MICROSTRUCTURE AND MECHANICAL PROPERTIES OF HOT-PRESSED Fe₃Al/Al₂O₃ AND Fe₃Al/TiC COMPOSITES

Katarzyna Pietrzak^{1,2*}, Dariusz Kalinski¹, Marcin Chmielewski¹

¹ Institute of Electronic Materials Technology, 133 Wolczynska Str., 01-919 Warsaw, Poland ² Institute of Fundamental Technological Research, Polish Academy of Sciences, 5B Pawinski Str., 02-106 Warsaw, Poland *e-mail of corresponding author: katarzyna.pietrzak@itme.edu.pl

Keywords: Fe₃Al/Al₂O₃ composites, Fe₃Al/TiC composites, hot pressing

Abstract

The aim of the presented paper is the development of the Fe_3Al/Al_2O_3 and Fe_3Al/TiC composites, processed by hot pressing in argon atmosphere. The effect of Al_2O_3 or TiC ceramic phase content on physical and mechanical properties of composites were investigated.

The paper presents the conditions of the newly developed technologies, results of microstructure investigations, measurements of the selected physical and mechanical properties such: density, hardness, bending strength, Young modulus and fracture toughness. The measured selected properties of $Fe_3Al/20vol.\%TiC$ were: hardness (HV_{10}) 4.8GPa, bending strength 1160MPa, Young modulus 190 GPa, fracture toughness 9.7 [MPa·m^{1/2}].

1 Introduction

The iron aluminides based on Fe₃Al and Fe₃Al have been among the most widely intermetallics and are promising candidates for many structural applications at elevated temperature in hard environments. This is due to a combination of their very good properties: low density, high resistance to sulfidation in H₂S and SO₂ gases, excellent oxidation resistance at a temperature up to 1200°C, high electrical resistivity that increases with temperature, good corrosion resistance in many aqueous environments, relatively high strength and good wear resistance. However, iron aluminides are brittle at room temperature although the toughness of Fe₃Al is higher than that of other aluminides such as TiAl and NiAl. Moreover, the strength and creep resistance above 550°C are degraded. Improvement in mechanical properties of iron aluminides has been attempted through modification of crystalline structure by means of macro-alloying (several percent) and process, yielding a more ductile structure [1]. Previous studies have demonstrated that chromium is the most effective solution to achieve environmental embrittlement in Fe-Al-Cr alloy system [2] while the addition of Si, Ta, Ce, Zr, Mo, Hf or Nb increases high temperature creep and tensile strength of Fe₃Al at the expense of room temperature ductility [2, 3]. Apart from these alloy design aspects, composites consisting of iron aluminides and ceramic reinforcement such as particles, whiskers, and fibers have been processed with the purpose of enhancing the mechanical properties of iron aluminides. [3, 4, 5]. The research results have indicated that Fe_3Al and Al_2O_3 have better characterization, as there is no interface phase between them [3]. Taking in consideration the analysis of thermodynamics calculation [4] we can state that the morphology of this interface (and in definitively the properties of composite) depends in great measure on the conditions of process. For this reason, researches have looked into the fabrication technology of Fe_3Al/Al_2O_3 composites and found that the composites with different ratio demand different sintering conditions.

The aim of this paper is the development of the Fe_3Al/Al_2O_3 and Fe_3Al/TiC composites, processed by hot pressing in argon atmosphere. The effect of Al_2O_3 or TiC ceramic phase content on physical and mechanical properties of composites were investigated.

2 Materials and testing methods

In the presented investigations the commercial powders of Fe₃Al (Alfa Aesar), TiC (Goodfellow) and Al₂O₃ (NewMet Koch) were used. The average grain size measured by Clemex (Television Image Analyses) showed the following values: $d_{Fe3Al}=9,52\mu m$, $d_{TiC}=2.29\mu m$, $d_{Al2O3}=3.29\mu m$. SEM images of staring materials are shown in Fig.1.

