Experimental comparison between upsetting characteristics of porous components prepared by Fe-based sintering technology

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Abstract. The investigation has been undertaken to develop and study the upsetting characteristics of iron-based sintered porous components by using elemental powders through powder metallurgical techniques. The paper presents experimental results of forming Fe-based sintered porous samples and upsetting it by two methods. The first method involves upsetting between two flat plates ( anvils ). The second method is based on the deformation of the material in closed-die. The study compares the graphs of variation of the force vs. displacement. The aim of the article is to compare the experimental characteristics of upsetting process of samples with different porosities. Metallic porous structure was obtained as a result of sintering processes Fe-based powders mixtures with addition of copper catalyst and Fe ( III ) oxide as a space holder. The sintering was proceeded at the temperature of 1130°C and 50 min. long, in dissociated ammonia protective gas. Some samples were sintered with addition of powdered activated carbon to increase its porosity properties and change chemical composition.

1 Introduction

Metallic foams are lightweight structural materials, however with enormous future potential for applications where lightweight combined with high stiffness and acceptable manufacturing costs are of prime interest. The performance of metallic foams, in particular those made of Fe, in various prototypes, such as foamed panels, sandwiches, complex 3-D - parts, casted and sintered foamed profiles has been discussed with respect to the expected and achieved goals. Scientific attempts to improve foam quality of any type of foam concentrate on the foam physics, i.e., bubble formation, foam nucleation, growth, stability, development or gas diffusion in the liquid state, where the foam structure is evolving [1-13]. Considering methods of production, metal foam structure characterises different level of homogeneity and comprises different characteristic features [1, 2]. Although they have very high potential, and wide fields of application, they are still not wide spread on the market. Some reviews about the applications of metallic foams and its several excellent properties of the material are available in the literature [14-19].
While structural applications take advantage of metal foams considering lightweight and specific mechanical properties, its functional applications were based on special functionality for instance a large open area in combination with very good thermal or electrical conductivity for heat dissipation or as electrode for batteries, respectively, and for overall application functionality [20-22].

Obviously applications of metal foams are strongly linked to the properties, and some of them are mainly related to those of the matrix metal itself, e.g., elasticity, temperature or corrosion resistance, etc., while others appear only in combination with the cellular structure, e.g., low density, large surface area or damping.

The foam structure is the characteristic feature of foam [23]. Mechanical properties depend mainly on the density, but are also based by the feature of the cellular structure i.e., cell connectivity, cell roundness, diameter distribution, etc.

Upsetting is defined as free forming, by which a workpiece segment is reduced dimension between usually plane, parallel platens (Figure 1). It also includes coining and heading. Closed-die forming, shape upsetting, or die heading involve tools which contain the intended shape wholly or in a part [24]. Based on elementary plasticity theory, upsetting forces can be determined approximately [24, 25, 26]. To do so, the distribution of the normal stress at the tool-workpiece interface is required. Substitution of the Tresca yield criterion [24]:

\[
\sigma_x - \sigma_z = \sigma_y
\]

With the help of the Tresca criterion the interface pressure is [24, 25]:

\[
\sigma_z = -\sigma_y \exp \left[ \frac{2\mu}{H} \left( \frac{d - x}{2} \right) \right]
\]

Figure 1 shows the interface pressure distributions provided by eq. 2. The upsetting force is obtained by integration of the pressure over the contact area A [24]:

\[
F_z = \int_A \sigma_z dA
\]

The aim of the article is to compare the experimental characteristics upsetting for samples with two different materials and various porosities.

2 Methodology

2.1 Fe foam preparing

Metallic precursor composition was made of iron-based powders: Distaloy SE, ASC 100.29 (by Höganäs company), and Fe₂O₃ powder with different compositions. As a reaction catalyst, Cu was used and Fe₂O₃ was used as a foaming agent-space holder. The samples were affected only by external gravity reactions but weren’t internally compacted. The mixture was sintered in a dissociated ammonia atmosphere at 1180°C for 45 minutes. After sintering, samples situated in furnace, were moved to an area where it was cooled in a protective atmosphere. Cooling took place in with an average speed of about 25 degrees.

| Powder       | Chemical Composition (Mas. %) | Average Particle Size (μm) |
|--------------|-------------------------------|-----------------------------|
| ASC 100.29   | C: <0.01, Cu: -, Ni: -, Mo: - | Balanced, 45–180           |
| Distaloy SE  | C: 1.5, Cu: 4, Ni: 0.5, Mo: -| Balanced, 45–180           |
per minute. To determine pore size and shape from the foam samples, the image analyser software was used. The properties of iron-based materials are shown in Table 1.

2.2 Machining
Placing an accurate sample matrix made of sintered foams required treatment which would give samples of repeating shape and dimensional accuracy. There’s been conducted reconnaissance study, which concluded that among the three-studied treatment technologies, meaning: laser processing [28], EDM [29] and machining, only the milling process gave the desired precision net shape and machining stability and the process of shaping the small depth of cut [30]. The process of forming test samples made of sintered foam consisted in a finishing mill for milling the front surface of the outer cylindrical samples sized Ø15x10mm.

