

DRY SLIDING FRICTION AND WEAR OF THE WC/TiC-CO IN CONTACT WITH Al_2O_3 FOR TWO SLIDING SPEEDS

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Abstract. *The paper examines the friction and wear behavior of four different WC/TiC-Co cermets, where three of them are composed of 5%, 10% and 15% TiC additions, and a WC-Co grade without TiC, taken as a reference material for comparison purpose. The principal aim is to improve wear resistance to high sliding speeds (hot rolling) of the WC-Co material as a reference by adding previously-listed percentage of TiC. The samples (cermets) were prepared according to the powder metallurgy procedure, which includes the preparation of the powder mixture, its compression shaping and liquid phase sintering. Sintering was carried out at 1460 °C, for 14 hours, in a reducing medium (H_2). The TiC materials are added in order to boost hardness of the WC-15Co cermet and, consequently, its resistance to wear under thermomechanical conditions. The experiments are conducted using a pin-on-disc tribometer in contact with Al_2O_3 alumina ball at two sliding speeds of 0.5m/s and 0.75m/s, at a high temperature of 450°C, and a 20 N load. It has been noticed that some recorded friction coefficients are unstable and exhibit many peaks during almost the whole friction test period. The obtained results from the SEM microscope show that the wear behavior of the new proposed material is improved, where it has been shown that, at the sliding speed of 0.75m/s, the greater the TiC percentage is, the lower the average friction coefficient will be. Also, for the speed of 0.5 m/s, the average friction coefficient is relatively more stable with the TiC percentage increase. Moreover, the obtained experimental results show an average wear rate decrease, with respect to reference grades (NA), that amounts to nearly 36% and 41% at the two sliding speeds P1 (0.5m/s) and P2 (0.75m/s), respectively.*

Key Words: Friction, Wear, Sliding Speed, Cermets, WC/TiC-Co

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1. INTRODUCTION

Titanium carbide (TiC) based cermets, used in many industrial applications, have received considerable attention due to several interesting properties at a high temperature, including a good tribological behavior, a better strength to weight ratio, and a higher wear resistance [1-3]. Also, the additions of TiC in the mixture of Co WC powders are justified by a better hot stability of titanium carbide compared to tungsten carbide. On the other hand, tungsten may produce volatile oxides which then accelerate the degradation of the surface of the material subjected to high temperature wear. The manufacture of cermets (WC-Co) could be accomplished by various techniques such as liquid phase sintering and conventional molding processes including injection molding. Yang et al. [4] give reviews of the additive manufacturing (AM) process, which appears to be an alternative to the traditional manufacturing processes able to produce parts with very complicated geometry. However, in this work we have adopted the classical technique based on liquid phase sintering because of its relative simplicity and good control of its parameters. Even though WC-Co cermets, without any addition, are widely employed as a cutting tool for high speed machining, hard metals and hard high-alloy steels, they exhibit poor oxidation resistance and corrosion [5, 6]. Also, it has been shown that these materials present surface degradation under high thermo-mechanical stresses and a high sliding speed [7]. Several authors have reported that adding TiC to tungsten carbide-cobalt (WC-Co) results in cermets with better performance just as they significantly enhance wear resistance of the WC/TiC cermets. Duman, et al. [8] investigated these performances in thermo-mechanical and high sliding speed applications.

Yang et al. [9] examined the impact of TiC addition on the oxidation behavior of the WC-Co cermet, following the appearance of complex oxides (W-Ti-O) which are more stable and more adherent than WO_3 oxide, volatile at medium temperature.

Furthermore, these WC/TiC grades show interesting properties, with respect to conventional WC-Co cemented carbides, including high hardness, good wear and oxidation resistance, high thermal and chemical stability, and a relatively low friction coefficient at a high temperature [10-12]. These materials are used in many industrial devices, involving high temperature dry contact components, such as hot-rolling dies, and aerospace equipment [13]. Furthermore, many authors have recently reported a considerable improvement of wear resistance of the WC-Co cermets, through the incorporation of TiC carbide particles [14-16]. The aim of the work is to examine the influence of the TiC addition on the cermet wear behavior at a high temperature for two different sliding speeds.

In this study, four grades, prepared by the procedure of powder metallurgy (WC, Co, TiC), are employed. The four shades of cermets were prepared with different ratios of WC/TiC-Co contents (5%, 10%, and 15%) of TiC including a reference grade without TiC (WC-Co). The tribological behaviors of these cermets were studied by severe friction tests for dry rotary sliding. Hence, this paper investigates the wear behavior of these WC-Co-TiC grades at a high temperature and high sliding speed, and compared to the reference WC-Co cermet. The experimental tests have been carried out under a load of 20 N at a temperature of 450°C using two sliding speeds of 0.50 m/s and 0.75 m/s. These parameters have been selected depending on the test machine (high temperature tribometer) characteristics and the use conditions of the WC-Co-TiC material. The chosen temperature represents an average value of the working temperature of the

considered material. For the two sliding speeds, our choice is due to the fact that the underlined material employs an average speed of about 0.5-0.75 m/s in hot rolling applications. The load of 20N is an average machine value. The obtained results have shown that adding TiC carbide particles has a positive effect on the wear behavior of the underlying cermets for the two considered speeds. Moreover, it has been noticed that addition of TiC stabilizes, somewhat, the friction coefficient even at a high speed. Also, we have observed the formation of an oxide film made of tungsten and cobalt for the two considered speeds of 0.50 m/s and 0.75 m/s, respectively. Also, Lemboub et al. [17] show by microstructure analysis the formation of a mixed carbide (W, Ti) C of « core-rim » type morphology.

