Mixture of iron and aluminum powders were used for fabrication of iron aluminide Fe3Al. The powder samples were compacted and vacuum sintered at temperatures of 1250, 1350, 1400 and 1450 °C, and also using hot forging at a temperature of 1050 °C and 1150 °C. It is shown, that the increase of density is observed only at a temperature of 1400 °C. At sintering temperature of 1450 °C the samples reach a density of 6.35 g/cm³, which is 94% of the theoretical value. Only the use of hot forging makes it possible to obtain nonporous samples. It is shown, that bending strength, fracture toughness and hardness increase with increase of sintering temperature, but higher values of this parameters have the samples, produced with use of hot forging.

**Key words:** INTERMETALLICS, POWDER, IRON ALUMINIDES, SINTERING, HOT FORGING, STRENGTH.

1. Introduction

Intermetallides of Fe-Al system have a number of properties attractive for industrial applications: low specific gravity, high strength, chemical resistance, high corrosion resistance, high wear resistance and heat resistance, and relatively low cost [1, 2]. However, limited ductility at low temperatures and a sharp drop in strength above 600 °C are the main drawbacks for their use as structural materials.

The most common methods for the production of intermetallic materials, including iron aluminides, are related to the methods of foundry production: melting in induction furnaces, electroslag melting technology, method of smelting using Exo-Melt technology [3]. However, this process for the production of iron aluminides is multi-stage and energy-intensive.

In addition, for the production of iron aluminide, great attention is paid to the methods of powder metallurgy, which have a number of advantages: the ability to manage the microstructure, improve properties, and also the production of shaped products. Along with this, these methods consume less material, and exclude the process of further processing [4, 5].

In the production of iron aluminides by powder metallurgy methods, special attention should be given to the synthesis of the alloy from elementary Fe and Al powders, which in the correct composition form the required intermetallic compound FeAl in accordance with the phase diagram. One and progressive methods for the production of aluminides is the process of self-propagating high-temperature synthesis (SHS). Despite its great energy-saving advantage, this method mainly leads to the production of an intermetallic compound with a porous structure that requires further pressure treatment. Such methods of consolidation of powders are hot pressing, hot isostatic pressing (HIP), electric discharge sintering (SPS, PAS, PPS) [6-8].

Nevertheless, the limited information on the application of thermomechanical processes based on dynamic processing of materials such as forging and stamping leads to the need for complex studies in the development of new efficient technologies for obtaining products from the Fe-Al alloy system.

The aim of this work was to study the possibility of production of the intermetallic system of the Fe-Al system by sintering and impact consolidation of powder bodies, as well as the influence of the regimes of the methods used on the structure and properties of iron aluminides.

2. Materials and experimental procedure

Iron powder with a particle size of 45-160 μm and an aluminum powder with a particle size of 50-100 μm were used as the initial powders for the production of iron aluminide Fe3Al (fig.1).

A mixture of powders in a ratio of Fe + 14% Al (wt.) were mixed in a tumbling mixer for 60 minutes in alcohol. Cylindrical samples with a specific force of 600 MPa were extruded from the obtained mixture of powders. The obtained samples for fabrication of intermetallic compound were preliminarily synthesized by sintering in a vacuum of 0,0133 Pa at a temperature of 1050 °C with an isothermal holding time of 60 min. The rate of temperature rise was 10 °C/min. Further, the powder samples were compacted by free sintering at temperatures of 1250, 1350, 1400 and 1450 °C, and also using hot forging at a temperature of 1040 °C and 1150 °C with isothermal aging at a final temperature of 20 min.

**Fig.1. The initial powders of aluminum (a), iron (b) and powder mixture (c)**

After carrying out the corresponding technological operations, the density (by hydrostatic method) was studied on the obtained samples; electrical resistivity (by measuring the voltage drop on the test and reference samples); Vickers hardness with a load of 100 N on the hardness tester 2137 TU; bending strength on samples measuring 4×4×25 mm and distance between supports 20 mm; fracture toughness on samples measuring 4×3×20 mm with a distance between supports 10 mm; compression on samples measuring 4×4×8 mm. A crack in the sample was injected by an electric spark method with a wire 0,1 mm in diameter. Bending and fracture toughness tests were carried out on a Ceramtest System testing machine, and on compression on a UTM-100 machine. X-ray phase analysis of samples was performed on a DRON-3 diffractometer in Cu-Kα radiation. The structure and the surface of fracture of the obtained samples investigated by scanning electron microscope JEOL Superprobe 733. A local X-ray spectral analysis was performed on an X-ray microprobe MS-46 from SAMESA (France).
3. Results and discussion

During the heating of samples from a mixture of powders of Fe + 14 mass% Al at temperatures of 540-1040 °C, intermetallic compounds are formed. This synthesis is accompanied by an increase in the linear dimensions of the sample and a decrease in the density, as shown in fig. 2 and in table 1. The formation of pores and cracks in sintering in the intermetallides of the Fe-Al system is attributed to several reasons, including the Kirkendal effect, which consists of different rates of mutual diffusion of the elements.

