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Synthesis of $FeAl/Al_2O_3$ Composites by Thermite Reaction†

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$FeAl/Al_2O_3$ composites were synthesized by using *in-situ* thermite reaction of iron oxide and aluminum powder mixtures. The phase, microstructure of the composites were investigated by X-ray diffractometry, scanning electron microscopy combined with differential scanning calorimetry. The results show that a chemical rection in Fe_2O_3/Al system is possible and the $FeAl/Al_2O_3$ composites with the interpenetrating network structure are obtained calcined at 900 ºC for 60 min.

Key Words: $FeAl/Al_2O_3$ composites, Microstructure, Thermite reaction.

INTRODUCTION

Alumina, as an excellent construction ceramics, possesses the attractive properties of good hardness, chemical stability, refractory character and low density. However, it is brittle at low temperature for the lack of fundamental dislocation mobility and insufficient slip systems[1]. From the standpoint of material design, it is undoubtedly a good method to develop ceramic-matrix composite with metal or alloy dispersion because of their high strength and multi-functional properties[2,3]. Among the metal or alloy, the intermetallic compound FeAl possesses a high melting temperature (1250 ºC), good oxidation resistance, relatively low density (5.56 g·cm^{-3}) and high thermal conductivity. These properties make FeAl alloy a promising candidate for high temperature structural applications[4,5]. In addition, FeAl alloy has also been identified that its thermodynamics is compatible with alumina[6].

Recently, the alumina-metal composites were synthesied by many techniques, such as reactive milling[7], reactive metal infiltration[8], directed metal oxidation[9], reactive sintering[10], thermomechanical powder consolidation processes11, thermite reaction synthesis[12-14]. Among these techniques, the thermite reaction method is an attractive technique, which utilizes the exothermicity of solid-state reaction to produce advanced materials. It shows many advantages of relatively simple equipment, low energy and possibility to form intermediate phase. In the present paper, $FeAl/Al_2O_3$ composites were synthesized by using *in situ* thermite reaction method. Their microstructure of $FeAl/Al_2O_3$ composites were investigated.

EXPERIMENTAL

Reagent grade aluminum powders (*ca.* 74 µm) and iron oxide powders were weighed according to 4-6 molar ratio. The powders were thoroughly mixed by a planetary mill for 15 h and then the mixtures were pressed to pellets with a diameter of 32 mm under a uniaxiasl pressure of 60 MPa. The green compacts were calcined at 700, 850, 900 ºC for 1 h, respectively.

The phase of samples were identified by the X-ray diffraction spectrometer using CuK_α radiation (XRD-6000, Shimadzu). Its microstructure was observed through the scanning electron microscope (SEM, JSM-5900). The main constituent elements were determined by energy dispersive X-ray spectroscopy equipped in SEM. Thermal analyses were carried out by differential scanning calorimetry (SDT 2960, TA Co.).

RESULTS AND DISCUSSION

Phase of $FeAl/Al_2O_3$ composites: Fig. 1 shows XRD patterns of the samples obtained at different temperatures. It is found that the major phase of sample calcined at 700 ºC are Fe_2O_3 and Al. Diffraction peaks referred to Al_2O_3, Fe and/or FeAl are observed as it was calcined at 850 ºC for 1 h, which means that the chemical reaction in Fe_2O_3/Al system was initiated. It can be seen that all diffraction peaks are identified to be Al_2O_3 and FeAl as it was calcined at 900 ºC for 1 h. No other impurity peak is detected in the samples.

Microstructure of $FeAl/Al_2O_3$ composites: Fig. 2 shows SEM images of samples calcined at different temperatures.

