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High temperature fretting behaviour of plasma vapour deposition TiN coatings

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Abstract

Fretting tests (mode I) were performed on TiN coatings at test temperatures of 23–500 °C. The evolution of the coefficient of friction with the number of fretting cycles for tests performed in that temperature range was recorded. Differences in that evolution were analysed based on the frictional energy dissipated in the sliding contact during the fretting tests. That analysis demonstrated the role of dissipated frictional energy and thermal energy in initiating a structural modification of the debris. The structural modification of the debris coincides with the transition from high friction conditions taking place in the presence of amorphous debris to low friction conditions in the presence of nano-crystalline debris. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

Fretting is a wear process induced by the reciprocating movement at small displacement amplitudes of a counter-body, and takes place in many technical systems. The contact region remains to a large extent isolated from the surrounding atmosphere. The debris formed and the surface reactions induced during fretting, play a major role in the fretting wear process.

Nowadays it is generally accepted that tribological contacts need to be analysed based on a tribo-system approach. The energy dissipated by friction is the main input of energy in a tribo-system. Therefore all the energy-consuming processes in sliding contacts, inclusive wear, are directly or indirectly dependent on the dissipated energy. This is the basis for expressing the wear rate as a volumetric wear per unit of frictional energy dissipated during wear tests. In fretting tests, a cumulative dissipated energy is obtained by summing up the energy dissipated during the successive fretting cycles. The advantage of plotting the volumetric wear vs. the cumulative dissipated energy is that both are cumulative phenomena. As a result, fretting tests performed under different sets of test conditions, including the duration of the test, can be compared as long as the main wear mechanism remains unchanged [1].

That approach has been successfully used for the laboratory investigation of wear-resistant TiN coatings widely used on cutting and forming tools [2–7]. The effect of relative humidity, normal load, and counterface material, has been extensively studied by Mohrbacher et al. [4,5]. These studies demonstrated that relative humidity plays an important role on the oxidational wear of TiN in non-lubricated fretting tests. Recently, de Wit et al. [7] studied the mechanism associated with the changes in friction observed during non-lubricated fretting tests performed with TiN-coated steel sliding against corundum balls. An investigation of the material transfer to the corundum counter-body revealed that for fretting tests performed at room temperature, both amorphous and nano-crystalline debris are detected. The appearance of amorphous and/or nano-crystalline debris depends on the contact load and the environmental conditions, and also on the testing time at which these debris are formed. The structure of the debris determines also whether a high or a low coefficient of friction is noticed [7]. The structure of the

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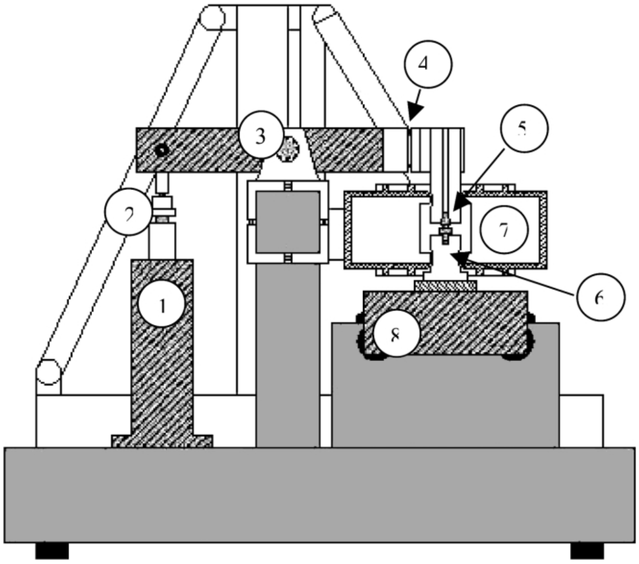

Fig. 1. Schematic diagram of the high temperature fretting test apparatus. 1, Normal-load actuator; 2, load-cell; 3, pivot; 4, quartz force-transducers; 5, upper specimen holder; 6, lower specimen holder; 7, oven; 8, motorised table.

debris evolves in tests performed at room temperature with the dissipated frictional energy, and this dissipated frictional energy is affected by environmental conditions, mainly the relative humidity [7].

