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MICROSTRUCTURE AND PHASE COMPOSITION OF SINTERED COBALT-IRON MATERIALS

MIKROSTRUKTURA I SKŁAD FAZOWY SPIEKANYCH MATERIAŁÓW KOBALT-ŻELAZO

This paper describes the effect of the sintering parameters, e.g. temperature and atmosphere, on the microstructure and phase composition of the cobalt-iron materials. Investigations were carried out on the specimens made of the mixtures of elemental cobalt and iron powders, containing 3, 6, 10, 15 and 25 wt.% iron addition. Samples were obtained by cold pressing under 400 MPa then sintered at 900 and 1150 °C for an hour. As a sintering atmospheres both flowing, purified hydrogen and nitrogen was used.

The obtained results have shown that density of Co-Fe materials strongly depends on iron content, whereas sintering atmosphere has a minor effect on their hardness. Microstructural investigations have shown that applied sintering atmosphere plays a significant role if grain size and cobalt's phase composition are taken under consideration.

Keywords: Cobalt-Iron, Sintering, Microstructure, Phase Composition

W artykule przedstawiono wyniki badań poświęconych wpływowi parametrów spiekania, tj. temperatury i atmosfery ochronnej na mikrostrukturę i skład fazowy materiałów kobalt-żelazo. Badaniom poddano próbki wykonane z mieszek elementarnych proszków kobaltu i żelaza, zawierających 3, 6, 10, 15 i 25% wag. Fe. Próbki wykonano techniką jednostronnego prasowania pod ciśnieniem 400 MPa i spiekania w temperaturze 900 oraz 1150 °C przez okres 1 godziny. Jako atmosfery ochronne zastosowano zarówno przepływający wodór, jak i azot. Otrzymane wyniki wykazały, że gęstość spieków Co-Fe jest silnie związana z zawartością żelaza, natomiast rodzaj atmosfery spiekania nie ma znaczącego wpływu na ich twardość. Badania strukturalne wykazały natomiast, że rodzaj zastosowanej atmosfery ochronnej ma zasadnicze znaczenie na wielkość ziarna spieków Co-Fe oraz skład fazowy spiekanego kobaltu.

1. Introduction

Due to high and unstable price of the cobalt on the market, the diamond impregnated tools manufacturers try to replace, event partially, this element with another one, which would be cheaper and commonly available. One of the element which can be partially used for cobalt is iron. Mixtures of cobalt and iron powders used in diamond impregnated tools are cheaper than pure cobalt powders and due to iron content, guarantee a better diamond-to-metal matrix bonding [1]. Additionally, cobalt-iron alloys can be heat treated in order to attain required microstructure and mechanical properties [2,3].

In some applications, especially for production of wire saw beads, cold pressing and pressureless sintering technique is used. As it has been found at [4], the cold press/sinter route has been found useful for consolidation of the powder mix containing 3 wt.% iron to near-full density in hydrogen, which results in high yield strength. It is therefore assumed that the conventional cold press/sinter process, being markedly cheaper than hot pressing, has a great potential for the cost-effective production of diamond impregnated wire saw beads containing low-iron, cobalt-base matrix.

During sintering, in order to prevent sintered parts from oxidizing, protective atmosphere, mainly hydrogen, or N_2-H_2 mixtures are used. A good reductive condition are required especially during sintering of oxidized powders. Sometimes, when pure powders are sintered, as protective atmosphere flowing nitrogen can be also used. Because of a very strong reductive character of hydrogen or N_2-H_2 mixture, the microstructure of sintered part, and in consequence its mechanical properties could be different if they would be sintered under inert gas at the same temperature. That is why, the main purpose of this study was to determine the effect of sintering parameters, e.g. temperature and atmosphere, on microstructure,

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phase composition and hardness of selected cobalt-iron materials.

