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Microwave-induced substitutional-combustion reaction of Fe_3O_4/Al ceramic matrix porous composite

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Abstract Microwave processing and substitutional-combustion reaction have been utilized to fabricate ceramic matrix porous composite from the thermite reaction of Fe3O4/Al system. Stoichiometric and mixtures with lower and over aluminum were tested. As this system was highly exothermic, the melting of reaction products and destruction the porous structure may occur. In order to avoid that, reaction coupled with a smaller driving force by controlling the microwave (MW) ignition condition at low temperature exotherm, where substitutional reaction occurs has been investigated. The phase and microstructure evolution during the reaction is analyzed by differential thermal analysis (DTA), X-ray diffraction (XRD), and scanning electron microscopy (SEM). Thermogram of the DTA analysis, irrespective of their mole ratio, recorded two exothermic peaks, one at ~1310 °C and another one at ~1370 °C. Fe and α-Al_2O_3 were the main products for the combusted mixture. Hercynite appeared as the major phase in the stoichiometric and slightly lower Al content mixtures due to incompleteness of reaction. In contrary, over aluminized mixture revealed the presence of $Al_{3.2}Fe$. When heated at 1360 °C, an additional FeO phase was observed. Mixtures with extremely low Al content showed the presence of unreacted Fe_3O_4 and some free Al due to the decrease of combustion velocity associated with a decrease in the sample exothermicities. Sample heated in electric furnace was dense. When heating by microwave, controlling the reaction progress at low temperature exotherm allowed the achievement of porous structure composite consisting of micron size iron particles well distributed and embedded in the hercynite and/or Al_2O_3 matrix.

Introduction

Combustion synthesis or self-propagating high temperature synthesis (SHS) is a fairly simple and economical technology which has been studied throughout the literature. The SHS reaction are suitable heat sources to several engineering application and has been applied in the processing of advanced materials, for instance the synthesis of ceramic reinforced metal-matrix composites [1, 2], shape memory alloys [3–5], and energetic nanocomposites [6–8]. The underlying basic of SHS relies on the ability of highly exothermic reaction to be self-sustaining, and therefore energetically efficient [9]. This type of reaction is often associated with large heat release which is sufficient to heat the product phase above their melting point [4, 7, 10]. Owing to large exothermic heat, the thermite reaction can generally be initiated locally. They can be ignited by a combustion wave from a chemical reaction [4, 7, 11], external heat source by an electric current or a laser beam [3, 5, 12, 13], plasma spraying [2] or by mechanical impact [14–20]. Once ignited, the combustion wave will propagate throughout the entire volume and convert the reactants to products. The products of the combustion synthesis reaction are normally extremely porous, e.g., typically 50% of the theoretical density [9, 21]. Such porous materials may have some application, e.g., filters and catalytic support structures, and perform for liquid metal infiltration in the production of ceramic–metal composites [21].

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The Fe_2O_3/Al mixture is a classical thermite system which has been studied intensely [2–8, 12]. Besides that, mechanochemically induced reaction of Fe_3O_4/Al system is also widely reported in the literatures [14–16, 19, 20]. The combustion products of this thermite comprise at least one heavy metallic phase (either metal or an alloy of the metal) and a light ceramic phase (alumina). However, by controlling adequately the processing variable, it is possible to obtain nanocomposites which are applicable as conducting magnetic materials [17] as well as high-temperature structure materials [18]. Durães et al. [7] published an extensive work on the radial combustion propagation in Fe_2O_3/Al thermite mixture to study the influence of reaction stoichiometry on the rate of propagation of combustion wave and reaction mechanism. As reported, the combustion front propagation rates increase with the increasing of Al content in the mixture, due to the increase of thermal conductivity in the reactants and products. Formation of Fe and α-Al_2O_3 as main phase and significant amount of $FeAl_2O_4$ and Fe_3Al phase was observed. Hercynite was detected with higher intensity in the product of mixture with lower ER (unitary equivalence ratio), conversely Fe_3Al intermetallic shows an opposite behavior. Dong et al. [2] combined self-propagating high temperature synthesis with plasma spraying to produced the composite coating based on Fe_2O_3/Al composite powder and revealed the presence of $FeAl_2O_4$ and Al_2O_3 as primarily phase and Fe, FeAl and Fe_3Al as secondary phases. Formation of FeAl and Fe_3Al was justified by the contact of Fe and Al melt in the Al excess region. Alongside with the SHS mode, some attention on the reaction milling of Fe_3O_4/Al system was reported by Botta et al. [14–16] and Takacs [19, 20]. Botta et al. [14–16] studied the effect of milling time and post treatment on the mechanochemically activated sample. They indentified the appearance of intermetallic phase, namely hercynite by adequately controlling the processing variable in the mechanochemical activation and in the final thermal treatment. Takacs [19, 20] on other hand, studied the stoichiometric effect on the mechanical alloying mixture, observed higher $FeAl_2O_4$ intensity in lower Al content mixtures and the presence of α-$FeAl_{0.33}$ and α-$FeAl_{0.89}$ in the over Al mixtures. Their finding was well collaborated with Durães et al. [7, 12].

