# PRESSURELESS SINTERING AND CHARACTERIZATION OF B$_4$C, TiC AND TiB$_2$-PARTICLE-REINFORCED TiAl$_3$-MATRIX COMPOSITES

## SINTRANJE IN KARAKTERIZACIJA KOMPOZITOV NA OSNOVI TiAl3, OJAČANIH Z DELCI B$_4$C, TiC IN TiB$_2$

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Dense (>95 % of theoretical density) TiAl$_3$-based composites reinforced with the volume fractions of B$_4$C or TiC 10–50 % and 10–30 % of TiB$_2$ particles were successfully prepared by combining pressureless reaction synthesis and reaction sintering. The first step was formation of TiAl$_3$ from the elemental powders by reaction between solid Ti and liquid Al (at 800 °C/1h and 1250 °C/3h in a static atmosphere of Ar with the volume fraction of H$_2$ 4 %). The reaction product obtained with a nominal composition of 88% TiAl$_3$-10%Al-2 % Ti, was milled and subsequently blended with the ceramic powders. The green compacts made from the blended powder mixture were reaction sintered at 1300 °C for 2 h in an Ar + 4 % H$_2$-rich environment using a vacuum furnace. The morphology of the TiAl$_3$ powders and the microstructure of the as-sintered composites were studied by scanning electron microscopy and X-ray diffraction analysis. The mechanical properties were measured by tensile tests performed at room temperature. In the case of reactive systems (TiAl$_3$-B$_4$C and TiAl$_3$-TiC), densification proceeds via formation of various secondary bonding phases, whilst in non-reactive system (TiAl$_3$-TiB$_2$) the process seems to proceed by infiltration of molten aluminium into pores. In this respect, densification of TiAl$_3$-TiB$_2$ samples with more than 30 % of ceramic particles was incomplete. Regarding the room temperature tensile properties of the composites, superior improvement was observed in TiAl$_3$-B$_4$C and TiAl$_3$-TiC samples with the volume fraction of ceramic particles 50 %. However, in TiAl$_3$-TiB$_2$ samples the improvement was significantly lower due to the lack of chemical affinity (wetting) between ceramic reinforcement and TiAl$_3$ matrix.

Key words: aluminium titanates, reaction synthesis, intermetallic matrix composites (imcs), ceramic particulate reinforcement, reaction sintering, metallographic characterization

Z reakcijskim sintranjem smo pripravili goste (>95 % teoretične gostote) kompozitne materiale na osnovi spojine TiAl$_3$, ojačane z anorganskimi delci s prostorninskimi deleži B$_4$C in TiC 10–50 % ter 10–30% TiB$_2$. V prvi fazi smo sintetizirali spojino TiAl$_3$ iz prahov elementov Ti in Al z reakcijo med trdnim Ti in tekočim Al pri 800 °C/1h in kasnejšim segrevanjem pri 1250 °C/3h v zaščitni atmosferi Ar s 4 H$_2$. Tako pripravljen vzorec, ki je vseboval 88 % TiAl$_3$, 10 % Al in 2 % Ti smo zmleli ter mu primešali keramične delce v določenem razmerju in dobro homogenizirali. Te izhodne mešanice smo enoosno stisnili in sintrali v pri 1300 °C 2 uri v zaščitni atmosferi Ar/H$_2$. Morfologijo izhodnih delcev TiAl$_3$ in mikrostrukturo pripravljenih vzorcev smo analizirali z elektronsko vrstično mikroskopijo in energijsko disperzijsko analizo ter rentgensko praškovno difrakcijo. Mehanske lastnosti smo določali z nateznim preizkusom pri sobni temperaturi. Med sintezo vzorcev na osnovi sistemov TiAl$_3$-B$_4$C in TiAl$_3$-TiC je zgoščevanje potekalo s tvorbo vmesnih faz, v sistemu TiAl$_3$ – TiB$_2$ pa z infiltracijo tekočega Al v pore in po mejah med zrni. V vzorcih TiAl$_3$ –TiB$_2$ z več kot 30 % TiB$_2$ je bilo takšno zgoščevanje nepopolno, zato so vzorci porozni. Glede na mehanske lastnosti pripravljenih kompozitnih materialov pri sobni temepraturi kažejo največje izboljšanje le-teh materiali s prostorninskim deležem 50 % B$_4$C in TiC. V sistemu TiAl$_3$-TiB$_2$ so bile izmerjene lastnosti nekoliko slabše zaradi odsotnosti kemijske reakcije med anorganskimi delci in TiAl$_3$ matrico.

