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Characteristics of Al-Fe sinters made by the powder metallurgy route

Charakterystyka spieków Al-Fe wytworzonych metodą metalurgii proszków

Abstract
The Al/Fe material was prepared by the powder metallurgy route with an additional intermediate stage which was a centrifuge of powder mixture. The application of the centrifuge stage was applied to obtain circular phase distribution of Al-rich phases in a sintered material. Iron powder with a particle size under 100 µm and aluminum powder with a particle size of about 25 µm, were used as starting materials. To determine the effect of centrifuging time on the distribution of Fe-Al particles, scanning electron microscopy (SEM, EDS) and XRD techniques were used. Microstructure observations show the influence of the centrifuging time on the distribution of Fe particles. It was observed that a longer centrifuging time caused changes in the ratio concentration of elements and allowed the growth of the intermetallic phase at the interface between solid Al and Fe particles.

Keywords: Al/Fe sinters, powder metallurgy, interface, intermetallic compounds, microstructure

Streszczenie
Spiekane materiały Al/Fe wytwarzano metodą metalurgii proszków z wprowadzeniem etapu pośredniego w postaci odwirowania mieszaniny proszku. Etap wirowania miał na celu uzyskanie kulistego rozkładu faz bogatych w Al w spiekanym materiale. Proszek żelaza o wielkości cząstek poniżej 100 µm i proszek aluminium o wielkości cząstek około 25 µm zastosowano jako materiały wyjściowe. W celu określenia wpływu czasu wirowania mieszanki na rozkład faz w wytworzonym materiale zastosowano techniki skaningowej mikroskopii elektronowej (SEM, EDS) i dyfraktometrii rentgenowskiej (XRD). Obserwacje mikrostruktury wykazały istotny wpływ czasu wirowania na mikrostrukturę uzyskanych spieków. Zaobserwowano, że dłuższy czas wirowania spowodował zmianę stosunku stężenia pierwiastków i pozwolił na wzrost fazy międzymetalicznej na granicy faz między stałymi cząstkami Al i Fe. Badania mikrotwardości obszarów spiekanego materiału świadczą o zwiększeniu twardości w obszarach nowo utworzonych faz międzymetalicznych.

Słowa kluczowe: spieki Al/Fe, metalurgia proszków, interfejs, związki międzymetaliczne, mikrostruktura

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1. Introduction

One of the most in demand engineering materials are aluminum matrix composites (AMCs) especially because of their unique attributes such as physical and mechanical properties, light weight and wide range of applications in the automotive and aerospace industries. All the properties of the material produced are connected with elemental material properties, the volume of the reinforcing phase, processing technique and parameters. The production of specific elements by means of the powder metallurgy (PM) route requires basic knowledge about the starting materials from researchers and designers, as well as an awareness of the specific interactions between connected components. The most common steps in this specific technique are mixing of fundamental powders, green compact production and sintering. All these steps have a direct effect on the density of the final product. Furthermore, this technology enables a wide selection of various chemical compositions, eliminates the problems of grain coarsening and inhomogeneity of chemical composition, and reduces the use of raw elements [1–3].

Materials like iron-aluminides are continually researched because of the rich amount of intermetallic compounds which can be obtained when applying the appropriate production conditions and participation ratio of Al:Fe. The production technology of Al:Fe elements, based on powder metallurgy, eliminates a number of limitations in the manufacturing process. However, the use of the PM technique inhibits phase transitions in the sintered material: the ratio of elements changes locally and depends on the degree of homogeneity of the mixture and the size of the powder particles of the starting material [4–7].

