

The Effect of Production Process on Properties of FeAl₂₀Si₂₀

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This work deals with comparison of two production methods on microstructure, phase composition and mechanical properties of intermetallic based on Fe-Al-Si which are appropriate materials for high-temperature applications. A conventional metallurgical process – casting – was compared with a novel processing method, based on ultra-high energy mechanical alloying (MA) and spark plasma sintering (SPS) in order to prepare FeAl₂₀Si₂₀ (in wt. %) alloy. The influence of production route was observed. Via MA + SPS can produce a material of the same phase composition but with significantly better mechanical properties. Negligible porosity, fine-grained and homogeneous structure are the reasons.

Keywords: Intermetallics, Mechanical properties, Powder metallurgy, Spark Plasma Sintering

1 Introduction

Due to the development of modern technologies, especially in the aviation or aerospace industry, the requirements for used materials are increasing. For example, in order to reduce the weight of the means of transport, interest in the development of light alloys with high strength is growing [1, 2]. For high temperature applications, materials based on intermetallic phases are developed [3]. Iron aluminides have become interesting since the 1930s, when corrosion resistance of alloy with more than about 18 at % Al was first described [4]. Intermetallic based on FeAl are relatively low-cost materials and have high specific strength, as well as excellent corrosion resistance at elevated temperatures under oxidizing, carburizing and sulfidizing atmospheres. These features make the FeAl intermetallic compound a very attractive material for industrial applications [5-7].

A relatively simple way to improve the utility of the material is to refine its grains [3]. Nanocrystalline materials, i.e. those having a grain size of up to 100 nm [8], exhibit markedly higher yield strength and hardness values compared to coarse-grained material of the same composition [9, 10]. On the other hand, the negative grain refinement effect was observed in creep behavior, where the coarse grain is better. The grain size is mostly associated with the production process. Conventional metallurgical method such as casting and forming often produce coarse grain materials due to low cooling rates. The solubility of the alloying elements in the matrix, unwanted segregation or the too high melting point of the components is also limiting [11-13]. However, there are already known manufacturing processes which allow one to overcome these problems [5]. Nanocrystalline materials can generally be prepared in several ways. The most commonly used processes for the preparation of nanocrystalline materials include powder metallurgy (PM) or intensive plastic deformation using the Equal-Channel Angular Pressing (ECAP) or High Pressure Torsion (HPT) method [9].

The PM method involves two main steps: the first one is powder production, the second powder compaction. There is a whole range of methods for making the powder and it is necessary to bear in mind that the method of

production affects the final powder morphology. Mechanical alloying, which means high-energy ball milling is a powder processing technique that allows preparation of homogeneous materials starting from blended elemental powder mixtures. The MA process leads to an alloy formation by solid-state reactions, which means that materials with very different melting temperatures can be easily alloyed [4, 10]. The mechanism of mechanical alloying is based on repeated cold welding of particles by plastic deformation and breaking these welds due to the high kinetic energy of balls [9, 14]. During the MA process, two steps can be observed: at first, the particles size is growing due to the formation of cold welds, consequently brittle particles are broken thanks to their strong deformation strengthening [5].

During sintering of the powder, the particles are joined together and the powder forms a compact material. For compacting, numerous processes can be used. Over the last few years, the method called Spark Plasma Sintering (SPS) plays leading role among them. In SPS method, the powder is placed in a graphite or tungsten carbide tool between two pressing punches being also electrodes. Electric current is applied directly through the uniaxially pressed powder. Simultaneous application of pressure and heat by Joule effect leads to rapid compaction of the powder. While applied static pressures up to hundreds of MPa allows to prepare near-full density compacts, high sintering rate and the lower sintering temperature can effectively restrain the grain growth [15-18].

2 Experimental

The intermetallic alloy FeAl₂₀Si₂₀, were prepared by two different production process. In the first case, vacuum arc melting and casting was chosen as one of the most common manufacturing methods. The second sample was prepared from elemental powders by mechanical alloying under a protective argon atmosphere (purity of 99.996 %) followed by Spark Plasma Sintering (SPS) compaction. The powder mixtures for milling contained 60 % Fe, 20 % Al and 20 % Si (by weight) and total batch was 5 g, it means that the ball-to-powder weight ratio was 70:1. The process was carried out in a planetary ball mill (Retsch

PM 100) for 4 h with change of rotation direction each 30 min.

