A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 54 2009 Issue 4

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# IRON-BASE MATERIALS MANUFACTURED FROM PREMIXED POWDERS BY THE HOT PRESS PROCESS

# MATERIAŁY WYTWARZANE METODĄ PRASOWANIA NA GORĄCO Z MIESZANEK PROSZKÓW NA BAZIE ŻELAZA

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. Fe-Cu, Fe-Cu-Sn and Fe-Ni-Cu-Sn premixed powders were used for the experiments. They were consolidated by the hot press process. The specimens were then subjected to density measurements and tested for Rockwell hardness and bending properties. Their microstructure were examined by the light microscopy (LM), scanning electron microscopy (SEM) and X-ray diffraction (XRD). Irrespective of the chemical composition near full densities were achieved after a 3 minute hold at 35MPa and 900°C. All examined materials showed a fine-grained microstructure. The Fe-Ni-Cu-Sn material was brittle but had the highest hardness and mechanical strength whereas the Fe-Cu-Sn alloys combined a relatively high hardness, yield strength and good ductility.
*Keywords*: Diamond impregnated tools, Hot pressing, Metallic matrix, PM iron-base alloys

Celem przeprowadzonych badań było ustalenie wpływu składu mieszanki proszków na mikrostrukturę i własności spieków na bazie żelaza, przeznaczonych na osnowę w narzędziowych materiałach metaliczno-diamentowych. Mieszanki proszków Fe-Cu, Fe-Cu-Sn i Fe-Ni-Cu-Sn zagęszczano metodą prasowania na gorąco. Spieki poddano pomiarom gęstości, twardości oraz próbie trójpunktowego zginania. Strukturę spieków badano z wykorzystaniem mikroskopii świetlnej (LM), skaningowej mikroskopii elektronowej (SEM) oraz rentgenowskiej analizy strukturalnej (XRD). Pomiary gęstości wykazały, że bez względu na wyjściowy skład mieszanki, wytrzymanie proszku przez 3 minuty w temperaturze 900°C i pod ciśnieniem 35MPa pozwoliło na uzyskanie materiału o bardzo niskiej porowatości. Wszystkie spieki posiadały drobnoziarnistą mikrostrukturę. Najwyższą twardość i własności wytrzymałościowe osiągnął materiał Fe-Ni-Cu-Sn, podczas gdy spieki Fe-Cu-Sn łączyły bardzo dobrą plastyczność ze stosunkowo wysoką twardością i granicą plastyczności.

## 1. Introduction

As the instability of the cobalt market continues with a steep increase in its market price witnessed in the first quarter of 2008 [1], the replacement of cobalt powders with cheaper ones has become a priority in the diamond tool manufacturing industry. Over the past decade the toolmakers have been offered a broad range of pre-alloyed copper-base and iron-base powders to substitute cobalt in the manufacture of diamond impregnated tool components (Table 1). The recent trend, however, is to use premixed powders on a broader scale [2,3], especially in the production of DIY and semi-professional tools, because they are markedly cheaper as compared with their pre-alloyed counterparts.

TABLE 1
Chemical composition and mean particle size of commercial cobalt substitutes [4,5]

| Designation | Nominal chemical composition (wt.%) | | | | Particle size (1) | Producer |
|---|---|---|---|---|---|---|
| | Fe | Cu | Co | Other | μm) | |
| *Next 100* | 25 | 50 | 25 | – | 0.8-1.5 | Eurotungstene |
| *Next 200* | 15 | 60 | 25 | – | 0.8-1.5 | |
| *Next 300* | 72 | 3 | 25 | – | ~4 | |
| *Keen 10* | n/a | n/a | 25 | n/a | ~2.5 | |
| *Keen 20* | n/a | n/a | 19 | n/a | ~3 | |
| *Cobalite 601* | 70 | 20 | 10 | – | ~5 | Umicore |
| *Cobalite HDR* | 66 | 7 | 27 | – | 6-7 | |
| *Cobalite CNF* | 68.4 | 26 | – | 3Sn; 2 W; 0.6$Y_2O_3$ | ~2 | |

(1) measured by the Fisher Subsieve Sizer

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The range of powder compositions with good consolidation characteristics has expanded in recent years to allow more matrix alloy choices. In the as-hot pressed condition the novel materials possess good mechanical strength and toughness, and meet demanding service requirements working as a matrix in diamond impregnated tools. This study compares the strength and ductility of Fe-Cu, Fe-Cu-Sn and Fe-Ni-Cu-Sn PM alloys made from premixed powders based on elemental carbonyl iron.