Ključne besede: aluminij titanati, reakcijska sinteza, kompoziti z intermetalno matrico, diskontinuirna ojačitev s keramičnimi delci, reakcijsko sintranje, metalografske preiskave

## 1 INTRODUCTION

Titanium aluminide alloys based on the equilibrium solid phases of the Ti- Al system and their composites reinforced with ceramic particles have been considered and are being developed for high-temperature applications in the automotive, aerospace, and power-generation industries. Such intermetallics based-matrix composites (IMCs) reinforced with ceramic particles generally achieved specific strength and specific stiffness, creep strength, toughness, and high-temperature strength resistance remarkably better than their as non-reinforced intermetallic counterparts [1,2]. Members of this class of advanced materials are of interest as potential high-temperature materials due to their high melting point, low density, good oxidation resistance, and high modulus of elasticity. Ultimately, bringing these attractive intermetallic materials into use largely depends upon the availability of practical processing routes.

Titanium aluminide IMCs reinforced with various ceramic particles (B$_4$C, TiC, TiB$_2$) can be effectively produced by powder processing, starting from commercial titanium aluminide powders blended with ceramic particulates [3]. Although this method offers near-net shapes with very little post-fabrication machining required, it is at the same time very expensive for

commercial application due to the high cost of titanium aluminide powders. Essentially, it is of great significance to develop an effective technique for preparation of titanium aluminide powders starting from cost-effective raw materials (e.g. by the exothermic reaction of liquid aluminium with solid titanium) and their conventional densification to fully dense alloys and composites [4]. Since such reactive synthesis and sintering aims at the formation of one or more aluminides, a prior knowledge of the formation of specific aluminide(s) and their microstructural development is particularly important for further commercialization of the process [5].

Among all the equilibrium solid phases of the Ti-Al system, only TiAl and Ti$_3$Al have been extensively used for alloys production and as a matrix in composites. This is because the Ti rich compounds Ti$_3$Al and TiAl exist over a wide range of composition while TiAl$_3$ exists as a very narrow range of compounds (an intermediate phase that appear as a vertical line in the binary phase diagram at some stoichiometric ratio of mole fractions of the elements) and any change in the definite composition of the mole fraction of elements (e.g. by oxidation) leads to its decomposition [6].

In the early stages of the development of titanium aluminide alloys and composites, the single step elemental powder metallurgical route has gained particular attention because near-net shape titanium aluminide alloy products could be fabricated by the consolidation and forming of blended Ti and Al elemental powders, followed by a subsequent reactive sintering process [7–11]. However, due to the large difference between the partial diffusion coefficients of Ti and Al, the synthesis of titanium aluminide alloys via reactive sintering follows the mechanism that Al atoms move faster into the Ti lattice then vice versa, thus leading during reactive sintering to the formation of Kirkendal diffusion pores [12–14]. Hot isostatic pressing (HIP) and refining of the Ti and Al reactants through cold deformation, such as extrusion and rolling of the Ti and Al powder mixture billet prior to reactive sintering, have been reported to be effective in eliminating the porosity of reactively sintered TiAl and Ti$_3$Al alloys. However, their high cost and low productivity make these techniques unsuitable for commercial use.