2. Experiments

As a starting material, a mixture of aluminum and iron powder was used. The aluminum powder particles had an average particle size of 25 μm and irregular shape which was obtained in a commercial atomization process (Fig. 1a). Iron particles had an average particle size of under 100 μm and an irregular shape with a sponge-like microstructure and large surface area (Fig. 1b), characteristic for the method of production [8].

a) b)

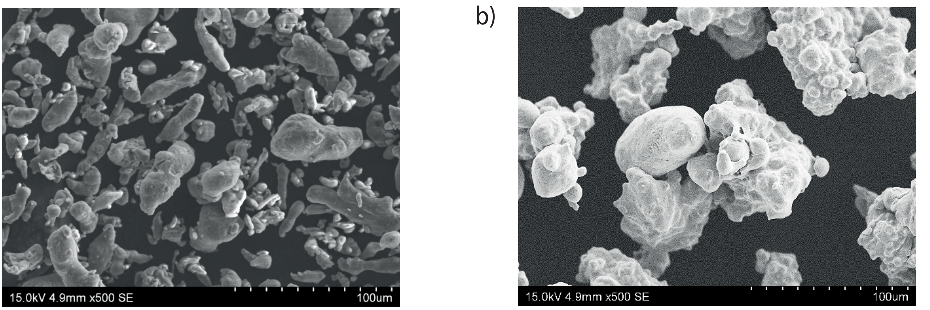

Fig. 1. Morphology of starting powders (SEM): a) aluminum; b) iron

Powders in 1:1 volume ratio were mixed for 20 minutes to obtain the desired homogenization and remove any clusters of powders (Fig. 2).

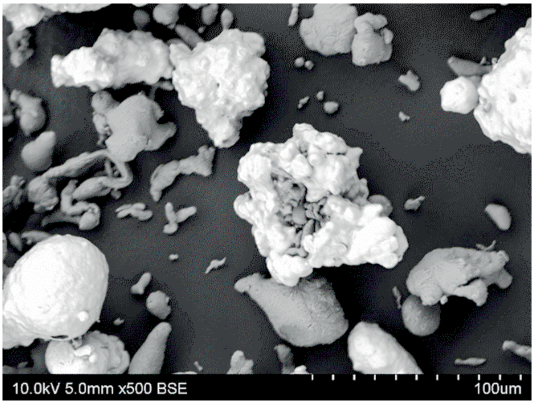

Fig. 2. Morphology of Al Fe powder mixture (SEM)

The Al-Fe mixture of powders was placed in a sleeve and centrifuged for 0 (material 1), 10 (material 2) and 20 minutes (material 3) respectively at 1200 rpm. It was an additional element aimed at obtaining a gradient distribution of particles. In the next step, the Al-Fe mixture was cold compacted at room temperature in a uniaxial press at 400 MPa. Sintering of all the green compacts was carried out using a ceramic furnace in a vacuum at 600°C for 6 hours. The process diagram is shown in Figure 3.

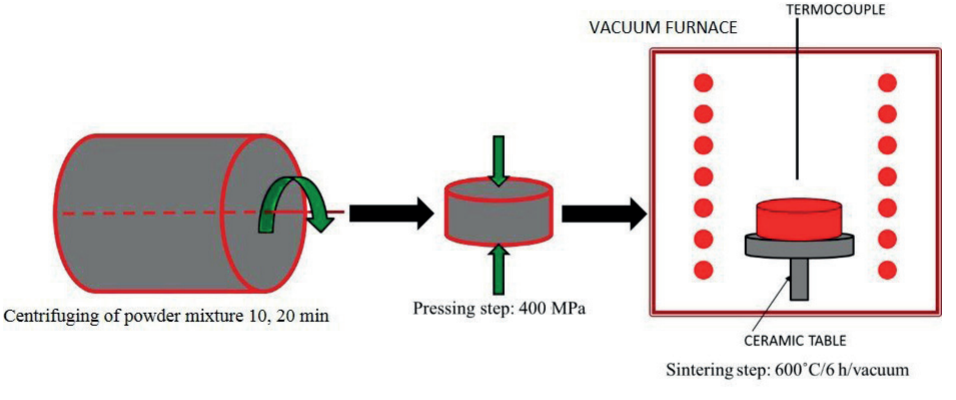

Fig. 3. Various stages of Al-Fe material production

X-ray diffraction (XRD) analysis was carried out on a diffractometer Bruker Discover D8 Advance with Ni filter and Cu target, Kα radiation. Densities of the composites were measured by using the Archimedes' method and compared with the theoretical densities to obtain varying degrees of densification. The microstructure of the prepared composites was examined by both an optical model OLYMPUS GX microscope and a scanning electron microscope (SEM, model: HITACHI S-3400N). The elemental analysis of the specimens was performed using an energy dispersive spectroscopy (EDS) microanalyses equipped on scanning electron microscope (SEM).

