, see Table 3. The powders were either milled in a planetary ball mill (Pulverisette 6, Fritsch GmbH, Germany) at ambient conditions or by vibration milling in a cryogenic mill (CryoMill, Retsch GmbH, Germany), while the grinding bowl was permanently cooled from the outside by gaseous nitrogen.

| Table 3. Preparation conditions of the initial powder mixtures and TMC designation of the consolidated samples. |
|---------------------------------------------------------------|
| **Method** | **Ball material** | **Ball diameter** | **BPR** | **Milling time** | **Rotating Speed/Frequency** | **TMC Designation** |
| **Planetary ball milling** | Cr-steel (hardened) | 10 mm | 5:1 | 4 h | 250 rpm | TMC-A |
| **Cryogenic milling** | Cr-steel (hardened) | 10 mm | 5:1 | 0.5 h | 30 Hz | TMC-B |
The powder mixtures were consolidated by SPS in an FCT-HP D 25/2-2 apparatus (FCT Systeme GmbH, Germany). Cylindrical samples with a diameter of 20 mm and a final height of 6 mm were sintered under vacuum atmosphere. The powders were heated up to 1200 °C at a heating rate of 100 K/min and kept at this temperature for 15 minutes. Parallel to the heating, the pressure was increased from 16 MPa at 450 °C to 51 MPa at 1200 °C and maintained constant during the dwell time. Parallel to the uncontrolled cooling, the pressure was reduced to 16 MPa within 15 minutes.

The density of the samples was measured using the Archimedes method in distilled water. For the microstructural investigations, samples were mechanically ground and polished. This was followed by chemical-mechanical polishing and subsequent etching with Kroll’s reagent for 20–40 seconds. Matrix grain size determination and size measurements of the reinforcement particles required etching with Kroll’s reagent for a shorter time and subsequently thermal etching according to the procedure proposed by Vander Voort [39]. Phase analysis was performed by X-ray diffraction as described in an earlier study [25].

The Young’s modulus was determined by measurement of the longitudinal and transversal wave velocities, employing the acoustical logging method on the faceground compact samples using a Hitachi V-1565 Oscilloscope (Hitachi, Japan) in combination with a Panametrics Model 5800 computer-controlled pulser/receiver (Panametrics GmbH, Germany). Due to the narrow sample diameter, the running periods of the acoustic sound waves were measured three times with an accuracy of at least 0.01 µs at the same position at each sample. A micrometer screw was used to determine the sample thickness between the faceground surfaces with an accuracy of 0.001 mm at three different sample positions. Finally, the wave velocities for Young’s modulus calculation were derived from the mean values of the measured sound wave running periods and the mean sample thickness. The Vickers hardness was determined from at least five indentations introduced with an indentation load of 294.2 N. For the compression tests, cylindrical samples with equal diameters and heights of 4 mm were cut from the sintered bodies by means of electrical discharge machining. Quasi-static compression tests at a strain rate of 10^-3/s were carried out at room temperature using an MTS 810 servo-hydraulic testing machine (MTS, USA). The compressive strain was calculated from the actual compression of the specimen, measured by a displacement gage. At least three compression tests were carried out for each composite to improve the statistical validation of the measured values.

3. Results and discussion

Figure 3 shows a comparison of the initial powder mixtures and the microstructures of the sintered samples for each milling condition, respectively. Especially, during the high energy ball milling the matrix particles undergo a strong plastic deformation, which is associated by pressing the smaller B₄C-particles into the matrix particle surfaces. Moreover this, coarse Ti-5553/B₄C particle agglomerates were formed through an increasing cold welding of the deformed matrix particles. In contrast, less plastic deformation of the matrix particles is observed during the cryogenic milling. Nevertheless, the high energy impact successfully avoids the formation of coarse B₄C particle agglomerates. However, the individual B₄C particles rather adhere to the matrix particle surfaces than they were pressed into them.

