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OBTAINING AND CHARACTERIZATION OF COATED AND UNCOATED SINTERED CERMET CUTTING TOOLS

Marius PINTILIE[1], Rami ŞABAN[2], Ioan NEDELCU[3], Irina CÂRCEANU[4], Alexandru GHIBAN[5]

Lucrarea prezinta preocuparile autorilor in ceea ce priveste producerea material Aliajele dure de tip Cermet pe baza carbură de titan / carbonitrura de titan şi carbură de wolfram sunt cunoscute pentru larga lor aplicabilitate, datorita proprietăților remarcabile:rezistenta la uzura, rezistenta de oxidare şi coroziune ridicate. In vederea imbunatatirii performantelor acest aliaje au fost selectate pentru investigatii in cadrul acestei lucrari compozitii cu conținut ridicat de TiCN până la 70%, 20 % liant Ni şi 10% WC. Reperele sinterizate tip Cermet obtinute experimental au fost acoperite cu straturi extra-durede tip TiC, TiCN folosind o noua tehnica PVD, ce include pulverizare magnetron combinata cu implantare ionica (CMSII).

Cermet hard metal alloys based on titanium carbide/titanium carbonitride and tungsten carbide are known for large their applicability because of remarkable properties of high wear, oxidation and corrosion resistance.In order to obtain performance compositions with high content of TiCN up to 70%, 20% concentrations of metallic binder Ni, and 10% of WC, where selected for investigation in this paper. The sintered cermets work pieces obtained experimentally were coated with extra-hard layers type TiC, TiCN using a new technique PVD including a magnetron sputtering combined with an ionic implant. The plasma was produced by a magnetron discharge and the procedure can be defined as a Combined Magnetron Sputtering and Ion Implantation (CMSII).

Keywords: hard alloys, cermet, extra hard layers, coating

[1] PhD Student, Faculty of Materials Science and Engineering, University POLITEHNICA of Bucharest, Romania, mariusnko@yahoo.com
[2] Professor, Dean of the Faculty of Materials Science and Engineering, University POLITEHNICA of Bucharest, saban@orangenet.ro
[3] Scientifique Researcher, *Prelucrări Metalurgice Prod SRL*, Bucharest, Romania prelucrari_metalurgice@yahoo.com
[4] Scientifique Researcher, Metallurgical Research Institute of Bucharest, Romania, irina_carceanu@yahoo.co.uk
[5] Polytechnic University Bucharest,PhD Student, Faculty of Materials Science and Engineering

1. Introduction

Cermets are materials with high resistance at higher temperatures, making special refractory materials resisting to corrosion in acid medium and a good wear resistance. They present a set of physical and mechanical properties, which allow them to be used in different fields extreme working conditions.

TiCN based cermets, which are dense and hard materials are important components of high-speed cutting tools [1,2]. Such tools require simultaneously high values for the triplet: hardness (wear resistance) – toughness (bending resistance) – cutting rate and shock resistance, corrosion and high temperature oxidation properties too. These temperatures appear usually during their service due to the friction generated in the cutting process[3]. All these characteristics are to be ascribed to the mechanical properties of the hard phases, which are retained in the cermets [4]. A typical hard phase in cermets has a core/rim structure. Dissolution – reprecipitation is involved in the mechanism for the formation of the core/rim of structure, ruling out other processes, such as spinodal decomposition and diffusion [5,6,7].

2. Experimental conditions

In order to obtain performance cermet cutting tools one has to comply with special compositional requirements including a high content of TiCN (higher then 25 % - 70% of TiCN), 20% up to content of metallic binder Ni , and 10% of WC. In this research we have used $Ti(C_{0.7}N_{0.3})$ from Kennametal, Latrobe – USA, WC from Starck-Goslar, Germany, Ni from Novamet-Wyckoff –NJ, with the following weight ratio: $Ti(C_{0.7}N_{0.3})$:WC:Ni =70:10:20.

The characteristics of starting materials determined by complying with the international standards as presented in table 1.

