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SINTERED Fe-Ni-Cu-Sn-C ALLOYS MADE OF BALL-MILLED POWDERS

SPIEKANE MATERIAŁY Fe-Ni-Cu-Sn-C OTRZYMANE Z MIELONYCH PROSZKÓW

The main objective of this paper was to perform sinterability studies of ball-milled Fe-12%Ni-6.4%Cu-1.6%Sn-0.6%C powders. A mixture of precisely weighed amounts of elemental iron, nickel and graphite, and pre-alloyed 80/20 bronze powders was ball-milled for 8, 30 and 120 hours. After cold-pressing at 400 MPa the specimens were sintered at 900°C for 30 minutes in a reducing atmosphere and subsequently tested for density and hardness as well as subjected to structural studies using scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis.

The obtained results showed that only the powder ball-milled for 120 hours yielded a near pore-free material after consolidation by cold pressing and pressureless sintering at 900°C. As such, it could have a potential to be used in production of diamond-impregnated wire saw beads.

Keywords: sintered diamond tools, matrix, sintering, microstructure

Głównym celem pracy było zbadanie przydatności mielonych proszków w młynie kulowym do wytwarzania spiekanych materiałów Fe-12%Ni-6.4%Cu-1.6%Sn-0.6%C. Mieszanę elementarnych proszków żelaza, niklu oraz grafitu wraz z proszkiem brązu B20 poddano mieleniu w młynie kulowym przez okres 8, 30 i 120 godzin. Prasowane pod ciśnieniem 400 MPa wypraski wykonane z mielonych proszków poddano spiekaniu w temperaturze 900°C przez 30 minut w atmosferze redukującej. Spieki zostały poddane badaniom gęstości, twardości oraz mikrostrukturalnym, obejmującym mikroskopię skaningową oraz rentgenowską analizę fazową.

Otrzymane wyniki badań wskazują, że tylko spieki wykonane z proszku mielonego przez 120 godzin odznaczają się niską porowatością i z tego powodu mogą stanowić potencjalny materiał osnowy spiekanych segmentów metaliczno-diamantowych przeznaczonych do produkcji pił linowych.

1. Introduction

Earlier studies on hot pressing of Fe-Ni-Cu-Sn-C powders have shown excellent wear resistance of the as-consolidated material. This self-hardening effect has been found beneficial in the production of sintered diamond-impregnated tools used for cutting abrasive rock and ceramics [1-7]. It should also be noted that the strain-induced martensitic reaction generates compressive stress in the material, which may potentially enhance retention of diamond crystals protruding from the working face of the tool [8].

Commercial interest in this particular application is growing as markedly more expensive and creating health hazards Co-WC powders have been commonly used until recently [9,10]. The latest trend is towards a broader acceptance of the conventional cold-press/sinter route for fabrication of sintered diamond tools. In particular, economic benefits can be obtained from its application in the mass production of wire saw beads [11]. Therefore the main objective of the present work is to study the as-sintered microstructure and properties of

inexpensive, ball-milled Fe-Ni-Cu-Sn-C powders with respect to their potential use in the production of diamond wire.

2. Experimental procedure and results

Four commercial powders (Fig.1) were used as starting materials, namely:

- carbon-reduced sponge iron (NC100.24 grade of Höganäs AB),
- carbonyl nickel (T210 grade of Vale),
- water atomized bronze containing 20% tin (25GR80/20-325 grade of ECKA),
- synthetic graphite (F10 grade of Timcal).

A powder containing 12% Ni, 0.6% C, 8% bronze, and balance Fe was mixed in a Turbula-type mixer for 10 minutes and milled for 8, 30 and 120 hours in a ball mill. The rotational speed of the mill vial was 91 rpm which corresponded to 70% of the critical speed. About 50% of the milling container was filled with 12 mm bearing steel balls and ~10:1 ball-to-powder weight ratio was used. After milling the powders were ex-

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amined for their particle size distribution, by means of the Fritsch laser particle sizer *Analysette 22*, apparent density and tap density, and subjected to microscopic examination.

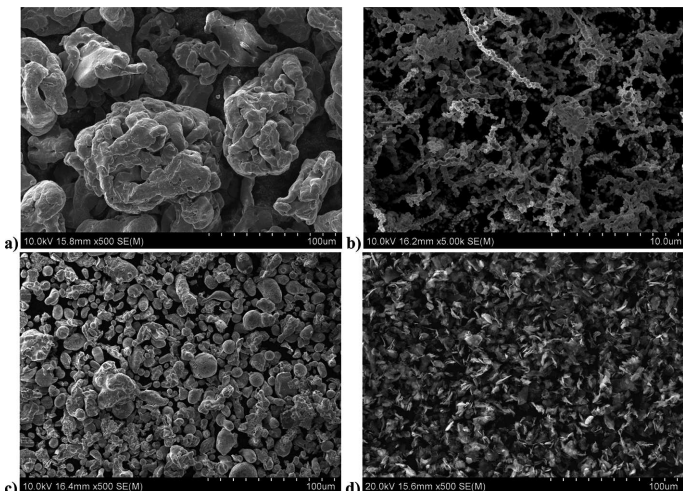


Fig. 1. (a) NC100.24 iron, (b) T210 nickel, (c) 25GR80/20-325 bronze and (d) F10 graphite

The results are shown in Figs 2 and 3, and in Table 1.