2. EXPERIMENTATION AND MATERIALS

2.1 Cermet preparation

The mean size of the particles and powder density of WC, Co and TiC, manufactured by NORINCO GROUP CHINA, are given in Table 1.

Table 1 The mean density and particle size of various powders WC, Co and TiC

Various powders	WC	Co	TiC
Particle size(μm)	1.1	2	4
Average density of the powder (g/cm ³)	4.5	<0.75	4

The employed cermets, made of various compositions, reported in Table 2, are prepared using a classical procedure of powder metallurgy. The Cobalt composition has been chosen to be 15% Co. This percentage allows better heat removal when the cermet is heated during operation and guarantees a compromise between hardness and toughness [18,19].

Table 2 Weight percentage of the samples

Grades	Composition in Wt.%		
	WC %	%CO	% TiC
NA (reference shade)	85	15	-
NB	80	15	5
NC	75	15	10
ND	70	15	15

In order to guarantee homogeneity of the microstructure of each shade, a grinding of the powder prepared in mass percentage of the shades is poured into a ball mill (Fig. 1). This operation makes it possible to obtain homogeneity of the different mixtures of the powder of the samples, prepared in a humid medium formed of C₂H₅OH alcohol during an average grinding of 24 hours.



Fig. 1 Powder ball mill

The mixture of powders is dried in an oscillating dryer (oven) intended for drying the powders of the composition (WC-Co-TiC). The average drying time of 30 to 40 min causes a vaporization of the alcohol C_2H_5OH ; then the dried powder is mixed with an adhesive to ensure good agglomeration of the three different powders of the composition. Finally, a forced sieving of the agglomerated powder allows the separation of other elements (large grain sizes) and obtaining grains of the same size.

2.1.1 The microstructure of grades before wear tests

The microstructure of grades before wear tests, depicted in Fig. 2, shows a SEM micrograph of the WC-Co-5TiC sinter which clearly distinguishes different constituents and their distribution in the structure.

The compression of the dried and sieved powder is carried out by a hydraulic press. The pressing values are related to the density (compaction) and the geometry of the specimen. The geometry of the specimen (15mm thick and 35mm in diameter) is calculated while considering the shrinkage of the specimen after cooling. Finally, an average pressing of 15 MPa is applied, followed by a steaming operation for an average duration of 3 to 4 hours in an oven in order to vaporize the glue of the test pieces.

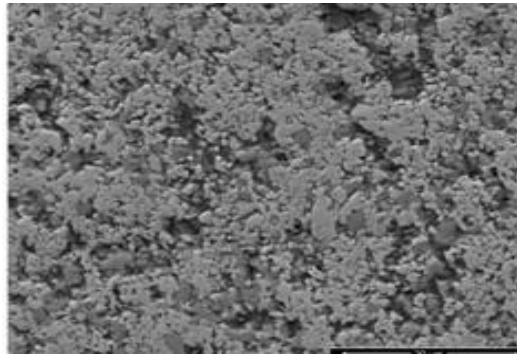


Fig. 2 Microstructure SEM image of grade (NB) 5% TiC before wear tests

The sample sintering cycle (cermets) is a standard cycle with three stages: heating to $600\text{ }^{\circ}\text{C}$ followed by a rise to $1460\text{ }^{\circ}\text{C}$, and cooling for an overall average time of 14 hours in the oven. The sintering operation consists of heating the test pieces to such a temperature that the powder grains weld together. The sintering cycle is carried out in a tubular chamber in the furnace blown by an argon gas in a non-oxidizing atmosphere ($Ar + H_2$).

2.1.2 Characterization of samples

The hardness and the density tests, carried out using INDUNO OLONA-VA-ITALY device and Archimedes method, respectively, have shown that the obtained samples comply with ISO standards as reported in Table 3. The microstructure of the wear track is analyzed by employing a Philips-branded FEG ESEM XL 30 microscope.

Table 3 The mean density and particle size of various powders WC, Co and TiC

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

2.2 XRD phase analysis

Fig. 3 reports the XRD diffraction spectrum obtained on the WC-5%TiC-Co cermet. The specifications of the used XRD machine are: (Operation angle range: 15-90°, step angle: 0.02°, KCuAnticathod: 0.154056 nm, power 3KW, Max Tube current 60 mA). Note that the identification of the phases was carried out through the use of Xpert high score plus and confirmed on the basis of the JCPDS - International Center for Diffraction Data 1996 files. Besides WC, which is the main phase of cermet, the figure shows peaks of the complex carbide Co₃W₃C, formed from the interaction between cobalt and WC during sintering in the liquid phase. The spectrum also highlights the simple TiC carbide, represented by very distinct peaks. The superposition of the WC and TiC peaks, corresponds to the mixed carbide (W, Ti) C, resulting from the solubility between the tungsten (WC) and titanium (TiC) monocarbons.