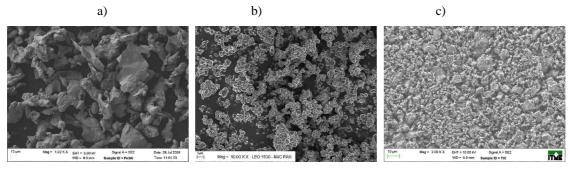


Figure 1. SEM images of starting materials: a) Fe₃Al, b) Al₂O₃, c) TiC.

Two compositions of powder mixtures with the following Fe₃Al to Al₂O₃ or TiC ratio (in vol.%) were prepared: $80Fe_3Al/20Al_2O_3(TiC)$, $70Fe_3Al/30Al_2O_3(TiC)$. They were obtained in a mechanical mixing process, using a planetary ball mill (Pulverisette 6, Fritsch) with tungsten carbide balls (\emptyset 10 mm). The process was conducted in the air at the different rotation speeds of 100 and 200 rpm and at a wide range of periods of time, from 1 to 8 hours. It has been found that with increasing mixing time and duration, the average grain size decreases. But the further prolongation of the mixing time to 8h did not result in appreciable changes in the morphology and composition of the powder mixtures. The ratio of the ball to the powder (BPR) was approximately 5:1. The SEM images and surface distribution of elements of powder mixtures after mixing process 200 rpm are shown in Fig.2, 3 and 4.



Figure 2. SEM images of powder mixtures Fe₃Al/30% Al₂O₃ (vol.%).

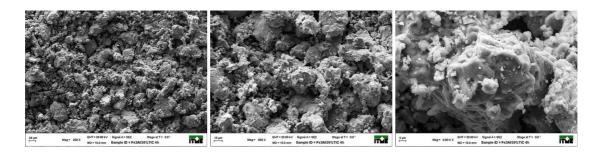
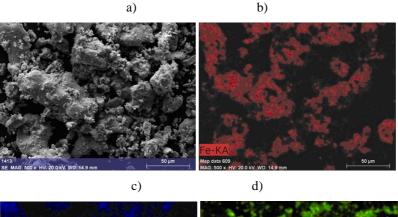


Figure 2. SEM images of powder mixtures Fe₃Al/20% TiC (vol.%). Mixing conditions: 200 rpm, time 4 hours.



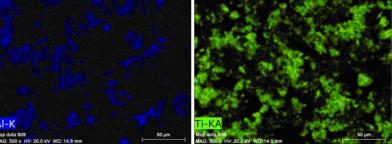


Figure 3. The microstructure (a) and surface distribution of Fe (b), Al (c) and Ti (d) of Fe₃Al/20%TiC (vol.%). powder mixtures.

After preliminary investigation, the process of sintering was performed for 30 min. under an argon atmosphere at 1150°C and an Astro Thermal Technology press was used at the pressure of 30 MPa. After the holding time, the samples were naturally cooled in the furnace to the room temperature before being removed.

The density of the obtained composites was measured according to the Archimedes method, and the porosity by using microscopy observation (Axivert 40 MAT) with Clemex software (Television Image Analyses). The measured average density of composites showed the following values $[g \cdot cm^{-3}]$: 6.2 - Fe₃Al/20vol.%TiC, 5.9 - Fe₃Al/30vol%TiC, 5.7 - Fe₃Al/20vol.%Al₂O₃, 5.4 - Fe₃Al/30vol%Al₂O₃.

The microstructure of produced composite materials was investigated using scanning electron microscope. The surface element distribution was determined by EDS, and the phase composition was examined using the XRD examination.

The hardness (HV10) was tested with a Vickers diamond indenter using a load of 49 N with loading time of 10 s. Each indentation was placed at least 10 diagonal lengths away from adjacent indentation. The hardness results were averaged over 10 indentations per specimens.

