JOURNAL OF THEORETICAL AND APPLIED MECHANICS
59, 3, pp. 509-513, Warsaw 2021
https://doi.org/10.15632/jtam-pl/138322

SHORT RESEARCH COMMUNICATION

FE-AL BASED COMPOSITE REINFORCED WITH ULTRA-FINE AL_2O_3 OXIDES FOR HIGH TEMPERATURE APPLICATIONS

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In this paper, an Fe-Al based composite reinforced with ultra-fine Al_2O_3 oxides was obtained through sintering of aluminium, iron and mullite ceramic powders using self-propagated high temperature synthesis (SHS). The powder mixture with a 50%wt. content of the ceramic reinforcement was cold pressed and subsequently subjected to the sintering process in vacuum at 1200°C for 25 minutes under external loading of 25 kN. The complex microstructure of the Fe-Al matrix reinforced with ultra-fine Al_2O_3 oxides was found to be desired in high temperature applications since only 3% of the relative weight gain was observed after 100 hours of annealing at 900°C.

Keywords: cermet composites, reaction synthesis, powder methods, electron microscopy

1. Introduction

Intermetallic-ceramic composites (IMCs) are unique materials that combine the properties of ceramics and metals. As a consequence, they are finding continuously increasing interest of the industry in high temperature applications (Kainer, 2006; Murali *et al.*, 2003). Among conventional sintering techniques for IMCs manufacturing, the reactive sintering (Novák *et al.*, 2010) and SHS (Murali *et al.*, 2003; Sritharan and Murali, 2001; Sritharan *et al.*, 2001) are commonly used in the laboratory scale. However, in all just mentioned techniques the reinforcing phase powders should have a favourably smaller than 1 μm radius in order to ensure their uniform distribution in the material volume. Such fine powders could be obtained using a high-energy milling processes (Prasad Yadav *et al.*, 2012; Han *et al.*, 2006). Among the variety of ICMs, Al_2O_3 reinforced composites possess the best mechanical properties (Avraham *et al.*, 2006; Zhu and Abbaschian, 2000). ICMs are also characterized by excellent thermal stability, which is desirable in high-temperature applications.

Based on the novel phase transformation model for powder mixtures subjected to the high temperature sintering, reported by the authors of a previous study (Kopec *et al.*, 2020), the Fe-Al based composite reinforced with a higher content of ultra-fine Al_2O_3 oxides were obtained through conventional sintering of aluminum, iron and particulate mullite ceramic powders using a SHS and modified sintering conditions. This composite was characterized by a specific microstructure that is desired in high temperature applications. Therefore, its heat resistance is studied in this research. Additionally, the application of the phase transformation model has enabled one to produce a multi-phase composite using a relative simple and conventional sintering technique.

2. Materials and methodology

The initial powder mixture used for the sintering contained 33%wt. of iron, 17%wt. of aluminium and 50% wt. of the reinforcing mullite ceramic. The higher content of reinforcing ceramics, in comparison to the previous study (Kopec *et al.*, 2020), was used to maintain thermal stability of the composite. The analysis of the Fe-Al equilibrium system enabled one to determine the specific content of aluminium and iron. Milling parameters were determined on the basis of literature analysis. The grinding jar and milling balls of 10 mm diameter were manufactured from 100Cr6 steel. Milling balls mass to feed powder mass ratio was equal to 10:1. The preparation of the composite from a powder mixture to sinter was presented in Fig. 1. The microstructural observations were performed using a FEI Scios Field Emission Gun Scanning Electron Microscope (FEG-SEM) operating at 20 kV. The Vickers hardness was determined for a constant diagonal of the indentation (20 μm), using Meyer's law (Matysik *et al.*, 2015) for the loads of 300 G, 500 G and 1000 G in order to limit the influence of elastic deformation on the results captured, which was related to the ISE effect (indentation size effect).

