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# PROCESSING AND CHARACTERIZATION OF Fe-Cu-Ni SINTERS PREPARED BY BALL MILLING AND HOT PRESSING

The main objective of the present work was to determine the effect of powder composition on microstructure and properties of iron-base materials used as matrices in diamond impregnated tools. The Fe-Cu-Ni powders premixed and ball-milled for 30 hours, were used for the experiments. The influence of manufacturing process parameters on microstructure and mechanical properties of produced sinters was investigated. Sintering was done by hot-pressing technique in graphite mould. The powders were consolidated to a virtually pore-free condition during 3 minutes hold at 35MPa and 900°C. Investigations of the sintered materials included: density, hardness, static tensile test and X-ray diffraction (XRD) analysis. Microstructural and fractographic observations were also made with a scanning electron microscope (SEM). The obtained results indicate that the sintered parts have a high density, close to the theoretical value, good plasticity, relatively high hardness and yield strength, and are characterized by a coarse-grained microstructure.

*Keywords:* matrix, diamond, hot pressing, diamond tools

## 1. Introduction

The cutting elements of tools, such as circular and wire saws, drills, milling cutters or grinding wheels have been traditionally fabricated by powder metallurgy (PM) consolidation of diamond-cobalt mixtures [1,2]. The first diamond circular saw blades for cutting stone were developed by Felix Fromholt in France in 1885. Progress in the tool manufacturing routes, by making use of PM techniques, resulted in developing diamond grit impregnated saw blades, which were put into operation around 1940. Metal bonded diamond composites were applied after World War II [3-5].

The tool selection depends on both properties of the workpiece material (its hardness and abrasivity) and cutting conditions (linear speed, cooling efficiency and cutting rate, etc.). These factors affect both the tool geometry as well as the composition and structure of diamond segments. The knowledge of these factors allows engineers to design a tool with a desired shape and diamond segments with an appropriate form, structure and composition.

The service life of diamond-impregnated segments is primarily dependent on the retention of diamond crystals and tribological properties of the metal bond (matrix). It is essential that the metal bond material should wear along with the diamond grits to induce self-sharpening of segments [6,7].

The recent advances in the manufacturing of cutting tools have resulted from the progress in PM technologies and sharp increase in industrial production of synthetic diamond has also been of great importance. In recent years China has become the biggest producer of synthetic diamond in the world. In 2010 China's production exceeded 7 billion carats. The yearly average increase rate has reached nearly 20% for the last 10 years. China's diamond accounts for over 90% of the world's production. It is exported to 58 countries [8].

The best and 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, excellent diamond retention, and satisfactory wear resistance. The major shortcoming of cobalt, however, is its high and unstable price, which has increasingly contributed to the tool production costs since the beginning of the new millennium, when diamond became a commodity product [9]. Cobalt is also a strategic metal because only a few countries produces it. Therefore it is no longer the first choice in many diamond tools' applications. Besides its price, cobalt may also cause toxicity problems.

Significant changes in the cost of raw materials as well as a relative decrease in the other production costs have exerted an increasing pressure on toolmakers to look for cheaper alterna-

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tives to cobalt powders [10]. To date, researchers have proposed a broad range of new iron-base alloys, which could be used as the matrix in diamond tools [11].

The main objective of this study was to determine the suitability of ball-milled Fe-Cu-Ni powder mixtures for fabrication of sintered 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:

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

Morphologies of the starting powders are shown in Fig. 1.

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 mixture was ball-milled for 30 hours in air 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 mass ratio was maintained at 10:1. The milling vial was rotated for 30 hours at 70% of the critical speed. The particle shape of as-mixed and the ball-milled powder is shown in Fig. 2.

The pre-mixed and ball-milled powders were tested for 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 Fig. 3.

Both the pre-mixed and 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 powder was held at 900°C and 35 MPa for 3 minutes in order to reduce the as-sintered porosity below 3%. The sintered FeCuNi specimens were subsequently subjected to X-ray phase analysis using a PANalytical Empyrean X-ray diffractometer using copper radiation (Cu$\lambda K\alpha$ = 1.5406 Å). The phases identified in the investigated samples are shown in Fig. 4.

