Fabrication of Fe / Al$_2$O$_3$ composite foam by sintering techniques

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Abstract. Composite foams of iron and aluminium oxide were prepared by sintering techniques. Al$_2$O$_3$ microsphere powders added during Fe foam formation were used as starting materials. Samples containing various compositions were prepared (Al$_2$O$_3$ microspheres bonding in Fe foam). The metallic porous structure was obtained by sintering on the basis of iron-based powder mixtures with the addition of a copper catalyst and Fe (III) oxide as a space carrier. Sintering was carried out at 1130°C and 50 minutes long, in a dissociated protective gas with ammonia. Observation of the microstructure and phase identification of the synthesized samples was carried out using optical microscopy (OM), scanning electron microscopy (SEM) and energy dispersion spectroscopy (EDS). The porosity of the Fe / Al$_2$O$_3$ composite products for the three different compositions was measured by means of the Cavalleri principle on transverse samples prepared from sintered materials.

1 Introduction
Metallic porous materials are a new class of engineering materials that are dynamically researched due to their innovative and useful properties [1-6]. The increase of interest in the porosity of materials was influenced by understanding of porous structures can be found in living organisms, for instance human bones or wood morphology, where there is unusual combination of properties: high rigidity with minimal weight. The development of metallic foams (as high porosity materials, ranging from 40% to 98% by volume) began more than 20 years ago [7]. These unique materials combine properties such as energy absorption, fluid permeability, wavelength, low thermal conductivity, and good insulating properties. They can be used as: kinetic energy absorbers, fluid filters and impurities, porous welding electrodes, high temperature sealers, heat exchangers [8 - 10]. As the field of metallic foams application dynamically grows, there are many methods of obtaining them [11-13]. To obtain new physical and chemical properties or improve the integrative properties of metallic foams, ceramic particles were added into metallic foams to form composite foams [14 - 16]. In addition because of low densities and low-pressure drops, metal foams offer excellent flow characteristics as catalyst structural supports. The Fe/Al$_2$O$_3$ system has recently received attention because of its potential magneto-transport properties and uses as surface coatings by granular films [17]. In addition, several researches have been carried out to investigate catalytic performance of Fe–Al$_2$O$_3$ system. [18,19] in work [20] Iron–alumina composite foam was prepared through combination of combustion synthesis (CS) and spark plasma sintering (SPS) techniques. Al, Fe$_2$O$_3$ and Fe
powders were used as starting materials. Samples containing 5–20 wt % (Al + Fe$_2$O$_3$) powders and balanced content Fe powder were sintered by SPS apparatus. Metal foam was formed by a typical base metal such as Al or Cu [21-24]. The subject of the experiment was the use test of producing techniques of the porous metal foams by reduction of metal oxides during sintering. The mixture was sintered in a dissociated ammonia atmosphere. Fe foam was prepared according to the method described in the Polish patent No. 199720 [25]. This allows for composing of irregular cellular structures with pores open or closed. The range of the porosity depends strongly on used materials, particle size and type of particulate material. However crucial influence on the porosity has the ratio between quantity of metal oxide powder and amount of matrix metal powder, which is a basic structure of produced sinter [26]. A new and promising method to produce open-cell metallic foams on the base of iron powder, low and high alloyed steel powders was the research objective. The foam material can be stacked and co-sintered with top layers to sandwich structures. The experiment used Ferrum powder ASC 100.29, Fe$_2$O$_3$ powder and Al$_2$O$_3$ microspheres to produce Fe-Al$_2$O$_3$ composite foam for potential magnetotransport and catalyst structural support applications. The object of the research was to examine new and promising method of fabrication an open-cell metallic Fe/Al$_2$O$_3$ composite foam.

2 Equipment and material

The initial stage for the production of porous sinter is to weigh and mix the powders together in appropriate proportions. There are four types of mixtures, labelled no. I, no. II and no. III. Table no. 1 shows types and proportions of powders that are components of each blend. Sample no. II disintegrated by too large share of microspheres Al$_2$O$_3$ in the mixture.

Table 1. Types and proportions of mixing powders.

| Sample designation | Composition | No. I | No. II | No. III |
|--------------------|-------------|------|-------|--------|
| ASC 100.29 [g]     | 140         | 140  | 140   |
| Cu [g]             | 8           | 8    | 8     |
| Fe$_2$O$_3$ [g]    | 16.8        | 16.8 | 16.8  |
| Al$_2$O$_3$[g]     | 3           | 12   | 6     |

Fe foam was prepared from mixture of iron powder ASC 100.29 (source Höganäs company) and iron oxide Fe$_2$O$_3$ + microspheres Al$_2$O$_3$. The sintering of prepared samples was done in a laboratory tube furnace at 1180°C, in a reducing gas shield of dissociated ammonia. Ammonia dissociates to nitrogen and hydrogen at 850°C in the presence of an iron catalyst. Such residual gases were directed to the sintering chamber into which a sample was placed. Hydrogen was a reducing oxide agent, iron oxide was reduced, thus providing protective atmosphere for the sintering process. Excess ammonia as well as the process derived products: the nitrogen and water vapour were subject to combustion. After sintering the samples situated in furnace were moved to an area where it was cooled in protective atmosphere. Cooling took place in with an average speed of about 25 degrees per minute. To determine pore size and shape from the foam specimens, the image analyse software was used.