The different morphologies of the initial powder mixtures strongly effect the local distribution of the reinforcing particles within the bulk TMCs. Obviously, the reinforcement particles in TMC-B were predominantly located in the vicinity of the former matrix particle surfaces, while less reinforcements were formed inside the matrix particle volume. This appearance is in agreement with the findings for a comparable powder mixture based on Ti-5553 and B₄C presented in an earlier study [26]. The microstructure of the TMC-A sample, shows a more homogeneously distribution of the reinforcement particles. This can be ascribed to the repeated deformation, fracture and cold welding of the matrix particles during powder preparation resulting in the formation of agglomerates with embedded B₄C particles.

A comparison of the mechanical properties between TMC-A and TMC-B is limited to the requirement of approximately equal phase contents with respect to the in-situ formed TiB- and TiC₀.₅-particles within the TMC samples. In other words, the completely solid-state reaction of the B₄C particles during
sintering has to be ensured, thus avoiding superimposed effects of different particle contents. Both composites exhibit almost the same density, see Table 4. This and the absence of unreacted B\(_4\)C clusters within the microstructure prove high degrees of B\(_4\)C conversion in the TMCs. The measured TiB- and TiC\(_{0.5}\) phase fractions only vary in the precision of the XRD-method resulting in almost identical total reinforcement particle volume fractions, designated as \(\Sigma(\text{TiB} + \text{TiC}_{0.5})\).

![Figure 3](image)

**Figure 3.** TMC-A and TMC-B – morphology of the initial powder mixtures (SEM-SE) and optical micrographs of the sintered composites showing different local distributions of the reinforcement particles.

| TMC | Density (g/cm\(^3\)) | Ti-bcc | Ti-hcp | TiB | TiC\(_{0.5}\) | \(\Sigma(\text{TiB} + \text{TiC}_{0.5})\) |
|-----|-----------------------|--------|--------|-----|-------------|------------------|
| TMC-A | 4.66 | 74 | 9 | 11 | 6 | 17 |
| TMC-B | 4.65 | 84 | – | 9 | 7 | 16 |

The measured phase fraction of \(\alpha\)-Ti (Ti-hcp) in TMC-A, may have resulted from multiple effects connected to the powder milling and sintering processes. Due to the high chemical affinity of titanium, oxygen and nitrogen, the repeated deformation and fracture of the matrix particles during the powder preparation in air atmosphere caused their contamination through interstitial solid solutioning, see Table
5. Oxygen and nitrogen have a strong α-phase stabilizing effect on titanium. The influence of carbon on the phase stability in titanium is similar to that of oxygen and nitrogen. However, solid solution of carbon within the matrix particles occurs first during the sintering process as a consequence of the $B_4C$-decomposition. Finally, an earlier study proved that the in-situ reaction with $B_4C$ effected a significant change in the chemical composition within the initial Ti-5553 particles, in particular leading to an increase of the aluminum concentration [25]. Next to the interstitials mentioned before, aluminum acts as a strong α-stabilizing element in titanium alloys as well. However, the matrix in TMC-B consisted of β-phase, only. Consequently, the β-phase destabilizing effects associated to the sintering process seem to have a much lower efficiency, than the powder contamination with oxygen and nitrogen.

Table 5. Oxygen- and nitrogen-concentrations within the Ti-5553 powder and the initial powder mixtures measured by hot gas extraction.

| Concentrations (wt.-%) | oxygen | nitrogen |
|------------------------|--------|----------|
| Powder: Ti-5553        | 0.130  | < 0.010  |
| Powder: TMC-A          | 0.503  | 0.250    |
| Powder: TMC-B          | 0.320  | 0.024    |

The measured microstructural parameters of the TMCs are given in Table 6. Identically sintered samples based on the unreinforced Ti-5553 powder reveal a β-phase grain size $d_0$ of 229 µm. Hence, the β-phase grain size $d_4$ within both composites was decreased in order of two magnitudes. The TiB-whiskers were characterized by their length $l$, diameter $d$ and aspect ratio $S$. Characteristically microstructure parameters of the plate-shaped TiC$_{0.5}$ particles are represented by their width $L_2$, thickness $t$ and aspect ratio $A = 2L_1/t$ including the particle length $2L_1$ according to the definition of Nardone and Prewo [40].

