Characterization of Fe-Cu-Ni composites sintered by hot-press

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Abstract. The main objective of the work was to determine the effects of powder composition on a microstructure and properties of iron-base materials used as matrices in diamond impregnated tools. Mixed powders and 30 hours Fe-Cu-Ni milled powders were used for the experiments. The influence of manufacturing process parameters on microstructure and mechanical properties of sinters was investigated. Sintering was performed in a graphite mould using a hot-pressing technique. The powders were consolidated to a virtually pore-free condition during 3 min holding under 35 MPa and at 900°C. The investigations included: density, hardness, static tensile test and X-ray diffraction (XRD) analysis. Observations were also made of microstructure and fracture surfaces of broken samples using a scanning electron microscope (SEM) were also conducted. The SEM observations revealed an evident dependence of grain size and microstructural homogeneity on milling time. The obtained test results indicate that the sinters are virtually densified, have good plasticity with relatively high hardness and yield strength, but sinters of non-ground powders are characterized by a coarse-grained microstructure.

1. Introduction

Cutting tools, such as circular and wire saws, drills, milling cutters or grinding wheels are traditionally fabricated by powder metallurgy (PM) consolidation of diamond-cobalt mixtures [1,2]. The progress in the tools manufacturing routes, using PM techniques, resulted in developing diamond grit impregnated saw blades, which were employed around 1940. Metal bonded diamond composites were applied after World War II [3-5].

A tool selection depends both on properties of a workpiece (its hardness and abrasion resistance) and cutting conditions (linear speed, cooling efficiency and cutting rate, etc.). Such factors affect both the tool geometry as well as the composition and structure of diamond segments, which allows engineers to design a tool with a desired shape and diamond segments with an appropriate form and composition. The lifespan of diamond-impregnated segments primarily depends on diamond crystals retention of and tribological properties of the metal matrix [6,7].

Recent advanced development in cutting tools manufacturing are due the progress in PM technology, but an increasing industrial production of synthetic diamond has also been of prime importance. China is the largest manufacturer of synthetic diamond in the world. In 2010, China’s production exceeded of 7 billion carats. The annual average increase rate has reached nearly 20% for the last 10 years. It
accounts for about 90% of the world’s production. China’s diamond is being exported to 58 countries and regions [8].

The best and the most common metal bond material is cobalt. It has been widely used in diamond tools for several decades. Cobalt combines good chemical compatibility with diamond at the processing temperatures, an excellent diamond retention, associated with a satisfactory wear resistance after some cutting operations. One of the major shortcomings of cobalt, however, is its high and unstable price, which has significantly affected tool production costs since the beginning of the new millennium, when diamond became a commodity product [9]. Nevertheless, cobalt is a strategic metal – produced only by few countries – and therefore cobalt is no longer the best choice for some applications. In addition, cobalt is toxic substance.

Significant changes in the cost of raw materials as well as a relative decrease in the other production costs have forced toolmakers to find cheaper alternatives [10].

Considering all the negative aspects, researchers have recently proposed new iron-based alloys which could be used as a matrix in diamond tools [11].

The main objective of the study was to determine the suitability of ball-milled Fe-Cu-Ni powder mixtures for fabrication of diamond-impregnated metal-matrix composites. The combined effects of chemical composition, powder milling conditions and sintering parameters on the as-consolidated microstructure and mechanical properties were studied. The obtained results were compared with properties of a hot pressed SMS (sub-micron size) grade cobalt powder.

2. Experimental procedure

The experimental powder mixture was made from:

- Höganäs NC100.24 grade, carbon-reduced iron powder (20-180 μm)
- ECKA CH-L10 grade, electrolytic copper powder (<45 μm)
- Vale T255 grade, carbonyl nickel powder (Fisher Sub-Sieve Size = 2.4 μm).

Morphologies of the starting powders are shown in figure 1.