The dissipated frictional energy is thus a key parameter determining the fretting behaviour of coatings like plasma vapour deposition (PVD) TiN. As a consequence, the test temperature ought to be an important parameter in fretting. Nevertheless, to our best knowledge, such an effect of temperature on the fretting behaviour of TiN PVD coatings has not yet been reported. In this paper, the fretting behaviour of TiN high-speed steel coatings sliding against corundum at temperatures up to 500 °C is presented and discussed.

2. Experimental

High-speed steel ASP23 flat specimens quenched and tempered at a hardness of 63 HRC were polished to a roughness R_a 0.02 μm and subsequently PVD-coated with TiN. The 4-μm-thick coatings were deposited using a Balzers triode ion-plating equipment (WTCM, Diepenbeek, Belgium). The roughness of the coating was 0.06 μm (R_a). Polished corundum balls with a diameter of 10 mm, a hardness of 2000 HVN, and a surface roughness R_a 0.2 μm, were used as a counter-body. Fretting tests mode I were carried out in a high temperature fretting test rig as outlined in Fig. 1 and described in detail in ref. [8]. The normal load, applied by an electro-magnetic linear actuator (1), was 10 N and remained constant during the tests. A triangular shape wave with a peak-to-peak displacement amplitude of 100 μm, and a frequency of 2 Hz was applied to the lower flat specimens by a linear motorised table guided by air bearings (8). A linear encoder with a resolution of 0.1 μm was used to assure feedback to the linear AC/DC motor. Quartz force transducers (4) were linked directly to the upper stationary ball specimen in order to measure the tangential force. All the tests were done in a background laboratory ambient air of 23 °C and 50% relative humidity. A specially designed oven (7) using $MoSi_2$ heating elements surrounded the fretting contact area. Ceramic specimen holders (5, 6) have been previewed to withstand temperatures up to 1200 °C. In this work, the temperature was varied between 60 °C and 500 °C, controlled by a thermocouple mounted on the TiN-coated specimen. This temperature is referred hereafter as the ‘test temperature’, and is not the real contact temperature at the sliding fretting contact area.

The tangential force and the displacement amplitude were measured on-line during the fretting tests at an acquisition rate of minimum 500 points/cycle. The number of cycles was selected in such a way that wear-through of the coating did not occur during the fretting tests performed in the temperature range mentioned above.

Prior to the fretting tests, the specimen and the counter-body were cleaned with acetone and alcohol. After the fretting tests, the morphology of the wear tracks was observed by scanning electron microscopy (SEM). Wear scars were investigated by laser profilometry (Rodenstock RM600), and the wear volume was obtained by integrating a cross-sectional profile areas as described earlier [9].

The tangential force was measured on-line during the fretting tests. The energy dissipated during the successive fretting loops was calculated based on the instantaneous values of the tangential force and displacement according to a procedure published earlier [5]. This cumulative dissipated energy was calculated for the different fretting tests performed.

3. Results

The evolution of the tangential force during the successive fretting cycles is shown as a fretting log in Fig. 2 for the case of a fretting test done at 100 °C. This fretting log represents hysteresis loops obtained on plotting the tangential force vs. displacement at discrete numbers of fretting cycles. The shape of the hysteresis loops does not change during the fretting tests and the loops are almost parallelograms, characteristic for fretting tests performed under gross slip. Starting as small loops, the width of these loops increases progressively up to a maximum value, and then decreases suddenly at approximately 1000 cycles for the case shown in Fig. 2. Hysteresis loops corresponding to cycle 1000 are

