ROLE OF SLIDING VELOCITY AND TEMPERATURE ON THE TRIBOLOGICAL BEHAVIOR OF BORON MODIFIED AS CAST Ti6Al4V ALLOYS

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ROLE OF SLIDING VELOCITY AND TEMPERATURE ON THE TRIBOLOGICAL BEHAVIOR OF BORON MODIFIED AS CAST Ti6Al4V ALLOYS

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Submitted in partial fulfillment of the requirements for the award of the degree **of**

Master of Technology

by **MOHIT DESHPANDE**



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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled **Role of sliding velocity and temperature on the tribological behavior of Boron modified as cast Ti6Al4V Alloys** in the partial fulfillment of the requirements for the award of the degree of **Master of Technology** and submitted in the **Discipline of MEMS, Indian Institute of Technology Indore**, is an authentic record of my own work carried out during the time period from July 2017 to June 2019 under the supervision of Dr. Eswara Prasad Korimilli, Assistant Professor, Department of Metallurgical and Material Science, Indian Institute of Technology Indore.

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

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v

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"I dedicate my work to my parents for their selfless love and my sister for her constant support".

ABSTRACT

Trace additions of boron addition into the Ti6Al4V melt reduce the grain size substantially and eliminate the most expensive step in the thermomechanical processing of Ti6Al4V. This has significant effects on the mechanical properties of the alloy like tensile strength, ultimate tensile strength, notch sensitivity, fatigue, fracture toughness, and creep performance. Titanium alloy components are subjected to severe contact loading conditions during their service. Thus, the tribological study of these alloys becomes extremely necessary. Wear experiments were done on the Pin-on-disk tribometer at varying speeds and temperatures. On carrying out wear experiments, a non-linear variation of wear rate was observed with increasing temperature while linear behavior was observed with sliding speed. Mixed behavior was obtained in the samples as several factors play a role in the wear. At 1 m/s the performance of 0.3B alloy was optimum, while at 2 m/s it was 0.55B. Both alloys at 4 m/s at elevated temperature of 573K showed worse behavior than base Ti6Al4V itself. This can be attributed to the presence of hard TiB phase in the boron modified alloys as well as to the oxides formed at 1 and 4 m/s. Presence of tribo-oxides put a marked effect on the behavior at low and high speeds. To characterize the wear tracks we used SEM, XRD, Optical profilometry and Optical microscopy. Distinct deformation features are observed in SEM analysis. High abrasive wear and delamination is obtained for boron modified alloys at high temperatures and speeds while adhesive features along with abrasive wear is obtained in Ti6Al4V alloy.

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1. Introduction

Titanium (Ti) and its alloys have been in widespread use in recent years. The fact that it's the fourth most abundant structural material in earth's crust after Aluminium, Magnesium, and Iron have led to the advancements in its processability and broadening of its usage [1]. It has the right blend of properties like low density, high strength to weight ratio, corrosion and oxidation resistance, elevated temperature strength, good fracture toughness [2]. These properties enable it to be applied to a range of applications such as aerospace industries, transportation, consumer goods sector, marine applications, petroleum refining, surgical implantations in bio-medical purposes [3] as shown in Figs. 1 and 2. The main attraction for its use in aerospace applications lies in its high strength to weight ratio while retaining them even at high temperatures. Airframes and engines typically employ Ti alloys for their weight saving property. In chemical and power industries, Ti is used due to its corrosion resistance. Its usage in offshore structures has increased in recent years [1]. The main reason being resistance to seawater (saline), drilling mud, and transport fluids. Also, the newer markets emerging for Ti are building applications like exterior walls, roofing materials. Apart from these, its usage in consumer products like spectacle frames, cameras, watches, jewelry and sporting goods is increasing [1]. In spite of this, the use of Titanium is limited to aerospace applications as Titanium alloys are very costly. About 60% of its cost goes to energy-intensive melting and fabrication processes for producing finished products from raw materials [1].

1.1 Structure and Classes of alloys

Ti exists in two different phases, namely α and β . It exists in a hexagonal close-packed, α (hcp) structure up to a temperature of 882°C, as shown in Fig. 1.2 and transforms into a body centered cubic structure, β (bcc) above the same temperature. Commercially pure Ti has low strength but excellent corrosion resistance [1] and used in applications where the strength is not an important factor. Hence, alloying elements are added to improve the strength without sacrificing the corrosion resistance. The alloying additions stabilize

either of the two phases. Aluminum and Oxygen are typical α stabilizers, while Vanadium, Iron, Molybdenum, Nickel, Palladium do so for β phase. A few other elements, like Tin, Zirconium, are neutral towards both. Fig. 1.3 shows the effect of alloying elements on the phase diagram of Titanium. Depending on these additions its alloys are classified into three categories: α , β and α + β alloys.

 α - Ti alloys have additions like Al, Zr, which enables them to be employed at higher as well as cryogenic temperatures [5]. These alloys are better at resisting high-temperature creep than β and $\alpha+\beta$ alloys. They have good weldability and exhibit strengthening with heat treatment. Typical α -Ti alloys are Ti-8Al-1Mo-1V, Ti-2.25Al-11Sn-5Zr-1Mo-0.2Si, Ti-5Al-6Sn-2Zr-1Mo-0.25Si, and Ti-6Al-2Sn-1.5Zr-1Mo-0.35Bi-0.15Si (Ti-11) [6].

 β - Ti alloys contain additions making them stable at room temperature. These alloys are prone to embrittlement by the ω (omega) phase [4] precipitated by elements like Fe, Cr, Mn, Nb, Mo, Cr, or V during the binary alloy formation. Although stable alloys like Ti-10V-2Fe-3Al have been developed recently, which gives higher strength and toughness and has the potential for using as an air-frames construction material. Another alloy Ti-35V-15Cr is developed for burn resistance, which is highly useful in designing gas turbine components [4].

 $\alpha+\beta$ alloys are the most widely used and commercially important Ti alloys, among which Ti-6Al-4V (referred to as Ti64) holds special importance. It retains both α and β phases which resolves the problem of hot-forming in α alloys. It has high tensile strength, and formability with a lower creep resistance and weldability compared to their α and β derivatives. It is mainly used in forged components like gas turbine fan blades in new Lockheed/Boeing F22 military aircraft [7]. It is also available in plate, rod, sheet, and wire forms. Heat treatment and strengthening mechanisms are employed for different applications. Some other alloys in this category namely Ti–6Al–6V–2Zr–0.7 (Fe, Cu) (Ti-662), Ti–6Al–2Sn–4Zr–6Mo (Ti-6246), Ti–4Al–4Sn–4Mo–0.5Si (IMI 551), and Ti–4.5Al–5Mo–1.5Cr (Corona-5). The mechanical properties of some of the well-known Ti alloys are listed in Table 1.1.

