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The tribological and mechanical properties of niobium carbides (NbC) bonded with cobalt or Fe₃Al

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ABSTRACT

The tribological and mechanical properties of niobium carbide bonded with 8 vol.-% (NbC-8Co), 12 vol.-% of cobalt (NbC-12Co) or 12 vol.-% of Fe₃Al (NbC-12Fe₃Al) are presented. Rotating discs made of metalbonded niobium carbide were mated against alumina (99.7%) under unlubricated (dry) unidirectional sliding tests (0.1 m/s to 12.0 m/s; 22 °C and 400 C) as well as in oscillation tests (f=20 Hz, Δx =0.2 mm, 2/50/98% rel. humidity, n=10⁵/10⁶ cycles). Microstructure and phase compositions were determined as well. The tribological data obtained were benchmarked with different ceramics, cermets, hard metals and thermally sprayed coatings, where NbC bonded with 8% and 12% Co presented above 7 m/s the lowest wear rates so far in such a benchmark. Binderless NbC (HP-NbC1) and the metal-bonded NbCs exhibited low wear rates under dry sliding associated with P ·V high load carrying capacities. NbC-based hard metal bonded with 12 vol.-% of Fe₃Al resulted in a higher hardness level than for 12 vol.-% cobalt. The tribological considerations and for closed tribosystems against established reference tribo-couples. © 2014 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY license (http://creativecommons.org/licenses/by/3.0/).

1. Introduction

Hard metals are among the most important powder metallurgical (PM) materials and solutions for wear protection, having a long and successful history. They stand synonymously with wear resistance. Historically, tungsten carbide (WC) has dominated the market for cutting tool materials.

Approximately 80% of the global tungsten production is mined in China, yielding an annual total of 75,000 t [1]. Niobium, a refractory metal like tungsten, offers a possibility in partially or even fully substituting tungsten in hard metals.

Although niobium carbide has been well known for decades, knowledge of its property profile remained limited. Its poor sintering ability and very high sintering temperatures may explain why little research has been done on this material. These obstacles can now be overcome by either hot pressing, high-frequency induction heated sintering or by plasma-spark sintering (SPS). On the other hand, demand for niobium has increased significantly over the last 45 years, particularly as a micro-alloying element in high strength and stainless steels. In such alloys, niobium forms dispersed micro- or nano-sized niobium carbide particles, controlling the microstructure and thus improving mechanical properties [2,3].

Yet, mechanical and particularly tribological properties of NbC remain largely unexplored. From an a priori contemplation, NbC is expected to be superior to WC in cutting tool applications, because at 1,225 °C NbC is nearly insoluble in Cr, Ni, Co or Fe [4,5], whereas WC is fully soluble under the same conditions. The high solubility of WC in these metals is responsible for the chemical wear of WC.

Recent studies [6] on hot-pressed (HP) and binderless NbC (HP-NbC1) indicated that pure niobium carbide has a high intrinsic wear resistance, when benchmarked against different ceramics, cermets, hard metals and thermally sprayed coatings. The HP-NbC as such is quite brittle. Consequently, 8 vol.-% and 12 vol.-% addition of cobalt binder as well as 12 vol.-% addition of Fe₃Al (cobalt-free) binder will improve properties, such as toughness and strength. Application of SPS technology allowed reducing the sintering temperature to 1,280 °C and the dwell time to 4 min, as will be shown in this article.

2. Experimental procedure

* Corresponding author. Tel.: +49 0 172 3959594. E-mail address: mathias.woydt@bam.de (M. Woydt). Different types of test samples for tribological and mechanical testing were prepared from discs by means of electrical discharge machining (see Fig. 1). The powders for densification of HP-NbC1

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and cobalt as well as Fe₃Al-bonded NbC were pure and essentially free of tantalum.

The planar surfaces of the tribological samples were finished by lapping or by polishing (see Table 1).

2.1. Niobium carbide bearing test samples

The details of the mechanical and metallurgical features for hot-pressed and binderless NbC (HP-NbC1) using NbC powder (d_{90} =18.12 µm) from CBMM are disseminated in reference [6]. This powder had a carbon content of 11.45 ± 0.65 wt.-% carbon.