Table 6. Measured microstructural parameters of the TMCs.

| TMC      | β-matrix | TiB       | TiC$_{0.5}$ |
|----------|----------|-----------|-------------|
|          | $d_1$ (µm) | $l$ (µm) | $d$ (µm) | $S$ | $L_2$ (µm) | $t$ (µm) | $A$ |
| TMC-A    | 4.8      | 15.4      | 1.3       | 13   | 2.4       | 1.6      | 1.7 |
| TMC-B    | 7.2      | 13.1      | 1.4       | 10   | 2.5       | 1.6      | 1.6 |

The TMC’s mechanical properties Young’s modulus, hardness und compressive yield strength increase with a more homogeneously distribution of the reinforcement particles, see Table 7. A Comparison of the compressive behavior is given by the compressive stress-strain curves in Figure 4.

Table 7. Measured hybrid Young’s modulus $E_c$, hybrid hardness $H_c$ and hybrid compressive yield strength at 0.2 % plastic deformation $\sigma_c$ of the TMCs.

| TMC     | $E_c$ (GPa) | $H_c$ (HV 30) | $\sigma_c$ (MPa) |
|---------|-------------|---------------|------------------|
| TMC-A   | 125         | 527 ± 13      | 1546 ± 14        |
| TMC-B   | 110         | 377 ± 8       | 1245 ± 19        |
Figure 4. Typical compressive stress–strain curves of TMCs.

Modelling of TMC’s mechanical properties was based on measured phase compositions and microstructure parameters. The partial destabilization of the β-phase in the matrix of TMC-A was considered for the matrix property calculations by applying a linear rule of mixture using the relative volume fractions of the α- and β-phase, respectively. This also includes the Hall-Petch strengthening effect due to the reduced β-grain size within both composites. Mechanical properties of the individual phases were taken from the literature (if available) or had to be determined in additional experiments. Input variables for the hybrid Young’s moduli and the hybrid hardness modelling are summarized in Table 8.

Table 8. Input variables for modelling of the hybrid Young’s modulus and hybrid hardness.

| Phase    | $E$ (GPa)  | Hardness (HV 30) | References |
|----------|------------|------------------|------------|
| Ti-bcc (Ti-5553) | 86   | $HV = 278HV + \frac{0.22HV\sqrt{m}}{\sqrt{d}}$ measured |
| Ti-hcp (Ti-5553) | 110  | 390   | [10, 41]   |
| TiB      | 450        | 1800             | [42, 43]   |
| TiC$_{0.5}$ | 265       | 2000             | [43, 44]   |

The Young’s moduli and the hardness of the subsystems TiC$_p$/matrix and TiB$_w$/matrix (RoHM) as well as the effective matrix and their reinforcement by TiB$_w$ (Fu-method) were calculated according to the Halpin-Tsai equations [11, 30, 45], thus considering the microstructural morphology of the individual reinforcement phases. Another fact that could not be ignored was the misorientation between TiB-whiskers and the load direction. The Halpin-Tsai equations are applicable just for short-fibres and whiskers, that are aligned parallel to the load line. Ryu et al. found, that the whisker orientation influence can be expressed in form of an effective aspect ratio $S_{eff,F(\theta)}$ considering the probability density function of misaligned whiskers $F(\theta)$, where $\theta$ is the misorientation angle [46]. $S_{eff,F(\theta)}$ is calculated as below:

$$S_{eff,F(\theta)} = \int_{0}^{90^\circ} \left[ S \cos^2(\theta) + \left(\frac{3\pi-4}{3\pi}\right) \left(1 + \frac{1}{S}\right) \cos^2(\theta) \right] F(\theta) d\theta$$  

(3)
For randomly aligned whiskers – like in the present TMCs – the probability density function is reduced to
\[ F(\theta) = \frac{2}{\pi} \] \[ [47]. \] A comparison between measured and modelled values is given in Table 9. Measured values of \( E_c \) and \( H_c \) were overestimated by the RoHM and the Fu-method, respectively. However, this overestimation increases significantly, as the local distribution of the reinforcement particles within the matrix becomes more inhomogeneous.