Mechanically alloyed powders were compacted by Spark Plasma Sintering (SPS) under the following conditions: sintering temperature of 1000 °C, heating rate of 300 °C.min⁻¹ to 900 °C and then 100 °C.min⁻¹ to 1000 °C, 10 min at the sintering temperature, pressure of 48 MPa. The sample was then slowly cooled in the chamber (50 °C.min⁻¹) to prevent its cracking.

The phase composition was examined by X-ray diffraction analysis using PANalytical X'Pert Pro diffractometer. Microstructure of samples was observed after etching by modified Kroll's reagent (10 ml HNO₃, 5 ml HF, 85 ml H₂O) on metallographic microscope Olympus PME3, as well as TESCAN VEGA 3 LMU scanning electron microscope equipped with Oxford Instruments X-max 20 mm² EDS detector with Aztec software package. Hardness were measured by Vickers method with the load of 30 kg (HV30). Each measurement was repeated ten times. Mechanical properties of the materials were measured using LabTest 5.250SP1-VM universal loading machine.

Fracture toughness has also been measured. However, direct measurement techniques were not used for this property, but fracture toughness was evaluated based on the lengths of the cracks after Vickers indentation, by Palmquist method. 10 imprints with a load of 1 kg were applied to the polished surface and subsequently the lengths of the cracks were measured. Indentations were observed with the Olympus PME3 optical microscope. Fracture toughness was calculated using equation (1):

$$K_{Ic} = 0,016 \cdot \left(\frac{E}{HV} \right)^{\frac{1}{2}} \cdot \left(\frac{F}{c^2} \right)^{\frac{1}{2}} \left[\text{Pa} \cdot \text{m}^{\frac{1}{2}} \right] \quad (1)$$

Where:

E...modulus of elasticity [Pa]

HV...Vickers hardness [-]

F...the load [N]

c...half the length of the crack after the impression [m] (Figure 1).

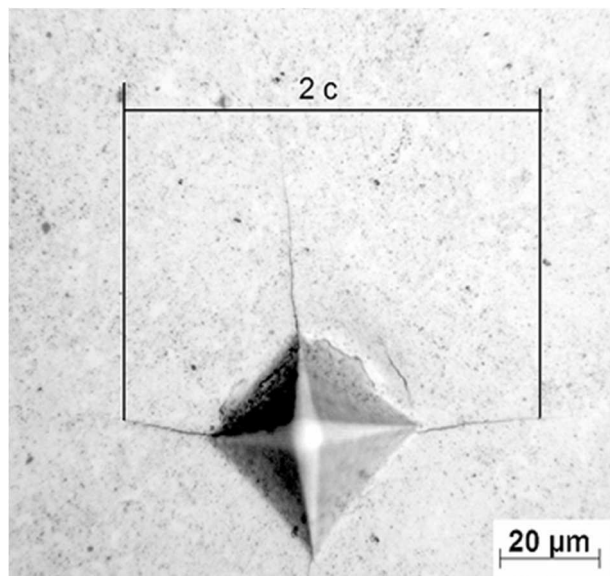


Fig. 1 Measurement of fracture toughness

3 Results and discussion

Phase composition of the individual alloy was determined by X-ray diffraction. It is interesting, that both of alloy had the same phase composition (Tab. 1), however in the case of sample prepared by MA+SPS, Si was substituted by Al up to 25 % in the phase Fe₃Si. The same phenomenon was achieved for cast alloy but to a lesser extent. The ternary phase of Fe-Al-Si is probably Fe₃Al₂Si₃. Precise identification is difficult because of overlap of diffraction lines, when the broadening due to fine grain structure is the reason.

Tab. 1 Phase composition of prepared alloys

	Phase		
	FeSi	Fe ₃ Si	Fe ₃ Al ₂ Si ₃
Fe-Al-Si (SPS)			
Fe-Al-Si (cast)			

However microstructure of each material was completely different. Fig. 2 shows the microstructure of samples acquired by an optical microscope. In the sample prepared by SPS method, ultrafine and homogenous structure was observed. Also porosity was very low, only 0.06 ± 0.02 area % by image analysis. It means that the density of the prepared material was very close to the theoretical density. In the cast sample there was a noticeably greater inhomogeneity of the microstructure, we can see a matrix in which all three elements are located - Fe₃Al₂Si₃, then light areas that represent the silicides - FeSi, and the darker parts, which represent also silicides - Fe₃Si. We can even see much higher ratio of porosity - 5.7 ± 4.0 % and the cracks that are present in the material after casting (see Fig. 2b).