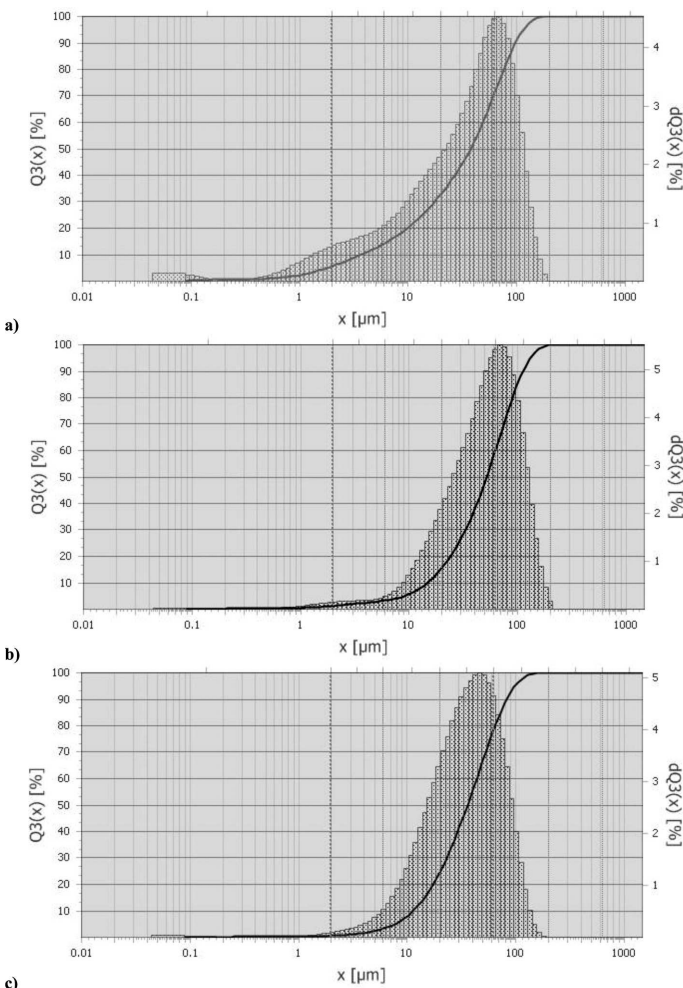


Fig. 2. Particle size distributions after milling for (a) 8 hours, (b) 30 hours and (c) 120 hours

TABLE 1

Particle size distributions, apparent densities and tap densities of the ball-milled powders

| Particle size, μm | Cumulative frequency, % | | |
|------------------------------|------------------------------------|------------------------------------|------------------------------------|
| | 8 hours | 30 hours | 120 hours |
| 2 | 5.7 | 1.3 | 0.6 |
| 5 | 12.5 | 2.9 | 2.5 |
| 10 | 20.2 | 5.7 | 8.0 |
| 20 | 33.0 | 15.7 | 24.7 |
| 30 | 43.2 | 26.9 | 41.4 |
| 50 | 61.0 | 48.1 | 67.2 |
| 80 | 82.3 | 74.0 | 89.0 |
| 100 | 91.1 | 85.4 | 95.5 |
| 150 | 99.2 | 97.8 | 99.8 |
| 200 | 100 | 99.9 | 100 |
| Mode | 66 μm | 70 μm | 45 μm |
| Apparent density | 2.92 g/cm³ | 1.76 g/cm³ | 1.18 g/cm³ |
| Tap density | 3.85 g/cm³ | 2.83 g/cm³ | 2.18 g/cm³ |