## 2. Experimental procedure and results

Seven elemental powders and pre-alloyed bronze were used to manufacture specimens for bending tests. The powders are shown in Figure 1.

Six combinations of the raw powders were prepared by mixing in a Turbula mixer, for 30 minutes, and used to fabricate rectangular bending bars, 4.8 mm high, 6.5 mm wide and 40 mm long, by hot pressing in a graphite mould at 900°C. In each case the powder was held for 3 minutes at the peak temperature under a pressure of 35 MPa.

Four specimens of each composition were produced in each, single hot pressing cycle. They were subjected to density measurements and hardness tests. The results are presented in Table 2.

TABLE 2

Effect of material composition on density and hardness (2)

| No. | Composition (powder ingredients) | Mean density, ($g/cm^3$) | Hardness RB |
|---|---|---|---|
| *1* | 85% *Fe CN* + 15% *MT 100* | 7.90 ± 0.56 | 77.0 ± 4.0 |
| *2* | 85% *ABC 100.30* + 15% *25 GR-325* | 7.94 ± 0.07 | 58.2 ± 8.1 |
| *3* | 85% *Fe CN* + 15% *25 GR-325* | 7.94 ± 0.05 | 96.2 ± 3.3 |
| *4* | 85% *Fe CN* + 12.75% *CH-L 10* + 2.25% *30 GN-350* | 7.94 ± 0.02 | 94.8 ± 1.9 |
| *5* | 85% *Fe CN* + 12.75% *MT 100* + 2.25% *30 GN-350* | 7.93 ± 0.08 | 95.9 ± 2.7 |
| *6* | 75% *Fe CN* + 15% *25 GR-325* + 10% *T110* | 8.02 ± 0.09 | 111.7 ± 3.1 (HRC 43.4 ± 3.5) |

(2) confidence intervals estimated at the 95% confidence level

The specimens were afterwards tested for transverse rupture strength, 0.2% offset yield strength and amount of plastic deformation at failure, using the three-point bending test. The bending bars were supported by two HSS rods, 3.5 mm in diameter and lying 30 mm apart, and loaded perpendicular to the hot pressing axis, using a similar HSS rod positioned midway between the supports The results are included in Table 3. Selected bending curves, typical of each material, are shown in Figure 2, whereas all curves obtained for the nickel containing material No. 6 are compared with a curve typical of hot pressed *Extrafine Co* powder [6] in Figure 3.

TABLE 3

Effect of material composition on bending properties (2)

| Material No. | Transverse rupture strength (MPa) | 0.2% offset yield strength (MPa) | Plastic strain (%) |
|---|---|---|---|
| *1* | | 618 ± 28 | |
| *2* | None of | 333 ± 41 | None of |
| *3* | specimens | 1033 ± 74 | speciments |
| *4* | broke | 995 ± 12 | broke |
| *5* | | 995 ± 53 | |
| *6* | 1830 ± 153 | 1381 ± 544 | 1.64 ± 2.81 (0.67 ÷ 2.78) |

(2) confidence intervals estimated at the 95% confidence level

All the materials were subjected to microscopic examinations. Typical microstructures observed on diamond polished and etched transverse sections are shown in Figure 4.

Selected fractured specimens, representing materials No.3 and No.6, were also examined by means of XRD for phase compositions. The X-ray diffraction patterns are presented in Figure 5.