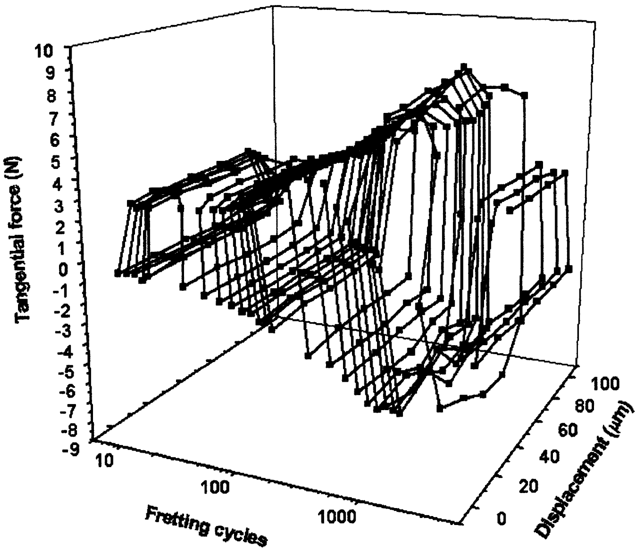

Fig. 2. Fretting log for the case of a fretting test done in ambient air at a temperature of 100 °C. The normal load was 10 N and the displacement amplitude was 100 μm.

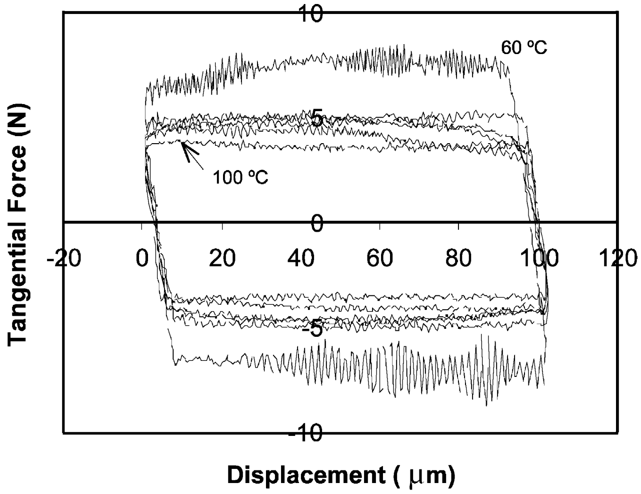

Fig. 3. Hysteresis loops corresponding to cycle 1000 for fretting tests performed at test temperatures varying between 60 °C and 500 °C (normal load 10 N, displacement amplitude 100 μm).

shown in Fig. 3 for fretting tests performed at test temperatures between 60 °C and 500 °C. The coefficient of friction was derived from these hysteresis loops as the ratio between the mean value of the tangential force and the normal load applied. Since the variation of the tangential force is small in the tests performed during this study, the mean value of the coefficient of friction is used. That mean value was calculated from the hysteresis loop area since all tests were performed under gross slip conditions, and since the displacement controller is very reliable. The evolution of the mean coefficient of friction during the fretting tests is shown in Fig. 4 for tests performed at temperatures between 60 °C and 500 °C. The use of a logarithmic scale for the number of cycles allows a better observation of the variation of the coefficient of friction during the running-in phase. The evolution of the coefficient of friction with the number of cycles agrees well with previous work [4], and is characterised by five periods. The first one is a running-in period with a low and constant coefficient of friction, followed by a second period where a rise of the coefficient of friction takes place. During the third period, the coefficient of friction reaches high values and remains more or less constant. Finally, a rapid drop of the coefficient of friction takes place and a steady state period is established till wear-through occurs. Besides this general evolution appearing in all tests performed, some major differences between the tests were noticed. So, e.g. the coefficient of friction increases in the running-in period with increasing test temperature. The maximum value of the coefficient of friction in the third period is 0.7 for fretting tests done at 60 °C, while a value near 0.5 is noticed at all the other test temperatures. Finally, the number of fretting cycles at which the fourth period starts, decreases with increasing test temperature. Only the fretting test done at 500 °C is an exception to this trend.