2. Experimental

Commercially available *extrafine* cobalt and *carbonyl* iron powder were used as a starting materials. Following mixtures (weight percent):
1 – Co;
2 – Co + 3% Fe;
3 – Co + 6% Fe;
4 – Co + 10% Fe;
5 – Co + 15% Fe;
6 – Co + 25% Fe.
were obtained by mixing in Turbula shaker mixer for an hour. From the mixtures, samples 5x5x15 mm were produced by cold pressing under 400 MPa. Then specimens were sintered in differential Netzesch 402E dilatometer both at 900 and 1150 °C. As a sintering atmosphere either flowing hydrogen or nitrogen was applied. The as-sintered specimens were then analysed for their density, porosity and Rockwell HRB hardness. Obtained results are summarised in Table 1 and presented on Fig. 1 and 2.

TABLE 1

Density, porosity and hardness of the examined materials

Mixture	Sintering temperature °C	Sintered under hydrogen				Sintered under nitrogen			
		Density g/cm^3	Relative density %	Porosity %	Hardness HRB	Density g/cm^3	Relative density %	Porosity %	Hardness HRB
Co	900	8.11	91.7	8.3	92	8.06	91.1	8.9	92
	1150	8.20	92.6	7.4	87	7.89	89.2	10.8	87
Co3Fe	900	7.93	89.9	10.1	87	7.87	89.3	10.7	90
	1150	8.14	92.2	7.8	81	7.29	82.6	17.4	73
Co6Fe	900	7.81	88.8	11.2	75	7.77	88.4	11.6	82
	1150	8.14	92.6	7.4	68	7.67	87.3	12.7	72
Co10Fe	900	7.68	87.8	12.2	57	7.52	85.9	14.1	56
	1150	8.06	92.1	7.9	45	7.21	82.4	17.6	52
Co15Fe	900	7.56	86.9	13.1	59	7.28	83.7	16.3	51
	1150	7.90	90.9	9.1	41	7.57	87.0	13.0	45
Co25Fe	900	7.55	87.8	12.2	81	7.02	81.6	18.4	65
	1150	7.86	91.4	8.6	89	7.50	87.2	12.8	80

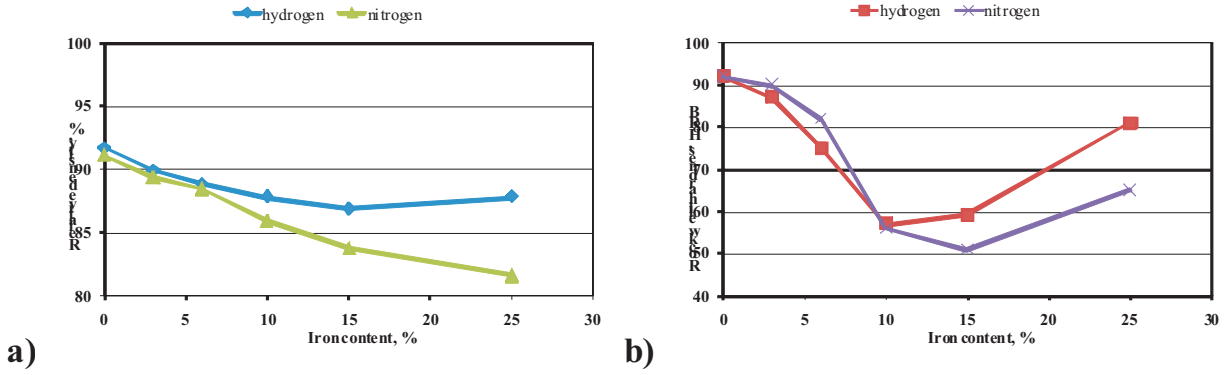

Fig. 1. Relative density (a) and Rockwell hardness (b) of the Co-Fe materials sintered at 900 °C