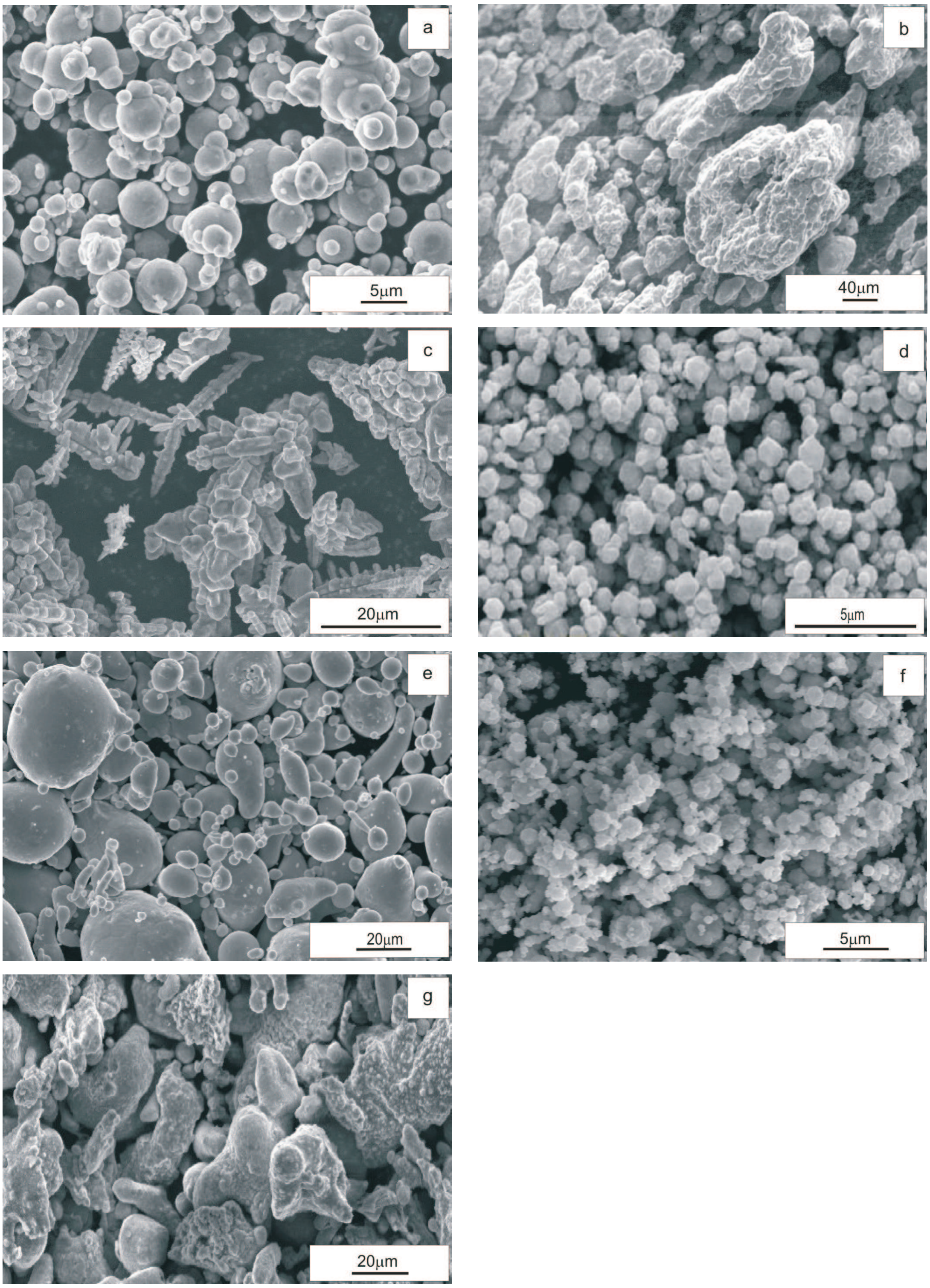


Fig. 1. SEM micrographs of experimental powders: (a) carbonyl iron *Fe CN* (BASF) (b) atomised iron *ABC 100.30* (Höganäs) (c) electrolytic copper *CH-L 10* (ECKA) (d) fine reduced copper *MT 100* (ECKA) (e) atomised tin *30 GN-350* (ECKA) (f) carbonyl nickel *T110* (INCO) (g) atomised tin bronze 85/15 *25 GR-325* (ECKA)