![Figure 1](image1.png)

**Figure 1.** Experimental powders: (a) NC100.24; (b) CH-L10 and (c) T255.
Prior to consolidation, the powder mixture containing 60% Fe, 28% Cu and 12% Ni was prepared by blending the constituent powders in a Turbula-type mixer for 30 minutes. Then, the powders were ball-milled for 30 hours in ethyl alcohol with a small amount of glycerol using the EnviSense RJM-102 laboratory mill. The milling vial was filled to half of its volume with 12 mm diameter 100Cr6 steel balls. The ball-to-powder weight ratio was 10:1. The milling vial was set to run at 150 rpm, which is about 70% of the critical rotational speed. The particle shape of as-mixed and the ball-milled powder is shown in figure 2.

![SEM micrograph of the particle shapes of a) mixed powders and b) the 30-hour ball milled powders.](image)

The mixed powders and ball-milled powders were tested for their particle size distribution, by means of the HELOS (H2769) & RODOS laser particle sizer with WINDOX 5 software enabling the measurement of particle sizes in the range of 0.1 μm-2000 μm. The results are shown in figure 3.

![Particle size distribution of the starting mixed powders and ball milled for 30 h.](image)

The primary mixture and the ball-milled powders were subjected to consolidation by hot pressing in a graphite mould. The hot pressing process was performed in the ARGA CAR1001 hot press furnace in nitrogen. The pressing temperature was properly selected to obtain the as-sintered porosity lower than 3%. Hence, the powder was held at 900°C and 35 MPa for 3 minutes.

The sinters made from the mixed and the ball-milled powders were subjected to an X-ray phase analysis using a PANalytical Empyrean X-ray diffractometer using a copper anode lamp (Cu Kα1 = 1.5406 Å). The identified phases of the samples are shown in figure 4.

The sintered specimens were first tested for density and hardness. The density measurements involved weighing the specimens in air and water using the WPA120 hydrostatic weighing system,
The results were also used to assess the as-sintered porosity. The hardness of the sinters was measured using the Vickers method for the load of 10 kG. The results are summarized in table 1.

| Fe-Cu-Ni material | Density [g/cm³] | Theoretical density [g/cm³] | Porosity [%] | HV10  |
|-------------------|----------------|-----------------------------|--------------|-------|
| Mixed powders     | 7.82 ±0.03     | 8.25                        | 5.18 ± 0.20  | 157.6 ± 12.1 |
| Milled for 30 h   | 8.10 ± 0.02    | 8.25                        | 1.79 ± 0.25  | 300.9 ± 16.3 |

scatter intervals estimated at 90% confidence level

The specimens were then machined, by turning, to produce non-standard specimens for static tensile tests. The tensile strength tests were carried out using the INSTRON 4502 universal testing machine. The diameter of the gage section of specimens was 3.5 mm. The cross head speed was set to 0.5 mm/min. The specimen elongation was registered by means of an extensometer with a gauge length of 10 mm. The results were then used to calculate the offset yield strength R₀.₂, ultimate tensile strength Rₘ, and elongation ε. The results of the static tensile strength test and a typical stress-strain curve are given in table 2 and plotted in figure 5, respectively.

| Stress [MPa] | 0h | 30h |
|--------------|----|-----|
| 0%           | 0  | 0   |
| 5%           | 5  | 10  |
| 10%          | 10 | 20  |
| 15%          | 15 | 30  |

Figure 4. X-ray spectrum of the sinters obtained from (a) the mixed powders, (b) the ball milled powders.

Table 1. Density, porosity and hardness.

