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MANUFACTURED PARTS OF CERMET MATERIALS TYPE Ti ($C_{0.7}N_{0.3}$)-WC-Ni USED IN THE EXTRACTIVE INDUSTRY

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Sunt prezentate rezultatele experimentarilor preliminare de obţinere a unor amestecuri omogene de cermeţi din sistemul Ti($C_{0.7}N_{0.3}$)-WC-Ni prin procedee de formare în stare plastică şi anume prin procedeul de formare prin injecţie a pulberilor. Aliajele dure sinterizate pe baza de carbură de titan şi carbură de wolfram sunt cunoscute în special pentru larga lor aplicabilitate datorată în special proprietăţilor remarcabile de rezistenţă la uzură, rezistenţă la oxidare şi coroziune pe care acestea le posedă. Lucrarea prezintă cercetările experimentale de realizare a acestor materiale prin tehnici specifice metalurgiei pulberilor precum şi caracteristicile fizico-chimice, mecanice şi structurale ale reperelor sinterizate realizate din amestecuri de aliaje dure tip Cermet.

This paper presents some preliminary experimental results for the obtaining of homogenous mixtures based on cermets from Ti($C_{0.7}N_{0.3}$)-WC-Ni systems by powders injection molding technique. Hard metal sintered alloys based on titanium carbides and tungsten carbides are known for their wide applicability because of remarkable properties of high wear, oxidation and corrosion resistance.

The paper presents the experimental researches concerning the correlation between physical-mechanical and structural characteristics of these types of materials and the technological parameters.

Keyworks: cermet materials, powder injection molding, feedstock

1. Introduction

The service stresses specific for extremely complex mechanical cutting operations, gave a new impulse to the elaboration of new materials using and developing advanced technologies that permit the achievement of products with increased characteristics. These materials must comply simultaneously with high values: *hardness (wear resistance) - toughness (bending resistance) - cutting rate,*

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and shock resistance, and also corrosion and high temperature oxidation properties. High temperatures are specific to the "in-service" condition of these parts.

Cermets are materials with high resistance at high temperatures, with special resistance to corrosion in acid environment and with good wear resistance. They possess an ensemble of physical-mechanical properties that permit to be used in dissimilar area in extreme service condition. TiCN based cermets are dense and hard materials, but also important components of high-speed cutting tools [1, 2].

A comparison of the cutting performance of WC-Co conventional tool materials with that of the cermets reveals that cermets provide improved surface finishing excellent chip and tolerance control and offer geometric accuracy in the work pieces [3].

All of these characteristics are due to mechanical properties of hard phases that are found in cermets [4]. A typical hard phase within cermets has a core/rim structure. Dissolution-reprecipitation is involved in the core/rim formation mechanism ruling out other processes, such as spinodal decomposition and diffusion [5-7].

Powder injection molding (PIM) is a fast growing manufacturing method that bridges the manufacturing gap that other metal working technologies and shaping processes cannot do because of technology or cost. Metal injection molding (MIM) can be a very cost effective method for producing large quantities of precision parts [8].

The powder injection molding process which was firstly applied to plastic materials and later in the ceramic industry is now studied with the aim of obtaining cermets sintered products having an complex geometry.

The powder injection molding process consists of four basic stages: feedstock obtaining, injection molding, binder removal or debinding and sintering. Although each stage seems to be individual they are in some ways related, and the knowledge of each step is necessary to understand the whole process [9].

MIM advantages are:

- high shape complexity: the MIM process can produce more complex parts than either investment casting or traditional press and sinter techniques;
- low cost: machining operations can be eliminated;
- tight tolerances: dimensions of parts produced with our feedstock can be held to $\pm 0.001 \div 0.002$ inch/inch;
- high density: MIM parts produced by these techniques will have density levels between 97.5 – 99.5 %; these levels typically exceed traditional press and sinter techniques;

- high performances: the tensile strengths, elongations and hardness are superior to traditional press and sinter techniques, and comparable to investment casting or machined components [10].

