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INFLUENCE OF PROCESSING PARAMETERS AND DIFFERENT CONTENT OF TiB_2 CERAMICS ON THE PROPERTIES OF COMPOSITES SINTERED BY HIGH PRESSURE - HIGH TEMPERATURE (HP-HT) METHOD

WPŁYW PARAMETÓW PROCESU ORAZ CERAMIKI TiB2 NA WŁAŚCIOWSCI KOMPOZYTÓW SPIEKANYCH METODĄ WYSOKOCIŚNIENIOWĄ (HP-HT)

In this paper the properties of the austenitic stainless steel reinforced with various volume fractions of TiB_2 ceramics have been studied. The high pressure- high temperature (HP-HT) method of sintering was applied to the formation of composites. Samples were sintered at pressure of 5 and 7 ±0.2 GPa and temperatures of 1273 K and 1573 K. For the tested materials, the relative density, Young's modulus and hardness were measured. In order to investigate the structure changes, the scanning electron microscope was used. The obtained results show that the temperature and pressure influence on the mechanical and physical properties of the investigated composites.

Keywords: high pressure-high temperature (HP-HT) sintering, titanium diboride (TiB_2), austenitic stainless steel, properties

W prezentowanej pracy zbadano właściwości stali austenitycznej z różnym udziałem objętościowym ceramiki TiB_2. Do wytworzenia spieków kompozytowych proszków zastosowano spiekanie wysokociśnieniowe HP-HT. Próbki były spiekane przy ciśnieniu 5 oraz 7±0.2 GPa i temperatururze 1273 K oraz 1573 K. Kompozyty poddano badaniom gęstości, modułu Younga oraz twardości. Badania mikrostruktury przeprowadzono za pomocą skaningowej mikroskopii elektronowej. Uzyskane wyniki badań wykazały, że temperatura oraz ciśnienie wpływają na mechaniczne oraz fizyczne wł aściwości materiałów kompozytowych.

1. Introduction

Powder metallurgy (PM) offers effective technique for the fabrication of metal-ceramic composites and provide an unifrom distribution of particles in the matrix. Austenitic stainless steels are widely used as engineering materials in the industry owing to their excellent corrosion and oxidation resistance, high work-hardening, and good formability [1,2]. One of the major drawbacks of the austenitic stainless steels is their low yield strength, usually being 150-300 MPa in the annealed state, what limits their technological applications. Additionally, the AISI 316L stainless steel materials provide limited wear resistance due to their low hardness [1-3]. The incorporation of ceramic particles into steel matrix can improve the hardness and wear resistance in various ways. Therefore, steel-matrix composites with ceramic-particles reinforcement, which combine the toughness of metal and the hardness of ceramic, have been the subject of intensive investigation [4-8]. Ni *et al*. [6] reported that the TiC addition to the austenitic stainless steel bring beneficial effects on mechanical properties and oxidation resistance. It was showed, that both at ambient and elevated temperature, tensile strengths of austenitic stainless containing 5 vol.% TiC were notably higher than those of the matrix without TiC addition. Moreover, creep resistance of austenitic-stainless steels was also significantly increased by TiC addition at the elevated temperature of 923 K. Oxidation test at 1073 K revealed that TiC addition to the austenitic stainless steels raised the oxidation resistance of the steel remarkably. Akhtar [9] produced steel-matrix composite reinforced with TiB_2 and TiC reinforcements (30 to 70 wt. %) through the synthesis reaction from Ti, C and FeB. Author reported on the reciprocating sliding wear behavior of the 465-stainless-steel composite reinforced with the *in situ* synthesized TiB_2 and TiC particles. The results of this test showed that the wear loss decreased with increase in the reinforcement content. The wear mechanisms were polishing wear and microploughing for the composites containing high volume fraction of the reinforcement, whereas microploughing and grooving were the dominant wear mechanisms for the composites containing low volume fraction of the reinforcing particles. Few studies have reported the use to TiB_2 reinforcement in stainless-steel-matrix [10-13]. Titanium diboride is also an attractive ceramic material acting as a reinforcing phase in the stainless-steel-matrix composites. Among various ceramic particulates, titanium boride is expected to be one of the best reinforcements for steel matrix due to high melting tempera-

