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Investigation of the tribological behaviour of WC/TiC based cermets in contact with Al₂O₃ alumina under high temperature

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Abstract:

WC/TiC-based cermets are, generally, considered as potential alloys widely used in hot rolling industry because of their interesting properties, namely high resistance to wear and oxidation. This work was aimed at studying the tribological behaviour, at relatively high temperatur, of WC/TiC-based cermets prepared using the powder metallurgy procedure. Three WC/TiC-Co cermets were prepared with different titanium carbide (TiC) additions namely 5%, 10% and 15% [in weight percentage (wt.%)], and a tungsten carbide-cobalt (WC-Co) grade without TiC which was considered as a reference material, resulting in a total of four samples. Friction tests were carried out, at two different contact temperatures of 450°C and 650°C, using a tribometer and an alumina ball during 2 h 46 min with load and speed of 20 N and 0.5 m/s, respectively. The obtained friction coefficients indicate that WC/TiC-based grades are relatively stable compared to the reference grade which shows an unstable friction coefficient with many peaks. It was also found that wear rates decreased with increasing TiC content, but exhibited a noticeable increase with rising temperature. Moreover, and in order to characterise the tribological degradation, the wear tracks microstructure composed of 80% WC, 15% Co and 5% of TiC, were analysed using a scanning electron microscope (SEM) process. Consequently, an enhancement of the wear resistance at 650°C was observed, and oxides of various types rich in tungsten, cobalt and oxygen were identified through SEM/energy electron spectrometery (EDS) images.

Keywords: cermets, friction, thermo-mechanical, WC-Co-TiC, wear

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1 Introduction

Ceramic-metal cermets composed of tungsten carbide-cobalt (WC-Co) powder, commonly manufactured by conventional powder metallurgy technology, are traditionally employed as cutting tools [1], [2]. Owing to their interesting tribological behaviour under high thermo-mechanical stresses, these alloys have recently attracted particular interest and have undergone considerable development in various industrial applications. Generally, these WC/TiC-based cemented carbide constitute a substitute material for conventional WC-Co cemented carbides in different applications requiring several properties involving, for instance, high hardness, excellent wear resistance, low coefficient of friction (COF) at high temperature, oxidation resistance, high thermal and chemical stability [3], [4], [5], [6], [7], [8], [9], [10]. The microstructure of these cermets and their tribological behaviour have been the subject of many works over the past several years. These materials are used in many industrial applications, involving equipment components that are in dry contact at high temperature, such as hot-rolling dies and aerospace equipment [11], [12], [13], [14], [15], [16]. Although WC-Co cermet seems to be a very widely used in cutting tools for high speed machining, hard metals and hard high-alloy steels, it exhibits some performance limitations, such as poor oxidation resistance and corrosion [17], [18]. At high temperatures, alloy decarburisation is often caused by thermal decomposition or oxidation. The decarburisation of tungsten carbide (WC) leads to the appearance of fragile phases altering the mechanical properties of the material [19], [20]. Moreover, under high thermo-mechanical stresses and according to the applied load and contact temperature, the WC-Co-based cermets present several tribological problems, namely oxidation and

Abdelaziz Lakehal is the corresponding author. ©2018 Walter de Gruyter GmbH, Berlin/Boston. surface degradation [21], [22]. However, most of the studies relevant to the wear resistance of the considered material deal with metal cutting applications carried out at relatively low loads and speeds, where the working conditions are far from those of the hot rolling milling and other hot processes [16].

Among the ways that are supposed to improve the hot tribological behaviour of these materials, is the addition of stable carbide such as titanium carbide (TiC) [23], [24], [25], [26], [27]. TiC is well known to be very useful as a ceramic component in cermets because of its high melting point (3065°C), hardness and wear resistance [28], [29], [30], [31], [32]. Several recent studies have reported a noticeable improvement in tribological performance at room temperature following the introduction of TiC into the starting powder mixture (WC-Co) [1], [33], [34]. Also, the cemented carbides (WC-TiC-Co) reveal high hardness at high temperatures [12], [35], [36]. Several authors have clearly reported the enhancement of the wear resistance of WC-Co cermets, by incorporating carbide particles, such as TiC, to form cemented carbide [26], [37], [38]. The present work examines the issue of improving the WC-Co cermet lifetime through TiC addition. Mainly intended for mechanical systems with severe dry sliding contact (hot-rolling dies, hot stamping dies and aerospace equipments, ...), the underlying cermets are tested against an alumina ball at relatively high temperature with different levels of TiC (5%, 10%, and 15%). Furthermore, the heat tribological behaviour (friction, wear rate) of WC-Co-TiC and WC-Co reference cermets is evaluated using pin-on-disc friction tests against a hard ceramic alumina ball (Al₂O₃) at contact temperatures 450°C and 650°C, under a 20 N load, for a travelled distance of 5000 m. The alumina ball is employed in our experiments because it is inert and does not oxidise at high temperatures. Microstructural analysis was also carried out to confirm the degradation character and the wear type of the considered cermets.