![Fig. 2. The initial sample from a mixture of Fe + 14% Al (a) and after vacuum sintering at temperatures of 650 (b) and 1040 °C (c) with isothermal holding for 60 minutes](image)

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>Density γ, g/cm³</th>
<th>Weight loss ∆m, %</th>
<th>Changes in ∆d, %</th>
<th>∆h, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>540</td>
<td>5.71</td>
<td>5.67</td>
<td>0.076</td>
<td>0</td>
</tr>
<tr>
<td>650</td>
<td>5.72</td>
<td>3.02</td>
<td>0.069</td>
<td>-17.5</td>
</tr>
<tr>
<td>1040</td>
<td>5.73</td>
<td>3.18</td>
<td>0.06</td>
<td>-16.3</td>
</tr>
</tbody>
</table>

After repeated cold compaction of porous synthesized samples to a higher initial density component of 5.2-5.3 g/cm³, conducted further consolidation intermetallic obtained by vacuum sintering at higher temperatures and hot forging. In the case of sintering of samples from the synthesized powder of the intermetallic compound, an increase in the density is observed only at a temperature of 1400 °C from 5.3 to 5.7 g/cm³ (fig. 3). At sintering temperature of 1450 °C, the samples reach a density of 6.35 g/cm³, which is 94% of the theoretical value for the Fe₃Al intermetallide (6.72 g/cm³). The use of hot forging to compact the synthesized powder makes it possible to obtain nonporous samples at a temperature of 1050 °C, where the density is 6.7 g/cm³.

![Fig. 3. Density of the intermetallic compound after sintering and hot forging at different temperatures](image)

Poreless samples forged structure is illustrated in fig. 4. The structure of the intermetallic compound composed of large particles which consist of smaller grains. On forged structures, it is also possible to distinguish the fine boundaries between small grains, and the thicker boundaries between large particles (subgrains) that have been etched.

In the case of sintering of samples, a large residual porosity is observed in the structure at low compaction temperatures, which decreases substantially with increasing sintering temperature (fig. 5). Analysis of microstructures also shows that with increasing temperature, the particles of the material under study become coarsened, which is especially noticeable at a temperature of 1450 °C.

![Fig. 4. Structure of the intermetallic Fe-14Al after hot forging at 1050 °C (a) and 1150 °C (b)](image)

![Fig. 5. Structure of samples after sintering at temperatures: a – 1250, b – 1400, c – 1450 °C](image)

In addition, it is necessary to note the presence of an additional phase in the structure of the sintered intermetallic compound, which...
is observed in the material at all sintering temperatures. But at a temperature of 1450 °C these inclusions acquire the largest size. According to the local X-ray spectral analysis, these inclusions are the Fe₂₃Al₇₈C₀.₇₇ phase (table 2).

Table 2
Local x-ray spectral analysis of samples of sintered and forged samples at temperatures of 1450 and 1050 °C, respectively

<table>
<thead>
<tr>
<th>Fabrication mode</th>
<th>Temperature, °C</th>
<th>Element</th>
<th>% (mass.)</th>
<th>% (at.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hot forging</td>
<td>1050</td>
<td>Matrix</td>
<td>13,0</td>
<td>23,6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Fe</td>
<td>87,0</td>
<td>76,4</td>
</tr>
<tr>
<td>Sintering</td>
<td>1450</td>
<td>Matrix</td>
<td>13,0</td>
<td>23,7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Inclusions</td>
<td>Fe</td>
<td>87,0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Al</td>
<td>12,2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>C</td>
<td>3,5</td>
</tr>
</tbody>
</table>

The formation of the carbide phase due to the presence of a small amount of carbon in the original iron powder. Chemical analysis for carbon and oxygen of the initial iron, aluminum powders and their mixtures is presented in table 3.

