HOT FORGING OF POWDERED Fe3Al INTERMETALLIC ALLOYS

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Abstract: The capabilities of powder hot forging for manufacturing of FeAl intermetallics and effect of forging and following thermal treatment routines on their structure and properties had been investigated. Fe3Al intermetallic powders were produced by means of thermal synthesis at 1000 °C in vacuum from a mixture of Fe and Al elemental powders. Hot forging of consolidated preforms had been carried out from 1000, 1050, 1100 and 1150 °C and afterwards the hot forged preforms were subjected to supplementary sintering in vacuum at 1100-1450 °C. It is found, that thermal synthesis of Fe + 14 % Al powder mixture results in formation of Fe3Al phase. Sintering of hot forged specimens result in increasing of strength and crack growth resistance, which values enhance with increasing of sintering temperature. Otherwise the hardness of hot forged intermetallics decreases after their sintering. The influence of modes of treatment on the structure and properties of the materials was investigated. It has been established that the strength and fracture toughness of the intermetallics obtained from milled blend after hot forging had the higher values as compared with the alloy made from the batch without its milling.

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

Introduction

The development of modern technology requires implementation of new materials which combine the chemical resistance, high wear resistance and heat resistance, which possess intermetallic compounds, in particular - iron aluminate. These intermetallic compounds are becoming more attractive to modern industry due to their unique combination of physical, chemical and mechanical properties, such as low density, high corrosion resistance, heat resistance and heat-temperature strength [1-3]. However, the widespread commercial use of such materials obtained by casting is currently limited due to the inherent fragility, low values of ductility and toughness at room temperature [1, 2].

At the same time, there was a series of works, pointing to prospects for the use of products from Fe-Al intermetallic powder metallurgy techniques such as hot isostatic pressing, extrusion, hot forging or pressing and injection molding [4-6], spark plasma sintering (including the combination with mechanical alloying) [7, 8]. However, the limited information in this field leads to the need for comprehensive research in the development of new effective technologies for manufacturing products from Fe-Al intermetallic compounds and study of the impact of technological modes of manufacturing on the structure and properties of the resulting materials.

It is known, as well, that the basic physical and mechanical properties of materials and performance attributes are largely dependent on the size and morphology of the structural elements of the alloy and increases with increasing dispersion past due, in particular, intensive grinding of the initial powders accomplished by their mechanical activation [8-10].

In this regard, the aim of this work was to study the impact of technological modes of processing the initial powder mixture and thermomechanical processing of sintered iron aluminate with the use of methods of hot forging on the structure and properties of the resulting materials.

Experimental procedure

The initial powders of iron with particle size 80-160 microns, and aluminum having a particle size of 30-70 microns in the ratio (%, wt.) of 86Fe + 14Al were mixed in a tumbling mixer for 60 minutes in an alcohol.

Thermal synthesis of the intermetallic Fe3Al powder from the mixture of Fe and Al elemental powders was performed under vacuum at a temperature of 1000 °C with isothermal exposure 60 min.

As a result of synthesis porous sponge was received, which was crushed and the obtained alloy powders were pressed at 600 MPa to porous billet for subsequent hot forging. The billet was heated in argon to 1000, 1050, 1100 and 1150 °C and hot forging was performed in the semiclosed die on the screw arc-type stator press.

The samples after hot forging were divided into four groups, one of which did not respond to further heat treatment, and the other three were subjected to additional sintering in vacuum in accordance with the following modes:

- sintering at 1100 °C - 180 min.;
- sintering at 1300 °C - 10 min.;
- sintering at 1450 °C - 10 min.

To study the effects of dispersion and the morphology of the structural elements on the structure and properties of the intermetallic compounds for their preparation were used the powder mixtures of two types: in the first case the initial iron and aluminum powders were mixed in a tumbler mixer for 60 minutes (mixture I), in the second - the same initial mixture was milled in a planetary mill for 20 minutes in alcohol (mixture II).

The obtained after hot forging samples of both parties were subjected to the following modes of processing:

- mode 1 – hot forging without additional treatment;
- mode 2 – hot forging + sintering in vacuum at 1100 °C for 180 min.;
- mode 3 – hot forging + sintering in vacuum at 1300 °C for 10 min.