1.2 Deformation mechanisms in Ti alloys

Both phases of Ti undergo different modes of deformation and slip. The hexagonal phase α deforms by slip as well as twinning. Alpha Ti can slip on basal (0002), prismatic (1010) and pyramidal planes (1011) in the closest packed <1120> direction. This slip system is called the <a> system and is not enough to satisfy the Von Mises criteria of having five independent slip systems. Secondary slip can occur on the <c+a> slip system consists of first (1122) and second order (1011) pyramidal planes along <1123> direction. The summary of slip systems is given in Table 1.2. [8]. Now, the α -Ti favors slip on prism planes rather than basal planes unlike in other hcp materials like Zirconium. The reason being slight atomic distortion leading to a reduction in the c/a ratio which is 6% less for alpha Ti. This happens as when Ti alloys are cooled down from the β phase the densely packed (110) planes transform to the (0001) basal planes in α phase. This results in increased distance between basal planes as compared to the (110) planes in the β state, hence the atomic distortion.

The β phase has a larger number of slip systems and thus is more ductile than the α phase. These different slip systems result from the combination of {110} planes and <111> directions [8].

1.3 Thermomechanical processing of Ti alloys

When Ti alloys are cooled from β phase into the $\alpha+\beta$ phase field, at the β grain boundaries α laths starts nucleating. These laths keep growing until they meet other colonies of different orientation. Inside the colony, the laths are separated by the β matrix that is retained in the microstructure. It is commonly referred to as the β laths. The Burgers relationship governs each colony's orientation. This type of nucleation is called sympathetic nucleation

and growth [1]. The α and β laths are also called α , β lamellae and microstructure are referred to as lamellar structure as shown in Fig.1.4. In case of slow cooling rates, the size of an α colony may be as large as half of the size of the β grain. On increasing the cooling rate, the size of α laths decreases and new laths starts nucleating on the boundaries of previous colonies. This results in a basket weave structure also known as Widmanstätten structure. The higher amount of β stabilizing elements favor this type of structure when the cooling rate is kept constant.

After Ti is extracted from the sponge, it has to be melted to produce the ingots which are the starting material for mill products and remelt stock for Ti castings. Quite a few melt defects may occur during this process. Thus, quite expensive processes are implemented to produce high-quality ingots. This results in higher costs of finished Ti products.

After ingot production, there are three more steps for processing of alloys. The first one is homogenization, following which is the deformation process (can be rolling, forging), and the last one includes recrystallization and annealing. Typical processing cycle is shown in Fig. 1.5. Maintaining high temperature while in β working adds to the cost of the finished product.

1.4 Boron modified Ti and Ti64 alloys

Boron is an α -stabilizer with very low solubility in the solid phase of Titanium. Its low solubility results in the formation of intermetallic precipitates (TiB particles) which are very hard (250HV) and strengthens the matrix. Zhu et al. [9] studied the microstructural effect of boron addition to pure Ti and various other Ti alloys. It was reported that the grain size is significantly altered by adding boron in quantity as low as 0.01% by mass. Ti-B particles precipitate along the grain boundaries and form a necklace like structure at higher concentrations. These are fine needle-shaped particles whose length ranged from 5-40µm. It was suggested that solubility of Boron was very low in the solid phase and almost all of it was precipitated out as Ti-B particles. The microstructures for cast Ti alloys (Ti-0.5Sn-B)

with different Boron content are shown in Fig. 1.6. The structure was lamellar α phase and some β phase with no Boron but as it was increased to 0.46% structure changed to acicular α with β phase and dispersed TiB particles along prior β -grain boundaries. They found that the elongation was maximum around 0.1 to 0.15%. Further increasing the content, ductility reduced but strength kept rising. The same thing happens with Ti-64 alloys as well. The grain size gets refined, and on adding Boron up to 0.5 mass%, the structure changed to Widmanstätten structure consisting of fine α and β laths, as shown in Fig. 1.7.

1.5 Importance of the tribological study of Ti64 alloys

Tribology is described as the "science and technology of interacting surfaces in relative motion and of related subjects and practices" [10]. Tribology deals with friction, wear, and lubrication. Friction between materials is the leading cause of wear. To reduce wear, one needs to reduce friction between the mating surfaces by lubrication, coating the exposed layer by suitable material, chemical treatment. Wear can be defined as the increasing loss of material in the system under relative motion. Wear reduces the life and performance of a component and is responsible for increasing the overall cost of system in repairing or replacing an entire component. The loss of material reduces the dimensions, which in turn increases the clearance in between components. This causes vibrations, noise, reduced efficiency in the system, further increasing the wear. The wear debris produced is also harmful as sometimes they may contaminate the final product as in the food and beverages industries. If they remain trapped inside the system, they act as abrasives, which again increases wear. The damage by wear is multifold. Titanium components are employed in aircraft where sliding occurs between mating surfaces, like in engine components, gears, cams, bearings, aircraft's outer surface. The wear that is caused in this case is of usually abrasive or fretting type. The air passing through engine carries dirt particles which cause erosive wear when they move over surfaces within. These all factors point towards the importance of wear studies for the materials, it will not

only increase the performance and efficiency of the existing parts; it will also save the cost of replacing and repairing the parts that have undergone premature failure. Let us take a look at the different types of wear mechanisms that may occur during sliding.

1.6 Mechanisms of wear in metals

1.6.1 Adhesive wear

When two surfaces are sliding over one another, there is never a complete contact between then no matter how highly polished the surfaces are. There are surface irregularities that come in contact which are as small as 1/100th of the apparent area of contact. The plastic deformation and adhesion between the materials lead to the formation of cold weld junctions, and the adhesive wear depends on whether the strength of junction is higher or between the original material itself. Now, most metallic materials show a good adhesion tendency. If the adhesion is strong, a small amount of material gets stick to the counter material, as shown in Fig. 1.8. [11]. This fragment then undergoes shear with continued sliding, and therefore, the debris is of the form of flat plates mostly. Scoring is a severe form of adhesive wear that occurs when the welded particles are torn out due to overheating of the surfaces under high load/pressure as in mating surfaces of gears. Sliding forces tear the metal apart, and this forms a minute cavity on one surface while a protrusion on another. It is also called as galling, seizing or scuffing.

1.6.2 Oxidative wear

Formation of an oxide layer is a good way to reduce the adhesive wear. Metals are generally covered with an oxide layer as they readily react with oxygen. These oxides reduce adhesion and get worn off first. Now, the presence of oxides doesn't always protect the surface from wear. If the material beneath the oxide layer is soft, the harder counter-face may penetrate the oxide layer and remove the fresh material. Also, if the oxide layer is thin or the adhesion between the oxide layer and the material is low significant wear may still happen.

If on the other hand the material underneath the oxide layer is hard and has decent adhesion with the layer a small fragment of oxide will be removed from the surface. The layer now exposed will react with the oxygen and form oxide again. In this case, the wear rate is reduced drastically. The rate of oxide formation depends on the material properties, sliding conditions. In sliding conditions, the friction causes a rise in temperatures, which reduces the activation energy for oxide formation. Thus rapid oxidation occurs compared to static conditions [11]. The wear debris produced are finely powered oxide particles and wear surface is generally smooth, covered with patches of oxides that can be seen with naked eyes.

