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ROLE OF MICROSTRUCTURE DISCONTINUITIES IN THE SOFT MAGNETIC COMPOSITES WITH ALUMINIUM ALLOY ADDITION

ROLA NIEJEDNORODNOŚCI MIKROSTRUKTURY W MAGNETYCZNIE MIĘKKICH KOMPOZYTACH Z DODATKIEM STOPU ALUMINIUM

The paper focuses on the effect of both the aluminium alloy addition and microstructural inhomogeneities on the magnetic behaviour of soft magnetic materials tested at low frequencies. The IIPC material (base on the commercial Somaloy 1P powder) has been blended with different amounts of commercially aluminium alloy Alumix 321 (0, 5 and 10 wt %). Specimens with a different green density were obtained by pressing at a pressure in the range from 400 to 800 MPa. Different thermal treatments (in air or nitrogen at the maximum temperature of 500°C for 30 min) were carried out on the evaluated systems. The microstructure investigation revealed that for materials with high aluminium alloy contents, pores are located nearby or around the aluminium alloy particles. The heat treatment regime resulted in a coarse-grained structure with a small number of inclusions within the grains and at the grain boundaries. The comparison of the results indicated that the magnetic properties were considerably dependent on the microstructural inhomogeneities.

Keywords: Soft Magnetic Composites, Insulated Iron Powder Compound, Aluminium Alloy, Coercivity, Specific Losses

Przedmiotem pracy jest wpływ dodatku stopu aluminium oraz niejednorodności mikrostruktury na właściwości magnetyczne magnetycznie miękkich materiałów badanych przy niskich częstotliwościach. Materiał IIPC (na bazie komercyjnego proszku Somaloy 1P) został zmieszany z różnymi ilościami komercyjnego stopu aluminium ALUMIX 321 (0, 5 i 10% wag.). Próbki o różnych gęstościach nasypowych prasowano stosując ciśnienie w zakresie od 400 do 800 MPa. Zastosowano różne obróbki cieplne (w powietrzu i azocie) ustalając maksymalną temperaturę 500°C. Badania mikrostruktury pokazały, że dla materiałów o wysokiej zawartości stopu aluminium, pory są w pobliżu lub otaczają cząstki stopu aluminium. W wyniku obróbki cieplnej tworzy się gruboziarnista struktura z minimalną liczbą wtrąceń wewnątrz ziaren i na ich granicach. Porównanie wyników pokazuje, że właściwości magnetyczne zależą w znacznym stopniu od niejednorodności mikrostruktury.

1. Introduction

Soft magnetic materials (SMM) are relatively new materials in electromagnetic application [1, 2]. SMM consisted of heat-treated powder compacts formed by basically pure iron powder particles coated with a very thin electrically insulated layer. These materials have become increasingly popular during the last years due to several advantages, such as weight and size reduction. Weight can be reduced through several types of technology improvements: in materials, design techniques and fabrication processes. To establish the design rules, attention has to be paid to the electromagnetic loss characteristics of SMM. Additionally, insulated iron powder compounds (IIPC) offer several advantages over traditional materials in some applications; for example, the isotropic nature of the SMM combined with the unique shaping possibilities opens up for 3D-design solutions [3-10].

The summary of the advantages of IIPC parts include [11-16]: the ability to produce complex shapes to net shape

without waste of material, and the ability to tailor the magnetic properties to the specific application by controlling the material and the processing parameters. IIPC cannot be sintered, since each particle has to be electrically insulated from the other one. Nevertheless, since during compaction in the particles are subjected to stress, which deteriorates the soft magnetic properties, a heat treatment has to be applied to provide a stress relief. Magnetic properties of the IIPC are influenced by the amount and type of polymer and the particle size distribution of the iron powder. Iron powder polymer composites are used in the as compacted condition and exhibit low eddy current losses. Applications for these materials are AC magnetic devices that require the minimization of eddy current losses. One drawback of the iron powder polymer composites is the high coercive force of the as pressed component. This high coercive force increases the hysteresis losses dramatically, resulting in reduced magnetic performance at low frequencies.