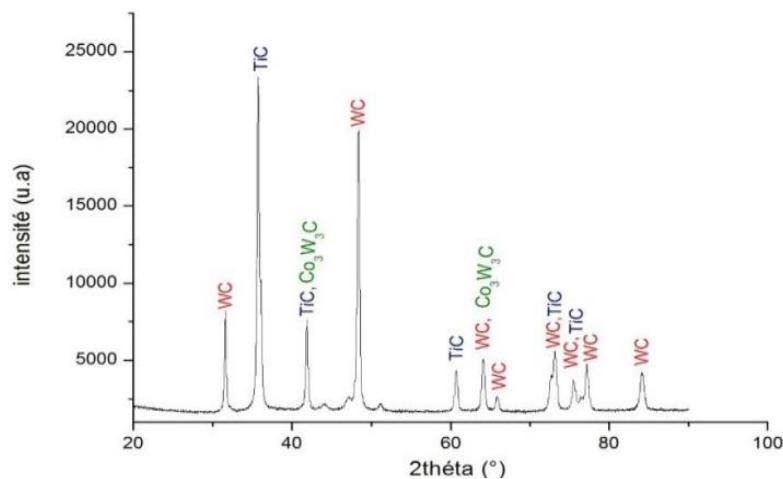


Fig. 3 XRD patterns of grade (NB) 5% TiC before wear tests

3. TESTS OF FRICTION

Wear tests were performed at 450°C for two sliding speeds and without lubricant on a CSM high temperature pin-on-disc tribometer (Fig. 4). The test process involving the Al_2O_3 ball and the two cermets (WC/TiC-Co) and the reference (WC-Co) samples have undergone friction and rotary sliding tests at a high temperature using a tribometer (CSM Instruments Switzerland) (Fig. 4)[20]. The four considered cermets have experienced tests, at two speeds (Table 4), using a 6 mm diameter alumina ball with hardness (HV) and density values of 1800 and 3.2, respectively. For each grade, three friction tests, corresponding to three radii, are carried out for two parameter sets P1 and P2 (force, temperature and sliding speed) (see Fig. 4). Thus, the estimated average value of the coefficient of friction is recorded.

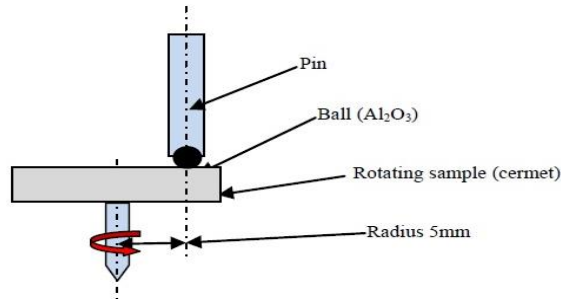


Fig. 4 Friction test process between the Al_2O_3 ball and the cermets

Table 4 Wear test employed parameters

Parameters	Thermo-mechanical parameters of wear tests					
	Applied load(N)	Sliding speed(m /s)	Contact temperature (°C)	Ambient temperature Contact (°C)	Distance traveled (m)	Time of test
P 1	20	0.5	450	20	5000	2h 46min
P 2	20	0.75	450	20	5000	1h 53min

A wear track of 5 mm radius and centered on the test cermet disc (Fig. 5) is obtained due to the ball rubbing against the sample surfaces.



Fig. 5 Friction wear track of the samples after tests

The software Tribometer module (Version 4.4.Q), with a sampling time of one second, is used to regularly record the force and coefficient of friction, humidity, the penetration depth of the ball, the grades and furnace temperatures (Fig. 6).