The fretting tests result in elliptical wear scars on TiN coated HSS steel samples. Fig. 5a,b shows such wear scars measured on TiN by contactless laser profilometry after fretting tests performed under gross slip conditions against a corundum ball for 5000 cycles at a test temperature of 60 °C. In the rather exceptional case that wear-through did occur within the number of fretting cycles selected (Fig. 5b), a large increase of the wear depth is noticed as pointed by an arrow in Fig. 5b. The wear loss was calculated from topographical analyses such as the ones shown in Fig. 5. The wear loss on TiN

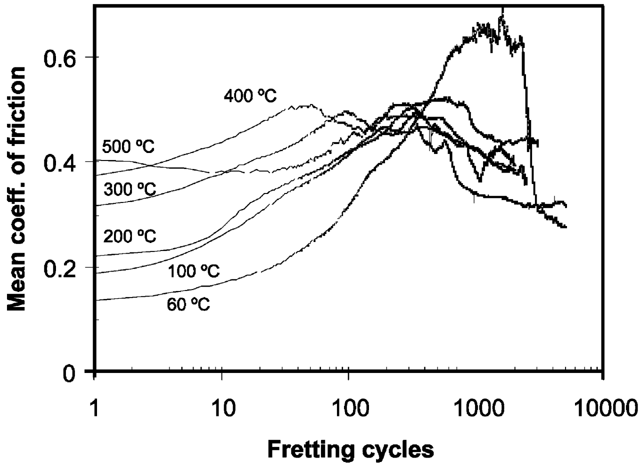

Fig. 4. Evolution of the mean coefficient of friction during the tests performed at temperatures in the range of 60 °C to 500 °C (normal load 10 N, displacement amplitude 100 μm).

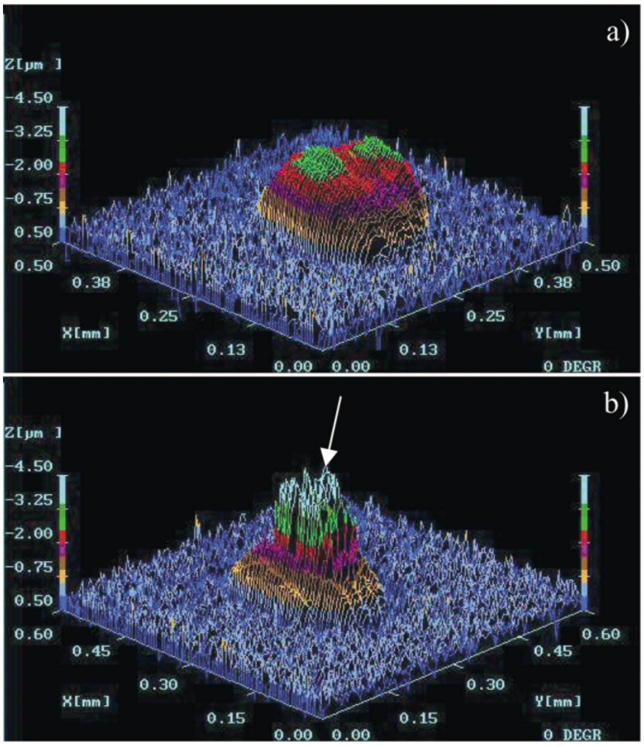

Fig. 5. Three-dimensional topography profile representation of the TiN fretting scar. Fretting test done at 100 °C for 5000 cycles at a normal load of 10 N and a lateral displacement of 100 µm. (a) Profilometry in the case wear-through did not take place. (b) Profilometery in the exceptional case of wear-through.

