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Tribological properties of TiB_2 and TiB_2–$MoSi_2$ ceramic composites

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Abstract

Recently, dense monolithic TiB_2 and TiB_2–20 wt.% $MoSi_2$ composites with high hardness (24–26 GPa) have been processed by hot pressing. To assess the tribological potential, the present study was performed in analyzing the influence of load on the fretting wear of TiB_2 and TiB_2–$MoSi_2$ composites against bearing steel. Under the investigated conditions, a higher coefficient of friction (COF) of 0.5–0.6 was recorded with all the materials with a lower COF at a higher load of 10 N. Detailed microstructural investigation of the worn surfaces was carried out using SEM–EDS and XRD in order to understand the fretting wear mechanisms. Severe wear (order of 10^{-5} $mm^3/N\,m$) was measured for the investigated materials under the selected fretting conditions with lower wear rate for TiB_2–20 wt.% $MoSi_2$ composite at all loads (2–10 N). While abrasive wear dominates the material removal process in the case of monolithic TiB_2, the tribochemical wear is observed to be the predominant wear mechanism for the composite.

Keyword: TiB_2–$MoSi_2$; Composites; Wear resistance; Friction

1. Introduction

Titanium diboride (TiB_2) is a non-oxide ceramic, having excellent combination of attractive properties: low density (4.52 g/cm^3), high hardness (∼22–25 GPa), high melting point (2200 °C), and high elastic modulus (∼500 GPa).[1] These characteristic properties make it a potential candidate-material for various structural applications. However, the brittleness and high costs of machinability along with poor sinterability and exaggerated grain growth at high temperature restrict the widespread use of monolithic TiB_2 in engineering applications. To overcome this problem, various metallic and non-metallic binders are used to obtain dense borides. In one of our recent work, $MoSi_2$ is used as a sintering additive to achieve dense borides via hot pressing route.[2]

The combination of high hardness and elastic modulus makes borides particularly attractive for tribological applications. Moreover, the low density of TiB_2 and its high elastic modulus makes this material a candidate for the construction of light-weight armor and other aero-applications. Extensive research has been carried out to understand the tribological behavior of hard materials like ceramics and cermets[3–15] with limited tribological work on TiB_2.[4,7,9,13] The friction coefficient and wear rate of composites depends on the type of matrix, reinforcement chemistry and volume, counterbody material and the experimental conditions, such as sliding speed, load and environment (humidity, atmosphere, etc.). The effect of particulate additions on the tribological performance of composites is complicated. Incorporation of a second phase need not necessarily result in improved wear performance. For example, the incorporation of TiB_2 into a SiC matrix increases the fracture toughness and the incorporation of SiC into a Si_3N_4 matrix improves the bend strength and fracture toughness, but in neither case the SiC addition results in improved wear performance.[4] However, the addition of either TiC or TiN to a silicon nitride matrix has shown to improve wear performance substantially

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under tribological conditions, where tribochemical wear is dominant.[4]

Several studies have been reported to investigate the wear behavior of advanced structural ceramics.[7–12] The fretting wear behavior of boride-containing composites under dry sliding conditions against bearing steel is reported and a tribochemical wear model is formulated.[9] The influence of fracture toughness, an important property for brittle materials, on the wear behavior of ceramics was also investigated.[10] The tribological behavior of TiC-based ceramics against high-speed steel was studied using a pin-on-disk tribometer.[11] Oxidative wear coupled with adhesive and abrasive wear was the major mechanism for material removal. The fretting wear behavior of TiB_2-containing composites against ball bearing steel in lubricating medium (water, oil) was also investigated and reported in literature.[13] In comparison to dry unlubricated conditions, lower coefficient of friction (COF) as well as two orders of magnitude lower wear rate was recorded for TiB_2 in water lubrication. The formation of lubricious tribochemcial layer leads to the improvement in tribological properties in water lubrication. Recently, the wear behavior of WC-based materials has been investigated by our research group and also reported elsewhere.[6,8] A transition in COF and wear rate with load was observed for WC–ZrO_2 composites, processed by pressureless sintering[6] and spark plasma sintering.[8] The tribochemical wear with the formation of WO_3 was observed as the dominant wear mechanism. Mukerji and Prakash studied the wear performance of sialon-based composites with varying amount of TiC or BN (up to 20 vol.%) against ball bearing grade steel under dry sliding conditions.[15]

Although tribological studies on several structural ceramics have been reported in the literature, none of the studies has evaluated the wear performance of TiB_2-based materials with non-metallic additives. There have hardly been any literature report on fretting tests of TiB_2–$MoSi_2$ composites against steel. In the present study, the fretting wear behavior of TiB_2, TiB_2–$MoSi_2$ composites fabricated via hot pressing was investigated under varying load (2–10 N).