The goal of the present paper is to study the possible improvement of mechanical properties of IIPC parts by means

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of the introduction of an aluminium alloy into the powder mix. Since the chosen aluminium alloy presents a sort of pre-sintering behaviour at 500°C, with the possibility of viscous flow around the IIPC, the heat treatment applied aims at providing an increase in the mechanical behaviour of the material, with a final good rigidity after the cooling process.

2. Experimental procedures

The IIPC material (commercial Somaloy 1P powder, Höganäs) has been blended with different amounts of aluminium alloy (commercial ready-to-press aluminium based powder Alumix 321 (Al - 0.95 wt. % Mg - 0.49 wt. % Si - 0.21 wt. % Cu - 0.07 wt. % Fe - 1.6 wt. % lubricant), Ecka Granules).

Powder mixtures were homogenized using a laboratory Turbula mixer for 20 min. Specimens with a different green density obtained using a 2000 kN hydraulic press, in a disc-shaped mould (Φ 40 mm) and unnotched impact energy $55 \times 10 \times 10 \text{ mm}^3$ specimens applying a pressure in the range 400, 600 and 800 MPa. Different thermal treatments (in air and in nitrogen at the maximum temperature of 500°C for 30 min) were carried out on the evaluated systems.

Both microstructure and porosity were investigated. The porosity was classified with the use of a Leica Qwin image analysis system with respect to area and mean diameter of the pores. For the determination of porosity characteristics 100x magnification were used. Densities were evaluated using the water displacement method (Archimede's principle), according to the ASTM B962 – 08 standard.

The system for the magnetic characterization is described in [17]. A power supply capable to produce sinusoidal waveforms up to 2 kHz has been adopted (Pacific supply, model: 360-AMX). This is critical aspect in order to correctly determine the core losses due to fundamental sinusoidal magnetization only, without considering any hysteresis and eddy current extra loss contributions due to the harmonic content.

3. Results

The soft magnetic composites reveal a different microstructure in terms of the pores. Numerous pores with different pore size diameters are found as well as pore clusters, mainly in the specimens mixed with aluminium alloy addition. A comparison among metallographic sections can be seen in following Figs. 1-9. In following Fig. 1 up to Fig. 9, showing different processing conditions, pores are easily detectable in the microstructures evidenced in black color, the IIPC matrix is in white color and aluminium base particles are in grey color.

