

Tribological performance of Ti_3SiC_2 containing 8% of TiC against the corundum

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1. Introduction

Ti_3SiC_2 has curious properties, intermediate between those of metals and ceramics, an unusual combination as many literatures showed [1-9]. Some of its key properties include excellent resistance to oxidation up to 1400°C , high thermal shock, resistance, high Young's modulus (325 GPa), relatively low hardness (4-5 GPa), high fracture toughness ($7-9 \text{ MPa}\sqrt{\text{m}}$) and good machinability with conventional tools [2, 10], it also has a good tribological property [11-15]. The bulk Ti_3SiC_2 could be appropriately applicable material for some tribological and wear-resistant applications. However the available studies, at the present, on Ti_3SiC_2 forerunner by Barsoum and his team [2-4,10,24], explain certain behaviours and predetermine experimental values. El-Raghy et al. [2] investigated the effects of grain size on the friction and wear for a high-purity Ti_3SiC_2 sample sliding against a 440°C stainless steel as counterpart in pin-on-disk type tester. The friction coefficient was as high as about 0.83, and the wear rates was in the range of $(4.25-1.34)\times 10^{-3} \text{ mm}^3/\text{Nm}$. Respectively, using a AISI 52100 steel pin as the counterpart in pin-on-disk tester, Sun and Zhou [16] measured the friction coefficient and wear rate of a Ti_3SiC_2 sample contained 7.0 wt.% of TiC impurities, the results showed that the steady friction coefficient is 0.4-0.5 and the wear rate is $9.9\times 10^{-5} \text{ mm}^3/\text{Nm}$. More recently, Zhai et al. investigated the high pure and TiC contain bulk Ti_3SiC_2 [17]. The highly pure sample, sliding dryly against low carbon steel disk, exhibits a changed friction coefficient in range of (0.09-0.53), and wear rate of $(0.6-2.5)\times 10^{-6} \text{ mm}^3/\text{Nm}$ for different sliding speeds of 5-60 m/s and normal pressures (0.1-0.8 MPa). However, the TiC- Ti_3SiC_2 exhibits a larger friction coefficient than the high-purity in the same test conditions. Many observations were reported by various authors about the tribological behaviour of Ti_3SiC_2 , and essentially in dry-friction. However many questions remain about different facets of its behaviour, which requests more investigation, preferably, in same condition against a different material for more understanding. From this point of view, in the present paper, the Ti_3SiC_2 contain TiC was carrying out against Alumina in the same test conditions as reported in reference [17], to make it easier to compare the results. It is also an occasion to present the modelling of behaviour by design of experiments method; which will allow us to predict a change of friction coefficient and wear rate, through a mathematical model of studied material during dry friction, in ambient air, and to predict consequently the optimal parameters of work.

2. Experimental procedure

The Ti_3SiC_2 sample was prepared by thermo-pressure method, which had been clearly described elsewhere [12]. The material was obtained from mixture elementary powders of silicon, titanium, and carbon, according to a molar ratio of Ti: 3; Si: 1; C: 2; and other elements, as reactive accelerators, in little quantity (< 0.1). The presence of TiC impurities in the sample was estimated to be 8% in volume. The surface measurement of TiC was done from micrographs obtained by using "Scion-Image" software, and identified by an ultraviolet lamp which could clearly show this element. The average of the percentages is calculated after several surface measurements of different sections from the same sample. Micrographs in Fig. 1 represent two microstructure images of Ti_3SiC_2 . Fig. 1, a is a micrograph observed using a scanning electron microscopy (SEM) exhibiting the highly pure of Ti_3SiC_2 sample. The Ti_3SiC_2 grains were relatively uniform in dimension with a plate-like shape. The average grain size was estimated to be about $20 \mu\text{m}$ and $25 \mu\text{m}$ in the elongated direction. Fig. 1, b is a typical SEM micrograph exhibiting the microstructures of the Ti_3SiC_2 sample which consists of dominant Ti_3SiC_2 grains and TiC grains detected by XRD analysis. The Ti_3SiC_2 grains were also plate-like, while the TiC grains exhibited an equiaxed shape. The average size was estimated to be about $25 \mu\text{m}$ for the Ti_3SiC_2 grains, and about $4-6 \mu\text{m}$ for the TiC grains [17]. The Ti_3SiC_2 -TiC is obtained by the addition of an infinitesimal quantity of trioxide of Beryllium to the powder. The study of the tribological behaviour of Ti_3SiC_2 with 8% TiC impurities was evaluated by exposing the samples, in circular block form, to the friction and wear tests on Pin-on-Disk (PoD) tribometer, with an interchangeable tray that depends on the size of the sample, and this last evidently depends on selected sliding speed (Fig. 2). The tests were investigated by using ball (about 6 mm on diameter) of corundum material, chosen for its resilient nature (high fracture toughness), sliding dryly on disk under a range of pressure 0.1-0.8 MPa at an ambient temperature of 20°C and a relative humidity of 20%. Both information about pressure and humidity appear on the display of tribometer. The sliding speeds from 5 to 60 m/s are obtained by placing the ball at a certain distance from the rotation axis of the sample combined with rotation speed. For example, to obtain $V = 60 \text{ m/s}$, one uses disc of diameter equal at 0.250 m with rotation speed of 4583.8 rpm obtained with a rheostat. The sliding distance traversed was chosen about 10000 m; a so long distance is necessary to study and extract maximum of informations