2. Experimental details

2.1. Materials

Monolithic TiB_2 and TiB_2–$MoSi_2$ composites containing 20 wt.% $MoSi_2$ particulate reinforcement were used in the present study. Monolithic TiB_2 with 97.5% ρ_{th} was obtained after hot pressing at 1800 °C for 1 h in vacuum, where as ~98% ρ_{th} density was obtained for TiB_2–20 wt.% $MoSi_2$ composite under similar hot pressing conditions at 1700 °C. XRD analysis indicated the predominant presence of TiB_2 and $MoSi_2$ in composite sample. The sizes of TiB_2 in monolith varied between 2 and 4 μm, while that in composite was around 1–3 μm. The particle sizes of $MoSi_2$ varied around 3–5 μm. The details of the processing, microstructure and properties are reported elsewhere.[2] Relative density and mechanical properties of the tested materials are tabulated in Table 1. While the hardness of the investigated materials varied between 23 and 27 GPa, the indentation toughness was in the range of 4–6 MPa $m^{1/2}$. Hot pressed pellets of diameter 10 mm were sectioned into 2 mm thick discs using high-speed diamond cutter. These discs were polished with emery papers (1/0, 2/0, 3/0, 4/0) and finally with diamond paste up to R_a 1 μm finish. The polished discs were ultrasonically cleaned in acetone for 10 min. Cleaned and polished monolithic TiB_2 and TiB_2-composites were used as flat (moving) materials. Eight millimeters diameter bearing grade (commercial SAE 52100 grade, hardness 63–65 HRc, data given by supplier) steel balls were used as counterbodies (stationary).

2.2. Wear tests and characterization

The fretting experiments were performed using a computer-controlled fretting machine (DUCOM TR281-M), which produces a linear relative oscillating motion with ball-on-flat configuration. By a stepper motor, the flat sample was made to oscillate with a relative linear displacement of constant stroke and frequency. An inductive displacement transducer was used to monitor the displacement of the flat sample, and a piezoelectric transducer to measure the friction force. Variation in tangential force was recorded and the corresponding coefficient of friction was calculated on-line with the help of a computer-based data acquisition system. The use of the same testing set-up has been made in our recent research on ceramics and composites.[6,8]

Prior to the fretting tests, both the flat and ball were ultrasonically cleaned in acetone. The fretting experiments were performed on monolithic TiB_2 and TiB_2–$MoSi_2$ composites against steel balls with varying load (P) of 2, 5 and 10 N at 8 Hz oscillating frequency and 100 μm linear stroke for a duration of 10,000 cycles. Also, the combination of the testing parameters resulted in the gross slip fretting contacts. All experiments were conducted in an ambient atmosphere at room temperature (30 ± 2 °C) with relative humidity (RH) of 45 ± 5%. The schematic of the test configuration is shown in Fig. 1.

Table 1
Relative density and mechanical properties of monolithic TiB_2 and TiB_2–$MoSi_2$ composites while monolithic TiB_2 is hot pressed at 1800 °C, the composite at 1700 °C

Material	Relative density (% of TD)	Vickers (H_v) (GPa)	Fracture toughness (K_{Ic}) (MPa $m^{1/2}$)
Monolithic TiB_2	97.5	25.9 ± 1.8	5.3 ± 0.9
TiB_2–20 wt.% $MoSi_2$	98.2	24.7 ± 2.7	5.2 ± 0.4

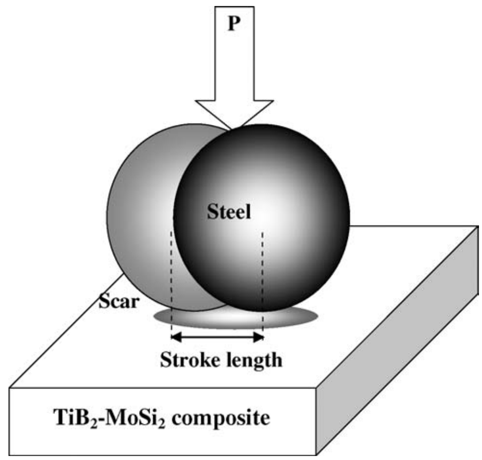


Fig. 1. Schematic of the fretting test configuration. The testing conditions: constants: stroke length 100 μm, oscillation frequency 8 Hz and cycles 10,000; variables: normal load (2–10 N).