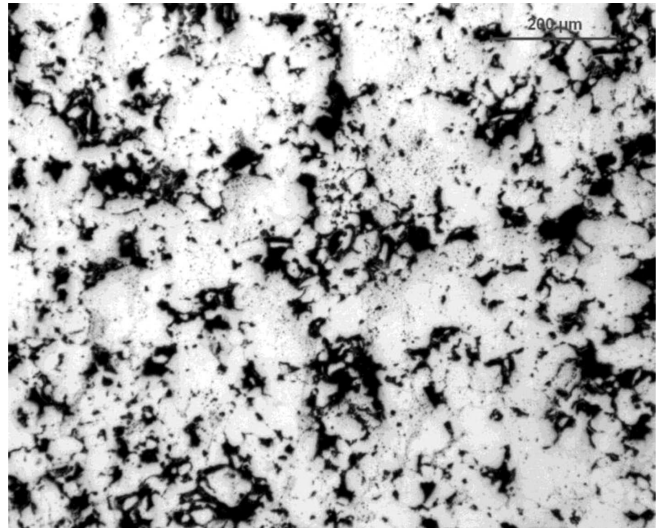


Fig. 1. Microstructure of IIPC system, pressing at 400 MPa

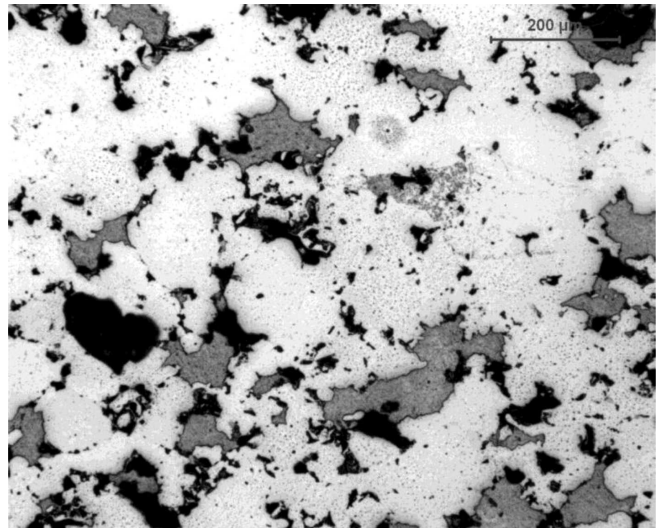


Fig. 2. Microstructure of IIPC + 5% Alumix 321 system, pressing at 400 MPa

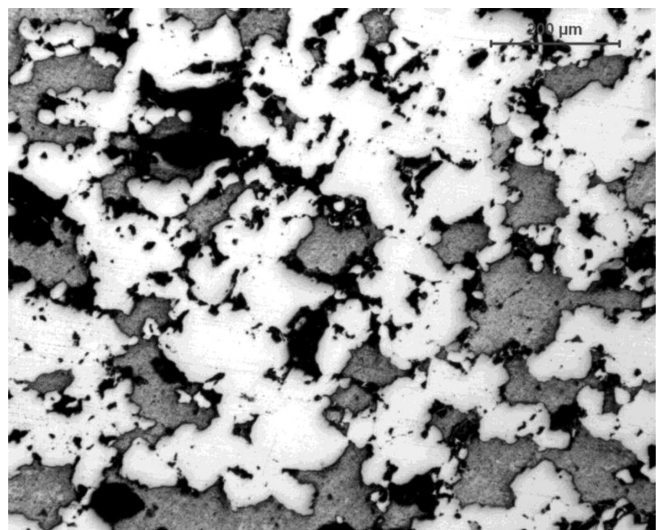


Fig. 3. Microstructure of IIPC + 10% Alumix 321 system, pressing at 400 MPa

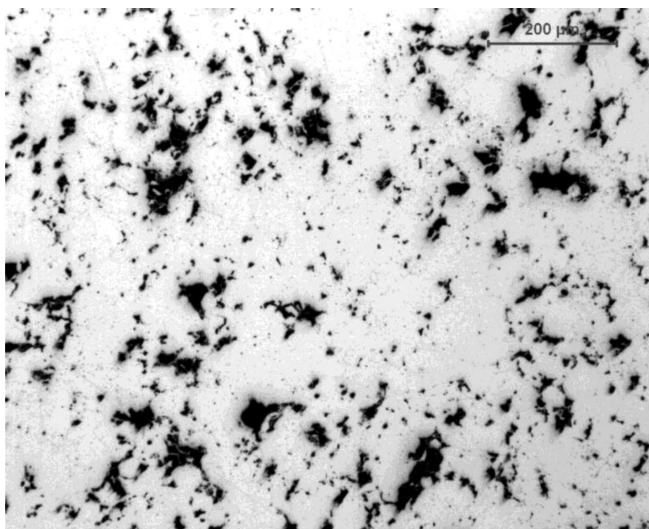


Fig. 4. Microstructure of IIPC system, pressing at 400 MPa and heat treated in air

