

WEAR BEHAVIOUR OF Ti6Al4V ALLOY AGAINST Al₂O₃ UNDER LINEAR RECIPROCATING SLIDING

F. ZIVIC^{a*}, M. BABIC^a, I. CVIJOVIC-ALAGIC^b,
S. MITROVIC^a, A. VENCL^c

^a Tribology Laboratory, Faculty of Mechanical Engineering, Kragujevac, Serbia
E-mail: zivic@kg.ac.rs; babic@kg.ac.rs; boban@kg.ac.rs

^b Vinca Institute of Nuclear Sciences, Belgrade, Serbia
E-mail: ivanac@vinca.rs

^c Tribology Laboratory, Faculty of Mechanical Engineering, Belgrade, Serbia
E-mail: avenc1@mas.bg.ac.rs

ABSTRACT

Tribological behaviour of four different heat-treated Ti6Al4V alloys, during linear reciprocating sliding against alumina, on the microscale was investigated. Experiments were carried out for dry sliding and in the Ringer solution, over a range of loads (100–1000 mN) and speeds (4–12 mm/s). The wear mechanisms were investigated based on observations of worn surfaces. Specific wear rates for tested Ti6Al4V alloy were of order of 10^{-7} – 10^{-4} mm³/N m. The lowest wear factor (order of 10^{-7} mm³/N m) was observed for the Ti6Al4V annealed for 1 h at 750°C in Ar atmosphere and then cooled down to room temperature in the furnace, tested in the Ringer solution. Load dependence of the wear factor exhibited transition characteristics. Wear mechanism has changed with change of load. The Ringer solution lowered wear factor for all tested conditions.

Keywords: Ti6Al4V, alumina, wear mechanism.

AIMS AND BACKGROUND

Tribological applications in many new fields of advanced materials applications, such as biomedical implants, impose growing challenges for engineering materials. Titanium and titanium alloys has been applied for medical purposes for years^{1–4}. Extensive research activities in the manufacturing process and surface modification of Ti alloys are undertaken to improve its properties. Ti alloys are applied in cases when high strength and low density are of primary importance. They are particularly interesting because of their excellent biocompatibility, the

* For correspondence.

low Young modulus and high corrosion resistance¹⁻⁶. Implants made of titanium alloys tend to develop structural and functional connection between the living bone and the surface of an implant (osseointegration). Despite their good mechanical and chemical properties and low density (4.5 g/cm³) the use of Ti alloys for structural applications is prevented by their poor wear resistance¹⁻¹⁰.

The tribological behaviour of Ti alloy is highly influenced by the flash temperatures generated at asperities contact⁷. High temperatures promote formation of a Ti surface oxide which influences tribological behaviour of the titanium alloys⁸. High flash temperatures also govern plastic deformation processes at interface zone⁷. Different thermal treatment procedures have been applied to obtain better characteristics of Ti alloys¹¹⁻¹³.

Sliding wear behaviour of Ti6Al4V alloy has been a subject to a relatively small number of investigations^{5,8-10}. However, the acting wear mechanisms during sliding wear of titanium alloys have not been sufficiently addressed and understood and needs to be further studied, as reported in literature by different authors^{5-8,12}. It is needed to better understand and systematically study of parameters and mechanisms influencing friction and wear of Ti6Al4V alloys and their dependencies on sliding speed and load, also in specific cases on the microscale.

Ceramics are regarded as favourable materials for joints or joint surface materials. Conventional ceramics such as alumina (Al₂O₃) have been evaluated due to their excellent properties of high strength, good biocompatibility and stability in physiological environments². Alumina can be polished to a high surface finish and has excellent wear resistance, and as such is often used for wear surfaces in joint replacement medical prostheses^{8,9}.

Within this study, testing of Ti6Al4V alloy against alumina has been done, by varying normal load and sliding speed. Alumina has significantly higher hardness than the titanium alloy, and shows outstanding abrasion resistance. Therefore, deformation processes during sliding can be assigned to Ti alloy only⁸. The aim of this study was to investigate tribological behaviour of Ti6Al4V alloy in contact with Al₂O₃, on the microscale, during linear reciprocating sliding, in regard to four different heat treatments of Ti6Al4V alloy, in dry conditions as well as using the Ringer solution as the environmental fluid. Wear response of heat-treated Ti6Al4V alloy has been analysed, based on the wear factor and using optical microscopy.