In a different paper [22], microwave energy was utilized for the ignition and combustion synthesis of composites. It was demonstrated that, when using microwave for ignition, under suitable processing condition the wave front can be controlled in contrast with the self-propagation in conventional combustion synthesis. When using microwave for ignition, the ignition temperature is reached in the center of the sample first and the combustion front propagates through the entire volume radially. The dependency of reaction on the thermal conductivity and density of the compact is greatly reduced in comparison with the conventional method [22]. Thus, only limited resources on utilizing microwave power for synthesis and control of the highly exothermic Fe_3O_4/Al system was available. As reported recently Lee et al. (Unpublished results), dense product was obtained when heating the mixture below 1200 °C, irrespective of heating method and mixture ratio. However, heating at 1265 °C in microwave furnace resulted in the formation of porous structure materials. In contrary, further increasing the heating temperature above 1380 °C lead to onset of combustion reaction, coincided with melting of reaction product. Thus, the authors argue the possibility of controlling the combustion wave front propagation just before the self-sustained combustion reaction occurred. In this study, the aim of this contribution is to analyze the reaction between Fe_3O_4 and Al for the production of porous microstructure composite material, which can serve as a catalyzed soot filters (CSF) by microwave induced substitutional-combustion synthesis. To investigate the controllability of microwave ignition and combustion behavior as postulated in Lee et al. (Unpublished results), heating of the mixture within the low temperature exotherm region, which corresponded to the low exothermic peak in thermal analysis, will be highlighted.

Experimental methods

Magnetite (~500 nm) and aluminum (200–300 μm) powders were used to compound the thermite mixtures. Table 1 presents the molar compositions of the prepared mixtures. SA’ and SB corresponds to stoichiometric

Table 1 Thermite mixtures compositions and heating conditions

Mixtures	Fe_3O_4:Al composition (mol)	Heating temperature (°C)	Holding time (min)	Note
SA’4	1:2.67	1265	10	Elec. heating
SB4	1:2.67	1265	10	MW heating
SB6	1:2.67	1360	–	MW heating
SB6ig	1:2.67	1380	–	MW heating (combusted)
SC1	1:1	1265	10	MW heating
SC6	1:1	1330	–	MW heating
SD1	1:2	1265	10	MW heating
SD5	1:2	1360	–	MW heating
SE1	1:3.33	1265	10	MW heating
SE5	1:3.33	1360	–	MW heating

reaction as expressed in Eq. 1, SC and SD corresponds to lower Al content and SE in excess of Al, respectively.

$$3Fe_3O_4 + 8Al \rightarrow 4Al_2O_3 + 9Fe \quad \left(\Delta H^{o}_{298} = -3359\ \text{kJ/mol}\right) \tag{1}$$

The thermite mixtures were weighted and uniaxially pressed into cylindrical green compact (Ø6.76 × 4.2 mm^2 thick). Since it is easy to heat both magnetite and aluminum in the H-field position, the green compact was put into a glass tube and placed at the H-field maximum position in a 2.45 GHz, 2 kW single mode microwave applicator as shown in Fig. 1. Temperature measurement was obtained using an optical pyrometer (minimum temperature measured = 400 °C). Samples were heated rapidly to a temperature corresponding to the exothermic peak obtained in DTA analysis. Two heating temperature namely, 1265 °C (with a soaking time of 10 min) and 1360 °C (which quenched immediately once the temperature was reached) was carried out at the low temperature exotherm to determine the controllability of microwave ignition and combustion. There were some temperature variations (range within 5–10 °C) due to the small composition inhomogeneities in the samples and/or non-controllable electromagnetic field distributions that contribute in perturbing the heat generation [23]. Difference thermal analysis (DTA) was performed in an EXSTAR SII analyzer under the flowing of N_2, with Pt holders, using a heating rate of 20 °C/min and samples of approximately 15 mg. The phase composition was analyzed by X-ray diffraction (XRD), using a RINT diffractometer with CuKα radiation at 40 kV and 100 mA ($\lambda = 0.154$ nm). In order to study the microstructure of the samples, a Philips scanning electron microscope (SEM) was used, and energy dispersion X-ray (EDX) was performed to quantify the composition of the selected area on the samples. For comparison, the stoichiometric mixture was heated under the same temperature setting by electric furnace. Off stoichiometric effect were studied using lower and over aluminized mixtures.