**Table 9.** Comparison of the measured and the modelled hybrid Young’s moduli \( E_c \) and hybrid hardness \( H_c \) (differences between model and experiment expressed as \( \Delta_{Mod-Exp} \)).

| TMC     | Hybrid Young’s modulus \( E_c \) (GPa) | Hybrid hardness \( H_c \) (HV 30) |
|---------|---------------------------------------|-----------------------------------|
|         | Experiment | RoHM \( (\Delta_{Mod-Exp}) \) | Fu-method \( (\Delta_{Mod-Exp}) \) | Experiment | RoHM \( (\Delta_{Mod-Exp}) \) | Fu-method \( (\Delta_{Mod-Exp}) \) |
| TMC-A   | 125        | 128 (+3)                       | 129 (+4)                       | 527 ± 13   | 561 (+34)                  | 562 (+35)                  |
| TMC-B   | 110        | 119 (+9)                       | 121 (+11)                      | 377 ± 8    | 520 (+143)                 | 522 (+145)                 |

A great plenty of effective strengthening mechanism could be identified in the present composites. This includes the grain boundary strengthening (Hall-Petch-effect) of the \( \beta \)-matrix grains \( \Delta \sigma_{H-P} \), the solid solution strengthening by interstitials (oxygen, nitrogen and carbon) \( \Delta \sigma_{ss} \), the load-transfer strengthening \( \Delta \sigma_{L-T} \) and the Orowan strengthening \( \Delta \sigma_{OR} \) of both, carbides and whiskers. In TMC-A the \( \beta \rightarrow \alpha \) phase transformation resulted in an increased matrix strength \( \sigma_{m0} \) by the amount of \( \Delta \sigma_{phase} \). A detailed description of the single calculation steps in determining the strengthening contributions is given by Grützner in [48]. The modelled values are listed in Table 10.

**Table 10.** Modelled strengthening contributions identified in the TMCs.

| TMC     | \( \Delta \sigma_{Phase} \) | \( \sigma_{m0} \) | \( \Delta \sigma_{H-P} \) | \( \Delta \sigma_{ss} \) | \( \Delta \sigma_{L-T(TiB)} \) | \( \Delta \sigma_{L-T(TiC)} \) | \( \Delta \sigma_{OR(TiB)} \) | \( \Delta \sigma_{OR(TiC)} \) |
|---------|----------------------------|-----------------|----------------|----------------|----------------------------|----------------------------|----------------|----------------|
| TMC-A   | 45                        | 965             | 451            | 178           | 358                       | 28                        | 14             | 12             |
| TMC-B   | 0                         | 920             | 400            | 114           | 237                       | 28                        | 12             | 14             |

As mentioned in the introduction, two distinct approaches are suggested in the literature, which enable the strength modelling of particle reinforced composites. One is the summation-approach, where the single contributions of particular strengthening mechanism are added to \( \sigma_{m0} \). According to the Clyne-method the final strength of a composite is calculated by summing the root of squares of all the individual strengthening contributions, as below

\[
\sigma_c = \sigma_{m0} + \sqrt{\sum \Delta \sigma_i^2} = \sigma_{m0} + \sqrt{\Delta \sigma_{H-P}^2 + \Delta \sigma_{L-T}^2 + \Delta \sigma_{ss}^2 + \Delta \sigma_{OR}^2 + \cdots} \]  \[ (4) \]

Table 11 shows a comparison of the modelled hybrid yield strengths for the composites with the measured values. Yield strengths were significantly overestimated by the summation approach. Hence, this approach is unsuitable for the current composites, which is associated with the superposition of the individual strengthening effects [31, 37]. These superposition effects are considered by the Clyne approach. However, precision decreases again, if the reinforcing particles were distributed more inhomogeneously within the matrix, see TMC-B in Table 11.
### Table 11. Comparison of the measured and the modelled hybrid compressive yield strength at 0.2% plastic deformation $\sigma_c$ of the TMCs (differences between model and experiment expressed as $\Delta_{\text{Mod} - \text{Exp}}$).