After each test, the worn surfaces of both the flat and the ball were observed using an optical microscope (Zeiss). The wear volumes of both the flat and ball were calculated from the measured wear scar diameters as per the equation given by Klaffke.[16] The use of this equation is reported to be justified for the present fretting conditions (providing errors less than 5%) when the wear scar diameter is larger than twice the Hertzian contact diameter, as was the case in our experiments. From the estimated wear volume, the specific wear rates [wear volume/(load × distance)] were calculated. Further detailed characterization of the worn surfaces was done using a scanning electron microscope (JEOL-JSM840). Prior to SEM observation, the worn samples were sputter-coated with a thin Au–Pd coating in order to obtain sufficient conductivity on the surface and avoid charging of the surface in the SEM. The compositional analysis of the tribolayer is carried out by EDS, attached with SEM. Additionally, the phase analysis of the worn surfaces of selected fretted samples was carried out using XRD.

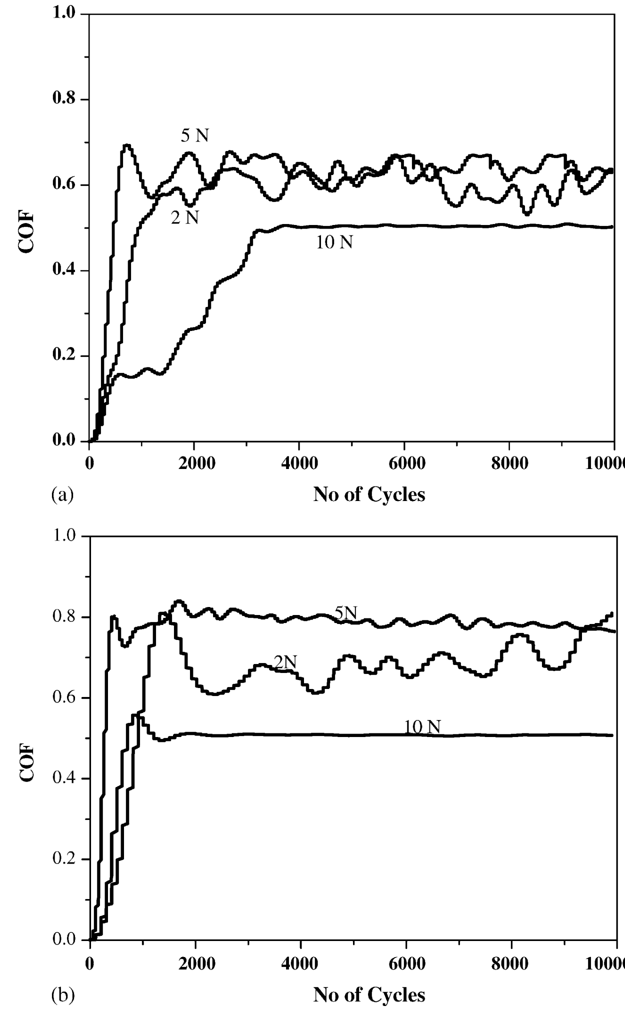


Fig. 2. The frictional behavior of (a) monolithic TiB_2 and (b) TiB_2–$MoSi_2$ (20 wt.%) composite, during fretting against bearing steel. Fretting conditions: 2–10 N load, 10,000 cycles, 8 Hz frequency and 100 μm stroke length.

3. Results

3.1. Friction and wear properties

The friction and wear properties of monolithic TiB_2 and its composites were studied under fretting contacts against bearing steel. The effect of the load was investigated by keeping other fretting variables (10,000 cycles, 100 μm amplitude and 8 Hz frequency) constant. The COF plots of TiB_2 and $MoSi_2$ reinforced TiB_2 at different loads are shown in Fig. 2. For monolithic TiB_2, COF increases to a high value in running-in-period (first 1000–3000 cycles) and subsequently attains a steady state value. Similar frictional behavior is also recorded with the composite. In both the materials, a slight fluctuation in measured COF is critically observed at 2 and 5 N; whereas such fluctuation is minimal at 10 N load. Also, a decrease in