The as-sintered specimens were also tested for density and hardness. The density measurements involved weighing the specimens in air and water (PN EN ISO 2738:2001) using the WPA120 hydrostatic weighing system. The results are summarized in Table 1.

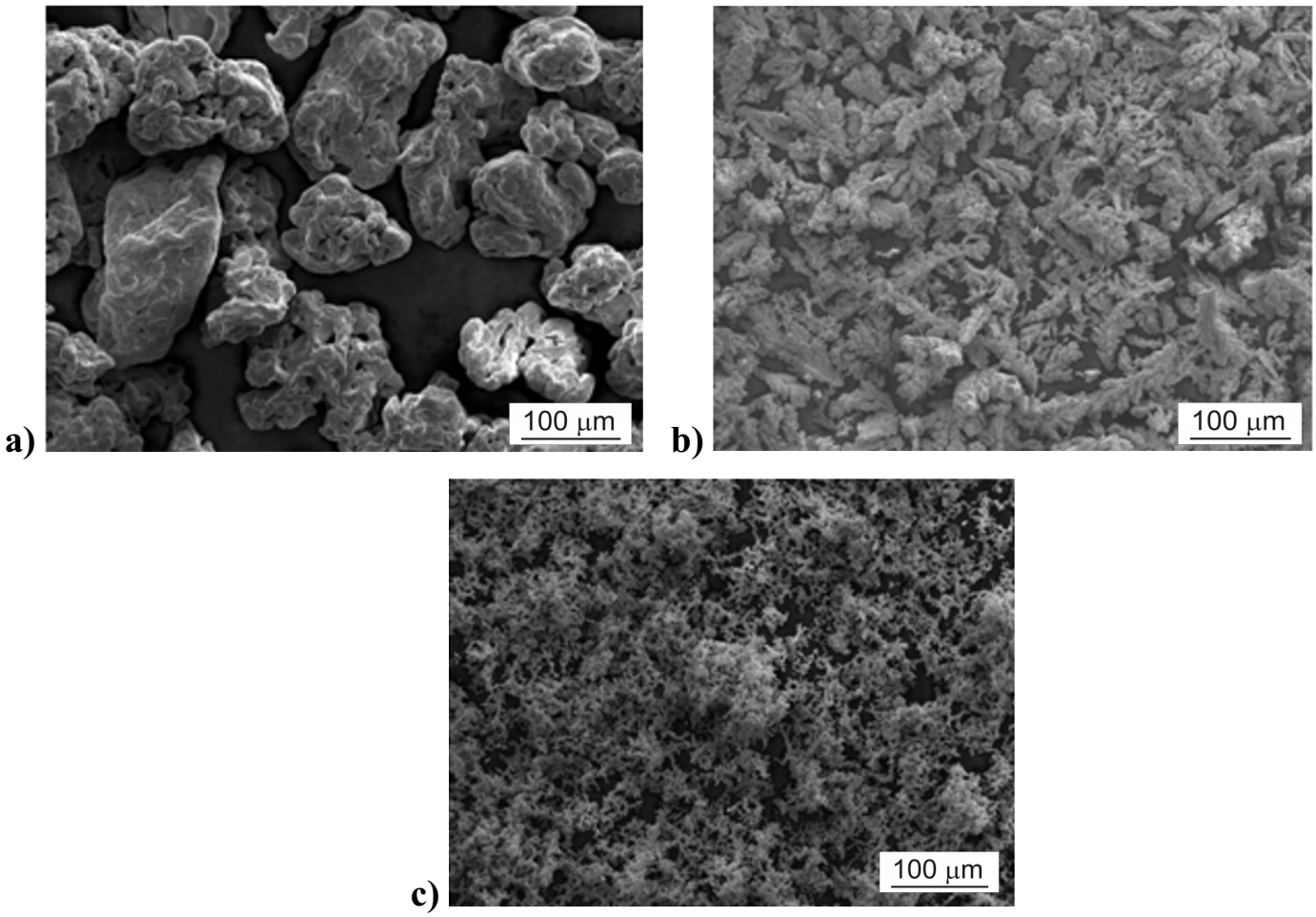


Fig. 1. Experimental powders: a) NC100.24; b) CH-L10; c) T255

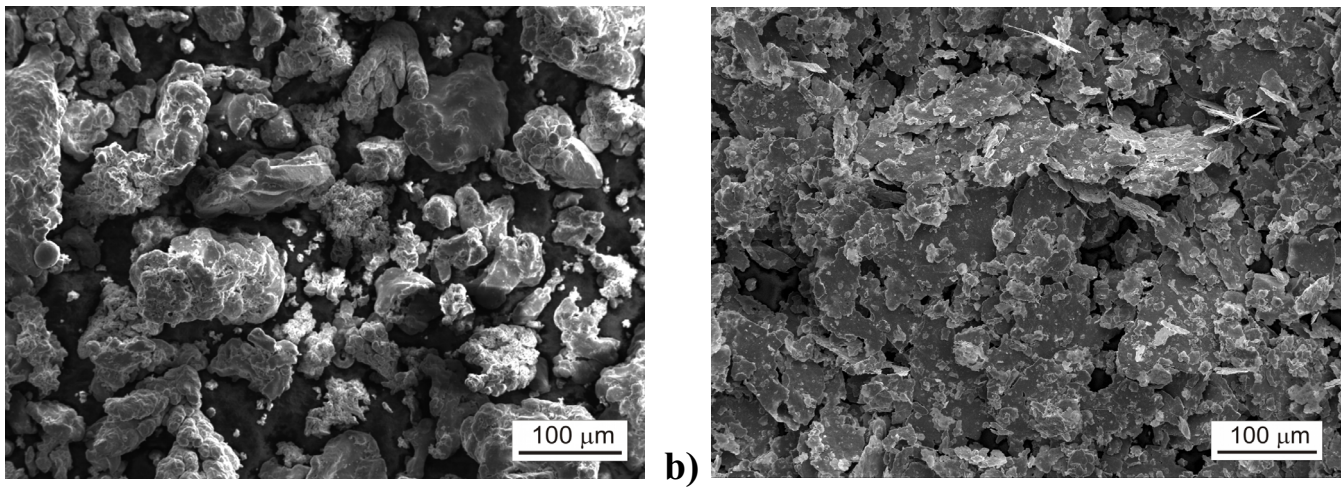


Fig. 2. Particle shapes of powders in as-mixed a) and ball-milled b) condition

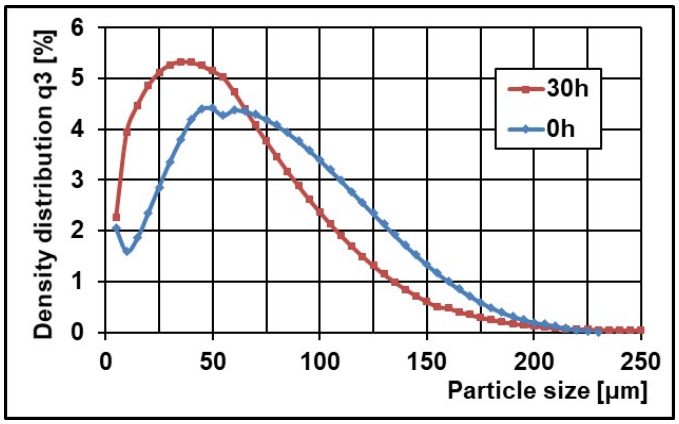


Fig. 3. Particle size distribution of the starting premixed powders and after milling for 30 hours

TABLE 1

As-sintered densities, porosities and hardness numbers

| Fe-Cu-Ni material made from | Density [g/cm$^3$] (1) | Theoretical density [g/cm$^3$] | Porosity [%] (1) | HV10 (1) |
|---|---|---|---|---|
| Premixed powders | 7.82 ±0.03 | 8.25 | 5.18 ± 0.20 | 157.6 ± 12.1 |
| Ball-milled for 30 h | 8.10± 0.02 | 8.25 | 1.79 ± 0.25 | 300.9 ± 16.3 |

(1) scatter intervals estimated at 90% confidence level

The specimens were then machined to produce non-standard specimens for static tensile tests. The tensile strength tests were carried out using the INSTRON 4502 universal testing machine