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ture, low density, outstanding tribological properties and good compatibility with the steel matrix. Its hardness (3400 HV) is greater than that of the more commonly used WC (2000 HV) and is almost as high as that of SiC (3500 HV) [14]. For example, Tjong and Lau [11] investigated composites reinforced with various volume fractions of TiB_2 particles which were fabricated by hot-isostatic pressing. The addition of the hard ceramic particles improve the mechanical strength of 304 stainless steels at the expense of its ductility. In general, the 0.2% offset yield strength of composites tend to increase with increasing TiB_2 volume content. Also, the hardness of stainless alloy appears to increase with increasing TiB_2 volume content.

In the present investigations, TiB_2 particles reinforced austenitic stainless steel has been fabricated using the high temperature-high pressure sintering and its microstructure, selected physical and mechanical properties have been studied.

2. Experiment

The starting materials used in this work were 99.9 wt.% TiB_2 with particle size of 2.5-3.5 μm (H.C. Starck) and commercial AISI 316L austenitic stainless steel with particle size of 25 μm (KAMB Import-Export). The stainless-steel powder have the chemical composition as follows: 17.20 wt.% Cr, 12.32 wt.% Ni, 2.02 wt.% Mo, 0.43 wt.% Mn, 0.89 wt.% Si, 0.03 wt.% S, 0.028 wt.% P, 0.03 wt.% C and balance of Fe. The composites were produced by mixing the powders in a turbula mixer for 12 hours.

Two materials were studied:

- AISI 316L stainless steel reinforced with 6 vol.% of TiB_2 particles
- AISI 316L stainless steel reinforced with 8 vol.% TiB_2 particles.

The composites were consolidated using the high pressure – high temperature (HP-HT) Bridgman type apparatus. Figure 1 presents the scheme of Bridgman-type HP-HT apparatus. The resulting mixtures were formed into discs (15 mm in diameter, 5 mm in high) by pressing in a steel matrix under the pressure of 200 MPa. In Table 1, the HP-HT sintering parameters for composites are showed.

TABLE 1

Parameters of sintering of composites using HP-HT method

Samples	Temperature [K]	Pressure GPa	Heating time [s]	Hold time [s]	Cooling time [s]
AISI 316L+ 6 vol.% TiB_2 and AISI 316L+ 8 vol.% TiB_2	1273	5	5	60	5
	1273	7	5	60	5
	1573	5	5	60	5
	1573	7	5	60	5

The application of the HP-HT sintering permit achieving simultaneously extreme high pressure (of the order of 100 GPa) and high temperatures during the process. Therefore, the sintering process proceeds much faster (usually in several minutes) than free sintering which usually takes few to several hours. The obtained sinters are characterized by a degree of densification reaching almost 100% and isotropic properties. The use of such conditions can also reduce the diffusion of particles and prevent grains growth [15,16].

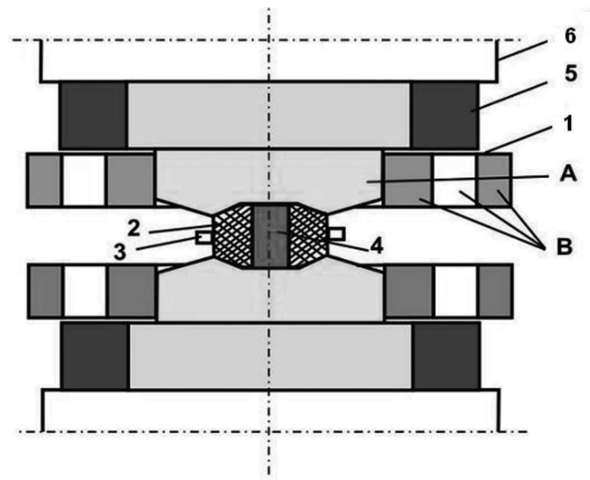


Fig. 1. Scheme of Bridgman-type, toroidal HP-HT apparatus: 1 – anvil (A – central part made of sintered carbides, B – supporting steel rings), 2 – pyrophyllite container, 3 – pyrophyllite gasket, 4 – material for sintering, 5 – punch, 6 – supporting plate [16]

