Ternary Fe-Al-Si Alloys Prepared by Mechanical Alloying and Spark Plasma Sintering

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Ordered aluminides and silicides of transition metals are interesting structural materials with good corrosion resistance and high-temperature mechanical properties. They are cheap and they allow us to avoid use of critical raw materials as is chromium. Nevertheless, they suffer by brittleness at ambient temperatures and growth of large grains at conventional casting as other intermetallics. To evade complicated, non-homogeneous microstructure with overgrown intermetallic grains in cast materials we selected powder metallurgy technique of spark plasma sintering (SPS). We investigate set of samples with composition FeAl20Si20 (in wt. %), FeAl35Si5, and FeAl30Si10.

The powders for sintering are prepared by mechanical alloying from elemental powders. There starts to appear binary phases after 1 h of mechanical alloying. It was silicides Fe3Si and FeSi and aluminid FeAl in the case of FeAl20Si20 alloy. It seems that after 8 h mixture reached the equilibrium state. All presented phases are binary, but they contain all three elements (the division is given by space group of respective phase). Phases are off stoichiometric and even their lattice parameters differs from tabled data. There appear FeAl and Fe2Al5 phases in the case of FeAl35Si5 alloy, but Fe2Al5 phase disappeared after 4 h of milling. The situation in FeAl30Si10 alloy was similar; there appear two phases FeAl and Fe2Si again and Fe2Si disappeared after 2 h of milling [1].

The mechanically alloyed powders were sintered using the SPS device FCT Systeme HP D 10 and sintered products were analysed by standard set of methods including XRD, SEM and TEM [2, 3]. The different phases were found in sintered samples than in mechanically alloyed powders. We found Fe3Si, FeSi and Fe3Al2Si3 phases in FeAl20Si20 alloy by XRD and EBSD. It was necessary to prepare reference samples by arc melting to evaluate EBSD data as there is again significant difference in lattice parameters between actual and tabled data. Such difference is given mainly by composition, rather than kinetics as arc melted samples can be used to evaluate SPS samples. Thus, reference samples were crushed to get powder for XRD and refined parameters from these samples were used to evaluate EBSD maps from both reference and sintered samples. The simultaneous acquisition of EDS and EBSD signal helped in segmentation of phases, which was more realistic than results refined just from EBSD data (there Fe3Al grains do not have realistic shapes), Figure 1. Nevertheless, each phase contains all elements even in sintered samples and segmentation by EDS data was simply impossible, because grains are not only off-stoichiometric, but even small (crystallites size from XRD were estimated 10-30 nm). Nanoparticles of oxides (probably due to the processing) and amorphous phase were observed by TEM.
The massive study of all available sintering parameters was performed for FeAl20Si20 alloy [4]. The Fe3Al and Fe2Al5 phases were observed in arc melted and sintered samples FeAl35Si5, contrary to powder, where Fe2Al5 phases disappeared [5].

References:

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Figure 1. FeAl20Si20 alloy. a) XRD pattern from powder of reference sample with three found phases FeSi, Fe3Si and Fe3Al2Si3. b) EBSD orientation map of reference, arc melted sample; c) phase composition of arc melted sample: yellow FeSi, green Fe3Si and red Fe3Al2Si3; d) EBSD orientation map of SPS sample; d) its phase composition; color codes for inverse pole figures of f) Fe3Al2Si3; g) Fe3Si and h) FeSi.