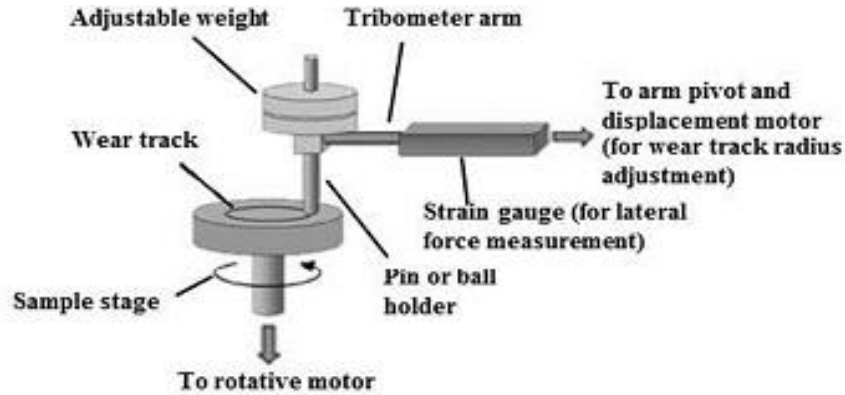


Fig. 6 Schematic illustration of the pin-on-disc tribometer

Two sliding speeds of 0.50m/s and 0.75m/s at 450°C are employed to test our WC/TiC-Co cermets usually employed in hot rolling tools. The test durations, for the 5000 m travelled distance, are 2 h 46 min and 1h 53 min for the two considered speeds of 0.50m/s and 0.75m/s, respectively. The choice of 450°C is due to the fact that this temperature could be considered as an average operating temperature of the cermet, intended for the preparation of hot rolling mill rollers. The selected distance will allow a wear normal regime operation of the materials and, therefore, obtain a significant wear volume.

4. WEAR RATE MEASURING PROCEDURE AND FRICTION TRACK CHARACTERIZATION

The track wear rates are measured by a profilometer (3D optical microscope Contour GT-I BrukerNano Surfaces Division Tucson, AZ USA). The wear track image, illustrated in (Fig. 5), is analyzed using the vision software. The analysis, using an optical profiler (Fig. 7), gives a geometrical profile of the sample track whose volume is characterized by three parameters namely, depth (in μm), width and perimeter (in μm).

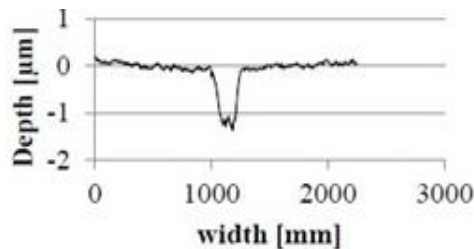


Fig. 7 Depth profile (μm) vs. wear track width (mm) and

Four images were taken at 90° from each other (Fig. 8), where a mean profile is obtained and the corresponding worn surface is calculated for each one. Then, an average worn surface is computed and multiplied by the track perimeter in order to obtain wear profile worn volume V [16,21,22]. Zhu, et al. [23] estimated the rate of wear (K) from the worn volume for a covered distance of 5000 m and using a load of 20 N. However, for a load P in (N) and a traveled distance D in (m), the wear rate is given as:

$$K = \frac{V}{P \cdot D} \quad (1)$$

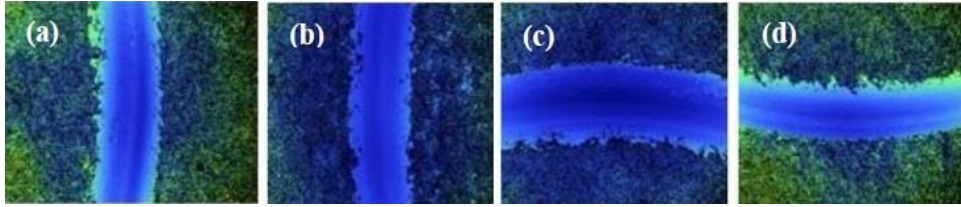


Fig. 8 The four optical profilometer measured sections spaced apart by 90°

5. RESULTS AND DISCUSSION

5.1 Friction and wear behavior of the cermets

Long-term friction tests of 5000 m covered distance for the WC-Co-TiC and WC-Co cermets, with severe tribological parameters, were conducted. For two parameters P1 (0.5 m/s) and P2 (0.75 m/s) (Table 4) used to test the considered cermets, no significant difference concerning the friction coefficients was observed between the two speeds. The long time period friction tests, for such hard materials, were chosen in order to surpass the running-in regime (severe wear) and achieve the mild wear regime. Employing parameters P1 and P2, friction coefficient (COF) against the travelled distance for four considered samples NB 5% TiC, NC 10% TiC, ND 15% TiC and reference NA with no TiC addition, are given in Fig. 9.

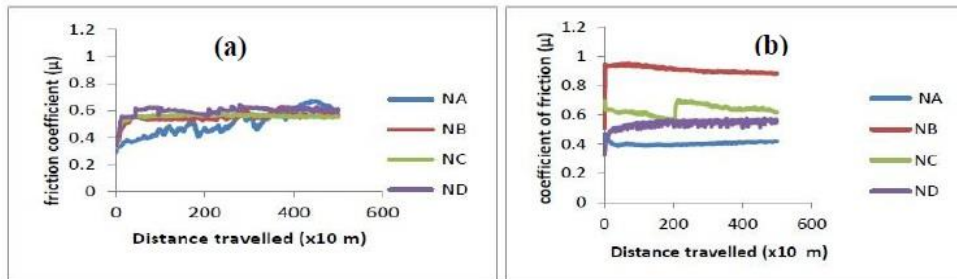


Fig. 9 Variation of the friction wear coefficient with respect to reference grade (NA) for two sliding speeds: a) P1 (0.5m/s) and b) P2 (0.75m/s)