Machining process was conducted without the use of coolant on the Hermle B300 milling center. A tool used to conduct the treatment was endmill, by Sandvik Coromant with a diameter of 10 mm. Trials cutting have been carried out with the following parameters: cutting speed \( v_c = 150 \text{ m/ min} \), feed rate \( v_f = 300 \text{ mm/ min} \), depth of cut \( a_p = 10 \text{ mm} \), infeed side \( a_e = 0.2 \text{ mm} \). The samples of Fe foam before and after machining are shown in Figure 2.

![Figure 2](image1.png)

Figure 2. Samples before machining (a) and after machining (b).

2.3 Upsetting
The experimental part of the research of upsetting was conducted at a special stand [27], which included the following:

- a tool for free upsetting and closed-die forming (Figure 3),
- LabTest 5.20SPI testing machine (LABORTECH firm), 20kN force (machine calibrated by PN-EN ISO 7500-1:2005 and meets the metrological requirements for class 0.5),
- computer stand with Test&Motion software (LABORTECH) to measure forces and displacements.

The materials for experimental investigations were Distaloy SE and ASC 100.29 rod segments, whose diameter was D=15 mm and high \( h_0=10.3 \text{ mm} \), (which corresponded to the relative coefficient \( h_0/D=0.69 \)).
3 Result and discussion

3.1 Porosity and microstructure

The microstructures of the porous metal foam were observed using an optical microscope from Nikon MA200. The view of samples obtained by means of optical microscopy is shown in Figure 4. The unetched samples were analysed. The obtained microstructures present open porosity and grain-to-grain connections. The measurement of porosity on the cross-section was obtained with NIS 4.20 imaging analysis system. The relative volume of particles was determined in accordance with the Cavalieri-Hacquert principle.

The research has shown that the porous metal foam with bulk density obtained from: ASC 100.29 resulted in a porosity of 62%, and obtained from Distaloy SE powder resulted in 75%. The shape of the sintered material particles and chemical composition affected the appearance of diffusion bridges creation. Earlier conducted studies consider metallic foams based on Fe [31, 32, 33] with the structure of considerably smaller pores. A similar distribution but for a much larger pore volume was obtained in using an organic foam precursor [34]. Nowadays, metallic porous materials have different structures and mechanical properties [35, 36]. To evaluate these properties, the test on samples of the obtained material should be performed. Most tests are conducted on samples of standard sizes. But when details of the porous materials have complex configurations of spatial elements, it’s difficult to implement the standard tests [34] and experimental tests on the samples of the details are needed to obtain actual structural and mechanical properties of the porous material and its spatial configuration.

3.2 Upsetting

Upsetting characteristic of all materials demonstrated the typical behaviour of elastic-plastic foam under compression. Compression tests all the materials showed the typical behaviour of a flexible foam in a compressed state (Figure 5). The compression process based on highly porous sintered Fe includes three stages: stage linear elastic, plastic deformation stage, and the stage compaction [37, 38]. Force-displacement curves of the samples 1 and 2 made from DISTALOY SE and ASC 100.29 are shown in Figure 3 which differs from that described in the literature for [37, 38]. At the beginning, the material has experienced a short-term elastic stage, the 5% compressive strain (which corresponded ratio Δl/h₀=0.05), where force increased linearly with increasing displacement Δl. Partially reversible deformation powders process takes place. Then the load slowly increase at higher displacement Δl in the next step of plastic deformation. As the load increases to 30% compressive strain, almost all of connections

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Figure 4. The microstructure of Fe foam samples made of Distaloy SE (a) and ASC 100.29 (b).

Figure 5. Force vs. displacement obtained for upsetting samples made from DISTALOY SE and ASC 100.29 at ratio h₀/D=0.69.
between the powder particles were plastically deformed. At this time, the pores become smaller and the shape of the pores changed irreversibly. Propagation strength within the material has been blocked due to the existence of pores in the sintering process, and therefore the load increased slowly with progressive displacement $\Delta l$. The maximum experimental values of loads obtained in upsetting of samples 1 and 2 made from Distaloy SE and ASC 100.29 at plastic deformation stage were analysed. The values were received at compressive strain $\varepsilon$=30%. The greatest force value was recorded in upsetting of sample 2 made from Distaloy SE ($F=6529.36\,N$). It was greater than the force for sample 1 made from ASC 100.29 ($F=5097.98\,N$) by approx. 30%. As a result of base analysis of changes in upsetting forces of samples 3 using ASC 100.29 in dies, it can be stated that together with an increase in relative ratio $\Delta l/h_0$ and compressive strain $\varepsilon$, the load increases exponentially for both examined materials, similarly with the Distaloy SE material.

4 Conclusions

Samples of porous sintered Fe with porosity up to 75 vol.% were successfully prepared using a powder metallurgical technique with Fe dioxide as a space-holder material. The influence of the initial iron powder size and iron oxide size on microstructural and mechanical characteristics was studied in details. The samples contained pores originating from bulk density and pores, which were formed by space holder (iron dioxide) decomposition. Increasing amounts of space holder in the initial mixture increased the total porosity. This clearly affects the course of loads during the upsetting and closed-die forming processes. In both cases, the diagrams characters of the curves of loads vs. strains are similar. The differences result from the use of a material with different mechanical properties and with various porosity.

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