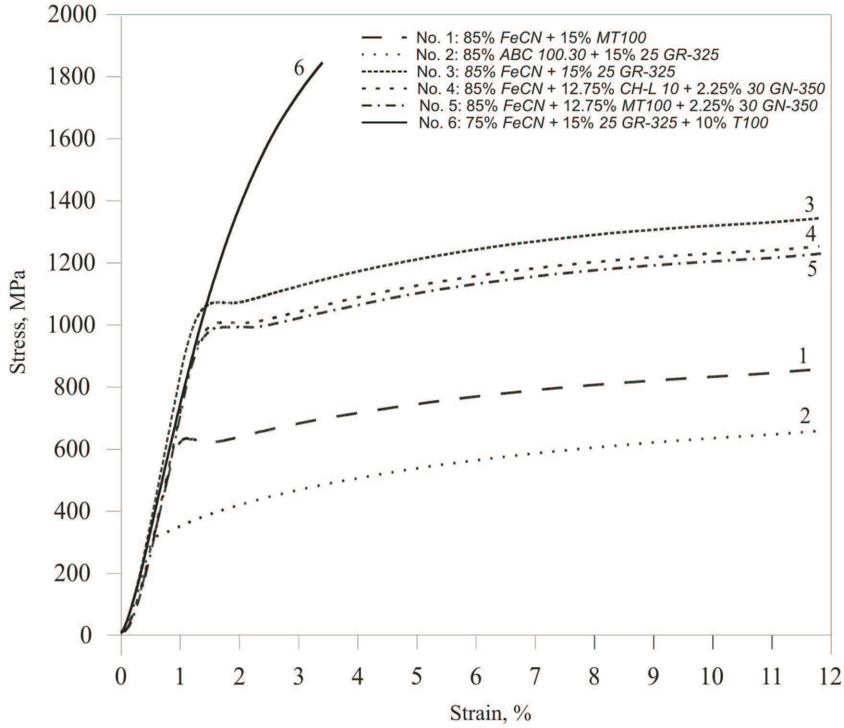


Fig. 2. The stress-strain behaviour of the investigated materials under 3-point bending conditions

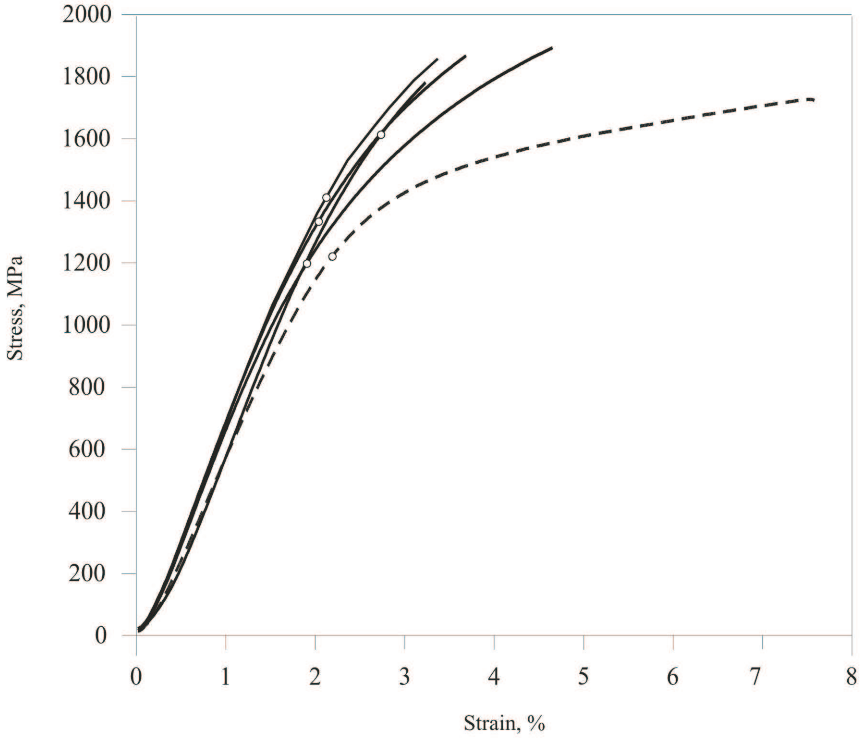


Fig. 3. The stress-strain behaviour of material No. 6 (solid curves) and hot pressed cobalt (dashed curve). White dots indicate the 0.2% offset yield strength

