

The influence of reinforcing particles on mechanical and tribological properties and microstructure of the steel-TiB₂ composites

I. Sulima ^{a,*}, L. Jaworska ^{a,b}, P. Wyżga ^b, M. Perek-Nowak ^c

^a Institute of Technology, Pedagogical University,
ul. Podchorążych 2, 30-084 Kraków, Poland

^b Institute of Advanced Manufacturing Technology,
ul. Wrocławska 37a, 30-011 Kraków, Poland

^c Faculty of Non-ferrous Metal, University of Science and Technology,
Al. Mickiewicza 30, 30-065 Kraków, Poland

* Corresponding author: E-mail address: isulima@ap.krakow.pl

Received 16.07.2011; published in revised form 01.09.2011

Properties

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-TiB₂ composites.

Design/methodology/approach: The austenitic AISI316L stainless steel reinforced with 10 vol.% and 20 vol.% TiB₂ 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. These 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.% TiB₂ 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 TiB₂ particles to steel caused significant reduction of the values of friction coefficient. The SEM studies of composites allowed to reveal TiB₂ phase along grain boundaries. In case of the composite with 20 vol.% TiB₂, 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-TiB₂ composites by high temperature methods. These results may be used to design new materials i.e. austenitic stainless steel reinforced with TiB₂ ceramic.

Originality/value: The work provide essential information on the effect of the TiB₂ particles on the mechanical and tribological properties of composites.

Keywords: Composites; Austenitic stainless steel; TiB₂; Sintering; Hardness; Compression strength; Friction coefficient

Reference to this paper should be given in the following way:

I. Sulima, L. Jaworska, P. Wyżga, M. Perek-Nowak, The influence of reinforcing particles on mechanical and tribological properties and microstructure of the steel-TiB₂ composites, Journal of Achievements in Materials and Manufacturing Engineering 48/1 (2011) 52-57.

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/cm³), superior Vickers hardness (3400 HV), good thermal conductivity ($\sim 110 W m^{-1} K^{-1}$ at 25°C) and high electrical conductivity ($22 \times 10^6 \Omega cm$). 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 use 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_2O_3 , TiO_2 and B_2O_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 were 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 taking the high temperatures into consideration. The improvement of the Young's modulus (218 GPa), tensile strength (885 MPa) and compression strength (1800 MPa) was obtained. However, the strain (elongation) decreased strongly from 45% to about 6% for the reinforced materials. The microstructural investigations indicated the homogeneous distribution of TiB_2 ceramic in the matrix. The presence of the Cr/Mo-rich phases near the TiB_2 grains was observed. Besides, Tjong *et al.* [14] investigated the properties of the AISI 304 stainless steel and composites reinforced with various volume fractions of TiB_2 particles (5 vol.%, 10 vol.%, 15 vol.% and 20 vol.%). The materials were fabricated by hot-isostatic pressing. It was showed, that with increasing TiB_2 content the hardness and tensile strength improved, but dropped the plasticity. Pin-on-disc abrasive measurements show that the wear resistance of 304 SS improves dramatically with increasing TiB_2 content. Tjong and Lau [15] investigated the dry sliding wear behaviour of AISI 304 stainless steel reinforced with 20 vol.% TiB_2 particles. The yield and tensile strengths of composites were determined to be 531 MPa and 619 MPa, respectively. The examinations showed that the addition of TiB_2 particles was 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 98-100% 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.

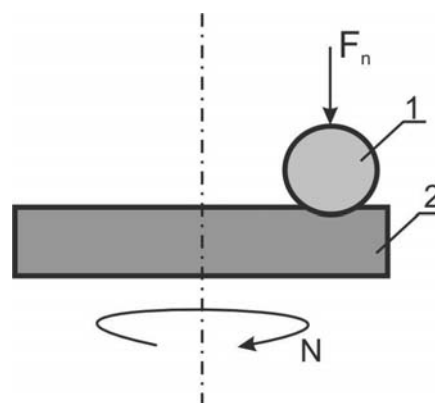


Fig. 1. Schematic of the ball-on-disk wear test system: 1 – ball; 2 – disk, F_n is the normal force on the ball

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 Dispersive Spectrometer EDS (NORAN System Six). The phase characterisation of materials was carried out by X-ray diffraction using Cu K_{α} 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.% TiB_2 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.% TiB_2 obtained at temperature of $1200^{\circ}C$ and pressure of 7 ± 0.2 GPa

Samples	Density (ρ_0) [g/cm ³]	ρ_0/ρ_{Teor}	Poisson's ratio
AISI 316L	7.54	---	0.3
AISI 316L + 10 vol.% TiB_2	7.29	96	0.28
AISI 316L + 20 vol.% TiB_2	6.99	96	0.27