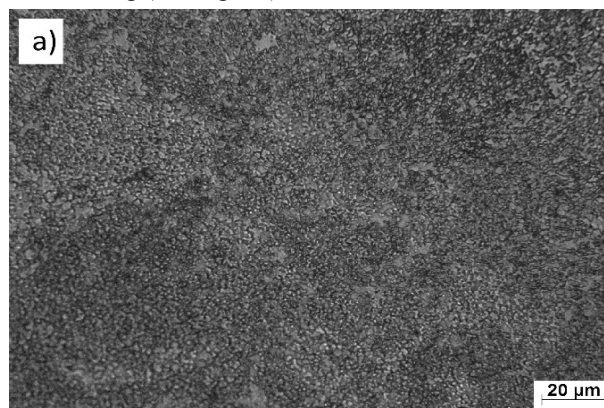


Fig. 2a Microstructure of sample prepared by SPS

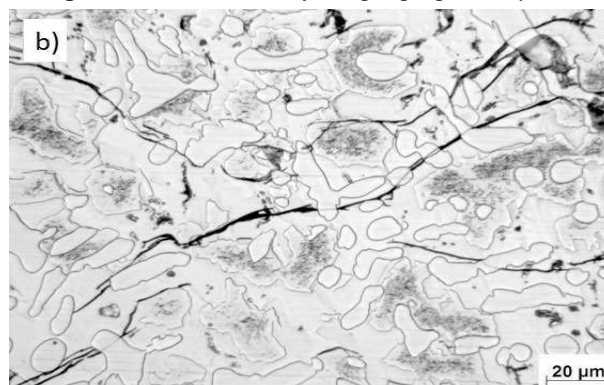


Fig. 2b Microstructure of sample prepared by casting

Although both materials had the same chemical, respectively phase composition as mentioned above, higher hardness (see Fig. 3) was observed for the sample prepared by mechanical alloying and subsequent compaction by the SPS method, finer structure and also strain hardening which is proceeded during mechanical alloying is the reason. The same trend was confirmed during the measurement of the fracture toughness (K_{Ic}), when K_{Ic} for material prepared via MA and SPS was $3.56 \pm 0.34 \text{ MPa}\cdot\text{m}^{1/2}$, however cast material showed fracture toughness up to ten times lower. In connection with the above results, we can state that refinement of the structure is indeed a suitable way to increase hardness and reduce brittleness at room temperature.

