

Microstructure of TiAl15Si15 Alloy Prepared by Powder Metallurgy

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This work deals with the microstructure of TiAl15Si15 intermetallic compound, which can be used as a high-temperature low-weight material especially for the automotive and aerospace industry. A combination of high mechanical properties, good oxidation resistance and low density of this material is a good potential for use this alloy in many applications. The TiAl15Si15 alloy was prepared by different techniques of powder metallurgy. In this work, microstructure after reactive sintering, reactive sintering in a combination of Spark Plasma Sintering and mechanical alloying followed by Spark Plasma Sintering was described. The results were compared with the same alloy prepared by arc melting.

Keywords: Reactive sintering, Mechanical alloying, Spark Plasma Sintering, Arc melting

1 Introduction

Titanium alloys with the other light-metals (aluminum or silicon) are very interesting materials for aerospace or cosmic industry, especially for use at high temperatures. They can find use as the structural materials, operating under static high-temperature loads [1,2]. Ti-Al-Si alloys have been widely considered for high-temperature application because they offer a combination of low density (3.9 - 4.2 g/cm³ [3]), good oxidation resistance and useful mechanical properties at higher temperatures than conventional titanium alloys [4-6]. Addition of niobium, chromium, vanadium and predominantly silicon to TiAl alloys improves the high-temperature behaviour of these alloys [7,8]. It is necessary to design a material, which has the composition as close as possible to the eutectic - that the eutectic phases are very fine. A big problem is the brittleness of silicides, so they need to be very fine or fibrous so that the fracture toughness of the material will be increased [4].

A lot of effort in finding solutions to improve low ductility at room temperature, strength and toughness at high temperatures has been expended and some solutions are very promising [9,10]. The principle of these methods is to control the microstructure by either microalloying [11] or thermo-mechanical processing [12], or by chemical composition [9,13]. The synthesis of TiAl intermetallic alloy is one of the most promising ways in the design of new metallic materials with high thermal stability and heat resistance. TiAl alloys can be prepared by conventional casting techniques, melting in an electric arc furnace under an argon atmosphere or by powder metallurgy techniques and others. Melting of TiAl alloys is a multi-step process, the process involves melting of an alloy, casting into the mold, extended isothermal annealing of ingot and controlled rapid crystallization of the molten metal [1]. The preparation of Ti-Al-Si alloys by melting metallurgy is very complicated, because of high melting points of intermetallic phases, exothermic reactions during the formation of these phases and high reactivity of the melt with the melting crucibles [14-16]. Sharp-edged coarse silicides of Ti₅Si₃ phases are formed by melting metallurgy of Ti-Al-Si alloys. Coarse oriented sharp-

edged silicides have the negative effect on mechanical properties because they decrease the fracture toughness of the resulting material [8,17]. Powder metallurgy is a promising method for preparation intermetallic alloys based on Ti-Al-Si system, it allows to produce components of very complicated shapes without the additional machining. Powder metallurgy enables to combine the elements with different melting points, different solubility or different density. The disadvantages of powder metallurgy are high prices of pure metal powders or limitation of resulting shape and size of products [18-21]. The using of reactive sintering (self-propagating high-temperature synthesis) or mechanical alloying in combination with subsequent compaction (in this case by Spark Plasma Sintering) allows the preparation of Ti-Al-Si alloys.

In this work, the development of the Ti-Al-Si alloys at UCT Prague was described. The microstructure of TiAl15Si15 alloy prepared by different powder metallurgy techniques was compared. The results were compared with the same alloy prepared by arc melting.

2 Experiment

The TiAl15Si15 intermetallic alloy was prepared by various techniques of powder metallurgy. First sample was prepared by reactive sintering. Powder of titanium, silicon and AlSi30 alloy were blended together. The mixture of powder was prepressed using a pressure of 420 MPa for 5 minutes. Prepressed samples were sealed in evacuated ampoules made of silica glass in order to proceed with the reactive sintering under vacuum. SHS of prepared samples was conducted in an electric resistance furnace at the temperature of 900 °C for 30 minutes. The second sample was prepared by reactive sintering in a combination with Spark Plasma Sintering. The reactive sintered sample was milled in a vibrating laboratory mill for 7 minutes. Milled powder was consolidated by Spark Plasma Sintering with a pressure of 48 MPa, temperature 1100 °C for 15 minutes. Heating rate was 100 °C/min and cooling rate was 50 °C/min. The third sample was prepared by mechanical alloying followed by Spark Plasma Sintering. The mixture of pure titanium, aluminium and silicon powders were placed in a steel vessel together

with milling balls (ball-to-powder weight ratio was 70:1) and milled under a protective argon atmosphere. The mechanical alloying was carried out in a planetary ball mill (Retsch PM 100 CM). Milling duration was 4 hours, rotation velocity was 400 min^{-1} and rotation direction was changed every 30 minutes. The reference sample was prepared in arc melting furnace Bühler MAM-5 in the Institute of Physics of the ASCR. The pieces of individual elements (purity 99.95 %) were the four times remelted under the argon atmosphere.