For parameter P1(0.5m/s) in (Fig.9a)) and after a short sliding distance of about 400 m, the COF for grades (NB), (NC) and (ND) exhibit a relatively slow variation with a mean value of $\mu=0.58$ which is smaller than that of reference grade (NA). Grade (NC) shows a better stability of the friction coefficient, with an average value of $\mu = 0.56$ along the whole test. Reference grade (NA) is practically unstable with an increasing COF showing small ripples from the point 1500 m up to the end of the friction test. The instability observed for NA (the basic composition, i.e. without the addition of TiC) on the coefficient of friction curve could be explained by brittleness of the angular-shaped WC particles where the constraints are concentrated. These are the cause of cracking of the rupture of the surface layers of the cermet during sliding. The debris thus formed constitutes a third body which intensifies and regenerates friction. In (Fig. 9.b) for parameter P2 (0.75m/s), the COF shows a stable variation with relatively large average values of about $\mu=0.4$ for NA, $\mu=0.9$ for NB. The friction behavior of sample NB containing 5% TiC could be attributed to structural heterogeneity due to poor distribution of TiC particles during the homogenization operation of the powder mixture. The COF of the NC grade drops sharply after 3000 m of the test which shows heterogeneity within this region, then rises and stabilizes, until the end of the test, around an average COF value of approximately $\mu = 0.6$. The ND grade indicates stability with an average value of $\mu = 0.57$, showing micro peaks along the whole test probably due to heterogeneity of the microstructure. Hence, for this large sliding speed of 0.75 m/s under the considered test conditions, it seems that the addition of TiC has a negative influence on the friction coefficient. The impact of the TiC addition on the cermet tribological behavior, at a high temperature, could be explained by the graph of (Fig.9) which exhibits a stabilization level during the surface degradation process.

On the other hand, the curve of the reference cermet, without addition of TiC, which presents, in a first stage, lower values of the COF, increases monotonically with the increase in the sliding distance. This will lead to much higher values compared to those recorded on the samples containing 5% TiC. This could be explained by the stabilization of the layer due to the formation of complex oxides.

5.2 Wear microstructure analysis

The microstructure analysis is represented by the SEM image as shown in Fig. 10. The microstructure of the wear trace of the WC-Co-5% TiC material after sliding against an Al_2O_3 alumina ball with two sliding speeds P1 (0.5m/s) and P2 (0.75m/s) shows wear mechanisms which produce two types of wear represented by three zones as shown in Figs. 10b) and d):

- Zone a: weak cracked oxidized zone, abrasion wear.
- Zone b: strongly oxidized and weakened zone, detachment of oxides, formation of cavities by decohesion.
- Zone c: weakly oxidized zone, homogeneous wear by abrasion.

In Figs. 10 a) and c) are shown the traces of the wear track for the two speeds which indicates the influence of the increase in speed Fig. 10c) an abrasive wear layer, which are micro grains on the track.

Also for P1 (0.5m/s) sliding velocity shows also that the observed wear track is essentially created by debris and tiny oxidized zones. The process of sliding velocity increasing P2 (0.75m/s) illustrate that the generated track is caused by larger oxidized

debris of the abrasive wear. An oxide layer is generated which facilitates the development of wear resistance proven by the observed stability variation and the obtained friction coefficient value. Moreover, in both Figs. 10b) and d) few hard metal micro cracks, in various directions, are noticed at the interface.

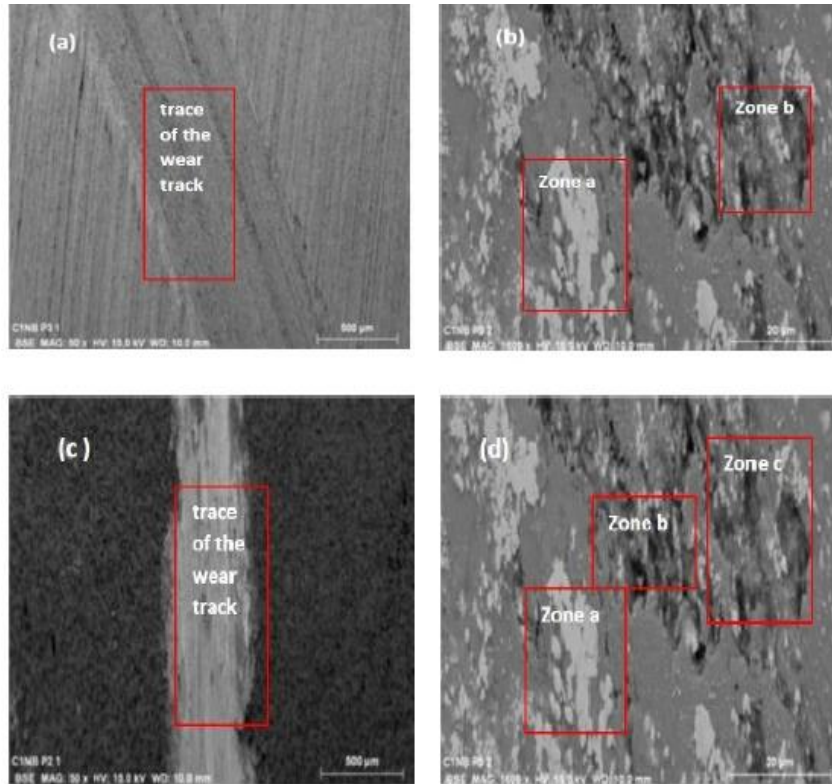


Fig. 10 SEM micrograph of the grade (NB) wear track for: a) and b) sliding speed P1 (0.5m/s), c) and d) sliding speed P2 (0.75m/s)

