THE TRIBOLOGICAL PROPERTIES OF SINTERED STEEL-MATRIX COMPOSITES

The tribological properties of composite materials reinforced with titanium diboride were investigated. Abrasion resistance tests were carried out at room temperature in a ball-on-disc system. Balls with a diameter of 3.14 mm were used as the counter-samples. The effect of the TiB₂ content and counter-sample material (Al₂O₃, Si₃N₄, ZrO₂, AISI52100 steel) on the coefficient of friction and wear rate of the sintered composites and 316L steel was determined. After the abrasion tests the sample surfaces were examined by scanning electron microscopy. The obtained results show that the tribological properties depend on the test conditions and content of the TiB₂ reinforcing phase.

Keywords: tribological properties, friction coefficient, composites, titanium diboride (TiB₂)

INTRODUCTION

Powder metallurgy is an effective and competitive technology to manufacture finished products from metals, ceramics and composites with very diverse properties [1, 2]. An important group of materials produced by powder metallurgy includes metal matrix composites dispersion-strengthened with particles, or with continuous and discontinuous (discrete) fibres [3-5]. The use of powder metallurgy to manufacture metal matrix composites enables a diffusion bond to be produced between the matrix and the reinforcement. It also offers vast possibilities in choosing the type, form and size of the reinforcing phase and allows products with the same chemical composition to be made, but with different values of density. Literature data [6-9] indicates a high level of interest in composites based on iron alloys reinforced with ceramic particles. The main reason for this interest is the low cost of steel production and high mechanical properties of the composites, additionally combined with satisfactory corrosion resistance. The ceramic phase introduced into the steel matrix improves the mechanical properties and wear resistance of the composites [10-13]. Among various ceramic materials, titanium boride is considered to be one of the best reinforcements for the steel matrix due to its high melting temperature (3225°C), low density (4.5 g/cm³), outstanding tribological properties and good compatibility with this matrix. TiB₂ ceramic materials are characterized by high hardness (3400 HV) and high corrosion resistance at temperatures up to 1400°C, high temperature chemical and structural stability, and resistance to thermal shock [14-16].

Tribological properties are important characteristics of structural materials. According to the definition, tribological wear is a kind of surface wear caused by friction processes. Under conditions of dry friction, abrasive wear is the dominant process [17]. The abrasive wear of composite materials depends on the morphology and volume fraction of the reinforcing phases, and also on the type, distribution and properties of these phases. The second group of factors responsible for the abrasive wear of composites includes the test conditions such as load, rotational speed, displacement, test tem-
perature, counter-sample material, and the test environment [17-20]. According to the literature [8, 21, 22], studies were carried out to ascertain the impact of TiB$_2$ on the tribological properties of stainless steels. Tjong and Lau [21] used a pin-on-disc machine to investigate the tribological properties of composites based on 304 steel with 20 vol.% TiB$_2$. The tests were carried out under different loads (15, 35 and 55 N) and at different speeds (1÷3 m/s). The results showed that the volumetric wear of the composite decreased with increasing the load or speed. It was observed that during the tribological tests, fragmentation (breakdown) of the TiB$_2$ particles into smaller pieces occurred. In another research work, Tjong and Lau [22] determined the abrasion resistance of sintered steel matrix composites with varying contents of TiB$_2$ ceramics. It was demonstrated that adding TiB$_2$ to the matrix significantly improved the abrasion resistance of the composite. Regardless of the test conditions used, the abrasion resistance of the composites containing ≥10 vol.% TiB$_2$ was ten times higher compared to the abrasion resistance of sintered 304 steel without the reinforcing phase.

**METHODS**

The raw materials used in this research were 316L austenitic stainless steel and 316L steel matrix composites containing 5 vol.% TiB$_2$ and 10 vol.% TiB$_2$. The materials were sintered using Spark Plasma Sintering (SPS/FAST). The sintering process was carried out at the pressure of 35 MPa and temperature of 1100°C for 5 minutes.