about behaviour of Ti_3SiC_2 , during a continuous cycle of friction. For every given test conditions, the friction coefficient and accurately mass loss of Ti_3SiC_2 block, were measured in one continuous friction process, and associated to the modelling process by the method quoted before in order to understand its behaviour during friction as well as the mechanisms which explain that.

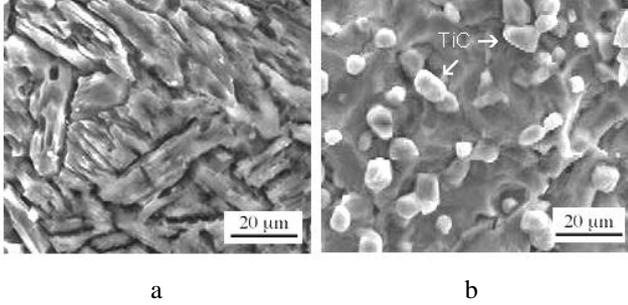


Fig. 1 Typical SEM micrographs showing microstructures of: a - the highly pure Ti_3SiC_2 and; b - Ti_3SiC_2 with TiC impurity



Fig. 2 Pin-on-Disk (PoD) tribometer from CSM instruments

3. Equation and modelling

In order to investigate the influence of the parameters action on the tribological behaviour of the Ti_3SiC_2 , the modelling by the design of experiments method seems adequate. It makes possible to predict other responses of friction coefficient and wear rate, basing of-course on test results already performed, in the experimental field. This method has the advantages to investigate the influence of each parameter separately as well as the influence of their interaction on the result. So it brings a solution that makes possible to minimize the number of experiments to carry out, and thus, saving time and money without sacrificing the precision of the results [19]. In the present study, we use the no-conventional design of experiments which makes possible to use data at our disposal without envisaging beforehand the number of experiments to carry out and without defining the parameters values, or without following a plan defined in advance [20]. The interest of response (friction coefficient & wear rate) modelling, by a polynomial, is to be able to calculate all the responses of study field without any required to additional experiments.

As the response y_i depends on two factors (sliding speed & pressure), which each of them has several levels, so it is judicious to express the estimated responses in matrix form as the following notation

$$[Y_{calculated}] = [X][a_i] \quad (1)$$

3.1. Model coefficients

Modelling is done by a polynomial of a second degree (1), which derives from the development of function in Taylor series, in which, each effect of parameter is represented by variable x_i attached to a coefficient a_i . Other coefficients a_{ij} are related to the interaction of effects x_i and x_j [21]. The general form of the polynomial model is written in the following form

$$y_i = a_0 + \sum_{i=1}^k a_i x_i + \sum_{i=1}^k a_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^k a_{ij} x_i x_j \quad (2)$$

The developed form of polynomial model is written as below

$$y_i = a_0 + a_1 x_{i,1} + a_2 x_{i,2} + a_{12} x_{i,1} x_{i,2} + a_{11} x_{i,1}^2 + a_{22} x_{i,2}^2 + e_i \quad (3)$$

where $x_{i,1}$ is sliding speed factor; $x_{i,2}$ is pressure factor; y_i is response factor and e_i is gap between measured and predicted values obtained from the model of friction coefficient for any cycle i of dry-friction experience.