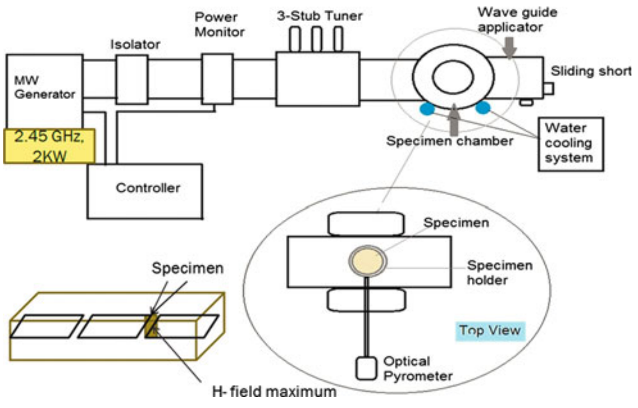


Fig. 1 Scheme of 2.45 GHz 2 kW single mode microwave applicator

Results and discussion

Thermal analysis

Figure 2 shows the DTA curve of three different stoichiometric mixture samples. Irrespective to their mole ratio, an endotherm was observed at 660 °C, attributed to the melting of Al with slightly higher intensity for the S3 due to the excess of Al volume in the sample. Two exotherm peaks were observed, one at ~1310 °C and another one at ~1370 °C. The smaller peak, i.e., the low temperature exotherm, signified the initiation and propagation of combustion by supply of microwave power, on another hand revealed suppression of combustion wave self-propagating by reducing the power. At this point, material with porous structure (Fig. 3a) can be obtained by MW heating. The second peak, i.e., the high temperature exotherm, was much bigger and showed the characteristic of sharp rise. It corresponded to the main combustion reaction, coincided with the melting of reaction products, and destroying of porous structure (Fig. 3b). At the low temperature exotherm, heat generated in the system was insufficient for the combustion wave front to be self-propagate, thus the spontaneous reaction progress is not possible. Constant dissipation of microwave energy to the sample is needed to force the combustion wave front to propagate. Owing to this, we take the advantage of microwave power to provide heat for the insufficient heat generation case, so as to control the combustion progress (propagation of combustion wave front) by controlling the incident power. Propagation of the combustion wave can be terminated by turning off the microwave supply and/or by pulsing the incident power for more precise control on the velocity of propagation of the combustion wave front. Our observation was in contrast with Botta et al. [15, 16], in which an

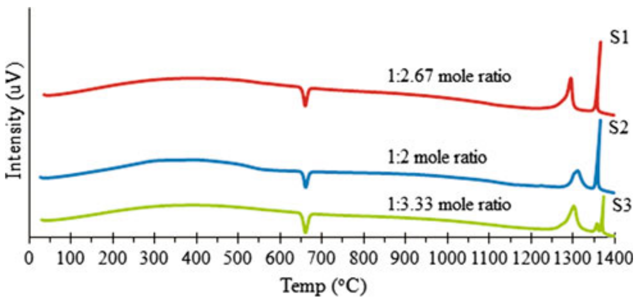


Fig. 2 Thermograms of Fe_3O_4/Al mixtures S1–S3 using a heating rate of 20 °C/min