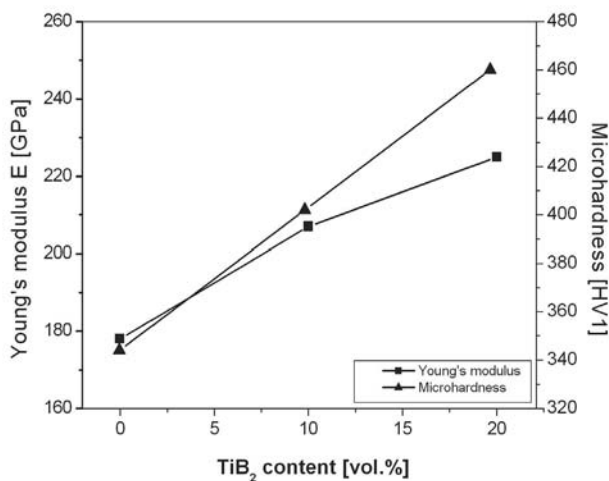


Fig. 2. The results of the microhardness and Young's modulus of composites as a function of TiB_2 content

The composites with 10 vol.% and 20 vol.% TiB_2 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 TiB_2 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.% TiB_2 , they are equal to 225 GPa and 460 HV1, respectively. In the case of composites with 10 vol.% TiB_2 , 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.

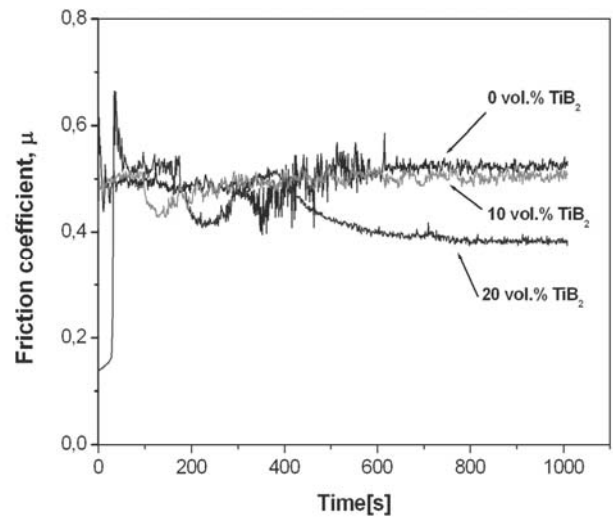


Fig. 3. Friction coefficient for austenitic AISI 316L stainless steel and composites reinforced with 10 vol.% and 20 vol.% TiB_2

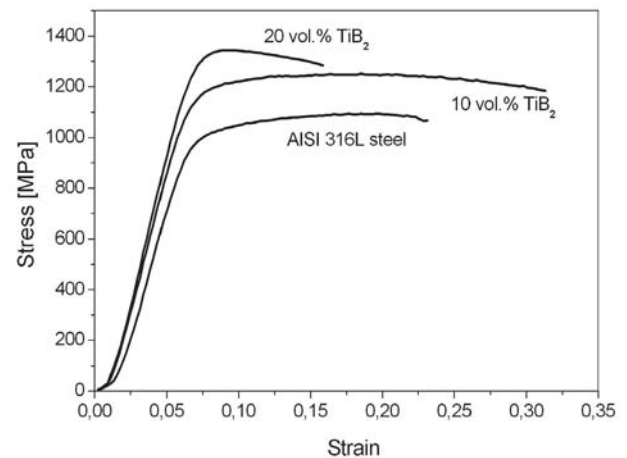


Fig. 4. The results of the compression tests for the austenitic AISI 316L stainless steel and steel- TiB_2 composites

