

High-temperature Behaviour of New Fe-Al-Si Alloy Produced by Powder Metallurgy

Pavel Novák, Kateřina Nová, Tomáš Vanka, Filip Průša

Department of Metals and Corrosion Engineering, University of Chemistry and Technology Prague, Technická 5, 166 28 Prague, Czech Republic. E-mail: Panovak@vscht.cz.

This work describes properties of newly developed Fe-Al-Si alloy for high-temperature applications. This alloy is a material composed of FeAl, FeSi and Fe₃Al₂Si₃ intermetallic compounds, which was prepared by powder metallurgy using mechanical alloying and spark plasma sintering. The oxidation resistance at the temperatures of 800 and 1000 °C was described, as well as the mechanical properties up to 700 °C. During oxidation, the alloy covers by aluminium oxide layer, which protects it against further oxidation. Concerning the mechanical properties, the yield strength and ultimate compressive strength increase in the temperature interval of 400 – 500 °C.

Keywords: iron aluminide; iron silicide; powder metallurgy; high temperature

1 Introduction

Bulk intermediary phases of nickel, iron or titanium with aluminium are modern materials for high-temperature applications. They are characterized by lower density and significantly improved thermal stability as compared with common iron or nickel alloys [1,2].

Alloys based on Fe-Al system are cheaper than Ti-Al alloys, used in modern jet engines of the airplanes, but have higher density (approximately 5.7 g.cm⁻³). These materials based Fe₃Al or FeAl ordered phases have very interesting properties such as the excellent chemical resistance to high-temperature oxidation, sulfidation and corrosion in highly oxidizing salts and anomalous temperature dependence of yield stress. In some alloys based on FeAl and Fe₃Al phases, the yield stress increases with temperature in a short temperature interval [3].

During the past few decades, powder metallurgy has become an alternative processing route of many materials including also intermetallics. Rapid solidification techniques, such as melt atomization, are problematic due to high melting points of intermetallics [4], extreme reactivity of the melts and poor compressibility and sinterability of the intermetallic powders obtained [5]. Advanced techniques, e.g. hot isostatic pressing, hot extrusion or spark plasma sintering, should be applied for compaction of these rapidly solidified powders [5].

Among the perspective production methods of intermetallics, mechanical alloying has attracted great interest. This method involves preparation of intermetallic compounds from the mixture of pure metals powders by high-energy ball milling, i.e. high energy milling of powder mixtures, where the phase composition changes from individual elemental powders to solid solutions, chemical compounds or amorphous materials [6]. During mechanical alloying, following stages happen: Joining of particles by plastic deformation, crushing of particles, formation of solid solutions and intermetallics [7]. In addition to the simple formation of alloy or intermetallic phase powders, mechanical alloying produces ultra-fine grained or even nanocrystalline powders due to severe plastic deformation during the process.

In this work, high temperature mechanical properties and oxidation resistance of FeAl₂₀Si₂₀ alloy (in wt. %)

prepared by mechanical alloying and spark plasma sintering was studied.

2 Experimental

FeAl₂₀Si₂₀ alloy (in wt. %) was prepared by the combination of ultra-high energy mechanical alloying and subsequent spark plasma sintering. Mechanical alloying was carried out in planetary ball mill (Retsch PM 100 CM) under following conditions, optimized in our previous paper dealing with the synthesis of intermetallics [6]:

- milling duration: 240 min,
- change of rotation direction each 30 min,
- rotation speed: 400 rpm,
- atmosphere: argon
- powder batch: 5 g
- ball-to-powder weight ratio: 70:1.
- initial components: Fe, Al and Si powders of a purity above 99.5 %, particle size <44 µm.

Microstructure of the alloy was studied by an Olympus PME3 light microscope after etching by modified Kroll's reagent (5 ml HNO₃, 10 ml HF and 85 ml H₂O).

Mechanical properties of the SPS-consolidated material were determined by the means of hardness. Compression tests were carried out using LabTest 5.250SP1-VM universal loading machine (produced by LaborTech) at following temperatures: room temperature, 400, 500, 600 and 700 °C with the initial deformation rate of 0,001 s⁻¹. Thermal stability was evaluated by the hardness measurement and microstructure observation after annealing at 800 and 1000 °C for 400 h.