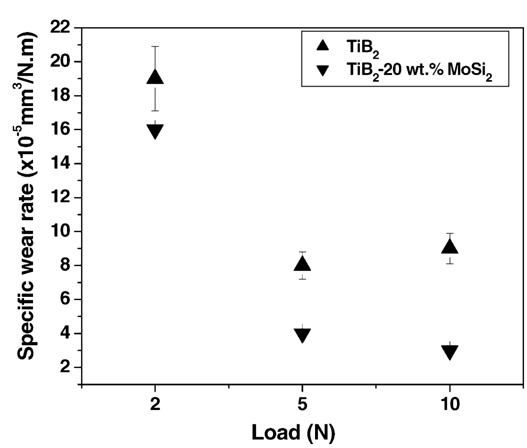
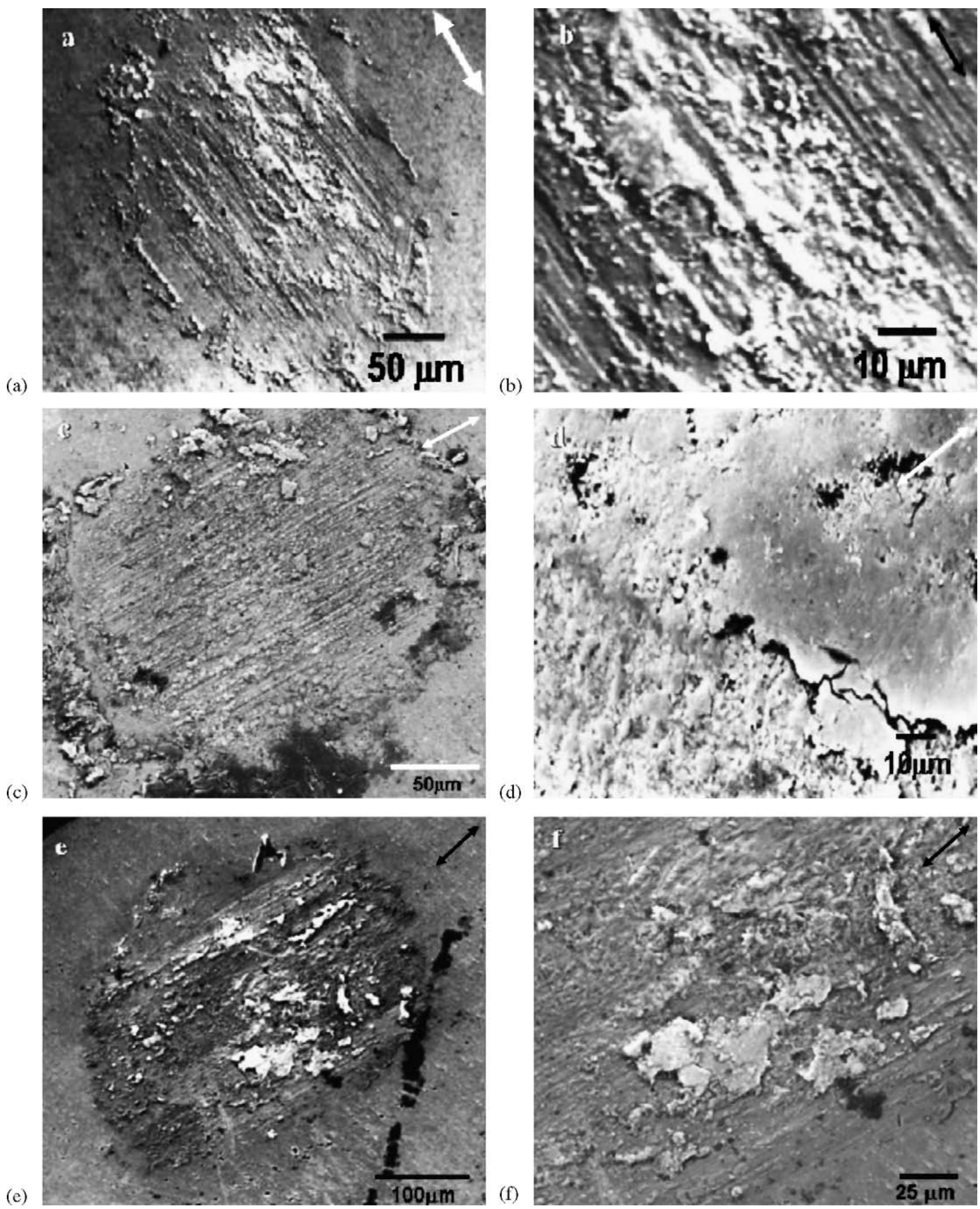


Fig. 3. Variation of specific wear rate with load for monolithic TiB_2 and TiB_2–$MoSi_2$ composites after fretting against bearing steel. Fretting conditions: 10,000 cycles, 8 Hz frequency and 100 μm stroke length.

steady state COF is observed for both monolithic TiB_2 and TiB_2 composite at 10 N load. The following detailed observations can be made from the COF data obtained with TiB_2 and its composites: (a) monolithic TiB_2 exhibits steady state COF values of 0.67 and 0.71 at 2 and 5 N, respectively. In contrast, steady state COF is lower and around 0.5 at 10 N load. Also, the attainment of steady state COF at 10 N load took place at the first 3000 cycles, while such a transition takes place earlier (first 1000–2000 cycles) at lower load of 2 and 5 N. (b) TiB_2–20 wt.% $MoSi_2$ composites exhibits steady state COF values of 0.67, 0.79 and 0.51 at 2, 5 and 10 N respectively. At 2, 5 and 10 N, peak COF values during running-in-period are 0.80, 0.80 and 0.55, respectively.