For obtained Fe₃Al/TiC (Al₂O₃) materials bending strength, fracture toughness and Young modulus were examined in a ZWICK 1446 strength machine at a support spacing of 40 mm

and a head travel speed of 1.0 mm/min. The samples intended for the bending strength and fracture toughness tests were sized at 5x5x50 mm, and the head load was 1 kN (bending strength) and 10 kN (fracture toughness). The fracture toughness was also tested by three-point bending. The samples were notched beams sized at 5x5x50 mm. The notch was made in the two stages: the samples were first notched to a depth of 0.9 mm using a wheel 0.2 mm wide and, then, deepened to about 1.1 mm (the total depth of the notch) with a wheel 0.023 mm wide. The straining rate, i.e. the head travel speed was 1.0 mm/min. The measurements of the Young modulus by the bending test (samples in the form of beams sized at 5x1x50 mm, support spacing – 40 mm) consisted of recording the load-induced deflection of the beam. The load ranged from 5 to 20 N.

3 Results

The selected results of obtained materials investigations (microstructure, physical and mechanical properties) are presented below.

Figures 4 - 9 show the microstructure of composites, their porosity and surface distribution of elements.

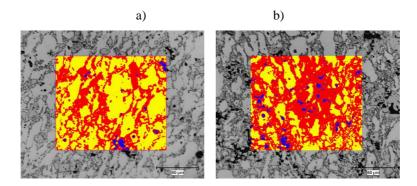


Figure 4. The image of porosity of Fe₃Al/TiC composites: a) Fe₃Al/20vol.%TiC, b) Fe₃Al/30vol%TiC; (yellow - Fe₃Al, red – TiC blue – porosity).

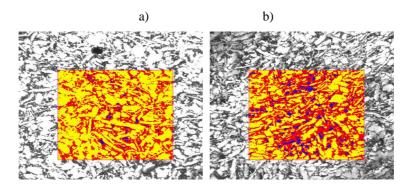


Figure 5. The image of porosity of Fe₃Al/Al₂O₃ composites: a) Fe₃Al/20vol.%Al₂O₃, b) Fe₃Al/30vol%Al₂O₃; (yellow - Fe₃Al, red - Al₂O₃, blue - porosity).

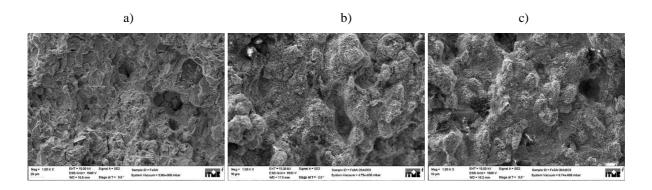


Figure 6. SEM images of a fracture: a) Fe₃Al, b) Fe₃Al/20vol.%Al₂O₃, c) Fe₃Al/30vol%Al₂O₃.

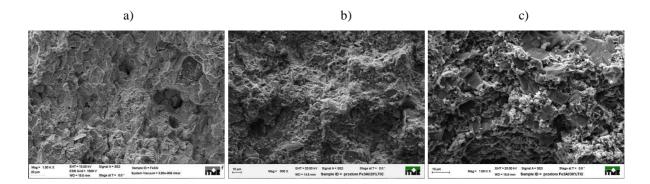


Figure 7. SEM images of a fracture: a) Fe₃Al, b) Fe₃Al/20vol.%TiC, c) Fe₃Al/30vol%TiC.

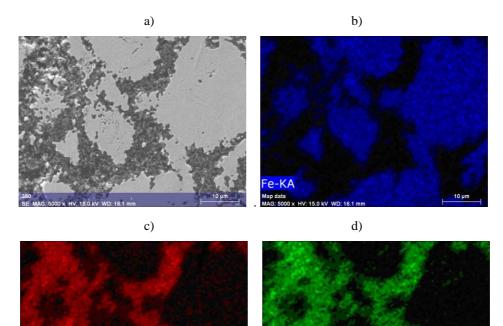


Figure 8. The microstructure (a) and surface distribution of Fe (b), Al (c) and O (d) of $Fe_3Al/20\% Al_2O_3$.