Commercially available NbC powder (Treibacher 100, Austria; FSSS=1.18 μm) and cobalt powder (Umicore grade Co-HMP, Belgium; FSSS=0.55 μm) were used for producing the cobalt bonded NbC samples. The granulometry of this NbC powder was d_{50} =1.72 μm and d_{90} =3.78 μm (using the Fraunhofer calculation model). The carbon content was 11.4 \pm 0.7 wt.-%. N2-atomized Fe₃Al powder (-325 mesh) from AGH University of Science and Technology, Krakow, Poland, was used for producing the NbC-12Fe₃Al samples.

University of Leuven (Katholieke Universiteit Leuven, Belgium) densified the powder mixtures of cobalt or Fe₃Al-bonded NbCs [7] to discs by solid-state sintering (SPS, type HP D 25/1, FCT Systeme, Germany). A pulsed electric current was applied with pulse/pause duration of 10/5 ms throughout all the experiments. The powder mixture was poured into a cylindrical graphite die with an outer diameter of 56 mm and sintered for 4 min. The conditions of densification are shown in Table 2. Graphite paper inserts were used to separate the graphite die/punch setup and the powder mixture. X-ray diffraction (XRD) and Electron Back Scattering Diffraction (EBSD) [8] identified cubic NbC in the SPS sintered discs (See Fig. 2).

The Field Emission Scanning Electron Microscopy (FESEM) image taken with SE detector and SEM image (see Fig. 2) revealed larger islands (situated deeper due to preparation) of the different metallic binders representing local inhomogeneity of the binder distribution. All metal-bonded NbC grades presented porosity-free NbC grains contrary to HP-NbC1 [6].

Table 1

Quantities describing the surface topographies of the tribological NbC-bearing samples.

Hard metals	Quantities of roughness [µm]				
	Rz	Ra	R _{pk}	R _k	R _{vk}
WC-6Ni HP-NbC1 (lapped) NbC-8Co (polished) NbC-8Co (lapped) NbC-12Co (polished) NbC-12Co (lapped) NbC-12Fe ₃ Al (polished)	0.176 3.065 0.038 1.342 0.042 0.910 0.054	0.026 0.384 0.005 0.177 0.007 0.120 0.008	0.013 0.143 0.007 0.213 0.007 0.220 0.010	0.071 0.948 0.016 0.577 0.022 0.380 0.027	0.051 0.944 0.009 0.219 0.012 0.150 0.011

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Hard metal	Temperature [°C]	Holding time	Pressure [MPa]	Constant heating rate [K/min]
SPS-NbC	2,000	12 min.	100	200
HP-NbC1 [5]	2,150	4 hours	50	10
SPS NbC-8Co	1,280	4 min.	40	100
SPS NbC-12Co	1,280	4 min.	30	100
SPS NbC-12Fe ₃ Al	1,300	4 min.	30	100



Fig. 1. Different samples machined by electro-discharge from discs; top: left for mechanical testing and right for cutting inserts; bottom: left for continuous and right for oscillating sliding.



Fig. 2. Morphology of NbC powder (top left), microstructure of NbC-8Co (FESEM, top right), microstructure of NbC-12Fe₃Al (SEM, bottom left) and XRD diffraction patterns (bottom right) of bonded NbCs (NbC-8Co, NbC-12Co and NbC-12Fe₃Al).

Bonding NbC with cobalt resulted in an average microhardness of $1,412 \pm 51$ HV0.2 for NbC-8Co and $1,410 \pm 13$ HV0.2 for NbC-12Co. Micro-hardness is considerably higher in NbC- $12Fe_{3}Al$ (1,633 ± 50 HV0.2) and in HP-NbC1 (1,681 ± 92 HV0.2). When performing HV5 measurements the Fe₃Al as intermetallic phase bonded NbC-12Fe₃Al appears to have a higher hardness (1,448 \pm 32 HV5) than the binderless HP-NbC1 (1,380 \pm 67 HV5) as seen in Fig. 3. NbC-based hard metal bonded with 12 vol.-% of Fe₃Al resulted in a higher hardness level than for 12 vol.-% cobalt at any indentation load. The strong load influence on microhardness for binderless NbC as shown in Fig. 3 is well established in literature and similarly applies for WC, W₂C, V₈C₇ or Mo₂C [9]. The effect is due to plastic deformation at room temperature under indentation via dislocation movements. In cubic carbides [10], the motion of dislocations occurs along the {111} planes in the direction $\langle 110 \rangle$.