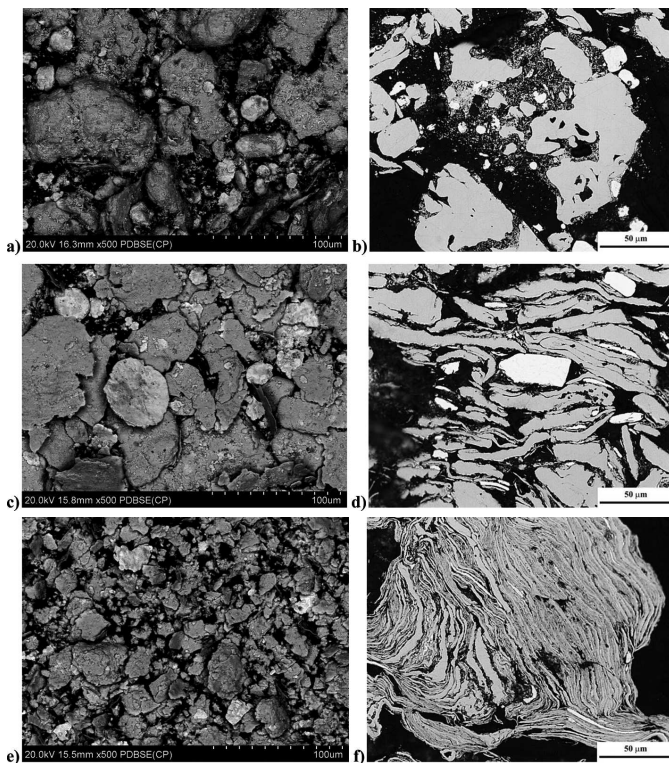


Fig. 3. (a,c,e) Particle shapes and (b,d,f) microstructures of the ball-milled powder after milling for (a,b) 8 hours, (c,d) 30 hours and (e,f) 120 hours

The ball-milled powders were subsequently cold pressed at 400 MPa in a rigid 7×40 mm die. The green compacts were subsequently sintered at 900°C for 30 minutes in hydrogen and slowly cooled down to room temperature with the furnace. Between 900 and 350°C the average cooling rate was 1 K/s. The as-sintered specimens were tested for density by the water displacement technique, Rockwell hardness and Vickers microhardness. The results are summarised in Table 2.

TABLE 2
 Properties of sintered Fe-Ni-(Cu-Sn)-C specimens

| Milling time, hours | Density, g/cm ³ | Hardness, RB * | Microhardness, HV0,5 * |
|---------------------|----------------------------|----------------|------------------------|
| 8 | 6.30 | 71±8 | 167±65 |
| 30 | 6.34 | 93±7 | 206±48 |
| 120 | 7.34 | 101±4 | 381±81 |

* – scatter intervals estimated at 90% confidence level

Selected specimens representing the three milling time variants were used to prepare metallographic cross-sections for microstructural studies by means of a scanning electron microscope fitted with an energy dispersive X-ray spectrometer (SEM-EDS).

The micrographs are presented in Figs 4 and 5.

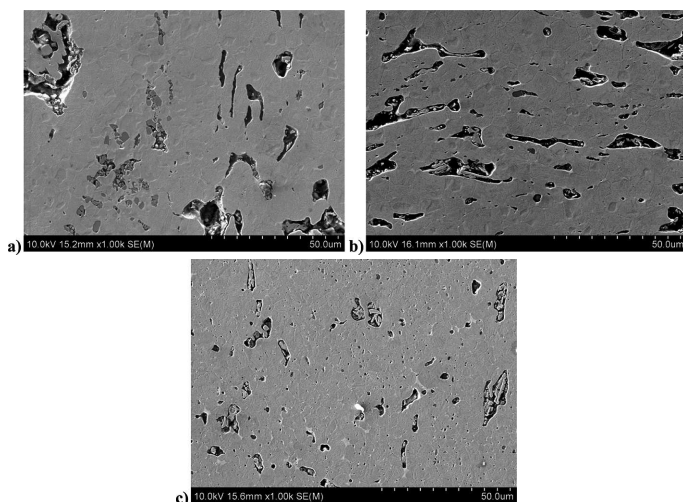


Fig. 4. SEM micrographs of specimens made of powders milled for (a) 8 hours, (b) 30 hours and (c) 120 hours

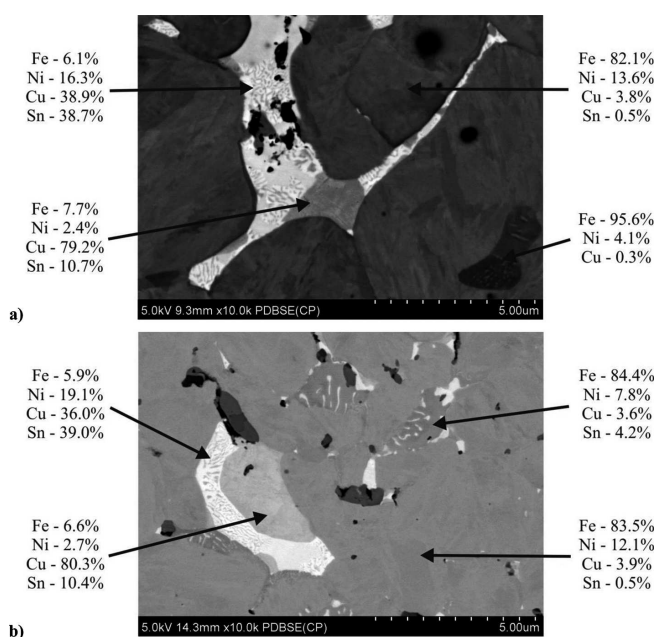


Fig. 5. EDS analysis of specimens obtained from powders milled for (a) 8 and (b) 120 hours

