# ABSTRACT

The term “Tribology” was coined in 1966, which deals with the study of interacting surfaces in relative motion and encompasses the phenomena of friction, and wear. The technological significance of the field can be gauged by the fact that most machine failures are caused by the wear of machine elements, which renders them unsuitable to perform their intended function and necessitates their replacement. Wear is the most common cause of material waste and loss of mechanical performance, and any reduction in wear can result in significant economic savings. Friction is a major source of wear and energy loss. Improved friction control is expected to result in significant cost savings. It is estimated that one-third of the world's current energy resources are required to overcome friction in some form or another.

Titanium Matrix Composites (TMCs) combine favorable mechanical properties, good corrosion resistance, and high-temperature durability. Due to the excellent corrosion resistance, low density, appropriate mechanical properties, and high biocompatibility, titanium, and its alloys are widely used in the chemical, submarine, aeronautical, and biomedical industries. However, the relatively low hardness and poor wear resistance restrict their applications in the components where wear is a problem. A number of Ti matrix composites (TMCs) containing different hard phases as the reinforcements are being fabricated and researched to overcome this. This class of MMCs have are attractive and a lot of attention is being paid towards their development because of (a) the availability of different types of reinforcement at reasonable prices, (b) the successful development of manufacturing processes to produce MMCs with reproducible structure and properties, and (c) the availability of standard or near-standard metal

working methods that can be used to fabricate these MMCs.

The present study has been conducted to investigate the tribological behavior of Ti-based composites containing different Vol.% of TiB synthesized via vacuum arc melting technique, by performing dry sliding reciprocating wear tests at different loads and frequencies. Further, looking into the present trend of research, the present study also envisages unraveling the friction and wear behavior of titanium-based composites containing different Vol.% of TiB. The composites containing different amounts of TiB have been synthesized at the same sintering temperatures to determine the optimum TiB Vol.% based on the examination of their mechanical properties and tribological performance. Titanium-based composites containing different contents of $TiB_w$ have been prepared at this optimized sintering Vol.% and their friction and wear characteristics may be evaluated under different working conditions to explore the effect of reinforcement content on the coefficient of friction and wear rate. Pure titanium-based composites containing different amounts of $TiB_w$ have also been prepared and tested under similar conditions for the purpose of comparison taking pure Ti as the base sample. The study also intends to establish the prevailing mechanisms of wear under the conditions used in the investigation for this new class of composites.

The thesis has been organized in the following six chapters:

**Chapter 1** contains the introductory remarks highlighting the technological importance of the problem under investigation.

**Chapter 2** provides a critical review of the published literature on composites and their tribological properties. It begins with a brief description on the composites and their processing techniques, with a special focus on vacuum arc melting which has been used in the present study. This is followed by a description on tribology, the various types of wear and the parameters that affect the phenomenon of wear including a brief overview of the fundamentals of friction and some basic friction and wear theories. The chapter also covers the details of the studies conducted for synthesizing the Ti-based composites containing different second-phase reinforcements and the evaluation of their mechanical properties. A detailed description of the friction and wear behavior of Ti-TiB composites also forms a part of this chapter. The chapter also presents the gap in the published research, which has helped in the formulation of the problem and ends with the objectives of the present study.

**Chapter 3** outlines the details of experimental procedures followed in the current investigation. In the present study, the metal powders used for making the sample were Ti powder (99.4% pure; average particle size of 100 µm; Alfa Aesar, USA), $TiB_2$ powder (99.5% pure; average particle size of 325 µm; Alfa Aesar, USA), Fe (99.7% pure; average particle size of 6 µm; Thermo Fisher Scientific, USA). Two sets of composites have been synthesized using two different routes i.e., spark plasma sintering (SPS) and vacuum arc melting (VAM). In the first set five composites have been varying fabricated by varying Fe (at a fixed B at. %) and Boron (at a fixed Fe at. %) content from 10-30 at.%, and are designated as TiBFe1010 (10% B and 10 % Fe), TiBFe1020 (10 % B and 20 % Fe), TiBFe1030 (10 % B and 30 % Fe), TiBFe2010(20 % B and 10 % Fe) and TiBFe3010 (30% B and 10% Fe). In another set, Ti-TiB composites containing five different volume percentages of TiB (50, 60, 70, 80, and 85 Vol.%) have been prepared by vacuum arc melting and are designated as TiB50, TiB60, TiB70, TiB80, and TiB85. Pure Ti specimens have also been prepared using the same process for the purpose of comparison. X-ray diffractometer and high resolution-scanning electron microscope (HR-SEM) equipped with energy dispersive spectroscopy have been used to characterize the composites. The details of the procedure used for tribo-testing of composites along with various parameters are also included in the chapter. All the tests have been performed using a Linear Reciprocating Ball-on-Flat Sliding Wear configuration against a counterface of bearing steel ball at different loads (10, 15, 20, and 25 N), with a stroke length of 1 mm at 5 Hz and at a frequency (4, 7, 10, and 15 Hz) with a stroke length 2mm according to ASTM G133. The chapter also provides the details of techniques like SEM, XRD and Raman spectroscopy used for the analysis of worn surfaces and wear debris of all the composites and counterface ball.