After implementation of each operations process for all of the obtained samples their density was investigated (by means of hydrostatic method), electrical resistivity (by measuring the voltage drop), the Vickers hardness with a load of 100 N, bending strength, fracture toughness. Bending and fracture tests was performed testing machine Ceramtest system. X-ray phase analysis of samples was performed on DRON-3 diffractometer in Co-Kα radiation. The structure of the material investigated by the scanning electron microscope JEOL Superprobe 733.

3. Results and Discussion

The results of XRD analysis of samples obtained after various processing steps showed that the synthesis of Fe-Al powders mixture at 1000 °C leads to the formation of intermetallic compound with an ordered B2 type structure, which is typical for phase FeAl, with interplanar spacings [110] constituting 2,044 Å (fig.1). Superstructural lines which characterize structure of the D03 phase FeAl, with interplanar spacings [110] constituting 2,044 Å (fig.1).

The density of the samples after forging at 1000 °C was about 6,62 g/cm3, and a further increase in temperature is almost unchanged (fig. 2). Subsequent sintering at 1100 °C and 1300 °C led to some decrease of density is more distinct for the samples forged at lower temperatures. Decrease of the porosity may be associated with degassing of samples during sintering in vacuum after forging and ordering process structure B2 type to type D03. Sintering of compacted samples at 1450 °C leads to increased...
density up to $6.71 - 6.72 \text{ g/cm}^3$ for all temperatures of forging that correspond to the theoretical density of the intermetallic Fe$_3$Al.

**Fig. 1.** X-ray diffraction pattern of a sample from a powder mixture Fe $+ 14\%$ Al after thermal synthesis and forging at a temperature of 1050 °C

The analysis of microstructure of samples after forging didn't show noticeable influence on its forging temperature, while their subsequent sintering led to a considerable increase of degree of interparticle fusion: in the continuous grid of brittle interparticle contacts of hot forged intermetallic (fig. 3a) after sintering at a relatively low temperature of 1100 °C interparticle fusion elements was observed (fig. 3b), while after sintering at increases temperature the interparticle mesh was converted markedly into a discontinuous (fig. 3c, d). Increasing of sintering temperature also leads to some consolidation of the structure.

Quality of borders or degree of contact interaction between particles in material are reflected indirectly by the characteristic of electrical resistivity. According to the results of investigations, the samples after forging showed some decrease in electrical resistivity with increasing of forging temperature to 1100−1150 °C (fig. 4), which may be caused by increasing the degree of adhesion between particles at higher temperatures. However at forging temperature of 1050 °C there is some increase in the electrical resistivity of the intermetallic compound, the increased porosity received after forging from 1050 °C samples (see fig. 2) can be one of the reasons of that. Thus, the defects which led to the increased value of the electrical resistivity of the intermetallic compound after forging from 1050 °C doesn't improve the subsequent sintering neither at high, nor at a low temperature.

Sintering of the samples compacted by forging shows decrease electrical resistivity from 129−132 mΩm·cm to 112−116 mΩm·cm, respectively for forged intermetallic compound before and after sintering. The reason for this decrease of electrical resistivity can both improved quality of interparticle borders and decrease in their length, as noted on fig. 3.

The characteristic of material strength also is sensitive to quality of interparticle borders. Bending tests of the samples showed that their strength after forging without subsequent sintering appeared at the level of 400 MPa and practically didn't depend on forging temperature (fig. 5, a). Sintering of the forged samples at 1100 °C resulted in an increase in strength to 620−700 MPa and the sintering temperature increases to 1300 and 1450 °C increased the level of strength of materials to 900−1050 of MPa. Presumably, this increase of strength associated with a change of state boundaries by improving the adhesion between the particles and a decrease in the length of such boundaries, which is particularly noticeable for higher sintering temperatures.

**Fig. 2.** Relationship between materials density and forging temperature for the samples after: 1 – hot forging (HF); 2 – hot forging + sintering at 1100 °C; 3 – forging + sintering at 1300 °C, 4 – forging + sintering at 1450 °C

**Fig. 3.** The structure of the samples after forging at 1100 °C (a) and subsequent sintering at 1100 °C (b); 1300 °C (c) and 1450 °C (d)

**Fig. 4.** Relationship between electrical resistivity and forging temperature for the samples after: 1 – hot forging (HF); 2 – hot forging + sintering at 1100 °C; 3 – forging + sintering at 1300 °C, 4 – forging + sintering at 1450 °C

**Fig. 5.** Relationship between bending strength and forging temperature for the samples after: 1 – hot forging (HF); 2 – hot forging + sintering at 1100 °C; 3 – forging + sintering at 1300 °C, 4 – forging + sintering at 1450 °C
Other characteristic which can estimate strength of boundaries and shows ability of material structure to resist crack promotion is a fracture toughness. Furthermore, the intermetallics are the materials of low ductility and fracture toughness is the most appropriate characteristic, that describes the behavior of brittle materials under loading.