EXPERIMENTAL

Materials. The material studied was Ti6Al4V alloy; chemical composition in mass percent of alloying elements: 6.1% Al, 3.95% V, 0.15% Fe, 0.14% O. Flat samples, 6.35×15.75×10.16 mm in size, were cut from industrially produced bars. Ti6Al4V samples were subjected to four different thermal treatments. The first and the second group of the samples (denoted by Ti64_1000_F and Ti64_1000_W further

in the text) were annealed at 1000°C for 1 h in a protective Ar atmosphere. Then, Ti64_1000_F sample was cooled down in the furnace to room temperature. Second group (Ti64_1000_W) was quenched in water afterwards. The third and the fourth groups of samples were annealed for 1 h at 750°C in Ar atmosphere. After annealing, the third sample group (denoted by Ti64_750_F) was cooled down to room temperature in the furnace, while the fourth one (denoted by Ti64_750_W) was quenched in water. All samples were then grounded with SiC paper (1000 grit) and cleaned for 30 min in an ultrasonic bath with ethyl alcohol, thus obtaining initial roughness of $R_a=0.3\ \mu\text{m}$. Final roughness of $R_a=0.07\ \mu\text{m}$ was obtained by polishing samples with diamond paste on abrading wheel.

Tribological tests. Sliding tests were carried out with a ball-on-flat configuration (Fig. 1) using CSM Nanotribometer Instrument. Tribological tests were performed with linear reciprocating module (stroke: 0.5 mm), by varying normal force values, F_n : 100, 250, 500, 750, and 1000 mN and maximum linear speed values, v : 4, 8, and 12 mm/s. Flat Ti alloy samples were in contact with a ball (1.5 mm diameter) made of Al_2O_3 . Duration of each test was 30 000 cycles (distance of 30 m), whereat one cycle is represented by full amplitude sliding distance (half amplitude, 0.25 mm). Selected sliding velocities lie in the range typically found in hip joints (0–50 mm/s). Two series of tests were realised: sliding in dry conditions and with the Ringer solution, as simulated body fluids. The Ringer solution was applied by immersing articulating surfaces totally in a solution. Composition of the Ringer solution is as follows (g per 1 l of water): NaCl – 8.6; KCl – 0.30; CaCl_2 – 0.33; Na^+ 147.00 mmol; K^+ 4.00 mmol; Ca^+ 2.25 mmol; Cl^+ 155.60 mmol.

Wear behaviour of the samples was monitored in terms of the wear scar width and length. Using the wear scar width and length and geometry of the contact pair (Fig. 1), the wear volume (in accordance with ASTM G133–02) and wear factor were calculated for each conducted test. Wear factor enables quantitative determination of contact conditions when the transition of mild to severe wear occurs.

Wear volume of the flat sample was quantitatively assessed for each test, after a total sliding distance of 30 m by measuring wear scar width and length

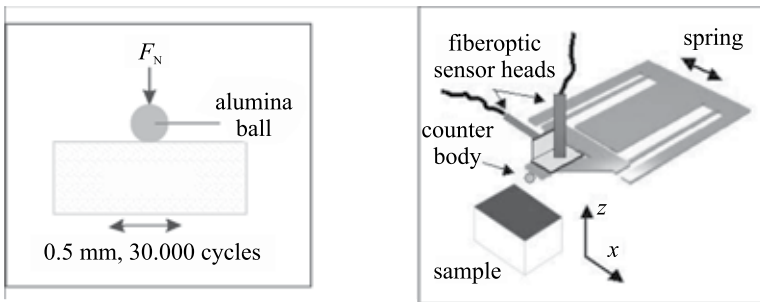


Fig. 1. Scheme of the contact pair geometry and nanotribometer contact geometry¹⁴

using optical microscopy (OM). Wear factor, i.e. a specific wear rate parameter, k (expressed in $\text{mm}^3/\text{N m}$) was calculated according to the following equation¹⁵:

$$k = V/(F s) \quad (1)$$

where F is the normal load; s – the sliding distance; V – the wear volume.

Wear volume of the flat sample, V in mm^3 , was calculated using average cross-sectional area of the wear scar and length of the wear scar, after simple geometrical calculation assuming that the cross-sectional area is represented by a flat segment of a sphere. Geometrical parameters of the sphere were adopted to correspond to that of the alumina ball in contact.

