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

Jaromír Kopeček^{1*}, František Laufek², Miroslav Karlík³, Petr Haušild³ Jaroslav Čech³, Andrea Školáková⁴, Kateřina Nová⁴, Boris Severa⁴, Jakub Šesták⁴, Pavel Novák⁴ and Filip Průša⁴

^{1.} Department of Functional Materials, Institute of Physics, The Czech Academy of Sciences, Na Slovance 1999/2, Prague, 182 00, Czech Republic.

^{2.} Czech Geological survey, Geologická 6, Prague 5, 152 00, Czech Republic

^{3.} Department of Materials, Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague, Trojanova 13, Prague, 120 00, Czech Republic.

^{4.} Department of Metals and Corrosion Engineering, University of Chemistry and Technology in Prague, Technická 5, Prague, 166 28, Czech Republic.

* Corresponding author: kopecek@fzu.cz

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 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

[3]. 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:

- [1] K. Nová et al, Metals 9 (2018) p. 20.
- [2] P. Haušild et al, Key Eng. Mater. 784 (2018) 15.
- [3] P. Haušild et al, Acta Phys. Pol. A 134 (2018) 724.
- [4] F. Průša at al, Mater. Sci Engn. A, submitted.

[5] This research was carried out in the frame of the project 17-07559S and of the project No. CZ.02.1.01/0.0/0.0/15_003/0000485, financed by ERDF; in part by the MEYS SAFMAT CZ.02.1.01/0.0/0.0/16_013/0001406, LO1409 and LM2015088 projects (SEM Tescan FERA 3 maintenance).



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.