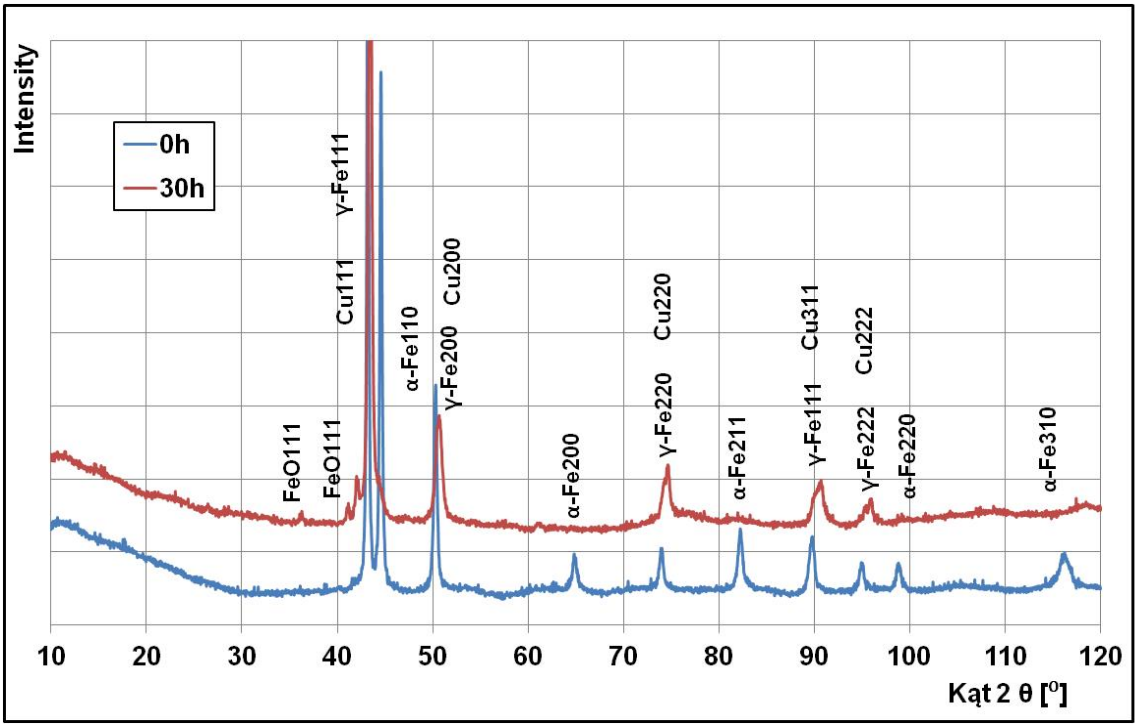


Fig. 4. X-ray diffractogram obtained for the premixed sinters (0 h) and after milling (30 h)

The diameter of the gage section of all 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 data acquired was used to calculate the offset yield strength ($R_{0.2}$), ultimate tensile strength ($R_m$) and elongation ($\varepsilon$). The results of the static tensile strength test and a typical stress-strain curve are given in Table 2 and plotted in Fig. 5, respectively.

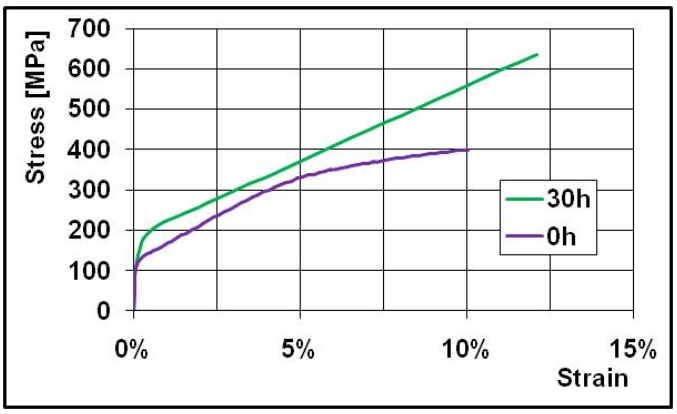


Fig. 5. Selected stress-strain curve for the premixed sinters (0 h) and after milling (30 h)

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. Typical fracture surfaces and microstructures are shown in Figs 6 and 7, respectively.