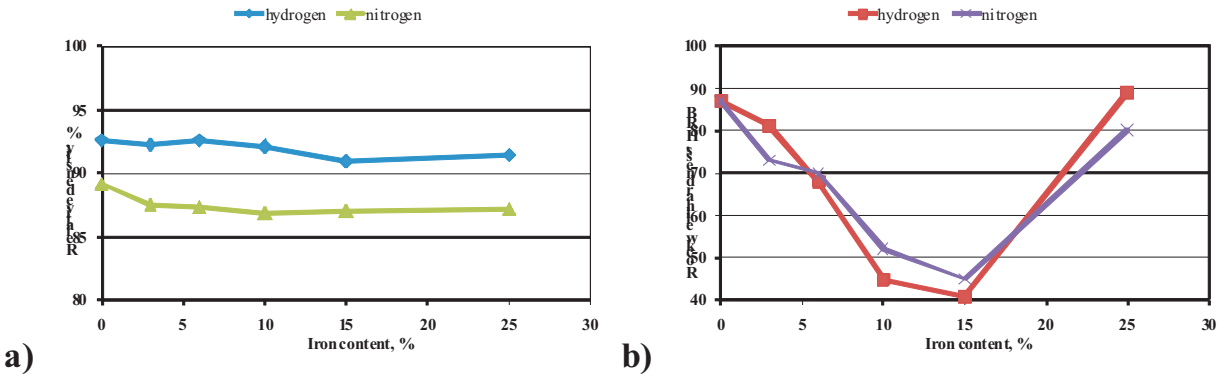

Fig. 2. Relative density (a) and Rockwell hardness (b) of the Co-Fe materials sintered at 1150 °C

Microstroctural observations were done on polished and etched cross-section of the sintered parts by means of light optical microscopy. In order to obtain the best results of the analysis, it was decided to use also a differential interference contrast (DIC) mode. Typical microstructures are pictured on Fig. 3 and 4.