The density of the composites was measured by the Archimedes method. The Young’s moduli of the composites were measured by ultrasound to determine the transverse and longitudinal wave velocity using the ultrasonic flaw detector Panametrics Epoch III. The phase constituents and the microstructures of the specimens were analyzed by X-ray diffraction (XRD, Brucker Discover D8) operating with Cu K$_\alpha$ radiation and scanning electron microscope (SEM, JEOL JSM 6610LV). The density of the composites was measured by the ultrasonic method of measuring the friction force is 0.2%. For each sintered material, three tribological tests were carried out. Table 1 gives the conditions and parameters under which the tribological tests were carried out by the ball-on-disk method. The friction coefficient was calculated from the following equation [23]:

$$\mu = \frac{F_f}{F_n}$$

where: $F_f$ - measured friction force [N], $F_n$ - applied normal force [N].

The specific wear rate according to the wear volume was calculated by means of equation (2) [23]:

$$W_{V(disc)} = \frac{V_{disc}}{F_n \cdot L}$$

where: $W_{V(disc)}$ - specific wear rate of the disc [mm$^3$/Nm], $V_{disc}$ - wear volume of the disc specimen [mm$^3$], $F_n$ - applied load [N], $L$ - sliding distance [m].

After testing, the worn surfaces were investigated using Scanning Electron Microscope (JEOL JSM 6610LV).

**RESULTS**

Figure 1a shows the microstructure of the 316L steel+10%TiB$_2$ composites. The microstructure of the composites is characterized by uniform distribution of the reinforcing TiB$_2$ phase (black areas) along the grain boundaries in the steel matrix. The X-ray diffraction
studies (Fig. 1b) of the composites confirmed the presence of TiB₂ particles, while the X-ray diffraction phase analysis of the matrix revealed some peaks derived from austenite (γ).

The coefficient of friction assumes different values depending on the type of counter-sample used (Al₂O₃, Si₃N₄, ZrO₂ and AISI52100 steel). The highest values of this coefficient, i.e. 0.70, 0.62 and 0.59, were obtained for the 316L steel, 316L steel+5%TiB₂ composite and 316L steel+10%TiB₂ composite, respectively, when the counter-sample made of AISI52100 steel was used in the tests. The lowest values of the coefficient were obtained by the sintered composites and Al₂O₃ used as the counter-sample in the tests (Table 3, Fig. 3).

Compared to 316L steel without reinforcement, introducing the reinforcing phase into the steel matrix reduces the wear rate of the composites (Fig. 4). For example, in the tests with the Al₂O₃ counter-sample, the mass loss and specific wear rate \( W_{\text{dis}} \) obtained for 316L steel were 0.0319 g and 823.23 \( \times 10^{-6} \) mm³/N⋅m, respectively. For the composite materials with 10 vol.% TiB₂, the mass loss and the wear rate were 0.0171 g and 482.37 \( \times 10^{-6} \) mm³/N⋅m, respectively. This demonstrates the higher wear resistance of the 10 vol.% TiB₂ composites. The titanium diboride particles protect the steel matrix during friction, reducing its wear. In the composites containing 10 vol.% TiB₂, the removal of material was less severe. The same trend was observed in other samples subjected to tribological tests carried out with the counter-samples made of AISI52100 steel, Si₃N₄ and ZrO₂.

From the results of the tribological tests, it follows that the highest values of wear rate were obtained for the 316L steel and the composites tested for abrasion resistance with the Al₂O₃ balls. On the other hand, the lowest values of wear rate were obtained during tests using the steel balls. A similar tendency was observed in the relationship between the mass loss and type of counter-sample used. Differences in the obtained values of the mass loss and wear rate might be due to differences in the hardness of the material used for the

<table>
<thead>
<tr>
<th>Sintered materials</th>
<th>Apparent density ( \rho ) [g/cm³]</th>
<th>Young’s modulus ( E ) [GPa]</th>
<th>Microhardness HV0.3</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L steel</td>
<td>7.91±0.03</td>
<td>99</td>
<td>225±5</td>
</tr>
<tr>
<td>316L steel+5%TiB₂</td>
<td>7.51±0.03</td>
<td>99</td>
<td>396±8</td>
</tr>
<tr>
<td>316L steel+10%TiB₂</td>
<td>7.08±0.03</td>
<td>97</td>
<td>475±9</td>
</tr>
</tbody>
</table>