Fig.11 illustrates the same SEM/EDS analysis at points (a), (b) and (c) at sliding speed P1(0.5m/s) of grade (NB) WC-Co-5%TiC showing spectra of different peaks containing elements (C, O, Al, Ti, Co, W) whose composition percentages are given in Table 5. The figure shows homogeneity of small debris of tungsten oxide whose existence is confirmed by the elements of the ball (O, Al) (Table 5) found in the wear track. Hence, this analysis may prove that the presence of TiC in the grade (NB) has delayed the oxidation, improving, therefore, the wear and oxidation resistance.

The microstructure analysis using SEM/ EDS at points (a), (b) (c) and (d) with sliding speed P2(0.75m/s) of grade (NB) WC-Co-5%TiC (Fig.12) illustrates the formation of an oxidized layer (tribofilm) composed, mainly, of an oxide rich in cobalt at points (a) and (c), and an oxide rich in tungsten at points (b) and (d) as reported in Table 6. Furthermore, the analysis reveals that the formed oxide is adherent, compact and spread over the whole wear track (oxide film formation) which boosts the wear resistance at high

temperature. Besides, the EDS spectra of the wear tracks shows transfer of a small amount of the material (Al) from the alumina ball to the track indicating the occurrence of a wear process. The structure of the WC-5% TiC-Co cermet, obtained by liquid phase sintering, is shown in Fig. 11. It consists of a set of WC particles welded by the complex compound Co_3W_3C . Other WC particles bind to TiC, through a dissolution-precipitation mechanism in liquid phase, to form the mixed carbide (W, Ti) C which acquires a "core-rim" morphology.

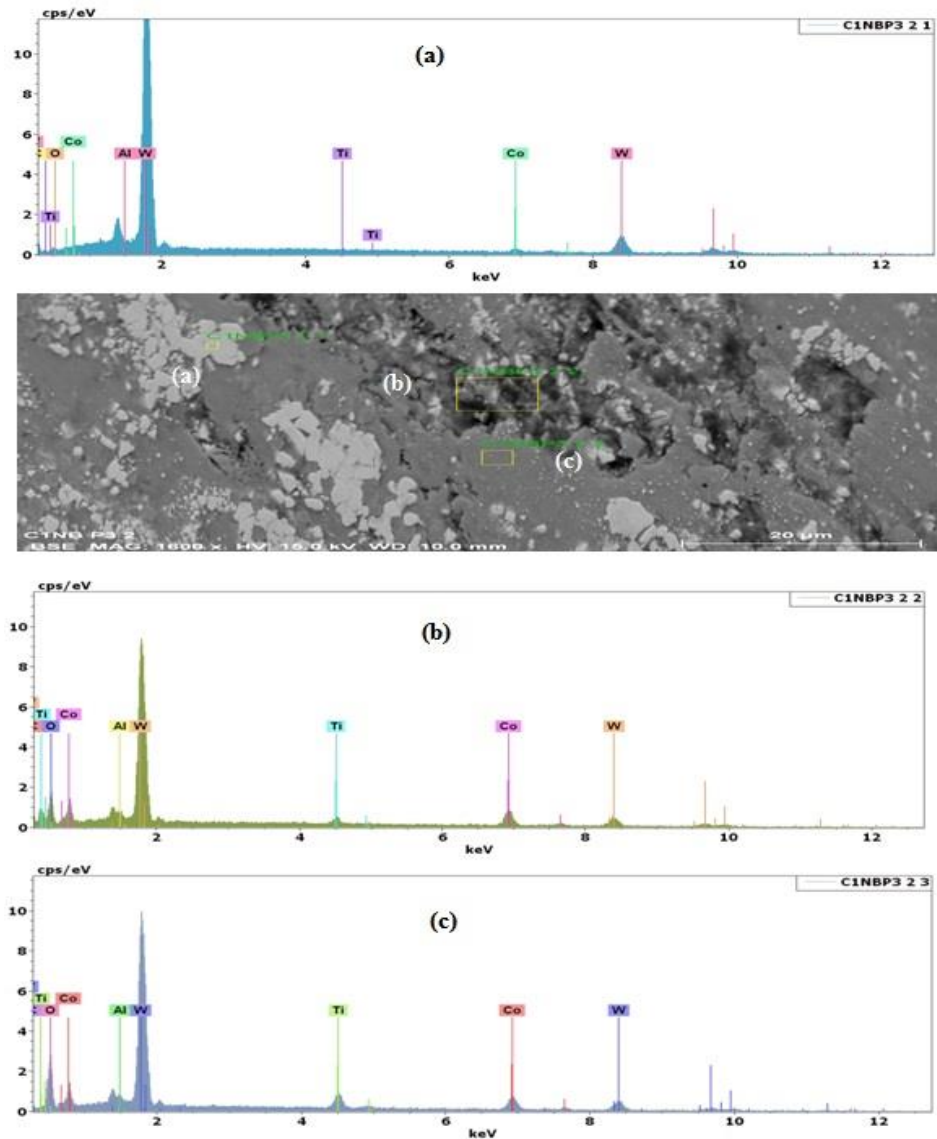


Fig. 11 SEM/EDS micrographs of the grade (NB) cermet wear track at sliding speed P1 (0.5m/s)