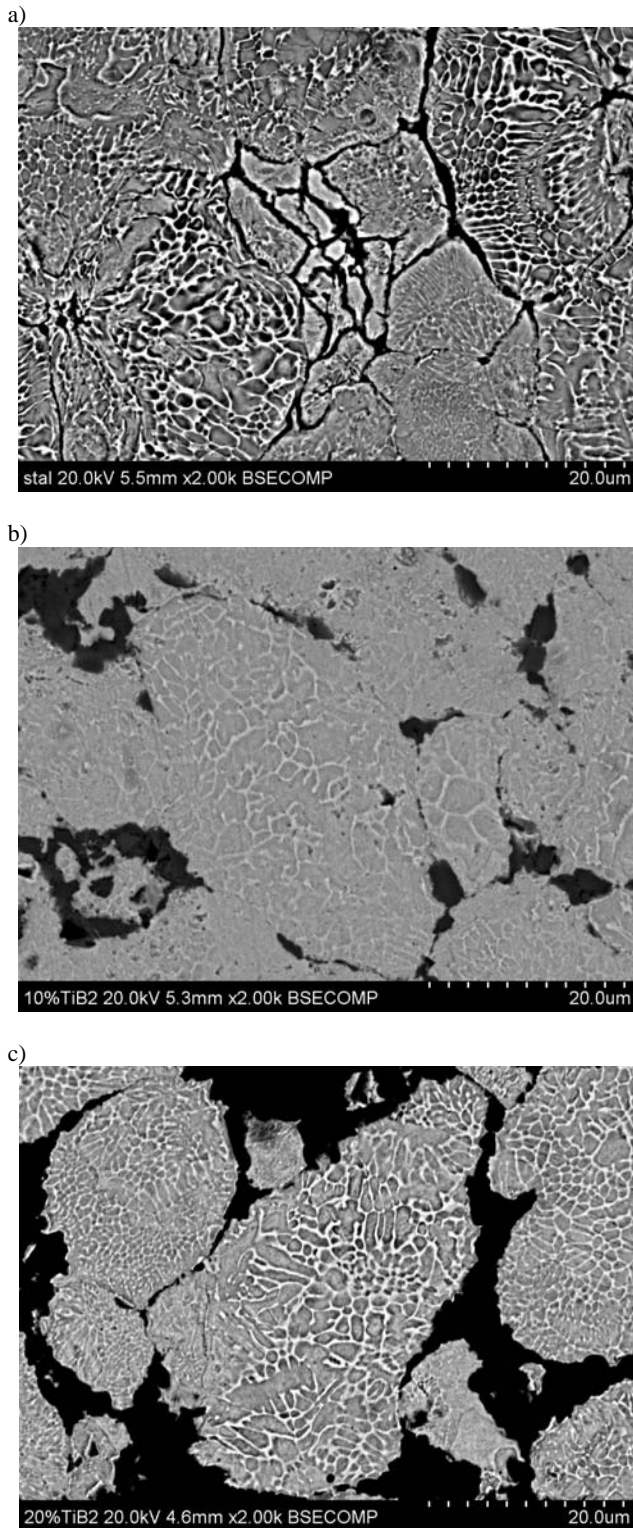


Fig. 5. The SEM microstructure of the a) austenitic AISI 316L stainless steel and composites with: b) 10 vol.% TiB₂ and c) 20 vol.% TiB₂ (after etching)

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 AISI316L 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.

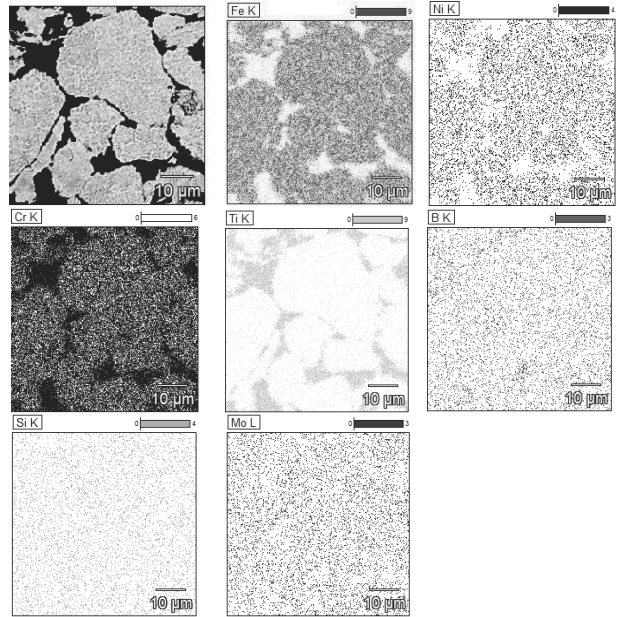


Fig. 6. SEM image of the composite with 20 vol.% TiB₂ and corresponding distribution maps of elements: Fe, Ni, Cr, Ti, Mo, B and Si