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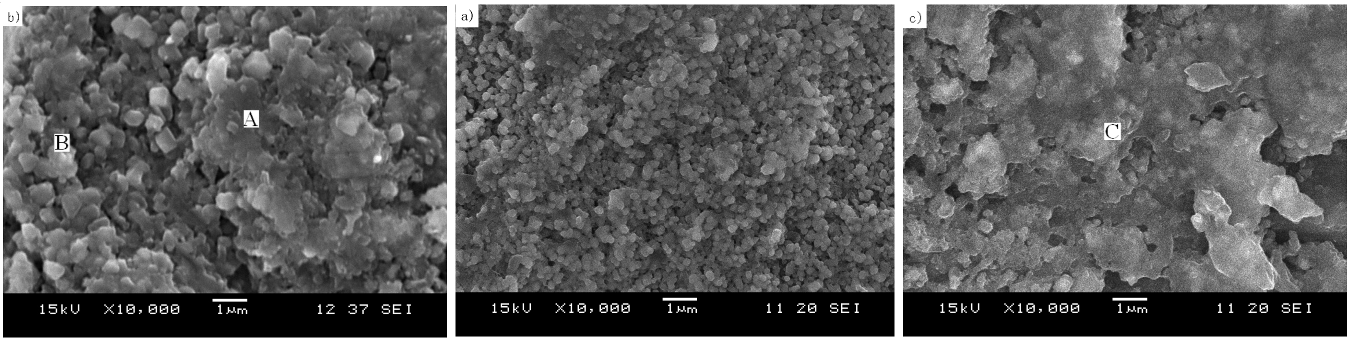

Fig. 2. SEM images of samples calcined at different temperatures. a) 700 ºC, b) 850 ºC, c) 900 ºC

Firstly, aluminium begins to melt and spread on the surface of Fe_2O_3 particles at 700 ºC [Fig. 2 (a)]. According to compositions of the samples (Table-1), it is verified that the gray region A is Al_2O_3 particles and white region B is FeAl particles in Fig. 2(b). The microstructure of Fe_2O_3/Al system becomes dense for the chemical reaction of Fe_2O_3/Al system was initiated at 850 ºC. Fig. 2(c) shows a typical image of a sample calcined at 900 ºC for 1 h and one can see that FeAl/Al_2O_3 composites with interpenetrating networks structure can be obtained.

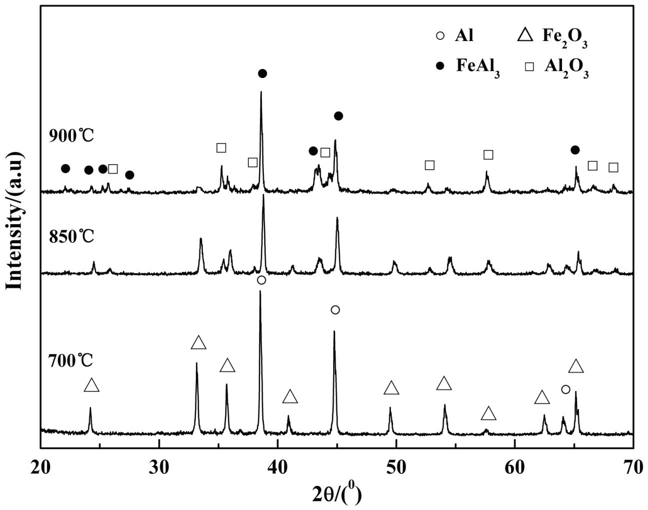

Fig. 1. XRD patterens of samples calcined at different temperatures for 1 h

TABLE-1
COMPOSITIONS OF THE REGIONS IN Fig. 2 (Wt %)

Region	Al	Fe	Other element	Total content
A	90.31	7.50	2.19	100
B	49.66	47.66	2.37	100
C	44.00	53.79	2.21	100

Differential scanning calorimetry analyses: Fig.3 shows the differential scanning calorimetry curve of the green compact at heating rate of 10 ºC/min. There are one endothermic peak (P_{Endo}) and one exothermic peak (P_{Exo}) in the curve. The observed endothermic peak locating at 660 °C can be reasonably ascribed to the melting of aluminium, the exothermic peak can be ascribed to the thermite reaction of Fe_2O_3/Al system.

The standard free energy of formation[15] can be given by:

$$\Delta G^0_f = -RT \ln k_T \qquad (1)$$

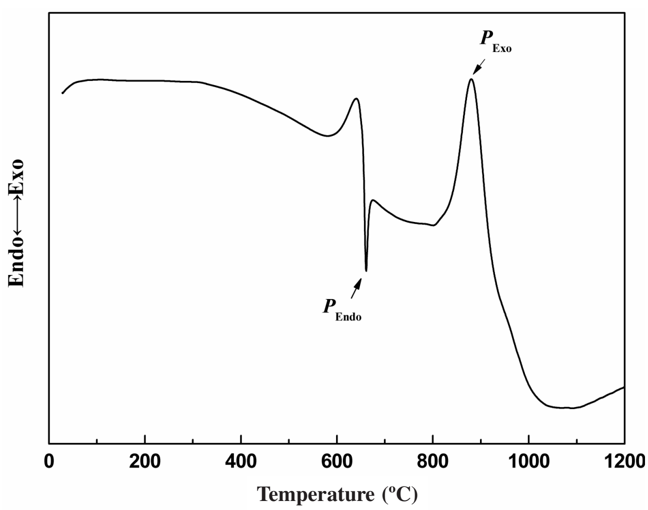

Fig. 3. Differential scanning calorimetry curves of Fe_2O_3/Al powder mixture at heating rates of 10 ºC/min