3. Results and discussion

3.1. Microstructure observations

As shown in Figure 4, different microstructures were obtained for the samples with different centrifuging times. In Figure 4a, the microstructure of the sample is presented, with the homogeneous dispersion of Al-Fe powders. The material which was centrifuged for 10 minutes and sintered (Fig. 4b), contains three areas: mixture of sintered Al-Fe particles in the outer and inner part of the sample, and the area rich in the newly-formed phase in the middle part of the samples.

For specimens which were centrifuged for 20 minutes and sintered (Fig. 4c), two areas were observed: Al-Fe particles in the outer of the sample, and the area rich in the newly-formed phase in the middle part of the sample.

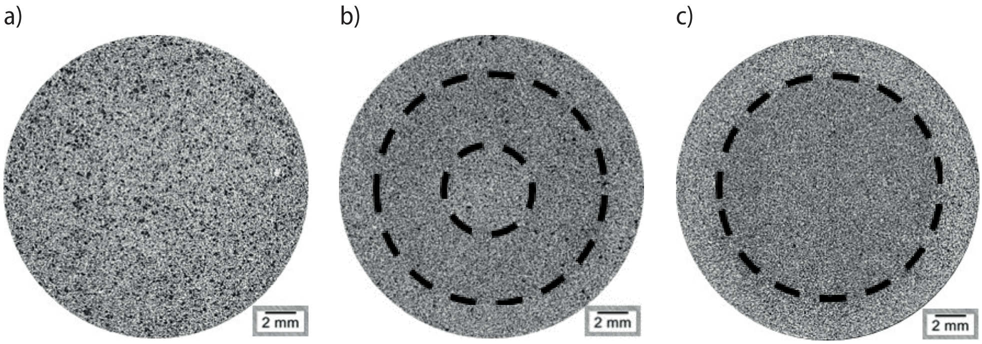

Fig. 4. Microstructure changes after sintering of samples depended on the concentration of each elements and centrifuging process of: a) 0 min; b) 10 min; c) 20 min

Observations performed under SEM microscopy revealed (Fig. 5) that material 1 contained three areas: white – which is the iron phase (Tab. 1 pt. 1), grey dark areas – Al phase (Tab. 1 pt. 2), and black spots which are the porosity. If we compare them with the materials obtained after the centrifuging and sintering process, it is clearly visible that a newly-formed phase occurs (Fig. 6). The EDX analysis revealed (Tab. 2, pt. 3) \ ~72 at.%

aluminum and ~28 at.% of iron, corresponding to the phase Fe_2Al_5 which has the highest cohesion energy among the phases of the Al-Fe system and its formation is most likely under these conditions.

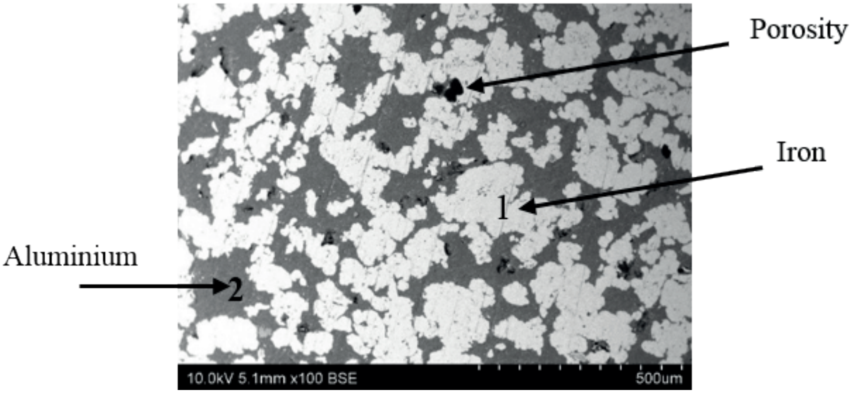

Fig. 5. Microstructure of Al-Fe material without the centrifuging process (SEM)