Table 1

Raw powders characteristics for the starting materials

Material	Flowing rate [s/50g]	Apparent density [g/cm^3]	Particle size FSSS [μm]
$Ti(C_{0.7}N_{0.3})$	not flowing	1.45 ± 0.01	3.4 ± 0.01
WC	not flowing	3.0 ± 0,01	1.87 ± 0.01
Ni	not flowing	2.46± 0,01	4.2 ± 0.01

The samples were prepared by means of a conventional P/M technique; that is, the powders were weighed, then milled with ceramic balls in acetone for 36 hours. These mixture was dried in a vacuum oven for 24 hours. The dried powder was sieved through a 100 mesh (≤ 100 μm) sieve to improve the packing density of the green compacts. In table 2 are presented the physical and mechanical characteristics of the experimental mixtures.

The powder mixtures were, then, binded with paraffin type A (3%), sieved in a semidry state by a sieve with 0,1 mm mesh size and dried, finally obtainig a mixture " ready to be pressed". To evaluate the compact ability, it was necessary to know the specific pressure optimal for the pressing. Specific pressure is comprised between 100-250 MPa according to nature and composition of the powders, geometry and size of the parts, type and the quality of the die.

Table 2

Physical-technological characteristics of cermet type $Ti(C_{0.7}N_{0.3})$ – WC-Ni

Type of material	C_{tot} [%]	C_{lib} [%]	Ni [%]	γ [g/cm³]	HV 50 [Kgf/mm²]	HRA	$\sigma_î$ [Kgf/mm²]	E [Kgf/mm²]
$Ti(C_{0.7}N_{0.3}$WC-Ni	15.05	1.0	19.9	5.90	1850	92.3	75±20	40000

The press executed a bilateral stress on a die of steel with circular section, having the surface of 1 cm^2, respectively on a die of steel with rectangular section (6.26x43) mm^2, for obtaining the green samples. The applied specific pressure was 1250N/mm^2 for the pulverulent mixtures cermet type $Ti(C_{0.7}N_{0.3})$-WC-Ni.

Minimum 15 cylindrical green compacts, respectively 10 specimens from each composition, have been obtained. The average densities of as raw oblates made of experimental mixtures cermet were 6,10 g/cm^3. Binder elimination has been carried out in Siemens-Plania furnace, in atmosphere of hydrogen, using as agent of package alumina previously burned at 1450^0C, during 5 hours, for assume thermic stability. The agent of package was then sieved (< 0.61μm).

The parameters of deliation presintering process were :-atmosphere hydrogen;- temperature of pre-sintering (800-900) ^oC;- maintaining to temperature of pre-sintering: 30 min.;- total time of cycle for binder elimination - presintering: 8h 30min.

Following the operation of binder elimination – presintering, it was found that the established parameters permitted an uniform elimination of binder without cracking. The pressed grades, without binder and presintered were subject to the sinterisation in view to obtain the complex of properties required for the material.

The operation was carriedcoutin a vacuum heating induction furnace, with medium frequency Balzers type currents, the operation parameters being as follows:-sintering temperature: the values were in the limits of (1450^0C-1500^0C); - sintering atmosphere: vacuum;-the holding time at sintering temperature was 60 min.;-total cycle time: 8 h.

3. Results

In order to characterize the sintered compacts the following physical-mechanical characteristics have been determined: HV hardness; density (g/cm^3); transverse fracture resistance (N/mm^2); sintering contraction. In table 3 are presented the characteristics of the sintered compacts obtained from homogeneous mixtures of cermet type powders.[8]

Table 3

Characteristics of sintered cermet type $Ti(C_{0.7}N_{0.3})$ – WC-Ni

Type of material	Density [g/cm^3]	Fracture strength [N/mm^2]	HV $_{50}$	Contraction on the direction of applying the pressing force C_h [%]
$Ti(C_{0.7}N_{0.3})$-WC-Ni cermet grade	6.01	1110	1850	5.485

The structural characteristics were investigated by scanning electron microscopy (SEM). The sintered grades were prepared for metallographic testing in order to: - determine the presence, type and repartition of pores as well as to put in evidence the microstructure. The samples for analyse were examined at the electron microscope at a magnification X2000 (see Fig.s 1). The microstructural analyse was carried out by emphasizing, step by step, the phases by Murakami attack.