2 Materials and experimental procedure

2.1 Preparation of cermets

The cermets we used were produced by a conventional process of powder metallurgy. They are shown in Figure 1. The various compositions in weight percentage (wt.%) are shown in Table 1. The WC, Co and TiC powders are commercial products manufactured by (Norinco Group, China). The average particle size and the density of the powders are, respectively, 1.1 μ m and 4.5 g/cm³ for WC, 2 μ m and <0.75 g/cm³ for Co, 4 μ m and 4 g/cm³ for TiC. After weighing, powders were mixed in a ball mill for 24 h with the addition of methanol (C₂H₅OH). The mixture is dried in an electric furnace (YZM-40, China) for 30 min at a temperature of 70°C.



Figure 1: Grade WC-Co-TiC cermet obtained by the sintering process.

Table 1:	Composition	by weight	of the powder	mixtures of	cermets
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Grades			Composition in Wt.%
	WC%	% CO	% TiC
NA (reference)	85	15	_
NB	80	15	5
NC	75	15	10
ND	70	15	15

An organic binding agent, namely the polyvinyl alcohol (PVA), is added to WC, Co and TiC powder, and the resulting mixture is sieved through an $80-\mu m$ mesh to avoid the presence of any agglomerates in the final

mixture. Samples (of diameter 35.5 mm and thickness 14.5 mm) from these mixtures are compacted using a hydraulic press of type (Hefei Metalforming Machine Tool Works, China) under a load of 15 MPa.

To remove the PVA organic binder, a steaming at 400°C of the cermets for 3–4 h is carried out. The sintering, carried out in a non-oxidising atmosphere (argon + hydrogen), proceeds through two heating stages, first at 600°C, then at 1430°C, with an overall heating and cooling time of about 10 h in the furnace under hydrogen pressure. Then the surfaces of the obtained samples are subjected to flat grinding. The measured porosity on polished samples was found to be consistent with ISO4505 standard. The hardness was measured using a durometer on a Hardness Testing Instruments Machine (Induno Olona, Varese, Italy), and the sample densities were determined according to Archimedes' method. The measured density and hardness of the samples are practically consistent with ISO compositions (see Table 2) [1]. The characterisation of the cermets microstructure is carried out using a Philips-branded FEG ESEM XL 30 scanning microscope (Germany).

Table 2: Density and hardness of cermets.

Grades	Density (g/cm ³)	Hardness (HRC)
NA (reference)	13.86	69
NB	13.48	71.6
NC	11.74	74.4
ND	10.7	77.7

2.2 Friction tests

Polished (WC/TiC-Co) and reference (WC-Co) cermet samples (Table 1) were subjected to rotary sliding tests of friction at high temperature on a pin-on-disc tribometer, with a linear speed of 0.5 m/s and an applied load 20 N(CSM Instruments, Switzerland). The four samples of cermets were tested (Table 1), at two different temperatures (Table 3). The chosen temperatures 450°C and 650°C represent, respectively, the maximum and the minimum surface temperature values of our WC/TiC-Co based hot rolling deformation tools. The duration of the tests was chosen to be 2 h 46 min because it has been noticed that, during friction experiments, the first wear track appeared after about 2 h (Figure 3). The alumina ball used for the friction tests has a diameter of 6 mm, a density and hardness values as reported in Table 4. Then, to avoid the premature adhesive wear of the ball, the surface, in direct contact with the sample, is slightly changed after each step of the friction tests.

Table 3: Thermo-mechanical parameters of wear tests applied to each grade.

Parameters	Thermo-mechanical parameters of wear tests					
	Applied load (N)	Sliding speed (m/s)	Contact temperature (°C)	Distance travelled (m)	Time of test	
P 1 P 2	20 20	0.5 0.5	450 650	5000 5000	2 h 46 min 2 h 46 min	

Table 4: Density and hardness properties of an alumina Al₂O₃ ball.