Table 1. Results of EDX analysis of point shot for Figure 5

pt.	Al [at.%]	Fe [at.%]
1	–	100
2	99.42	0.58

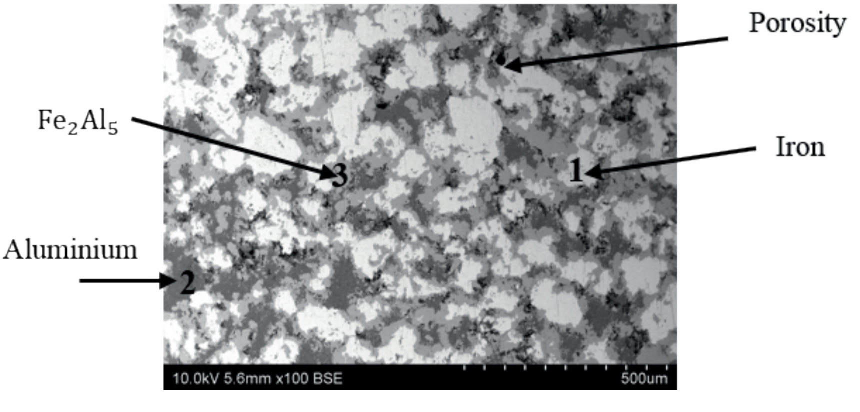

Fig. 6. Microstructure of Al-Fe material centrifuged 20 min and sintered – middle part of the samples (SEM)

Table 2. Results of EDX analysis of point shot for Figure 6

pt	Al [at.%]	Fe [at.%]
1	0.34	99.66
2	98.59	1.41
3	72.04	27.96

3.2. XRD analysis

X-ray diffraction (XRD) analysis confirms that in areas rich in aluminum and iron phase was observed under SEM microscope and EDX, no new phase occurs (Fig. 7).

For the materials obtained by centrifuging and sintering method and where the phase of Fe_2Al_5 was defined, the XRD measurement confirmed its occurrence as shown in Figure 8.

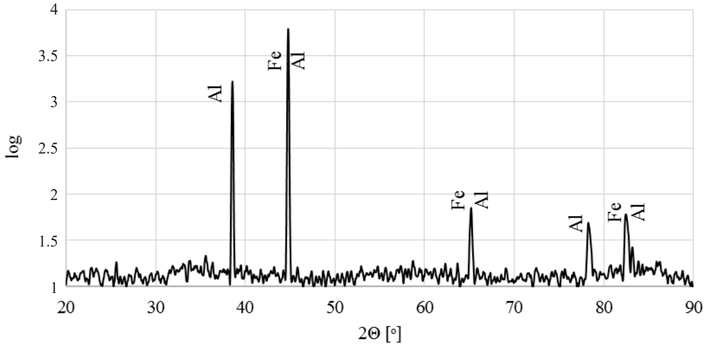

Fig. 7. Results of phase composition analysis of the centrifuged sample (20 min) outer part of the sample

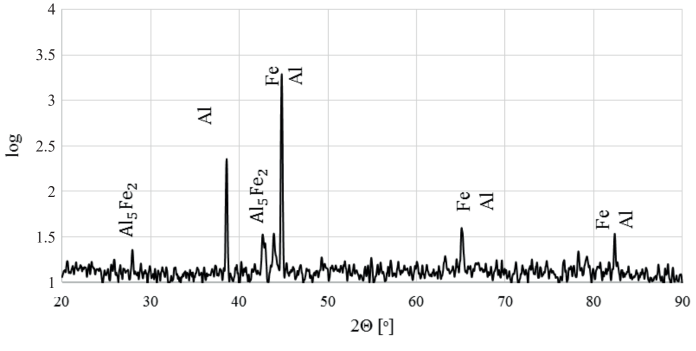

Fig. 8. Results of phase composition analysis of the centrifuged sample (20 min) middle part of the sample

3.3. Modelling development

From the application point of view (e.g. drills) the best microstructure would be a hard outer side of the sample and a ductile center. It means that Fe concentration should be at the maximum at the edge of the sample, as shown in Figure 9. The full line in Figure 9 shows measured Fe concentration in the investigated samples. The dashed line in Figure 9 shows the desired concentration of Fe.