The wear volumes of TiB_2 and its composites are computed by measuring the wear scar diameter in the transverse direction on the worn samples and following the Klaffke's equation.[16] The specific wear rates, calculated based on the wear volume, load and total fretted distance are plotted in Fig. 3. For both the investigated materials, the wear rate decreases with load. Independent of the load (2–10 N), the wear



(a) (b) (c) (d) (e) (f)

Fig. 4. SEM images showing the overview and details of the worn surfaces on monolithic TiB_2 at different loads: (a and b) 2 N, (c and d) 5 N and (e and f) 10 N (overall and detailed). Arrow indicates the fretting direction. Fretting conditions: 10,000 cycles, 8 Hz frequency and 100 µm stroke length. Counterbody: bearing steel.

rate of the composite is found to be lower than that of the monoliths under the selected fretting conditions. The wear of steel was observed to be more than that of the ceramic in all cases and is evident from the large difference in hardness. The steel balls also suffer extensive fretting damage after testing against monolithic TiB_2 with increased severity as the load is increased from 2 to 10 N. The transverse wear scar diameter on steel, an indicative parameter for severity of wear damage, increases from 700 to 900 μm as load increases from 2 to 5 N. The steel balls also show extensive fretting damage after testing against TiB_2 composite. The wear scar transverse diameter increases systematically from 282.5 to 426 μm as load increases from 2 to 5 N.

Summarizing the tribological data, no clear correlation between COF and load is observed for the investigated materials. Also, the frictional behavior of the investigated tribocouple at the highest load of 10 N is stable as can be realized from the absence of any observable fluctuations in the friction plot. However, the frictional behavior of monolithic TiB_2 as well as of TiB_2–20 wt.% $MoSi_2$ is similar to that of steels, as the steady state COF for self-mated steel is reported to vary commonly between 0.5 and 0.6.[11] Based on the measured wear data, it is clear that the addition of 20 wt.% $MoSi_2$ decreases the wear rate of the composites vis-à-vis TiB_2 monoliths. Additionally, a systematic decrease in wear rate with load is observed for both the monolithic TiB_2 and its composites.

3.2. Topography observation of the worn surfaces and analysis of tribolayer

Detailed microstructural investigation using SEM was performed in order to understand the fretting wear mechanism for monolithic TiB_2 and its composites. The overall topographical features of worn scars formed on monolithic TiB_2 flats are shown in Fig. 4. The abrasion was commonly noticed under all loads. At 2 N load, a non-protective fractured tribolayer was observed (Fig. 5a and b). Similarly at 5 N, severe wear took place due to the non-protective nature of discontinuous tribolayer, which is removed by delamination. At the highest load of 10 N, extensive wear occurred due to similar delamination of the surface. EDS analysis revealed the presence of B, O, Ti and Fe on the worn surface of monolithic TiB_2 at all loads (Fig. 5). An interesting observation is that the relative peak intensity of Fe and O increases with the increasing load. The presence of O-peak indicates the tribo-oxidation of ceramic as well as the transfer of iron oxide from steel ball. The material transfer from steel is quite likely due to lower hardness of steel relative to TiB_2 ceramic. The major wear mechanisms for TiB_2/steel appear to be abrasion, adhesion and tribochemical wear.