	Density (g/cm ³)	Hardness (HV)
Ball properties	3.2	1800

The ball rubs against the surfaces of the test samples in such a way that the wear track is a circle of radius 5 mm, centred on the test cermet (disc) (Figure 2). The friction force, the friction coefficient, the humidity, the track temperature and the furnace temperature are regularly recorded throughout with the acquisition time in seconds using the software (Tribometer module/Version 4.4.Q).



Figure 2: Process of the friction test between ball in Al_2O_3 (pin) and cermets (disc).

2.3 Procedure for measuring wear rates and characterization of friction tracks

The procedure for measuring track wear rates was carried out using a surface profilometer [the threedimensional (3D) optical microscope Contour GT-I Bruker Nano Surfaces Division, Tucson, AZ, USA]. The analysis procedure consists in using software (vision) to analyse the wear track image (Figure 3). This analysis gives a geometrical relief of the wear track (profile of the worn track) of the test sample. The wear track volume is characterised by the depth (in μ m) measured with respect to the surface of the sample, the width (in mm) and the perimeter (in mm) of the wear track given by an optical profiler (Figure 4). Four equally spaced measurements were taken on each circle track (90° between two successive points) (Figure 5) [39], [40], [41].



Figure 3: Trace of the wear track after the friction test.







Figure 5: The four sections (A–D) taken by the optical profiler of each track spaced apart by 90° for measuring the average used volume.

The average of the four wear profile worn volume V of the test samples was calculated. The wear rate (K) was then deduced from the worn volume for a travelled distance D = 5000 m and an applied load P = 20 N [18], [26], [42], [43],

$$K = V/(P \times D) \left(\mu m^3 / N / m\right) \tag{1}$$

The microstructure study was carried out to observe the degradation behaviour of the wear tracks for the WC/TiC-based materials, where the NB grade is taken as an example. It is worth noticing that a complete microstructure analysis is beyond the scoop of the present work. The microstructure analysis, realised at the wear zone level following the friction tests, was conducted using a scanning electron microscope (SEM) (Philips XL FEG-ESEM, Germany) equipped with a dispersive energy electron spectrometer (EDS). This analysis allows us to find the different elements quantified at different points in the area of the worn tracks.

3 Results and discussion

3.1 Friction and wear behaviours

The results of the long-term friction tests of 2 h 46 min, for 5000 m travelled distance of dry sliding were chosen for this type of materials (cermets), with grades of WC-Co-TiC and WC-Co reference, of high tribological performances. Very little data concerning friction coefficients are available for testing hard metals in contact with alumina at high temperatures between P1(450°C) and P2(650°C). The relatively long time period friction tests that we choose was motivated by our aim to go beyond the running-in regime (quick wear) and reach the working wear. The COF against to travelled distance in metres of different grades NB 5% TiC, NC 10% TiC and ND 15% TiC according to the reference NA without TiC under applied temperature between P1(450°C) and P2(650°C) are given in Figure 6.



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Figure 6: Comparison of the different TiC contents of the (WC-Co-TiC) cermets compared to the WC-Co (NA) reference to according COF for sliding distance (5000 m) obtained under applied temperature between P1(450°C) and P2(650°C) : (A) (NB) 5% TiC, (B) (NC) 10% TiC and (C) (ND) 15% TiC.

The sliding of different samples against alumina (Al_2O_3) shows that the COF has the same trend and the smallest value, for grades (NB), (NC) and (ND) with parameter P2(650°C) according to the reference grade (NA). This COF reached a stable variation with average value $\mu = 0.42$ in a short time relatively after short sliding distances of 300 m, these will be discussed in more detail in Sections 3 and 4. The friction coefficient was divided into two stages, for grades (NB), (NC) and (ND) for parameter P1(450°C) according to the reference grade (NA). This variation of COF average value $\mu = 0.42$ shows a more rapid decrease at the start of the test and relatively stable stage from point 600 m. The reference grade (NA) is unstable, for the parameter P2(650°C), the COF does not stop increasing over the whole distance. With the other parameter, P1(450°C), micro oscillations appear starting from the point 1500 m until the end of the test. These initial instabilities likely change the wear mechanism types (see Sections 3 and 4).