In order to predict the best microstructure according to Figure 9, a model was developed. It was a 1D iterative model where the assumption was made that Fe particles can move inside an Al matrix. However, the Al matrix can hamper the movement of the smallest Fe particles. The movement of the Fe powder particles leads to the formation of a ring (rich with Fe_2Al_5 intermetallic phase) around the sample center, as seen in the pictures above (Fig. 4).

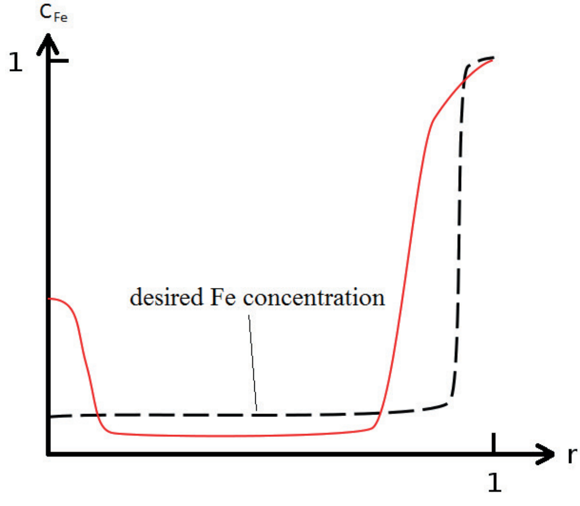

Fig. 9. The diagram showing the desired Fe concentration

In this model, a distance (*r*) from the center of a sample of Fe particle is a function of its mass (*m*), rotation speed (ω), actual distance to center (*ri*), centrifuging time (*tc*) and contraction force of Al matrix (FeAl):

$$ri + 1 = G(m, \omega, ri, tc) - FmAl \tag{1}$$

The FeAl parameter in Equation (1) was a fixed value based on the experimental observations. The results of the simulations are shown in Figure 10.

During the simulations, 1000 Fe particles were generated with a random distance to the center of sample (*r*). In the calculations there was fixed rotation speed of the process which was 600 rotations per minute. The contraction force was equal to $1.5 \cdot 10^{-10}$ N. In the present investigation, three variants were considered. Variant A where Fe particles had size greater than 100 μm, variant B where Fe particles had size between 100 μm and 40 μm, and variant C where Fe particles had size below 40 μm. The results of simulations for A, B and C cases of Fe particles size are shown in Figure 10. It is seen that for case C, there is no desired concentration of Fe particles. Case B exhibits the lowest concentration of Fe particles in the center part of the samples than cases A and C but, in case A, the outer part of the sample, while rich in Fe particles, is also the thinnest.

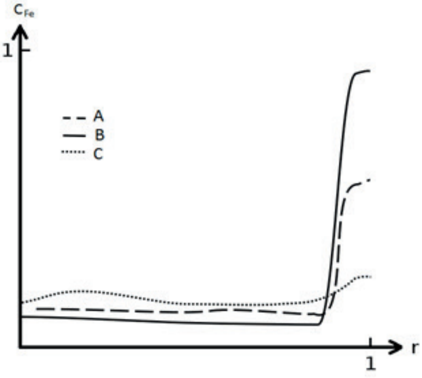

Fig. 10. The results of simulations for A, B and C case of Fe particles size

4. Conclusions

The proper selection of a mixture of Fe particle size affects the speed of movement of the particles in the powder mixture.

Parameters of the centrifugation process have a significant influence on the concentration of elementary components.

Al-Fe composite material with gradient distribution of intermetallic phase was obtained after centrifugation process, pressing and sintering in a vacuum.

The rings shown in Figure 4 appear to be intermetallic compounds, this may be caused by changes in the concentration of Fe/Al starting components.

The simulations show that using the developed model the distribution of Fe particles in samples can be predicted. However, the model still needs some improvements.

Acknowledgements

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