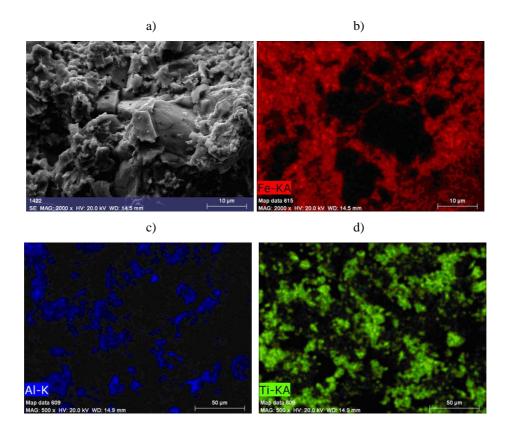


Figure 9. The microstructure (a) and surface distribution of Fe (b), Al (c) and Ti (d) of Fe₃Al/20% TiC.

Material (vol.%)	Young Moduls [GPa]	Bending strength [MPa]	K _{IC} [MPa·m ^{1/2}]	Hardness HV ₁₀₀ [GPa]
Fe ₃ A1	152.9	1547.0	35.2	3.16
Fe ₃ Al/20vol.%Al ₂ O ₃	74.0	526.7	5.3	3.76
Fe ₃ Al/30vol.%Al ₂ O ₃	53.9	281.0	3.1	3.12
Fe ₃ Al/20vol.%TiC	190.5	1157.0	9.7	4.1
Fe ₃ Al/30vol.%TiC	192.7	880.0	7.6	4.8

The obtained results of measurements of mechanical properties are presented in tables 1.

Table 1. Properties of hot-pressed Fe₃Al-matrix composites.

4 Conclusions

The effect of the technological parameters on the properties of the constituent powders and of the Fe₃Al/Al₂O₃ and Fe₃Al/TiC hot-pressed composites from a mixture of these powders with different composition was examined and the parameters were optimized. The experiments have shown that the physical and mechanical properties (such as e.g., density, resulting porosity, bending strength, Young modulus, fracture toughness and hardness) of the composite closely depend on the parameters of both the mixing and hot-press processes. From the point of view of the uniformity of the distribution of the ceramic reinforcement (TiC or Al₂O₃) within the Fe₃Al matrix, the most advantageous mixing parameters were found to be: the rotational speed ω =200 rev/min, mixing time t= 4 h, the ball-to-powder weight ratio (BPR) of 5:1 and the mill ball size of 10 mm. With the powder mixtures prepared at the

higher speed $\omega_2 = 200$ rev/min, the composites had a higher relative density (and hence lower porosity), better bending strength, and higher hardness. Their structure was more homogeneous and more compact.

The decisive improvement of the bending strength and hardness of the composite was achieved after optimization of the sintering process, in particular by increasing the sintering temperature.

The more positive results were observed for the Fe_3Al/TiC composites ($Fe_3Al/20\%$ TiC sintering degree 97\%) than for Fe_3Al/Al_2O_3 (sintering degree 93%).

The future works will be focused on investigations of high energetic method of mixing process (mechanical alloying), possible small amount of additives to the mixture of powders and using of other hot-pressure process conditions (higher pressure).

Acknowledgement

The results presented in this paper have been obtained within the project "KomCerMet" (contract no. POIG.01.03.01-14-013/08-00 with the Polish Ministry of Science and Higher Education) in the framework of the Operational Program Innovative Economy 2007-2013.

References

- [1] Uenishi K., Kobayashi K.F.: Processing of intermetallic compounds for structural applications at high temperature *Intermetallics* **4**, pp. 95-101, (1996).
- [2] Liu C.T., George E.P., Sikka V.K, Deevi S.C., in: Stoloff N.S., Jones R.H. (Eds.), Processing and Design Issues in High Temperature Materials, The Mineral, Metals and Materials Society, pp. 139-155, (1997).
- [3] M.R. Ghomashchi: 'Al₂O₃ reinforced Al/Ni intermetallic matrix composite by reactive sintering' *J. of Mater. Sci.* **30**, pp. 2849-2854, (1995).
- [4] Sun K., Li A., Cui X., Yin Y., Li P. Sintering technology research of Fe₃Al ceramic composites, *Materials Processing Technology*, **113**, pp. 482-485, (2001).
- [5] Peng L.M., Li H., Wang J.H., Gong M., high strength and high toughness ceramic-iron aluminide (Fe₃Al) composites, *Materials Letters*, **60**, pp. 883-887, (2006).