The microstructure of prepared samples was observed by metallographic optical microscope Olympus PME3 and by electron microscope TESCAN VEGA 3 LMU equipped with EDS analyzer (SEM-EDS). The porosity and pore size were evaluated using the Lucia 4.8 image analyzer.

3 Results and discussion

First, the TiAl15Si15 intermetallic alloy was prepared by reactive sintering. The mixture of powders was pressed on a tensile LabTest 5.250SP1-VM and SHS process was carried out in an electric resistance furnace in silica ampoules at 900°C for 30 min. The microstructure of TiAl15Si15 alloy (Fig. 1) is characterized by a homogeneous structure with fine sharp-edged Ti_5Si_3 silicides (lighter parts) and TiAl matrix (darker parts on the figure). Formation of silicide Ti_5Si_3 is given by its high thermodynamic stability and the high mutual affinity of titanium and silicon. The small black spots on the Fig. 1 are pores, the porosity of this alloy is not so high, 0.56 vol. % (but there are very big pores, it is not possible to prepare larger compact material). Porosity was measured on optical microscope images using the Lucia 4.8 image analyzer. It is assumed that the volume fraction shall be equal to the flat rate with the isotropic distribution of pores that can be assumed in these alloys.

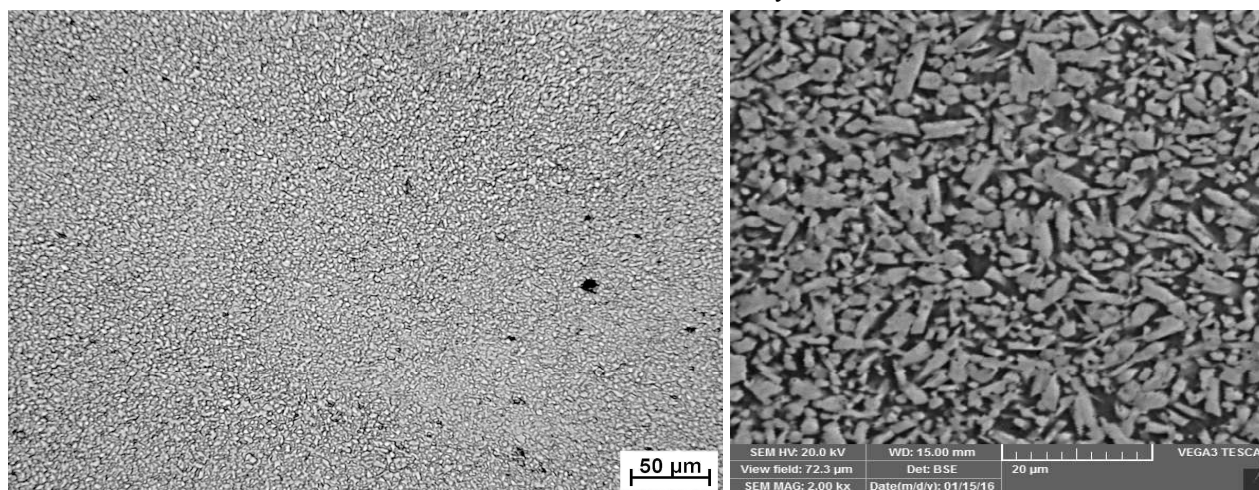


Fig. 1 Microstructure of TiAl15Si15 alloy prepared by reactive sintering

The microstructure of the TiAl15Si15 after reactive sintering and Spark Plasma Sintering is shown in Fig. 2. The phase composition does not change during the Spark Plasma Sintering process, it is the same as after reactive sintering. The particles of titanium silicides are not so sharp-edged, the shape of this particles is more rounded. This morphology of titanium silicides particles is more

acceptable due to better mechanical properties of the final structure. In the scanning electron micrograph, it is possible to see many small microcracks in the particles of titanium silicides Ti_5Si_3 . The cracks are probably caused by the effect of high pressure during SPS and they have been initiated and propagated by the thermal expansion of the particles of silicides. The porosity is 2.66 vol. %.