In the current attempt to optimize the cost and quality of IMCs, a two stage composite fabrication process was considered. The first step in composite production was the cost-effective synthesis of titanium aluminide powders from liquid aluminium and solid titanium, while the second step was the reactive sintering of the cold compacted as-received titanium aluminide powders blended with ceramic particles (B$_4$C, TiB$_2$ and TiC) to composite samples. The main expectation was that porosity due to Kirkendal diffusion could be reduced below 5 % by refining the microstructure with ceramic particulate reinforcement and by selecting from the titanium aluminides the most reactive constituents, such as TiAl$_3$.

TiAl$_3$ was found to be attractive for at least two reasons: (i) it is the main product of the reaction between liquid aluminium and solid titanium, which is one of the most economical ways of production of titanium aluminides, and (ii) it is also a reactive, non-stable phase (Daltonides) which could react with ceramic reinforcement, enabling formation of fully dense composite structures.

Hence, in the present paper we report the fabrication and characterization of titanium aluminide-based IMCs made by a pressureless two-step procedure: (i) the reactive synthesis of titanium aluminide powders with nominal composition 25Ti : 75Al by reactive synthesis from liquid aluminium and titanium powder in combination with milling and, (ii) reactive sintering of cold compacted 25Ti : 75Al powders blended with ceramic particles (B$_4$C, TiB$_2$ and TiC) to fully dense composite samples.

## 2 EXPERIMENTAL

TiAl$_3$ powder was synthesized by high temperature reaction synthesis from the elemental powders. As a first step, a homogeneous powder mixture of titanium (purity 99.5 % and average particle size 30 μm) and aluminium (purity 99.7 % and particle size 100 μm) with a composition corresponding to stoichiometric TiAl$_3$, 25 % mole fractions of Ti and 75 % Al, was prepared in a planetary mill. After room temperature uniaxial and cold isostatic compaction of the mixtures, the green pellets obtained were heated in a vacuum furnace (800 °C/1h and 1250 °C/3h), in a static atmosphere of Ar + 4 % H$_2$. This was followed by cooling the pellets to room temperature and subsequent milling of the reaction products in an attrition mill. The milling time was 4 h, and the grinding media-to-powder ratio was 2:1.

Finally, composites were formulated by blending the synthesized TiAl$_3$ powder with commercial ceramic powders (B$_4$C, TiC and TiB$_2$) in appropriate amounts to create IMC-based matrices with the volume fractions (10, 20, 30, 40 and 50) % of B$_4$C, TiC or TiB$_2$ discontinuous reinforcement.

The powder blends were thoroughly mixed in a planetary mill and subsequently cold compacted. In all cases, reaction sintering of the compacts was conducted at 1300 °C for 2 h in an Ar + 4 % H$_2$-rich environment using a vacuum furnace.

The as-synthesized composite samples were cut, machined and polished in accordance with standard procedures.

Microstructural characterization of titanium aluminide powders, as well as sintered composites, was performed by optical and scanning electron microscopy (OM and SEM with EDS), whereas X-ray diffraction (XRD) measurements were applied to the samples to identify the phases and their crystal structures.

The specimens for OM observation were electrolytically polished in a solution of 95 % CH$_3$COOH and

5 % $HClO_4$, and then etched in a solution of 5 % $HNO_3$, 15 % HF, and 80% $H_2O$. The main grain sizes were measured by the linear intercept method.

The specimens for XRD were abraded with SiC paper and were then subjected to diffraction using $CuK_a$ radiation.

Quantitative determination of the volume percentage of titanium aluminide and secondary phases involved in the matrix, ceramic particles and retained porosity was performed by analysing optical and scanning electron micrographs of polished composite bars using the point counting method and image analysis and processing software.

Composite density measurements were carried out in accordance with Archimedes' principle, applying distilled water as the immersion fluid.

The initial density of the green compacts was calculated from the mass and geometry of the samples.

The tensile properties (tensile strength, 0.2 % tensile yield strength and elongation) of the composite specimens were determined in accordance with the ASTM test method, E8M-96. The tensile tests were conducted on cylindrical tension-test specimens 3.5 mm in diameter and 16 mm gauge length using an automated servo-hydraulic tensile testing machine with a crosshead speed of 0.254 mm/60 s.

