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Effect of hot forging on microstructure and tensile properties of Ti-TiB based composites produced by casting

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Abstract

Microstructure and mechanical behavior of near eutectic Ti-1.5 wt.% B and hypereutectic Ti-2B wt.% B composite materials obtained by casting have been investigated. Commercially pure titanium was used as a matrix material. Homogeneously distributed TiB whiskers were revealed in the as-cast composite materials. Multiple isothermal 2-D forging of the composites was carried out in the temperature range of the beta phase field. The hot forging led to effective alignment of boride whiskers with retaining a high aspect ratio. Tensile mechanical tests in ascast and forged conditions were carried out at room and elevated temperatures. The composites demonstrated much higher strength in comparison with the matrix material without drastic ductility reduction. The effect of boride orientation and morphology on the tensile properties of the composite materials is discussed.

Key words: titanium matrix composites; microstructure; TiB whiskers; tensile properties

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1. Introduction

Strength, stiffness and wear resistance of Ti and Ti-alloys in a wide temperature range can be improved through composite route using continuous/discontinuous reinforcements [1-10]. Matrix and reinforcing materials are usually chosen based on the following criterions [1,2]: i) both materials should have low density, ii) elasticity modulus of the reinforcing material should be much higher than that of the matrix material (taking into account the comparatively low elastic modulus of Ti-alloys), iii) matrix and reinforcing materials should have similar thermal expansion coefficients, iv) the materials should be chemically stable with respect to each other to avoid the formation of unfavorable phases along boundaries between matrix and reinforcing materials. Composite materials based on Ti and Ti-alloys are reinforced by fibers or particles of TiB₂, B₄C, TiN, SiC, TiB, TiC or Al₂O₃ compounds [3-6]. The physical properties of some of these compounds are listed in Table 1. Most investigated composite materials based on Ti-alloys and reinforced by Si- or Al₂O₃-fibers or TiC-particles demonstrate improved strength, stiffness and wear resistance [2-5]. However because of high titanium reactivity all of these reinforcements lead to the formation of one or more reaction products at the interface that decrease the mechanical properties of the composite material [2,7]. The mentioned requirements are fulfilled best in the case of the TiB compound (lattice B27). This compound is characterized by high elasticity modulus, its thermal expansion coefficient is similar to that of Ti-alloys and the compound is chemically stable with respect to the matrix Ti-alloy [1,2,6-8] (Table 1).

In accordance with the binary Ti-B phase diagram [2], titanium alloys with a boron content of more than about 1.5 wt.% (hypereutectic alloys) are classified as discontinuously reinforced composite materials. Their fabrication techniques are usually based on powder metallurgy and sintering that provides high microstructural homogeneity and more or less appropriate properties [2,6-13]. In recent years, conventional casting has attracted great attention because of the easy of fabrication and low cost [14-20]. In this case, TiB whiskers are formed in-situ during casting [15]. The following regularities have been established in respect of near eutectic Ti-TiB based composite materials obtained by casting: i) the boron addition in an amount of around 1.5 wt.% leads to formation of TiB whiskers throughout the material resulting in refinement of as-cast structure [15,16]; ii) the morphology and distribution of the TiB whiskers are dependent on cooling conditions during casting [16]; iii) the presence of borides, borides together with carbides or lanthanum oxides increase strength, creep resistance but significantly reduce lowtemperature ductility in cast condition. Forging, sheet rolling and hot extrusion in the β or in the upper part of the α + β phase field followed by heat treatment can lead to properly balanced mechanical properties and particularly acceptable low-temperature ductility [14-20]. No doubt that a success of casting route is dependent on the boron amount which can be added without dramatic loss in ductility. A critical issue is also associated with thermomechanical treatment of the composite material. On the one hand, this can be used for reorientation of borides along the intended straining direction; on the other hand, thermomechanical treatment can break the boride whiskers resulting in a reduction of the aspect ratio and strengthening efficiency of the titanium borides [16-19,21-23].

The present work was aimed at a study of microstructure and mechanical properties of near eutectic and hypereutectic composites based on commercially pure Ti and TiB prepared by casting. Multiple two-directional (2-D) forging was applied as thermomechanical treatment to obtain preferentially oriented borides. Tensile properties of the composites were compared with those of the matrix alloy.

