The influence of reinforcing particles on mechanical and tribological properties and microstructure of the steel-TiB$_2$ composites

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**ABSTRACT**

**Purpose:** The present work aims to investigate the effect of the reinforcing ceramic particles on the mechanical and tribological properties and microstructure of the steel-TiB$_2$ composites.

**Design/methodology/approach:** The austenitic AISI316L stainless steel reinforced with 10 vol.% and 20 vol.% TiB$_2$ particles was produced using the high temperature-high pressure (HT-HP) method. The sintering process was carried out at pressure of 7.0±0.2 GPa and temperature of 1200°C for 60 seconds. Density of sintered materials was measured according to the Archimedes principle. Mechanical properties were determined by Vickers hardness and compression test. The friction coefficient was measured using ball-on-disk method. This tests were realized at room temperature. Microstructural observations were carried out using scanning electron microscopy.

**Findings:** The materials were characterized by very high level of consolidation, which was equal to 96% for composites with 10 vol.% and 20 vol.% TiB$_2$ particles. The results show that the composites exhibited higher Young’s modulus, Vickers hardness and compression strength when compared with conventionally austenitic AISI316L stainless steel. The addition of 20 vol.% of TiB$_2$ particles to steel caused significant reduction of the values of friction coefficient. The SEM studies of composites revealed TiB$_2$ phase along grain boundaries. In case of the composite with 20 vol.% TiB$_2$, the continuous layer of ceramic along the grain boundaries was observed.

**Practical implications:** The obtained test results may be used to optimize the sintering process of the steel-TiB$_2$ composites by high temperature methods. These results may be used to design new materials i.e. austenitic stainless steel reinforced with TiB$_2$ ceramic.

**Originality/value:** The work provides essential information on the effect of the TiB$_2$ particles on the mechanical and tribological properties of composites.

**Keywords:** Composites; Austenitic stainless steel; TiB$_2$; Sintering; Hardness; Compression strength; Friction coefficient

**Reference to this paper should be given in the following way:**
1. Introduction

Diboride titanium is considered as the best reinforcement for the following two reasons: firstly, it exhibits an high Young’s modulus (345-409 GPa), secondly, unlike most other ceramic reinforcements, which are reactive in molten iron, TiB$_2$ is stable in liquid Fe. Moreover, titanium diboride has the high melting point (3225°C), low density (4.5 g/cm$^3$), superior Vickers hardness (3400 HV), good thermal conductivity (~110 Wm$^{-1}$ K$^{-1}$ at 25°C) and high electrical conductivity (22 x10$^6$ Ω cm). TiB$_2$ ceramic is characterized by the good corrosion resistance, tribological properties and considerable chemical stability [1-3].

In recent years, many studies were reported on the use of TiB$_2$ ceramic as reinforcing phase in composites, among others having matrix of iron, aluminium, copper, cobalt and their alloys [4-11]. The authors focused mainly on studies on the effect of number of reinforcing phases on the properties and microstructure of the composites. These works were reported also on the effect of the various techniques for the fabrication of composites with TiB$_2$ particulate reinforcements [6, 9, 11, 12]. Anal et al. [5] attempted to synthesize TiB$_2$-reinforced Fe-based composites by aluminothermic reduction of Fe$_2$O$_3$, TiO$_2$ and B$_2$O$_3$. The examination also included the evaluation of the effect of heat treatment on the microstructure and abrasive wear resistance of the composite materials. The composite, synthesized by this process, possesses high hardness and high temperature stability. The abrasive wear resistance of the composite was compared with a standard wear resistant material, i.e. high-chromium white cast iron. It was found to be better than that of the standard material. The composite was also characterized by the good high temperature stability. Nahme et al. [13] studied the mechanical properties of the 316L austenitic stainless steel reinforced with 15 vol.% TiB$_2$ ceramic. The yield and tensile strength of composites were increased with increasing TiB$_2$ content. The additional TiB$_2$ particles were very effective to improve the wear resistance and ductility of austenitic stainless steel. The volumetric wear of the composite tended to decrease with increasing applied normal loads or with sliding velocity. The influence of the temperature and pressure on the properties and microstructure of the austenitic AISI 316L stainless steel reinforced with 1 vol.% and 2 vol.% TiB$_2$ ceramic was studied by Sulima et al. [16, 17]. They reported that the application of higher temperature and higher pressure resulted in higher hardness and Young modulus. All specimens which were HT-HP sintered at temperature of 850°C-1300°C and pressure of 5 GPa and 7 GPa show densities between 95-100% of the theoretical density. The microstructural investigations indicated that the TiB$_2$ ceramic was distributed along the grain boundaries.