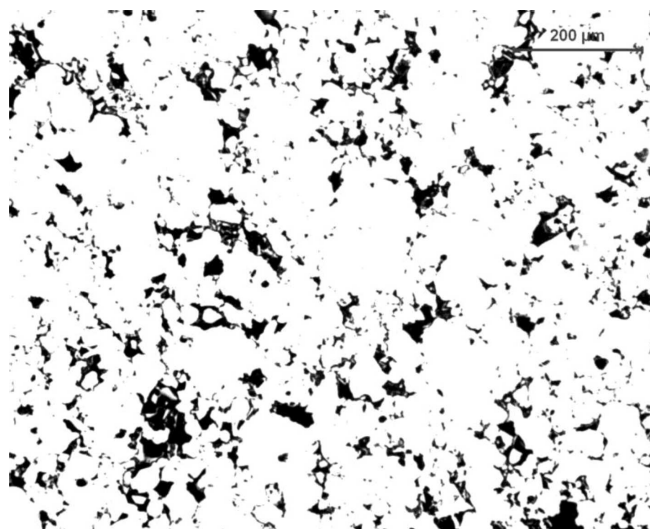


Fig. 7. Microstructure of IIPC system, pressing at 400 MPa and heat treated in nitrogen

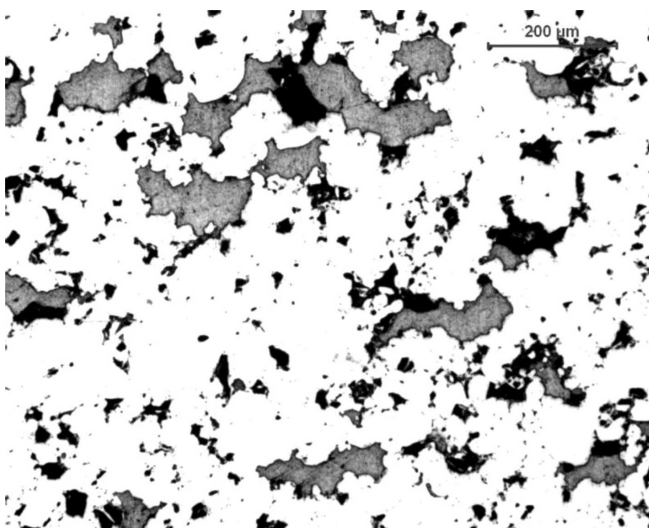


Fig. 5. Microstructure of IIPC + 5% Alumix 321 system, pressing at 400 MPa and heat treated in air

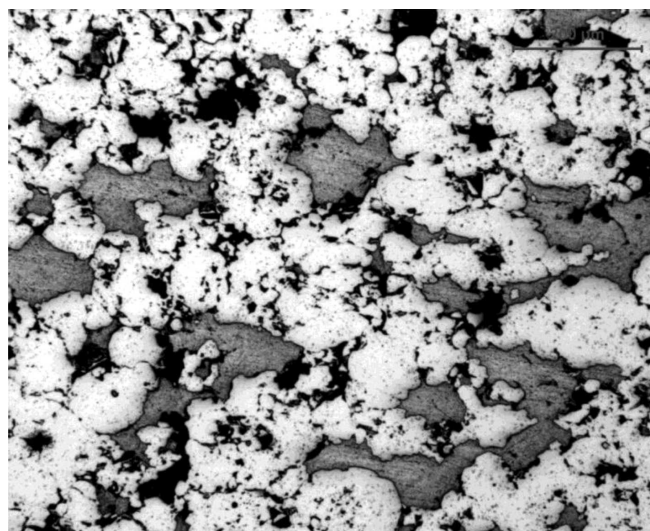


Fig. 8. Microstructure of IIPC + 5% Alumix 321 system, pressing at 400 MPa and heat treated in nitrogen