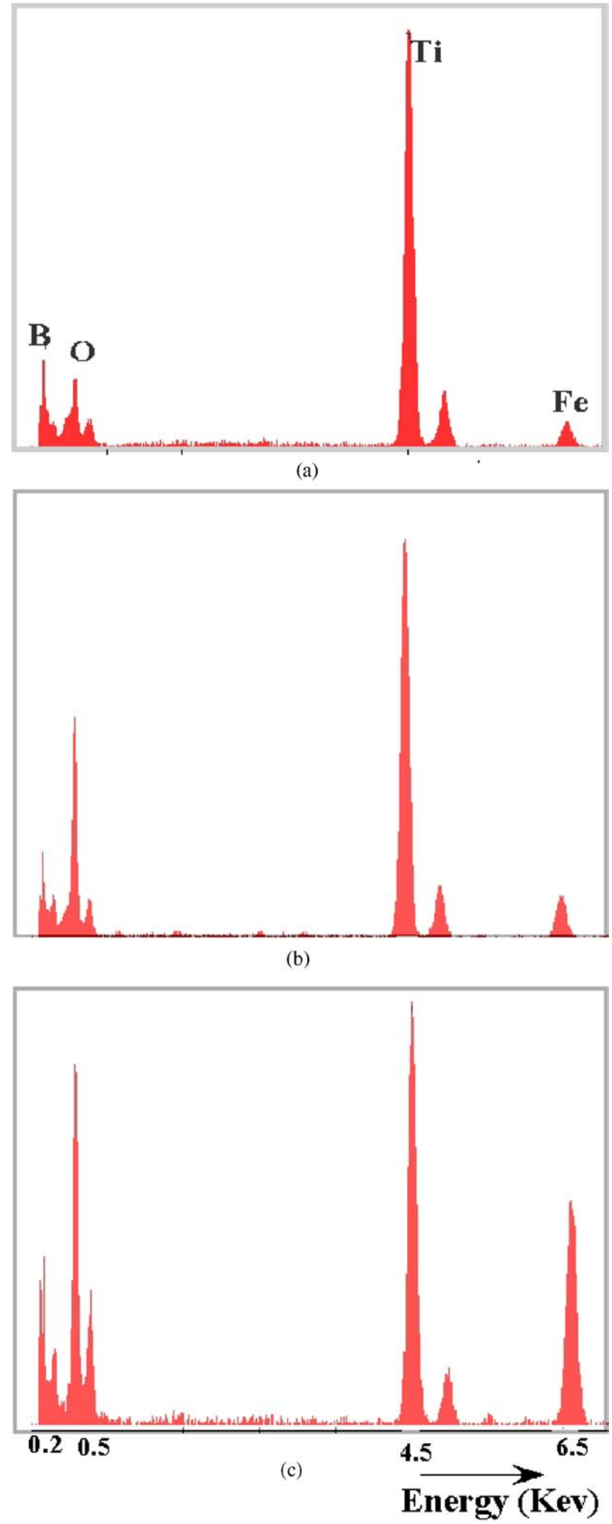


Fig. 5. EDX analysis on the tribolayer of monolithic TiB_2 at different loads: 2 N (a), 5 N (b) and 10 N (c). Fretting conditions: 10,000 cycles, 8 Hz frequency and 100 μm stroke length. Counterbody: bearing steel.

Fig. 6 shows the overall and detailed view of the fretted scars on TiB_2–20 wt.% $MoSi_2$ composite. In contrast to observations made with worn TiB_2, distinctly different observation was made with TiB_2–$MoSi_2$ composite. Under all loads (2–10 N), the worn surfaces are commonly covered with a rather protective tribochemical layer. The important features