Table 2. Selected properties of sintered steel and composites

The tribological properties of sintered steel-matrix composites
counter-sample. According to literature data, the hardness of Al$_2$O$_3$, Si$_3$N$_4$, ZrO$_2$ and steel is 2200 HV, 2100 HV, 1300 HV and 850 HV [25, 26], respectively. The friction ball made of Al$_2$O$_3$ is characterized by the highest hardness, hence it can easily penetrate the material and remove it from the surface exposed to wear.

**Table 3. Weight loss, coefficient of friction and specific wear rate of sintered steel and composites**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ball</th>
<th>Mass loss $m$ [g]</th>
<th>Relative weight loss $\Delta m$ [%]</th>
<th>Specific wear rate $W_s \times 10^6$ [mm$^3$/Nm]</th>
<th>Coefficient of friction $\mu$ [-]</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L steel</td>
<td>Al$_2$O$_3$</td>
<td>0.0319</td>
<td>1.45</td>
<td>823.23</td>
<td>0.55</td>
</tr>
<tr>
<td></td>
<td>Si$_3$N$_4$</td>
<td>0.0290</td>
<td>1.06</td>
<td>748.39</td>
<td>0.58</td>
</tr>
<tr>
<td></td>
<td>ZrO$_2$</td>
<td>0.0234</td>
<td>0.89</td>
<td>603.87</td>
<td>0.55</td>
</tr>
<tr>
<td></td>
<td>steel</td>
<td>0.0131</td>
<td>0.17</td>
<td>338.06</td>
<td>0.70</td>
</tr>
<tr>
<td>316L steel + 5%TiB$_2$</td>
<td>Al$_2$O$_3$</td>
<td>0.0221</td>
<td>0.65</td>
<td>593.29</td>
<td>0.40</td>
</tr>
<tr>
<td></td>
<td>Si$_3$N$_4$</td>
<td>0.0208</td>
<td>0.57</td>
<td>558.23</td>
<td>0.48</td>
</tr>
<tr>
<td></td>
<td>ZrO$_2$</td>
<td>0.0167</td>
<td>0.50</td>
<td>448.32</td>
<td>0.48</td>
</tr>
<tr>
<td></td>
<td>steel</td>
<td>0.0091</td>
<td>0.09</td>
<td>244.30</td>
<td>0.62</td>
</tr>
<tr>
<td>316L steel + 10%TiB$_2$</td>
<td>Al$_2$O$_3$</td>
<td>0.0171</td>
<td>0.53</td>
<td>482.37</td>
<td>0.37</td>
</tr>
<tr>
<td></td>
<td>Si$_3$N$_4$</td>
<td>0.0153</td>
<td>0.44</td>
<td>431.59</td>
<td>0.41</td>
</tr>
<tr>
<td></td>
<td>ZrO$_2$</td>
<td>0.0140</td>
<td>0.45</td>
<td>394.92</td>
<td>0.43</td>
</tr>
<tr>
<td></td>
<td>steel</td>
<td>0.0071</td>
<td>0.05</td>
<td>200.28</td>
<td>0.59</td>
</tr>
</tbody>
</table>

**Fig. 2. Typical COF curves of:** a) 316L steel, b) 316L steel+5% TiB$_2$ and c) 316L steel+10% TiB$_2$ as function of testing time, measured using different balls

**Fig. 3. Coefficient of friction as function of TiB$_2$ content, measured using different balls

**Fig. 4. Variation of specific wear rate as function of TiB$_2$ content, measured using different balls

**Rys. 2. Współczynnik tarcia w funkcji czasu testu, zmierzony przy zastosowaniu różnych kulek (przeciwpróbek)

**Rys. 3. Współczynnik tarcia w funkcji zawartości TiB$_2$, zmierzony przy zastosowaniu różnych kulek (przeciwpróbek)

**Rys. 4. Zmiana wskaźnika zużycia w funkcji zawartości TiB$_2$, zmierzona przy zastosowaniu różnych kulek (przeciwpróbek)
The tribological properties of sintered steel-matrix composites