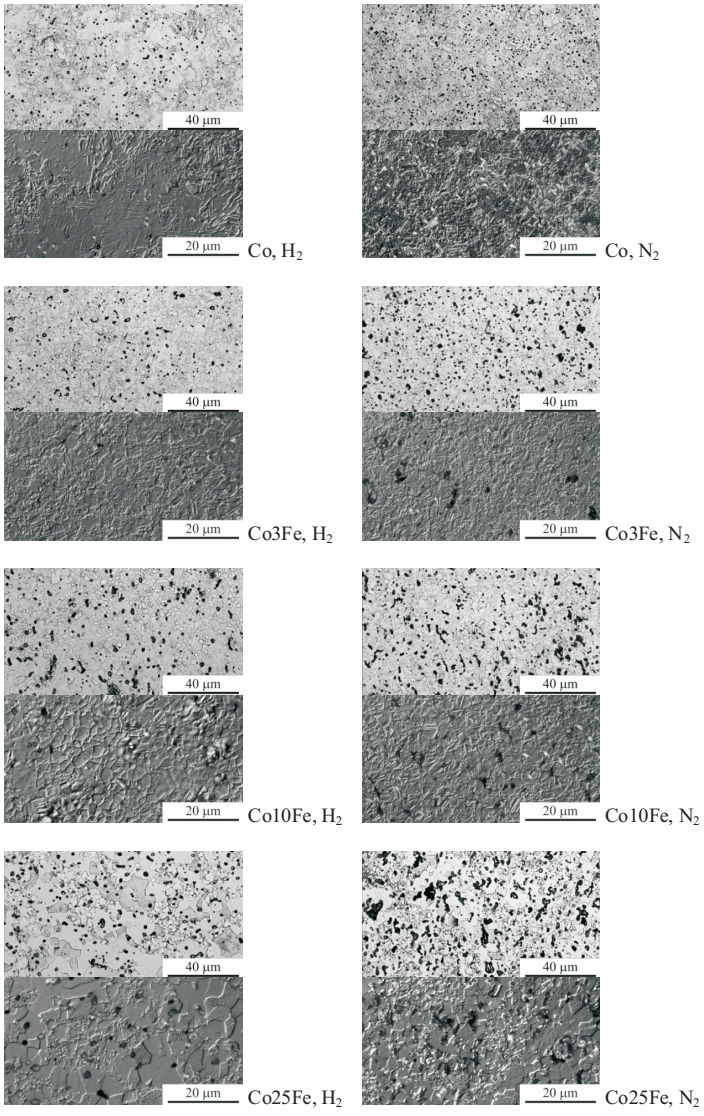

Fig. 3. Microstructures of the selected Co-Fe materials sintered at 900 °C

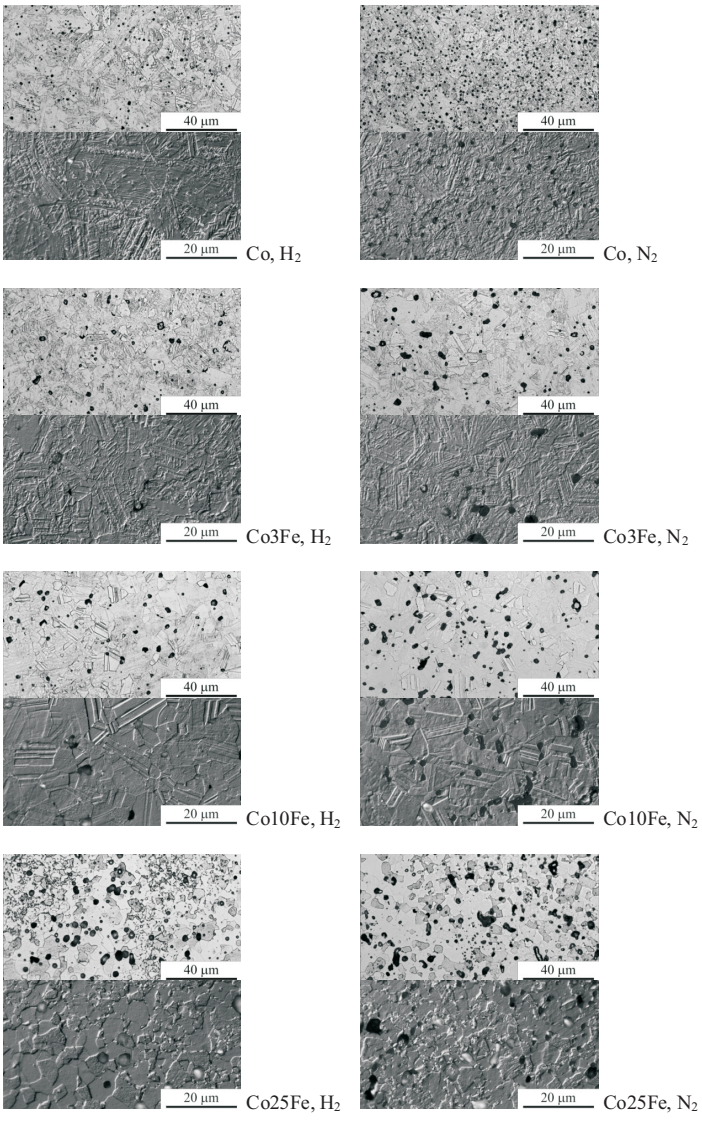

Fig. 4. Microstructures of the selected Co-Fe materials sintered at 1150 °C

The investigated materials were also subjected for X-ray phase analysis (XRD). Prior to the analysis, the specimens were ground on SiC papers and then electropolished in dilute sulphuric acid in order to remove the work-hardened layer. Recorded traces are presented on Fig. 5 and 6.

