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
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MICROSTRUCTURE AND MECHANICAL PROPERTIES OF FINE-GRAINED IRON PROCESSED BY HYDROEXTRUSION

CHARAKTERYSTYKA MIKROSTRUKTURY I WŁAŚCIWOŚCI MECHANICZNYCH ULTRADROBNOZIARNISTEGO SPIEKU ŻELAZA ODKSZTAŁCONEGO METODĄ WYCISKANIA HYDROSTATYCZNEGO

The aim of this study was to characterize the microstructure and properties of fine-grained iron. The samples were prepared by sintering at 1000°C ±5°C for 30 minutes, followed by Severe Plastic Deformation via Hydrostatic Extrusion (HE). After the sintering the rod samples were capsulated to prevent damage during extrusion. HE has been applied as a final consolidation process of iron powders. The HE process was carried out on two extrusion passes. The rods of 8 mm in diameter were obtained as a final product after hydroextrusion. The total cumulative strain was 2.3.

Materials in three states were investigated: sintered, sintered and deformed by one hydrostatic extrusion pass, and two hydrostatic extrusion passes. The results were compared with the results obtained for coarse-grained Armco iron. The measurements showed that the grain size was reduced from 3.5 μm after sintering to 180±81 nm after the 2nd extrusion pass. The tensile strength and microhardeness increased with cumulative strain of the material. The impact test carried out at -196°C showed transformation of the fracture mechanism from cleavage for coarse-grained structure to ductile for extruded material.

Keywords: iron, powder metallurgy, mechanical properties, low temperatures, plastic deformation, hydrostatic extrusion

W pracy scharakteryzowano mikrostrukturę i mechaniczne właściwości ultradrobnoziarnistego spieku żelaza. Próbki przygotowano w dwóch etapach – wypraski spieczono w temperaturze 1000°C ±5°C w czasie 30 minut, a następnie spieki odkształcono metodą wyciskania hydrostatycznego [HE]. W celu uniknięcia pękania spieków proces wyciskania przeprowadzono na próbkach uprzednio zakapsułkowanych. Proces HE przebiegał w dwóch etapach a skumulowane odkształcenie wyniosło 2.3. średnica końcowego produktu wyniosła 8 mm.

W pracy przedstawiono wyniki badań zarówno spieku, jak i materiału po poszczególnych wyciskaniach hydrostatycznych. Wyniki badań ujawniły redukcję rozmiaru ziaren żelaza z 3.5 μm po spiekaniu do 180 nm po drugim wyciskaniu HE. Zarówno wytrzymałość ja i mikrotwardość wzrosły ze stopniem skumulowanego odkształcenia próbek.

Wyniki badań udarnościowych porównano z wynikami otrzymanymi dla gruboziarnistego żelaza Armco. Badania udarności metodą Charpiego przeprowadzono na próbkach uprzednio chłodzonych w ciekłym azocie. Obserwacje mikroskopowe przełomów próbek po udarności wykazały zmianę charakteru pękania żelaza z łupliwego dla materiału gruboziarnistego na ciągliwy dla materiału ultradrobnoziarnistego.

1. Introduction

Sample preparation by powder metallurgy usually proceeds in three main steps: powder preparing, compaction and sintering. The final product quality and properties are determined by the powders properties and sintering parameters[1,2]. Additionally, the product properties are influenced by the powder characteristic such as particle shape and size, structure and surface development [3]. The product properties achieved by powder metallurgy are similar with those materials obtained by conventional forging methods.

The main advantage of powder metallurgy is reduction of machining process which results in a decrease in the cost of production. What is more, this method gives an opportunity to use finer grain size powders to produce fine grained structure products.

The porosity of the final product is the biggest disadvantage of materials produced by powder metallurgy [4,5]. The porosity leads to the decrease of mechanical properties (eg. strength, ductility, fracture toughness). The reduction of the porosity and the strengthening of materials can be caused by the grain refinement. The grain boundaries are the barriers for dislocation motion.

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The grain refinement can be achieved by hydrostatic extrusion process (HE)[5,6]. This method was used to strengthen a lot of materials such as: steel, aluminum alloys, copper[7,8]. After HE, grain size become finer than in the original state and the rod properties along the transverse section were uniform. The iron sinter made of two kinds of powders was chosen.

It was expected that the applied method of materials processing will cause the porosity reduction and will increase mechanical properties and elongation to failure of the final product in comparison to the commercially used coarse-grained Armco iron.