Microstructural observations taken at elevated magnifications have revealed that the bronze partially melts during the sintering cycle. According to the Cu-Sn phase diagram, in bronze containing 20% Sn the peritectic reaction occurs at 798°C. As a result a Cu-13.5% Sn solid solution and tin-rich liquid are formed. The melting bronze dissolves some iron from the surface and causes rounding of the iron grains. As it is evident from Fig. 5, nickel dissolves readily in the liquid phase and solidifies forming eutectic structures.

In order to determine the volume fractions of (γ Fe) and (α Fe) the polished metallographic specimens were subjected to XRD phase analysis. The results are shown in Fig. 6.

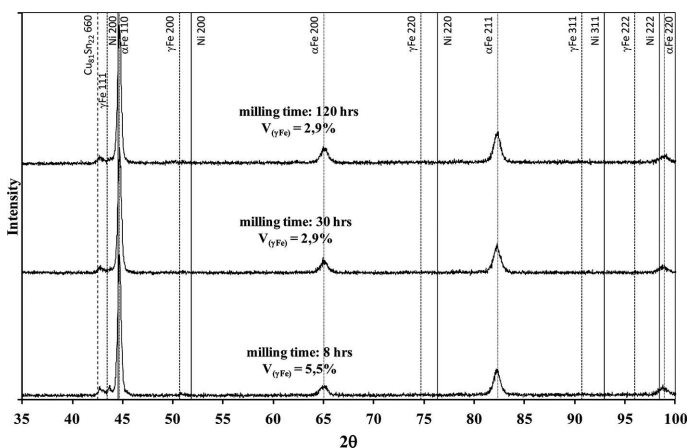


Fig. 6. XRD patterns. The volume fractions of (γ Fe) were calculated assuming that $V_{(\alpha Fe)} + V_{(\gamma Fe)} = 100\%$ (no account taken of other phases)

3. Discussion

During milling the powder particles were repeatedly flattened, work-hardened, fractured, and welded together. After 30 or 120 hours of milling time the particles showed flake shape (Figs 3c and 3e) and a characteristic layered microstructure with a very small lamellar spacing (Fig. 3f). Due to welding coarser particles were created. This increased the average particle size of the powder milled for 30 hours. Prolonged milling for 120 hours resulted in breakage of work-hardened particles and decreased the average particle size (Table 1). The change in particle shape and size with longer time of milling also resulted in decreasing the apparent density and tap density. The flake shape of powders milled for 30 and 120 hours had a major effect on their compaction properties. Although with careful handling it was possible to prepare test specimens, their green strength was insufficient for further processing under industrial conditions. Therefore the ball-milled powders would require granulation prior to cold compaction. The sintering behaviour of the investigated powders was markedly affected by the milling time. The best sinterability was obtained after 120 hours of milling and was manifested by the highest as-sintered density and hardness. The results given in Table 2 indicate that generally the investigated powders cannot be consolidated to full density by means of free sintering for 30 minutes at 900°C in hydrogen but specimens made of the powder milled for 120 hours reach the closed porosity range (~8%).

The microstructural examinations of the metallographic sections revealed relatively high internal porosity (Figs 4a and 4b) and microstructural inhomogeneity of the tested specimens (Fig. 5). Those manufactured from the powder milled for 120 hours showed lower porosity (Fig. 4c) and fine-grained microstructure both resulting in high hardness (101 RB) and microhardness (381 HV).

The XRD analysis indicated that in a pressureless sintered alloys the volume fraction of retained austenite ranges from 2.9 to 5.5% and is markedly lower compared to their hot-pressed counterparts.

4. Conclusions

1. Ball-milling results in flaky powders having layered microstructure with very fine lamellar spacing obtained after milling for 120 hours.
2. Green compacts made of a powder milled for 120 hours can be pressureless sintered to a closed porosity condition at merely 900°C for 30 minutes in hydrogen.
3. The volume fraction of retained austenite in the investigated alloys is generally lower than 5.5%.
4. The relatively good combination of low porosity and high hardness makes the Fe-Ni-Cu-Sn-C powder milled for 120 hours a potential low cost candidate material to substitute cobalt and cobalt alloys as a matrix in diamond-impregnated wire saw beads. Its insufficient compactibility seems to be a side issue as powders are routinely granulated prior to cold-pressing in high-volume manufacturing of diamond wire beads.

Acknowledgements

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