2. Experimental

2.1. Initial materials

The VT1-0 alloy (Russian alloy, analogue of Grade 2) and the VT1-0 alloy doped by 1.5 and 2.0 wt.% B were taken as starting materials. The alloy compositions are given in Table 2. For the sake of simplicity, the VT1-0, VT1-0-1.5B and VT1-0-2B alloys are designated in the text as Ti, Ti-1.5B and Ti-2B, respectively.

The composite materials were melted in a laboratory arc-melting furnace under argon atmosphere using the commercial Ti and boron powder. To have appropriate homogeneity, the ingots were remelted at least 7 times. The boron powder with purity 99.5% was supplied from the Russian enterprise OAO Aviabor. The Ti-alloy free of boron was obtained by remelting of the commercial Ti-alloy. 100-gram ingots of the Ti- and composite materials with an approximate size of \emptyset 45×15 mm were prepared.

2.2. Thermomechanical treatment

Before thermomechanical treatment the as-cast materials were cut off to make four flat faces. Thermomechanical treatment consisted of multiple 2-D forging under isothermal conditions at T=950°C and $\varepsilon'=10^{-2}-10^{-3}$ s⁻¹ with a total strain e≈3. As a result, the initial as-cast materials were transformed into the workpieces with a size of about 75×18×14 mm³. Both of the composite materials were forged in the same conditions. The obtained forgings were annealed at T=950°C (1 h.) followed by furnace cooling and then ground to remove the oxide layer. Flat specimens for tensile tests were cut out of the workpieces so that the specimen tension axis was parallel to the workpiece length.

2.3. Microstructural examination

For microstructural observations, the compressed specimens were cut parallel to their compression axes along their diameter and the cross section was studied. The forgings of the composite materials were examined in longitudinal and transversal sections. Microstructural examinations were carried out using optical and scanning electron microscopy (SEM) in secondary electron (SE) or back-scattering electron (BSE) mode. Before SEM studying, the specimen surfaces were subjected to polishing or polishing and etching. The etchant composition was 5% HF + 15% HNO₃ + 80% distilled H₂O. The volume fraction of borides was measured by the systematic point count method using polished specimens. The aspect ratio of the TiB whiskers (the ratio of length to diameter) was evaluated using deep-etched specimens. The tensile fracture behavior was studied taking into consideration flat surfaces and fracture surfaces of tensile strained specimens. X-ray diffraction (XRD) measurement was carried out using Co- K_{α} radiation.

2.4. Mechanical tests

Compression and tensile specimens were prepared by electrospark cutting followed by fine grinding of work surfaces. The compression tests were performed for one of the composite materials (Ti-1.5B) at T=700-1000°C with an initial strain rate of $\varepsilon'=10^{-3}$ s⁻¹ to an engineering strain of 60%. The specimen dimensions were 8×5×5 mm³. Two specimens were tested at each temperature. The maximum true stress σ_{max} , and the yield strength $\sigma_{1.25}$, corresponding to a plastic strain of 1.25% were determined from the tests.

Flat specimens having a gauge section of $10\times3.5\times1.5$ mm³ were used for tensile tests. Between three to five specimens per point were tested. The tensile tests were performed at T=20-500°C with an initial strain rate of $\varepsilon'=10^{-3}$ s⁻¹. The ultimate tensile strength σ_{UTS} , the yield strength $\sigma_{0.2}$, and elongation to rupture δ were determined from the tests. The reduction of area was defined after tensile testing at 20 and 300°C; at higher temperatures the reduction of area was not measured because of oxidation.

3. Results and discussion

3.1. Initial as-cast materials

Figs. 1a-c represent BSE and SE images of the Ti-alloy, Ti-1.5B and Ti-2B composites in as-cast conditions. The microstructure of the matrix alloy is characterized by coarse α -colonies with a size d~100-1000 μ m. Colonies have jagged boundaries, which are typical of α -Ti after fast cooling from the β -phase. The microstructures of the Ti-1.5B and Ti-2B composites consist

of α -Ti matrix and homogeneously distributed boride (TiB) whiskers having random orientations and morphology. The whisker size was mostly in the range $\emptyset(0.5-5)\times(10-200) \ \mu\text{m}$ in Ti-1.5B and $\emptyset(0.5-15)\times(10-800) \ \mu\text{m}$ in Ti-2B, respectively. Since the Ti-2B composite is hypereutectic, there were primary borides reaching up to 15 μ m in diameter and 800 μ m in length; sometimes the borides had plate-like morphology with a width up to 10-15 μ m. The volume fraction of whiskers was 8.3 and 11% in Ti-1.5B and Ti-2B, respectively. The aspect ratio of whiskers in Ti-1.5B averaged around 32. The colony size and the length of α -lamellae in the composites are limited by a distance between boride whiskers, which did not exceed 10-30 μ m. XRD analysis performed for Ti-1.5B confirmed the presence of the TiB phase (Fig. 1d).