Images of the composite surfaces after the tribological tests using different counter-samples (Al₂O₃, Si₃N₄ and ZrO₂) are shown in Figures 5-7. The examined surfaces bear traces of adhesive and abrasive wear. In the wear track zone, as a result of the tribological tests, protrusions and irregularities on the surface are first fused together and then sheared, which indicates the adhesive type of wear. This type of wear usually occurs at low speeds and high pressures, especially under conditions of dry friction when there is no lubrication. Adhesive wear is defined as a type of wear occurring on the surfaces of rubbing bodies as a result of the formation of local adhesive bonds which are then broken (destroyed) by metal particles that are detached or stuck to the surface [18, 27]. Figures 5-7 show traces of wear as a result of fusion of the 1st type. During the tribological tests, fusing of the ball and disc materials took place, followed by decohesion of the steel. Some fragments of the bridge of fusion remaining on the ball surface could make scratches on the sample surface.

Fig. 5. View of 316L steel surface after rubbing against ball of: a) Al₂O₃, b) Si₃N₄, c) ZrO₂
Rys. 5. Widok powierzchni stali 316L po badaniach ścieralności z kulką: a) Al₂O₃, b) Si₃N₄, c) ZrO₂

Fig. 6. View of surface of 316L steel+5%TiB₂ composites after rubbing against ball of: a) Al₂O₃, b) Si₃N₄, c) ZrO₂
Rys. 6. Widok powierzchni kompozytu stal+5%TiB₂ po badaniach ścieralności z kulką: a) Al₂O₃, b) Si₃N₄, c) ZrO₂

Fig. 7. View of surface of 316L steel+10%TiB₂ composites after rubbing against ball of: a) Al₂O₃, b) Si₃N₄, c) ZrO₂
Rys. 7. Widok powierzchni kompozytu stal+10%TiB₂ po badaniach ścieralności z kulką: a) Al₂O₃, b) Si₃N₄, c) ZrO₂
Figures 8-10 show micrographs of the ball surface after tribological testing of the 316L steel + 10% TiB$_2$ composites. Material worn off the tested sample (steel matrix) was observed on all the surfaces of the ceramic balls. After the tribological tests, there were no signs of wear on the surfaces of the Al$_2$O$_3$ or Si$_3$N$_4$ balls (Figs. 9 and 10). Visible traces of wear were observed only on the surface of the ball made of ZrO$_2$ (Fig. 8). The hardness of Al$_2$O$_3$, Si$_3$N$_4$ and ZrO$_2$ is 2200 HV, 2100 HV and 1300 HV, respectively [25]. Ceramic materials based on ZrO$_2$ are characterized by the lowest hardness, which can affect the surface wear of the ball.

In the case of sintered composites, the second mechanism that intensifies the wear process may be adhesive wear, as evidenced by scratches and furrows in the wear track zone (Figs. 6 and 7). Their presence may be due to the effect of tearing the reinforcing phase (TiB$_2$) out of the matrix and loose transfer of ceramic particles along the wear track (between the sample and counter-sample contact surfaces). The transferred ceramic particles can scratch the surface of the cooperating sample or cause plastic deformation of the matrix.

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CONCLUSIONS

The tribological behaviour of 316L steel+TiB₂ composites was studied by the ball-on-disc method. The increase in TiB₂ content improves the physical and mechanical properties of the composites. It also has a beneficial effect on the wear resistance. The coefficient of friction and the wear rate decrease with the increasing content of TiB₂. The use of steel balls and ceramic balls made of Al₂O₃, Si₃N₄ and ZrO₂ in the tribological tests allowed different values of the coefficient of friction and wear rate to be obtained from the examined materials. The wear mechanisms were adhesive and abrasive in all the examined materials.

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REFERENCES