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

TABLE 2

Static tensile test results

| Fe-Cu-Ni material made from | Offset yield strength $R_{0.2}$ [MPa] (1) | Ultimate tensile strength $R_m$ [MPa] (1) | Elongation $\varepsilon$ [%] (1) |
|---|---|---|---|
| Premixed powders | 137.0 ± 10 | 396.9 ± 7.5 | 10.0 ± 0.20 |
| Ball-milled for 30 h | 229.0 ± 15 | 634.9 ± 13.3 | 12.1 ± 0.45 |

(1) scatter intervals estimated at 90% confidence level

TABLE 3

Chemical compositions in selected areas shown in Fig. 7 (weight %)

| | Premixed powders | | | | Ball-milled for 30 h | | | |
|---|---|---|---|---|---|---|---|---|
| | O | Fe | Cu | Ni | O | Fe | Cu | Ni |
| (Fe) | — | 73.9 | 21.2 | 4.9 | — | 85.97 | 3.99 | 10.04 |
| (Cu) | — | 2.31 | 97.5 | 0.19 | — | 5.75 | 89.38 | 4.87 |
| (Oxides) | 17.84 | 75.08 | 1.86 | 5.22 | 19.04 | 72.73 | 4.25 | 3.98 |

## 3. Discussion

The objective of the present study was to fabricate sintered materials using inexpensive iron-based powders and to assess their potential applicability in sintered diamond impregnated tools. The hot pressing parameters were carefully selected in order to obtain the as-sintered density approaching the theoretical one (Table 1). It was found that the premixed and ball-milled powders could be consolidated to a virtually pore-free state by holding them for 3 minute at 900°C under a pressure of 35 MPa.

The addition of nickel and copper to iron resulted in a combination of relatively high mechanical strength and good ductility of the investigated alloys. After consolidation, the alloys possess high hardness, which markedly increases for the ball-milled powder. Both materials show an excellent combination of high hardness and ductility. The experimental data indicates that the as-sintered samples reach near-full density levels, good ductil-

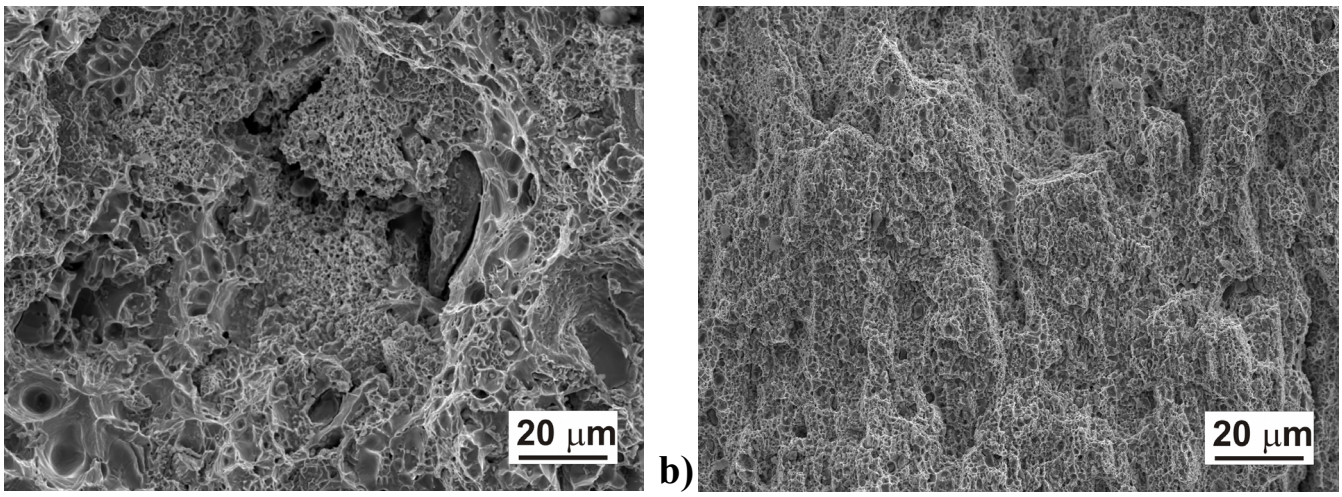


Fig. 6. Typical fracture surface of tensile specimens obtained from the premixed (a) and ball-milled powder (b)