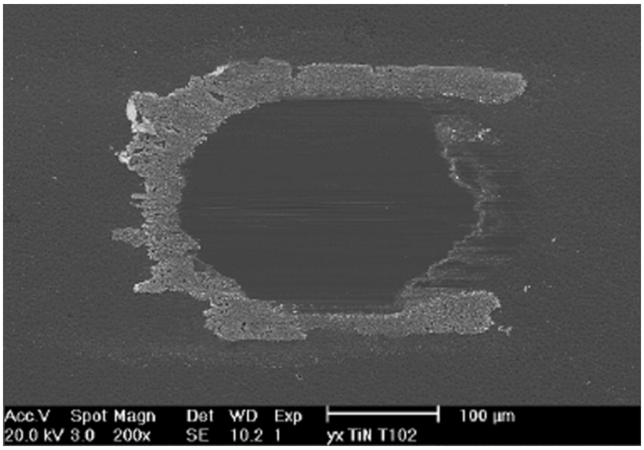

Fig. 7. Morphology of TiN fretting scar observed in SEM. Fretting test done at 100 °C for 5000 cycles at a normal load of 10 N and a displacement amplitude of 100 µm.

coatings tested at different test temperatures is plotted in Fig. 6 against the cumulative dissipated energy. In Fig. 6, data published by Mohrbacher et al. [5] on a similar material combination but tested at 23 °C, are represented as a dotted line. Mohrbacher found a linear relationship between the volumetric wear loss and the cumulative dissipated energy for fretting tests performed under gross slip conditions in ambient air of 23 °C, and at different relative humidity, normal loads, and frequencies. The test done presently at 60 °C fits well with the data of Mohrbacher, notwithstanding the fact that a completely different fretting test equipment was used with a different rigidity and frame structure. Even more

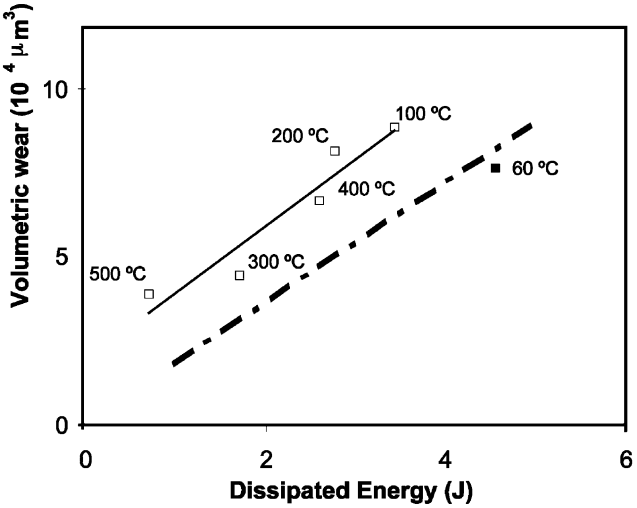

Fig. 6. Wear volume of TiN coatings as a function of the cumulative dissipated energy. Dashed line represents the results obtained at 23 °C by Mohrbacher et al. [6].

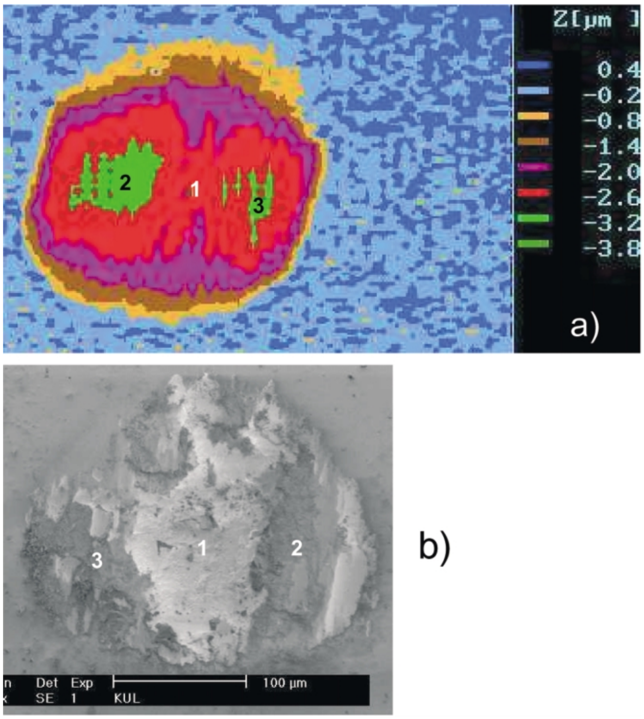

Fig. 8. Wear scars of test temperature of 100 °C. (a) Three-dimensional profile of a wear scar on the flat TiN surface, (b) SEM of the corresponding wear track on the corundum ball.