3.2. Effect of forging processing on microstructure of the Ti-1.5 and Ti-2B composites

Thermomechanical treatment in respect of Ti-TiB composites is required not only to convert ingot shape but also to control TiB morphology and orientation. In addition, the forgeability of the composite materials should be tested before forging processing. Therefore, the hot compression tests were performed for one of the composites before forging. The compression tests results are given in Table 3. The yield strength of the composite was around two times higher than that of the matrix alloy. Nevertheless the composite specimens were strained to ε =60% without any cracks.

Figs. 2a,b illustrate BSE images of the Ti-1.5B composite after hot compression at T=800 and 1000°C, respectively. One can see that the boride whiskers tended to align perpendicularly to the deformation axis. This process at 700-800°C was accompanied by intensive breaking of the boride whiskers: their length in the middle of the deformed specimens decreased from 10-200 to 5-50 μ m (Fig. 2a). During deformation in the β phase field, at T=900-1000°C, the boride whiskers were appreciably less damaged (Fig. 2b). Reasoning from the compression tests, forging processing was performed in the β phase field, at T=950°C.

Figs. 3a-d represent SE and BSE images of the Ti-1.5B and Ti-2B composites in the forged conditions. 2-D forging led to alignment of the borides predominantly perpendicular to the forging directions. Figs. 3c-d illustrate the transversal sections of the forged workpieces. It is seen that the borides mostly look like hexagons that testifies to their alignment along the workpiece length. Nevertheless, not all boride whiskers were strictly aligned perpendicular to the both forging directions.

The forging procedure also led to partial breaking of the boride whiskers (Figs. 3a,b). Particularly, the primary borides in Ti-2B were broken and their length did not exceed 180 μ m. The aspect ratio of TiB in Ti-1.5B and Ti-2B after 2-D forging averaged around 20. The mean values of length and diameter of the boride whiskers after forging were slightly higher in Ti-2B

than in Ti-1.5B. Note that precise evaluation of the aspect ratio is not possible because not all borides were exactly oriented perpendicular to the both forging directions (parallel to a SEM picture under study) (Figs. 3a,b). Nevertheless, it can be concluded that 2-D forging did not lead to significant breaking of the borides that is reflected by the average aspect ratio. This can be explained by the fact that the strength of ceramic TiB whiskers at T=950°C is much higher than that of the matrix titanium, so many of the TiB whiskers were probably not broken during forging.

Since the forging and final annealing temperature was relatively high (in the single β phase field), the average size of α -phase colonies did not seem to change in the composites after processing in comparison with that in the as-cast composites.

3.3. Tensile tests

The tensile tests performed in the temperature range of T=20-500°C showed appreciably higher strength and lower ductility of the composites as compared with the matrix alloy (Fig. 4). As was demonstrated for Ti-1.5B, 2-D forging resulted in a marked increase of strength and ductility against the as-cast composite material. Evidently, this should be ascribed to alignment of the boride whiskers after forging processing. With increasing the boron content from 1.5 to 2% the strength slightly increased and the ductility decreased both at room and elevated temperatures. Note that even at room temperature the elongation obtained for Ti-2B was always higher than 5% and averaged 7.6%. As the test temperature increased, the ductility of the alloys increased reaching maximum values at 300°C and then decreased; that can be attributed to oxidation of specimens during testing. At T=400-500°C the ductility of the composite materials was found to be approximately the same irrespective of the boride content, morphology and orientation. Presumably, this is associated with increasing the interphase Ti-TiB surfaces extent, which promoted oxidation of the composites in the course of tensile testing.