In Figs. 1 the morphology of particles of powders from grade type $Ti(C_{0.7}N_{0.3})$-WC-Ni is presented; fine grain powders are emphasized, with relative uniform repartition of compounds. (the ring like form of particles permits to obtain a higher degree of packing for the powder particles). In Fig. 2 a schematic representation of core/rim structure[4] is depicted.

From powder grade $Ti(C_{0.7}N_{0.3})$-WC-Ni cermet type number of 45 sintered cutting tools with different shapes have been produced, that were plated with protective and functional layers type TiC, TiCN. The coatings with extra-hard layers type TiC, TiCN, on a base hard alloy Cermet type were obtained using a new technique PVD using a magnetron sputtering combined with an ionic implant.

Murakami's reagent X2000

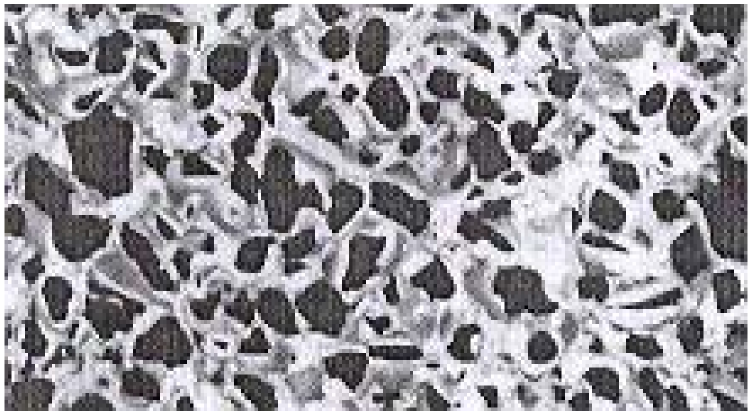


Fig.1. The morphology of powders from $Ti(C_{0.7}N_{0.3})$-WC-Ni

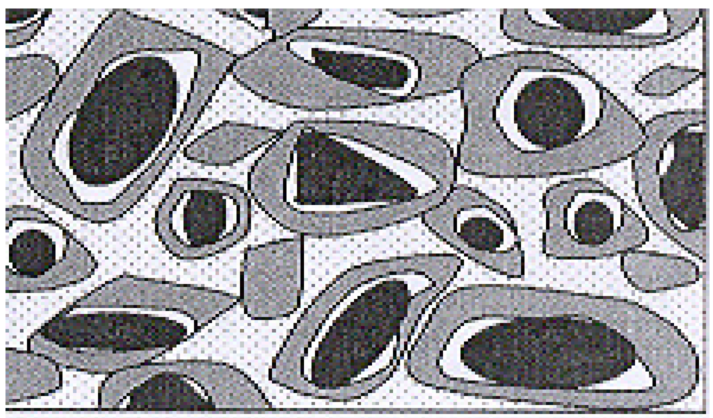


Fig.2. A schematic representation of core/rim structure

The plasma is produced by a magnetron discharge and the procedure can be defined as a Combined Magnetron Sputtering and Ion Implantation (CMSII). The system combines the advantages of hard coating deposition with those of ion implantation. A schematic diagram of the experimental arrangement we have used in the experiments is shown in Fig. 3.[9]

The treatment chamber (1), with water cooled walls, has a diameter of 300 mm and a height of 420 mm. On the bottom plate a circular magnetron (2) with an active target of ~ 40 cm^2 is mounted. A rotating workpiece holder (3) is mounted on the top plate of the chamber by means of a ceramic insulator (4) tested to 150 kV. A thermocouple, which is introduced through the workpiece holder, monitors the temperature of the load during the process.The workpiece holder is connected with the cantilever support (5), which can accommodate four cantilevers (6).