The WC-6Ni (E204; C7P) used for comparison was from Sandvik Hard Materials and had a micro-hardness of 1,482 \pm 40 HV 0.2.

2.2. Mechanical properties

The four-point bending strength $(3 \times 4 \times 45 \text{ mm})$ at 22.3 °C (rel. humidity=33%) was determined in an INSTRON machine equipped with 5000 N load cell meeting class 1 according to EN 10002-2 and by using a loading device (cross head speed=0,8 mm/min) as required by DIN EN 843-1. The statistical Weibull analysis was performed according to DIN EN 843-5:2007 and is shown in Fig. 4. Higher strengths and Weibull moduli are expected from future grades with a more homogeneous distribution of metallic binders and the NbC grains. The trend in improving the strength by bonding NbC with



Fig. 3. Micro-hardness as function of indentation load for binderless and metalbonded NbCs.

cobalt or Fe₃Al is clearly visible. The strength level for NbC-12Co is already remarkable for a non-optimized demonstration grade.

The elastic modulus was determined on longer bars having the same shape as bending bars using the resonance method with a piezoelectric emitter-receiver in an ELASTOTRON 2000 machine (See Fig. 5). The calculated elastic modulus at room temperature for HP-NbC1 was 477 GPa [6], for NbC-8Co 443 GPa, for NbC-12Co 437 GPa and for NbC-12Fe₃Al 447 GPa using ASTM E1875-2008. At 1,000 °C, the elastic modulus of NbC-8Co decreased only to







Fig. 5. Elastic and bulk modulus versus temperature (in vacuum) of cobalt- and Fe₃Al-bonded NbCs in comparison to binderless WC and NbC.

382 GPa, for NbC-12Co to 368 GPa and for NbC-12Fe₃Al to 379 GPa. The data shown in Fig. 5 for binderless tungsten carbide were taken from reference [11] and for binderless NbC_{0.95} from reference [12].

2.3. Tribometers

The tribometers for unidirectional sliding [13,14] and oscillating [15] sliding are proprietary developments of BAM and the details are disclosed elsewhere [13,15]. They comply with ASTM G99 (DIN 50324) and with DIN EN 1071-13:2010. The wear volumes of stationary and rotating/oscillating specimen were calculated from stylus profilometry and the wear scar diameters by using ASTM D7755-11. The wear rate k_{ν} is defined as the ratio of volumetric wear to the product of load F_n and the sliding distance s. The coefficient of friction (CoF) and the total linear wear of both tribo-elements (specimen) were recorded continuously. One test per combination of parameters was performed, because the testing philosophy at BAM is to screen over a wide range of operating conditions rather than doing repeated tests, except at specific points.

2.3.1. High-temperature tribometer

Sintered alumina (99,7%) bodies were used as stationary spherical (toroids with R_1 =21 mm and R_2 =21 mm) specimens with polished surfaces (R_{pk} =0.019 µm), which were pressed against the planar surfaces of the rotating NbC. A normal force of 10N was applied, resulting in an initial Hertzian contact pressure P_{0max} of approximately 660 MPa. The sliding distance was 5,000 m. Experiments were performed at 23 °C and 400 °C in air (rel. humidity at RT approx. 35%) with sliding speeds of 0.1, 0.3, 1.0, 3.0, 7.5/8.0 and 10/12 m/s. The resolution limit of the wear rate for the rotating specimen corresponds to about 10^{-8} mm³/N · m.

2.3.2. Oscillating tribometer

The polished ball (\emptyset =10 mm; alumina 99.7% or 100Cr6H=SAE E52100) in the oscillating tribometer is fixed at the top of a lever with an integrated load cell for the measurement of friction force. The ball (non-rotating) is positioned on a disc that is fixed on a table, oscillating at 20 Hz and a stroke of 0.2 mm as well as is at 22 °C loaded by a dead weight acting as the normal force (F_N =10 N) perpendicular to the sliding direction. The tests were run under three relative humidity levels of 2%, 50% and 98% up to one million cycles. The sensitivity of a couple against the impact of

tribo-oxidation induced by humidity can be effectively quantified under dry oscillation.