It is worth noting that the difference in the σ_{UTS} values of the composites and the matrix alloy at room temperature is higher than the difference in the $\sigma_{0.2}$ values in spite of the fact that the ductility of the composites was lower than that of the matrix alloy. It is indicative of significant strengthening during straining caused by the borides. This strengthening effect is observed in spite of breaking of the borides during straining.

The reduction of area after tensile tests at 20 and 300°C strongly decreased in the composites especially in cast Ti-1.5B having random boride orientations as compared with the matrix alloy (Fig. 4d). Another interesting feature was the fact that the ψ and δ values were approximately the same for cast Ti-1.5B, about 5 and 9% at 20 and 300°C, respectively, while in the forged composites having predominant orientation of borides along the tensile axis the ψ

values were appreciably higher than the corresponding elongations (Figs. 4c,d). In other words, in Ti-1.5B with random boride orientations the strain developed till fracture almost without necking, while the composite materials in the forged conditions showed appreciable necking. Thus, predominant orientation of the boride whiskers along the tensile axis led to more ductile fracture.

Table 4 summarizes the microstructural parameters and room temperature tensile properties of the composites compared with the Ti-alloy. The orientation of the boride whiskers thus plays a key role in both tensile strength and ductility of the composite materials.

It is known that the increment in strength of discontinuously reinforced metal matrix composite materials is dependent upon the shape and volume fraction of the reinforcement, the orientation of the whiskers and cohesive strength between the matrix and reinforcement materials. A shear-lag model based on the load-transfer concept between matrix and reinforcement materials can be applied to evaluate the increase in yield strength Δ YS [22-25]:

$$\Delta YS = YS_m \times 0.5 \times V \times L/D \times C \tag{1}$$

where YS_m is the yield strength of the matrix alloy, V and L/D are the volume fraction and the aspect ratio of TiB whiskers, respectively, C is the whisker orientation factor, which can be varied from 0 to 1. Note that strengthening caused by matrix microstructure refinement is apparently negligible since the α -colony size, as mentioned above, was not practically changed after forging and annealing.

Taking into account Eq. (1) and the data represented in Table 4, the orientation factor value can be calculated. The calculation gives C=0.25 in as-cast Ti-1.5B, C=0.8 and C=0.71 in the forged Ti-1.5B and Ti-2B composites, respectively. Thus, 2-D forging in the β phase field, at T=950°C, resulted in effective alignment of boride whiskers (the C value increased from 0.25 to 0.71-0.8) and maintenance of a high aspect ratio of the TiB whiskers that promoted the strengthening effect. Note that 2-D forging (or drawing) is one of the typical hot working operations in respect of titanium based alloys.

It seems that similar forging processing can be applied for composite Ti-TiB based alloys with another matrix material. For instance in the case of the Ti64 matrix alloy, the Δ YS value in accordance with Eq. (1) (taking into account the YS_m value in as-cast condition as 827 MPa [16], V=0.083, L/D=20 and C=0.8) can be near 550 MPa that gives the yield strength of the Ti64-1.5B composite as high as 1377 MPa. Ivasishin and coauthors obtained this outstanding result in Ti64-1.55B after 3-D forging, rolling, beta solution treatment and ageing [16]. However the high strength of Ti64-1.55B was attributed not only to load-sharing by TiB but also to strengthening of the matrix material owing to hot working and subsequent strengthening heat treatment [16]

while the strengthening in the Ti-1.5B and Ti-2B composites was only provided by a loadsharing mechanism due to high modulus and strength of the borides.

Thus, the increment in yield strength due to the presence of the boride whiskers in the Ti-TiB based composites can be significant if appropriate thermomechanical treatment is applied. In the case of the titanium matrix material multiple 2-D forging at T=950°C and $\varepsilon'=10^{-2}-10^{-3}$ s⁻¹ was found to be a promising method providing effective alignment of the boride whiskers with simultaneous retaining a high aspect ratio. The increase in the boron content from 1.5 to 2 wt.% led to additional strengthening without drastic reduction of the ductility (Table 4). However the efficiency of strengthening in the hypereutectic composite was slightly lower than in the near eutectic composite due to a lower value of the orientation factor C. Lower ductility of Ti-2B comparing to Ti-1.5B (Table 4) should be evidently ascribed to coarser borides and to a higher amount of unfavorably oriented boride whiskers in Ti-2B.