The fracture toughness assessment of the samples after hot forging shown that they possess $K_{IC}$ values at $10\text{−}11 \text{ MPa}\cdot\text{m}^{1/2}$ for the whole range of forging temperatures (1000-1150 °C) (fig. 6). Their sintering allowed to increase this characteristic and with the increase of sintering temperature there is a marked increase in fracture toughness of $14\text{−}15 \text{ MPa}\cdot\text{m}^{1/2}$ after sintering at 1100 °C, till $17\text{−}20 \text{ MPa}\cdot\text{m}^{1/2}$ – at 1300 °C and till $22\text{−}33 \text{ MPa}\cdot\text{m}^{1/2}$ – at 1450 °C, which, in our opinion, is associated with a significant improvement in the quality of interparticle boundaries.

Unlike previous characteristics of the intermetallics, which had insignificant dependence on forging temperature, evaluation of hardness values indicate an increase in the latter with increasing temperature deformation. In case of sintering of hot forged samples there is a decrease of hardness in compare with unsintered materials (fig. 7).

When studying the influence of preliminary processing of initial powder mixtures on the structure and properties of the resulting material it was shown that milling in a planetary mill did not lead to a noticeable refinement of mixture components compared to mixing in a tumbling mixer (fig. 8). However, due to intense exposure of local plastic deformation of powder particles the significant amount of lamellar conglomerates appeared in the blend composition that is caused by rather high plasticity of mixture components.

The analysis of results the density of the samples after each mode of processing showed that application of hot forging for densification of iron aluminide obtained from a mixture of Fe and Al elemental powders (mixture I) and the mixture after milling in a planetary mill (mixture II) allowed to obtain intermetallics density of 6,57 g/cm$^3$ and 6,37 g/cm$^3$ respectively (fig. 9a).

Application of vacuum sintering for samples after forging led to some reduction in density - to 6,52 g/cm$^3$ and 6,32 g/cm$^3$ for the alloys obtained from the first and second mixtures, respectively, which may be due to the partial degassing of the samples so and by the formation of oxide phases in the grain boundaries in the presence of residual oxygen in the powder.
Local X-ray analysis of the intermetallic phases showed that the plates consist of iron with aluminum and dark layers are oxide phase, which can be attributed to the nonstoichiometric spinel Fe₆Al₂O₉. The component ratio indicates that the composition of plates consists of several phases - solid solution and intermetallic compound, i.e. B2+α and D0₃+α. Furthermore, an increase in the sintering of aluminum and oxygen content in the oxide phase, which further shifts the phase composition of lamellar particles to the area of the solid solution α.

It is obvious that the emergence of oxide phases in the alloy should be manifested in the performance characteristics of the electrical resistivity, which indirectly reflects the degree of interparticle interaction in the material. For all modes of processing the rougher due to the presence of intergranular oxide layer resistivity material with lamellar structure higher than the electrical resistance of the intermetallic forged initial powders (fig. 9b). Thus, if the intermetallic compounds made from untreated powders (mixture I), there is a clear trend noticeable decrease conductivity after annealing of hot-forging preforms and with increasing the sintering temperature for the material obtained from milled powders (mixture II), influence on the sintering resistivity values slightly.

Noticeable increase in the oxygen content for the material after intensive milling in a planetary mill compared with the initial powder and the material, obtained from the simple powder mixture was confirmed by the data in Table. 1.