**Chapter 4** begins with the presentation of results on the structure and property characterization of TiTiBFe composites, i.e., TiBFe1010, TiBFe1020, TiBFe1030, TiBFe2010, and TiBFe3010 synthesized via spark plasma sintering, namely XRD patterns of composite reveal the peaks corresponding to the presence of TiB phase FeTi, and Ti, which indicate that there is no oxidation of the powders during sintering. A dense and compacted structure with a higher volume fraction of TiB and FeTi was observed. The hardness of the composite increases with the increase of both FeTi and TiB phases but with TiB phases the hardness value is high. in comparison to 30 wt.% Fe as measured by a Vickers hardness.

The chapter also contains the results and discussion pertaining to the friction and wear characteristics of TiBFe1010, TiBFe1020, TiBFe1030, TiBFe2010, and TiBFe3010 composites. The results on tribological performance have been evaluated by carrying out reciprocating wear tests at different loads of 10, 15, 20, and 25 N and a fixed frequency of 5 Hz, and results are presented in two parts: (i) TiBFe1010, TiBFe1020, TiBFe1030 composites having fixed Boron and varying Fe and (ii) TiBFe1010, TiBFe2010, and TiBFe3010 containing fixed Fe but varying amount of B. All the composites i.e., TiBFe1010, TiBFe1020, TiBFe1030, TiBFe2010, and TiBFe3010 have been observed to possess a typical microstructure containing β-Ti, TiB and intermetallic

FeTi. The hardness of the composites increased from 488 to 871 $HV_{0.1}$ with the addition of Fe from 10 to 30 at. %, which has been attributed to the increasing amount of the formation of FeTi. The coefficient of friction decreased with an increasing amount of Fe at each of the loads i.e., 10, 15, 20, and 25 N, and the composite containing 30 at. % Fe, i.e., TiBFe1030 showed the lowest coefficient of friction. However, wear rate decreased with increasing Fe content from 10 to 20 at. % followed by an increase again for 30 at. % addition at each of the loads. The lowest wear rate was observed for TiBFe1020 having 20 at. % Fe despite its lower hardness than TiBFe1030 and has been attributed to dominating effect of the transfer layer present over the sliding surface which shields the underlying substrate from direct metal-metal contact. The hardness of composites containing 10 to 30 at. % B at a fixed amount of Fe increased from 488 to 964 $HV_{0.1}$. The composite TiBFe3010 showed the lowest coefficient of friction (between 0.51 and 0.44) and wear rate (TiBFe3010) with increasing load (ranging from 0.41 - 1.32 $\times 10^{-4}$ $mm^3/m$) due to increasing TiB content. The dominating mechanisms for have been found to be delamination and ploughing as revealed by morphologies of the worn surfaces.

**Chapter 5** describes the results of the structure and property characterization of Ti-TiB campsites synthesized via vacuum arc melting. Ti-TiB composites have been prepared by varying the $TiB_2$ wt.% in the Ti matrix to get different volume fractions of TiB, namely TiB50, TiB60, TiB70, TiB80, and TiB85. XRD patterns of composite reveal the peaks corresponding to the presence of TiB phase and Ti, indicating that melting of powders occurred without any oxidation or disintegration. The SEM micrographs of the microstructure of TiB50, TiB60, TiB70, TiB80, and TiB85 composites revealed relatively dense structures with two different shapes of TiB (i) whiskers and (ii) blocky in the Ti matrix. However, the composites with higher volume fraction of TiB were observed to be denser than lower volume fraction composites. The hardness has been observed to increase with an increase in TiB content in the composites i.e., 256 HV for pure Ti to 895 HV for TiB85.