Figure 5. Stress-strain curve.
Table 2. Static tensile test results\textsuperscript{a}.

| Fe-Cu-Ni material          | Offset yield strength R\textsubscript{0.2} [MPa] | Ultimate tensile strength R\textsubscript{m} [MPa] | Elongation \(\varepsilon\) [%] |
|---------------------------|-----------------------------------------------|-----------------------------------------------|----------------------------------|
| Mixed powders             | 137 ±10                                       | 396.9 ±7.5                                    | 10.0 ±0.20                      |
| Milling for 30 h          | 229 ±15                                       | 634.9 ±13.3                                   | 12.1 ±0.45                      |

\textsuperscript{a}scatter intervals estimated at 90% confidence level

The fractured specimens were examined fractographically using the JSM-7100F scanning electron microscope fitted with an OXFORD INSTRUMENTS X-Max-AZtec EDS system. The grip sections of tensile specimens were also used to produce metallographic specimens for microstructural observations. A typical fracture surface and microstructure are shown in figure 6(a) and figure 6(b), respectively.

![Figure 6. Typical fracture surface after a tensile strength test obtained for (a) the mixed powders and (b) the milled powders.](image)

The microstructures of the experimental alloys were examined and analyzed by means of a SEM fitted with an energy dispersive spectroscopy (EDS) system (figures 7(a) and 7(b)). Table 3 presents chemical compositions obtained from the EDS analysis in micro-areas indicated in figure 7.

![Figure 7. Microstructure of the sintered Fe-Cu-Ni material, (a) mixed powders and (b) ball-milled powders.](image)
The objective of the study was to fabricate sintered materials using inexpensive iron-based powders and to assess their potential application as a sintered matrix in diamond impregnated tools. The hot-press parameters were carefully selected to obtain the as-sintered density approaching the nearly full density (table 1). It was found that the mixed and ball-milled powders could be consolidated to a virtually pore-free state by the hot press route at the temperature of 900°C during 3 h under the pressure of 35 MPa. During milling, the powder particles are repeatedly flattened, work-hardened and fractured, and welded together. After 30-hour ball milling, they became flaky in shape (figure 2).

After consolidation, the alloys possess high hardness, which considerably increases during milling. The ball-milled material shows a good combination of high strength and ductility. The obtained test results indicate that the produced sinters have roughly theoretical density, good plasticity with relatively high hardness and yield strength, but sinters premixed are characterized by a coarse-grained microstructure.

Nevertheless, the microscopic examination of the metallographic sections revealed marked differences in microstructural inhomogeneity of the tested specimens. The ones manufactured from the mixed powders showed a highly inhomogeneous distribution of structural components. After milling, a strong grain refinement was discovered. The sintered microstructure of the material made of the milled powder for 30 h was generally fine sized (figure 7(b)). Both the ground and unground material revealed the presence of following phases: solid solution-type Fe, Cu, and oxides.

The XRD analysis showed that the α-Fe peaks of the sinters obtained from the milled powders disappeared. There are lines indicating the occurrence of γ-Fe iron which are connected with copper lines. The sinters obtained from milled powders revealed weak lines of FeO (II) oxide. However, there is no line of Ni, which indicates the diffusion of Ni atoms to Fe and Cu phases (figure 4).

The fractographic examination of the sinters produced from the milled powders showed ductile dimpled fracture surfaces. Whereas, for the specimens obtained from the mixed powders the fracture surfaces varied, with ductile fracture being predominant (figure 6).

The addition of nickel and copper to iron resulted in high hardness and tensile strength of the alloy and acceptable ductility.

4. Conclusion
The experimental data imply that the material obtained from the powders ball-milled for 30 h and hot-pressed at 900°C is characterized by a fine-grained microstructure and inhomogeneous microstructure. It should be emphasized that although the analysed material shows slightly lower strength and ductility as compared with cobalt [9-11], its hardness and offset yield strength are sufficiently high to meet the criteria for less demanding applications, such as professional general-purpose tools.

Undoubtedly, the tested Fe-Cu-Ni material deserves further attention because of its attractive price and the ease of consolidation by hot-pressing. Presumably its strength and plastic properties can be improved by fine tuning its chemical and powder composition as well as the powder processing and consolidation conditions.

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