## 3 RESULTS AND DISCUSSION

### 3.1 Morphology and chemical composition of laboratory prepared titanium aluminide powders

The morphology of laboratory prepared $TiAl_3$ powders, obtained by milling the pelletized products of reactive synthesis, is presented in **Figure 1**. As evident, the $TiAl_3$ powder obtained is non-agglomerated, with well shaped individual particles of particle size mostly below 10 μm.

The morphology of the raw titanium powder, **Figure 2**, is very similar with the morphology of synthesized $TiAl_3$. The main difference is in the average particle size,
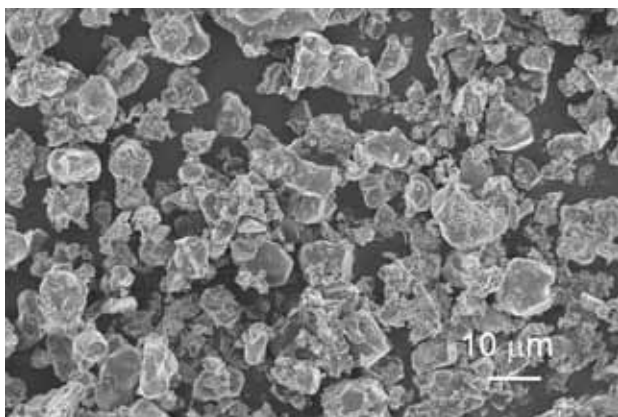


**Figure 1:** SEM micrograph of $TiAl_3$ after milling
**Slika 1:** SEM-posnetek $TiAl_3$-delcev po mletju
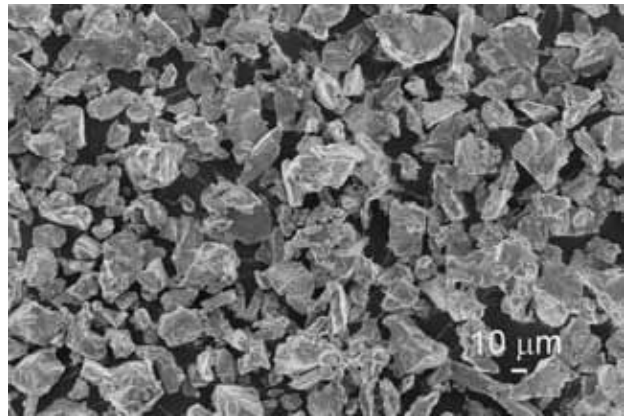


**Figure 2:** SEM micrograph of as-received commercial Ti powder
**Slika 2:** SEM-posnetek komercialnega Ti-prahu

which in the case of the as-received titanium powder is about three times larger.

The typical phase composition in synthesized $TiAl_3$ is reported in **Table 1**.

**Table 1:** Phase composition in laboratory prepared $TiAl_3$ powder
**Tabela 1:** Fazna sestava laboratorijsko pripravljenega $TiAl_3$ prahu

| Phase | Volume fraction (*f/%*) |
|---|---|
| $TiAl_3$ | 87 ± 5 |
| Al | 10 ± 1 |
| Ti | 2 ± 0.2 |
| Other phases | 1 ± 0.1 |

### 3.2 Microstructural development in IMCs

Generally, the microstructure of IMCs consists of an intermetallic matrix (based on an ordered intermetallic compound or a multiphase combination of intermetallic compounds), the ceramic particulate reinforcement and the interfacial region with secondary phases formed during reactive sintering by chemical reactions proceeding between the ceramic reinforcement, $TiAl_3$ and/or non-reacted Al and Ti. As minor phases, solidified Al inclusions and individual titanium particles are also involved in the composite microstructure.