3.4. Fracture behavior

The flat surfaces of tensile strained specimens of the composites were investigated. Figs. 5a,b illustrate formation of macroscopic cracks caused by unfavorably oriented borides and breaking of a coarse boride after tensile testing. The fact that the boride whiskers were fractured during the tensile test suggests that the whiskers bore the load, which transferred from the matrix material (Figs. 5c,d). Evidently, this is a result of high strength of coherent boundaries between the matrix and reinforcement materials [23].

The following conclusions can be done from Fig. 5: i) large cracks and particularly cracks of a critical size were initiated within the matrix material between unfavorably oriented borides (Fig. 5a) or within large unfavorably oriented borides (Fig. 5b); ii) the borides oriented parallel or near parallel to the tensile axis were broken during straining but did not lead to formation of large cracks (even in the case of rough borides) (Figs. 5c,d); iii) the strain was localized near the fracture zone. As can be seen (Figs. 5c,d), the boride breaking occurred much more extensively near the fracture zone than far from it.

Fig. 6 represents fracture surfaces of tensile specimens of the composites in the cast and forged conditions. One can see that the fracture behavior depended strongly on the boride whiskers orientation. Even in the as-cast Ti-1.5B composite the fracture behavior was predominantly ductile if the boride orientation was relatively favorable (Fig. 6a). On the contrary, unfavorable orientation of the borides led to brittle cleavage fracture (Fig. 6b). In the forged Ti-1.5B and Ti-2B composites the fracture behavior was much more ductile than in as-cast Ti-1.5B. One can see that ductile dimples are characteristic of the fracture surfaces especially in the case of favorable boride whiskers orientation (Figs. 6c). It is evident that

unfavorably oriented coarse boride whiskers, especially plate-like whiskers, promoted brittle cleavage fracture (Figs. 6d).

Thus, the fracture behavior of the Ti-TiB composites is strongly dependent upon the boride whiskers orientation. If the TiB whiskers are oriented parallel to the tensile axis, the fracture behavior becomes near completely ductile.

One can conclude that alloying of titanium and titanium alloys by boron combined with 2-D hot forging in the temperature range of the β phase field can be used for manufacturing of discontinuously reinforced Ti-TiB based composites with aligned boride whiskers having a high aspect ratio. Comparing with Ti-TiB based composites having a random boride whiskers orientation, this will provide significant improvement in both ductile and strength properties.

4. Conclusions

Near eutectic Ti-1.5B and hypereutectic Ti-2B composite materials have been obtained by casting to explore the relationships between processing, microstructure and tensile properties. The following conclusions can be drawn from the study:

• Whisker reinforced Ti-1.5B and Ti-2B composite materials were produced in-situ using the arc-melting technique. The borides were homogeneously distributed throughout the cast materials and their volume fraction was 8.3 and 11%, respectively.

• Hot compression tests showed that the boride whiskers tended to align perpendicularly to the deformation axis. At T=700-800°C this process was accompanied by intensive breaking of the boride whiskers, whereas at T=900-1000°C the boride whiskers were appreciably less damaged.

• Multiple isothermal 2-D forging at T=950°C and $\varepsilon'=10^{-2}-10^{-3}$ s⁻¹ applied for the Ti-1.5B and Ti-2B composites led to effective alignment of boride whiskers with retaining a high aspect ratio. Presumably, this is due to the fact that the deformation was fulfilled in the β phase field and at relatively low strain rates. As was demonstrated for Ti-1.5B, this resulted in significant improvements in the strength and ductility as compared to the composite material with randomly oriented boride whiskers. Particularly, the elongations as high as δ =13.3 and 7.6% were obtained at room temperature in the forged Ti-1.5B and Ti-2B composites, respectively.

• The strengthening effect was interpreted in terms of a shear-lag model based on the load-transfer concept between matrix and reinforcement materials. In accordance with this model, the whisker orientation factor after forging was evaluated as C=0.8 and 0.71 in Ti-1.5B and Ti-2B, respectively.

The obtained results suggest that similar thermomechanical processing, 2-D forging in the temperature range of the β phase field, might be effective for composite Ti-TiB based materials

with the matrix material made of industrial titanium alloys. In this case an additional contribution to strengthening can be obtained due to strengthening of the matrix material if appropriate heat treatment is applied after 2-D forging. The Ti-TiB based composite materials obtained via casting and 2-D forging can be apparently considered for engineering applications.