In the present paper, the influence of 10 vol.% and 20 vol.% of TiB$_2$ ceramic on the mechanical and tribological properties and microstructure of the composites was studied.

2. Experimental procedure

In the present investigations, the TiB$_2$ powder (H.C. Strack, average grain size below 2.5-3.5 µm, purity of 99.9%) and AISI 316L steel powder (Hoganas, average grain size of about 45 µm) were used. The chemical composition of the applied stainless steel powders is following: 16.9 wt % Cr, 12.15 wt % Ni, 2.1 wt % Mo, 0.9 wt % Si, 0.7 wt % Mn, 0.03 wt % C and balance Fe.

Two different composites were studied: AISI 316L stainless steel reinforced with 10 vol.% and 20 vol.% TiB$_2$. The composites were produced by mixing the powders in a turbula mixer for 6 hours. The resulting mixtures were formed into discs (15 mm in diameter, 5 mm high) by pressing in a steel matrix under pressure of 200 MPa. For the densification of materials the high temperature-high pressure (HT-HP) Bridgman type apparatus was used. The samples were sintered at temperature of 1200°C and pressure of 7 ± 0.2 GPa for 60 seconds.

The densities of the sintered specimens were measured by Archimedes water immersion method. Young’s modulus of the samples was measured basing on the velocity of the ultrasonic waves transition through the sample using ultrasonic flaw detector Panametrics Epoch III. The accuracy of the calculated Young’s modulus was estimated at 2 %.

Sintered samples were prepared by lapping on a cast iron plate with diamond paste and etching. The Vickers indentation tests were performed on compacts using FM-7 microhardness tester. The applied load for non-graded materials was 0.98 N. The compression test was carried out using INSTRON TT-DM machine at strain rates of about 10$^{-3}$ s$^{-1}$. This tests were conducted with specimen of 3 mm in diameter and 4.5 mm in length.

Fig. 1. Schematic of the ball-on-disk wear test system: 1 – ball; 2 – disk, $F_n$ is the normal force on the ball.
Tribological tests were carried out using the UMT-2T (producer CETR, USA) Ball-on-Disk tribotester. The schematic diagram of this method is present in Figure 1. Ball was made of WC with diameter of 3.175 mm. The tests were conducted at room temperature under load of 100 N for sliding speeds of 0.1 m/s and a total sliding distance of 100 m for test duration of 1000 s.

The microstructures and chemical composition were observed using Hitachi S-3400N scanning electron microscope (SEM) with Energy Depressive Spectrometer EDS (NORAN System Six). The phase characterisation of materials was carried out by X-ray diffraction using Cu Kα radiation and by energy dispersive X-ray microanalyser (EDS).

### 3. Results and discussion

The results of studies on some physical and mechanical properties of the austenitic AISI 316L stainless steel and composites with 10 vol.% and 20 vol.% TiB₂ ceramic are presented in Table 1 and Figure 2.

**Table 1.** The properties of austenitic AISI 316L stainless steel and composites reinforced with 10 vol.% and 20 vol.% TiB₂ obtained at temperature of 1200°C and pressure of 7 ± 0.2 GPa.