grinding for 5 min with rotating speed of 450 RPM	→	mixing for 30 min in a turbulent static mixer	→	single-sided pressing in a 25 mm diameter die using a conventional hydraulic press at 900 MPa for 5 min	→	sintering in graphite dies and a vacuum chamber at 1200°C for 25 min with external load of 25 kN

Fig. 1. Schema of the composite preparation

3. Results and discussion

A microstructure of the sintered composite consisted of large, non-fragmented SiO_2 and Al_2O_3 reinforcing oxides in an $FeAl_3$ matrix (Figs. 2a,b). The phase transformation model for composites reinforced with a 20%wt. content of the mullite ceramics assumed in (Kopec *et al.*, 2020) was confirmed for the material investigated. The energy dispersive X-ray microanalysis performed in the area of the infiltrated SiO_2 ceramic (Figs. 2c,d, Table 1) shown an increased amount of oxygen and aluminum in the dark grey areas marked as "1", indicating the dominant content of aluminum oxide ceramics. On the other hand, diffusion of silicon atoms from SiO_2 ceramics to the intermetallic matrix found during infiltration into the porous ceramics led to formation of $FeAl_3$ and $Al_{4.5}FeSi$ (τ_6) phases. The τ_6 phase was mainly observed in the areas of ceramic reinforcement. The sintering temperature of 1200°C was high enough to initiate porous silica infiltration, which subsequently led to defragmentation of primary SiO_2 precipitates, Fig. 2c. The primary $FeAl_3$ matrix was transformed into the ternary $Al_{4.5}FeSi$ (τ_6) phase (Fig. 2c). Additionally, diffusion of oxygen atoms from SiO_2 ceramics to active aluminum atoms from the metallic liquid was observed, and dispersive Al_2O_3 particles were formed (Fig. 2d). It was concluded that the mechanism of growth of a new fine Al_2O_3 aluminum oxide precipitates confirmed correctness of the phase transformation model assumed in (Kopec *et al.*, 2020) for composites with the 50%wt. of ceramic reinforcement.