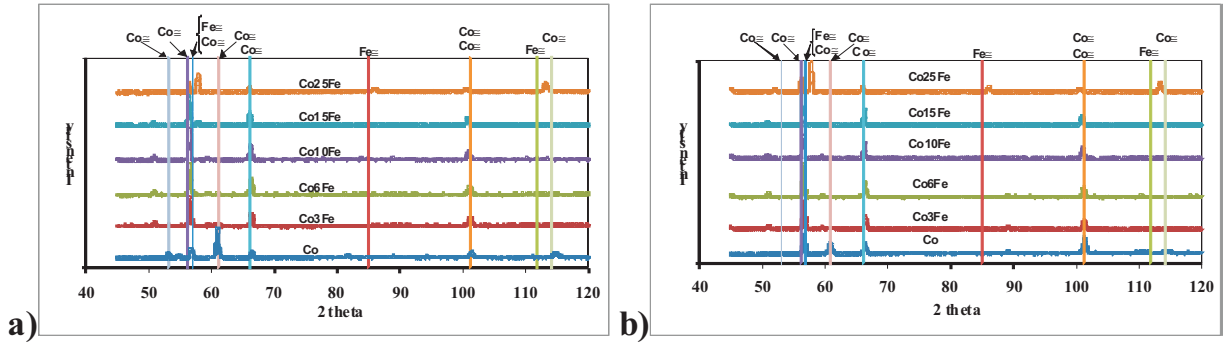

Fig. 5. X-ray traces of the Co-Fe materials sintered under hydrogen (a) and nitrogen (b) at 900 °C

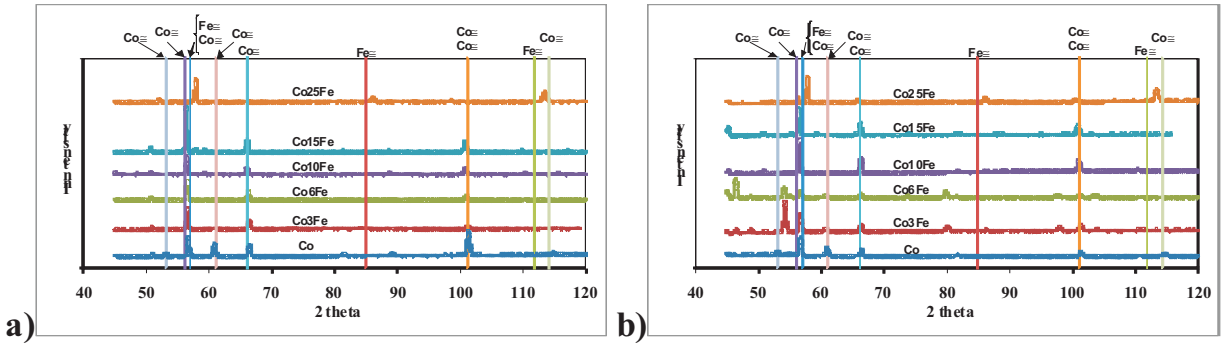

Fig. 6. X-ray traces of the Co-Fe materials sintered under hydrogen (a) and nitrogen (b) at 1150 °C

XRD was also used to determine an effect of sintering temperature and atmosphere on the volume fraction of the fcc phase (V_{fcc}) in sintered cobalt. V_{fcc} was calculated using the following equation:

$$V_{fcc} = \frac{I_{(200)}}{I_{(200)} + 0.505 \cdot I_{(10\bar{1}1)}} \cdot 100\%, \qquad (1)$$

where: $I_{(200)}$ and $I_{(10\bar{1}1)}$ are the integrated intensities of the (200) and $(10\bar{1}1)$ diffraction lines recorded for the fcc and hcp structures, respectively. Obtained results are summarised in Table 2.

TABLE 1

Co fcc phase content

Sintering temperature, °C	***900***		***1150***	
Sintering atmosphere	hydrogen	nitrogen	hydrogen	nitrogen
Co fcc phase content, %	27.6	59.1	63.5	56.4

3. Discussion

The density of sintered Co-Fe materials in most cases is strongly depended on the iron content, applied sintering temperature and atmosphere. Materials sintered at 900 °C show markedly lower density in comparison with their counterparts sintered at 1150 °C. Also, by increasing iron content, the density of the as-sintered Co-Fe parts decreases. The effect of applied atmosphere is evident, when density of Co-Fe materials sintered at 1150 °C is considered. The density of materials sintered under hydrogen is 4 to 12% higher than after sintering under nitrogen. In the case of lower sintering temperature, the effect of applied atmosphere is not so strong. The densities of cobalt and materials containing 3-6% Fe sintered under hydrogen is similar to the densities achieved after sintering under nitrogen. The considerably differences in densities are observed for the materials containing higher iron addition (10-25%).