2. Materials experimental procedure

The iron sinters were investigated. The samples were prepared by sintering, followed by Hydrostatic Extrusion. Two kinds of iron powders delivered by KAMB Import-Export were used to prepare the samples. The characteristic of the powders is presented in Table 1.

TABLE 1

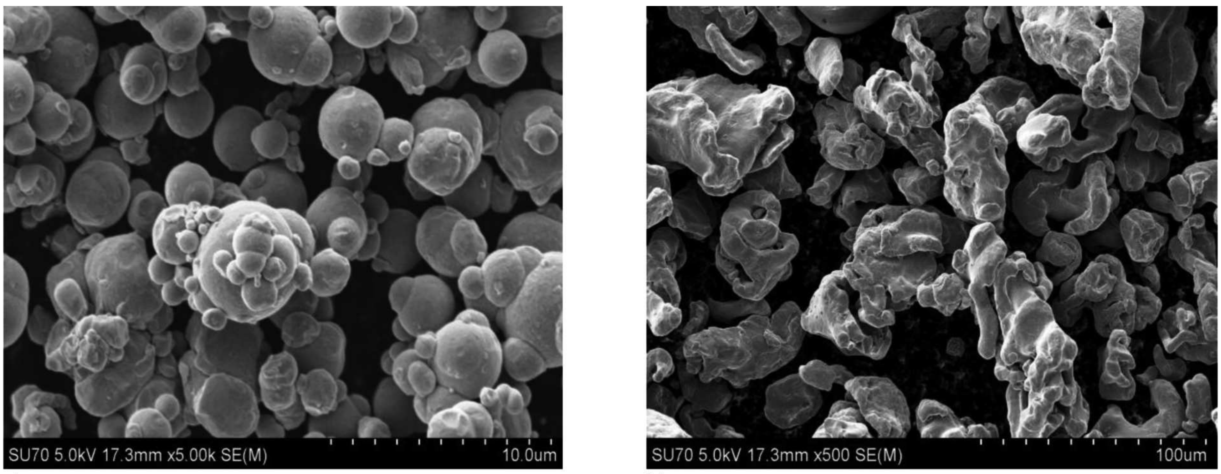
The powder characteristic

parameter	“FeM”	Iron powder “FeD”
chemical composition (producer data) [%]	99.5 Fe,0.04C	99 Fe, 0.01C
particle size [um]	6.8	35.1
particle shape (PN-EN ISO 3252)	spheroidal	irregular, nodular
Density [g/cm^3]	7.79±0.0044	7.82 ±0.0015
BET Multiplot	0.29	0.06

The crystallite size was measured by X-ray diffraction using CuKα radiation. To estimate crystallite size the Scherrer’s and Williamson-Hall’s equations were used. The particles size was analyzed by the small angle scattering method in isopropanol environment. Before the measurement ultrasounds were used to reduce the agglomerates.

Firstly, the weighed powder was milled for 2 hours without using any grinding balls. Secondly, the powders mixtures were consolidated by isostatic pressing at 210 MPa. Thirdly, the samples were sintered at 1000°C ± 5°C for 30 minutes using protective atmosphere (endothermic propane gas with dew-point +10°C). After the sintering the rod samples were capsulated to prevent damage during extrusion. The liquid medium which surrounds the sample during extrusion process is under pressure. The liquid medium penetrates into the pores of the material which causes the damage to the extruded samples. Finally, the samples were extruded via hydrostatic extrusion. HE was carried out in 2 passes with the final true strain 2.3. In the first pass the core diameter was reduced from 25 mm to 11 mm, and in the second one: from 11 mm to 8 mm. The maximal achieved extrusion pressure was 963 MPa.

The density of all fabricated materials was investigated using Archimedes’ method. The microstructure was investigated using light microscope as well as electron microscope. The transverse and longitudinal sections of the samples were examined. The samples in three conditions: sintered, sintered and deformed by one hydrostatic extrusion pass, and two hydrostatic extrusion passes were observed by light microscope. The samples used for observation were grinded and polished mechanically by Struers equipment then etched. Etching time was chosen experimentally for each sample. The samples after two extrusion passes were examined by JOEL JEM 1200 EX II transmission electron microscope. For TEM investigation thin foils were prepared. The foil preparation proceeded by rolling 3 mm in diameter roller,



a) b)

Fig. 1. The powder morphology – a) powder Fe ”M” , b) powder Fe ”D”