This modelling kind has advantages of: noting, through the coefficients values a_i , the predominance of each parameter compared to another; solving easily the i expressions by a matrix calculation using adapted software, and to have the possibility of analysing the interactions between the effects of parameters. The treatment of design of experiments consists on estimating, by the method of least squares, the P coefficients of a mathematical model while their number is lower than that of experiments performed [21]. By using "Matlab" for treatment of Eq. (2), we obtain the coefficients values by the following equation

$$a = ({}^tXX)^{-1} {}^tX y \quad (4)$$

where a is coefficient matrix; $({}^tXX)^{-1}$ is inverse of matrix (XX) ; tX is transpose of matrix X and y is experimental measured response.

3.2. Mathematical modelling

After estimating coefficients of models (friction; wear), we can now establish the mathematical models that linked response to factors. The models take the following forms

$$y_{Friction} = 0.213315 - 0.13918 x_1 + 0.0348933 x_2 + 0.140659 x_1^2 - 0.0226041 x_2^2 + 0.01782 x_1 x_2 \quad (5)$$

$$y_{Wear} = 1.9895 + 0.7616 x_1 + 0.3229 x_2 - 0.4421 x_1^2 + 0.0421 x_2^2 + 0.5765 x_1 x_2 \quad (6)$$

where $y_{Friction}$ is a predicted value of friction; y_{Wear} is predicted values of wear; x_1 is sliding speed value; and x_2 is applied pressure value.

Through the estimating vales of models coeffi-

cients and as a preliminary analysis, we can already indicate that the pressure acts slightly compared to sliding speed parameter in both friction and wear response.

3.3. Model quality

The Eq. (5) drifting from a statistical method is only an approximation of reality, from where appear the differences between the experimental and the prediction values. The estimate of coefficients of the second-degree polynomial model is based on test results, which are particular values with a random variable, for each treatment of design of experiments. So we have to make a judgment, by the quality of model coefficients, on the obtained results. We distinguish two types of qualities which are as follows.

3.3.1. Model validity

It makes possible to know the degree of rapprochement of the predicted values compared to the measured values. This coefficient translates the contribution of the model in the restitution of the measured response variation. It can be calculated by the relation (6).

$$R^2 = 1 - \frac{SSE}{SST} \quad (7)$$

where SSE is sum of squares of errors and SST is sum of squares total [22, 23].

In our case, the model validity coefficient is 0.950 and 0.830 for friction coefficient and wear rate model, it is estimated very good since it checks the following relation

$$0 \leq R^2 \leq 1. \quad (8)$$

3.3.2. Model reproducibility

It makes possible to judge the ability of the polynomial model to predict the response without carrying out other tests than those already done. Its theoretical value can vary between minus infinity ($-\infty$) and (+1), but the model is considered reproducible [23], if the value of Q^2 is close to 1.

$$Q^2 = 1 - \frac{PRESS}{TSS} \quad (9)$$

$$-\infty < Q^2 \leq 1 \quad (10)$$

where $PRESS$ is prediction residual error sum of squares and TSS is total sum of squares.

The models reproducibility obtained by applying Eq. (9), of which Q^2 is 0.916 and 0.694 for friction coefficient and wear rate successively, are even good since both check Eq. (10).

The results mentioned before show that the quality of mathematical models obtained is satisfactory. So it means that the new responses values, obtained from models, will be close to experimental values.

4. Graphical analysis of the results

In this part, we will expose a series of results basing of course on the theory of design of experiments. The

curves below interpret the tribological behaviour of Ti_3SiC_2 , under the effect of the external parameters, during dry friction; they are traced in "Excel" and "Matlab".

4.1. Friction behaviour

Fig. 3 shows an exposition of real values of friction coefficient as a function of the applied pressure with different values of sliding speed: low, average and high. The linear values of sliding speed of: 5, 20, 40 and 60 m/s has been given by taking the significant values of the responses amongst others of many experimental responses, we have also taken into account the minimizing of the number of experiments to be carried out. Two types of curves are presented, the evolution of friction coefficient increases appreciably in most of them, except for 20 m/s which is rough, and continuous increasing for 5 and 60 m/s sliding speeds, however, it decreases considerably in the rest, notably on the 20 and 40 m/s curves. On the other hand, the friction coefficient exhibited from the Ti_3SiC_2 , appears less sensitive to the change induced by the pressure applied compared to that of the sliding speed. The variation of change is only about 0.1 in pressure-dependent but it exceeds 0.3 when the sliding speed increased from 5 to 60 m/s for a given pressure. A similar tendency variation of friction coefficient is exhibited, in reference [17], in which the highly pure Ti_3SiC_2 sliding dryly against the low carbon steel.