interesting is to note that for the experimental data obtained at test temperatures between 100 and 500 °C in ambient air, a linear dependence is also noticed between dissipated energy and volumetric wear loss. The best linear fit for these high temperature fretting tests, has a slope comparable to the one reported by Mohrbacher, but the line is shifted so that a slightly higher wear loss is taking place at a given amount of dissipated energy.

Typical fretting wear scars morphologies are shown in Figs. 7 and 8. Fig. 7 shows the wear scar on the TiN coated surface after a fretting test done at 100 °C for 5000 cycles. Fig. 8a shows the profilometry of that wear scar on the TiN-coated surface, and Fig. 8b the morphology of the corresponding wear scar on the corundum counter-body. The morphology of the wear scar on the TiN coated surface is characterised by a central contact area exhibiting fine grooves and showing traces of some polishing action by the debris, as well as accumulated amounts of debris located at the periphery of the contact zone. These debris are quite small. On the corundum counter-body, a transfer layer adheres well in the contact area. EDX-analyses revealed the presence of Ti and O in this transferred material. On comparing Fig. 8a,b, it appears that the transfer layer sticking to defined areas of the ball leads to a reduction of the wear of the corresponding TiN counterparts. In fact, the central part of the ball contact area, referred as 1 in Fig. 8b, has a corresponding area on the counter-face (Fig. 8a) exhibiting a low wear compared with the surrounding regions 2 and 3 without any evidence of transferred material on the ball scar.

4. Discussion

The shift in the fretting wear data obtained in at temperatures of 100 °C up to 400 °C in comparison with the fretting data obtained at 23 °C by Mohrbacher et al. (see Fig. 6), indicates that the wear degradation process is influenced by the test temperature in that range. The linear relationship between the volumetric wear and dissipated energy for fretting tests performed either at low (range of 23 °C up to 60 °C) or at high temperature (range of 100 °C up to 400 °C), indicates that the test temperature does not basically affect the wear mechanism. That lateral displacement of the curve indicates that the achievement of a given volumetric wear loss requires lesser dissipated frictional energy in fretting tests done at high temperature than in fretting tests done at low temperature. Having a closer look on the evolution of the coefficient of friction during fretting tests, we notice that the coefficient of friction remains low at the start of the fretting tests performed either at low or high temperature. The general evolution of the coefficient of friction with the number of fretting cycles is similar for all fretting tests done. The coefficient of friction progressively increases until a maximum value is reached, followed by a more steep decrease. However, two major differences are noticed between tests performed at different test temperatures (see Fig. 9). The first difference is that the coefficient of friction at the start of the fretting tests increases with increasing test temperature. Secondly, the number of fretting cycles necessary to achieve the drop in the coefficient of friction once the maximum value is reached (see points B), decreases with increasing test temperature.