Samples were prepared for testing in accordance with ASTM F86–01. Each specimen was thoroughly cleaned by ethyl alcohol, then cleaned in ultrasonic bath for 60 min and dried in hot air afterwards. Then, samples were cleaned in isopropyl alcohol, by staying in the solution for 60 min and dried in a hot air. Samples were stored in a desiccator, prior to testing. The repeatability of the results for replicate tests was satisfying (coefficient of variation of wear scar width and length was under 5%).

RESULTS

The variation of the wear factor, k , as a function of applied load and sliding speed in dry and lubricated conditions, for tested Ti6Al4V alloy specimens, is illustrated in Fig. 2. Calculated k values were of order of 10^{-7} – 10^{-4} $\text{mm}^3/\text{N m}$. Generally, surface plots (Fig. 2) suggest that Ti alloy specimens exhibited different wear behaviour depending on the applied heat treatment. Ti64_750_F sample (annealed for 1 h at 750°C in Ar atmosphere and then cooled down to room temperature in the furnace) had lower wear factor than other tested Ti alloy samples in all combinations of applied loads and sliding speeds, both in dry sliding and with the Ringer solution. It exhibited the wear factor of order of 10^{-7} – 10^{-5} $\text{mm}^3/\text{N m}$.

The wear factor difference between heat-treated Ti alloy samples, is especially pronounced in area of low loads. Generally, wear level was the lowest for the highest applied load. Initially, wear level increased with increasing the load up to approximately 250 or 500 mN and then decreased with further load increase. Wear factor values are lower in the Ringer solution, under all load conditions and it was more prominent under low loads. Surface plots (Fig. 2.) suggest that the wear rate of the Ti alloy, regardless of applied heat treatment, increased with sliding speed at lower loads and for the highest load, had no influence.

Optical micrographs of worn scars produced during sliding are shown in Fig. 3. It can be seen that dark black wear debris is covering the wear scar, differently distributed around the sliding paths showed in Fig. 3a, b. Four different patterns of wear debris distribution were obtained for four applied heat treatments. In case of Ti64_1000_F (Fig. 3a), coarse wear debris were generated and large