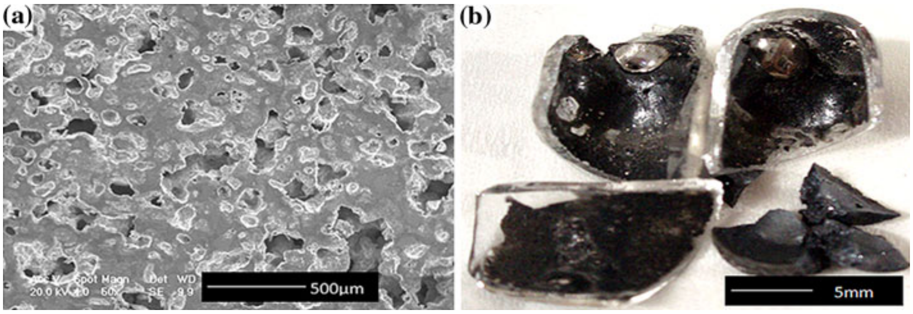


Fig. 3 Image of sample heated at different temperature: **a** low temperature exotherm region, obtaining porous structure material, **b** high temperature exotherm region, melting, and destroying of porous structure

exotherm at approximately 600 °C due to oxidation of part of the Al was reported. Owing to the high heating rate, lesser residual time was available at any temperature, thus the melting plateau as well as the T_{ig} will shift toward higher temperature [11]. Beside that, temperature measurement method may also contribute to this variation. With microwave energy, the heat is generated internally and propagates outward in a radial manner. Thus, temperature at the surface is lower compared to the center area. This explained the temperature variation in the experiment as compared with the results obtained from DTA analysis.

Temperature profile

Figure 4a, b depicts the time–temperature profiles of stoichiometric mixture compacts heated at three different conditions, namely 1265, 1360, and 1380 °C, respectively. These heating conditions were set according to the results obtained from the DTA analysis to evaluate the controllability of microwave ignition and substitutional-combustion of Fe_3O_4/Al system. When heating the mixture compact at 1265 °C (at the shoulder of lower temperature exotherm), it demonstrates the ability to maintain/hold the heating temperature without pulsing the incident power. Reaction occurred without any perceptible thermal explosion at all. When the heating temperature was increased to 1360 °C, the profile showed a plateau signifying the initiation of ignition and a small rise of temperature. At this state, it is impossible to hold the heating temperature. Propagation of the ignited combustion wave front can be terminated by turning off the microwave power due to the low velocity of combustion wave. Porous structure materials as shown in Fig. 3a can be obtained. In contrast, commencement of main combustion occurs when heating the mixture compact up to 1380 °C (the high temperature exotherm). Initiation of ignition and a sharp rise to the peak of combustion (T_c) which surpassed the melting point of reaction products was observed. Control of the combustion wave propagation was impossible, even though the microwave power has been cut off. The combustion wave front propagated rapidly in a self-sustained manner and the combustion reaction was completed in less than a second. Mixture compact underwent melting of products due to high combustion temperature (Fig. 3b). Based on this initial result, experiment on the effect of different stoichiometric ratio to the end product was carried out.

XRD analysis

X-ray diffractograms in Fig. 5a, b show the change of products formed by the reaction in different thermite mixtures heated to 1265 and 1360 °C. X-ray diffractograms for mixtures heated at 1265 °C revealed the presence of Fe and $FeAl_2O_4$ (hercynite) as main phase in SB4 and SD1. Al_2O_3 and Fe–Al intermetallic are also significant phases. The unambiguous identification of other possible phases is hindered by the overlap and broadening of the diffraction lines. However, the shoulder at around 44.2°

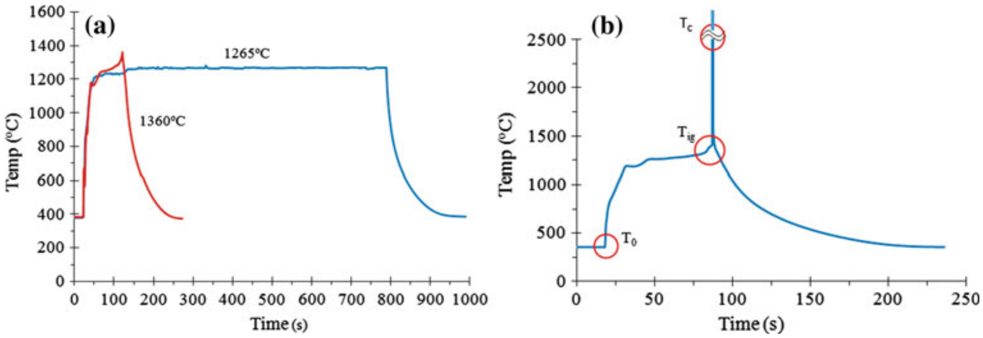


Fig. 4 Temperature–time plot for sample heated to: **a** 1265 °C hold for 10 min and 1360 °C without holding and **b** 1380 °C combusted