3. Tribological results

The following tribological data under dry friction were compared with homologous results issued from the tribological database TRIBOCOLLECT of BAM for thermally sprayed coatings [14,16], self-mated ceramics, ceramic composites [17] and steels as well as mated with stationary specimen in alumina. The tribological characteristics of these materials are summarized in [18]. The colored areas indicate the ranges established with different grades of the indicated material system.

3.1. Dry sliding

The frictional level of NbC grades in Fig. 6 compares well with different tungsten carbide-based or Cr_2C_3 -based hard metals or monolithic alumina and thus qualifies these for traction and frictional applications rather than for low friction bearings. At RT, the friction of HP-NbC1 increased with increasing sliding speeds, whereas metal-bonded NbCs presented an opposite trend. At 400 °C, the friction decreased for all NbC grades and hard metal grades with increasing sliding speed, but were on average lower than those for WC grades. The friction of NbC grades at high sliding speed is a favorable property for a cutting tool, reducing the cutting forces, thus achieving a given cutting performance at reduced machine power.

Fig. 7 illuminates the total wear rate (sum of stationary (Al_2O_3) and rotating (NbC) specimen). HP-NbC1 comprised a particularly high wear resistance, especially at RT, which is more or less independent of sliding velocity. The wear resistance of HP-NbC1 at RT and low speeds is one of the highest. The metal-bonded NbC grades at RT displayed a rather constant evolution of the wear rate with increasing sliding speed. The wear rates of the metal bonded

NbCs decreased with sliding speed by one order of magnitude to low wear rates at high sliding speeds. At 8 m/s at RT, the wear rates k_v of the rotating disc in NbC-8Co were outstandingly low with $k_V=4.4/7.8 \cdot 10^{-7}$ mm³/N·m, when compared to all other ceramics and hard metals. The wear rates of the rotating discs made from NbC-12Co reached values of $k_V=9.6 \cdot 10^{-7}$ mm³/N·m at RT and 12 m/s.

At 400 °C, the dry sliding wear resistances of tribo-active materials ($Ti_{n-2}Cr_2O_{2n-1}$ -phases, (Ti,Mo)(C,N)), binderless HP-NbC1, metal-bonded NbCs and thermally sprayed Cr_2O_3 or WC-based hard metals ranged between 10^{-7} mm³/N·m and $5 \cdot 10^{-6}$ mm³/N·m on a level known from the regime of mixed/ boundary lubrication. Wear resistance under dry sliding of NbC grades is better than that of Cr_3C_2 and similar to or better than that of WC-based systems.

Fig. 8 represents the load carrying capacity expressed as $P \cdot V$ values (contact pressure times sliding velocity), for all NbC grades increased at room temperature from 1-2 MPa \cdot m/s at 0.1 m/s up to 100 MPa \cdot m/s at 8.0 m/s, because tribo-oxidation was enhanced with increasing sliding speed (or generated frictional heat). In contrast at 400 °C, the P \cdot V values ranged more or less on the same level as at RT.

First, the two NbC grades follow the same evolution, independent of ambient temperature, which indicates that tribo-oxidation of niobium carbide dominates this behavior. It is remarkable that the P·V values, or μ ·P·V, of these NbC grades increase with increasing sliding speed. As a result, as shown in Fig. 8, NbC presented an unusually high load carrying capacity.

Normally, the P · V values [18,19] of dry sliding couples decrease with increasing sliding speed. Triboactive materials [14,16], like $Ti_{n-2}Cr_2O_{2n-1}$ and (Ti,Mo)(C,N), represent the nearest neighbor to NbC grades having slightly lower P · V values or maximum frictional heat flows. It has to be taken into consideration that NbC has a very high melting point (3,522 °C). Hot-pressed Nb₂O₅ is relatively soft, having a hardness of only 500 HV0.2 and Nb₂O₅ has a melting temperature of 1,512 °C without sublimating. In contrast, WO₃ formed by tribo-oxidation on WC begins to sublimate above 800 °C.



Fig. 6. Coefficient of friction of HP-NbC1 and of cobalt- or Fe₃Al-bonded NbCs compared to different ceramics and hard metals [10,13,15] under dry friction at RT and 400 °C.



Fig. 7. Total wear coefficients of HP-NbC1 and of cobalt- or Fe₃Al-bonded NbCs compared to different ceramics and hard metals [10,13,15] under dry friction at RT and 400 °C.