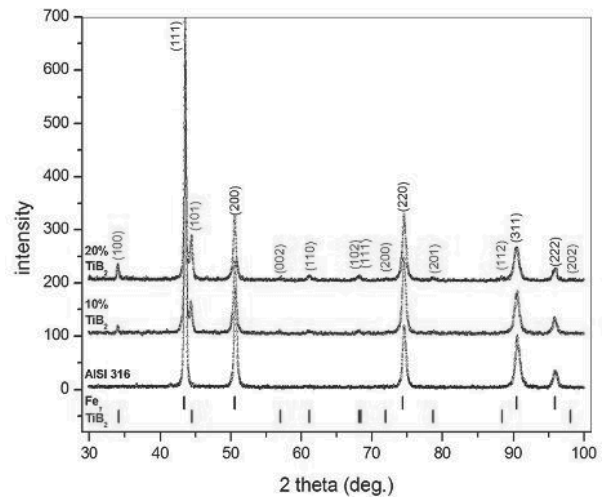


Fig. 7. X-ray diffraction pattern of the sintered materials obtained at temperature of 1200°C and pressure of 7 ± 0.2 GPa

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 TiB_2 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 TiB_2 phase on the compression strength. The increase of the compression strength with the increase of TiB_2 phase content was observed. The following values of the compression strength were obtained for the composites with 10 vol. % and 20 vol. % TiB_2 , 1250 MPa and 1350 MPa, respectively. For the comparison, the compression strength of the sintered AISI 316L stainless steel was 1092 MPa.

The microstructure of the austenitic AISI 316L stainless steel and composites with 10 vol.% and 20 vol. % TiB_2 after etching is given in Figure 5. The EDS analysis revealed the presence of TiB_2 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.% TiB_2 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.% TiB_2 , the continuous layer of the ceramic phase along the grain boundaries was observed.