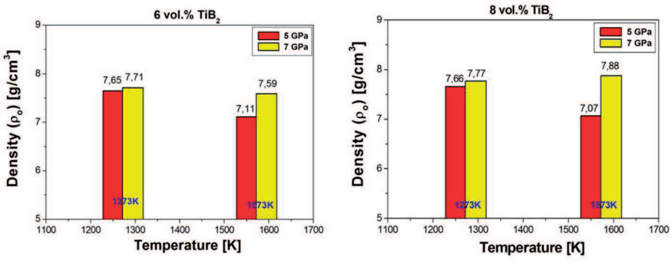
Density was determined by weighing in air and water using Archimedes method. Young's modulus of the composites were measured basing on the velocity of the ultrasonic waves transition through the sample using ultrasonic flaw detector Panametrics Epoch III. The accuracy of calculated Young's modulus is estimated at 2%. The Vickers indentation tests were performed on a polished surface of samples using FM-7 microhardness tester. Five hardness measurements with indentation load of 2.94 N were carried out for each sample. Standard deviations of HV0. 3 values were no more than 4% of the average values.

For morphological characterization of the composites JEOL JSM 6610LV scanning electron microscope (SEM) were used. EDS technique (AZtec) was applied to determine the chemical composition of sintered materials. Also, the phase compositions of selected samples were analyzed by X-ray diffraction (XRD) using Cu Kα radiation with a scintilation detector (Brucker Discover D8).

3. Results and discussion

The densification data of the composites at various sintering temperatures and pressures is presented in Fig. 2. The results reveal that both composites with 6 vol.% TiB_2 and 8 vol.% TiB_2 exhibit very high densification at the sintering temperatures of 1273 K (at pressure of 5 and 7 GPa) and 1573 K (at pressure of 7 GPa). The values of relative density of the composites obtained in this sintering parameters corresponding to 98-100% of the theoretical density (7.75 g/cm^3 and 7.68 g/cm^3, respectively). The exceptions are following conditions of sintering: temperature of 1573K and pressure of 5 GPa. Under these conditions lower relative density of 7,11g/cm^3 and 7.07 g/cm^3 was received for composites with

6 vol.% TiB_2 and 8 vol.% TiB_2, respectively. These values correspond to the 92% of the theoretical density.

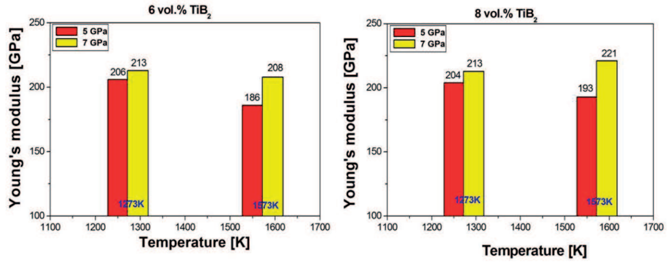


a) b)

Fig. 2. Densities of composites as a function of composition, temperature and pressure of sintering: a) 6 vol.% TiB_2 and b) 8 vol.% TiB_2