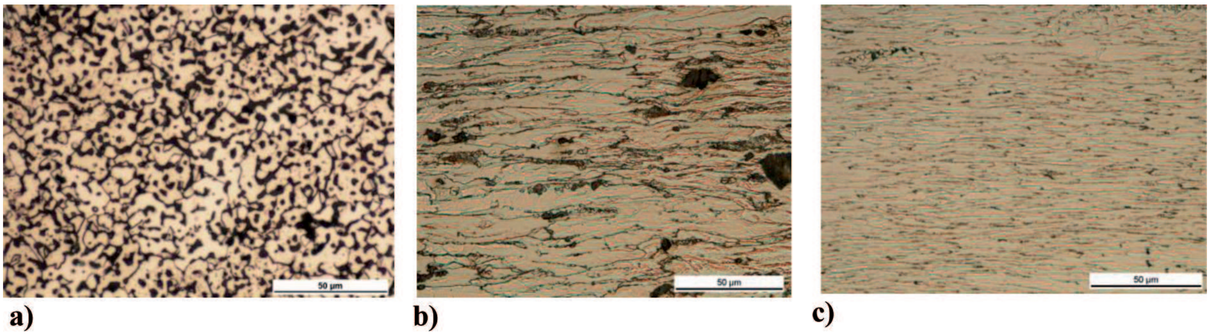


Fig. 2. The sinters' microstructure observed on longitudinal section by light microscope – a) sinter, b) sinter after one extrusion pass, c) sinter after two extrusion passes

cutting into slices, grinding and ion polishing. The pictures obtained using light and transmission electron microscopy were used for quantitative analysis of the microstructure. The Micrometer v 0.91_01b program, developed at the Materials Engineering Department, was used for the analysis.

The ultimate tensile strength, yield strength and elongation to failure were measured at room temperature. The tensile tests were carried out using Q-TEST/10 universal test stand with 10^{-3} s^{-1} strain rate. Vickers microhardeness tests were carried out using Zwick- HV0.2-15 hardness tester. Microhardeness was measured under 0.2 KG load, applied for 15 s on the polished samples across the transverse section. The impact toughness tests were carried out using RESIL 5.5 impact tester. The impact toughness test was evaluated at liquid nitrogen temperature. The sample size was 3x4x32 mm.

3. Results and discussion

3.1. Density

Theoretical iron density used for experiment is 7.8 g/cm^3. The results of samples' relative density measurement are shown in Table 2. The density increased after extrusion. Sinters which contain pores achieve lower mechanical properties due to stress concentrations presence.

TABLE 2
The samples average relative density compared with theoretical density or iron

	Sintered [%]	one pass extruded [%]	two pass extruded [%]
"FeM"	76.0	96.5	96.0
"FeD"	78.6	92.9	92.8

3.2. Microstructure

Microstructure (e.g. Fig 2 a-c) revealed by light microscopy shows a decrease in grain size and material porosity which had been confirmed by density measurement. The grains were elongated along the extrusion direction. The pores were also elongated. To characterize the microstructure and estimate the grain size reduction caused by hydroextrusion process, the samples after second extrusion pass were examined by TEM. The structure of material is presented in the Fig 3. The investigation revealed substantial deformation of the material, which is typical for materials deformed by severe plastic deformation methods. Elongated grains were observed in the microstructure.

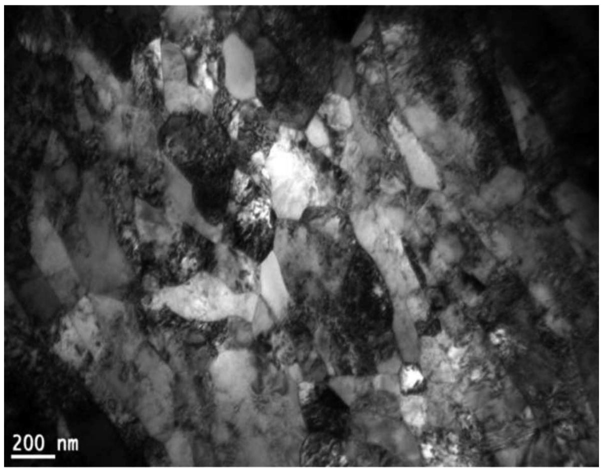


Fig. 3. The sinter microstructure observed on longitudinal section by TEM

The grains size were measured on the transverse samples section. After 2nd extrusion pass (true stain 2.3) average iron grains were around 180 ±81 nm. The values of the determined quantitative parameters for the microstructure of the investigated materials are shown in Table 3. The grain size decreased with the true strain increase. After hydrostatic extrusion the grain size was similar for both kinds of the samples.