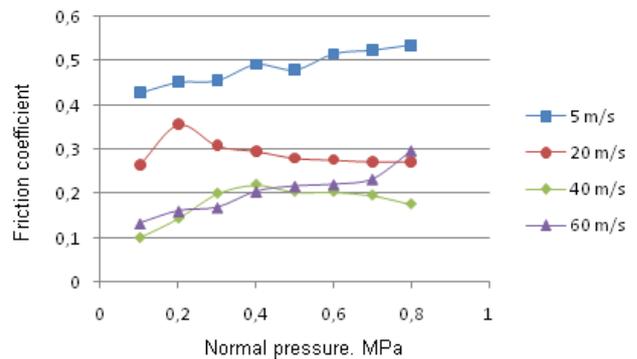


Fig. 3 Friction coefficients of Ti_3SiC_2 as a function of sliding speeds under the normal pressure

Among the advantages of design of experiments method is that the interaction between parameters is better investigated through the different graphical representations. Figs. 4, 5 show typical curves of simultaneous effect of the two parameters (sliding speed and pressure applied) on the friction coefficient variation, which are usually called Response-surface (Fig. 4) in 3D, and Iso-response contours (Fig. 5) in 2D, these last are only projection of response surfaces on the down plane. In addition to the experimental values on the graph (Fig. 3), there are, on the response surfaces and the lines of Iso-response plot, others values of friction coefficient calculated before by the mathematical model Eq. (5). Therefore, the response-surface and Iso-response exhibit descriptive and predictive values.

Through this we clearly see the evolution of the friction coefficient according to the parameters. At the initial stage of a continuous friction process, we note that for any selected sliding speed up to 23 m/s, the friction coefficient f remains almost constant and changes very

little with the increase in pressure between 0.1 MPa and 0.8 MPa; the contours of Iso-response remain nearly vertical on the graph for this case. It confirms once again that the pressure acts very little on the change of friction coefficient. On the other hand, we note, beyond a quoted speed, that the interaction speed-pressure is very visible since the contours are inclined and curved. As an example, by analysing the graph (Fig. 5) from bottom to top, for a speed of 40 m/s with increasing pressure, we report a different values of friction coefficient from 0.112 to 0.203 which means that the coefficient changed a several time compared to the first case. Generally, the graphs show, that the coefficient of friction decrease with increase of sliding speed on a large part of experimental field. However, there is an increase of the coefficient for a pressure of 0.54 MPa up to 0.8 MPa with high sliding speeds (57-60 m/s); that shall prove curious properties of the studied material.

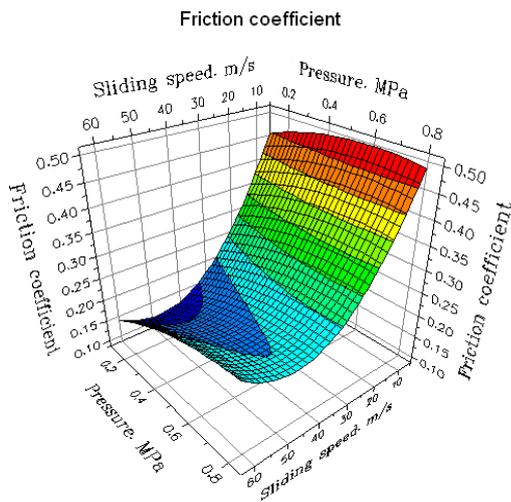


Fig. 4 Response-surface plot showing the effect of sliding speeds and normal pressure on friction coefficient