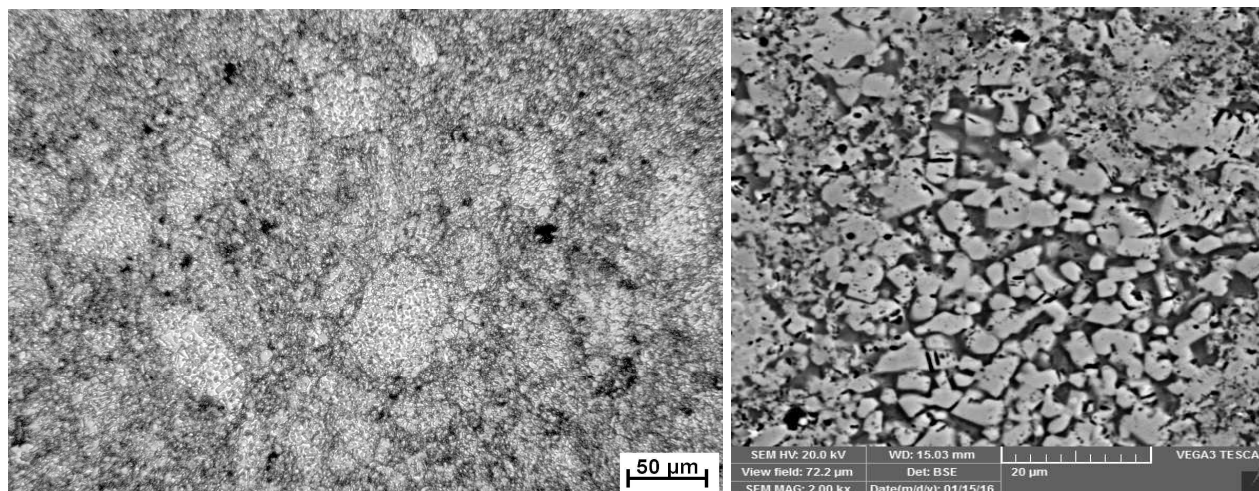


Fig. 2 Microstructure of TiAl15Si15 alloy prepared by reactive sintering and Spark Plasma Sintering

According to previous research, four hours of mechanical alloying of the TiAl15Si15 alloy from pure metal powders was chosen as a sufficient time to form powder consisted of only intermetallic phases. The milled powder was then consolidated by Spark Plasma Sintering with a pressure of 48 MPa, temperature 1100 °C for 15 minutes. The heating rate was chosen 100 °C/min and cooling rate was 50 °C/min. The scratch patterns were made from the compacted tablets, which were subsequently etched by the Kroll's agent to visualize the structure of the present aluminides and silicides. These aluminides have reduced chemical resistance and therefore they are visible as darker areas in micrographs. Silicides, on the other hand, are lighter. Due to the preparation of the samples by mechanical alloying

followed by compacting by SPS, a very fine-grained non-porous structure was gained.

The compacted TiAl15Si15 (Fig. 3) is formed by titanium silicides (Ti_5Si_3) in titanium aluminide matrix (TiAl , TiAl_2). The formation of titanium silicide is conditioned by the high affinity of silicon and titanium. The remaining titanium reacts with aluminium to the formation of the TiAl matrix. Except Ti_5Si_3 and TiAl , the TiAl15Si15 alloy contains also TiAl_2 phase, which also forms the matrix of this alloy. In the microstructure of compacted TiAl15Si15 intermetallic alloy, it is possible to see also the small light particles of iron, which are a contamination from milling steel container and steel milling balls. The porosity of this alloy is also very low, 0.42 vol. %.