The COF took the average values $\mu = 0.41$ and $\mu = 0.58$, over the entire test distance for any grade of parameters P2(650°C) and parameters P1(450°C), respectively. All these results indicate that the COF, for a given grade, if the contact temperature increases then the COF decreases and vice versa.

The comparison of the COF variation for the different grades with TiC against an alumina ball (Al₂O₃) according to the reference grade (NA) (Figure 7) indicates a medium and stable friction coefficient reaching a stable value $\mu = 0.58$ and $\mu = 41$ relatively in a short time, after 350 m of dry sliding. We also observed that the variation of the grade (NC) is relatively the most stable over the whole distance. We notice that these results indicate that the presence of TiC proves the COF stability of different grades according to the reference grade (NA) for the parameters P1(450°C) and P2(650°C).



Figure 7: Comparison of the variation of the (COF) of different grades according to the reference grade (NA) for the two temperature:(A) (P1) (450°C), (B) (P2) (650°C) for a distance travelled (5000 m).

3.2 Wear rate

Comparison of the wear rate of the different grade cermets with TiC according to the reference grade WC-CO indicates that increasing the content of TiC by 5% implies an average decreasing by 20% of the wear rate for both P1(450°C) and P2(650°C) (Figure 8). However, the wear rate decreases with the increase of the TiC content with respect to the reference grade (NA). The increase in temperature has an important effect on the wear rate: increasing temperature from P1(450°C) to P2(650°C) results in nearly 350% increase in total wear rate for the four samples. It is worth noticing that the grade ND (15% TiC content) exhibits the lowest wear rate with K = 378 μ m³/Nm and K = 2446 μ m³/Nm for the two temperatures P1(450°C) and P2(650°C), respectively. On the

other hand, the reference grade (NA) shows a more important wear rate, with values of K = $774 \,\mu m^3/Nm$ and K = $4115 \,\mu m^3/Nm$ corresponding to P1(450° C) and P2(650° C), respectively.



Figure 8: Comparison of the wear rate of different grades cermets according to the reference grade (NA) for the two temperature P1(450°C) and P2(650°C) for a distance travelled (5000 m).

3.3 Microstructure analysis for wear

SEM micrograph images of the wear track of grade (NB) WC-Co-5% TiC cermets corresponding to P1(450°C) shows that the wear track is generated mainly by debris and small oxidised zones (Figure 9A). The one corresponding to P2(650°C) (Figure 9B) shows that the track is generated by oxidised debris, which indicates the influence of temperature. An oxide layer is generated, this layer enables the formation of wear resistance, this is confirmed by the value and stability of the obtained COF variation.



Figure 9: SEM micrograph of the wear track of grade (NB) WC-Co-5% TiC cermets after sliding against Al $_2O_3$: to according of the two temperatures: (A) P1(450°C) and (B) P2(650°C) after (5000 m) of sliding.

SEM/EDS micrographs of the wear tracks of grade (NB) WC-Co-5% TiC cermets after sliding against alumina for long-term friction tests (2 h 46 min) are shown in Figure 10 and Figure 11 and discontinuous layers were formed on the wear tracks. A few micro cracks of hard metal in various directions are observed at the interface. SEM/EDS analyses at points (a–c) (Figure 10) show spectra of different peaks containing elements (C, O, Al, Ti, Co, W) (Table 5). This analysis indicates homogeneity of smaller wear debris formed in the wear track and points of tungsten oxides at the various points of the track. The existence of these wear debris is confirmed by the elements of the ball (O, Al) (Table 5) found in the track. These analyses also indicate that the presence of TiC in the grade (NB) at temperature (450°C) has delayed the oxidation which leads to an improve of the resistance to wear, the friction and the resistance to oxidation.



Figure 10: SEM micrographs of the wear track of grade (NB) WC-Co-5% TiC cermet when sliding against Al_2O_3 : EDS spectrums using temperature P1(T = 450°C) after (5000 m) of sliding.



Figure 11: SEM micrographs of the wear track of grade (NB) WC-Co-5% TiC cermet when sliding against Al_2O_3 : EDS spectrums using temperature P2(T = 650°C) after (5000 m) of sliding.

Table 5: Different elements of the spectra (a-c) SEM/EDS in different points of the used track for temperature P1(450°C).