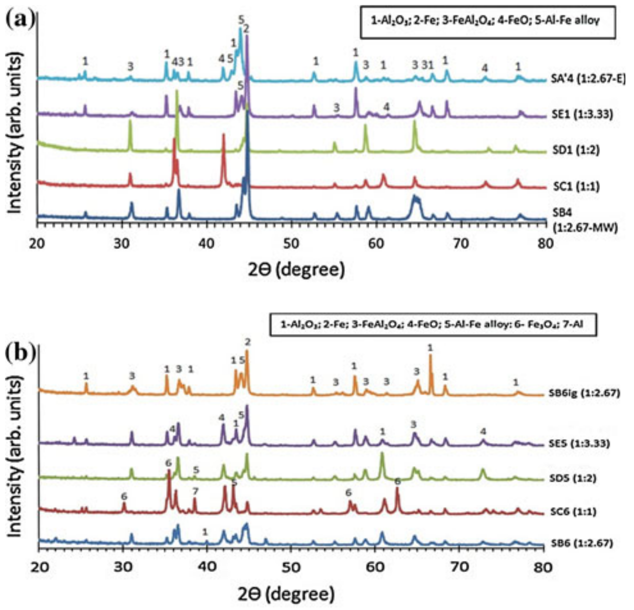


Fig. 5 X-ray diffractrograms of products formed in different thermite mixtures heated at: **a** 1265 °C and **b** 1360 °C (*SB6ig was the combusted sample)

may originate from $Al_{3.2}Fe$. Owing to the similarity in Fe_3Al (45-1203), Al_3Fe (38-1147), FeAl (33-20), and Fe_3Al_2 (45-982) JSPDC-ICDD database patterns, the assignment of these intermetallic phase was in doubt. Consequently, any phases related to Fe_3Al, FeAl, and Fe_3Al_2 was named as Fe–Al intermetallic/alloy thereafter. Hercynite was widely recognized as the intermediate product in the thermite reaction. Formation of this intermediate phase was mainly dependent on the ratio of Fe_3O_4/Al and FeO/Al or through an appropriate control of the mechanochemical and thermal condition [14–17]. According to Durães et al. [7], the presence of $FeAl_2O_4$ and Fe_3Al clearly depends on the Fe_2O_3:Al ratio. $FeAl_2O_4$ was detected in higher intensity when Al content in the mixture is below the stoichiometric amount. In contrast, Fe_3Al intermetallics phase being detected with higher intensity in the product of over-aluminized sample. The mentioned phenomena were also observed by Takacs [19] in the reduction of magnetite by aluminum induced by mechanical alloying. Results obtained in this study were in good agreement with Durães et al. [7] and Takacs [19] in the case of hercynite. However, the same behavior was not observed in the case of Fe_3Al. Contrary, increasing of Al_2O_3 intensities is observed in SE1 products instead of Fe–Al intermetallic. This may be due to different experimental method and/or starting materials used in this study. As indicated in the X-ray diffractograms (Fig. 5a), the intensity of hercynite intermediate phase increase almost double in the product of SD as compared to SB4. Further decrease of aluminum content in the mixtures (SC1) resulted in a shortage of aluminum and the formation of FeO as main phase (Eq. 2). Simultaneously, Al_2O_3 becomes almost absent due to the formation of hercynite by the reaction of Eq. 3. This led to decrease of the reduced metallic iron. Conversely, increase of the aluminum content from SD1 to SB4 mixture led to an increase in the reduced metallic iron. Furthermore, there is an increase in the amount of Al_2O_3 align with the Al content in the mixtures. Instead of Fe, formation of Fe–Al intermetallic as main phase was observed in product of sample heated in electric furnace. Further studies will be carried out to clarify this.