Table 1. Chemical composition of the composite specific areas

Wt.%	Al	Si	Fe	O
1	61.3	–	8.7	30.0
2	62.4	2.9	34.7	–
3	55.7	15.0	29.3	–

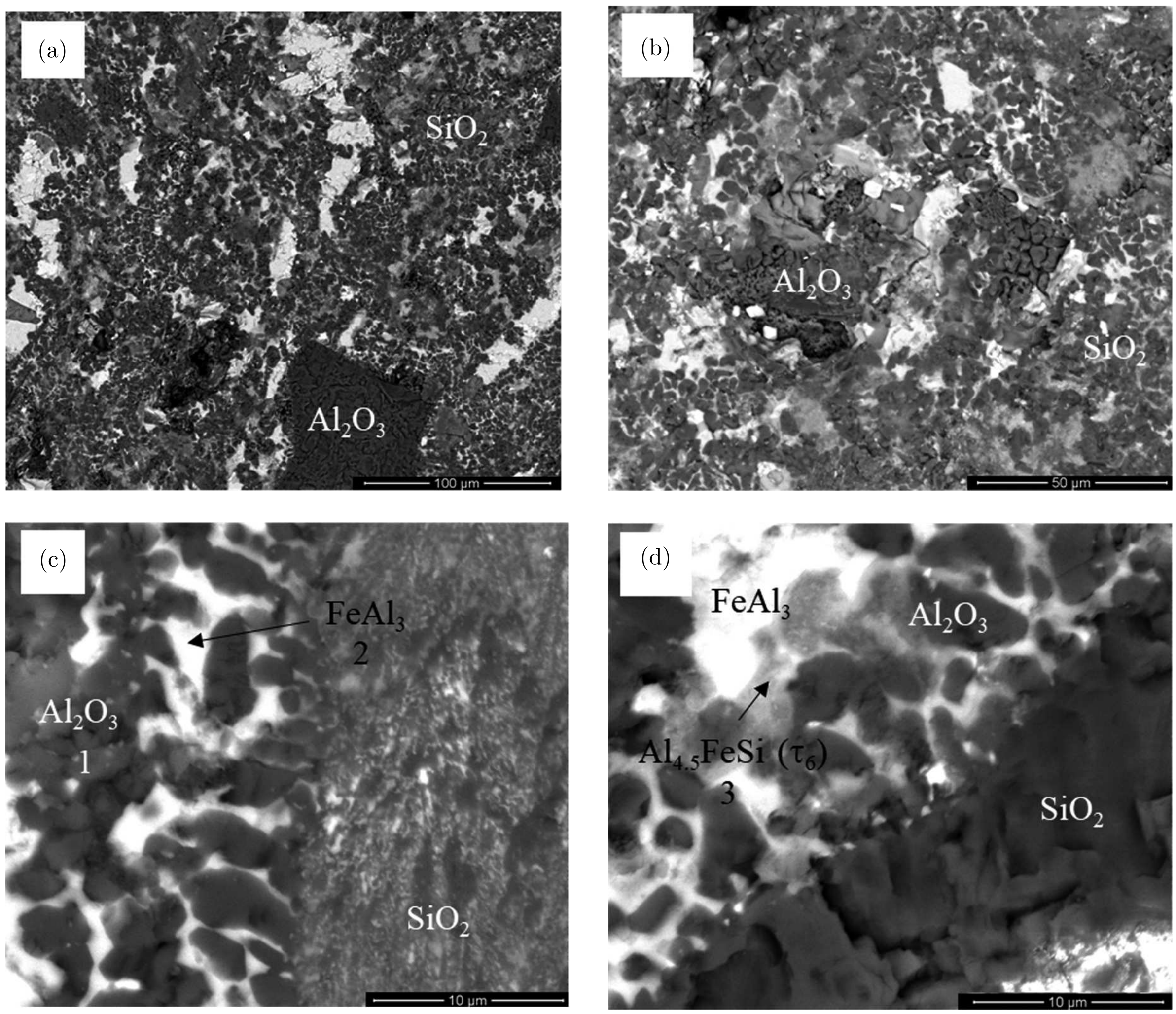

Fig. 2. Microstructure of the composite with a 50%wt. content of mullite ceramics after sintering at 1200°C for 25 minutes (a); mechanically bonded Al_2O_3/SiO_2 conglomerates (b); initiation of defragmentation of primary SiO_2 precipitates (c); infiltration of silica ceramics and growth of the fine Al_2O_3 oxides in the $FeAl_3$ matrix (d)

