Properties of hot-pressed sinters obtained from Fe, Cu and Ni powders

Właściwości spieków otrzymanych techniką prasowania na gorąco z proszków Fe, Cu i Ni

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The results of studies upon properties of sinter obtained from elementary mixtures of iron, copper and nickel powders have been subjected to grinding in a ball mill for 60 hrs. The sinters have been made by hot-pressing technique in graphite matrix. Their studies included density and hardness measurements and static tensile test. Electron microscope has been also applied to observe the microstructure and fracture surfaces of broken samples. The research has been aimed at determining the suitability of manufactured composites for the production of metallic-diamond tools. Their properties have been compared with those of sinter produced from a commercial mixture of powders.

KEYWORDS: diamond tools, powder mixture, sinter, matrix

Progress in the production of tools for cutting, drilling and machining of materials depends on the development of powder metallurgy technology and development of synthetic diamond production on an industrial scale. In the selection of the tool, properties of the workpiece and the machining conditions should be considered. Workpieces for saws, drills, milling cutters and grinding wheels are sinters of cobalt with diamond.

Technically, cobalt has many advantages [1–4]. Sintered cobalt alloys are characterized by high strength and good plasticity. Cobalt matrix exhibits very good retention properties [5–7]. The disadvantage of cobalt is its high and unstable price. For this reason, tool manufacturers are looking for cheaper matrix materials that could replace cobalt [7–9].

For the manufacture of metallic-diamond tools, mixtures of cheaper elemental powders, carbonyl iron powder or reduced iron with copper or bronze were used. Research has shown that the sintered materials have a coarse and non-homogeneous structure, which results in a deterioration of their strength properties [5]. The fine particle microstructure of the sinter is a precursor to the microstructure of the powder particles, which inhibits grain growth during sintering [10].

The main purpose of the study was to investigate the suitability of Fe, Cu and Ni powder mixture in a ball mill for the production of sintered metal-diamond composites. The influence of process parameters on microstructure

and mechanical properties of sinters was investigated. These properties were compared with the properties of sinter produced from a commercial mixture of powders.

Methodology and research results

Sinters achieved by means of hot pressing of mixtures of elementary iron, copper and nickel powder milled in a ball mill for 60 hrs, was used for the tests. Mixtures contained:

- reduced iron powder NC 100.24 (Höganäs) with a particle size of 20÷180 μm,
- CH-L 10 electrolytic copper powder (ECKA) with particle size <45 µm,
- carbonyl nickel powder T255 (Vale) with Fisher's replacement diameter of 2.4 μm.

The shape and composition of the powder particles used for the tests are shown in figs. 1*a*-*c*.

Prior to consolidation, the powders were mixed in the appropriate proportions for 30 min in a Turbula mixer. The mass fraction of the individual powders was 60% Fe, 28% Cu and 12% Ni. Subsequently, the powder was ground in an RJM-102 ball mill made by EnviSense, in an air atmosphere, in a container filled with 50% balls of 12 mm diameter made of 100Cr6 steel. The mass ratio of the balls to the powder ground was 10:1. The rotational speed of the drum was about 70% of the critical speed. The shape and particle size of the powders after grinding are shown in fig. 2.

The hot pressing process was carried out in a graphite matrix under nitrogen, using the CAR100 press of the Italian company ARGA. The pressing temperature was chosen so as to obtain sinters with a porosity of no more than 5%. The powder was held for 3 min at a maximum temperature of 900°C and a maximum pressure of 35 MPa.

For comparative purposes, sinter from a commercial powder mixture of 33% Fe, 33% Cu and 33% Ni was produced. Pressing was done at 850 °C and the other parameters were the same as for 60% Fe, 28% Cu and 12% Ni sinter. The hot pressing parameters recommended by the manufacturer were used.

The obtained sinter was subjected to density measurements using a WPA120 hydrostatic balance – according to PN-EN ISO 2738:2001 standard. On the basis of density measurements, the porosity of tested sinters (table I) was determined.

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Fig. 1. Photographs of powders used for tests: a) Fe, b) Cu, c) Ni

Strength tests were carried out on a universal strength machine type INSTRON 4502. The traverse speed was set at 0.5 mm/min. Diameter in the measuring part was 3.5 mm and the elongation of the specimens was recorded using a 10 mm extensometer. Based on the recorded data, the calculated yield strength $R_{0,2}$, tensile strength R_m and relative elongation ε were calculated. The results of the static tensile test and the stretching curves of the tested sinter are presented in table II and fig. 3.

Subsequent measurements of the elasticity constants were carried out by acoustic method and the

hardness of sinters was determined according to Vickers at a load of 10 kG. The results of measurements are shown in table III.



Fig. 2. Mixture of Fe, Cu and Ni after grinding for 60 h

The retention properties of the matrix are directly related to its thermal expansion. Knowledge of the coefficient of thermal expansion of the matrix is needed, among others, to simulate the diamond particle retention in the matrix [5, 6]. Dependence of this coefficient on the temperature (within the range of hot pressing) was determined by the dilatometric method using a Netzsch dilatometer. The dilatometric curve for 60Fe28Cu12Ni sinter is shown in fig. 4.