Isothermal oxidation tests were carried out at 800 and 1000 °C in air. Oxidation rate was determined from weight gains caused by the oxide formation on the surface of thermally exposed samples. In the oxidation tests, the samples were heated continuously in alumina crucibles for 100, 200, 300 and 400 h, followed by air-cooling.

3 Results and discussion

Microstructure of the FeAl₂₀Si₂₀ alloy is composed of iron silicides particles (Fe₃Si, FeSi) in a matrix of Al₂Fe₃Si₃ ternary phase, see Fig. 1. The size of silicide particles does not exceed 5 µm. Their geometry is almost rounded, which is good for the resulting mechanical properties of the material.

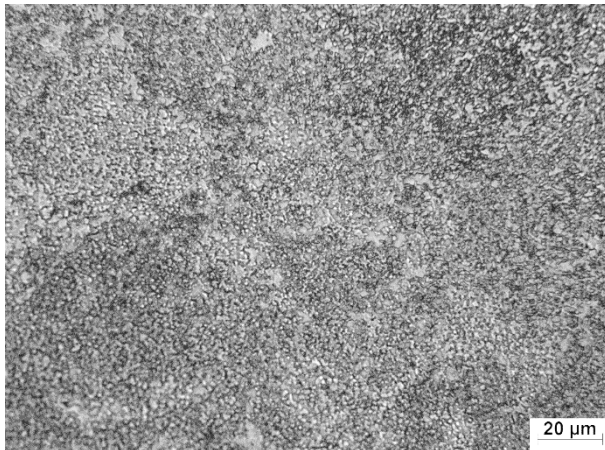


Fig. 1 Microstructure of FeAl₂₀Si₂₀ alloy prepared by mechanical alloying and spark plasma sintering

The yield strength of the alloy at room temperature is 1071 ± 110 MPa, while the ultimate compressive strength reaches 1085 ± 115 MPa. The fact that yield strength is very close to the ultimate compressive strength implies that the material is very brittle. Upon heating, the same behaviour can be observed also at 400 °C. At higher temperatures, larger plasticity was observed. In addition, the yield strength and ultimate compressive strength increase up to 1163 and 1508 MPa at 500 °C, respectively (Fig. 2). This behaviour has been previously observed for aluminide-based alloys [3] and very weakly also in Fe₃Si iron silicide [8]. In the case of ternary Fe₃Al₂Si₃ phase, which forms a matrix of the alloy, the yield strength anomaly has never been reported before.

The anomaly of yield strength and ultimate compressive strength at the temperatures around 500 °C opens interesting range of applications. These temperatures are common for exhaust valves of diesel internal combustion

engines. During normal operation, the temperature reaches approx. 400 °C and during the cleaning procedure of the filter of solid particles it increases to approx. 500 °C [9]. These conditions were proved to be optimal for the use of this alloy.

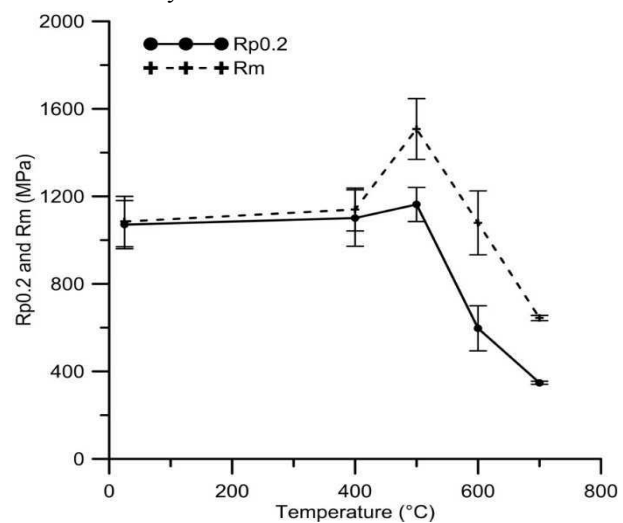


Fig. 2 Temperature dependence of mechanical properties in compression (yield strength Rp0.2 and ultimate compressive strength Rm) of FeAl₂₀Si₂₀ alloy

Hardness of the FeAl₂₀Si₂₀ alloy vs. duration of annealing at 800 and 1000 °C is presented in Fig. 3. It can be seen that the hardness does not change significantly after the exposure at both 800 and 1000 °C. This fact was confirmed by observation of microstructure after annealing, because no significant coarsening of silicide particles was observed even after the exposure for 400 h, see Fig. 4.