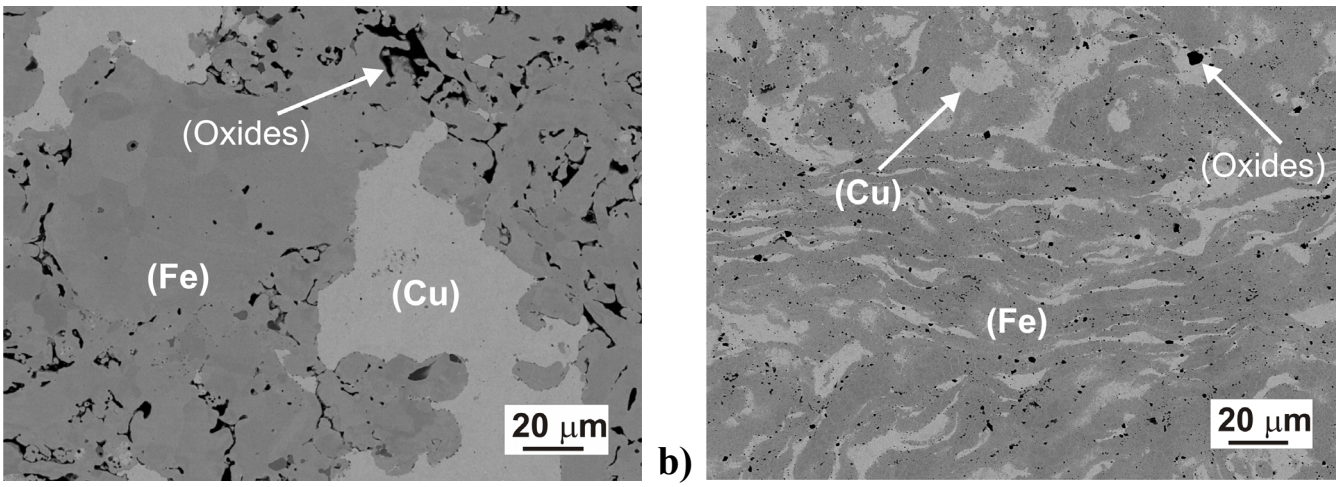


Fig. 7. Microstructure of Fe-Cu-Ni obtained from: a) premixed powders, b) ball-milled powders

ity, relatively high hardness and yield strength. The specimens obtained from premixed powders are characterized by a coarse-grained microstructure, which apparently impairs their hardness and mechanical strength.

The microscopic examination of the metallographic sections have revealed marked differences in microstructural homogeneity of the tested specimens. Parts manufactured from premixed powders show a highly inhomogeneous distribution of structural components. After milling a strong grain refinement had been observed. The as-sintered microstructure of material made from the ball-milled powder is markedly finer as compared with the premixed counterparts (Fig. 7b). Both alloys revealed presence of ferrite, (Cu) and oxides.

The XRD analysis showed that the α-Fe peaks of specimens obtained from the ball-milled powders disappeared. There are lines indicating the occurrence of γ-Fe iron, however they overlap with the copper lines. The specimens obtained from ball-milled powders revealed weak lines of FeO (II) oxide. There are no lines of Ni, which implies diffusion of Ni atoms into (Fe) and (Cu) solusions (Fig. 4).

The fractographs presented in Fig. 6 show ductile dimpled fracture surfaces. The fracture surfaces in the specimen made from the ball-milled powder is finer and more homogeneous (Fig. 6b).

## 4. Conclusion

The experimental data imply that the material obtained from the powders ball-milled for 30 hours and hot consolidated to near-full density at 900°C is characterized by a fine-grained, relatively inhomogeneous microstructure. It should be emphasized that although the studied material shows slightly lower strength and ductility as compared with cobalt, its hardness and ductility 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 ease of consolidation through hot pressing. Presumably its mechanical strength can be improved by fine tuning the chemical composition as well as the powder processing and consolidation conditions.

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