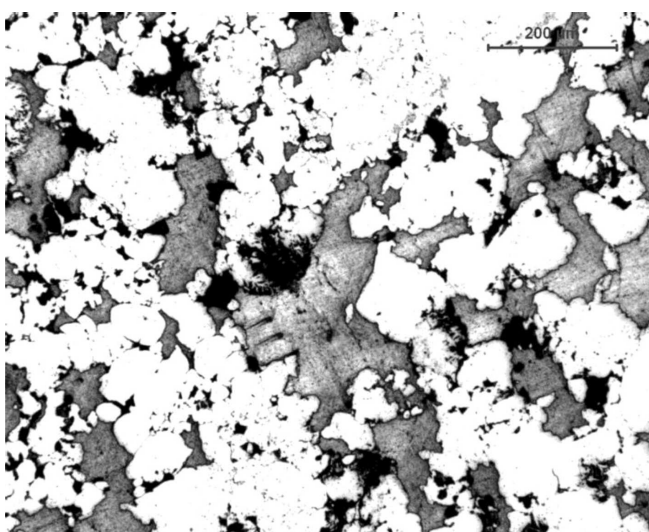


Fig. 6. Microstructure of IIPC + 10% Alumix 321 system, pressing at 400 MPa and heat treated in air

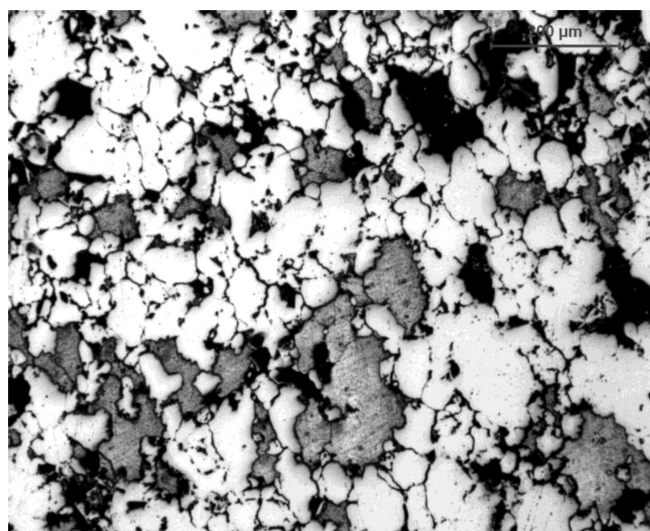


Fig. 9. Microstructure of IIPC + 10% Alumix 321 system, pressing at 400 MPa and heat treated in nitrogen

The microstructure, shown in Fig. 4, referring to the material in the green state, reveals the outlines of the original IIPC powder particles and the pores. In the pressed state, pores act as crack initiators and due to their presence the distribution of stress is inhomogeneous across the cross section and leads to the reduction of the effective load bearing area.

The lower pressing pressure (400 MPa) creates flattened pores that subsequently contribute to anisotropic dimensional change during heat treating, as visible in Fig. 5 and Fig. 6, referring to the introduction of aluminium based powders into the mix. For materials with higher aluminium contents, pores located nearby or around the aluminium alloy particles.

Heat treatment regime results in a coarse-grained structure with some inclusions within the grains and at the grain boundaries. In terms of bonding evolution between the adjacent particles, the microstructures analyses revealed that heat treatment of aluminum alloys are more effective when carried out in nitrogen. The following Fig. 10 shows the effect of the applied pressing pressure on the final porosity level within the different produced samples, in the as-pressed state and after the heat treatments in air and nitrogen.

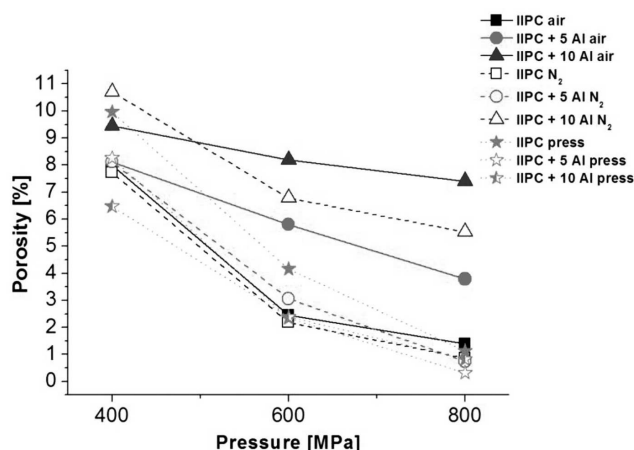


Fig. 10. The porosity differences in the investigated systems

For the materials treated in nitrogen the porosity is lower than for the structures treated in air. The amount of pores seems to be increasing with increasing aluminium alloy additions, the worst results deriving in case of the addition of 10 wt. % of aluminium alloy.