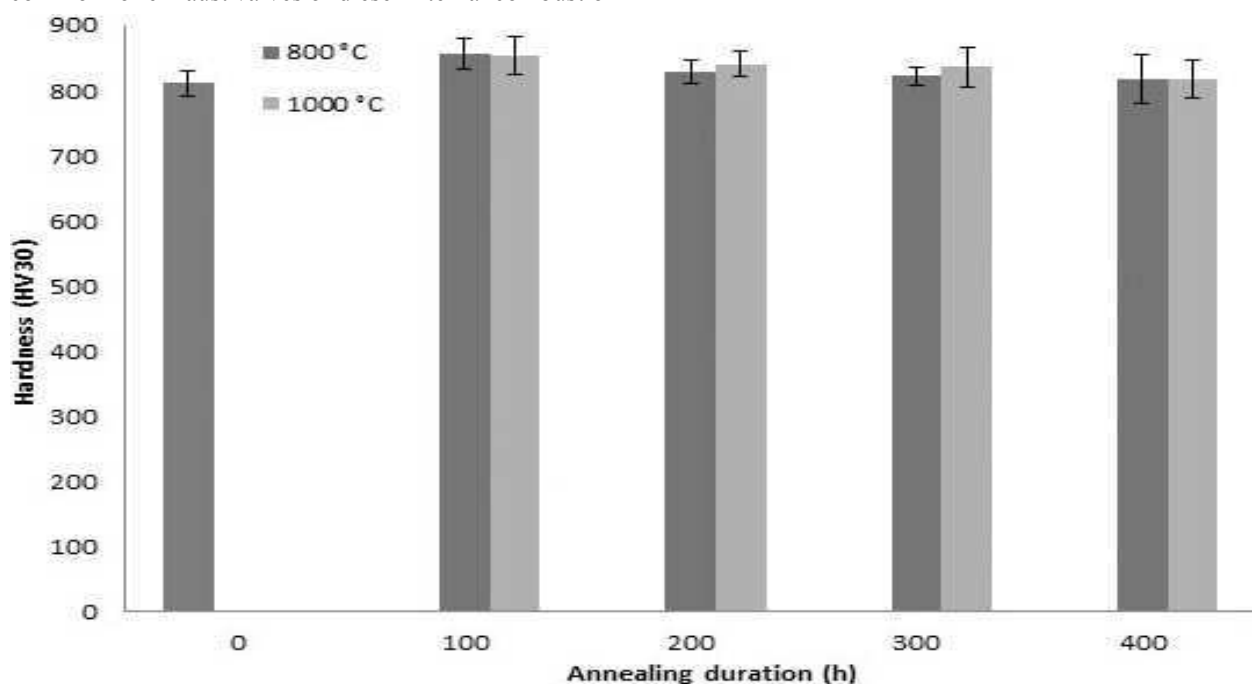


Fig. 3 Hardness of FeAl₂₀Si₂₀ alloy vs. duration of annealing at 800 and 1000 °C

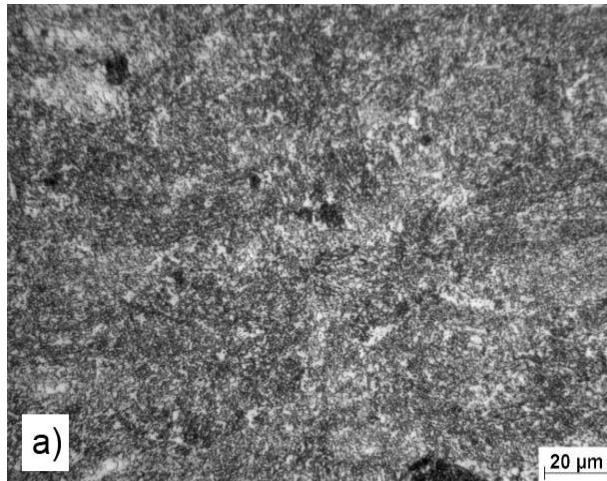


Fig. 4a Microstructure of FeAl₂₀Si₂₀ alloy after annealing at 800 °C for 400 h

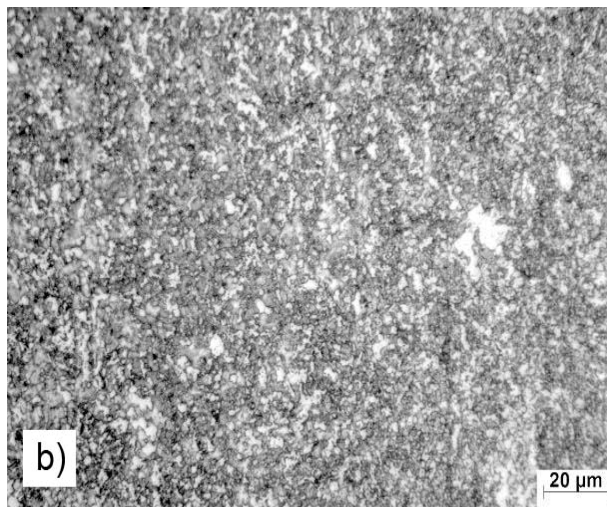


Fig. 4b Microstructure of FeAl₂₀Si₂₀ alloy after annealing at 1000 °C for 400 h

The oxidation rate of the alloy, represented by weight gains caused by isothermal oxidation, is approx. two times lower at 800 °C than at 1000 °C (Fig. 5). The growth of the oxide layer at 800 °C is linear at the beginning and after 300 h it follows the parabolic shape, see Fig. 5. It means that the early stages of oxidation are controlled by chemical reaction and therefore it follows the linear law. After reaching certain thickness and compactness of the oxide layer, the oxidation is controlled