TABLE 3

The results of grain size measurements

	d [μm]	CV
“FeM”_0HE	3.57	0.42
“FeM”_1HE	2.47	0.39
“FeM”_2HE	0.18	0.46
“FeD”_0HE	7.37	0.63
“FeD”_1HE	3.10	0.52
“FeD”_2HE	0.19	0.44

3.3. Mechanical properties

The mechanical properties were determined by three methods: Vickers microhardeness test, tensile test and impact toughness test.

The results of Vickers microhardness measurements are shown in Table 4. The microhardness increased with the true strain increase. The “FeM” iron achieved higher microhareness value than iron “FeD” after sintering as well as after extrusion. The deformation via extrusion caused an increase in microhardness of the samples.

TABLE 4

The Vickers microhardness

Microhardness ± standard deviation	Sintered [%]	one pass extruded [%]	two pass extruded [%]
“FeM”	108.14±15.2	248.4±8.1	270.2±10
“FeD”	104.63±31.4	241.9±11.6	256.8±11.3

Table 5 shows the tensile test results of the iron samples in three conditions: sintered, sintered and deformed by one HE pass and two HE passes. The results show an increase in strength of the extruded samples. The elongation to failure decreased with the true strain increase. The samples which were produced from fine-grain powders achieved higher yield and tensile strength than the samples produced from coarse-grained powders.