The combined evaluation of microstructure and porosity shows that the treatment in nitrogen can better affect structure homogeneity as well as pore size distribution if compared to the treatment in air.

Fig. 11 presents the dependence of density on the applied processing conditions.

The summary of magnetic properties is given in the TABLE 1.

The coercivity and the remanence tend to increase with increasing applied pressure; this is also attributed to the enhanced densification and promotes porosity reduction. The addition of aluminium alloy, however, it determines a decrease of remanence and an increase in coercivity. Comparison of the results indicates that the magnetic properties are considerably dependent on the structural state of the alloy. Several authors [15, 18-20] underline that the behaviour of powders during

the pressing and heat treatment is an important issue to get a suitable magnetic properties.

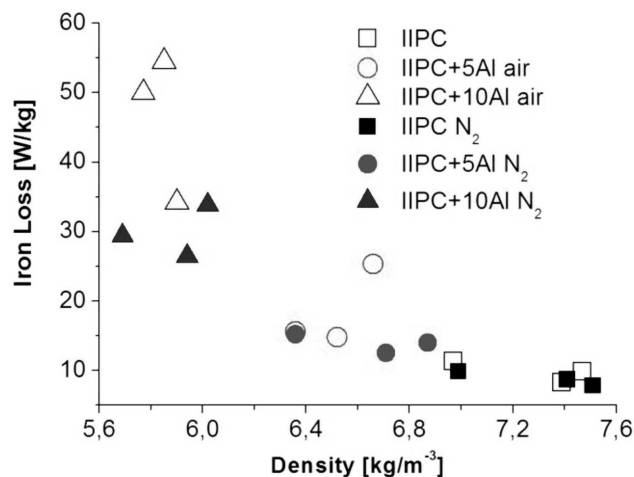


Fig. 11. The dependence of density on the applied processing conditions

4. Discussion

TABLE 1 shows the magnetic properties of studied materials. As previously pointed out, in the investigated microstructures some discontinuities occur, such as interconnected and residual porosity. It is well-known that porosity has a negative effect on the magnetic properties such as the magnetic induction and permeability as well the coercive force.

The analysis of porosity reveals a difference between the materials processed with air and nitrogen atmosphere.

The detail examination revealed that the interface between adjacent particles even after compaction and heat treatment are partially contaminated with impurities such as oxide and/or nitride. Therefore the grain boundary segregation gives rise to weakening of sinter necks.

Aluminium oxides (mainly for the material treated in air) and aluminium nitrides (mainly for the material treated in nitrogen) spreading between IIPC matrix were detected. Magnesium, one of the elements in the considered aluminium alloy, can work as a reducing agent in this system, even at the solid state. The presence of magnesium had a positive impact on the heat treatment process [21, 22], which includes reducing the oxide. This promotion mainly comes from its reductive reaction with the oxide on Al surface to form spinel ($MgAl_2O_4$). Schubert et al. [23] underline that segregation process of magnesium was already revealed at 400°C. Moreover, revealed incorporation of nitrogen atoms enhances the destruction of Al_2O_3 by potential formation of intermediate oxy-nitrides and subsequent AlN (the aluminium alloy powder particle surfaces decorated with some rosette-like precipitations).

Aluminium oxides and/or nitrides act like mechanical barriers to plastic deformation and prevent further interlocking of aluminium alloy particles. Therefore, the lack of metallic interparticle bonding was evident.