by the diffusion of oxygen through the oxide layer, thus following the parabolic law. On the other hand, the oxidation at 1000 °C follows the parabolic law right from the beginning of the oxidation test (Fig. 5). The reason for this behaviour can be found in the phase composition of the oxide layer. At 800 °C the γ -Al₂O₃ (cubic) is formed, being nearly immediately transformed to δ -Al₂O₃ (tetragonal or orthorhombic). It induces cracks in the oxide layer and lowers the protective effect of the layer in the initial stage of the oxidation. On the other hand, at 1000 °C α -Al₂O₃ (corundum, trigonal) is formed, which is stable and compact and therefore it acts as the oxidation barrier right from the beginning of the exposure [10].

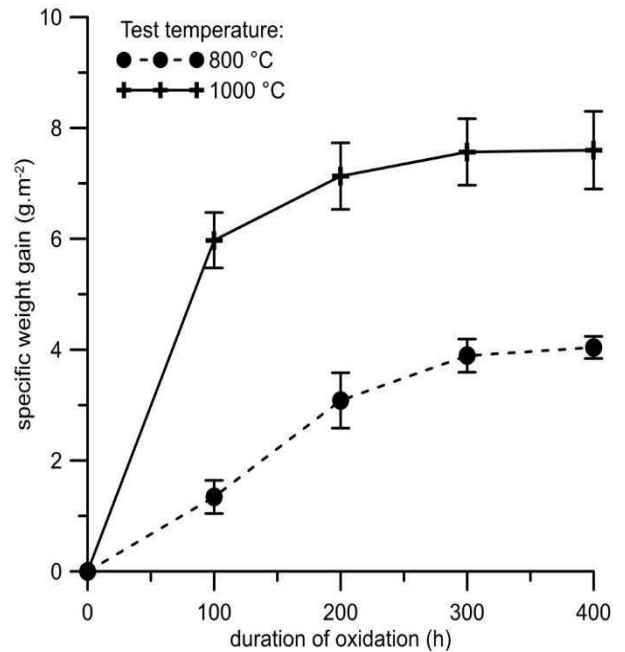


Fig. 5 Dependence of specific weight gain on duration of oxidation at 800 and 1000 °C