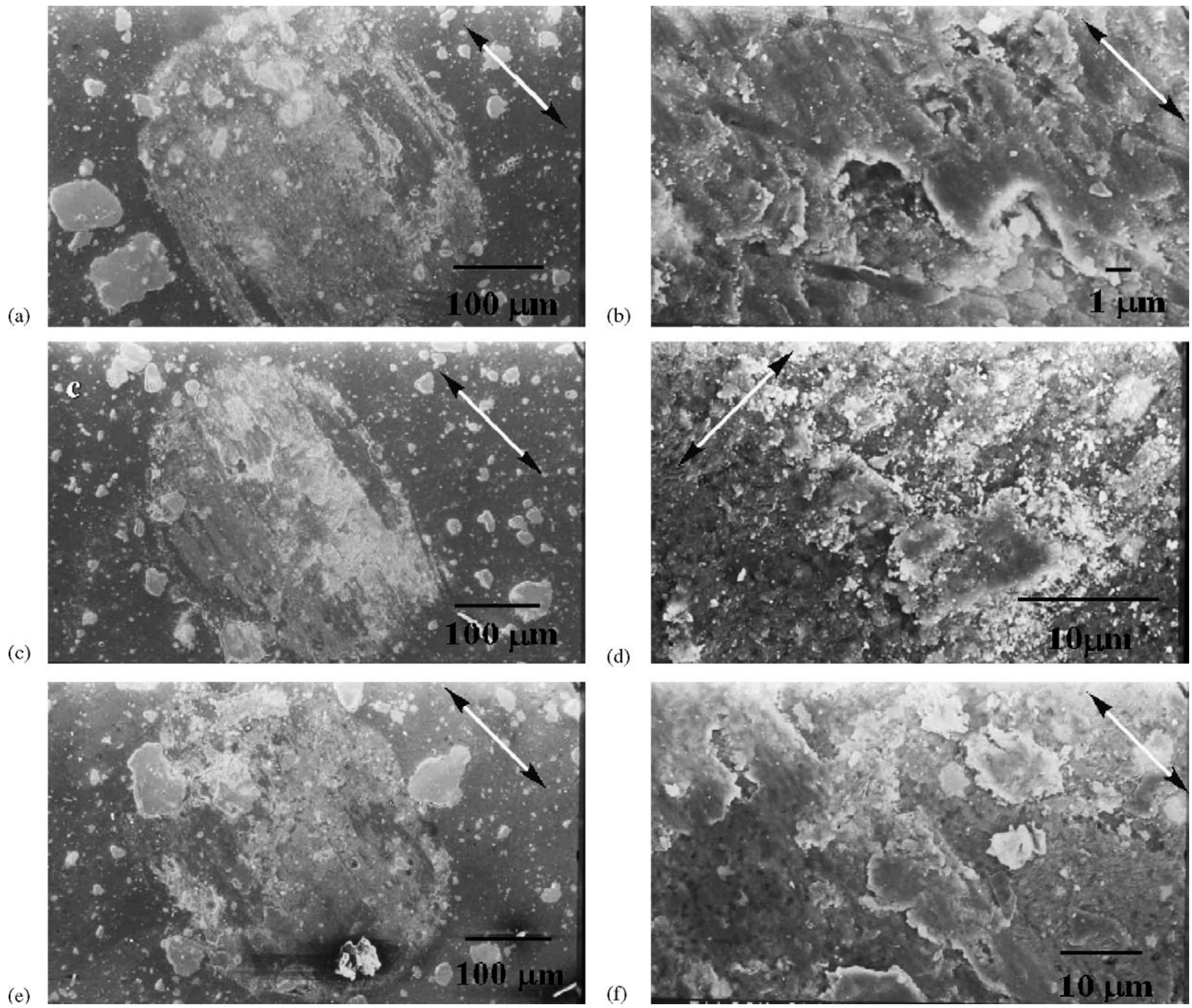


Fig. 6. SEM micrographs reveals the overview as well as details of the fretted worn surfaces of TiB_2–$MoSi_2$ (20 wt.%) composite at varying loads of 2 N (a and b), 5 N (c and d) and 10 N (e and f). Arrow indicates the fretting direction. Fretting conditions: 10,000 cycles, 8 Hz frequency and 100 μm stroke length. Counterbody: bearing steel.

like accumulation of oxide debris at the periphery of scar, smearing, fracture and the removal of oxide layer (as sheets) are illustrated in Fig. 6. It was observed that formation and smearing of tribochemical reaction product start at 2 N itself. The formation of sheet-like wear debris (50–70 μm size) results from the fracturing of tribolayer at some locations. Often finer debris particles (5–10 μm size) are also found scattered around the wear pits. Detailed look at the tribolayer reveals the cracking of tribolayer even at lowest load of 2 N (Fig. 6b). At an intermediate load of 5 N, entrapment of compacted debris particles on to the abrasive grooves is observed (Fig. 6d). At the highest load of 10 N, the cracking along the fretting direction and fracturing of tribochemical layer can be noticed (Fig. 6f).

XRD analysis of the worn surfaces on both monolith and composite after fretting at 10 N load is carried out and the results are plotted in Fig. 7. As could be seen in Fig. 7a, TiO_2, B_2O_3, Fe_2O_3 and $FeTiO_3$ are detected on the fretting scar, in addition to bulk TiB_2 phase. It should be noted that neither TiO_2 nor B_2O_3 is detected on the as-hot pressed surface, i.e. the virgin surface. Similar formation of tribo-oxides is also observed in the case of composite (Fig. 7b). The presence of MoO_3 and SiO_2 is additionally detected after fretting against steel. Iron oxides are not commonly recorded from worn composite within the detection limit of XRD.

4. Discussion

Based on the obtained friction and the wear data as well as SEM–EDS observations, XRD analysis of the mechanism of wear can be discussed. The wear of materials are governed by the material parameters (hardness, toughness), operating parameters (load, sliding velocity, humidity, temperature, etc.). Among the mechanical properties, the hardness is an important property determining the deformation of materials

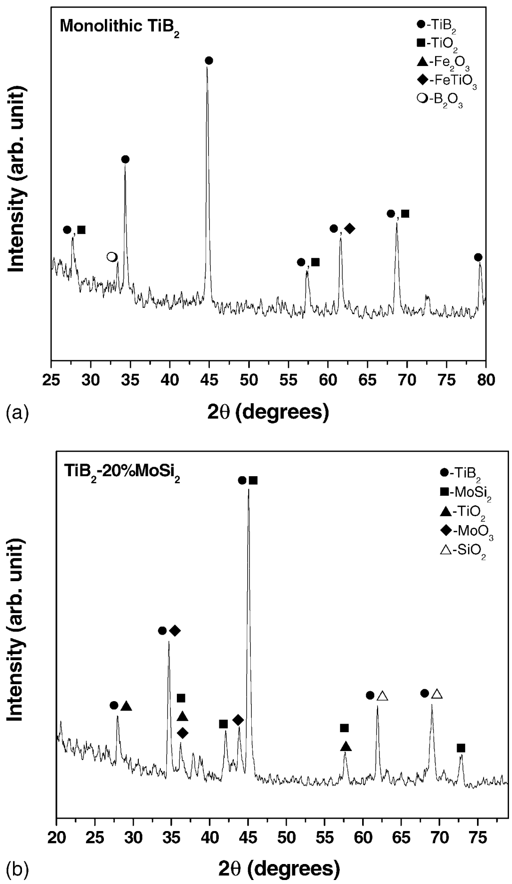


Fig. 7. XRD spectra obtained from the worn surfaces on monolithic TiB_2 (a) and TiB_2–20 wt.% $MoSi_2$ composite (b), after fretting against steel at 10 N load. Fretting conditions: 10,000 cycles, 8 Hz frequency and 100 μm stroke length.