#### 3.2.1 $B_4C$-$Ti_3Al$ samples

Reactive sintering of $B_4C$-TiAl and $B_4C$-$Ti_3Al$ composite samples resulted in specimens with densities higher than 95 % of T. D. As in densification, the boron carbide particulate reinforcement reacted with the intermetallic matrix forming numerous secondary phases ($TiB_2$, TiC, $Al_3BC$), well detectable in the microstructure of sintered samples – **Figure 3**.

In the $B_4C$-$Ti_3Al$ and $B_4C$-TiAl samples intensive chemical reactivity between the composite constituents was observed during high temperature densification due to the fact that boron carbide reacts with both molten aluminium and solid titanium [15,16].
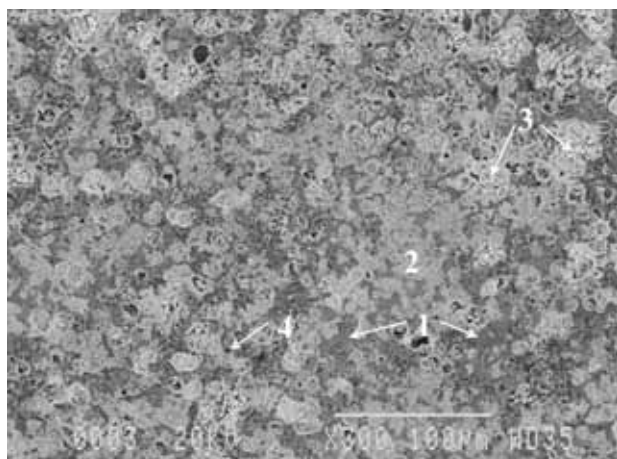
**Figure 3:** SEM micrograph of sintered $TiAl_3$-40 % $B_4C$ composite sample with various phases identified by XRD: (1) Al, $Al_4C_3$, $Al_3BC$ (2) AlTi, $AlB_2$, (3) $TiB_2$ and (4) $B_4C$

**Slika 3:** SEM posnetek sintranega $TiAl_3$-40 % $B_4C$-vzorca kompozita z različnimi fazami, identificiranimi z XRD: (1) Al, $Al_4C_3$, $Al_3BC$ (2) AlTi, $AlB_2$, (3) $TiB_2$ in (4) $B_4C$
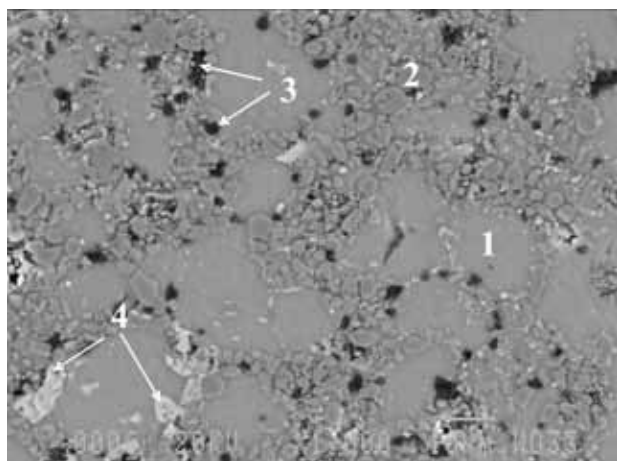
Boron carbide reacts at high temperature (1300 °C) with aluminium, forming mainly $Al_3BC/Al_4C_3$ and some $AlB_2$ (Reaction 1):

$$5B_4C_{(s)} + 19Al_{(l)} = 2Al_3BC_{(s)} + Al_4C_{3(s)} + 9AlB_2 \quad (1)$$

However, boron carbide also reacts with solid titanium particles producing secondary TiC and $TiB_2$:

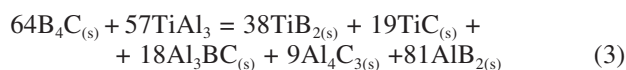$$B_4C_{(s)} + 3Ti_{(s)} = 2TiB_{2(s)} + TiC_{(s)} \quad (2)$$