4 Conclusion

In this work, the FeAl₂₀Si₂₀ alloy (in wt. %) was prepared by the combination of mechanical alloying and spark plasma sintering. The alloy exhibited very fine structure, composed of silicides (Fe₃Si and FeSi) and ternary phase (Fe₃Al₂Si₃). The ultimate compressive strength of the alloy was nearly 1100 MPa at room temperature. Due to anomalous behaviour, it increased to approx. 1500 MPa at 500 °C. The FeAl₂₀Si₂₀ alloy exhibits excellent oxidation resistance at 800 and 1000 °C in the air and excellent thermal stability at these temperatures. Due to superior oxidation resistance and mechanical behaviour at the appropriate temperatures, the material could be applicable in automotive industry for the manufacture of exhaust valves of diesel engines.

Acknowledgement

This research was financially supported by Czech Science Foundation, project No. 17-07559S.

References

- [1] LEE, D., SANTELLA, M.L., ANDERSON, I.M., PHARR G.M. (2005) Thermal aging effects on the microstructure and short-term oxidation behavior of a cast Ni₃Al alloy. In: *Intermetallics*, Vol. 13, pp. 187–196.
- [2] MORRIS, D.G., MUNOZ-MORRIS M.A. (2007) Development of creep-resistant iron aluminides. *Materials Science and Engineering A462*, pp. 45–52.
- [3] MORRIS, D.G., MUNOZ-MORRIS, M.A. (2010) A re-examination of the pinning mechanisms responsible for the stress anomaly in FeAl intermetallics, *Intermetallics*, Vol. 18, pp. 1279–1284.

- [4] MASSALSKI, T.B. (1990) *Binary Alloy Phase Diagrams*, ASM Int.: Materials Park.
- [5] VAN DYCK, S., DELAEY, L., FROYEN, L., BUEKENHOUT, L. (1995) Reactive powder metallurgy of Ni₃Si-based alloys. In: *Intermetallics*, Vol. 3, 309–314.
- [6] NOVÁK, P., ŠKOLÁKOVÁ, A., VOJTĚCH, V., KNAISLOVÁ, A., POKORNÝ, P., MORAVEC, H., KOPEČEK, J., KARLÍK, M., KUBATÍK, T.F. (2014) Application of Microscopy and X-ray Diffraction in Optimization of the Production of NiTi Alloy by Powder Metallurgy, In: *Manufacturing Technology*, Vol. 14, pp. 387-392.
- [7] VALALIK, M., NOVÁK, P., KUBATÍK, T.F., VOJTĚCH, D. (2015) Unconventional Method of Preparation Intermetallic Phases Fe-Al by Mechanical Alloying in Comparison to Reactive Sintering, In: *Manufacturing Technology*, Vol. 15, pp. 105-109.
- [8] SCHAEFER, H.E., FRENNER, K., WURSCHUM R. (1999) High-temperature atomic defect properties and diffusion processes in intermetallic compounds, In: *Intermetallics*, Vol. 7, pp. 277-287.
- [9] CASTELLANO, J., CHAUDHARI, A., AND BROMHAM, J. (2013) Adaptive Temperature Control for Diesel Particulate Filter Regeneration, *SAE Technical Paper* 2013-01-0517, doi:10.4271/2013-01-0517.
- [10] NOVÁK, P., ZELINKOVÁ, M., ŠERÁK, J., MICHALCOVÁ, A., NOVÁK, M., VOJTĚCH, D. (2011) Oxidation resistance of SHS Fe-Al-Si alloys at 800 °C in air, In: *Intermetallics*, Vol. 19, pp. 1306-1312.

10.21062/ujep/95.2018/a/1213-2489/MT/18/2/299

Copyright © 2018. Published by Manufacturing Technology. All rights reserved.