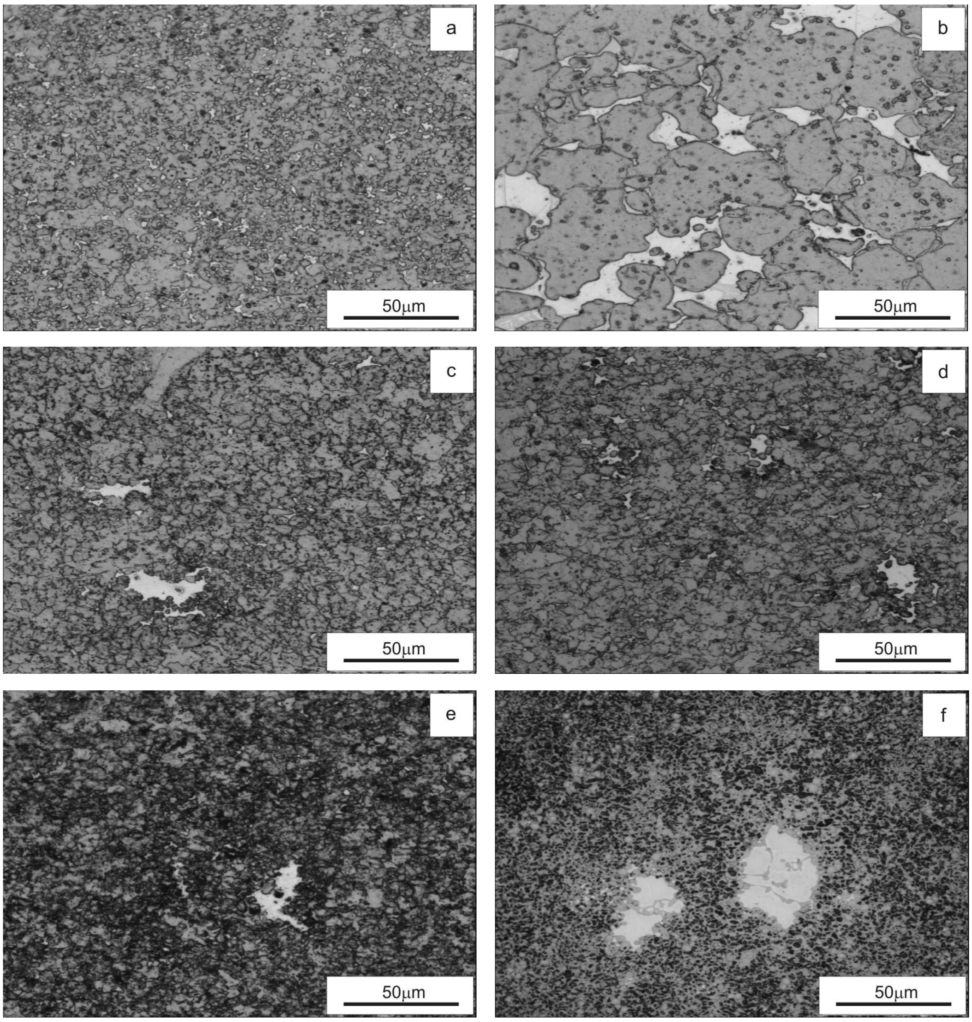


Fig. 4. LM microstructures of selected specimens hot pressed of: (a) 85% *Fe CN* + 15% *MT 100* (No.1) (b) 85% *ABC 100.30* + 15% 25 *GR-325* (No.2) (c) 85% *Fe CN* + 15% *25 GR-325* (No.3) (d) 85% *Fe CN* + 12.75% *CH-L 10* + 2.25% *30 GN-350* (No.4) (e) 85% *Fe CN* + 12.75% *MT 100* + 2.25% 30 *GN-350* (No.5) (f) 75% *Fe CN* + 15% *25 GR-325* + 10% *T110* (No.6)