$$Fe_3O_4 + (2/3)Al \rightarrow 3FeO + (1/3)Al_2O_3 \qquad (2)$$

$$FeO + Al_2O_3 \rightarrow FeAl_2O_4 \qquad (3)$$

Products formed in the sample heated up to 1360 °C (at the low temperature exotherm), is found to be qualitatively similar to those heated at 1265 °C with a behavior nearly the same as before, except for the SC6 mixture (Fig. 5b). However, it was observed that the hercynite intensities' increase with oxygen is less steep. Formation of an additional phase, namely FeO-appeared as one of the major phases in these products. Unreacted reactant, ascribed to Fe_3O_4 with the highest intensities among all the products and Al in low concentration were detected in the product of SC6 mixture. As mentioned above, further decrease in aluminum content to a Fe_3O_4:Al ratio of 1:1, resulted in a shortage of aluminum which lowers the extent of reaction. Reduction of Fe_3O_4 was then suppressed due to the decrease of combustion velocity, associated with a decrease in the sample exothermicities, originated by the decrease in the thermite reaction completeness. Heating of the SC6 mixture above 1340 °C lead to the commencement of main combustion and the reactive mixture undergoes a violent exothermic self-sustained reaction. Very little Fe and Al_2O_3 phase was detected at the same time that hercynite was almost absence. An Al-rich Al–Fe intermetallic phase, $Al_{3.2}Fe$ appeared in the sample with intensity comparable to those FeO main peaks. Presumably, melted Al liquid spread throughout the sample because of the capillary forces and wetted the reduced Fe that led to formation of $Al_{3.2}Fe$ phase. Diminution of the products intensities, reaching about 40–50% was observed as compared to the products of mixtures heated at 1265 °C. This can be associated with the different heating condition as revealed by the temperature–time profile in Fig. 4a. Despite the temperature difference, time allowed for reaction in the microwave applicator played an important role. As the samples were heated rapidly to the set temperature and quenched immediately just after the desired temperature was reached, lowering of crystallinity may occur due to high cooling rate and abrupt change in temperature.

Increase of heating temperature approaching to high temperature exotherm region, result in self-sustained combustion reaction (Fig. 4b), coincided with the melting of reaction product and destruction of porous structure (Fig. 3b). X-ray diffraction pattern of the combusted product sample (SB6ig mixture) revealed the presence of α-Al_2O_3 and Fe as main phases and low concentration of non-stoichiometry hercynite originated by some substitution of Al with the Fe atoms, in the type Fe $[Al_xFe_{2-x}]O_4$, with $0 \leq x \leq 2$ [15] as secondary phase.

Microstructure evaluation

Figure 6a, b display the microstructure changes of different thermite mixtures heated at 1265 and 1360 °C, respectively. Despite the molar ratio difference, mostly all samples revealed the presence of intermediate phases except for the combusted sample (Fig. 6b(e)). Occurrence of these intermediate phases could not be avoided except at higher temperature of combustion and at the expense of product shape and porosity [24]. In addition, lack of homogeneity in mixing and composition fluctuation due to coarse particle size will lead to the multiphase product also [11, 25]. When heated in microwave, stoichiometric sample and sample with slightly lower in Al content heated at 1265 °C showed micron sized iron particles, elongated and irregular in size and shape, uniformly dispersed and distributed within hercynite matrix. On the other hand, the formation of these well distributed iron particles have not been observed in others sample. Product of over aluminized mixtures showed the formation of Fe, Al_2O_3, and Fe–Al intermetallic. Fe–Al intermetallic phases was formed when Fe is in contact with melted aluminum [7, 26]. Sample with lowest Al content denote the formation of FeO as the matrix phase and Fe as secondary phase surrounded by hercynite. Owing to the formation of the FeO phase, concentration of Fe phase was greatly reduced. This observation collaborated well with the results obtained from XRD analysis. Figure 6a(f) depicts the representative microstructures of porous composite for Fe_3O_4/Al system that was obtained by microwave heating at 1265 °C for 10 min. It had an interconnected and uniform porous structure, indicating the pre-ignition of combustion wave as compared to electric furnace, where the sample was dense and with closed pore structure. This may be due to the kinetic enhancement by microwave heating.