3 Micro and macro structure investigation

To illustrate fusion structures, investigators used the optical microscopy and SEM methods. For tests of metallographic specimens, Nikon MA 200 Eclipse microscope with the image analysis system NIS 4.20 was used. All samples were mounted in a vacuum, using Buehler EpoThin resin during
preparation process for the protection of the porous structure. SEM examination was performed using the JEOL JSM 7100F microscope (with Schottky field emission) with EDS OXFORD X-MAX microprobe. The results showed that there is a possibility of obtaining the binding between the Fe foam and Al₂O₃ microsphere.

4 The observation with optical microscope and image analysis.

The porosity was studied using an NIS 4.20 image analyser. Figure 1 shows an image of the porous structures of formed Fe sample and Al₂O₃ microsphere binding.

![Sample No. I](image1.png) ![Sample No. III](image2.png)

**Figure 1.** The porous samples OM microstructures (mag. 100x). Not etched.

Considerable expansion of the surface inside the structure was achieved. It should be noted that non-uniform distribution of pores was due to diversity of the composition powder particle size. There are bridges connecting porous layer with a steel substrate surface. The relative volume of particles was determined in accordance with the Cavalieri-Hacquert principal. The result of the measure of the porous metal foam showed the porosity in the range of 51-59% with bulk density. The chemical composition and shape of the sintered material particles affect the creation of the diffusion bridges. A quantitative analysis of the sample microstructure was conducted to determine its porosity. It was necessary to determine the number of pores per unit considering polished surface for different cross-sections. 5 areas so called *Region of Interests* (ROI) of the structure were analysed. The average porosity was: 51% for specimen no. I and was 59% for specimen no. III. Fig. 2. shows location of ROI for analysed specimen No. I.

![Figure 2.](image3.png)

**Figure 2.** The analysis of ROI of sample No. I.

5 The observation with SEM microscope and EDS analysis.

Figure 3 shows the SEM micrographs of sintered foam surfaces of composition no. I (Figure 3), imaged using the JEOL JSM 7100F electron microscope. The depth of the field obtained by SEM allows us to assess the complex structure of the foam with visible parent material, voids forming porosity and Al₂O₃ microspheres.
Figure 4 shows the microstructures of sample No. III of metallic foam (500x and 1000x magnification) with invisible parent material, voids forming porosity and Al₂O₃ microspheres.

Figure 3. SEM photography of sample No. I (mag. 500x and 1000x).

Figure 4. SEM photography of sample No. III (mag. 500x and 1000x).

Figure 5. The microstructure of the elements SEM and EDS distribution for Fe, Al and O, (samples No. I and III).

During the SEM observation and the EDS microlens tests, there was no diffusion of the surface finish from the Fe / Fe₂O₃ mixture to the Al₂O₃ microsphere material. Lack of diffusion is evident in case
of Fe / Al₂O₃ association. Fig. 5 shows the microstructure of the elements SEM and EDS distribution for Fe, Al and O. Connections inside the Fe foam obtained by sintering in dissociated ammonia have shown morphological characteristics of the foam connections with open cells [17, 18]. The pores remained open to the outside. Similarly to works [17-20], a combination with acceptable parameters was obtained. When using techniques such as "joining metals" [21], the metal used for binding the foam pores, causes it’s closing. However, any comparison with literature data from papers [17-21] is difficult to perform due to significant differences in the materials and techniques used. Available publications consider bindings such as foam-foam and foam-ceramics and foam-base metal configurations [27-30]. Metallographic examination and EDS analysis have demonstrated the possibility of using sintering techniques for materials difficult to bind, such as metallic foams with a non-metallic component of Al₂O₃.

6 Conclusions

There is a wide range of possible applications for presented materials, e.g. as heat exchangers, filters or catalysts. New and promising method of producing open-cell metallic foams on the base of iron powder, low and high alloyed steel powders as well as nickel alloy powder was used. Macroscopic and microscopic observations were provided insight into the morphology of the structure. In comparison with literature reports presenting other methods of producing porous composites Fe/Al₂O₃, relatively simple and available manufacturing technology was used. The obtained effects are encouraging and allow assuming that the compositional composites can be made on the basis of other sintered metal powders using the technology used.

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