where T is temperature, k_T is equilibrium constant, for α-Al_2O_3, $\Delta G^0_f = -1120500 + 214.2\,T$, while for Fe_2O_3, $= -540600 + 170.3\,T$. The standard free energy of the formation (ΔG^0_f) of α-Al_2O_3 and Fe_2O_3 *versus* temperature is calculated and shown in Fig. 4.

The following equation is employed to predict strictly if the chemical reaction occurs:

$$\Delta G_f = \Delta G^0_f + RT \ln \frac{a_{Fe}}{a^2_{Al}} \qquad (2)$$

and a_{Fe} and a_{Al} are the activities of iron and aluminum in the system. Generally, it is reasonable to predict from ΔG^0_f if the

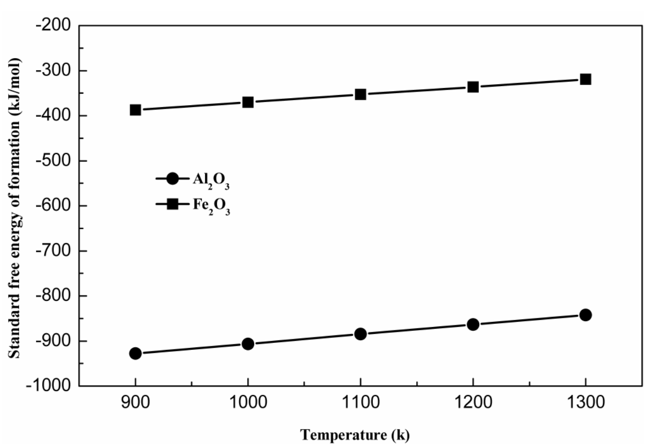

Fig. 4. Standard free energy of the formation of α-Al_2O_3 and Fe_2O_3 conclusions

reaction occurs, when ΔG^0_f is negative and its absolute value is large enough. Fig. 4 shows the possibility of the chemical reaction in Fe_2O_3/Al system.

Fe_2O_3/Al system is well known by the exothermic reaction, as it is submitted to thermal treatments. The thermite reaction equation is:

$$Fe_2O_3 + 2Al \rightarrow 2Fe + Al_2O_3 \quad (3)$$

and the enthalpy is:

$$\Delta H_T = \Delta H_O + \int \Delta C_p dT \quad (4)$$

where ΔH_0 is the enthalpy at standard state. The ΔH_0 is - 847.63 $kJ \cdot mol^{-1}$ Al_2O_3 for eqn. 3. ΔC_p is the difference of the heat capacity between the products and the reactants and for reaction eqn. 3. $\Delta C_p = a + b \cdot 10^{-3}T + c \cdot 10^5 T^{-2}$. Table-2 gives the values of ΔH_T for difference temperatures. It is also verified that there are strong exothermic effect in Fe_2O_3/Al system. Actually, the FeAl/Al_2O_3 composites are obtained, which is attributed to the thermite reaction of Fe_2O_3/Al system.

TABLE-2
EXOTHERMIC VALUES OF REACTION Eq. (3)
AT DIFFERENT TEMPERATURES

Temperature T (K)	933	1000	1100	1200	1300
ΔH ($kJ \cdot mol^{-1} Al_2O_3$)	-911.91	-900.56	-916.96	-935.32	-955.13

Conclusion

Aluminum and iron oxide powders were used to prepare FeAl/Al_2O_3 composites by *in situ* thermite reaction. The chemical reaction is feasible thermodynamically in Fe_2O_3/Al system. Thermite reaction in Fe_2O_3/Al system is ignited as the temperature is above 850 ºC. FeAl/Al_2O_3 composites with interpenetrating network structure are obtainned at temperature of 900 ºC for 1 h.

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