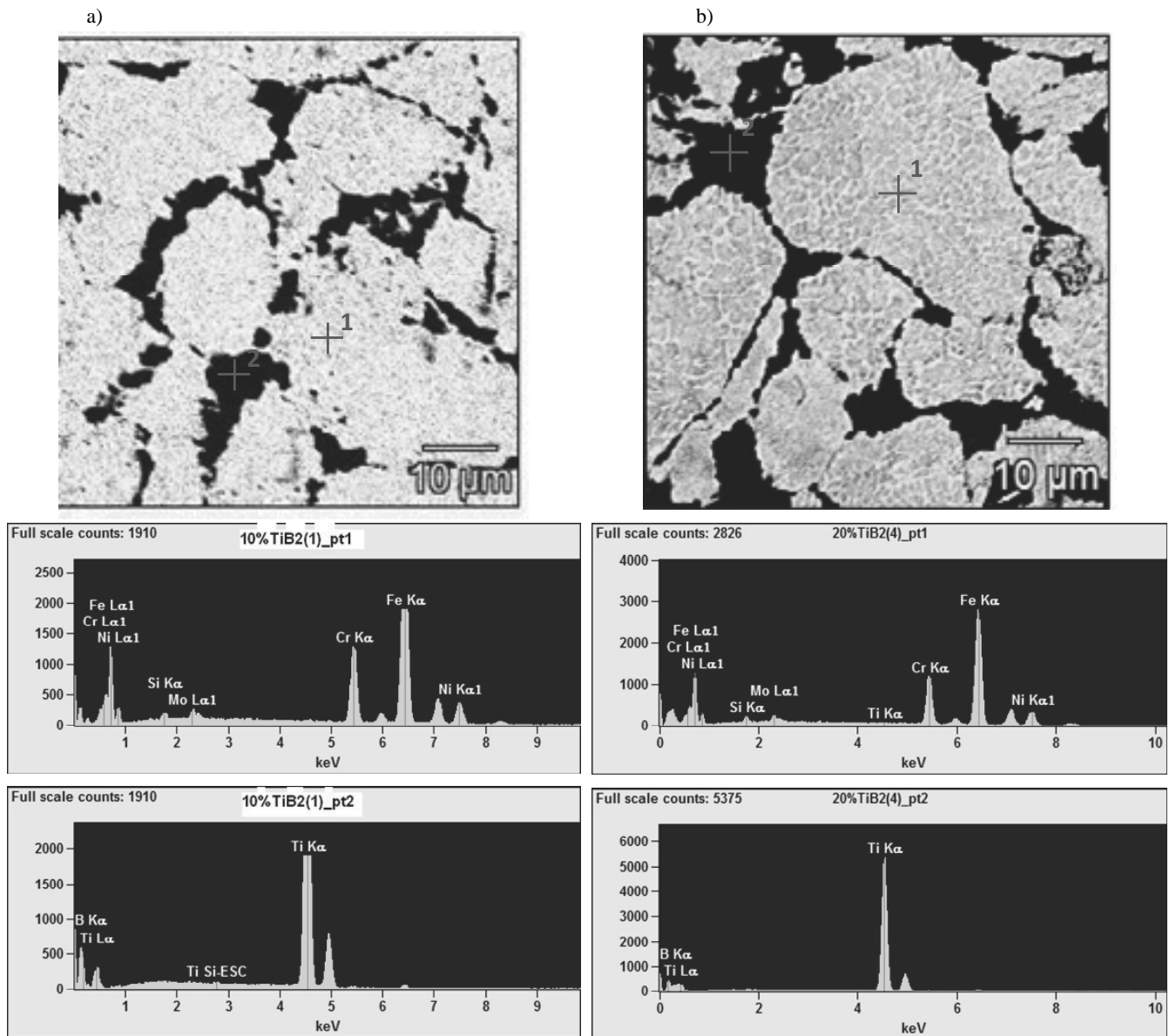


Fig. 8. Results of EDS microanalysis of sintered composites – SEM images with EDS spectra respectively for: a) 10 vol.% TiB_2 (after etching), b) 20 vol.% TiB_2 (after etching)

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 this 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

- [1] J.F. Shackelford, W. Alexander (Eds.), CRC Materials Science and Engineering Handbook, Third Edition, CRC Press, 2001, 509.
- [2] C. Subramanian, T.S.R.Ch. Murthy, A.K. Suri, Synthesis and consolidation of titanium diboride, *International Journal of Refractory Metals and Hard Materials* 25 (2007) 345-350.
- [3] W. Weimin, F. Zhengyi, W. Hao, Y. Runzhang, Influence of hot pressing sintering temperature and time on microstructure and mechanical properties of TiB₂ ceramics, *Journal of the European Ceramic Society* 22 (2002) 1045-1049.
- [4] E. Fraś, A. Janas, A. Kolbus, Cast in situ composite reinforced with titanium boride particles, *Composites 1* (2002) 23-27.
- [5] A. Anal, T.K. Bandyopadhyay, K. Das, Synthesis and characterization of TiB₂-reinforced iron-based composites, *Journal of Materials Processing Technology* 172 (2006) 70-76.
- [6] S.C. Tjong, K.F. Tam, Mechanical and thermal expansion behavior of hiped aluminium-TiB₂ composites, *Materials Chemistry and Physics* 97 (2006) 91-97.
- [7] A. Pettersson, P. Magnusson, P. Lundberg, M. Nygren, Titanium-titanium diboride composites as part of a gradient armour material, *International Journal of Impact Engineering* 32 (2005) 387-399.
- [8] A. Farid, S. Guo, F. Cui, P. Feng, T. Lin, TiB₂ and TiC stainless steel matrix composites, *Materials Letter* 61 (2007) 189-191.
- [9] A. Farid, Microstructure evolution and wear properties of in situ synthesized TiB₂ and TiC reinforced steel matrix composites, *Journal of Alloys and Compounds* 459 (2008) 491-497.
- [10] D.H. Bacon, L. Edwards, J.E. Moffatt, M.E. Fitzpatrick, Synchrotron X-ray diffraction measurements of internal stresses during loading of steel-based metal matrix composites reinforced with TiB₂ particles, *Acta Materialia* 59 (2011) 3373-3383.
- [11] I. Sulima, P. Figiel, M. Suśniak, M. Świątek, Sintering of TiB₂-Al composites using HP-HT method, *Archives of Materials Science and Engineering* 33/2 (2008) 117-206.
- [12] L. Jaworska, L. Stobierski, A. Twardowska, D. Królicka, Preparation of materials based on {Ti-Si-C} system using high temperature - high pressure method, *Proceedings of the 13th International Scientific Conference "Achievements in Mechanical and Materials Engineering" AMME'2005, Gliwice - Wisła, 2005*, 275-278.
- [13] H. Nahme, E. Lach, A. Tarran, Mechanical property under high dynamic loading and microstructure evaluation of TiB₂ particle-reinforced stainless steel, *Journal of Materials Science* 44 (2009) 463-468.
- [14] S.C. Tjong, K.C. Lau, Abrasion resistance of stainless-steel composites reinforced with hard TiB₂ particle, *Composites Science and Technology* 60 (2000) 1141-1146.
- [15] S.C. Tjong, K.C. Lau, Sliding wear of stainless steel matrix composite reinforced with TiB₂ particles, *Materials Letters* 41 (1999) 153-158.
- [16] I. Sulima, P. Klimczyk, P. Hyjek, The influence of the sintering conditions on the properties of the stainless steel reinforced with TiB₂ ceramics, *Archives of Materials Science and Engineering* 39/2 (2009) 103-106.
- [17] I. Sulima, P. Figiel, L. Jaworska, P. Hyjek, The properties of AISI 316L stainless steel reinforced with TiB₂ ceramics sintered by the HT-HP process, *Materials Engineering* 1 (2011) 40-43.