| TMC    | Experiment $\sigma_c$ (MPa) | Summation approach $\Delta_{\text{Mod} - \text{Exp}}$ (MPa) | Clyne approach $\Delta_{\text{Mod} - \text{Exp}}$ (MPa) |
|--------|-----------------------------|-------------------------------------------------------------|----------------------------------------------------------|
| TMC-A  | 1546 ± 14                   | 2033 (487)                                                  | 1585 (39)                                                |
| TMC-B  | 1245 ± 19                   | 1725 (480)                                                  | 1400 (155)                                               |

### 4. Conclusions
Mechanical properties of hybrid TiB/TiC particle-reinforced Ti-5Al-5Mo-5V-3Cr matrix composites were strongly influenced by the local reinforcement particle distribution. Despite almost identical total reinforcement particle volume fractions, the composite exhibiting a more homogeneously reinforcement particle distribution within the matrix reveals higher measured values of Young’s modulus, hardness and compressive yield strength. Furthermore, these mechanical properties were modelled taking into account the phase composition as well as microstructural parameters and the chemical composition of individual phases. The rule of hybrid mixtures (RoHM) and the Fu-method – both considering the hybrid TiB-whisker and TiC-particle reinforcement – were used for modelling of the Young’s modulus and the hardness, respectively. Concerning the compressive yield strength of the composite, it was necessary to identify the particular effective strengthening mechanisms. The strengthening contributions of these mechanisms represent input parameters for the compressive yield strength modelling in accordance with the summation approach and the Clyne-approach. With pronounced agglomeration of the reinforcement particles the measured values of the hybrid Young’s modulus and the hybrid hardness were overestimated by the RoHM and the Fu-method. The hybrid compressive yield strength of the composites was modelled more appropriately using the Clyne-approach. However, with increasing clustering of the reinforcement particles the measured values of the compressive yield strength were overestimated by the Clyne-approach as well.

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### References
[1] Gorsse S and Miracle D B 2003 Mechanical properties of Ti-6Al-4V/TiB composites with randomly oriented and aligned TiB reinforcements *Acta Mater.* **51** 2427–42
[2] Yang Z F, Lu W J, Xu D, Qin J N and Zhang D 2006 In situ synthesis of hybrid and multidimensional titanium matrix composites *J. Alloys Compd.* **419** 76–80
[3] Yanbin L, Yong L, Huiping T, Bin W and Bin L 2011 Fabrication and mechanical properties of in situ TiC/Ti metal matrix composites *J. Alloys Compd.* **509** 3592–601
[4] Huang L J, Geng L, Xu H Y and Peng H X 2011 In situ TiC particles reinforced Ti6Al4V matrix composite with a network reinforcement architecture *Mat. Sci. Eng. A* **528** 2859–62
[5] Nag S, Banerjee R, Srinivasan R, Hwang J Y, Harper M and Fraser H L 2009 $\omega$-Assisted nucleation and growth of $\alpha$ precipitates in the Ti–5Al–5Mo–5V–3Cr–0.5Fe $\beta$ titanium alloy *Acta Mater.* **57** 2136–47
[6] Veeck S, Lee D, Boyer R and Briggs R 2004 The castability of Ti-5553 alloy *Adv. Mater. Process.* **162** 47–9
[7] Cotton J D, Hellenbrand P J, Bryan D J, Bayha T D, Leder M and Levin I 2014 The Effect of Hydrogen on the Fracture Toughness of Ti-5Mo-5V-5Al-3Cr TMS 2014 143rd Annual Meeting & Exhibition, Annual Meeting Supplemental Proceedings ed The Minerals, Metals & Materials Society 1st edn (Springer International Publishing)

[8] Fanning J C 2005 Properties of TIMETAL 555 (Ti-5Al-5Mo-5V-3Cr-0.6Fe) J. Mater. Eng. Perform. 14 788–91

[9] Nyakana S L, Fanning J C and Boyer R R 2005 Quick reference guide for β titanium alloys in the 00s J. Mater. Eng. Perform. 14 799–811