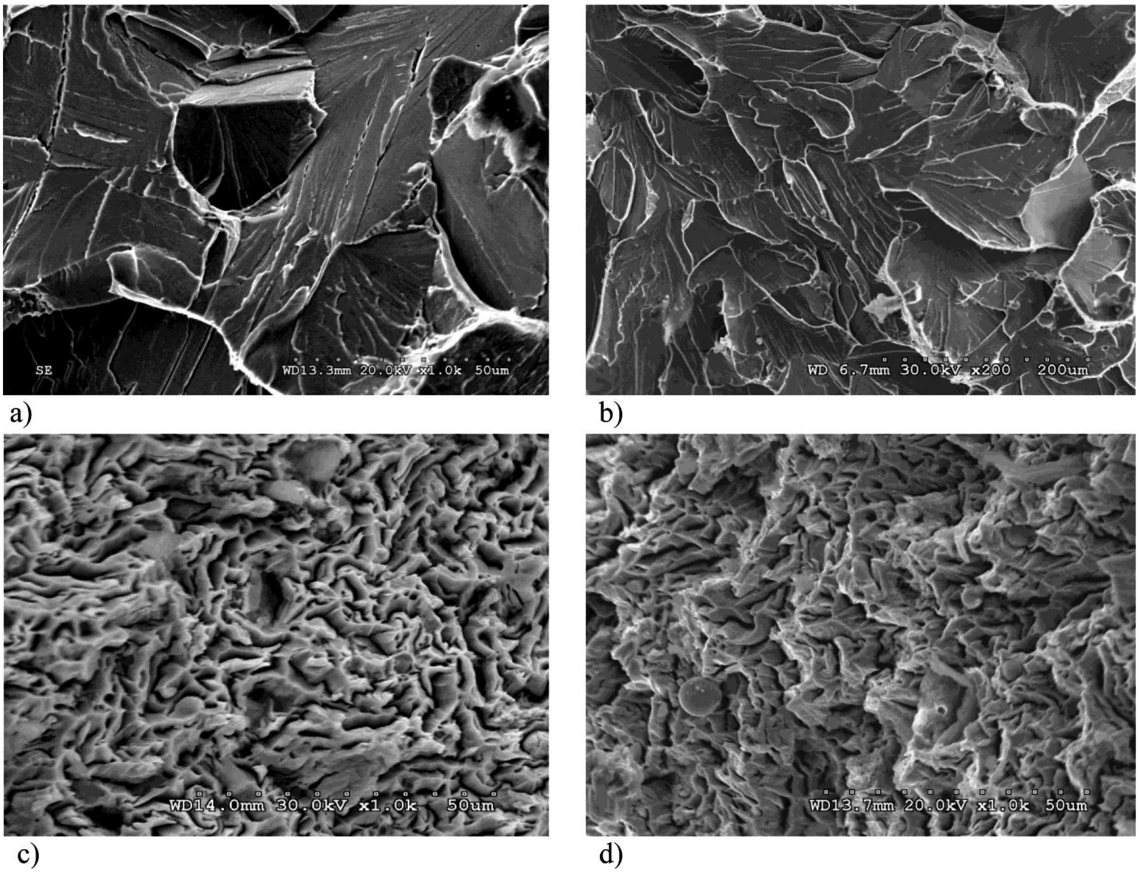


Fig. 4. The fracture surface of the samples after impact toughness test- a, b) iron Armco tested at liquid nitrogen temperature, c) iron sinter tested at liquid nitrogen temperature, d) iron sinter tested at room temperature

TABLE 5
Average Mechanical Properties of Samples

Sample ± standard deviation	Re [MPa]	Rm [MPa]	A [%]
"FeM"_0HE	137.75±8.01	177.77±0.01	4.29
"FeM"_1HE	779.70±22.56	815.74±30.65	1.68
"FeM"_2HE	851.77±56.57	855.95±56.72	1.32
„FeD"_0HE	73.71±8.91	78.24±8.75	0.60
„FeD"_1HE	380.18±21.76	383.63±21.27	0.06
„FeD"_2HE	431.42±45.90	433.93±45.48	0.06

The Charpy impact toughness tests of samples after extrusion process and of Armco iron samples were done. There were four samples from each state investigated. The results of tests carried out at liquid nitrogen temperature are shown in Table 6.

TABLE 6
The results from impact toughness test carried out at liquid nitrogen temperature

Sample ±standard deviation	KC [J/cm^2]
"FeM" _1HE	1.63±0.09
"FeM" _2HE	2.35±0.33
"FeD" _1HE	1.89±0.25
"FeD" _2HE	2.25±0.13
Armco	1.13±0.07

The fracture surface of coarse-grained iron samples broken at liquid nitrogen temperature usually is cleavage which (Fig. 4a-b). In our investigation the fracture surface of the ultra-fine-grained iron broken at room temperature and at nitrogen temperature was ductile (Fig. 4c-d).

4. Summary and conclusions

The iron samples in three states were investigated: sintered, sintered and deformed by one hydrostatic extrusion pass, and two hydrostatic extrusion passes. The density, microstructure analysis and mechanical properties were carried out to investigate the properties of the materials. The mechanical properties such as tensile strength, impact toughness and Vickers microhardeness were carried out at room temperature. Additionally impact tests were carried out at liquid nitrogen temperature.

The ultrafine-grained material was produced by the experiment. The final grain size obtained by sintering, followed by HE was about 180±81 nm. The grain size refinement was produced by hydro static extrusion. HE decreased the porosity of the material. This confirms that HE can be used for consolidation of powders materials. After hydrostatic extrusion process material density increased and ultrafine-grained structure was obtained. The ultrafine-grained structure caused the decrease in the mechanical properties such as yield strength and tensile strength at room temperature as well as impact toughness at liquid nitrogen temperature. The impact test carried out at -196°C showed transformation of the fracture mechanism from cleavage for coarse-grained structure to ductile for extruded material.

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REFERENCES

[1] J. Grosseau-Poussard, X. Milhet, C. Huvier, J.F. Dinhut, J. Mater. Process. Tech. **205**, 151-159 (2008).
[2] H. Li, J. Zhang, Y. Pei, Z. Zhao, Z. Ma, J. Iron. Steel. Res. Int. **18**, 11-15 (2011).
[3] D. Poquillon, V. Baco-Carles, Ph. Tailhades, E. Andrieu, Powder Technol. **126**, 75-84 (2002).
[4] L.M. Tham, L. Su, L. Cheng, M. Gupta, Mater. Res. Bull. **34**, 71-79 (1999).
[5] S. Lee, C.Y. Yoon, H.J. Park, S.S. Kim, E.J. Kim, T.H. Choi, K.Y. Rhee, J. Mater. Process. Tech. **191**, 396-399 (2007).
[6] L.M Tham, M. Gupta, L. Cheng, Mater. Sci. Eng. A **326**, 355-363 (2002).
[7] L. He, L.F. Allard, E. Ma, Scripta Mater. **42**, 517-523 (2000).
[8] M. Andasmas, P. Langlois, N. Fagnon, Th. Chauveau, A. Hendaoui, D. Vrel, Powder technol. **207**, 304-310 (2011).

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