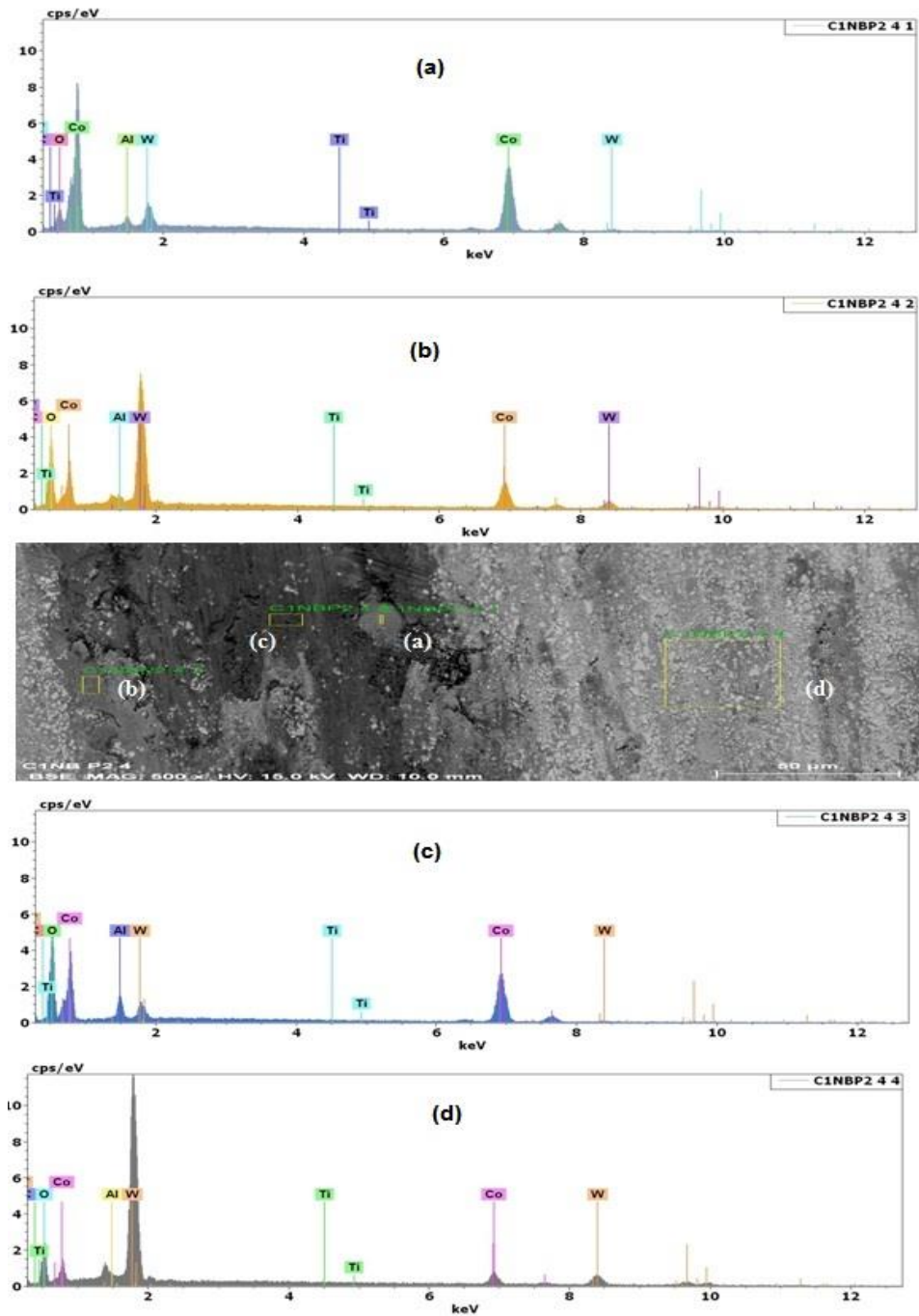


Fig. 12 SEM/EDS micrographs of the grade (NB) cermet wear track at sliding speed P2 (0.75m/s)

Table 5 The spectra (a, b, c) elements on different points for the sliding speed P1(0.50m/s) tracks

Elements	C	O	Al	Ti	Co	W	Total
Spectrum (a)	7.024561	0.68357	0.112209	0.064895	2.608032	89.50673	100%
Spectrum (b)	6.597754	11.20896	1.057664	3.137928	19.13305	58.86465	100%
Spectrum (c)	5.511438	10.03151	0.691847	3.510701	12.28522	67.96928	100%