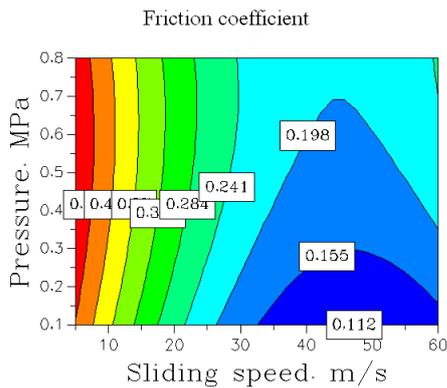


Fig. 5 Iso-response plot of friction coefficient as a function of sliding speeds under normal pressure

4.2. Wear behaviour

The Ti_3SiC_2 wear rates were investigated by weighting the mass loss for every given test conditions, then the data are reported on the graph (Fig. 6) as a function of sliding speed under normal pressure from 0.1 to 0.8 MPa. The way curves behave, appears almost as in the precedent ones, showing that the wear rate and friction coefficient have a similar dependence to the pressure, but it is different for the sliding speed; in fact, an increase on the

speed results in decrease of friction coefficient however, the wear rate increases. It is thus noticeable that the wear rate is less sensitive to the pressure compared to the sliding speed which is much depending. The wear rate varies in range of $(0.6-2.5) \times 10^{-6} \text{ mm}^3/\text{Nm}$ under normal pressure from 0.1 to 0.8 MPa with different sliding speed levels, except for a high speed of 60 m/s, which increases to $3.8 \times 10^{-6} \text{ mm}^3/\text{Nm}$.

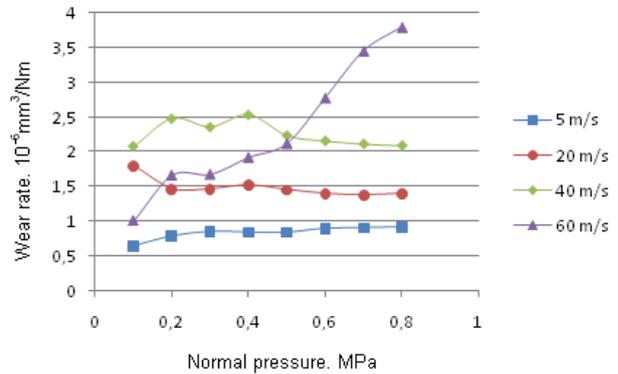


Fig. 6 Wear rates of Ti_3SiC_2 as a function of sliding speeds under the normal pressure

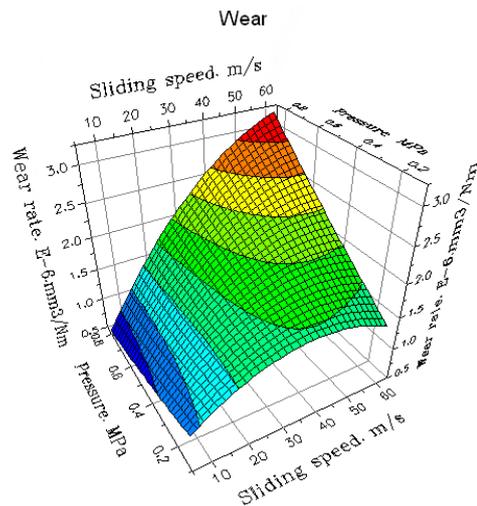


Fig. 7 Response-surface plot showing the effect of sliding speeds and normal pressure on wear rate

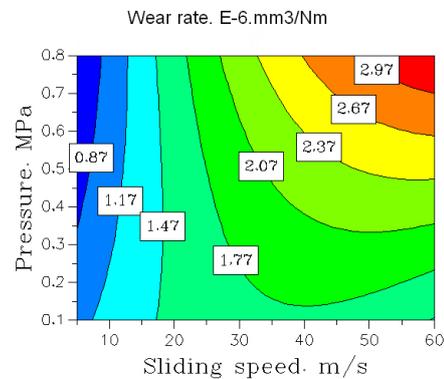


Fig. 8 Iso-response plot of wear rate as a function of sliding speeds under normal pressure

A simultaneous effect of both parameters discussed before, are illustrate on a typical representation of model equation in Response-surface (Fig. 7) and Iso-

responses contours (Fig. 8).