1.6.3 Abrasive wear

Abrasive wear also is known as cutting wear is a very frequent phenomenon occurring in the industry. In it, when a hard particle slides and rolls over a surface under pressure. The hard particles can enter in the system from sources like a contaminated lubricant, dirt or sand from castings, metal wear debris. Abrasive wear is classified as –

- a) Two body abrasion This happens under metal-metal contact where one is harder than the other. The asperities on the harder surface penetrate or plows into, the softer one cutting through it. Two body abrasion always exists since the perfectly polished surface can never be obtained. It can easily transform into three body abrasion as the work hardened wear debris produced are sometimes trapped between the mating surfaces. The mechanism in this wear is micro plowing or cutting wherein small cracks are produced in the softer matrix. The wear is identified by grooves or scratches, as shown in Fig. 1.9. Wear debris appear as small chips.
- b) Three body abrasion This happens when a foreign particle is trapped between the mating surfaces and abrades either one or both of them.
 Sometimes the particle is embedded into the softer surface and abrades

the opposite one. This type of abrasion is found in machines working in desert areas or mining industries.

1.6.4 Delamination wear

Delamination is a severe type of wear in which the material is removed in the form of thin sheets/layers. The dislocations that are parallel to the surface are acted upon by image forces which removes them. As a result of this, the sub-surface layer undergoes work hardening better than the surface layer [12]. Continuous sliding causes dislocations to pile up over some distance from the top layer. If a hard phase is present in the material, these dislocations move around it to form voids. These voids then undergo shearing and coalesce to form a crack parallel to the surface. With further sliding the crack travels to the top surface and material is removed as a thin sheet. The schematic representation of the wear is shown in Fig. 1.10 [12].



Fig. 1.1 a) Classification of metals based on density, b) Various uses of Titanium and its alloys in aero-engines c) Titanium usage in automotive components and d) Dental applications [13].



Fig. 1.2 a) α (hcp) phase below 882°C and b) β (bcc) phase of Titanium above 882°C [1].



Fig. 1.3 Effect of alloying additions on the α and β phases of Titanium [1].

Alloy	Processing	Yield	Ultimat	Elongati	Fracture
	/Manufact	strengt	е	on	toughne
	uring	h	strength	(in %)	SS
		(MPa)	(MPa)	(111 70)	(MPa-
					m ^{1/2})
Τί-64(α+β) [14]	Standard	833.9	900.2	6	126
	Ti-64 HIP				
	cycle				
$Ti_64(\alpha+\beta)$	HID'od	979	910	80	
Π-0+(α+ β)	THE EQ	020	510	0.9	
	mill				
	annealed				
0.05B [14]	"	878.7	943.6	8.5	73
0.1B [14]	"	900	965.8	7.3	63

0.4B [14]	"	913.3	972.5	5.2	38
Ti-64-1B	Cast+extr ude	1045	1138	10	
Ti-64-1B	PA (pre- alloyed) powder metallurg Y processing	1079	1124	13	
Ti-64-1.55B [2]	As-cast	999	1033	0.9	
	Forged	1065	1128	2.7	
	Rolled	1170	1230	6	
	Extruded	1185	1200	5	
Ti-6Al-2Sn-4Zr- 2Mo(near α alloy) [15]	Duplex annealed	827	896	10	
Ti-6242 [16]	Heat treated @ 970°C (alloy @480°C)	565	735	25	
Ti-5Al-5V-5Mo- 3Cr (near β alloy) [17]	As-Cast and HIP'ed	1055	1159	9	

Ti-5553 [18]	Solution treated and aged	1150	1250	12	55
Ti-5553 [18]	BASCA treatment	950	1100	6	70

Table 1.1. Mechanical properties of various Ti-64-xB alloys and some important α , β alloys.



Table 1.2. Slip modes in α-Titanium



Fig. 1.4 Typical $\alpha+\beta$ lamellar microstructure observed under a) Light microscope b) TEM



Fig. 1.5. Typical thermomechanical processing cycle of $\alpha+\beta$ Ti alloys.



Fig. 1.6. Optical microstructures of as cast Ti alloys with different Boron content.



Fig.1.7. Microstructures for Ti64 alloys with increasing Boron content a) 0, b) 0.05, c) 0.1 and d) 0.4 wt%, the grey background is α -phase, the bright phase is β and black is TiB.



Fig. 1.8. Schematic diagram showing steps leading to adhesive wear between two surfaces.



Fig. 1.9. Schematic diagram depicting two and three body abrasive wear.



Fig. 1.10. Delamination wear [12].

2 Literature survey

It has been reported in the literature that Ti64 alloys have poor wear resistance. Tamirisakandala et al. [2] have found that the low solubility of Boron leads to a reduction in the lattice embrittlement issues caused by other interstitial elements like C, H or O. Ti-B phase diagram suggests that TiB is precipitated when added below 18.4 weight %. The formation occurs via a eutectic reaction $L \rightarrow \beta$ + TiB as shown in Fig.2.1. TiB particles increase the stiffness and strength of the matrix without sacrificing the density. The structure of TiB is orthorhombic, and they grow in short whiskers. Its thermal expansion coefficient is similar to Ti, and hence, it doesn't impart any residual stresses during solidification. It is also the most stable boride phase in Ti-B system, and its chemical compatibility is excellent [2]. Even with the benefits of boron addition, it must be kept below a certain limit as increasing addition causes coarser Ti-B precipitates, which are brittle. These particles cause fracture behaviour to change from ductile to brittle and results in premature failure. Microstructural studies showed that on subsequent heat treatment the grain growth was restricted in alloys with Boron which confirms that strength and stiffness are retained in Boron modified alloys. Also, while cooling the alloy form higher temperature, it was observed that equiaxed microstructure was obtained in Ti-64-xB alloy instead of lamellar. The equiaxed structure is obtained only through reheating above the β transus; thus, the addition of boron eliminates the need of $\alpha+\beta$ hot working. It was reported by them that addition of 0.1% boron reduces the grain size of as-cast Ti-64 from 1700 to 200µm, Ti-6Al-2Sn-4Zr-2Mo (Ti-6242) from 550 to 50µm and Ti-5Al-5V-5Mo-3Cr (Ti-5553) from 300 to 50µm. The graphs of grain size vs Boron concentration show a sharp decline in the range of 0 to 0.1% suggesting that a critical value of concentration is required for obtaining large grain refinement. As shown in Fig. 2.2 [2] boron addition beyond 0.1% doesn't cause much reduction.