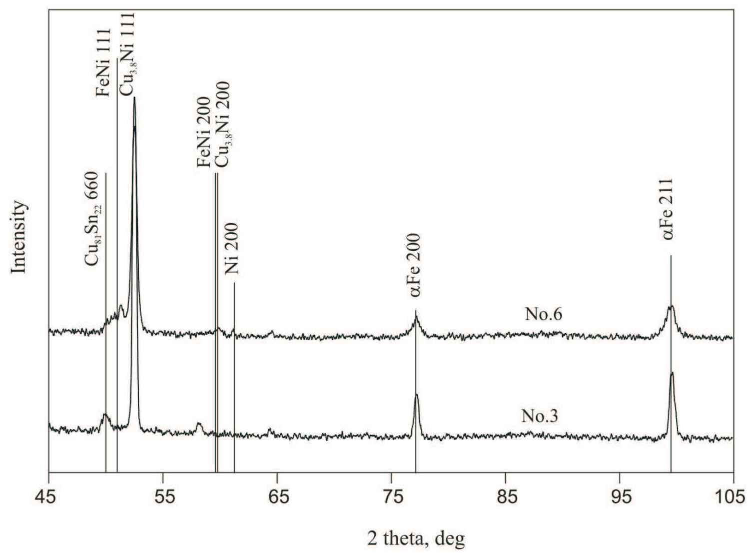


Fig. 5. X-ray diffraction patterns of materials No. 3 (85% Fe *CN* + 15% 25 *GR-325*) and No. 6 (75% *Fe CN* + 15% 25 *GR-325* + 10% *T110*)

## 3. Discussion

The specimen densities included in Table 2 show an excellent hot pressing behaviour of the tested powders. Except for material No. 1, there is a strong probability (0.975) of reaching near-full density (max. 2% porosity) upon holding the mixed powders for 3 minutes at 900°C under a pressure of 35 MPa.

The hardness and yield strength of the tested materials vary to a large extent depending on the particle size and chemical composition of the starting powders. The lowest mechanical strength has the material made of the coarse atomised iron powder (*ABC 100.30*). The application of fine carbonyl iron powder (*Fe CN*) results in a marked increase in hardness and yield strength of the material. Further increase in strength is possible by adding nickel, but only at the cost of ductility.

The highest mechanical properties have materials No. 3 and No. 6 obtained from the carbonyl iron *Fe CN*, pre-alloyed tin bronze 25 *GR-325* and, in the latter case, carbonyl nickel *T110*. Material No. 3 has a fairly uniform microstructure (see Figure 4) and exhibits a combination of a relatively high hardness and yield strength, and good ductility.

By substituting 10% iron with nickel it is possible to further increase the hardness and yield strength of the material beyond the benchmark values set by cobalt (see Figure 3), although the nickel addition markedly impairs the specimen's plasticity in the bending test (see Table 3). Seemingly, the rise in hardness and strength has been brought about by the formation of solid solutions containing nickel as it is evident from Figures 5 and 6.