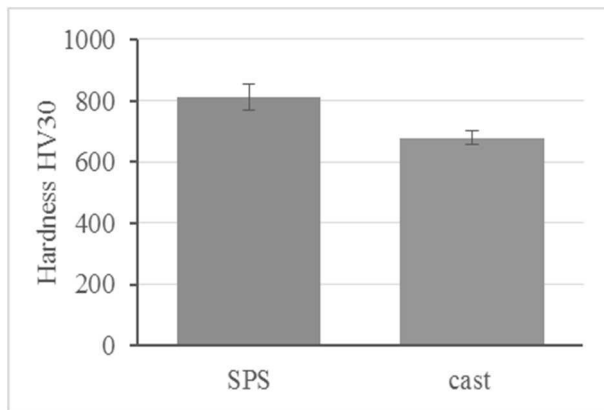


Fig. 3 Hardness (HV30) vs. manufacturing process

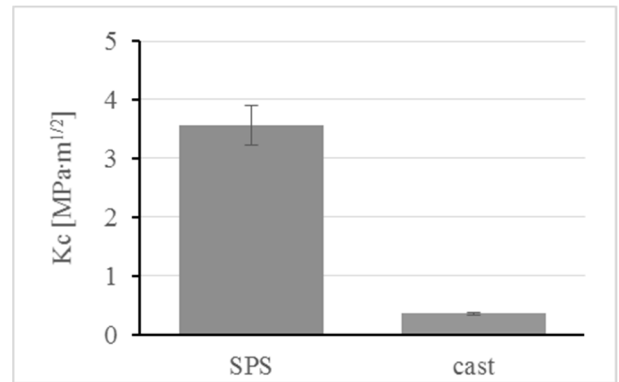


Fig. 4 Fracture toughness (K_{Ic}) vs. manufacturing process

Mechanical testing revealed that the finer structure as well as strain hardening have significant effect on the hardness and even more pronounced influence on the yield/ultimate strength, as presented in Fig. 4. The reason probably lies in the large number of boundaries, which act as barriers to the movement of defect or propagation of cracks. About strain hardening, during mechanical alloying the material is plastically deformed and the number of lattice defects increases, the dislocation slip is becoming more and more challenging, which leads to hardening of the material.

From the perspective of cast structure, the problems were the cracks present in the material already after casting. These defects easily propagate under applied stress and lead to material failure at low strain.

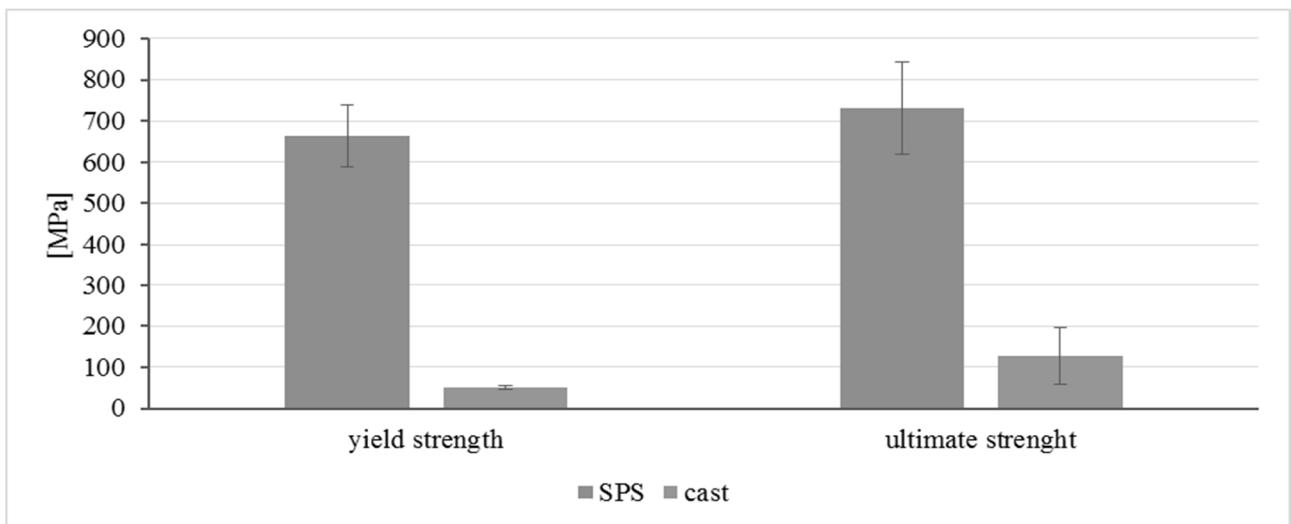


Fig. 4 Yield and ultimate compressive strength vs. manufacturing process

4 Conclusion

By using of mechanical alloying and Spark Plasma Sintering it is possible to prepare intermetallic alloys based on the Fe-Al-Si system without cracks with a porosity close to the theoretical density ($0.056 \pm 0.015\%$). Structure of the obtained material consists of mixture of FeSi, Fe₃Si and Fe₃Al₂Si₃. Cast alloy had the same phase composition, but microstructure was coarser with visible defects and also much higher porosity. These are the rea-

sons why hardness, fracture toughness as well as yield/ultimate strength were inferior in the case of cast material. The sample prepared by MA + SPS exhibits a high hardness of $811 \pm 42 \text{ HV}_{30}$, which is about 20 % more than of the cast material. The fracture toughness of MA+SPS prepared and cast FeAl₂₀Si₂₀ alloy was 3.56 ± 0.33 and $0.35 \pm 0.02 \text{ MPa}\cdot\text{m}^{1/2}$, respectively. Prepared FeAl₂₀Si₂₀ alloy is a promising material that will be tested further (e.g. other mechanical properties or resistance against oxidation and also corrosion resistance).

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