[10] Wagner V, Baili M, Dessein G and Lallement D 2010 Experimental characterization of behavior laws for titanium alloys: application to Ti5553 Key Eng. Mater. 446 147–55

[11] Tjong S C and Mai Y W 2008 Processing-structure-property aspects of particulate- and whisker-reinforced titanium matrix composites Compos. Sci. Technol. 68 583–601

[12] Li G, Li D, Liu Y, Wang Q and Guan S Microstructure and Mechanical Properties of Ti-35V-15Cr-0.05C Nonburning Titanium Alloy J. Mater. Sci. Technol. 14 583–4

[13] Ma Z Y, Tjong S C and Gen L 2000 In-situ Ti-TiB metal-matrix composite prepared by a re-active pressing process Scripta Mater. 42 367–73

[14] Soboyejo W O, Lederich R J and Sastry SML 1994 Mechanical behavior of damage tolerant TiB whisker-reinforced in situ titanium matrix composites Acta Metall. Mater. 42 2579–91

[15] Ni D R, Geng L, Zhang J and Zheng Z Z 2006 Effect of B4C particle size on microstructure of in situ titanium matrix composites prepared by reactive processing of Ti-B4C system Scripta Mater. 55 429–32

[16] Feng H, Zhou Y, Jia D, Meng Q and Rao J 2006 Growth mechanism of in situ TiB whiskers in spark plasma sintered TiB/Ti metal matrix composites Cryst. Growth Des. 6 1626–30

[17] Carman A, Zhang L C, Ivasishin O M, Savvakin D G, Matviychuk M V and Pereloma E V 2011 Role of alloying elements in microstructure evolution and alloying elements behaviour during sintering of a near-β titanium alloy Mat. Sci. Eng. A 528 1686–93

[18] Vadayar K S, Rani S D and Prasad V V 2013 Microstructural and mechanical characteristics of in-situ titanium metal matrix composites Int. J. Theor. Appl. Res. Mech. Eng. 2 12–6

[19] Zhang X, Liu W, Zhang D, Wu R, Bian Y and Fang P 1999 In situ technique for synthesizing (TiB+TiC)/Ti composites Scripta Mater. 41 39–46

[20] Brodkin D, Kalidindi S R, Barsoum M W and Zavaliangos A 1996 Microstructural evolution during transient plastic phase processing of titanium carbide-titanium boride composites J. Am. Ceram. Soc. 79 1945–52

[21] Mogilevsky P, Gutmanas E Y, Gotman I and Telle R 1995 Reactive formation of coatings at boron carbide interface with Ti and Cr powders J. Eur. Ceram. Soc. 15 527–35

[22] Zhao H and Cheng Y-B 1999 Formation of TiB2-TiC composites by reactive sintering Ceram. Int. 25 353–8

[23] Vallauri D, Atlas Adrián I C and Chrysanthou A 2008 TiC-TiB2: composites: A review of phase relationships, processing and properties J. Eur. Ceram. Soc. 28 1697–713

[24] Jia L, Wang X, Chen B, Imai H, Li S, Lu Z and Kondoh K 2016 Microstructural evolution and competitive reaction behavior of Ti-B4C system under solid-state sintering J. Alloys Compd. 687 1004–11

[25] Grützner S, Krüger L, Schimpf C, Radajewski M and Schneider I 2018 Microstructure and mechanical properties of in-situ TiB/TiC particle-reinforced Ti-5Al-5Mo-5V-3Cr composites synthesized by spark plasma sintering Metall. Mater. Trans. A 49 5671-5682

[26] Grützner S, Krüger L, Radajewski M and Schneider I 2018 Characterization of in-situ TiB/TiC particle-reinforced Ti-5Al-5Mo-5V-3Cr matrix composites synthesized by solid-state reaction with B4C and graphite through SPS Metals 8 377