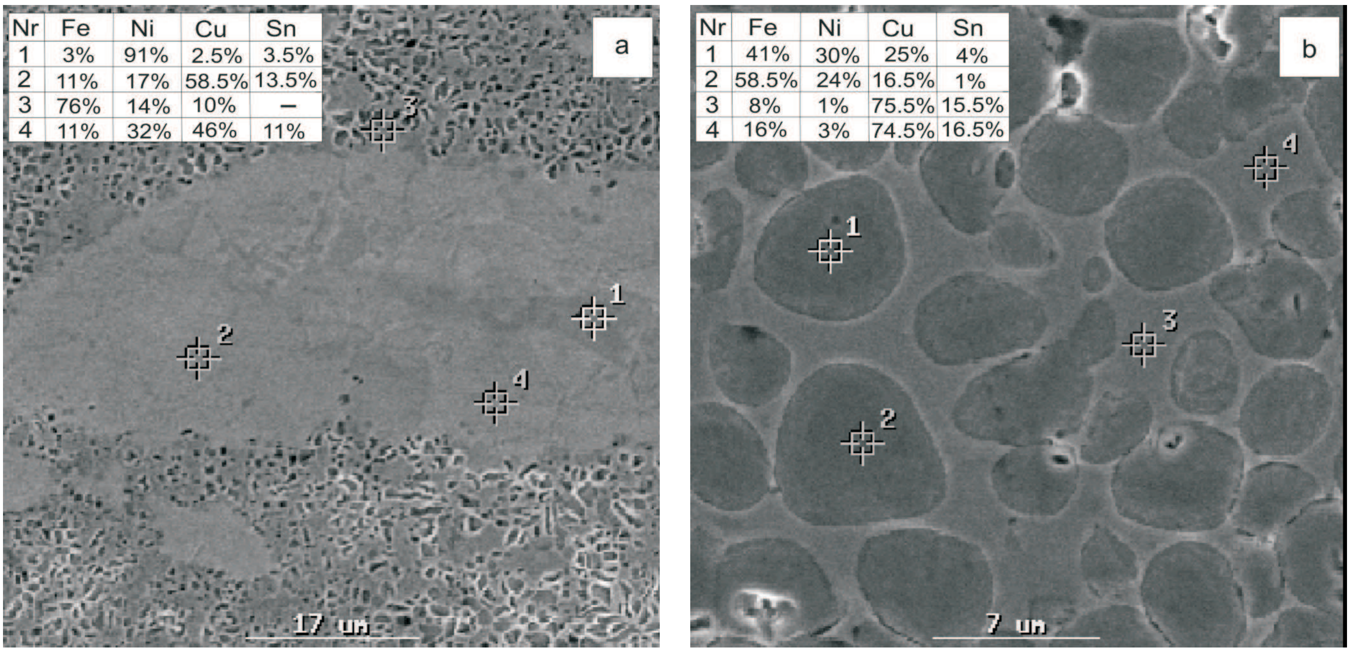


Fig. 6. Examples of electron probe micro analysis carried out in selected areas of a metallographic specimen of material No. 6

The data given in Table 3, for material No. 6, as well as the stress-strain curves illustrated in Figure 3 demonstrate a wide dispersion of the offset yield values. The apparent reason for this is imperfect mixing of the starting powders which results in non-uniform microstructure of the material, as exemplified in Figure 7.