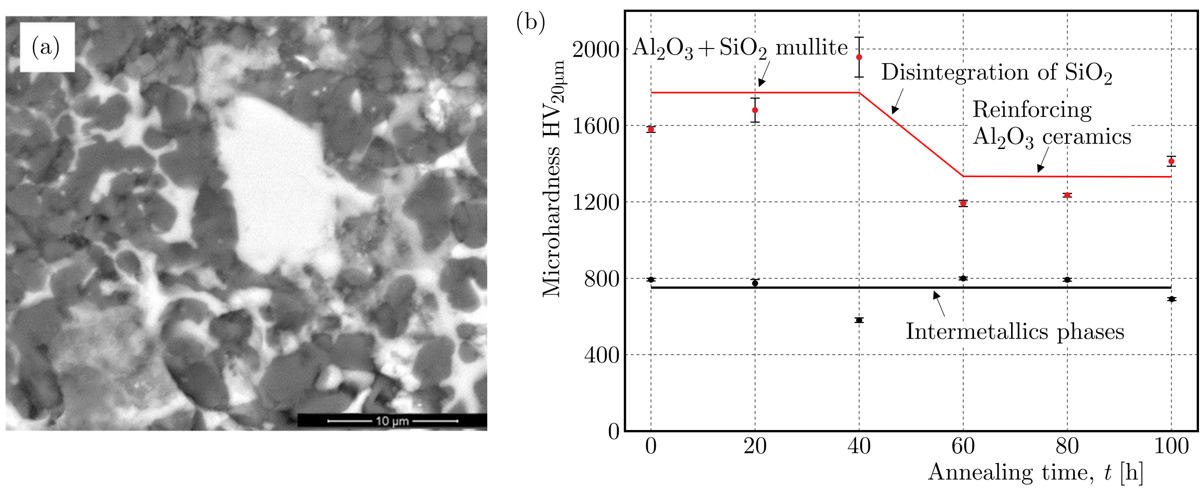

Fig. 3. Quasi-eutectic structure matrix in the areas of fragmented particles of the SiO_2 ceramic composite obtained after the process of homogenization at 600°C for 100 h (a); hardness as a function of annealing homogenization time at temperature of 600°C in the ceramic matrix and reinforced composite containing 50%wt. mullite (b)

It should be mentioned, however, that the sintering process did not enable full transformation of the microstructure to the $Al_{4.5}FeSi$ (τ_6) matrix reinforced with nanoparticles of Al_2O_3. In order to provide sufficient conditions for phase transformation, a homogenization process for 100 hours at 600°C was proposed. The correctness of the homogenization process assumed was verified through microstructural analysis and microhardness testing performed on the homogenized composite after specific periods of time (Fig. 3). It was found that under the annealing conditions, decomposition of SiO_2 and formation of reinforcing Al_2O_3 precipitates occurred in a shorter time period of 40-60 hours compared to the composite with a 70% ceramic content (Kopeć, 2015). The phase transformations were also confirmed by the microhardness results since specific regions of microstructural evolution were distinguished in Fig. 3b.

Anticipating high-temperature applications of the fabricated composites, the sintered samples were subjected to heat resistance test at 900°C in the air atmosphere. During the first 10 hours of the test, the weight of sintered samples was measured every hour. After 10 hours, the weight was measured every 5 or 15 hours until the total test time of 100 hours was reached. The composite exhibited excellent susceptibility to forming a durable and tight scale. The formed annealing scale protects the sinter material against high-temperature chemical corrosion at 900°C. A maximum weight increase of 3% was obtained after 50 hours of annealing (Fig. 4) and no major changes in the sample weight were observed. The fabricated composite was characterized by excellent durability and tightness of the protective scale as no splintering and increase in the weight was observed.

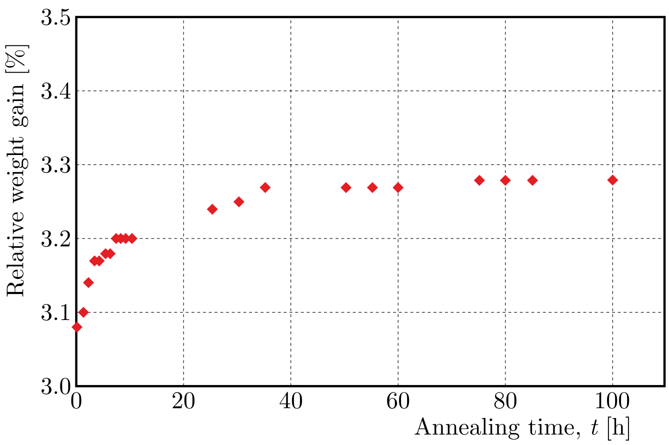

Fig. 4. Relative weight gain during homogenization at 900°C for 100 hours

4. Conclusions

An Fe-Al based composite reinforced with a 50%wt. content of the mullite ceramic was characterized by high hardness and superior heat resistance at 900°C in the air atmosphere as only 3% of the relative weight gain was observed after 100 hours annealing. Heat resistance remained extremely important for materials operating under complex conditions of ultra-high temperatures and the air atmosphere. It was found that the multi-phase microstructure of an $Al_{4.5}FeSi$ (τ_6) matrix reinforced with Al_2O_3 nanoparticles subjected to high temperature exhibited excellent durability and tightness of the protective scale and, therefore, was found to be very promising for potential high temperature applications.

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Manuscript received March 24, 2021; accepted for print May 13, 2021