[27] Zhang Q, Wu G, Chen G, Jiang L and Luan B 2003 The thermal expansion and mechanical properties of high reinforcement content SiCp/Al composites fabricated by squeeze casting technology Compos. Part A Appl. Sci. Manuf. 34 1023–7
[28] Geiger A L and Jackson M 1989 Low expansion MMCs boost avionics *Adv. Mater. Process.* 7 23–30
[29] Rice R W, McMillan P W and Stryjak A J 1979 Internal stress dependence of the hardness of crystallized glasses *J. Mater. Sci.* 14 2768–72
[30] Goyal R K, Tiwari A N and Negi Y S 2008 Microhardness of PEEK/ceramic micro- and nano-composites: Correlation with Halpin-Tsai model *Mat. Sci. Eng. A* 491 230–6
[31] Sanaty-Zadeh A 2012 Comparison between current models for the strength of particulate-reinforced metal matrix nanocomposites with emphasis on consideration of Hall–Petch effect *Mat. Sci. Eng. A* 531 112–8
[32] Jia L, Chen B, Li S, Imai H, Takahashi M and Kondoh K 2014 Stability of strengthening effect of in situ formed TiCp and TiBw on the elevated temperature strength of (TiCp+TiBw)/Ti composites *J. Alloys Compd.* 614 29–34
[33] Callister W D and Rethwisch D G 2014 *Materials science and engineering: An introduction* 9th edn (Hoboken, NJ: Wiley)
[34] Ozerov M, Klimova M, Stepanov N and Zherebtsov S 2018 Microstructure evolution of a Ti/TiB metal-matrix composite during high temperature deformation *Mater. Phys. Mech.* 38 54–63
[35] Ozerov M, Klimova M, Sokolovsky V, Stepanov N, Popov A, Boldin M and Zherebtsov S 2019 Evolution of microstructure and mechanical properties of Ti/TiB metal-matrix composite during isothermal multiaxial forging *J. Alloys Compd.* 770 840–8
[36] Hull D and Clyne T W 1996 *An Introduction to composite materials* (Cambridge: Cambridge University Press)
[37] Casati R and Vedani M 2014 Metal matrix composites reinforced by nano-particles—A review *Metals* 4 65–83
[38] Fu S Y, Xu G and Mai Y W 2002 On the elastic modulus of hybrid particle/short-fiber/polymer composites *Compos. Part B Eng.* 33 291–9
[39] Vander Voort G 2015 *Metallographic preparation of titanium and its alloys* (Tech Notes 3)
[40] Nardone V C and Prewo K M 1986 On the strength of discontinuous silicon carbide reinforced aluminum composites *Scr. Metall.* 20 43–8
[41] Opini V C, Salvador C A, Campo K N, Lopes E S, Chaves R R and Caram R 2016 α phase precipitation and mechanical properties of Nb-modified Ti-5553 alloy *Mat. Sci. Eng. A* 670 112–21
[42] Madtha S, Lee C and Ravi Chandran K S 2008 Physical and mechanical properties of nanostructured titanium boride (TiB) ceramic *J. Am. Ceram. Soc.* 91 1319–21
[43] Ravi Chandran K S and Panda K B 2002 Titanium composites with TiB whiskers *Adv. Mater. Process.* 160 59–62
[44] Samsonov G V, Naumenko V Y and Okhremchuk L N 1971 Herstellung und Eigenschaften von Karbiden der Übergangsmetalle in ihren Homogenitätsbereichen *Phys. Stat. Sol. (a)* 6 201–11
[45] Leblanc J L 2010 *Filled polymers – Science and industrial applications* (Boca Raton: CRC Press)
[46] Ryu H J, Cha S I and Hong S H 2003 Generalized shear-lag model for load transfer in SiC/Al metal-matrix composites *J. Mater. Res.* 18 2851–8
[47] Fukuda H and Chou T W 1982 A probabilistic theory of the strength of short-fibre composites with variable fibre length and orientation *J. Mater. Sci.* 17 1003–11
[48] Grützner S 2020 *Festphasenreaktionsverhalten bei der SPS-Synthese von in-situ TiB/TiC-partikelverstärktem Ti–5Al–5Mo–5V–3Cr* (Freiberger Forschungshefte. B, Werkstofftechnologie vol 388) 1st edn (Freiberg: Technische Universität Bergakademie Freiberg)