<table>
<thead>
<tr>
<th>Samples</th>
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The composites with 10 vol.% and 20 vol.% TiB₂ reached density of 7.29 g/cm³ and 6.99 g/cm³, respectively. This values corresponding to 96% of the theoretical density (7.61 g/cm³ and 7.27 g/cm³). It was observed that the density of composites decrease with the increasing of TiB₂ phase content. It results from much lower density of titanium diboride then stainless steel used for sintering process.

The results of the studies (Fig. 2) indicated that the Young modulus and the hardness of composites increase with titanium diboride content. The highest value of the Young modulus and hardness have composites with 20 vol.% TiB₂, they are equal to 225 GPa and 460 HV1, respectively. In the case of composites with 10 vol.% TiB₂, the Young modulus and hardness are 207 GPa and 402 HV1. For comparison, the Young modulus and Vickers hardness of the austenitic AISI 316L stainless steel are 178 GPa and 344 HV1, respectively.

**Fig. 3.** Friction coefficient for austenitic AISI 316L stainless steel and composites reinforced with 10 vol.% and 20 vol.% TiB₂.

Friction coefficient (μ) measured by using ball-on-disc method is presented in Figure 3. The results showed that the friction coefficient of the composites decreases with increasing TiB₂ content. In the case of the austenitic AISI 316L stainless steel and composite with 10 vol.% TiB₂ the values of the friction coefficient were very similar: 0.5 and 0.53, respectively. However, the value of the friction coefficient for the composite with 20 vol.% TiB₂ decreases to 0.37.

**Fig. 4.** The results of the compression tests for the austenitic AISI 316L stainless steel and steel-TiB₂ composites.
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This is an effect of additions of the TiB₂ particles which caused the increase of Vickers hardness of composites. In the...
composites, material removal is slow because the hard TiB₂ particles resist the plastic deformation and provide protection to the steel matrix. In case of the austenitic AISI316L stainless steel, the WC ball can penetrate easily during sliding.

Figure 4 shows the influence of content of TiB₂ phase on the compression strength. The increase of the compression strength with the increase of TiB₂ phase content was observed. The following values of the compression strength were obtained for the composites with 10 vol. % and 20 vol. % TiB₂: 1250 MPa and 1350 MPa, respectively. For the comparison, the compression strength of the sintered AISI316L stainless steel was 1092 MPa.

The microstructure of the austenitic AISI 316L stainless steel and composites with 10 vol.% and 20 vol. % TiB₂ after etching is given in Figure 5. The EDS analysis revealed the presence of TiB₂ phase (black areas) distributed along grain boundaries (Figs. 6, 8). The results of analysis obtained from the X-ray diffraction spectra were consistent with microscopic examination (Fig. 7). The samples with 10 vol.% TiB₂ were characterized by irregular distribution of ceramic phase in the matrix. Large agglomerates of the ceramic phase were detected at the grain boundaries (Fig. 5b). However, in the case of the composite with 20 vol.% TiB₂, the continuous layer of the ceramic phase along the grain boundaries was observed.

![Image](image_url)

Fig. 8. Results of EDS microanalysis of sintered composites – SEM images with EDS spectra respectively for: a) 10 vol.% TiB₂ (after etching), b) 20 vol.% TiB₂ (after etching)
4. Conclusions

Two variants of the steel-TiB₂ composites with 10 vol.% and 20 vol.% TiB₂ phase were obtained by high temperature-high pressure (HT-HP) method. The results of investigations of these composites were compared with the results on the matrix material.

The addition of the TiB₂ particles into the austenitic AISI 316L stainless steel is a good route to improve the mechanical properties of these materials. The increasing Vickers hardness and Young’s modulus of the composites with increasing the TiB₂ phase content was observed. The resulting composite showed the increase in the compression strength when compared to the unreinforced alloy. Tribological measurements showed that a friction coefficient of the composites increased with the increasing TiB₂ content.

The highest properties were obtained for the austenitic AISI 316L stainless steel reinforced with 20 vol.% TiB₂ ceramics. For this composite, the Young modulus, Vickers hardness, compression strength and friction coefficient achieved values of: 225 GPa, 460 HV1, 1350 MPa and 0.37, respectively.

References