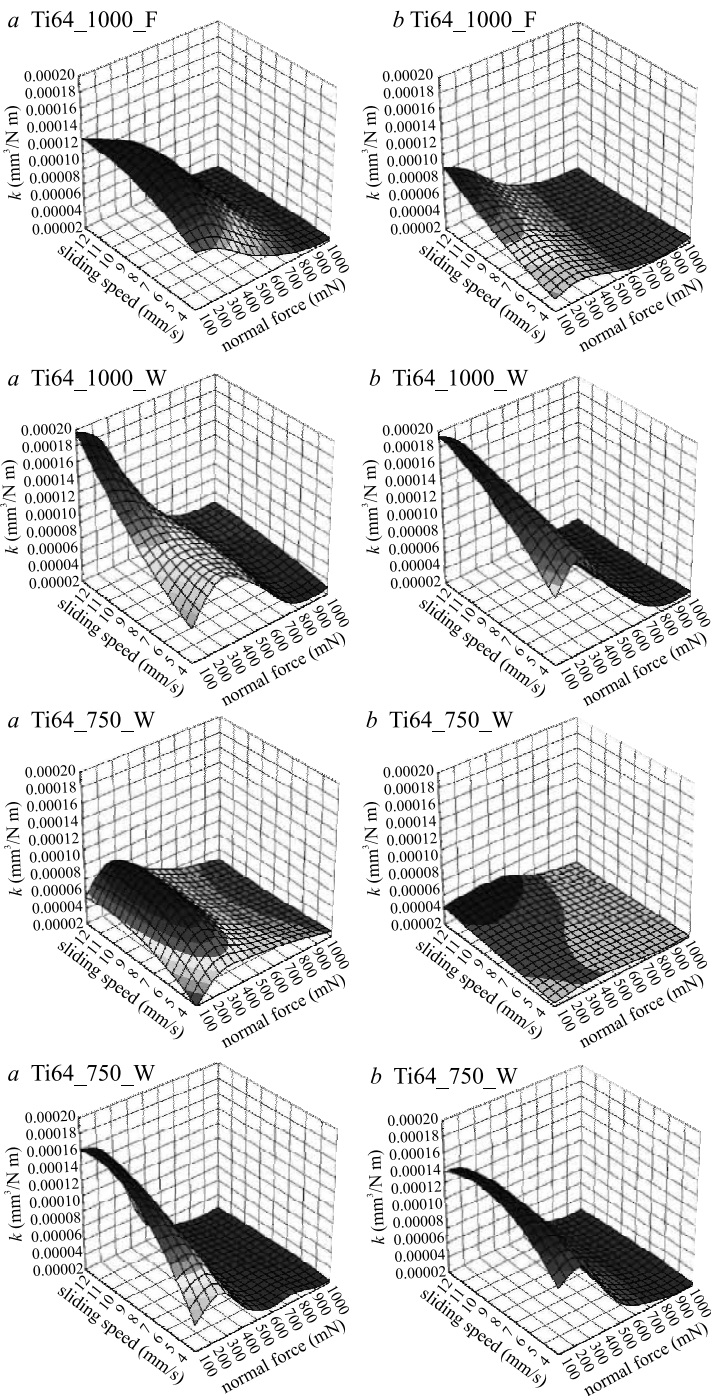


Fig. 2. Specific wear rate parameter: *a* – dry sliding, *b* – the sliding with the Ringer solution

debris agglomerates appeared far from the track. In case of Ti64_750_F, fine wear debris were observed (Fig. 3b), uniformly distributed along the wear track. Loose wear debris are gathered at the leading edge of the wear track.

Cleaned worn scars on Ti64_1000_F under different loads are shown in Fig. 4. Transition feature of load dependence can be clearly noticed, by different

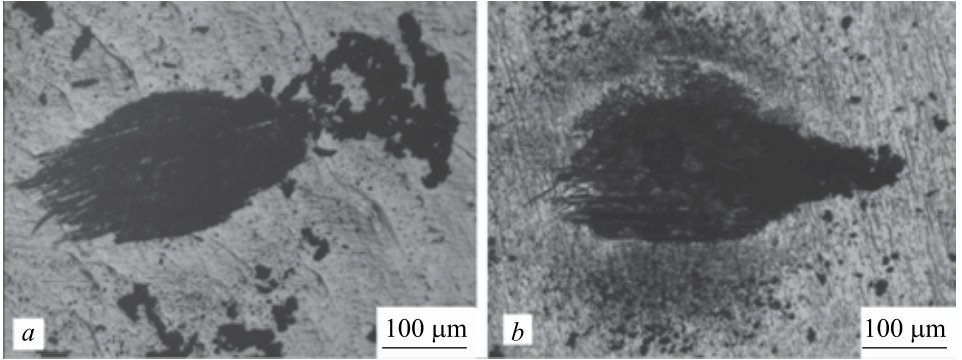


Fig. 3. Wear tracks on Ti6Al4V samples after sliding in dry condition (500 mN, 4 mm/s): *a* – Ti64_1000_F sample, *b* – Ti64_750_F sample