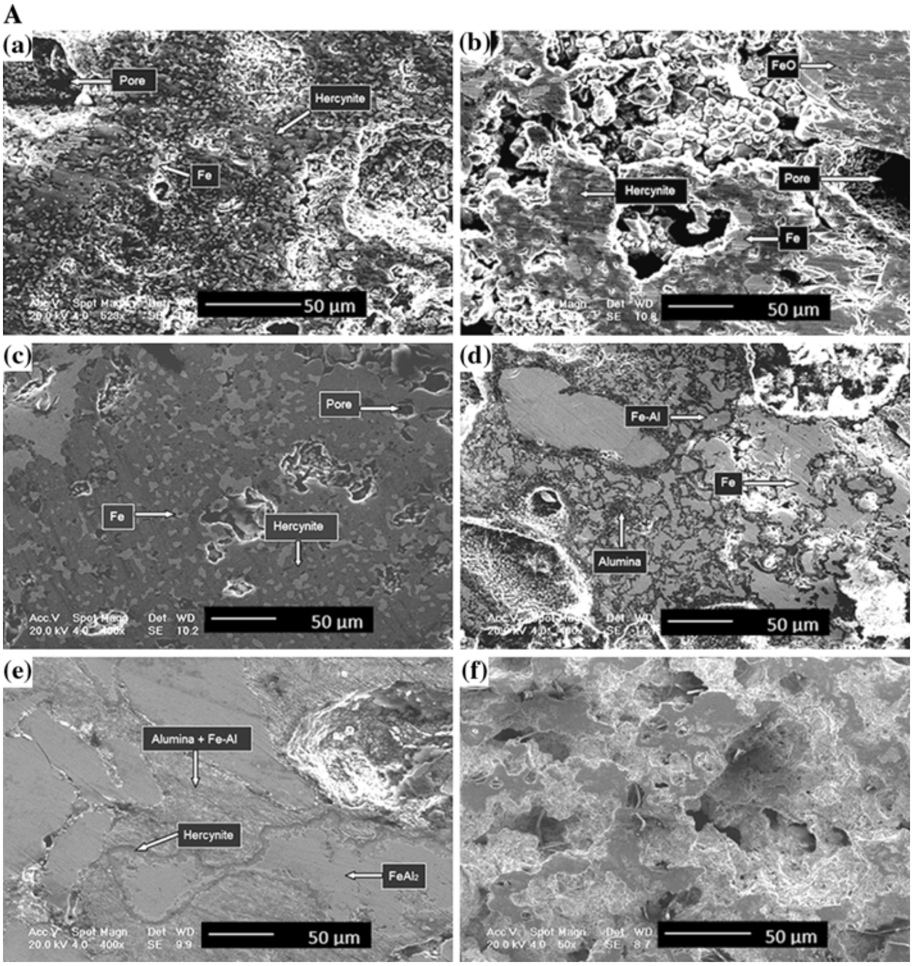
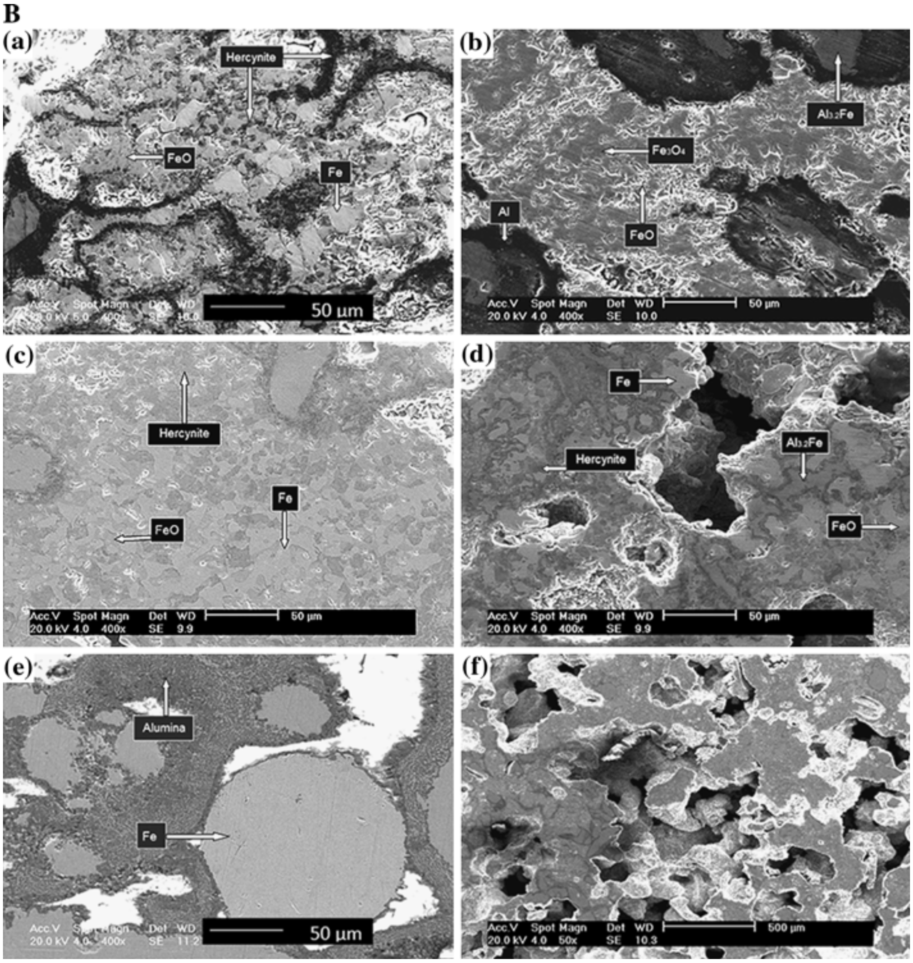