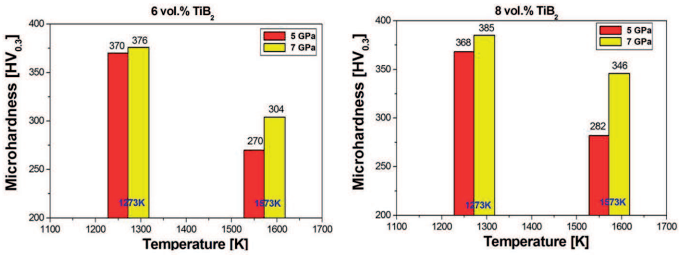
Fig. 3 shows the relationship between sintering parameters and Young's modulus. It is interesting that for lower temperature of sintering the higher values of the Young's modulus were obtained (for example Fig. 3a). The exception is the use of the temperature of 1573 K and pressure 7 GPa, because for this conditions of sintering the Young modulus reaches the highest values of 221 GPa. Generally, the increase in pressure causes an increase in the value of the Young's modulus when used with the same sintering temperature. The higher values of Young modulus were observed for samples with higher content of TiB_2 which were sintered at the temperature of 1573 K. The composites with 8 vol.% TiB_2 received the values of Young modulus: 193 GPa and 221 GPa at pressure of 5 and 7 GPa (1573 K), respectively.

Variations in Vickers hardness of composites with different content of TiB_2 are presented in Figure 4. It was found that the sintering temperature has a significant effect on the hardness of the composites. The higher value of hardness have composites which were sintered at the lower temperature. In the case of composites with 6 vol.% and 8 vol.% TiB_2 the higher values of hardness are 376 GPa and 385 GPa, respectively. The application of higher temperature causes a drop in hardness, especially at pressure of 5 GPa. The increase of TiB_2 content did not induce significant increase in Vickers hardness; standard deviations of HV0.3 values were no more than 4% of the average values. The results of hardness for the composites with 6 vol.% and 8 vol.% TiB_2 are very similar and are within the limits of measuring error. The increase of hardness with the increase of TiB_2 content was observed only at temperature of 1573K and pressure of 7 GPa. The hardness of the composites with 6 vol.% TiB_2 was measured as 304 HV0.3, which rose to 346 HV0.3 with the addition of 8 vol.% TiB_2. Generally, the worst combination of physical and mechanical properties was achieved at temperature of 1573 K and pressure of 5 GPa. The results suggest that the application of lower temperature is sufficient for good quality HP-HT sintering of the composites with 6 vol.% and 8 vol.% TiB_2. The use of lower temperature can also reduce the cost of the sintering process. Further studies will be carried out to evaluate the effects of sintering parameters and different content of TiB_2 ceramics on mechanical and tribological properties of composites.



a) b)

Fig. 3. Effect of sintering parameters and different content of TiB_2 particles in the matrix on Young's modulus of composites: a) 6 vol.% TiB_2 and b) 8 vol.% TiB_2



a) b)

Fig. 4. Effect of processing parameters and different content of TiB_2 particles in the matrix on microhardness of composites: a) 6 vol.% TiB_2 and b) 8 vol.% TiB_2

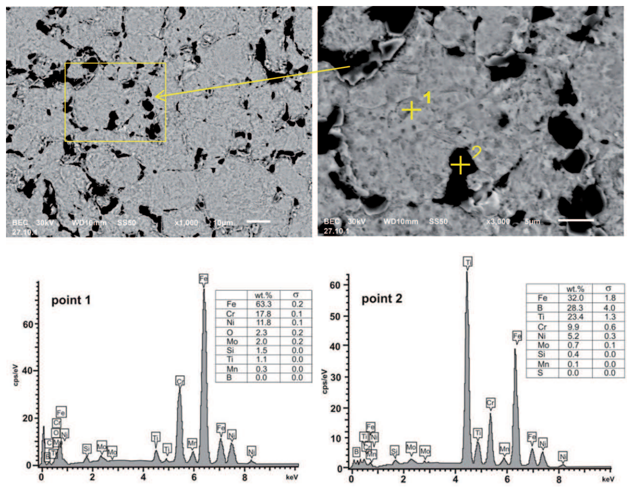


Fig. 5. The SEM micrograph of the composites with 6 vol.% TiB_2 after HP-HT sintering at temperature of 1273K and pressure of 7±0.2 GPa