at tribocontacts. In general, the metals undergo plastic deformation because of lower hardness (fully hardened steel ~7 GPa). In contrast, ceramics suffer fracture at the tribosurfaces because of limited plasticity in ceramics.[18] Also, looking at Table 1, it should be clear that the difference in hardness between the investigated materials is not considered to have any observable influence on tribological properties. The fracture toughness, an important parameter determining wear resistance for brittle ceramics,[18] is almost the same for both monolith as well as composite. In the present work, all the experiments are carried out under identical conditions with varying load.

An important factor largely controlling the reported tribological behavior of ceramics and ceramic-based composites is their interaction with atmosphere to form tribochemical oxides. This is particularly more important for non-oxide ceramics like TiB_2. The tribochemical wear of ceramics are closely related with surface chemistry and physics of the tribological process. Tribochemical wear is a form of oxidative wear like in metals that primarily occurs under nominally unlubricated conditions of sliding.[19] As far as the tribological properties are concerned, it is usually, but not always, beneficial by forming a tribochemical layer. In monolithic TiB_2 and TiB_2–20 wt.% $MoSi_2$, oxidation of the ceramic phases as well as steel is the cause for the high wear rate and friction. While tribo-oxidation wear is the major wear mechanism for all the investigated materials, the adhesive and the abrasive wear by brittle microfracture of tribolayer also contribute to more wear in the case of monolithic TiB_2 and TiB_2–20 wt.% $MoSi_2$ composites.

Based on SEM–EDS and XRD results, the possible tribochemical reactions that can occur during fretting actions include:

$$2TiB_2 + 5O_2 \rightarrow 2TiO_2 + 2B_2O_3 \quad (1)$$

$$2MoSi_2 + 7O_2 \rightarrow 2MoO_3 + 4SiO_2 \quad (2)$$

$$FeO + TiO_2 \rightarrow FeTiO_3 \quad (3)$$

Comparing Figs. 5 and 7, it is clear that TiO_2, B_2O_3, Fe_2O_3 and $FeTiO_3$ containing tribochemical layer is non-protective and spalls from the contacting interface. In one of the earlier investigations, So et al.[17] reported that higher the pressure and sliding speed, the thicker was the tribochemical layers and the shorter was the time interval for a layer to be extruded from the contact interface. In the present study, similar phenomenon is expected to take place and cause a higher COF. Although similar oxides form in the composites also with the exception of Fe-oxides (not detected), but additionally, the presence of MoO_3 and SiO_2 is also detected. It is quite plausible that SiO_2-rich tribolayer probably behaves like a rather protective layer, covering almost the entire tribosurface. In the fretting wear process, formation of tribo-oxidation products and subsequent fracture and removal of debris/tribochemical layer results in the observed high COF, as noticed in monolithic TiB_2 and TiB_2–20 wt.% $MoSi_2$ composite. However, since the tribochemical layer formed on the fretted surface of the composite is better protective than in monolithic TiB_2, the wear rate is decreased with 20 wt.% $MoSi_2$ addition.

5. Conclusions

In an effort to evaluate the tribological potential, the fretting wear experiments were carried out on monolithic TiB_2 and TiB_2–20% $MoSi_2$ composites against bearing steel under varying load. The important observations are as follows:

(a) Both monolithic TiB_2/steel and TiB_2–20 wt.% $MoSi_2$ composite/steel tribocouple exhibit similar frictional behavior with steady state COF varying in the range of 0.5–0.8 under the selected fretting conditions. Lower COF of 0.5 for both the fretting couple is measured at the highest load of 10 N.
(b) A specific wear rate is observed to decrease with the increase in load for the investigated materials. Lower wear rate is measured with the composite as compared to monolith at all loads.
(c) Under the investigated fretting condition, material transfer from steel counterbody as well as tribo-oxidation of

the constituent ceramic phases occurred at the fretted interface. SEM–EDS analysis revealed higher amount of transferred Fe on monolithic TiB_2 with increasing load. On the worn surfaces, the presence of TiO_2, B_2O_3 as well as $FeTiO_3$ is recorded in monolithic TiB_2, whereas additional formation of SiO_2 and MoO_3 is observed due to $MoSi_2$ addition in composite.

(d) While the tribochemical wear is found to play a major role in the removal of materials for both the tribocouple, the abrasive wear, in addition to tibochemical wear is found to contribute to more wear for monolithic TiB_2.

Acknowledgement

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