The chapter also contains the results and discussion pertaining to the friction and wear characteristics of Ti, TiB50, TiB60, TiB70, TiB80, and TiB85 composites at different loads (10, 15, 20 and 25 N) and frequencies (4, 7, 10 and 15 Hz). The coefficient of friction (COF) has been found to fluctuate with time with relatively larger fluctuations in amplitude for all the composites in comparison to pure Ti. The composites have shown a run-in period having larger fluctuations before attaining a steady state. The duration of run-in period at a fixed frequency has been observed to depend on the composition and the load. However, the variation of coefficient of friction is observed to be steady for Ti at all loads with almost no running-in stage. The steady state coefficient of friction has not shown any particular trend of variation with load and composition at all frequencies i.e., 4, 7, 10, and 15 Hz. Both the coefficient of friction and wear rate increased with increasing load for all the composites. At a given load, the coefficient of friction and the wear rate of the composites decreased from 50 to 60 Vol.% TiB followed by an increase for 70 Vol.% and a reduction thereafter till 80 Vol.% and remained almost the same thereafter till 85 Vol.% TiB.

The average coefficient of friction for Ti, TiB50, TiB60, TiB70, TiB80, and TiB85 composites has been found to decrease with increasing load with the exception of Ti, which has shown an increase in coefficient of friction from 10 to 25 N a frequency of 7 Hz. However, TiB80 has shown a lower COF at all the loads in comparison to TiB50, TiB60, TiB70, and TiB85 with a minimum COF of 0.40 and maximum COF of 0.51 at 10 N. The observed behavior has been attributed to the presence of a transfer layer of oxides of debris over the surface which prevents direct contact between mating bodies and provides easy shearing junctions at the interface. The variation of wear rate with load did not shown any particular trend in composites whereas it has been observed to increase with increasing load for pure Ti. The wear rate of TiB50 increased from 10 to 15 N then decreased till 25 N. TiB60 showed an increase in wear rate from 10 to 20 N thereafter decreased a little at 25 N, while TiB70, TiB80 and TiB85 showed similar trends i.e. their wear rate increased from 10 N to 15 N followed by a decrease till 25 N with a minimum value of 0.18 x $10^{-4}$ $mm^3/m$. This has been attributed to the formation of a well compacted transfer layer and its degree of compaction which prevents the wear of the underlying substrate. The operative mechanism of wear has been found to be a mixture of ploughing, adhesion, and oxidation for pure Ti whereas the same for composites is adhesion, oxidation, delamination, and abrasion for all loads and frequencies.

The average coefficient of friction for pure Ti, TiB50, TiB60, TiB70, TiB80, and TiB85 decreased with increasing load from10 to 25 N for a frequency of 10 Hz. The composite TiB80 showed the lowest COF among all and pure Ti showed the highest. The wear rate of pure Ti increased with increasing load Ti and it had the highest wear rate among all the materials. The composites did not reveal any specific trend of variation of wear rate with load. Among all composites, TiB80 demonstrated the lowest wear rate and the observed behavior has been attributed to the presence of a transfer layer containing lubricious of oxides. The mechanism of wear is observed to be a combination of a ploughing, abrasion, and oxidation for Ti and whereas the same for composites was a mix of delamination, abrasion, adhesion, and oxidation. However, at a frequency of 15 Hz, the average COF is found to increase with increasing load for pure Ti whereas it is observed to either decrease of remain constant for composites with increasing load. TiB80 showed the lowest coefficient of friction at all the loads. At relatively lower loads of 10 and 15 N pure Ti also showed almost same COF as TiB80. The wear rate did not show any specific trend of variation with load for at 15 Hz also. The wear rate decreased with an increase in load for TiB60, TiB80, and TiB85 whereas for TiB50 and TiB70 it decreased from 10 to 15 N followed by an increase at 20 N before decreasing further at 25 N. The wear rate shown by pure Ti is significantly higher in comparison to composites which reflects the effect of the addition of TiB. Among all the composites, TiB85 composite has shown the lowest rate of wear at all the loads and the lowest rate of $0.23\times10^{-4}mm^3/m$ is observed at 25 N. This has been attributed to the formation of a well compacted transfer layer and its degree of compaction which prevents the wear of the underlying substrate. The mechanism of wear for composite is a mixture of ploughing, abrasion, adhesion and oxidation whereas for Ti ploughing, delamination and oxidation are the primary mechanisms of wear.

The composite having 80 Vol.% TiB showed the lowest coefficient of friction and wear rate among all the composites at 4, 7, and 10 Hz due to the formation of a transfer layer of wear debris over the surface, its degree of compaction, and the presence of lubricious oxides ($TiO_2$, $B_2O_3$, and $H_3BO_3$) as revealed by XPS. Whereas, at 15 Hz, TiB85 has shown a marginally lower coefficient of friction and wear rate among all composites.

**Chapter 6** presents the major conclusions of the present study pertaining to the microstructure and properties of composites along with their friction and wear characteristics under different loads, and frequency and the role of TiB in extending the effect of titanium matrix composites.