Typical microstructures of the HP-HTed composites are presented in Fig. 5 and 6. A more detailed EDS study were required to confirm only the presence of TiB_2 particles within the steel matrix (point 2 in the Fig. 5). SEM observation showed that all composites had uniform dispersion of fine TiB_2 particles in austenitic steel matrix. The TiB_2 particle size is about 2-5μm in diameter although some particles are much smaller. No porosity was observed in the microstructure, what results in very high densification of the composites. The phase composition of the composites was identified by X-ray diffraction analysis (Fig. 7). According to the XRD results, it is evident that the dark particulates can be identified as TiB_2. The EDS analysis results (Fig. 8) are in agreement with the XRD results. In order to explain the influence of sintering parameters on the properties of composites, the detailed structural investigations should be carried out. Taking into account the results of authors [17,18], the formation of a liquid phase during sintering at temperature of 1573 K is possible. It is formed by the eutectic reaction between solid solution (Fe-Mo) and borides.The phenomena occurring during the sintering process affect the properties of the materials.

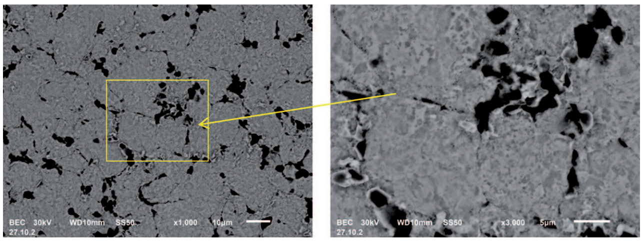


Fig. 6. The SEM micrograph of the composites with 8 vol.% TiB_2 after HP-HT sintering at temperature of 1273K and pressure of 7±0.2 GPa

According to the authors [19], the distribution of the added ceramic particles is very importance to the mechanical properties of the composite. The homogeneous distribution of the reinforcement ensures isotropic properties and uniform distribution of stresses into the material. On the other hand, clusters of reinforcing particles, resulting from insufficient mixing or electrostatic phenomena, influence on the deterioration of the properties of the composites by serving as sites for microporosity and crack initiation. Additionally, Pagounis *at al.* [20] revealed that the use of fine matrix powder causes a more uniform distribution of reinforcement.

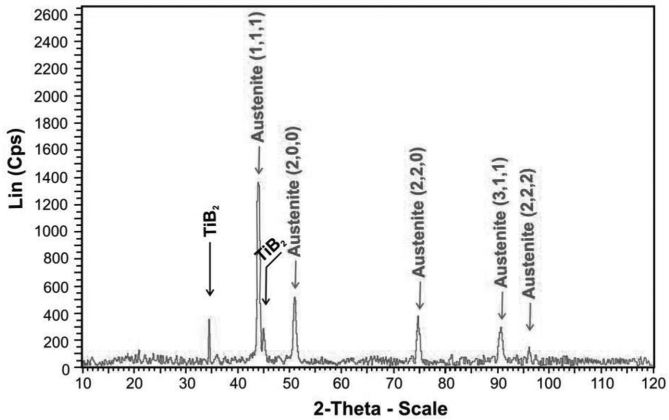


Fig. 7. XRD patterns of the composites with 8 vol.% TiB_2 particles (temperature of 1273K, pressure of 7±0.2 GPa)