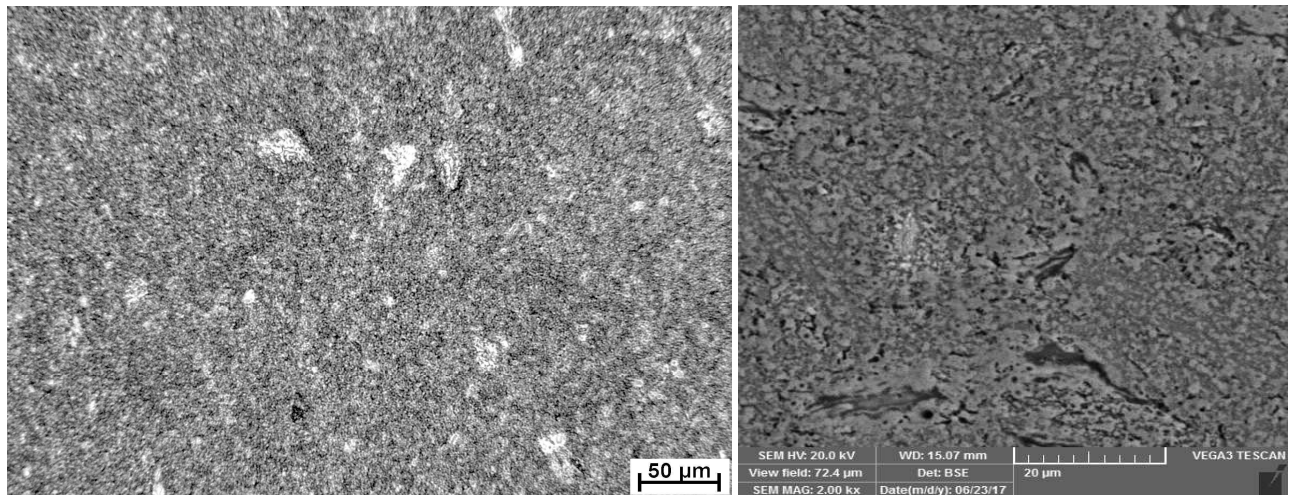


Fig. 3 Microstructure of TiAl15Si15 alloy prepared by mechanical alloying followed by Spark Plasma Sintering

The reference TiAl15Si15 alloy was prepared in an arc melting furnace under the argon atmosphere. The phase composition is the same as other TiAl15Si15 alloys, which are formed by Ti_5Si_3 silicides in TiAl matrix. The particles of titanium silicides are very coarse (Fig. 4). The microstructure is formed by various local morphology of titanium silicides, by primary silicides and silicides with

fibrous and lamellar morphology. Colonies are differently oriented depending on the local direction of heat transfer, which causes the stretching of silicides. Stretched silicides are cracked due to the high cooling rate after melting. The black particles in Figure 4 are pores. The pores in the cast alloy are concentrated in the middle of the sample.

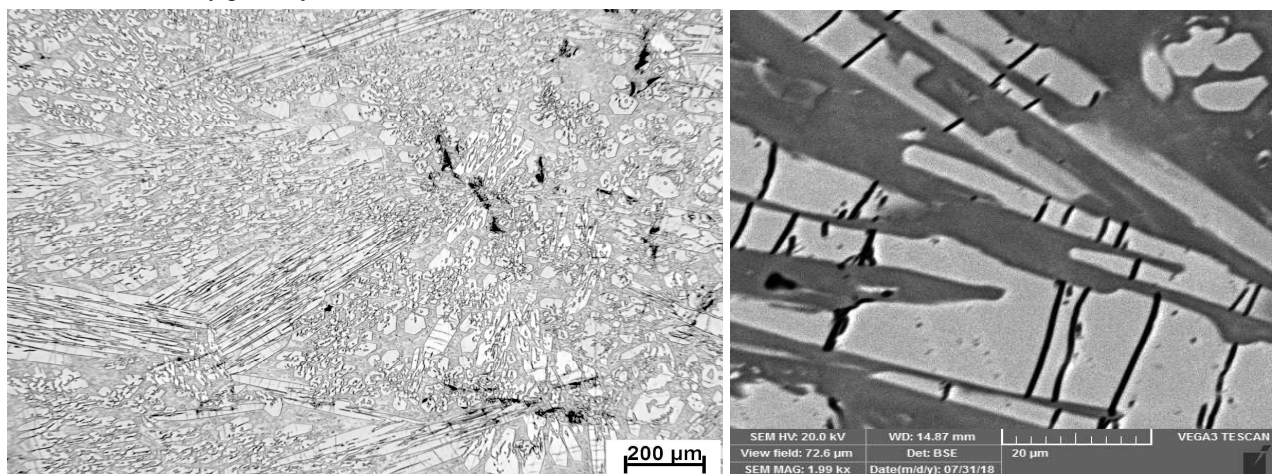


Fig. 4 Microstructure of TiAl15Si15 alloy prepared by arc melting

4 Conclusion

In this work, the microstructure of TiAl15Si15 alloy

prepared by various techniques of powder metallurgy was studied and each preparation methods were compared. The TiAl15Si15 alloy is always characterized by Ti_5Si_3

phase in TiAl matrix (in the case of mechanical alloying also TiAl₂ phase). The microstructure after reactive sintering is full of sharp-edged silicides, therefore, the other preparation step (Spark Plasma Sintering) was followed. Mechanical alloying followed by SPS brought better results, the microstructure was more homogeneous with finer phases. Arc melting of Ti-Al-Si alloys is not suitable due to the formation of very coarse silicides with many cracks.

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