2. Experimental conditions

In order to achieve the desired performance composition with high TiCN content (>25% TiCN) have been prepared. Actually we have investigated a composition containing 70% TiCN + 10% Ni binder + 10% WC powder. As raw materials we used $Ti(C_{0.7}N_{0.3})$ powder from Kennametal, Latrobe-USA, WC powder from Starck-Goslar ,Germany, Ni powder from Novamet-Wyckoff-NJ, in the following weight ratio: $Ti(C_{0.7}N_{0.3})$:WC:Ni = 70:10:20.

Powders characteristics were determined according to International Standards: SR EN 23923-1:1998, ISO 3923-1-Metallic powders – Determination of apparent density - Part 1: Funnel method.; SR EN ISO 4490:2003, Metallic powders - Determination of flow ability by means of a calibrated funnel (Hall flow meter). The particle size was determinated by using Fisher sub-sieve size (FSSS) method.

The characteristics of raw powders comply with requirements of the international standards are presented in Table 1.

Table 1

Raw powders characteristics

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

Samples were prepared by conventional P/M technique, i.e. powders were weighed, and then milled with ceramic balls in acetone medium for 36 hours. The mixture was dried in a vacuum oven for 24 hours. Dried powder was sieved through a 100 mesh (≤ 100 μm) sieve to improve the packing density of the green compacts. Table 2 are shows the physical - mechanical characteristics of the experimental mixture.

Table 2

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

Material type	C_{tot} [%]	C_{free} [%]	Ni [%]	Apparent density, [g/cm^3]	Particle size FSSS, [μm]
$Ti(C_{0.7}N_{0.3})$ WC-Ni	15.05	1.0	19.9	5.95± 0.01	5.9 ± 0.01

The powder mixture was binded with paraffin type A (3%), then sieved in semidry state with a sieve with 0,1 mm size and dried. Finally, a mixture “ready to be pressed” was obtained.

For feedstock obtaining a binder system was used which contains paraffin, bees wax and oleic acid (table 3).

Table 3

The characteristics of binder components

Binder component	Density g/cm^3	Melting temperature ° C
Paraffin	0.90	50-54
Bees wax	0.94-0.97	60-66
Oleic acid	1.2	14

The parameters of the feedstock prteparation (mixing – homogenizing) were as follows:

- work temperature: 70-80° C;
- time: 6 hours;

3. Results

For injection molding the volume fraction of powder in the feedstock based on $Ti(C_{0.7}N_{0.3})$ WC-Ni cermets powder and selected binder system was 0.40 according to 8% weight binder.

This feedstock presented at the injection molding temperature suitable rheological characteristics (low viscosity ≤ 2000 Pa.s) make possible the uniform filling of the dies cavities [8].

Parts having simple and complex shapes were produced by the injection molding process. The injection pressure was between 2-8 bar and the injection time was 5-20 s.

The injection temperature was about 70 °C (± 10 °C).

Thermal debinding was applied for the binder removal from injection molded parts, the main aim being the setup of an optimum debinding – presintering cycle. Debinding cycles were performed in vacuum or hydrogen atmosphere in the presence or absence of packing medium (alumina).

For the setup of debinding – presintering parameters the results of the differential thermal analysis were used. The conditions of measurements by differential analysis were:

- sample weight: 0.8 - 2.8 g;
- used medium: Ar; air;
- temperature range: 500-1000 °C;
- heating rate: 5 ° C/min… 10 ° C/ min;

The parameters of the debinding – presintering process which allowed the shape presentation of the parts and also a slow and gradual binder removal were as follows: -atmosphere: hydrogen (flow: 200 l/h); - packing medium: alumina; - average heating rate up to 500 °C: 0,5 °C/min; - average heating rate to 500 - 900 °C: 10 °C/min; - presintering temperature: 900 °C;

It was found that the established parameters permitted a uniform elimination of binder without cracking of the parts.

The presintered pressed grades without binder were subject to the sintering process in order to obtain the complex properties required for the material.