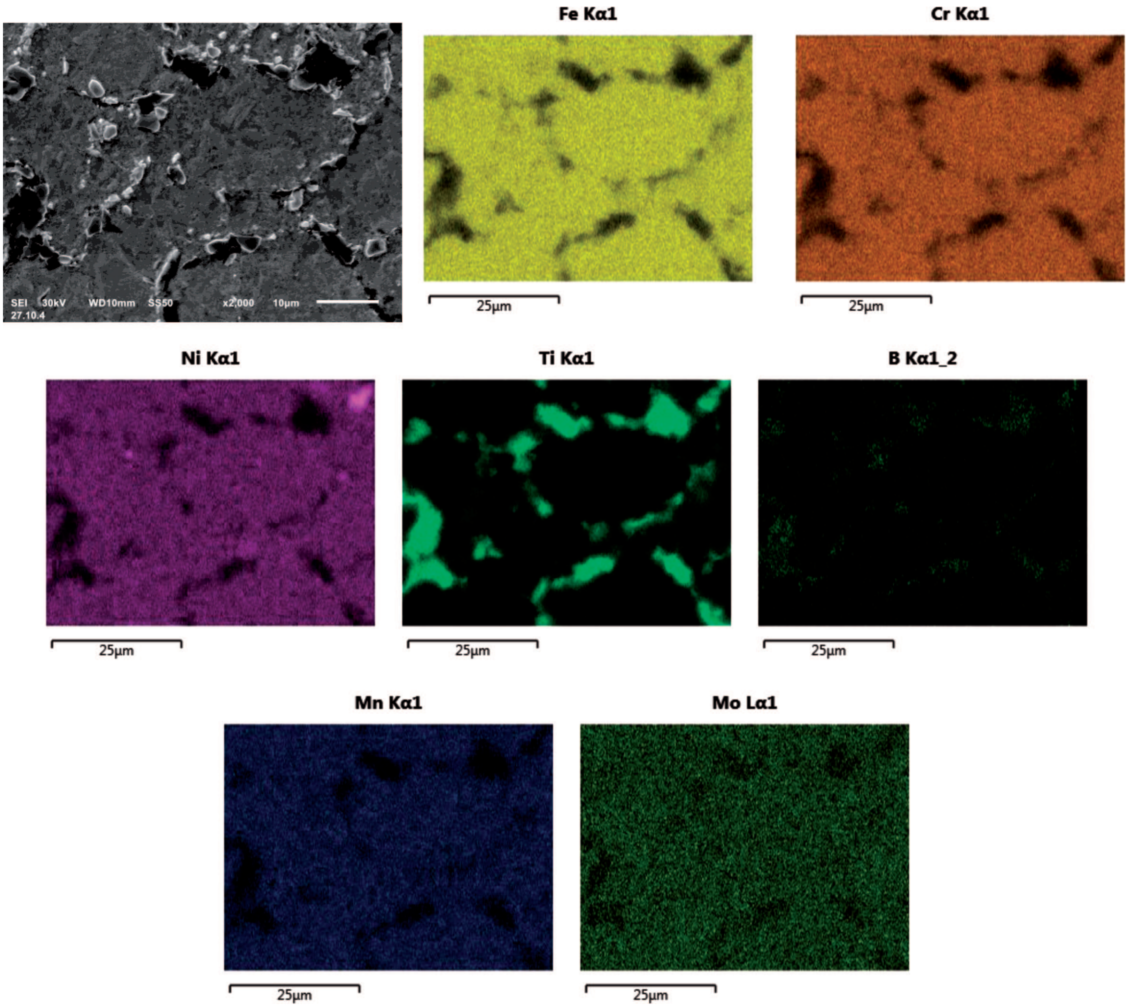


Fig. 8. The SEM micrograph of the composites with 8% vol. TiB_2 and corresponding distribution maps of elements: Fe, Ni, Cr, Ti, Mo, B and Mn (obtained at temperature of 1573K and pressure of 5±0.2 GPa)

4. Conclussions

The austenitic-steel-matrix composites reinforced with TiB_2 particles were obtained by HP-HT sintering technique. The effect of sintering process on the densification, Young's modulus, hardness and microstructure of the composites was investigated. From the results of the present investigation the following conclusions can be drawn:

1. The relative density of composites is above 98% of the theoretical density. Only in case of the temperature of 1573K and pressure of 5 GPa the values of density correspond to the 92% of the theoretical density.
2. It was showed that the properties of composites depend significantly on the temperature and pressure of sintering. Generally, the best combination of physical and mechanical properties of composites was achieved at temperature of 1273K and at pressure of 7 GPa.
3. The microstructure analysis reveals that fine TiB_2 particles with a size of about few μm are homogeneously distributed in the austenitic steel matrix.

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