Sen et al., [14] carried out experiments to understand the role of microstructure on tensile, fatigue properties of the boron-modified alloys. They found that all alloys showed elastic-perfectly plastic behavior with negligible work hardening. They calculated the grain and the lath sizes; the
lath size reduces by about 38%, which is smaller than the reduction in grain size, which decreased by order of magnitude up to 0.1% addition. Further adding boron till 0.4% reduces lath size by 10%. The reduction in lath size is attributed to the different thermal conductivity of TiB needles at grain boundaries from the matrix [10, Sen et al.]. This increases the nucleation rate of α laths and controls the lath size.

Tensile properties like elastic modulus, yield strength, ultimate tensile strength all were found to be increasing with the increase in boron content. The increase is, however, observed up to only 0.1% addition. Further addition showed a little rise in values. The increase in elastic modulus, yield, and ultimate tensile strength is around 8-10%. The variation of tensile properties is shown in Fig.2.3 [14]. Fracture toughness was found to be decreasing with an increase in boron concentration. It got lowered to 38 MPa m^{1/2} for 0.4 wt% boron from 126 MPa m^{1/2} for Ti-64. High magnification images revealed that fracture was controlled by cleavage of α laths. Also, due to the needle-like structure of Ti-B particles, they act as stress raisers. When crack propagates and meets them, stress concentration occurs, and material fails easily. This is also responsible for the reduced fracture toughness of alloy. It was observed from fractographs that the α lath length was the controlling parameter for the fracture mechanism in Ti64 alloys. The variation of fracture toughness in Ti-64 with boron wt% is given in Fig. 2.4.

Agrawal and Kartikeyan [19] studied the strain hardening phenomena in Ti-64-xB alloys. They found that the increase in yield strength 4% was much lower than the increase in ultimate tensile strength, which is 14%. This points towards an increase in the strain hardening in the Ti-64-xB alloys. They carried out slip trace studies through SEM and optical microscopy where they focused on slip transmission barriers and modes of slip. It was observed that the lath boundaries allowed the slip line transmission, whereas the colony boundaries resisted them. There is a lattice mismatching at colony boundaries and strain incompatibility which resisted the travel of slip lines. Hence alloys with low Boron content (<0.09%) undergoes single slip and localization while multiple slip was observed in alloys with higher Boron. Due to multiple slip, the deformation was more homogeneous, resulting in enhanced strain hardening for high Boron content alloys. Fig. 2.5 and 2.6 [19] show the evolution of slip traces in respective materials.

Molinari et al., [20] studied the dry sliding wear characteristics of Ti-64-xB alloys. They focused on studying the wear mechanisms responsible for the poor wear resistance of Ti64 alloys. They subjected the alloy under different loads in the range of 35-200 N and sliding speed range of 0.3-0.8 ms⁻¹. After that, they carried out the surface morphological and wear debris study. Wear volume increased with increasing loads, and there was a shift in the minimum value of wear volume towards lower sliding speeds, as depicted in Fig 2.7 [20]. Intermediate speeds always showed the minimum wear. The wear debris comprised of TiO and α -Ti at lower speeds while only α -Ti peaks were found at the highest speed in XRD analysis. The lowest wear speed debris consisted mainly of metallic particles. SEM analysis showed that at lower speeds material was removed in the form of small fragments whereas at higher speeds delamination of subsurface was observed. Optical microscopy images confirm the presence of two layers; a surface scale followed by a strained sub-surface layer. Micro-hardness data revealed high values on the surface scale, which dropped to the bulk matrix value with increasing depth. The surface scale thickness increased with increasing load. Secondary Ion Mass Spectroscopy (SIMS) analysis showed that the surface scale was found to be contaminated by Oxygen at lower speeds while it is negligible at higher speeds. The wear debris characteristics varied widely with load and sliding speeds. They concluded with separating the different types of wear with the minimum wear speed curve, as shown in Fig. 2.8.

Li et al. [21] carried out pin on disk wear experiments on Ti64 alloys and studied the tribo oxides formed and how did they affect the wear behavior. Tests were run at speeds of 0.5-4 ms⁻¹ at loads of 10-50 N with an interval of 20 N. They observed the wear rate decreased at first, then reached a maximum and further decreased on increasing the sliding speed, as shown in Fig. 2.9. A similar trend was observed for the friction coefficient as well. Their results were in accordance with the results of Molinari et al. [20] in that they also observed the absence of oxides at the intermediate speeds while multiple oxides were present at lowest and highest speeds. Optical

microstructures of the cross-sections, as shown in Fig. 2.10 [21] revealed, there always existed a surface layer which was different from the Ti-64 bulk. They found that the tribo layer was a mechanically mixed layer (MML) made up of a mixture of the wear debris from sliding metal, counter-face metal and reaction products of oxygen and wear debris. Its formation depends heavily on the sliding system and conditions. They concluded that wear mechanism changed from the combination of delamination wear and oxidative wear at a lower speed to delamination wear at 2.68 m/s, then to oxidative wear at 4 m/s. They also found that the sliding speed induced tribo-oxides have a protective function, which is in contrast to the traditional view.



Fig. 2.1 Ti-rich end of the Ti-B binary phase diagram showing solubility of boron in Titanium [2].



Fig.2.2 Grain-size vs boron concentration [2].



Fig. 2.3 Various tensile properties of Ti-64-xB alloys with varying boron concentration [14].



Fig. 2.4 Fracture toughness variation of Ti-64 with boron wt% [14].



Fig. 2.5 Evolution of slip traces in Ti-64-0.4B alloy. Multiple slip lines appear at higher strains [19].



Fig. 2.6 Evolution of slip traces in Ti-64-0.09B alloy. Higher strains show multiple slip patterns [19].



Fig. 2.7. Wear volume vs. sliding speed at different loads [20]



Fig.2.8. Qualitative wear map showing predominating wear mechanisms [20].



Fig. 2.9 Wear rate vs. sliding velocity with increasing loads [21].



Fig. 2.10 Optical microstructures of cross-sections of Ti-64 alloys at 30N normal load a) 0.75m/s, b) 2.68m/s and c) 4m/s [21].

3. Experimental Methods

This chapter discusses the materials used, sample preparation, characterization techniques, and experiments performed used to obtain the results.

3.1 Materials

Materials preferred for this study are the ingots of Ti6Al4V alloys produced by casting as received from Dayton, OH. The casted ingots of Ti6Al4V alloy were cut into the standard specimen size of 60 X 60 X 25 mm for performing the wear experiments. Induction skull melting (ISM) process is preferred to melt the parent material Ti64. This ISM uses a high-temperature induction coil in a controlled atmosphere to liquify the metal and offers superior advantages like the high yield of molten metal and less melting time as compared to other conventional melting processes. The unique thing about ISM is, it uses a watercooled, copper crucible to melt a metallic charge which then can be segmented cylindrical walls. The molten metal solidifies against these segmented walls forming a thin layer (skull/skeleton) on the surface. Highly reactive metals such as Titanium, Zirconium, etc. can be melted in induction skull melting furnace. After melting the charge, boron is added in the elemental form, ensuring the uniforming mixing in the melt. The impurities and porosities induced during solidification process are removed by heat treating the casts using Hot Isostatic Pressing (HIS). This HIS process contains the heating of solidified metal up to 1173 K followed by a holding time of two hours with a set pressure of 100 MPa. As such, three compositions were made and used for wear experiments with a % wt of Boron 0, 0.3, and 0.55. These boron added alloys are cut in the shape of pins having dimensions 8 mm in diameter and 40 mm in length. A highly efficient wire cut EDM process was preferred to cut the pins from as received blocks of Ti6Al4V alloys due to less material loss and superior surface finish. Materials used for performing the experiments is shown in Fig. 3.1.