Table. 1. The oxygen content in the initial powders of iron and aluminum and intermetallic compounds of two compositions after sintering at a temperature 1000 °C

<table>
<thead>
<tr>
<th>Powder</th>
<th>Al</th>
<th>Fe</th>
<th>Mixture I</th>
<th>Mixture II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen content, %</td>
<td>0.2</td>
<td>0.4</td>
<td>0.2</td>
<td>0.9</td>
</tr>
</tbody>
</table>

The data of microstructural analysis of hot forged intermetallics had shown, that after forging of initial powders (mixture I) structure is observed with near-equiaxed particulate size of 50-150 microns (fig. 10.a). After forging of milled powders (mixture II) structure seems like substantially lamellar with 10-20 microns thickness of lamellas (fig. 10,b). Thereby in the structure of the intermetallics obtained from milled powders the lamellar particles thin layers of obviously oxide phase were observed. Subsequent sintering the samples at 1100 °C and 1300 °C led to some enlargement of the structure as well as a marked increase in the degree of interparticle splicing of material obtained from unmilled powders: in a continuous grid of brittle interparticle contacts in hot forged intermetallic (fig. 10a) interparticle splice elements after sintering at a relatively low temperature of 1100 °C is observed (fig. 10,c), and with increasing of sintering temperature to 1300 °C the mesh was markedly transformed into discontinuous (fig. 10, e).

In case of use of the milled powders noted effect is shown significantly more weakly (fig. 10, b,d,e).

Taking into account the higher density and smaller values conductivity for intermetallics obtained from mixture I (not subjected to preliminary intensive grinding) would suggest, and the possibility of providing with such technological scheme and the higher the strength characteristics of the alloy. However, the results of bending tests have shown that the material obtained from mixture II, despite significant intergranular oxide layers, reaching a thickness of 5 microns, after the hot forging has a higher strength (~1200 MPa) as compared with an alloy made from mixture I (870 MPa) (fig. 9c).

These results well correlate with the data on influence of sintering on change of structure of intermetallic compounds shown in fig. 10. Decrease electrical resistivity of intermetallic compound obtained after sintering mixture I apparently due both to the improvement of the contact between the particles and the processes of coalescence of smaller pores into larger at the boundaries between the particles during sintering, as well as decrease in the total duration of the borders due to coarsening of the particles (fig. 10 , a, c, e).

At the same time, insignificant effect sintering the structure of the grain boundaries for materials obtained from milled mixture II (fig. 10, b, d, f) similarly reflected on the nature of the change in the resistance.

Fig. 9. The effect of processing modes and type of original powder mixture on density (a) and the basic physical and mechanical properties (b-e) of intermetallics

Fig. 10. The structure of the mixture intermetallic compounds I (a, c, e) and II (b, d, f) after hot forging (a, b) and subsequent annealing at 1100 °C (c, d) and 1300 °C (e, f)
The reason for this phenomenon seems to be something that was born and propagating crack under load in the event of the lamellar structure of intermetallic compound is extremely difficult to fully pass on the grain boundary and go around the plate-like particles, as in the case of destruction of material with considerably less texturing structure.

Sintering the samples after forging at 1100 °C causes decrease of strength and fracture toughness for both alloys (fig. 9, c,d) from removing during sintering strain hardening effects and thermal stresses, and increase the sintering temperature to 1300 °C leads to an increase of these characteristics improve due to quality and strength of phase boundaries, as evidenced by a decrease in the electrical resistivity of alloys, sintered at high (1300 °C) temperature (fig. 9b).

The hardness of the intermetallics obtained from the charge I after forging as well as after the subsequent sintering in both modes was higher compared to the intermetallics made from batch II, subjected to milling (fig. 9, e). Sintering after forging leads to a marked decrease of hardness of both alloys, the value of which decreases with increasing the thermal treatment temperature.

Conclusions
1) Thermal synthesis of Fe + 14% Al powders mixture at 1000 °C leads to the formation of intermetallics with an ordered structure, which is maintained even after hot forging.

2) Forging temperature in the investigated temperature range have not any significant impact on the structure of the resulting intermetallics, while their subsequent sintering leads to significant increase in the degree of interparticle splicing. The solid brittle mesh interparticle contacts for hot forged material with increased sintering temperature to 1300 - 1450 °C is converted into discontinuous.

3) The sintering after hot forging of the samples increases material strength and fracture strength values that increase with increasing the sintering temperature, while the hardness after sintering of hot forging of intermetallic reduced.

4) Preliminary processing of Fe and Al elemental powders mixture in a planetary mill essentially changes the morphology both the initial mixture and the structure of intermetallics after hot forging.

5) The strength of the intermetallics obtained from milled charge after hot forging had higher values (~1200 MPa) as compared with the alloy made from powder mixture without milling (870 MPa). Similar pattern is observed for the values of fracture strength of intermetallics produced from powders of various morphologies.

References
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