Table 6 The spectra (a, b, c) elements on different points for the sliding speed P2(0.75m/s) tracks

Elements	C	O	Al	Ti	Co	W	Total
Spectrum (a)	4.010348	5.024438	1.187481	0	81.87428	7.903455	100%
Spectrum (b)	3.343464	20.52027	1.055945	0.350977	31.37472	43.35462	100%
Spectrum (c)	4.194662	22.07891	3.678074	0.131206	65.34973	4.567423	100%
Spectrum (d)	4.003289	14.01891	0.652586	0.105596	13.12244	68.09717	100%

5.3 Wear rate

Comparison of the wear rate for the three considered grade cermets, with respect to reference grade (WC-CO), in terms of TiC addition and using two sliding speeds P1(0.5m/s) and P2(0.75m/s), is illustrated in Fig. 13. It is clear that adding TiC results in a considerable decrease in the wear rate as reported in Table 7. Also, for the three cases, without TiC, 5% TiC and 10% TiC, the average difference of the wear rate between the two speeds is about 55%, whereas for 15% TiC addition, the wear rate difference has decreased to nearly 39%. In other words, it can be concluded that increasing the TiC percentage may reduce the speed influence on the wear rate.

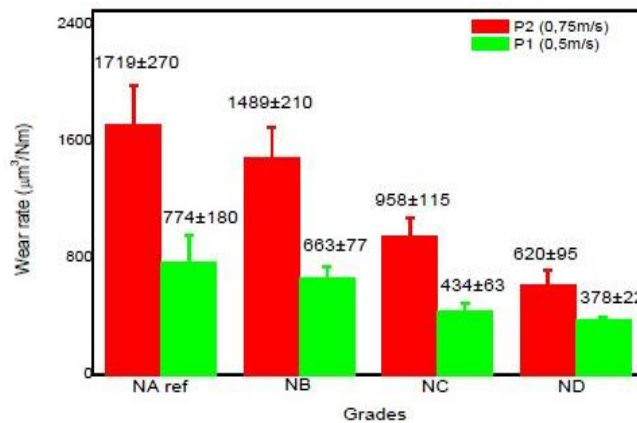


Fig. 13 Wear rate of different grades cermets according to reference grade (NA) for two sliding speeds P1(0.5m/s) and P2(0.75m/s)

Table 7 Wear rate decrease in % with respect to reference grades (NA) at two sliding speeds P1(0.5m/s) and P2(0.75m/s) for the travelled distance of 5000 m

Sliding speed (m/s)	TiC percentage (%)		
	5%	10%	15%
0.50	14.3%	43.9%	51.2%
0.75	13.3%	44.3%	63.9%

The improvement in the wear resistance of the WC-Co cermet, observed during the addition of TiC, could be explained as follows: titanium carbide modifies the morphology of the WC-particles, which passes from an angular or faceted shape to a rounded shape. According to several previous works [24-26], the absence of sharp angles on the rounded particle eliminates the concentration of stresses and, therefore, the risks of failure by propagation of brittle cracks in the material. This leads to a significant enhancement in resistance. On the other hand, the appearance of a "Core-Rim" type structure following a phenomenon of dissolution of fine particles of carbides and reprecipitation of mixed carbide during sintering in the liquid phase could have a positive effect on the wear resistance of the cermet. The hardness of the cermet will then increase with the rate of the TiC additions.

6. CONCLUSION

In this work a comparative analysis of the friction and wear rate for three cermets NB, NC and ND with TiC addition ratios of 5%, 10% and 15%, respectively, has been carried out. The comparison has been conducted with respect to a reference sample NA, with no TiC addition, for dry rotational sliding friction tests against Al_2O_3 ball, at two sliding speeds P1(0.5m/s) and P2(0.75m/s) and a fixed temperature $T=450^\circ C$. In addition, using SEM and EDS micrographs, the NB grade microstructure has been examined. From the obtained experimental results, some conclusions are drawn:

- For the sliding speed of 0.5 m/s, the friction coefficient of grade (NC) has revealed a better stability with respect to (NB) and (ND) cermets which, in turn, have shown an acceptable stability of the COF coefficient. Reference grade (NA) is practically unstable with an increasing COF with small ripples.
- For the second speed of 0.75m/s, the COF has exhibited a quite stable variation for the four considered cermets, with a relatively large average value due, probably, to TiC addition.
- The addition of TiC has resulted in a significant decrease of the wear rate. Also, adding TiC has clearly reduced the influence of the speed on the wear rate.
- Through SEM/EDS images of grade (NB) wear tracks, an oxidation layer made, mostly, of tungsten, cobalt and oxygen, has been noticed. The formed layer (tribofilm), is compact, adherent and spread over the whole wear track improving, therefore, the wear resistance at the considered temperature of $T=450^\circ C$. Moreover, these images show the formation of mixed carbide (W, Ti) C with a "core-rim" morphology type.

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