3.2 Experimental Procedure

3.2.1 Equipment

The wear experiments are performed on a pin on disk tribometer of Ducom make. The pin-shaped samples are held in a sample holder, and wear occurs when the sample slides against the rotating hardened EN-31 steel disc. The power supply for the rotation of the disk is supplied through an electric motor controlled manually by the control unit. A lever arm with various weights is provided at the back side of the machine which loads the samples mechanically. Real-Time monitoring and data acquisition of frictional force are done with the help of piezoelectric transducer. High temperature wear experiments are carried out in a perforated chamber, heating both sample and disc in the range of room temperature to 300°C. The schematic of a tribometer is shown in Fig. 3.2 [22]

3.2.2 Wear Experiments

High temperature wear experiments are performed on pin disc tribometer in the range of 300 K to 573K. The sample and disc both are heated up to the predetermined temperature followed by a dwell time of 10 to 15 min to ensure the complete homogenization. The temperature is controlled with the help of a thermocouple mounted inside the chamber. The disc was allowed to rotate at a constant speed of 650 rpm, having a constant load of 25 N on the sample. To observe the wear tracks on the samples, the pin was held at an offset distance of 60 mm from the center of the disc. The setup was allowed to run for 30 minutes, and the total distance traveled by the pin was recorded as approximately 3675 m. To ensure complete contact is maintained between the pin and the disc, the surface of the pins are grounded by emery papers up to 600 μ m grit size and then washed with ethanol for obtaining a clean surface. Tests are performed by following the ASTM G99/95a course of action [23].

The real-time monitoring of wear was done with the help of a linear variable differential transformer (LVDT) attached to the lever arm. After each experiment, the surface of the disk is polished with the emery paper of $600 \,\mu\text{m}$

grit size. The pin is then sectioned with the help of an abrasive cutter up to 2 mm depth for further examination.

3.2.3 Data interpretation/calculation

The test parameters that we get include -

Time – time recorded for a particular data point.

Load - dead weight applied to produce normal force.

Wear – loss of material/reduction in the height of the specimen.

Speed – the rotational speed of the disc.

Friction force – force at the contact point of pin and disc.

Temperature – the constant temperature inside the chamber.

The results of the sliding experiments are expressed in the form of wear rate. The wear rate is calculated as either volume loss per unit time or volume loss per unit distance traveled. Volume loss (V) can be calculated from the following equation –

$$\mathbf{V} = \boldsymbol{\pi}^* \mathbf{r}^{2*} \mathbf{w}^* \mathbf{10}^{-3} \,(\mathrm{mm}^3) \tag{1}$$

Where r = radius of the pin (in mm)

w = wear of the pin (in μ m), obtained from the experiment.

Distance traveled by the pin is obtained from the following equation

$$\mathbf{D} = \mathbf{R}^* \mathbf{t}^* \boldsymbol{\pi}^* \mathbf{d} / \mathbf{60} \ (\mathrm{m}) \tag{2}$$

Where R = rotational speed of disc (rpm)

t = time (sec)

d = Wear track diameter (mm) = 60 mm

Wear rate (w) is then calculated as,

$$\mathbf{w} = \mathbf{V}/\mathbf{D} \ (\mathbf{m}\mathbf{m}^3/\mathbf{m}) \tag{3}$$

3.3 Characterization techniques

3.3.1 X-ray diffraction spectroscopy (XRD)

The possible phases and crystal structure identification of Ti6Al4V alloys before and after wear experiments are performed using X-ray diffraction (XRD) study. X-ray diffractometer uses an X-ray detector consists of a cathode ray tube (CRT), and area detector mounted on the arm to collect the diffraction patterns. The sample and the detector can be rotated to enable scanning at all the possible angles. A typical XRD setup used in this study is shown in Fig.3.3.

Monochromatic x-rays are generated in a cathode ray tube by heating the filament. Once it reaches sufficient temperature, it generates high-speed electrons which then strike on to the target material. This produces characteristic X-ray spectra. These x-rays are then collimated and directed on to the sample. In the current work, XRD patterns of as received and worn out Ti6Al4V alloys are obtained using X-ray diffractometer of Smart Lab make which works on the Bragg's principle of diffraction. In this work, the XRD patterns were obtained in the range of 10 to 80° angle to observe the presence of any oxides in the specimens. The observed XRD peaks of as received and worn out Ti6Al4V alloy are matched with JCPDS (Joint Commission on Powder The observed peak intensity and constructive interference are obtained from the following equation and presented in Fig. 3.4.

$n\lambda = 2d*sin\theta$

where λ = wavelength of the x-ray

- d = interplanar spacing between the crystal planes
- θ = diffraction angle
- n = integer number

3.3.2 Scanning electron microscopy (SEM)

A scanning electron microscope uses an electron for magnification. The wavelength of visible light limits an optical microscope's magnification; thus, SEM is used where even higher magnification is required. It is extremely useful while working with materials on a nanoscale. These materials are far too small for getting a detailed image from a regular optical microscope.

SEM like XRD also uses X-rays to strike the specimen with electrons. These electrons when interacting with the sample, produces various other signals such as secondary electrons (produces SEM images), back-scattered electrons (BSE), diffracted back-scattered electrons (EBSD) used to determine crystal structures and orientation), photons (characteristic x-rays that are used for elemental analysis and continuum X-rays), visible light (cathodoluminescence), and heat [24]. Secondary electrons and BSE are most commonly used for imaging purposes. While SE are used to study the morphology and topology of the surface, BSE are used to illustrate the contrast between the multiple phases present in the specimen.

3.3.3 Optical microscopy

The microstructural analysis of subsurface of worn out Ti6Al4V alloy is performed using an inverted optical microscope of Carl Zeiss make. The purpose of carrying out optical microstructural examination of the subsurface is to confirm the behavior of material just beneath the worn out surface.