It should be remembered that the representations are traced by using experimental and prediction values of wear rate; those last are calculated before by the mathematical model Eq. (6). Therefore, the response-surface and Iso-response exhibit descriptive and predictive values. It constitute an interesting strong point of designs of experiments method, we can clearly see the evolution of wear rate depending of sliding speed and pressure. This evolution was animated by coloured areas; it converges towards the red zone with the increase in wear rate, and towards the blue zone with the decrease. Through this we remark, in some zone corresponding to the low sliding speed, that the wear rate exhibits a very slight change, the increase not exceed 0.3 when the sliding speed increased from 5 to 20 m/s under a given normal pressure from 0.1 to 0.8 MPa; which means that the pressure acts very little on the change of wear rate with low sliding speed. However, the variation of wear rate is remarkable, it exceeded 0.6 with increasing in sliding speed and it is very important with increase of interaction speed-pressure for a high sliding speed with which we record high values of wear rate (up to $3.8 \times 10^{-6} \text{ mm}^3/\text{Nm}$). Of what confirms once again that the sliding speed remain the dominating factor, and as interaction has a big affect on the increase of wear rate.

5. Discussion

The results discussed on the present study shows that the friction coefficient and even the wear rate depend much, on first order, on sliding speed then on interaction speed-pressure. The Ti_3SiC_2 with 8% of TiC impurities exhibits a higher values of friction coefficient and relatively low values of wear rate for the low sliding speed, it is only in this case, in which they vary inversely in such a way that when the pressure increases, the friction coefficient increases and the wear rate decreases. However, they vary simultaneously in the rest of the results for average and high sliding speed under given normal pressure. A similar tendency variation of tribological behaviour of the studied material against corundum was exhibited, in reference [17], in which the highly pure Ti_3SiC_2 sliding dryly against the low carbon steel. The results reported from this last, were almost the same even the difference in sample and counterpart proportions. In fact, the impurities composition in the samples and the nature of counterpart materials, in the studied case, played the role of compensating factors, that is, the Ti_3SiC_2 contains 8% of TiC impurities, and as demonstrated elsewhere [12, 16], the hard TiC grain would increase the friction coefficient and the wear rate, however the corundum exhibits a lowly sliding resistance; it is noticeable that the rest of the test conditions were similar. On the other hand, the changing magnitude was larger in the second test with the same composition of sample of Ti_3SiC_2 in [17], which it is, in fact, related essentially to the counterpart chosen, since it is the only difference between conditions tests. It is worth to discuss the related factors resulting such response, to understand the dominant mechanism. The tribological behaviour, as discussed in many researches [1-18], is closely related to the mechanical, thermal and physicochemical factors. In our case, the corundum, because of its excellent mechanical properties, supposed that it would minimise the adverse effects as: the material transfer, frictional heat and chemi-

cal reaction; due to its high hardness, high temperature resistance and chemical inertness and as a biomaterial which it is used for special applications. However, a certain number of primarily physicochemical mechanisms enable the appearance of chemical composition on the surface of the material; they also act on the friction coefficient. By then, those compounds form films which are intercalated between the counterpart and the material, playing a role of a 3rd body. The tribological behaviour of this material becomes ambiguous at a given moment without understanding precisely how. However an explanation can be given for the various stages of variation of friction. Positively, on the first stage, the highly pure Ti_3SiC_2 exhibited a high friction coefficient and a quite low wear rate when it dryly slides against the corundum particularly under a higher sliding speed. Beside, the coefficient of friction is in perpetual reduction between 10 m/s and 40 m/s under every normal pressure, essentially, when two surfaces slide together, most of the work done by friction is turned into heat; consequently, the increase in heat, during friction, can modify the mechanical properties and metallurgical of sliding surfaces, thus providing a self-antifricion mechanism that results in reducing of the friction coefficient. Additionally, the heat favours the oxide formation which covers places on the sample's surface. This layer insulates partially and gradually the Ti_3SiC_2 from the corundum, therefore the coefficient decreases. Some literatures [24, 25] have shown that Ti_3SiC_2 can be oxidised at a certain temperature in air during a contact. An oxide film consisting of SiO_2 and TiO_2 is formed on the Ti_3SiC_2 surface; it is dense and adhesive which attributed in the decrease of friction coefficient [26, 27]. On the other hand, the remarkable increase of the friction coefficient for the high sliding speed under the high pressure beyond 0.4 MPa, whereas it decreases before this value, is probably caused by wrenching and sweeping of the oxides film which is rejected out, from the contact surface, by motion, then again the TiC grains reappear on the surface and incorporate in contact with corundum. So that leads to an increase in the friction coefficient which reaches the value of 0.245 under sliding speed of 60 m/s and a pressure of 0.8 MPa. The increase in temperature, during friction, seems also be a promoter factor, the growth rate of oxide film increases exponentially with the temperature [28]. However, it is limited by the diffusion speed of the slow-reacting element [29, 30]. Nevertheless, it was difficult for a film to be maintained on the friction surface at sufficiently high temperature, this is due to the change of its specific viscosity and the forces applied, in cause of high sliding speed that supports the wrenching. It is conceivable that a good oxide film with a higher percentage of coverage results in a smaller coefficient of friction, and a poor oxide film with a lower percentage of coverage results in a larger friction coefficient.