Moreover, from thermodynamic point of view, boron carbide could also react with $TiAl_3$ causing its decomposition. The possible overall stoichiometric reaction between boron carbide and particulate $TiAl_3$ is the following:

$$64B_4C_{(s)} + 57TiAl_3 = 38TiB_{2(s)} + 19TiC_{(s)} +$$
$$+ 18Al_3BC_{(s)} + 9Al_4C_{3(s)} + 81AlB_{2(s)} \quad (3)$$

### 3.2.2 $TiB_2$-$TiAl_3$ samples

$TiB_2$ is stable at 1300 °C in contact with both $TiAl_3$ and aluminium. Because of that, dense composite samples, **Figure 4**, are obtained only if a sufficient amount of elemental aluminium is present in the system as a liquid phase. The experimental findings suggest that the necessary amount of aluminium is about one third of the volume fraction of ceramic reinforcement. Sintering of $TiAl_3$-$TiB_2$ composites with a larger amount of ceramic reinforcement (>30 %) and without proper addition of elemental aluminium resulted in samples with a huge fraction of Kirkendall diffusion pores, as it is shown in **Figure 5**.
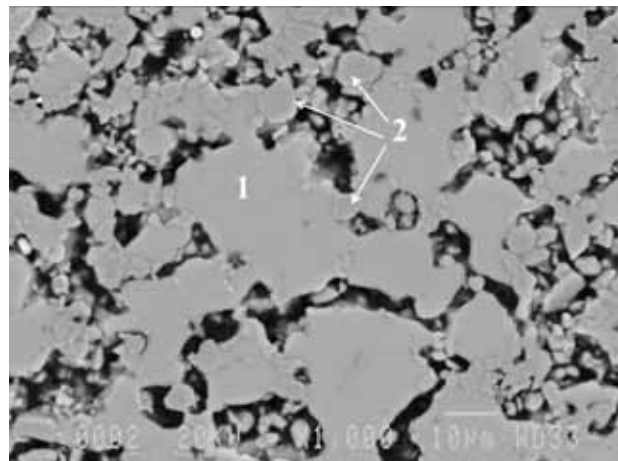
### 3.2.3 TiC-$Ti_3Al$ samples

In this system, the best results of pressureless reactive sintering were experimentally obtained. Almost all the sintered samples, including these with a high amount (50 %) of ceramic reinforcement, had a retained porosity below 5 %, among which many were fully dense.

As in the case of $B_4C$, TiC may also react with $TiAl_3$ and elemental aluminium. The products of these reactions are various Ti-Al-C phases (e.g. $Ti_3AlC$, $Ti_3AlC_2$ and $Ti_2AlC$) and $Al_4C_3$. The presence of these phases was confirmed by SEM-EDS at the matrix-reinforcement interface of the fabricated composites, **Figures 6, 7**.

### 3.3 Mechanical properties

The results of room temperature tensile tests on the composite samples are listed in **Table 2**. As a result of matrix reinforcement, significant improvements in Young's modulus, tensile strength and ultimate tensile
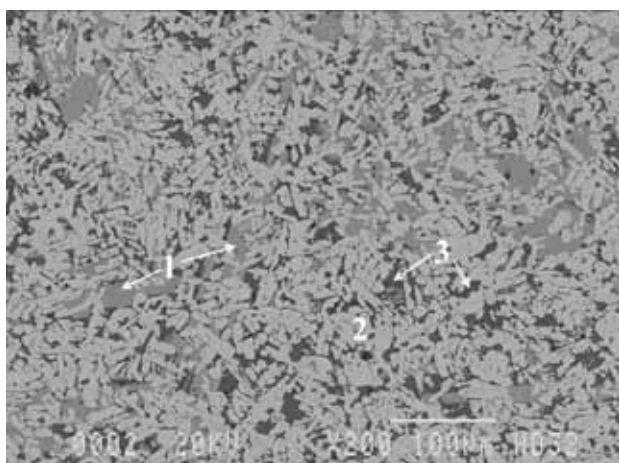