Table 3
Chemical analysis of powders and mixtures of Fe-14Al before and after synthesis

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Fe</th>
<th>Initial mixture</th>
<th>Sintering at 1050 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>O₂</td>
<td>0,2</td>
<td>0,4</td>
<td>0,5</td>
<td>0,2</td>
</tr>
<tr>
<td>C</td>
<td>0,08</td>
<td>0,07</td>
<td>0,07</td>
<td>0,07</td>
</tr>
</tbody>
</table>

As can be seen from table 3, about 13 wt.% Al is included in the composition of the synthesized intermetallic compound, which means that the obtained material is in the region of α-Fe + Fe₃Al according to the binary phase diagram of the Fe-Al system. [9]

The X-ray diffraction analysis of Fe-14Al intermetallic showed (fig. 6) that the material in addition to the Fe₃Al phase contains traces of the carbide phase Fe₃Al₇₈C₀.₇₇. The intermetallic phase of Fe₃Al was identified by the interplanar distance [220] of 0.33 Å, and also by the reflex [200], which indicates the intermetallic structure is ordered in type B2.

Fig. 6. X-ray diffraction pattern of sample from a powder mixture Fe+14%Al after thermal synthesis and forging at a temperature of 1050 °C

To produce a powder intermetallic with high strength characteristics, in addition to providing a low porosity, it is also necessary to create qualitative boundaries between particles, especially when powder consolidation occurs at low temperatures compared to conventional casting and remelting technologies, where the condensation temperature of the Fe₃Al intermetallic compound exceeds 1530 °C. The state of the boundaries, or the degree of interparticle interaction can be indirectly estimated from the characteristic of the resistivity. In the case of sintering the material under study, the resistivity decreases with an increase of the compaction temperature from 235 μΩ·cm at 1250 °C to 122 μΩ·cm at 1450 °C (fig. 7,a). In this case, the reason for the decrease in the specific resistivity can be both improvement of the contact formation between the particles and a decrease in the porosity during sintering. The specific electrical resistivity of the stamped samples corresponds to a value of 122-125 μΩ·cm. If we compare the results obtained with the data of other authors, then it should be noted that the resistivity of the Fe₃Al intermetallic compound obtained by remelting can be from 100 μΩ·cm for a D₀₃ type structure up to 130 μΩ·cm for a B₂ type structure [10]. Taking into account that the obtained intermetallic compound belongs to the region of α-Fe + Fe₃Al and consists of lattices with different types of ordering – A₂ and B₂, the obtained level of specific electrical resistance agrees with the known results of other authors. In addition, it is necessary to pay attention to the porosity between the particles of the forged samples, which can also affect the ability of the electric signal transmission between the structure elements.

The quality of the boundaries in the case of powder bodies can have a significant effect on the strength of such materials. The bending test of intermetallic samples obtained by sintering and hot forging had shown that the sintered samples show low strength (less than 400 MPa) at low sintering temperatures (fig. 7,b). Only at sintering temperature of 1450 °C samples reach 950-980 MPa of bend strength. The reason for the sharp increase in the strength of the composite sintered at 1450 °C is, in our opinion, an increase in the density of the composite and a change in the quality of the boundaries in the material. The porous samples obtained by forging at 1050 °C showed a strength of 890 MPa, and the increase in the consolidation temperature of powders to 1150 °C, the strength rises to 1060 MPa, which is most likely due to the improvement of interparticle interaction and, consequently, the quality of the boundaries. Another strength parameter that characterizes the ability of a structure to resist crack propagation is fracture toughness. As in the case of strength, the fracture toughness shows a small growth with an increase in the sintering temperature of the intermetallic compound from 7 MPa·m²/² at 1250 °C, 11.5 MPa·m²/² at 1400 °C (fig. 7,c). And when the sintering temperature increases to 1450 °C, the crack resistance reaches values of 24.6 MPa·m²/². For forged samples, we also observe a similar change in the crack resistance at a strength of 1050 °C; crack resistance is 20.9 MPa·m²/², and at 1150 °C, 25.1 MPa·m²/². The least quality of boundaries affects the hardness characteristics of the material. But tests have shown that the hardness with temperature change and the compaction method changes in the same way as the two previous mechanical characteristics. When sintering the intermetallic compound in the temperature range 1250-1400 °C, the hardness of the material changes from 780 to 945 MPa, and when the temperature reaches 1455 °C, the hardness increases to 1845 MPa (fig. 7,d). Samples that forged at 1050 °C show the hardness of 1200 MPa, which increases to 2270 MPa with increasing forging temperature to 1150 °C. Comparison of the hardness of the sintered and forged Fe₇₆.₄Al₂₃.₆ intermetallic with the data of other researchers shows their similarity. For example, it is known from [11] that the solid solution of aluminum in iron Fe₇₆.₄Al₃.₆ has a hardness of 1940 MPa, and the intermetallic with ordering of the structure as D₀₃ Fe₇₆.₄Al₃.₆ is 2350 MPa.