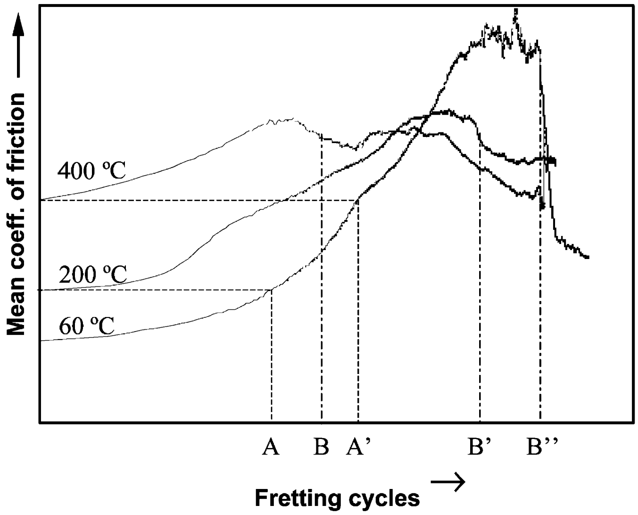

Fig. 9. Outline of the friction evolution to identify the points used in the energetic analysis. The three curves shown are taken from Fig. 4 and correspond to fretting tests done at 60, 200 and 400 °C, respectively.

To get a better insight into these phenomena, two typical amounts of frictional energy dissipated in the fretting contacts were calculated. The first one is used to explain the increase of the coefficient of friction at the start of the fretting tests with test temperature. Hereto the fretting test performed at 60 °C was taken as a reference. The frictional energy dissipated in that fretting test was calculated up to the time that a coefficient of friction is reached corresponding to the coefficient of friction at the start of a fretting test performed at a higher temperature. It was obtained by integrating the fretting hysteresis loops in the reference test up to the cycle corresponding to point A in Fig. 9. That energy can be considered as the equivalent energy in the reference test required to create in the contact area, a material surface condition similar to the one on the TiN sample at the start of the fretting test at the corresponding higher test temperature. That equivalent energy is plotted in Fig. 10 as a function of the temperature difference with the reference fretting test at 60 °C. A linear relationship is found for fretting tests performed in the range of 200–500 °C. This indicates that the

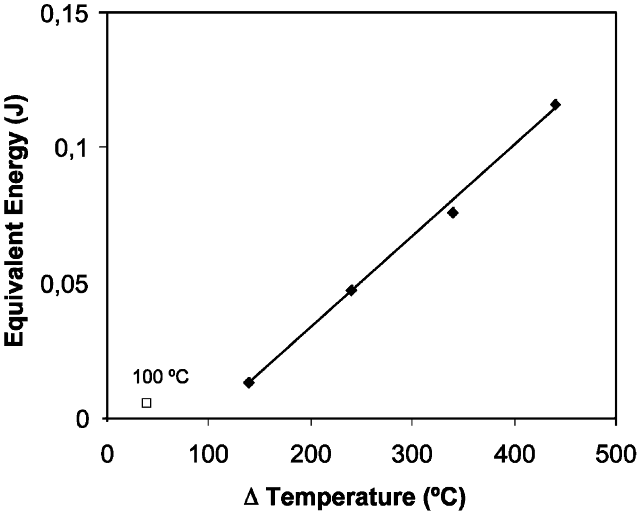

Fig. 10. Effect of the test temperature on the equivalent energy necessary to reach the starting value of coefficient of friction at 60 °C (see point A in Fig. 9) for fretting tests performed at 10 N and 100 μm displacement amplitude.

surface modification induced on TiN during a fretting test, is equivalent to a thermal oxidation requiring a threshold contact temperature of approximately 160 °C (ΔT of 100 °C above the temperature of the reference test).

The second one is the frictional energy dissipated in the fretting contact up to the time that the drop in the coefficient of friction takes place (see points B in Fig. 9). This dissipated frictional energy was calculated for fretting tests performed at test temperatures between 60 and 400 °C (Fig. 11). That dissipated frictional energy induces the transformation of amorphous debris formed in the sliding contact during the first three periods of the fretting tests (see Fig. 4), into nano-crystalline ones. Such a transformation has also be reported by de Wit et al. [7]. That dissipated frictional energy decreases linearly with increasing temperature (Fig. 11). The input of thermal energy takes thus up a part of the energy required for initiating the crystallisation of the amorphous titania debris. It has to be noticed that the results obtained at a test temperature of 500 °C, do not fit with the data obtained at lower test temperatures. That can be explained by looking on previous work on TiN [7,10] that showed that the evolution of the coefficient of friction depends on the formation of a transfer layer on the surface of the counter-body. The formation of such a transfer layer consisting of titanium and oxygen, was described by Singer [11] as originating from debris resulting from the degradation of the TiN coating. The achievement of a constant coefficient of friction depends then on the evolution in the structure of that transfer layer. Both de Wit et al. [7] and Hsu et al. [10] found out that the heating up of TiN in an oven at 500 °C, induces the crystallisation of TiN into Ti-O. In view of these facts, the results of the fretting test shown in Figs. 4 and 11 can be explained as follows:

- during fretting tests done at 60 °C, the crystallisation of the debris is induced by the frictional energy dissipated in the sliding contacts. Initiation of crystallisation requires a large number of fretting cycles, and the coefficient of friction remains high during a large number of fretting cycles;
- during fretting tests performed between 100 °C and 400 °C, the dissipated frictional energy required to induce crystallisation decreases since thermal oxidation becomes more active, and the coefficient of friction lowers after a smaller number of fretting cycles;
- for fretting tests performed at 500 °C and higher, the frictional behaviour is different right from the start of the fretting tests. This is linked to a spontaneous thermal oxidation of TiN on exposure to ambient air at these temperatures as reported previously [7,10], so that the crystallisation of the debris does not require any additional frictional energy.

From these experimental facts, it may be concluded that the test temperature during fretting tests acts as an additional input of energy contributing to the crystallisation of the debris. At increasing test temperature, the frictional energy required to induce crystallisation lowers. This conclusion agrees well with the oxidation mechanism put forward by Mohrbacher et al. [5] to explain the fretting wear of TiN coatings. The test temperature thus appears as an additional input of energy for crystallisation of the debris, proportional to the test temperature increase.

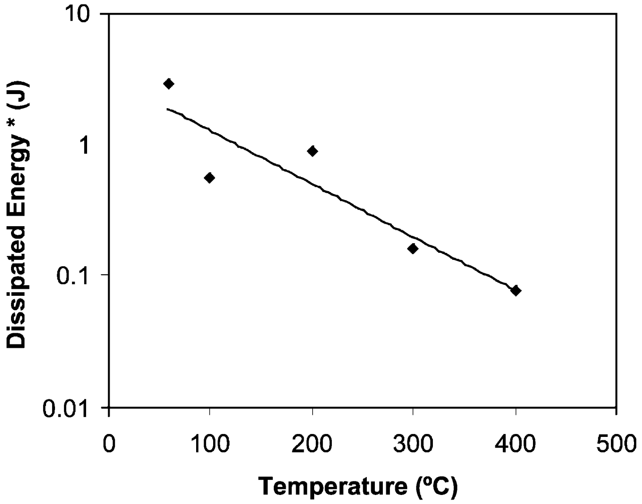

Fig. 11. Cumulative dissipated energy up to the start of the drop of the coefficient of friction, (see points B in Fig. 9) for fretting tests performed at 10 N and 100 μm displacement amplitude.

5. Conclusions

The effect of temperature on the fretting behaviour of PVD TiN coatings sliding against corundum was investigated up to 500 °C. The experimental results could be explained based on a structural transformation from amorphous to nano-crystalline debris originating from the TiN coating that takes place at a certain moment during fretting tests. This transformation of debris governs the evolution from a high to a low coefficient of friction, and determines the extent of the fretting wear on TiN. The energy needed to activate that structural transformation of the debris, is shown to originate not only from the frictional energy dissipated in the sliding contacts during the fretting tests, but also from the external heat input when fretting tests are performed at temperatures above room temperature. At increasing test temperature in the range of 60 °C up to 400 °C, the external heat reduces accordingly the frictional energy needed to get low friction nano-crystalline debris on TiN. For fretting tests performed at temperatures above 500 °C, the oxidation of TiN on exposure to the ambient air is sufficient to create an oxide layer on top of TiN.

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