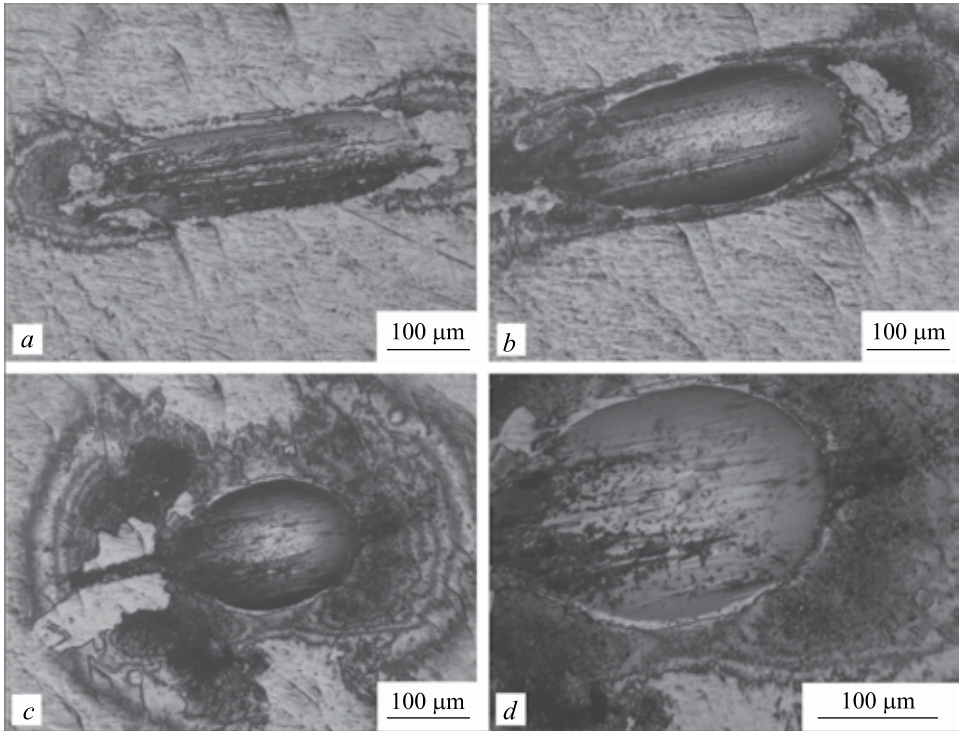


Fig. 4. Wear tracks on Ti64_1000_F after sliding, at different loads, in dry condition (4 mm/s): *a* – 100 mN, *b* – 500 mN, *c* and *d* – 1000 mN

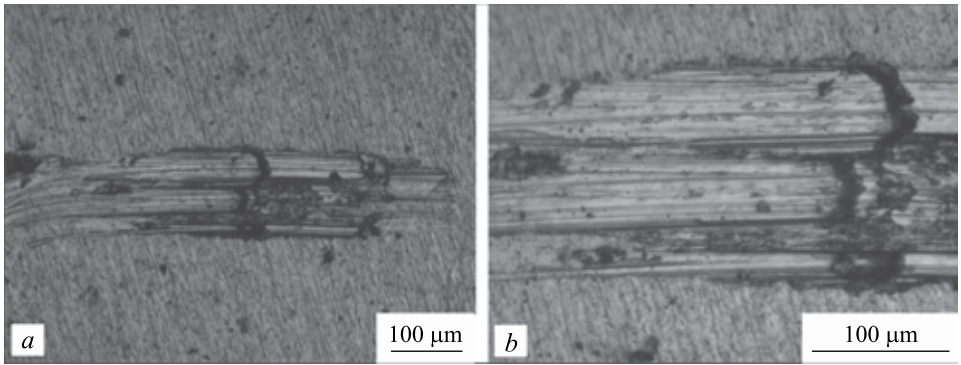


Fig. 5. Worn tracks on Ti64_750_F (1000 mN; 4 mm/s; the Ringer solution): central zone of the track (a); the same zone with higher magnification (b)

shape of the wear scar. The wear scar in case of 1000 mN load (Fig. 4c, d) is the smallest one. It can be clearly seen that the end regions of the wear scar was pulled up by adhesion and narrow delaminated area of irregular shape is surrounding the wear track. Higher magnification shows pits, cracks and delaminated plates, besides grooves, throughout the wear track (Fig. 4d).

The worn track on Ti alloy, after sliding in the Ringer solution, is shown in Fig. 5. Central zone of the track in Fig. 5a is shown with higher magnification in Fig. 5b. Deep longitudinal grooves can be clearly seen, whereat they appear ‘broken’ along the direction of sliding, mainly in the central region, where the sliding velocity is at its maximum. This phenomenon was observed at the highest load, for all tested samples. It can also be noticed that the worn scars (Fig. 5) did not exhibit either coarse or accumulated wear debris, but only small quantities of very fine wear debris.

DISCUSSION

The obtained results show that tribological improvement obtained by heat treatment applied in case of Ti64_750_F could be explained by formation of such a grain structure which promotes ductile fracture behaviour of surface layers. That, in return, promotes more favourable wear properties, corresponding to fine wear particles generation. Fine wear debris generated in case of Ti64_750_F (Fig. 3b) contribute to further wear in significantly less extent than clusters of coarse particles, which appeared in case of Ti64_1000_F (Fig. 3a). Specific wear rate presented in Fig. 2 also indicates significantly lower wear level in case of Ti64_750_F (more than 50% decrease of k factor), for experimental conditions showed in Fig. 3. Dong and Bell⁸ identified TiO, T₂O₃ and TiO₂ oxides after sliding of Ti6Al4V disk against alumina ball. Also, tribochemical reactions occurring between alumina and Ti6Al4V, under friction-generated temperatures in the contact zone produce Ti₃Al and TiAl, as reported in Ref. 8. Titanium oxides pro-

mote lower level of wear since they act as a protective barrier between alumina and titanium, according to many studies^{3,5,6,8}. On the other hand, intermetallics, such as Ti₃Al and TiAl promote extensive wear, because they additionally score the surfaces in contact^{7,8}. It is evident from results obtained within this study that heat treatment applied in case of Ti64_750_F provides fine wear particles resulting in lower wear level.