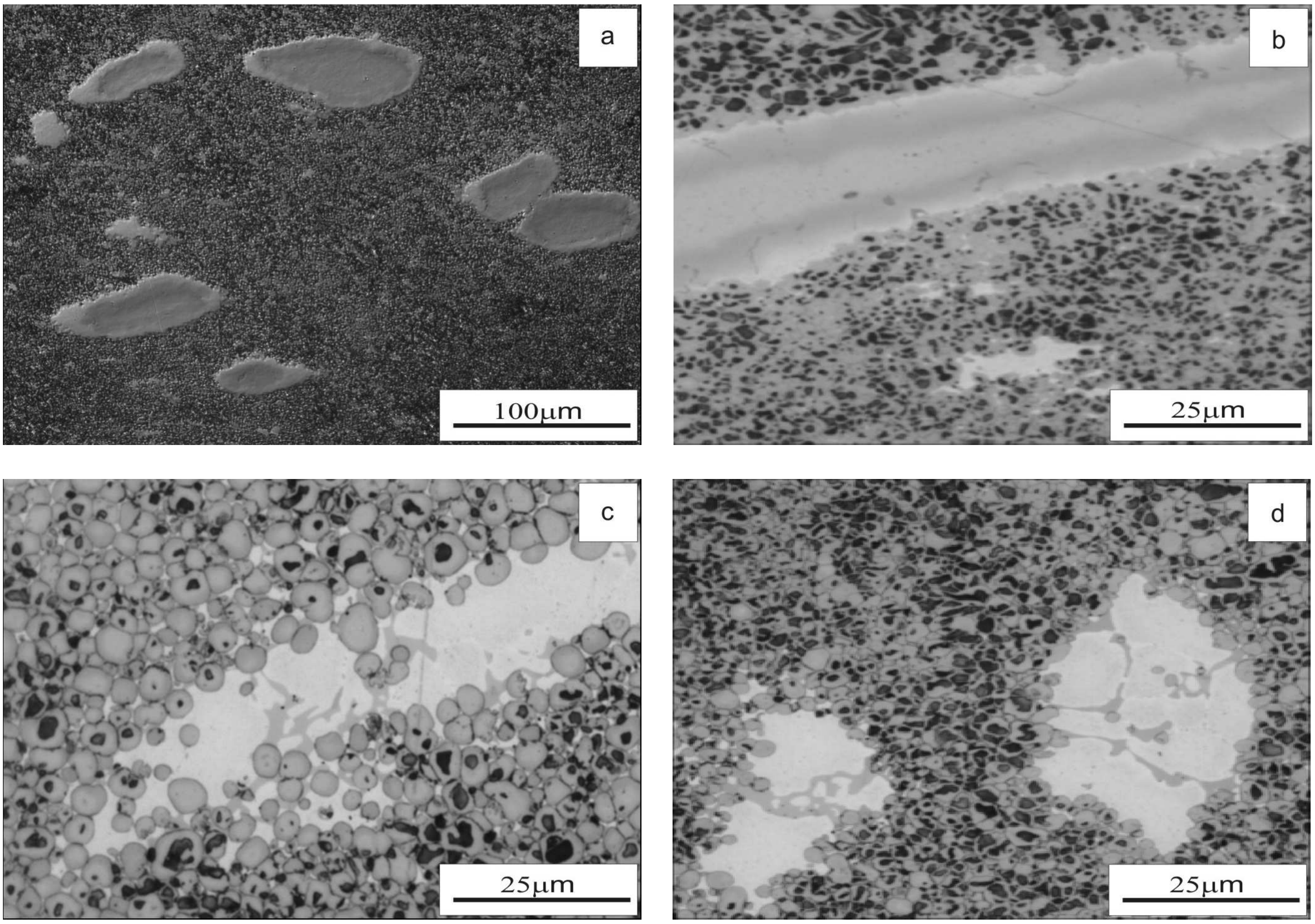


Fig. 7. LM micrographs showing microstructures in various areas of a metallographic specimen of material No. 6: (a) inclusions of nickel and copper-rich phases in an iron-rich matrix (b) large plate-like inclusion with a nickel-rich core (c) rounded iron and nickel-rich inclusions formed in abundant liquid (as in Figure 6b) (d) fibrous nickel-rich phase (grey) within a copper-rich lakes (as in Figure 6a)

## 4. Conclusions

1. The experimental work has shown that by holding the tested premixed Fe-Cu-Sn-(Ni) powders for 3 minutes at 900°C and 35 MPa it is possible to obtain nearly pore-free material.
2. The microstructure and properties of the material depend on the premix composition:
   a) the fine carbonyl iron-base material has markedly higher hardness and mechanical strength as compared with its coarse atomised iron-base counterpart;
   b) the addition of 2.25 wt.% tin to a Fe-Cu premix considerably increases the hardness and offset yield strength of the consolidated material;
   c) the particle size of a copper powder used has negligible effect on the properties of the consolidated material;
   d) the specimens fabricated from premixes containing elemental copper and tin have noticeably lower hardness and mechanical strength than specimens having the same bulk chemical compositions and made from a premix containing pre-alloyed bronze, although the difference is statistically insignificant;
   e) the addition of 10 wt.% nickel to a Fe-Cu-Sn premix markedly increases the hardness and offset yield strength of the material at the expense of its plasticity.
3. The tested Fe-Cu and Fe-Cu-Sn materials exhibit large ductilities.
4. The tested Fe-Ni-Cu-Sn premix poses mixing problems which results in a complex phase composition and non-uniform microstructure of the material upon consolidation.

**Acknowledgements**

The authors gratefully acknowledge Professor V. Ratuszek and Dr M. Paćko for their able assistance with the X-ray diffraction and mechanical tests as well as Messrs Cedric Sheridan of Foxmet S.A., Luxemburg, and Tom Stephenson of Valeinco, Canada, for providing powders. The work was supported by the Polish Ministry of Science & Higher Education through contract 11.11.110.788.

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*Received: 10 March 2009.*