The relative density of the samples made of cobalt sintered at 900 °C under hydrogen is 0.7% higher in comparison to their counterparts sintered under nitrogen, whereas relative density of the Co25Fe material sintered under hydrogen is 7.6% higher than sintered under nitrogen.

Cobalt samples sintered at 1150 °C under hydrogen show 3.8% higher density than sintered under nitrogen. In the case of Co25Fe, sintering under hydrogen leads to 4.8% higher relative density in comparison to the material sintered under nitrogen.

When the effect of iron content on relative density of the materials sintered at 900 °C under hydrogen is considered, it can be seen, that relative density of pure cobalt is about 4.4% higher in comparison to the Co25Fe material, and only 1.3% higher after sintering at 1150 °C. But if nitrogen is used as sintering atmosphere, the relative densities of cobalt sintered at the 900 °C and 1150 °C are 11.6% and 2.3% higher, respectively, in comparison with relative densities of the Co25Fe material. This indicates, that during sintering Co-Fe materials under nitrogen, the diffusion process is inhibited by the unreduced cobalt's and iron oxides. It is noteworthy that materials

containing iron additions are characterised by relative density over 90% after sintering at 1150°C under hydrogen, whereas pure cobalt specimens show such relative density after sintering at 900 °C.
The hardness of the investigated materials strongly depends on the iron content irrespective of sintering atmosphere. It is interesting that despite differences in the relative density of the parts sintered under hydrogen or nitrogen, examined materials show similar Rockwell hardness, except Co25Fe material, which hardness is markedly lower after sintering under nitrogen. The lowest hardness was recorded for the materials with 10-15% of iron addition. The microstructure of the investigated materials can be described as homogenous. As it is illustrated on Fig. 4, a grain-grow process took place when materials were sintered at higher temperature. It is evident that microstructure of specimens sintered at 1150 °C consist of greater grains with high amount of the twins formed during recrystallisation.
There are minor differences between microstructures of the materials sintered under hydrogen or nitrogen, but microstructure of Co-Fe materials sintered under nitrogen is characterised by higher amount of oxides, which hinder the grain growth and recrystallisation process.
Results obtained from the X-ray diffraction – Fig. 6, have proved that even small addition of iron stabilises the cobalt's α phase. The shifted position of the peaks with regard to the patterns is a consequence of formation of a cobalt-iron solid solution. Formation of this solid solution is accelerated by the higher sintering temperature. The calculated volume fraction of the *fcc* phase indicates that sintering at higher temperature under hydrogen leads to higher volume of the cobalt's *fcc* phase. This behaviour is the effect of grain grow process and it is in agreement with already published data [5,6]. When cobalt sintered under nitrogen is considered, the volume of the *fcc* phase is almost independent of the sintering temperature. One of the explanation of this effect can be that an average grain size of the sintered cobalt at 900 and 1150 °C is similar.

4. Conclusion remarks

The undertaken investigations and obtained results have shown that density of as-sintered Co-Fe materials strongly depends on the iron content. It is possible to obtain a very high relative density of the Co-(3-25%)Fe materials after sintering at 1150 °C under hydrogen. Due to inert behavior of the nitrogen, the reduction of cobalt's and iron oxides is impossible, that is why the sintered Co-Fe materials under nitrogen show markedly lower relative density. What is interesting, the applied sintering atmosphere has a minor effect on the hardness of the Co-Fe parts, both after sintering at 900 °C or 1150 °C. The results obtained from the X-ray diffraction have proved that formation of the cobalt-iron solid solution, on the one hand is accelerated by the higher sintering temperature, but on the other is hindered by the unreduced oxides. The calculated volume fraction of the cobalt's fcc phase show that phase composition depends on the applied sintering atmosphere.

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