3.4 Sample preparation

The as-received Ti6Al4V alloys are cut into the cylindrical disc shape having diameter 8 mm and thickness of 2 mm. These cylindrical disc-shaped samples

are then hot mounted using SimpliMet 3000 mounting press of Buehler make. The samples are placed in the mounting mold and mounted with resin powder. The hot mounting of samples is performed under high pressure applied pneumatically and high temperature. Due to the high temperature of the mounting chamber, resin powder starts melting and covers the sample in predetermined shape and size. The hot mounted samples are ground on EcoMet polishing machine of Buhler make with the grit size 220, 320, 400, 500, 600, 800, 1200, 1500, 2000, and 2500 μ m. The fine polishing of all the Ti6Al4V alloy samples is done gently with the slow speed (150 rpm) around 2-3 minutes. The orientation of the sample is altered by 90° on each paper to cancel out the scratches produced while grinding. This is followed by diamond polishing with a three μ m diamond paste. The specimens are then ultrasonically cleaned in ethanol for 5 minutes and then dried in air.

Chemical etching is performed on the specimen after metallographic preparation using a suitable etchant. For Titanium alloys, Kroll's reagent, which is a mixture of 1.5ml hydrofluoric acid, 2.5ml nitric acid, and 46ml distilled water, is used. The surface of the specimen is gently wiped with a cotton swab dipped in the solution for around 15-20 seconds. This process is followed by rinsing of the sample in running tap water for 10 seconds. This process oxidizes the grain, lath, and colony boundaries, highlighting them and enables us to see the microstructure.



Fig. 3.1 a) Samples used for hardness experiments, b) Pins used for wear tests.



Fig. 3.2 The schematic diagram illustrating the key components of the pin on disc tribometer.



4-Circle Gonoimeter (Eulerian or Kappa Geometry)





Fig. 3.4 Bragg's law. The two rays exhibit constructive interference when the distance between the two parallel paths differs by an integer number of wavelength.

4. Results

In this chapter, we present the wear results and the microstructural and topographical analysis of all the alloys used in the study.

4.1 Initial Microstructure

The initial microstructures of the three alloys are shown in Fig. 4.1. Ti64 alloys have a lamellar microstructure (β -annealed) consisting of alternate layers of hexagonal close-packed (hcp) α and body-centered cubic (bcc) β layers distributed in prior β -grain boundaries. The α -laths are thicker than β -laths and have a dark appearance as opposed to the light, almost white color of the latter. Several of the α , β colonies are inside a single prior β grain with random orientation. The plate-like α - β laths that make up the colonies are, however, oriented in preferred directions according to Burger's orientation relationship between β and α . The α laths intersect each other in a pattern similar to a basket weave, therefore also known as a basket weave structure. Trace boron additions to the Ti64 alloy introduces thin TiB whiskers with aspect ratio 9~10 along the grain boundaries, forming a necklace like structure. The thickness of the whiskers varies from 2-5 µm. These TiB particles are efficient strengthners as well as play a huge role in the grain refinement by acting as heterogeneous nucleation sites in the matrix during solidification. Boron addition also alters the lath size.

4.2 Initial properties

The variation of hardness with the B content for as-cast Ti64 alloys is shown in the Fig.4.2. The plot indicates that the hardness increases almost linearly with the boron concentration. This is due to the fact that with the increase in boron concentration, the volume fraction of TiB particles also increases. Sen et al., [14] calculated the volume fraction for different alloy compositions and found that it follows the equation -

$$V_{TiB} = 5.23 * wt.\% boron \tag{1}$$

They also calculated the grain size and found that it is reduced by almost 91% by addition of only 0.1% boron while a further add up to 0.55% reduces it by

around 10%. Similarly, the lath size is reduced by 52% up to 0.55% addition. The Fig. 4.3 shows the variation of lath sizes with increasing boron content.

4.3 Wear data analysis

The pin on disk wear test gives wear, w, as height loss of the pin in microns vs. time, t. The variation of wear loss with time at a peak load of 25N, the temperature of 300 K, and a velocity of 2.4. m/s is shown in Fig. 4.3. The initial part of the curve is erratic and nonlinear, and after some amount of time, it becomes linear. The period in between is known as the break-off period. The initial part of the curve indicates that the contact between the pin and the disk surface is incomplete, which results in uneven wear of the pin. The incomplete contact may be due to the irregularities on the pin surface, which arise during specimen preparation. Although the pin surfaces are polished before the experiments, it is difficult to establish a conforming contact between the pin and the disk surface. As the test progresses, the irregularities are worn off, resulting in complete contact between the surfaces. The wear of the surface becomes more or less uniform, and it attains a steady state. The oscillations on the data could be attributed to the formation of the wear debris which lies between the pin and disk and thus causing serrations in the data. In the steady state, it is possible to describe the relationship between wear loss and time by a linear fit. It is also possible to obtain the coefficient of friction by dividing the tangential force with the normal force. The friction coefficient is plotted against the distance traveled by the pin in Fig. 4.4 and is observed to be constant throughout the test after steady-state conditions are achieved. The COF values for the given set of experimental conditions (T- 300 K, sliding velocity 2.4 m/s) for the three alloy compositions is found to be in the range 0.17-0.26 with highest for 0.3 B and lowest for 0.55 B. At a given sliding velocity, several factors, e.g. the local increase in temperature, formation of TiO, presence of wear debris between the pin and disc, internal microstructure of the material, etc., influences the COF values.

Wear rate, η is calculated to compare the different results. It is defined as volume loss per unit distance traveled by the pin (mm³/m). Three types of graphs are

plotted here, wear rate against the experimental parameters temperature, concentration, and sliding velocity, as shown in Fig. 4.5, 4.6, and 4.7.

Fig. 4.5 shows the wear rate variation with respect to temperature for different alloy composition at 2.04 m/s. The slope of the curve changes in all temperature ranges indicating that different wear mechanisms are taking place in each range. It is observed that the n decreases by about 10% from 300K to 373K. This is in complete contrast with the pin heating experiments where η increased for the same range. This may be due to the heating of both the pin and the disk in our experiments as the disk loses some of its hardness on getting heated. Further from the temperature range of 373 to 473K the η is almost constant for 0 and 0.55B alloys but decreases by 10% for 0.3B to the lowest value. This is again contradictory to pin heating results where although the η still increased its increase was reduced to 2.2% from the 37.5% for the 300-373K range. For the range from 473 to 573K the wear rate increases for 0 and 0.3B alloys by 10 and 16.57% respectively while for 0.55B it is decreased by 2.5% which is negligible. Ti64 has the highest wear rate for 573K, similar to pin heating experiments. Graphs indicate 0.55B has a higher wear rate than 0.3B at all temperatures except 573K where the two alloys have similar rates. 0 and 0.3B have similar performance from 300 to 450K. After 450K wear rate decreases for 0.3B being the lowest at 473K and then again increases up to 573K.