Different studies⁵⁻⁹ indicate that change of contact conditions (load, sliding speed) between Ti alloy and alumina results in change of governing wear mechanism. Wear factor diagrams and optical micrographs presented in this study also suggest that significant change of wear level can only be attributed to the change of wear mechanism with change of load. The effect of the load on the wear factor of the Ti alloy showing transition characteristic has also been reported by Dong and Bell⁸. They also reported that sliding speed dependence showed similar transition features. However, results obtained in our study do not suggest transitional feature of the sliding speed. Difference of obtained results occurs probably due to a fact that range of applied sliding speed was significantly lower in our study (4–12 mm/s) compared to range of sliding speed they observed (0.0625–1 m/s).

Visual observation of the worn surfaces of the Ti6Al4V samples indicates longitudinal grooves, scratches and irregular pits of different sizes (Fig. 4). End regions of the wear scars appear as jagged. It is obvious that abrasion and adhesion processes, as well as tribochemical oxidation, occurred during the sliding, what is in accordance with findings of other authors⁷⁻⁹. The observed grooves and scratches clearly characterise the occurrence of abrasive wear (Fig. 4b). Loose wear debris within the contact zone additionally score the surface, as it is ejected from the wear track. Delaminated areas indicate adhesion (Fig. 4c, d). Small black, randomly positioned plateaus appearing ‘gnarled’ when cleaned indicate fretted surfaces, i.e. tribo-oxidation (Fig. 4a). Larger areas of delaminated surface can be observed at the ends of the worn track (Fig. 4). This is in accordance with accumulation of loose debris, at the starting and/or ending edges of the wear track (Fig. 3), which promotes higher level of scoring and galling within these zones.

Wear factor values at 100 mN load (Fig. 4a) are significantly lower than those at 500 mN (Fig. 4b) and optical micrographs indicate tribochemical oxidation at 100 mN in higher extent than at 500 mN. Abrasive wear was dominant at 500 mN (Fig. 4b) and wear factor is the highest. Further load increase to 1000 mN promotes further change of wear mechanism. High load contribute to tribochemical reactions between Ti alloy and alumina, due to increase of the real contact area, probably resulting in delamination wear as a governing mechanism. Also, locally high flash temperatures, at asperities contacts, can be expected under high load.

According to many studies^{7,8,16,17}, when Ti alloy slide at high speed or under a high load, a high flash temperatures can be expected (up to 350°C for experimental conditions in this study). According to Hager et al.¹⁶, at 450°C the Ti6Al4V

specimens experience approximately 50% reduction in tensile strength, 40% reduction in compressive and shear strength, and 20% reduction in elastic modulus. Molinari et al.⁹ reported that the yield strength of Ti6Al4V alloy is highly sensitive to temperature (950 MPa, at room temperature; 650 MPa at 200°C and 450 MPa at 500°C). Consequently, Ti sample undergo material softening within the interface zone¹⁶. Temperature increase influences extensive plastic deformation and mechanical alloying of the materials in contact and favour plowing, galling and friction welding processes at asperities contact.

Some authors reported that tribological behaviour is controlled mainly by Ti surface deformation characteristics⁷. It can be clearly seen (Fig. 4c) that increase of load to 1000 mN produced delamination. The smallest quantities of wear debris were generated for 1000 mN, indicating that the governing process, in regard to wear, for these experimental conditions is plastic deformation, accompanied with very mild adhesive wear. Obtained results indicate that when wear debris are present at the contact zone in larger extent, abrasive wear mechanism was dominant and wear rate is the highest.

Presence of the Ringer solution produced lower wear factors (Fig. 2) and rather different appearance of wear tracks (Fig. 5), compared to dry conditions (Fig. 4). It appears that the Ringer solution provides surfaces in contact with sufficient lubrication. It seems that corrosion processes associated with influence of the Ringer fluid on Ti alloy³ do not play significant role under our experimental conditions. It is possible that the Ringer fluid contributes to reduction of abrasive wear, by removing wear debris away from the contact zone, thus inhibiting third-body abrasion by loose hard particles. It also decreases effects of adhesive wear because of the lubricating effect between surfaces in contact.