The experiments reported here have been carried out with plain carbon steel foils having a length of 35 mm, a width of 5 mm and a thickness of 0.6 mm or 0.9 mm. A side window (7) allowed the cantilever to be seen from outside when the discharge is on. The bending of the cantilever during the coating deposition was monitored by means of a small telescope (8) positioned at 0.7 m from the axis of the chamber. The precision of this instrument in measuring the deviation δ was 0.05 mm, which represents 1-2% of the typical maximum measured deviation. A protective shield was used to prevent coating of the window.[10]

Typical parameters of the high voltage pulse discharge were U=45kV, τ = 20 μs, ύ = 25 Hz, while the average current density on the magnetron target was 45 mA/cm^2. The deposition method CMSII was successfully used to obtain layers from TiN but it can be used also to obtain multiphase layers is during deposition are used two reactive gases. The two types of reactive layers are introduced into the technological enclosure by a distribution system that has calibrated needle valves, mounted on the way of the two gases so that the flows of the two gases can be precisely controlled and independently.

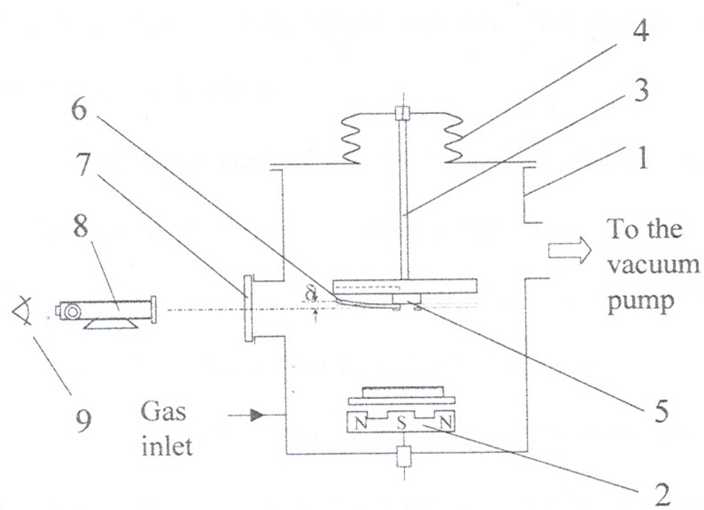


Fig.3. Schematic representation of the experimental setup for in situ measurement of the internal stress within the hard coatings: 1. treatment chamber; 2. magnetron; 3. workpiece holder; 4. ceramic insulator; 5. cantilever support; 6. cantilever; 7. side window; 8. small telescope; 9. observer

The TiC layers were obtained by using as a reactive gas the butane (C_4H_{10}) and to obtain layers of TiCN it was used a gaseous mixture of C_4H_{10} and N_2. The micro-hardness measurements were performed with a metallographic microscope Epytip 2 type having a Hanemann micro-indenter. The pressing loads were of 40 respectively 100 gf (400, respectively 1000mN).The micro-hardness measurements for TiCN layers have indicated the fact that for the layers with structure predominant of Ti_2N the micro-hardness is large, being around 3000 $HV_{0.04}$, in relation to ~ 1700 $HV_{0.04}$ when TiC phase is majoritary. Table 4 presents the micro-hardness values obtained for the three types of layers, the sub-layer material being a cermet compound.

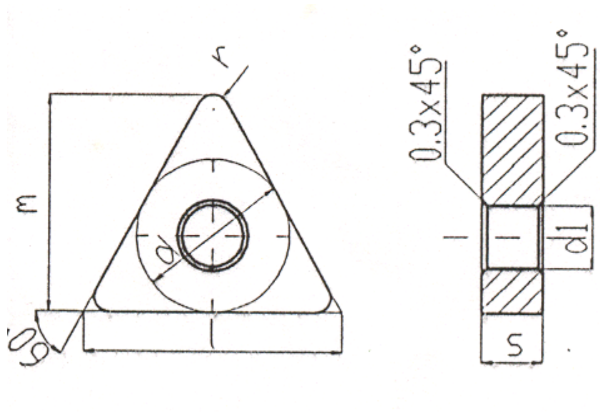
Table 4

The microhardness values obtained for the two types of surface layers

Layer type	$HV_{0.04}$	$HV_{0.1}$
TiC	1800 – 2500	1800 – 2100
TiCN	2200 – 3116	1950 – 2200
Layer material (comparatively)	-	1339-1559