Fig. 4.6 shows the variation of wear rate for different Ti-64 alloys with the sliding velocity at 300K. It is clear from the plot that η increases with sliding velocity for all the alloys. η is nearly the same for all at lower speeds; however, there is a clear distinction between η at a higher speed of 4 m/s. η increased by about three times for 0.0 and 0.3B alloys when velocity was increased from 1 to 2 and then 2 to 4 m/s. For 0.55B alloy the increase in η was halved for velocity range from 2 to 4 m/s. At 4 m/s the wear rate is highest for 0.0B and lowest for 0.55B alloy. A surprising result is obtained at 573K where pure Ti-64 has the lowest wear rate at highest speed, and 0.3B has the highest wear rate.

Fig. 4.8 shows the variation of coefficient of friction, f (COF) with temperature for all alloys at 2.04 m/s. The COF shows a more or less increasing trend with temperature neglecting the small differences in the wear rates for 0B and 0.3B

alloys up to 450K. An interesting observation is that for 0.55B alloy despite the decreasing trend in wear rate with temperature, the COF is increasing with it.

4.4 SEM analysis

SEM analysis of the wear tracks on all the samples. It gave us an idea of macroscopic wear mechanisms occurring on the samples. The worn surfaces are shown in Fig. 4.9. Distinct wear features can be seen on the alloys such as adhesive transfer of the material in the form of flat platelets for lower temperatures and velocity. While at high temperature and velocities abrasive features such as deep grooves, material pull-outs, and

delamination is seen. It is observed that with increasing boron concentration, the tendency for abrasive and delamination wear is more in the form of closely spaced grooves and detachment of large particles from the subsurface layers. Also, the grooves are deeper and wider for increased boron content in the alloy.

4.5 Profilometer analysis

The contact profilometer analysis of the wear samples was done. There is a general trend of increase in the average value of surface roughness R_a with boron content and temperature. There is an increasing trend with sliding velocity too, but there are some exceptions to it. The possible reasons for the anomalous behavior of some samples will be discussed in the next chapter.

4.6 XRD analysis

XRD spectra of the wear samples are shown in Fig. 4.10. The peaks are matched with the standard crystallographic data. TiO peaks are found at 1 and 4 m/s for all the samples at all the temperatures indicating the formation of tribo oxides while at the intermediate speed of 2 m/s no such peaks are found. Other than TiO α -Ti peaks are found in all samples.

4.7 Subsurface microstructures

Cross-sectional optical micrographs were taken for selected samples. The micrographs are shown in Fig. 4.11. Very less kinking is observed in comparison to the pin heating experiments. In addition to this, a separate layer is observed for samples run at 1 and 4 m/s speeds above the wear surface.







Fig. 4.1 Optical microstructures of boron modified Ti64 alloys with (a) 0.0B, (b) 0.3B and (c) 0.55B



Fig. 4.2 Hardness variation of alloys with increasing boron concentration.



Fig. 4.3. Variation of lath sizes with increasing boron concentration.



Fig. 4.4 Variation of wear, w with time, t.



Fig. 4.5 Variation of friction coefficient with sliding distance.



Fig. 4.6 Variation of wear rate, η with temperature, T for different Ti-64-B alloys at 2.04 m/s.



Fig. 4.7 Variation of wear rate, η with sliding velocity, v_s at 300K for Ti-64-xB alloys.



Fig. 4.8 Wear rate variation with sliding velocity at 573K for Ti-64-xB alloys.



Fig. 4.9 Coefficient of friction with temperature for the alloys at 2.04 m/s.



Fig. 4.10 SEM images for worn surfaces tested at 2 m/s, 25N of composition (a), (d) 0B, (b), (e) 0.3B and (c), (f) 0.55B at 300 and 573K temperature respectively.



Fig. 4.11 SEM images for worn surfaces tested at 1 m/s, 25N of composition (a), (d) 0B, (b), (e) 0.3B and (c), (f) 0.55B at 300 and 573K temperature respectively.



Fig. 4.12 SEM images for worn surfaces tested at 4 m/s, 25N of composition (a), (d) 0B, (b), (e) 0.3B and (c), (f) 0.55B at 300 and 573K temperature respectively.



Fig. 4.13. Representative contact profilometer images for the worn surfaces tested at 2 m/s, 25N and composition and temperature of (a), (b) 0B, (c), (d) 0.3B and (e), (f) 0.55B at 300 and 573K respectively.





Fig. 4.14 XRD patterns of the alloys at (a) 300K, (b) 573K tested at 2 m/s, 25N.



Fig. 4.15 XRD pattern obtained for samples tested at 25N, 1m/s at 573K.



Fig. 4.16 Subsurface microstructures of alloys tested at 25N, 2.04 m/s with composition and temperature of (a), (b) 0B, (c), (d) 0.3B and (e), (f) 0.55B at 300 and 573K respectively.
5. Discussion

In this chapter, we will try to analyze the results critically and give probable justifications.

5.1 Analysis of pin on disk results

In Fig. 4.4 we see the wear rate(η) change with temperature in 0, 0.3, and 0.55B alloys. All three alloys show a distinct behavior. For the temperature range of 300 to 473K 0 and 0.3B alloys have similar wear rates while 0.55B has larger rate than both alloys. This is similar to pin heating experiments. However, since in chamber heating, we are heating both the pin and the disk, the disk loses some of its hardness on getting heated, and thus we have obtained lower values of wear rates by about 34% at all temperatures. For temperature from 473 to 573K wear rate is increasing for 0 and 0.3 but decreasing for the 0.55B alloy. In fact, 0.55B continuously shows a decreasing trend in η . This may be attributed to the hardness of the specimens wherein 0.55B is the hardest (35HRC) while 0B has the least (29-30 HRC), so, hardness lost is least for 0.55B. It also has the highest volume fraction of TiB particles as well, which are almost twice as harder as base Ti64. Table 5.1 shows the effect of different properties on wear rate.

Factors affecting the wear rate (increasing)	Effect on the wear rate	Effect on COF
Hardness	t	t
Elasticity modulus	ŧ	↓
Sliding velocity	Ļ1	↓†
Fracture toughness	t	NA
Temperature	↑ ↓	† ↓
Nirmal load	t	1

Table 5.1. Effects of various factors/properties on wear rate and COF.

From table 5.1, we see that η also decreases with increasing elastic modulus. 0.55B also has the highest elastic modulus of all the three alloys, which might explain its decreasing trend. It has a higher η at temperatures 300 to 473K as TiB particles often come out and act as the abrasive particles themselves, leading to three body abrasion as stated earlier.

In Fig. 4.6 and 4.7 the wear rate is increasing with the increase in sliding velocities. According to research conducted by Molinari et al. [] on increasing sliding speed, the contact time decreases but the flash temperature at the contact point increases. Due to the increase in temperature wear increases because of the thermal softening as the yield point of material decreases significantly. At room temperature, the η is lowest for 0.55B and highest for 0B whereas at 573K 0B possess the lowest η . This sudden decrease can be better explained in the following sections.