Elements	С	0	Al	Ti	Со	W	Total
Spectrum (a)	4.462546	16.71093	1.074495	3.98432	14.15275	59.61496	100%
Spectrum (b)	4.699013	13.98472	0.692324	3.110177	15.38206	62.1317	100%
Spectrum (c)	11.14747	8.063785	1.807032	3.929418	18.09596	56.95634	100%

Analysis by SEM/EDS of the microstructure at points (a–d) (Figure 11) shows the formation of an oxidised layer (tribofilm) forming mainly an oxide rich in tungsten (W) at points (b–d) and an oxide rich in cobalt (Co) at point (a) (Table 6). This analysis also indicates that the formed oxide is compact and adherent, distributed over the entire wear track (formation of an oxide film) which improves the resistance to wear at high temperature. EDS spectrum on wear tracks proves that there is a process of material (Al) transfer from the alumina ball to the track which would indicate a three-body wear mechanism.

Table 6: Different elements of the spectra (a–d) SEM/EDS in different points of the used track for temperature P2(650°C).

Elements	С	0	Al	Ti	Со	W	Total
Spectrum (a)	1.71275	21.71036	0.482545	0.309294	47.63304	28.152	100%
Spectrum (b)	2.623737	17.85586	0.378818	1.314193	19.72604	58.10135	100%
Spectrum (c)	2.012118	18.3842	0.585716	2.241436	17.24254	59.53399	100%
Spectrum (d)	2.018057	19.57532	0.790104	3.33697	16.54762	57.73193	100%

3.4 Mechanism of tribological degradation

The wear mechanisms at high temperature are more complex in materials of high hardness (cermets) such as those used in the case at hand. According to the obtained SEM images, the conducted friction tests at high temperature have resulted in a tribological degradation mechanism in the sample NB: At the beginning, the dominant mechanism is plastic deformation of the cobalt. The oxidation of cermet components implies the formation of several oxides in the wear track, giving rise to fragile cracking followed by debris detachment. This mechanism, accelerated by increasing the temperature, forms a tribolayer (forming an oxide layer) which indicates wear resistance at high temperature (650°C) and resulting in a significant friction coefficient drop. In other words, as the wear formation processes a tribolayer area (a wear resistance layer), from third-body wears debris, and is generated. Moreover, this analysis shows that the formation of the major chemical component within the tribolayer is due to the ambient atmosphere. Also, traces of Ti and of W coming from TiC and WC, respectively, are observed. We have noticed fine TiC particles which are fragments of the original TiC formed by cracking and Co binder release. These particles precipitated at the hard metal interface under high temperature, confirming, therefore, wear resistance of the considered cermets.

4 Conclusion

In this article we have presented a comparison of the friction and wear of cermets with different ratios WC/TiC-Co (5%, 10%, 15%) of TiC, including a reference grade (WC-Co) without TiC for dry rotational sliding friction tests against an alumina ball, at two different temperature parameters P1(450°C) and P2(650°C). Here are the observations and conclusions obtained:

- 1. A low and more stable COF was found for the parameter P2(650°C) of the grade (NB) with an average value $\mu = 0.41$ and for the parameters P1(450°C) of the grade (NC) with an average value $\mu = 0.55$.
- 2. Comparison of the COF of each nuance for the two temperatures (450°C) and (650°C) with respect to the reference nuance (NA) confirms the stability of the COF of the different grades with TiC.
- 3. Reference grade (NA) without TiC show an unstable friction coefficient either constantly increasing or with numerous peaks during the test period for the various tribological parameters P1(450°C) and P2(650°C).
- 4. The wear rate of the grades with TiC (NB, NC, and ND) is lower with respect to the grade without TiC (NA), and the grade (ND) has the lowest rate. Also, it has been found that the reference grade, without TiC, shows the maximum wear rate.
- 5. The micrographs pictures SEM/EDS of wear tracks of the grade (NB) are used to determine the process of tribological degradation. It shows the beginning of a generated oxidation layer giving a wear resistance at P2(650°C). This is confirmed by the values and the stability of the coefficients of frictions found. The presence of TiC gives rise to a compacted layer (tribofilm), rich in tungsten, cobalt and oxygen (resistance of wear).
- 6. This work confirms the incorporation of TiC, which improves the tribological behaviour (friction, wear) under high temperature, improving the lifetime of WC/TiC-based cermets. The investigation carried out in this paper shows that WC/TiC-based cermets have a promising tribological characteristics under high thermo-mechanical conditions for real applications.

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