The operation was realized in a vacuum induction heating furnace type Balzers, with intermediate frequency currents, the operation parameters being:

- sintering temperature: the values were in the limits of 1400 °C-1450 °C;
- the holding time at sintering temperature was: 60 min;
- total cycle time: 8 hours;
- packing medium: alumina.

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 analysis were examined at a magnification X2000 (fig. 1).

The micro-structural analysis was carried out by emphasizing, step by step, the phases by Murakami attack.

In Fig. 1 the morphology of powder particles from grade type $Ti(C_{0.7}N_{0.3})$ WC-Ni is presented; fine grain powders are put in evidence with relative uniform repartition of compounds (the rounded particles permits to realize a higher degree of packing of powder particles).

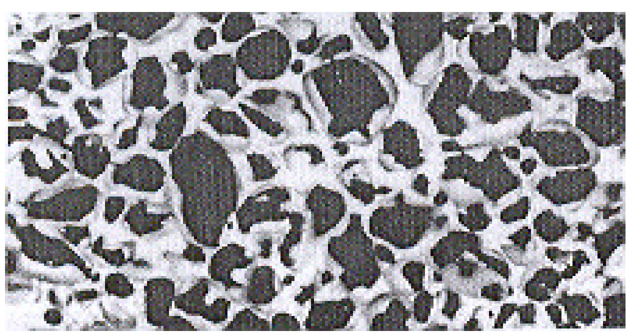

Fig. 1. The morphology of powders for $Ti(C_{0.7}N_{0.3})$ WC-Ni;
Murakami's reagent X2000

In order to characterize the sintered grades the following physical-mechanical characteristics were measured: hardness HV; density (g/cm^3); transversal fracture resistance (N/mm^2); sintering contraction. In table 4 are presented the characteristics of the sintered grades manufactured from homogeneous sintered mixtures of cermet type powders [8].

Table 4

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

Material type	Density [g/cm^3]	Fracture strength [N/mm^2]	HV_{50}	Contraction along the direction of applied pressing force C_h [%]
Ti($C_{0.7}N_{0.3}$)-WC-Ni cermet grade	6.01	1110	1850	5.485

In Fig. 2 some manufactured parts used in the extractive industry, that we have obtained by powder injection molding are presented [11].

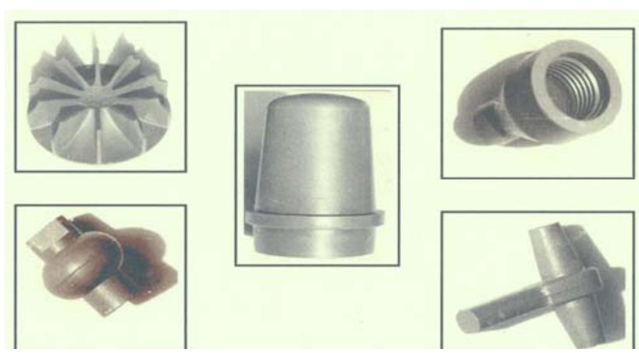

Fig. 2**.** Some manufactured parts realized by powder injection molding, used in the extractive industry [4]

4. Conclusions

The experiments lead to the following conclusions:

●The rheological characteristics of the feedstock for a uniform filling of the die’s cavities were suitable for a volume fraction of powder of 0.40 at a temperature of 70 °C (± 10 °C);

●The results of the differential thermal analysis were necessary to setup the parameters of the debinding – presintering process;

●The presence of the packing medium (alumina) allowed a gradual debinding with the retaining of the parts shapes;

●The low heating rates 0.5 °C/min also allowed a slow and gradual binder removal without holes or cracks appearance;

●The experiments allowed to determine the factors that have significant influences on the characteristics of Cermets materials with matrix role, i.e.: forming parameters; composition of gaseous environments, as well as of thermal and time parameters of heat treatment processes;
●The values of the physical – mechanical characteristics of the analyzed samples were in accordance with those specified in the supplier's control certificates which allowed the obtaining of maximum results in industrial exploitation conditions of the products.

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