The different type of inclusions present in the microstructures treated in air or nitrogen confirms these assumptions. It is evident that heat treatment conditions strongly influence

TABLE 1

The summary of magnetic properties

Pressure	Material	Density	Core Loss	Remanence	Coercivity
[MPa]		[kg/m ⁻³]	[W/Kg]	[T]	[A/m]
	Pressing				
400	IIPC	7,02	10,33	0,017	189
600	IIPC	7,41	7,85	0,027	209
800	IIPC	7,55	7,59	0,032	217
400	IIPC+5 wt. % Al alloys	6,36	12,8	0,01	194
600	IIPC+5 wt. % Al alloys	6,66	17,93	0,011	289
800	IIPC+5 wt. % Al alloys	6,52	27,48	0,019	422
400	IIPC+10 wt. % Al alloys	5,77	36,26	0,003	333
600	IIPC+10 wt. % Al alloys	5,9	62,66	0,007	399
800	IIPC+10 wt. % Al alloys	5,87	34,22	0,01	331
	Pressing and heat treatment in air				
400	IIPC	6,97	11,3	0,021	184
600	IIPC	7,39	8,3	0,027	205
800	IIPC	7,47	9,85	0,023	218
400	IIPC+5 wt. % Al alloys	6,36	15,6	0,007	196
600	IIPC+5 wt. % Al alloys	6,52	14,8	0,012	220
800	IIPC+5 wt. % Al alloys	6,66	25,3	0,023	312
400	IIPC+10 wt. % Al alloys	5,77	50	0,005	227
600	IIPC+10 wt. % Al alloys	5,85	54,5	0,006	310
800	IIPC+10 wt. % Al alloys	5,9	34,2	0,01	331
	Pressing and heat treatment in nitrogen				
400	IIPC	6,99	9,85	0,024	164
600	IIPC	7,41	8,7	0,02	230
800	IIPC	7,51	7,8	0,032	234
400	IIPC+5 wt. % Al alloys	6,36	15,2	0,007	165
600	IIPC+5 wt. % Al alloys	6,71	12,5	0,014	209
800	IIPC+5 wt. % Al alloys	6,87	14	0,015	265
400	IIPC+10 wt. % Al alloys	5,69	29,4	0,005	182
600	IIPC+10 wt. % Al alloys	5,94	26,4	0,006	267
800	IIPC+10 wt. % Al alloys	6,02	33,8	0,01	312

the pore size distribution. It is possible to explain the role of the pore sizes as follows: the degrees of local stress multiplication close to pore boundaries and between pores depend on pore shapes in relation to the direction of principal stress and on the degree of overlap of stress fields arising from neighbouring pores. The main reasons for which differences occur among the presented soft magnetic composites mainly derive from the pore clusters. Pore clusters arise with addition of aluminium alloy to the IIPC matrix and strongly depend on the atmosphere purity. It means that inclusions can greatly influence the pore size and shape distribution, mainly in interparticle bonding evolution, the strength of which can be decreased markedly by grain boundary segregation.

The application of increasing pressing pressure increases the produced dislocations and stress fields at the part surface. Moreover, it can disrupt the coating layer covering the original iron powder particles. Higher compacting pressures cause a greater volume reduction and, consequently, a sensible decrease of present porosities, typically resulting in a demagnetization field effect. The movement of domain boundaries is impeded by microstructural discontinuities, such as porosity, grain boundaries, oxides, nitrides as well as stress fields.

5. Conclusions

The results showed that:

1. The increase of aluminium alloy influences the pores distribution, so that for higher alloy addition pores are located nearby or around the aluminium alloy particles. The detail examination revealed that the interfaces between adjacent particles are partially contaminated with impurities such as oxide and/or nitride. Therefore, the grain boundary segregation gives rise to weakening of sinter necks.

2. The coercivity and the remanence increased with increasing applied pressure. This effect can also be attributed to the enhanced densification and promote porosity reduction. However, the subsequent decrease of remanence and increase of coercivity are observed in terms of addition of aluminium alloys.

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