Fig. 4.8 shows the coefficient of friction with temperature. It is increasing with the temperature for all the cases. We see that COF increases rapidly from room temperature to 373K, and it is almost constant at higher temperatures. This is because at elevated temperature, the possibility of oxidation of Ti64 increases, which may reduce the COF as oxides have a lubricating effect. Also, material softening at higher temperatures tend to increase COF. At 2 m/s as we will see in the coming section that no oxides were found, so COF was always increasing in our case.

5.2 Wear morphologies of the worn surfaces

The wear morphologies for the worn out surfaces at room and high temperature are presented in Fig. 4.10. As we have already stated the wear for our samples is lower than pin heating experiments, we can see this clearly in SEM images as well. While abrasive wear with intensive shearing was observed for pin heating, here we have slight adhesive features with abrasive wear at room temperature. The shearing is mild at room temperature with shallow grooves. Grooves are formed due to the sliding action of wear particles and debris which acts as abrasives themselves. Due to TiB particles present in alloys, this phenomenon is dominant. At high-temperature severe wear with delamination is observed. The grooves are much deeper and wider, indicating high abrasive wear caused by the debonded TiB particles. Debonding of the TiB depends on the sliding conditions, temperature, load, velocity. High temperature, along with velocity causes huge pile-up stresses that further causes the removal of these particles. Also, it is stated earlier that the coefficient of thermal expansion is different for TiB and Ti matrix (TiB- 6.4x10⁻⁶/K and Ti64- 8.6x10⁻⁶/K at 250°C [25]). At elevated temperatures, its effect can be enhanced. In Fig. 4.10 (f) we can see

the material removed in the form of pull outs which can be attributed to the plowing action of TiB. The hard particles when penetrate the matrix they cause cracks which propagate into the subsurface, which then coalesce, and material is removed in the form of laminates [12]. For 0.55B, the wear rate is lowest at a high temperature, which is a contradiction, as it has the highest probability of debonding. But SEM image shows that there is much less delamination seen in the alloy. This can be the combined result of high hardness, the elastic modulus of the alloy together with the loss of hardness by the disk at high temperature.

For the 1 m/s tests, we have observed that wear rate is decreasing on increasing temperature. This is evident from SEM images in Fig. 4.10. Abrasive grooves can be seen in all the images however, they are more pronounced at room temperature. No delamination is observed as the speed is low. The reduced wear can be attributed to the presence of oxides (prevalently TiO) confirmed by the peak obtained in XRD results presented in Fig. 4.15. A similar pattern was observed in samples at 4 m/s. TiO is found in the samples for speeds of 1 and 4 m/s. But none such peak was present at 2 m/s. XRD pattern for samples tested at 2 m/s is given in Fig 4.15. We can say that the wear is oxidative for 1 and 4 m/s and it is metallic at 2 m/s.

Much research has been conducted to determine the effect of tribo-oxides on wear behavior. Alam and Haseeb [26] concluded that no protective oxide layer and severe delamination is responsible for the low wear resistance of Ti64 alloy. Qiu et al. [27,28] carried a tribo study in Ti64 in the speed range of 30-70 m/s. Oxides formed in the order of TiO, TiO₂, and V₂O₃. He stated that low wear resistance was due to the loosen oxide layer. Above researchers concluded that Ti64 has loose and brittle oxide layers that do not provide a protective function. However, in research conducted by Chelliah et al. [29], they found that wear rate was decreasing, then increasing for a speed range of 0.01-1 and 1-1.4 m/s respectively. They concluded that behavior was governed by a combination of tribo-oxides and strain response and that oxides can play a protective role too. Molinari et al. [20] found the presence of oxides at low and high speed but none at intermediate speed (speeds being 0.3, 0.5 and 0.8 m/s). They found the wear debris mainly consisted of oxide particles at 0.3, metallic and some oxide fragments at 0.5 and mostly metallic at 0.8 m/s. Their results confirmed that Ti64 has a low resistance to plastic deformation even at low loads. This causes instability of the surface layers, which leads to decreased protection from oxide layers. X.X Li et al. [21] conducted experiments on Ti64 in the range of 0.5-4 m/s. They stated that tribo-oxides and tribo-layers are two separate things. Tribooxides are formed by the combination of metal wear debris and oxygen. Whereas, tribo layers are mechanically mixed layers (MML) produced when tribo-oxides and metal debris combine. Thus, when tribo-oxides are present in tribo-layers, they affect the wear behavior.

Further, they found that the formation of tribo-oxides increased with sliding speed. This is due to the increased frictional heat. But, tribo-layers formation depended on whether oxides were worn away due to centrifugal forces or remained adhered to the surface under sliding conditions. These tribo-layers posses lubrication tendency and can avoid metal-metal adhesion, thereby reducing wear while movable oxide particles caused abrasion and increasing wear.

Now in our results, we obtained no oxides at 2 m/s. These may have happened as with the increase in speed the centrifugal force drove away most of the oxides formed, and hence tribo layer did not form. However, at 4 m/s, as stated by X.X. Li [21] the frictional heat that was produced was able to trap oxide particles along with the high pressure and temperature, which resulted in the formation of the tribo layer. So wear rate for 0B decreased at 4 m/s at 573K due to the reasons mentioned above. Now, not much decrease was obtained in the 0.3 and 0.55B alloys. TiB particles may be responsible for this as at high temperature and sliding speed debonding may increase, causing TiB particles to remove or break-down the tribo-layers.

We see in Fig.4.11 the representative profilometer images. We can see that the spacing of asperities is decreasing with increasing boron concentration, which is due to the reduction in grain size with the same. We can relate this with the higher abrasive wear for the 0.3 and 0.55B alloy.

5.3 Subsurface microstructures

The subsurface optical micrographs for the alloys are presented in Fig. 4.16. Much less kinking is observed as compared to pin heating experiments except for 0B alloy. Kinking of laths happens due to high shear stresses at the pin and disk contacting point. This is a dominant mechanism which is responsible for material removal. Higher kinking suggests material can sustain higher shear stresses without breaking away. Due to chamber heating, the shear stresses were lower than pin heating, and thus much less kinking was observed.

6. Conclusion and future scope

Alloy compositions containing boron are complex structures. They show varying wear behavior under varying conditions of sliding. Since they contain a hard second phase in the form of TiB particles, it is difficult to predict their wear behavior.

In our experiments, 0.55B possess better mechanical properties, but the alloys show mixed results depending on the sliding conditions. The general trend with increasing velocity is increase in wear rate. However, with increasing temperature alloys show mixed results. At 1m/s 0.3B alloy has the lowest wear rate; at 2 m/s 0.55B and 4 m/s 0B has the lowest at the temperature of 573K. We observed oxide formation at low and high speeds; none was found at medium speed. The formation of oxides, along with hard TiB particles, is suspected to be responsible for these contrasting results.

Other compositions of Ti64 alloy like 0.06, 0.08, 0.09, 0.11B needs to be explored for their wear behavior. With much less volume fraction of TiB particles than 0.3 and 0.55B alloys, these alloys may provide better results for same conditions.

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