Fig. 8. Maximum frictional power loss of NbC grades under dry sliding.

As can be seen in Fig. 7, the wear resistances of cobalt and Fe₃Al-bonded NbCs at RT and high sliding velocities are the highest. The high melting point of NbC and of Nb₂O₅, when compared with WC and the sublimation of WO₃, represents a tribological advantage under high temperature conditions at the cutting edge of a tool according to the shown wear resistances.

The wear tracks of HP-NbC1 obtained at RT and 400 °C are represented in reference [5]. NbC is a beneficial material for closed tribosystems under dry sliding, because no wear particles visibly agglomerated in the wear tracks. The observation that no agglomerates were found in the wear tracks is in line with the low wear rates of 10^{-6} mm³/N · m.

3.2. Dry oscillation

The tribological profile (wear rates or wear coefficients as " K_v " versus coefficients of friction as "CoF") is displayed separately for polished counterbodies of alumina in Fig. 8 and for ball bearing steel (100Cr6, SAE E52100) in Fig. 9. The arrows indicate the effect of increasing the relative humidity from 2% to 98%. The tribological



Fig. 9. Total wear rates with the associated coefficient of friction of alumina balls dry oscillating on discs of different materials with the influence of the relative humidity.

profiles of steels and the ceramic samples including WC-6Ni, HP-NbC1 as well as cobalt and Fe₃Al-bonded NbCs are sensitive to relative humidity. The degree and the trend depend on the counterpart either in the case of the polished alumina ball ($\emptyset = 10 \text{ mm}$) or of the polished 100Cr6 ($\emptyset = 10 \text{ mm}$). In contrast, the wear rates and coefficients of friction of binderless NbC (here: HP-NbC1) are practically insensitive to relative humidity when oscillating against 100Cr6.

In 100Cr6 steel or other ferrous alloys, tribo-oxidative [20] formation of Fe₂O₃ and/or hydrolyzed to α -, β - or γ -FeOOH and Fe (OH)₂ dominate under dry oscillation at RT. Generally, and particularly in comparison to polished WC-6Ni, the wear resistance of NbC grades under dry oscillation is high, having K_v values of 10^{-6} mm³/N·m. The wear rates under dry oscillation of binderless NbC (HP-NbC1) and the cobalt-bonded NbC grades are quite similar (Fig. 10).



Fig. 10. Total wear rates with the associated coefficient of friction of balls of bearing steel dry oscillating on discs of different materials with the influence of the relative humidity.

The presence of cobalt (NbC-8Co; NbC-12Co) and Fe₃Al binders (NbC-12Fe₃Al) in NbC grades increases the sensitivity of their frictional behavior to relative humidity. The coefficient of friction decreases under increasing relative humidity. The wear rates remain unchanged for cobalt-bonded NbC. The Fe₃Al binder further enhanced the influence of relative humidity on friction. Fig. 10.

4. Conclusions

The wear resistance presented by binderless NbC and NbC grades bonded by cobalt or ironaluminide (Fe₃Al) can easily compete with that of ceramics, "triboactive" materials and hard metals. Thus NbC qualifies as a member of the group of tribological materials with enhanced wear resistance. The room temperature wear rates of different NbC grades are low and less sensitive to increasing sliding speed. Remarkably, increasing sliding speeds to 8.0 m/s and above decreases the wear rate down to outstandingly low values of k_V of $2\text{--}7 \cdot 10^{-7}~mm^3/N \cdot m.$ The wear rates at 400 $^\circ C$ of NbC grades generally remained below 10⁻⁶ mm³/N · m, regardless of the applied sliding speed. The low wear rates of NbC were associated with high load carrying capacities (P-V-values), in excess of 100 MPa · m/s. The P · V values increase with increasing sliding speed. Under dry oscillation, the wear resistance of binderless NbC was insensitive to relative humidity for bearing steel (100Cr6=SAE E52100) as well as alumina counterbodies, whereas the coefficient of friction of metal-bonded NbC grades was reduced with increasing relative humidity indicating the impact or cobalt and Fe₃Al binders. The low solubility of NbC in metals [3,4] and the high P · V values are an ideal prerequisite for cutting tool materials. Furthermore, the achieved level in hardness and elastic modulus as well as the actual level in strength and toughness are sufficient to support the load at the cutting edge.

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