Evidence of the plastic flow can be clearly seen in Fig. 5 that is in consistence with previously stated, indicating low wear rate. Diagrams in Fig. 2 showed that wear level was the lowest for the highest applied load. This is in consistence with worn scars presented in Fig. 5, showing evidence of mild adhesive wear.

CONCLUSIONS

Four applied heat treatments of Ti6Al4V alloy caused four different wear levels for investigated Ti alloy. Annealing at 750°C in Ar atmosphere followed by cooling in the furnace to the room temperature, decreased wear factor by half as compared to other heat-treated samples.

Wear factor exhibited transitional characteristic. Initially, it increases with load increase to rapidly decrease to its smallest value, observed at the highest applied load. The Ringer solution lowered wear rate of observed Ti alloy in all experimental conditions.

Dominant wear mechanism changed with load change, from mild adhesive and tribochemical wear, at the lowest load, followed by severe abrasive wear ob-

served in range of middle load values, to very mild adhesive wear, at the highest applied normal load.

ACKNOWLEDGEMENTS

This study was financed by Ministry of Science and Technological Development, Serbia, project No 14005.

REFERENCES

1. J. BLACK, G. HASTINGS (Ed.): Handbook of Biomaterial Properties. Chapman & Hall, London, 1998.
2. M. GEETHA, A. K. SINGH, R. ASOKAMANI, A. K. GOGIA: Ti Based Biomaterials, the Ultimate Choice for Orthopaedic Implants – A Review. Progress in Materials Science, **54**, 397 (2009).
3. M. MASMOUDI, M. ASSOUL, M. WERY, R. ABDELHEDI, F. EL HALOUANI, G. MONTEIL: Friction and Wear Behaviour of cp Ti and Ti6Al4V Following Nitric Acid Passivation. Applied Surface Science, **253**, 2237 (2006).
4. J. D. BRONZINO (Ed.): The Biomedical Engineering Handbook, Medical Devices and Systems. CRC Press, Taylor & Francis Group, New York, 2006.
5. A. MOLINARI, G. STRAFFELINI, B. TESI, T. BACCI: Dry Sliding Wear Mechanism of the Ti6Al4V Alloy. Wear, **208**, 105 (1997).
6. G. STRAFFELINI, A. MOLINARI: Dry Sliding Wear of Ti–6Al–4V Alloy as Influenced by the Counterface and Sliding Conditions. Wear, **236**, 328 (1999).
7. M. LONG, H. J. RACK: Friction and Surface Behavior of Selected Titanium Alloys during Reciprocating-sliding Motion. Wear, **249**, 158 (2001).
8. H. DONG, T. BELL: Tribological Behaviour of Alumina Sliding Against Ti6Al4V in Unlubricated Contact. Wear, 874 (1999).
9. J. QU, P. J. BLAU, T. R. WATKINS, O. B. CAVIN, N. S. KULKARNI: Friction and Wear of Titanium Alloys Sliding against Metal, Polymer, and Ceramic Counterfaces. Wear, **258**, 1348 (2005).
10. K. G. BUDINSKI: Tribological Properties of Titanium Alloys. Wear, **151**, 203 (1991).
11. M. ZITNANSKY, L. CAPLOVIC: Effect of the Thermomechanical Treatment on the Structure of Titanium Alloy Ti6Al4V. J. of Materials Processing Technology, **157–158**, 643 (2004).
12. R. DING, Z. X. GUO, A. WILSON: Microstructural Evolution of a Ti–6Al–4V Alloy during Thermomechanical Processing. Materials Science and Engineering, **A327**, 233 (2002).
13. H. FUJII: Strengthening of $\alpha+\beta$ Titanium Alloys by Thermomechanical Processing. Materials Science and Engineering, **A243**, 103 (1998).
14. CSM Instruments SA, <http://www.csm-instruments.com/>
15. H. CZICHOS, T. SAITO, L. SMITH (Eds): Handbook of Materials Measurement Methods. Springer, Berlin, 2006.
16. C. H. Jr HAGER, J. H. SANDERS, S. SHARMA: Effect of High Temperature on the Characterization of Fretting Wear Regimes at Ti6Al4V Interfaces. Wear, **260**, 493 (2006).
17. W. S. LEE, C. F. LIN: High-temperature Deformation Behaviour of Ti6Al4V Alloy Evaluated by High Strain-rate Compression Tests. J. of Materials Processing Technology, **75**, 127 (1998).

Received 3 August 2010

Revised 2 October 2010