The lower values of microhardness of the layers obtained at 100 gf in respect to those obtained at pressing loads of 40 gf are due to the influence of sub-layer. This influence is obvious at reduced layer thickness and high pressing loads. The layer depths were measured on steel samples that were coated in the same time with the cutting tools.

The measurements were performed by sample sectioning and measuring with an optical microscope. The layers thickness were as follows: TiC layer 6.6 μm ± 0.2; TiCN layer 6.1μm ± 0.1. The inserts selected were TNMA 220412, made of $Ti(C_{0.7}N_{0.3})$-WC-Ni cermet and plated with protective and functional layers type TiC, TiCN. Fig. 4 presents the geometrical characteristics of inserts type TNMA 220412; Fig. 5 shows inserts tools coated with different layers by CMSII method.



L=22;
S=4.76 ± 0.025
D= 12.70± 0.05
D_1 =5.16± 0.025
M= 17.859 ± 0.013
R=1.2

Fig.4. Geometrical characteristics of inserts type TNMA 220412

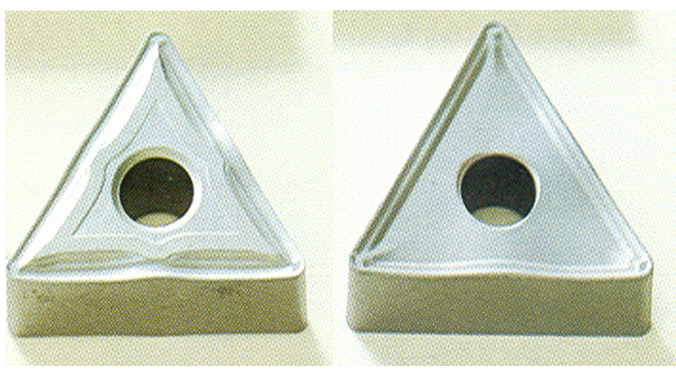


Fig.5. Inserts tools coated with different layers by CMSII method

4. Conclusions

The experiments lead to the following conclusions:

- the selected systems of ceramic -metallic materials were characterized from physical-mechanical, chemical and structural point of view in accordance to standards specific to powder metallurgy;
- the experiments allowed to determine the factors having significant influences on the characteristics of $Ti(C_{0.7}N_{0.3})$-WC-Ni cermet type materials with matrix role, i.e.: forming parameters; composition of

gaseous environments as well as of thermal and time parameters of heat treatment processes;

- from powder grade $Ti(C_{0.7}N_{0.3})$-WC-Ni cermet type have been obtained sintered cutting tools with different shapes, that were plated with protective and functional layers type TiC, TiCN;.
- the coatings with extra-hard layers type TiC, TiCN, on a base of $Ti(C_{0.7}N_{0.3})$-WC-Ni material type were obtained using a new technique PVD supposing a magnetron sputtering combined with an ionic implant;
- the microhardness measurements for TiCN layers indicated the fact that for the layers with structure predominant of Ti_2N the micro-hardness is large, being around 3000 $HV_{0.04}$, in relation to ~ 1700 $HV_{0.04}$ when TiC phase is major;
- the more reduced values of micro-hardness of layers obtained at 100 gf in respect to those obtained et pressing loads of 40 gf are due to the influence of sub-layer. This influence is obvious at reduced layer thickness and high pressing loads;
- the layers thickness were as follows: TiC layer 6.6 μm ± 0.2; TiCN layer 6.1μm ± 0.1;
- the cermet type powder mixtures investigated in this paper are recommended to be used to manufacture cutting tools for automated machines, at very high cutting rates, in conditions of dried processing.

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