**Figure 4:** Microstructure of a pressureless sintered $TiAl_3$-30 %$TiB_2$ -10 % Al composite. The microstructural constituents are: (1) $Al_3Ti$, (2) $TiB_2$ (3) Al and (4) traces of $Al_2O_3$

**Slika 4:** Mikrostruktura $TiAl_3$-30 % $TiB_2$ -10 % Al-kompozita, sintranega pri atmosferskem tlaku. Mikrostrukturni gradniki so: (1) $Al_3Ti$, (2) $TiB_2$ (3) Al in (4) sledovi $Al_2O_3$



**Figure 5:** Scanning electron micrograph of pressureless sintered $TiAl_3$-50 % $TiB_2$ composite with a significant number of Kirkendall diffusion pores: (1) $Al_3Ti$ and (2) $TiB_2$

**Slika 5:** SEM-posnetek $TiAl_3$-50 % $TiB_2$ kompozita sintranega pri atmosferskem tlaku s številnimi Kirkendallovimi porami: (1) $Al_3Ti$ in (2) $TiB_2$
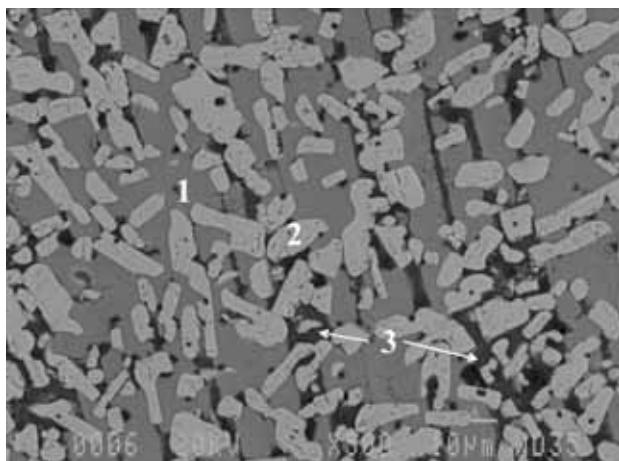
**Figure 6:** SEM micrograph of a TiAl$_3$-30 % TiC composite: (1) TiAl$_3$ matrix, (2) Ti-Al-C phase and (3) Al
**Slika 6:** SEM-posnetek TiAl$_3$-30 % TiC kompozita: (1) TiAl$_3$ matrica, (2) Ti-Al-C faza in (3) Al



**Figure 7:** Microstructure developed in TiAl$_3$-40 % TiC: (1) TiAl$_3$, (2) Ti-Al-C and (3) Al with up to 5 % of Ti
**Slika 7:** Mikrostruktura TiAl$_3$-40 % TiC-kompozita: (1) TiAl$_3$, (2) Ti-Al-C in (3) Al z do 5 % Ti

**Table 2:** Average room temperature tensile properties of various laboratory prepared TiAl$_3$-B$_4$C, TiC, TiB$_2$ composite samples
**Tabela 2:** Povprečne vrednosti mehanskih lastnosti (dobljene pri sobni temperaturi) kompozitov na osnovi TiAl$_3$, ojačanih z B$_4$C, TiC, TiB$_2$