Although the influence of film and the temperature are visible, the presence of the TiC impurities seems to be a dominant factor. The comparison between the studies exposed in the quoted references (for Ti_3SiC_2 pure) and our results (for Ti_3SiC_2 with 8% of TiC impurities in hexagonal form plates formed by a reaction between Ti and C), confirms this assumption. These plates, which seem to be harder than the mother-material, increase the roughness of the friction surface; that causes a change of the tribological behaviour of the studied material, in other words a higher

friction coefficient.

6. Conclusions

1. The Ti_3SiC_2 with 8% of TiC impurities, sliding dryly against corundum, exhibits variant friction coefficient and wear rate of (0.53-0.1) and $(0.6-2.5) \times 10^{-6}$ mm³/Nm respectively, for a change of sliding speed of 5-60 m/s under normal pressure from 0.1 MPa to 0.8 MPa.

2. The modelling of tribological behaviour of Ti_2SiC_3 , by the design of experiments method, enabled us to display and illustrate the evaluation results graphically, under different facets; and advantageously, to have a prediction values of the parameters acting externally, without exceeding the experiment field between maximum and minimal values of sliding speed and pressure.

3. In addition to the remarkable dependence of the behaviour of the friction coefficient and the wear rate on sliding speed, pressure applied and their interaction; the hard TiC impurities, which are formed in material, can contribute in the interlocking tribological action on the Ti_3SiC_2 .

4. The knowledge of variation of cited parameters makes that the material in question can be controlled for how long it can perform satisfactorily or on its performance quality.

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Ti_3SiC_2 TURINČIO 8% TiC TRIBOLOGINIAI PRIVALUMAI LYGINANT SU KORUNDU

R e z i u m ė

Ištirta Ti_3SiC_2 , turinčio 8% TiC priemaišų, tribologinė elgsena esant sausam slydimui. Bandymai atlikti su tribometru, naudojant korundą kaip tiriamosios medžiagos, kurios santykinis drėgnumas kambario temperatūroje 20%, publikatą. Rezultatai rodo mažėjantį trinties koeficientą (0.53–0.1) ir didėjantį dilimo intensyvumą $(0.6–2.5) \times 10^{-6} \text{ mm}^3/\text{Nm}$, kai slydimo greitis didėja nuo 5 iki 60 m/s esant normaliam slėgiui 0.1–0.8 MPa. Eksperimento matematinis modelis paaiškina tribologinę Ti_3SiC_2 elgseną kreivėmis ir grafikais, iliustruojančiais įvairius pramonėje pasitaikančius atvejus.

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TRIBOLOGICAL PERFORMANCE OF Ti_3SiC_2 CONTAINING 8% TiC AGAINST THE CORUNDUM

S u m m a r y

The tribological behaviour of Ti_3SiC_2 containing 8% of TiC impurities, in dry-sliding, was experimentally investigated. The tests were performed on a tribometer, using corundum as a counterpart against a studied material, at room temperature with a relative humidity of 20%. The results show a decreasing friction coefficient (0.53-0.1) and an increasing wear rate $(0.6–2.5) \times 10^{-6} \text{ mm}^3/\text{Nm}$, with a sliding speed increasing from 5 to 60 m/s and under a normal pressure in the range of 0.1–0.8 MPa. The mathematical model, obtained through a modelling by design of experiment method, explains the tribological behaviour of Ti_3SiC_2 with curves and graphs that show the different cases of its use in industry.

Keywords: Ti_3SiC_2 , tribological behaviour, dry-sliding, antifriction effect.

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