Separately, we consider strength tests for compression and tension. The compression tests, as well as the investigation of the previous characteristics of the obtained material, were carried out for all samples. Tensile tests were carried out only on samples sintered at 1450 °C and forged specimens, since the samples, which were sintered at low temperatures, had low strength and it was difficult to prepare samples with necks 3-4 mm in diameter for this type of test.

The compressive loading of sintered samples showed that the strength of the intermetallic compound increased insignificantly from 470 to 605 MPa with increasing sintering temperature from 1250 to 1400 °C (fig. 8,a), and when the compaction temperature reaches 1450 °C, the strength rises to 855 MPa, which corresponds to the trend of variation and previous characteristics in the
preparation of the intermetallic sintering. Forged specimens showed higher strength values than the sintered samples, which was 990 MPa for consolidated samples at 1050 °C and 1060 MPa for samples compacted at 1150 °C. Yield strength for deformation of 0.2%, sintered and forged intermetallic samples repeats the nature of the change in compressive strength and is shown in fig. 8.b. It should be noted here that the yield strength of the specimens stamped at different temperatures is almost the same. In the case of sintering, the yield stress is also observed, regardless of the compaction temperature, despite the low compressive strength. The yield strength of samples of sintered at 1400 °C and 1450 °C differs by almost two times—315 and 595, respectively.

Analysis of the fracture surfaces of sintered and forged samples after the tensile test shows that in the specimens stamped at 1050 °C the main fracture crack passes mainly along the boundary between the particles (fig. 9.a). Only in some places there are elements of the cleavage of the particle. Those, in this case, the strength of the intermetallic compound is determined by the strength and quality of the boundaries between the particles. Fig. 4.a shows the boundaries between particles that have been etched and the grain boundaries inside the particle that have not been etched, which can indicate the presence of impurities at the particle boundaries, which reduce the quality of contact and adhesion strength between the particles.

With an increase in the temperature of forging of the intermetallic compound to 1150 °C, changes occur also in the case of material failure. In fig. 9.b, a predominantly intercrystalline fracture is observed, but with a significantly larger proportion of the trans-crystalline component at the fracture surface, in comparison with the previous case. Those, improves the adhesion between particles and the crack extends not only along the boundary between the particles, but also breaks up large particles. In the case of the destruction of the intermetallic sintered at 1450 °C (fig. 9.c), a transcryalline fracture is observed, i.e. these patterns are characterized by a crooked (brook) nature of destruction. Only in some places on the surface of the destruction of such samples, the boundaries between the particles are noticeable. This is explained by the fact that at 1450 °C significant changes occur in the structure of the material due to the enlargement of the particles and the recrystallization of a large number of boundaries in this case.

**Conclusions**

1. Vacuum sintering of Fe-14% Al powdered samples allows to get noticeable increase in density only from temperatures of no less than 1400 °C. After sintering at temperature of 1450 °C the samples reach a density of 6.35 g/cm³, which is 94 % of the theoretical value. Fully dense compacts (6.7 g/cm³) had been obtain with use of porous performs hot forging from a temperature of 1050–1150 °C.

2. With increase of sintering temperature from 1250 °C to 1450 °C, electrical resistivity of intermetallic compound decreases from 235 μΩ·cm to 122 μΩ·cm, that is stipulated by intensification of
interparticle interaction, improvement of state of the boundaries and decrease of samples porosity. Meanwhile the particles of the material become coarsened, which is especially noticeable at a temperature of 1450 °C. The specific electrical resistivity of hot forged samples is 122-125 μΩ·cm.

3. Bending strength, fracture toughness and hardness increase with increase of sintering temperature, but higher values of this parameters have the samples, produced with use of hot forging.

Fig. 9. Destruction surface of sintered and forged samples after tensile tests: a – hot forging 1050 °C; b – hot forging 1150 °C; c – sintering at 1450 °C

References