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Figure captions

Fig. 1. (a) BSE and (b,c) SE images of the (a) Ti-alloy, (b) Ti-1.5B and (c) Ti-2B composites in as-cast condition and (d) XRD spectrum obtained for Ti-1.5B in as-cast condition; b,c – deepetched specimens.

Fig. 2. BSE images of the Ti-1.5B composite in hot deformed conditions: a and b - middle parts of specimens deformed at T=800 and 1000°C, respectively (ϵ =60%). The deformation axis is vertical.

Fig. 3. (a,b) SE and (c-d) BSE images of the (a,c) Ti-1.5B and (b,d) Ti-2B composites after 2-D forging: a,b – longitudinal sections of the forgings, c,d – transversal sections of the forgings; a,b – deep-etched specimens.

Fig. 4. Temperature dependences of (a) yield strength $\sigma_{0.2}$, (b) ultimate tensile strength σ_{UTS} , (c) elongation to rupture δ and (d) reduction of area ψ obtained for the Ti-alloy, Ti-1.5B and Ti-2B composites.

Fig. 5. SE images obtained from the flat surfaces of specimens tensile tested at room temperature: a,b – formation of large cracks (a) between or (b) within unfavorably oriented boride whiskers, c,d – breaking of the borides (c) near and (d) far from the fracture zone; a – Ti-1.5B, b-d – Ti-2B. The tensile axis is horizontal.

Fig. 6. Fracture surfaces of specimens of the (a,b) Ti-1.5B and (c,d) Ti-2B composites after tensile straining at room temperature: a,b - as-cast condition with random boride whiskers orientation (a - relatively favorable and b - unfavorable boride orientation); c,d - forged conditions with predominantly aligned boride whiskers (the whiskers are mostly located perpendicular to the picture plane); d – the effect of individual unfavorably oriented coarse borides on the fracture behavior.



Fig. 1. (a) BSE and (b,c) SE images of the (a) Ti-alloy, (b) Ti-1.5B and (c) Ti-2B composites in as-cast condition and (d) XRD spectrum obtained for Ti-1.5B in as-cast condition; b,c – deepetched specimens.



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Characteristics	Ti	TiB	TiB ₂	TiC	SiC
Density, g/cm ³	4.57	4.56	4.52	4.92	3.21
Elasticity modulus, GPa	110	550	529	460	420
Thermal expansion coefficient at $20^{\circ}C(\times 10^{-6})$, K ⁻¹	8.8	8.6	6.4	7.4	4.3

Properties of the Ti, TiB, TiB₂, TiC and SiC compounds [7]

Material compositions (in wt.%)

Material	Fe	С	Si	Ν	Ti	0	Η	В	Other impurities
Ti	< 0.18	< 0.07	<0.1	< 0.04	99.24-99.7	< 0.12	< 0.01	-	<0.3
Ti-1.5B	same						1.5	Same	
Ti-2B	same					2.0	Same		

Compression mechanical properties of the Ti-1.5B composite at T=700-1000 $^{\circ}$ C compared with the Ti-alloy

T, °C	Ti			Ti-1.5B		
	σ_{max} , MPa	σ _{1.25} , MPa	ε, %	σ_{max} , MPa	σ _{1.25} , MPa	ε, %
700	115	90	>60	166	165	>60
800	63	50	>60	100	99	>60
900	40	30	>60	71	70	>60
1000	6	5.5	>60	25	24	>60

Summary of the microstructural parameters and room temperature tensile properties of the Ti-1.5B and Ti-2B composites compared with the Ti-alloy

Parameters and mechanical properties	Ti as-cast	Ti-1.5B as-cast	Ti-1.5B 2D-forging	Ti-2B 2D-forging
Size of α -	coarse	fine (fine (~10 µm)	
colonies, µm	(~100-1000 µm)		(- · · P····)	
Volume fraction	_		11	
of borides, %	-			
Orientation of	_	random	predominantly	predominantly
boride whiskers	-	Tandom	aligned	aligned
Average aspect	_	≈32	≈20	≈20
ratio		52	20	20
$\sigma_{0.2}$, MPa	315	420	522	559
σ_{UTS} , MPa	430	617	722	796
δ, %	17	5	13.3	7.6
ψ, %	54	6.5	23.1	19.7