Fig. 6 **A** Microstructure of samples with different Al: Fe_3O_4 mol ratio heated at 1265 °C: *a* 1:2.67. *b* 1:1. *c* 1:2. *d* 1:3.33. *e* 1:2.67 (Elec.). *f* porous structure; and **B** 1360 °C: *a* 1:2.67. *b* 1:1. *c* 1:2. *d* 1:3.33. *e* 1:2.67 (combusted). *f* porous structure

Fig. 6 continued



Samples heated to 1360 °C was quenched immediately just after the heating temperature was reached, except for SB6ig mixture which undergone full combustion. Microstructure of the specimens is shown in Fig. 6b. These specimens demonstrated the same behavior as specimens heated at 1265 °C, but with the occurrence of an additional phase, namely FeO, manifested in the incomplete reduction of Fe_3O_4 due to the time constrains which limit the extent of reaction. Dramatic changes in the microstructure were observed in SC6 mixture (Fig. 6b(b)) as compared to SC1 (Fig. 6a(b)). Unreacted Fe_3O_4 and some free Al was observed in the sample, indicating the incompleteness and inexothermicities of the reaction when Al content was extremely low in the thermite mixture. As mentioned in the phase analysis, shortage of aluminum will lower the extent of reaction, thus suppressing the reduction of Fe_3O_4 due to the decrease of combustion velocity with lower Al content and lower combustion temperature. Significant amount of $Al_{3.2}Fe$ phase was also present in the sample. Result of the atomic composition from the EDX quantitative analysis was Al-77.44 at.% Fe confirmed the formation of these Al-rich Fe–Al intermetallic, namely $Al_{3.2}Fe$ as revealed in the X-ray diffractograms.

When heating the sample approaching the high temperature exotherm (second exothermic peak in the DTA plot which show a characteristic of sharp rise), main combustion commenced, resulted in the formation of Fe and α-Al_2O_3 (Fig. 6b(e)) as expressed by Eq. 1. Highly porous multiphase structure sample was obtained by controlling the reaction progress at the low temperature exotherm to avoid the commencement of main combustion. The mentioned criteria of multiphase porous microstructure formation was also observed by Biswas et al. [11] in the fabrication of porous NiTi by thermal explosion mode of self-propagating high temperature synthesis (SHS). As highlighted, the first phase of solid-state diffusion was essential to achieve the near-net shape porous product, but inevitably contained some undesirable phases that necessitated for post-reaction heat treatment. As compared visually, mixtures heated at 1360 °C shows a better degree of porosity.

Conclusions

Synthesis of the Fe_3O_4/Al system have been carried out with a stoichiometry and non-stoichiometry composition in microwave and electric furnace by controlling the reaction progress to avoid the commencement of main combustion. Products formed in different mixture compositions by substitutional-combustion reaction were characterized by X-ray diffraction, energy-dispersive X-ray spectroscopy (EDX), and scanning electron microscopy (SEM) to investigate the influence of the mixtures' ratio on the reaction mechanism. The main products for the combusted mixture were identified as Fe and α-Al_2O_3. Hercynite was found as an intermediate product in almost all mixture products. It appeared as the major phase in the stoichiometric and slightly lower Al content mixtures with intensity comparable to Fe. Reaction product with excess Al content revealed the presence of $Al_{3.2}Fe$. An additional phase, namely FeO was observed in the products when heated at 1360 °C. Extremely low Al content mixtures revealed the presence of unreacted Fe_3O_4 and some free Al due to the decrease of combustion velocity and sample exothermicities. Significant amount of $Al_{3.2}Fe$ was also detected. Meanwhile, mixtures heated in electric furnace revealed the appearance of Fe–Al intermetallic as major product. None porous products are formed by conventional heating at this temperature. It is speculated that low temperature exothermic reaction occurred within the inner part of the specimens. Control of the reaction progress at the low temperature exotherm by manipulating the microwave power allowed the achievement of porous structure composite consisting of micron-sized iron particle, isolated and well-distributed and embedded in the hercynite and/or Al_2O_3 matrix.

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