| Volume fraction of ceramic reinforcement in TiAl$_3$ matrix $\varphi$/% | Retained porosity in sintered samples % | E/GPa | Tensile strength $\sigma$/MPa | 0.2 % tensile yield strength $\sigma$/MPa | Elongation in 50 mm $\Delta l$/% |
|---|---|---|---|---|---|
| 10-B$_4$C | 1.4±0.1 | 187±20 | 332±33 | 274±27 | 0.8±0.08 |
| 20-B$_4$C | 2.2±0.2 | 207±22 | 352±35 | 298±30 | 0.6 ±0.06 |
| 30-B$_4$C | 3.1±0.3 | 228±23 | 377±38 | 324±32 | 0.5±0.05 |
| 40-B$_4$C | 3.9±0.4 | 241±24 | 394±40 | 346±35 | 0.3±0.03 |
| 50-B$_4$C | 4.6±0.5 | 249±25 | 423±42 | 368±37 | 0.2±0.02 |
| 10-TiC | 1.1±0.1 | 218±22 | 374±37 | 329±33 | 0.5±0.05 |
| 20-TiC | 1.7±0.2 | 241±24 | 409±41 | 366±37 | 0.4±0.04 |
| 30-TiC | 1.9±0.2 | 270±27 | 453±45 | 400±40 | 0.3±0.03 |
| 40-TiC | 2.1±0.2 | 297±30 | 489±49 | 431±43 | 0.3±0.03 |
| 50-TiC | 2.4±0.2 | 321±31 | 513±51 | 467±47 | 0.2±0.02 |
| 10-TiB$_2$ | 3,9±0.4 | 149±15 | 298±30 | 209±21 | 0.7±0.07 |
| 20-TiB$_2$ | 4,6±0.5 | 167±17 | 318±32 | 229±23 | 0.5±0.05 |
| 30-TiB$_2$ | 5,0±0.5 | 188±19 | 331±33 | 248±25 | 0.4±0.04 |

yield. Reaction synthesis was performed at moderate temperature (1300 °C) for 2 h under a flowing atmosphere of Ar + 4 % H$_2$. The reaction product was milled in a conventional planetary mill, resulting in particulates with an average particle size of 10 μm.

Dense (>95 % of theoretical density) TiAl$_3$-based composites reinforced with 10–50 % of B$_4$C or TiC and 10–30 % of TiB$_2$ particles were successfully prepared by combining pressureless reaction synthesis and reaction sintering.

Irrespective of the ceramic reinforcement applied, dense composite samples reinforced with 10 % to 30 % of ceramic particulates were successfully obtained, revealing the significant industrial potential of this fabrication method. The retained porosity in sintered samples was less than 5 %.

The highest chemical affinity between ceramic reinforcement and matrix was observed in the TiAl$_3$-TiC and TiAl$_3$-B$_4$C systems, while TiAl$_3$-TiB$_2$ was chemically inert. The sintering of TiAl$_3$-TiC and TiAl$_3$-B$_4$C samples proceeds via formation of various secondary phases involved in the process of densification. Accordingly, TiAl$_3$-TiC and TiAl$_3$-B$_4$C samples with as much as 50 % of ceramic reinforcement were successfully pressurelessly sintered. On the other hand, sintering in TiAl$_3$-TiB$_2$ was achieved by the liquid phase (molten aluminium). Hence, in TiAl$_3$-TiB$_2$ samples with more than 30 % of ceramic particulates, under the applied sintering conditions Kirkendall diffusion pores were not reduced below 10 %.

Regarding the room temperature tensile properties of the composite samples, the highest improvement was obtained in TiAl$_3$-TiC and TiAl$_3$-B$_4$C samples with the highest amount (50 %) of ceramic reinforcement, while

strength of the fabricated composites were observed. At the same time, the highest improvement of tensile properties caused by reinforcement was observed in samples reinforced with TiC and B$_4$C and less in samples reinforced with TiB$_2$. Comparing the mechanical properties of composite samples with various volume fractions of ceramic particles in the matrix, it was found that Young's modulus, tensile strength and ultimate tensile strength increase while elongation decreases with an increasing fraction of ceramic reinforcement.

## 4 CONCLUSION

A TiAl$_3$ sintering mixture (87 % TiAl$_3$, 10 % Al and 1 % Ti) for composite fabrication was routinely reaction synthesized from the elements – (solid titanium particles and molten aluminium) with an almost 100 % reaction

in TiAl$_3$-TiB$_2$ samples the improvement was rather moderate. Generally, the improvement of tensile strength, tensile yield strength and modulus was found to correlate with amount of ceramic reinforcement in the matrix, irrespective of the sort of reinforcement. However, quite the opposite behaviour was found regarding elongation, where the introduction of ceramic particles into the intermetallic matrix led in all specimens to a significant reduction of ductility and toughness.

## Acknowledgment

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