From the dilatometric curve, the coefficient of thermal expansion α was determined (fig. 5). Comparison of coefficients for both sintered plates is included in table IV.

Subsequently, the stretched samples were subjected to fractographic and structural analysis – the JSM-7100F electronic scanning microscope integrated into the X-Max-AZtec XDS microanalysis system from Oxford Instruments was applied for this purpose.

Sinter	Density, g/cm ³	Theoretical density, g/cm ³	Porosity, %
60Fe28Cu12Ni	8.07 ±0.03	8.25	2.18 ±0.35
33Fe33Cu33Ni	8.18 ±0.02	8.55	4.33 ±0.23

TABLE I. Density and porosity of tested sinters

TABLE II. Strength parameters of sinters

Sinter	Yield strength limit <i>R</i> _{0.2} , MPa	Tensile strength <i>R</i> _m , MPa	Relative elongation <i>ε</i> , %
60Fe28Cu12Ni	355 ±15	716.6 ±16.7	9.35 ±0.45
33Fe33Cu33Ni	223 ±25	539.4 ±4.5	2.68 ±0.08

A representative fracture surface with a distinctly ductile character is shown in fig. 6 and the microstructure with the sintering phases is shown in fig. 7. Chemical composition of the marked sintering phases is given in table V.



Fig. 3. Stress curves of tested sinters

TABLE III. Parameters of elasticity and hardness

Sinter	Modulus of elasticity <i>E</i> , GPa	Poisson ratio v	Hardness HV10
60Fe28Cu12Ni	163	0.32	290.5 ±9.3
33Fe33Cu33Ni	164	0.32	208.0 ±17.0



Fig. 4. Dilatometric curve for 60Fe28Cu12Ni sinter

TABLE IV. Comparison of thermal expansion coefficients

Sinter	Temperature	Thermal expansion coefficient, K ⁻¹
60Fe28Cu12Ni	average for 900–100°C	19.78 · 10 ⁻⁶
60Fe28Cu12Ni	100°C	17.10 · 10 ⁻⁶
33Fe33Cu33Ni	average for 850–100°C	15.54 · 10 ⁻⁶
33Fe33Cu33Ni	100°C	13.95 · 10 ⁻⁶



Fig. 5. Thermal expansion coefficient for sinter 60Fe28Cu12Ni



Fig. 6. Fracture surface of sinter 60Fe28Cu12Ni



Fig. 7. Microstructure of sinter 60Fe28Cu12Ni

TABLE	V.	Chemical	compositi	on of	phases	in	sinter
60Fe28	Cu	12Ni					

Phase of sin- tering*	Chemical composition, % of weight			
	0	Fe	Cu	Ni
1		11,66	84,25	4,08
2		91,59	3,78	4,63
3	17,48	75,53	4,08	2,91

* According to designations in fig. 7.

Discussion of results and conclusions

In this paper, research was conducted to produce sinters that are made from inexpensive iron-based powders, as well as to assess their potential for the production of sintered diamond tools. The adopted hot pressing parameters enabled high density sintering, close to the theoretical density (table I).

It has been shown that the ground powders can be consolidated into a nearly pore-free state by hot pressing at 900°C. Sinter density varied between 8.05 g/cm³ and 8.11 g/cm³. As shown in table III, Fe-Ni-Cu alloy has a high hardness (290.5 HV10), tensile strength (716.6 MPa) and yield strength (355 ±15 MPa) with a maximum elongation of 9.35% (table II).

The fractographic studies show that the fracture surfaces of all stretched samples are of a dimpled, ductile character. An analysis of chemical composition of EDS, performed on metallographic extrusions made on sintered grits produced from grinding powders, revealed a complex multi-phase microstructure of sinters. It confirmed the presence of: Fe – 42÷52%, Cu – 48÷53%, Ni – 2÷3% by weight. As it can be seen in the picture of the microstructure (fig. 6), the alloy consists of a solution (α -Fe), solution of copper (Cu) – which is a solid solution of Fe and Ni in Cu – and mixtures of oxides (dark fields in the picture).

Conclusions

The material obtained from the ground powder for 60 hrs is very well sintered at 900°C. It is characterized by fine-grained microstructure with low chemical heterogeneity and high hardness and tensile strength. Addition of 12% nickel (by weight) to the alloy, significantly improved the hardness and strength of the alloy at the expense of its plasticity. The alloy properties can be modified by varying the chemical composition and grinding time.

Tested 60Fe28Cu12Ni sinter has better mechanical properties as compared to 33Fe33Cu33Ni sinter (table II and table III) and other iron-based sintering compounds containing Cu [5], but it is less effective than cobalt sinters [6].

The 60Fe28Cu12Ni sinter is remarkable due to its affordability, ease of consolidation through hot pressing and the ability to change over a wide range of strength and plastic properties. This material is characterized by high coefficient of thermal expansion (table IV), so that it has good retention properties, i.e. it holds the diamond particles well on the metallic-diamond work surface.

The coefficient of thermal expansion of the 60Fe28Cu12Ni sinter is significantly higher than that for 33Fe33Cu33Ni sinter (table IV) and greater than the cobalt expansion factor [6], which has good retention properties. This feature allows to consider the material being studied as a matrix in the manufacture of metal-diamond tools.

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