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Short communication

Characterization of microwave processed aluminium powder

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Abstract

Commercial Al powder was exposed to microwave radiation for 45 min. The as received and microwave heated Al powders were characterized by X-ray diffraction analysis (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), energy dispersive X-ray (EDX) analysis and transmission electron microscopy (TEM). XRD of the microwave treated Al powder confirmed the formation of Al–Al_2O_3 composite. FTIR studies and EDX analysis indicated the transformation of Al powder into Al–Al_2O_3 core–shell composite powder after microwave processing. SEM showed that the morphology of the microwave processed Al powder was quite different from that of the as-received Al powder. TEM image of the microwave treated Al powder supported the FTIR and EDX data.
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Keywords: A. Microwave processing; B. Composites; Electron microscopy; Spectroscopy

1. Introduction

Microwave heating has been used as an innovative sintering method for ceramics, polymers and composites [1]. Novel processing of materials such as development of functionally gradient materials, joining, synthesis of ceramic powder, glazing of coating, crystallization of bulk glass and glassy coating has been conducted using microwave heating [2–7]. Microwave energy is being utilized for the sintering of metal powders [8]. In addition, ceramic and metal nanopowders have been sintered by microwave heating [9].

Researchers have shown that finely divided metal powder can be efficiently heated by microwave energy [10–11]. The dielectric loss and eddy current loss have important roles in the heating of metals. Multiple scattering in the metal powder also leads to the absorption of the microwave energy [10]. The exact mechanism of microwave energy absorption by metallic particles is yet to be completely understood [8,11–12]. However, it has been unequivocally established that high conductivity samples can be much more effectively heated up in the magnetic field component of the microwave field [11]. The energy loss mechanisms are yet to be elucidated. However, it has been suggested that magnetic loss mechanism can be affected by contribution due to hysteresis, eddy currents, magnetic resonance and domain wall oscillation in the case of conductor materials [12]. The oxide coating on aluminium and Al–Al_2O_3 composite has been formed by microwave heating and reported elsewhere [13–15]. The objective of the present work was to form Al–Al_2O_3 core–shell composite by microwave processing.

2. Experimental

Commercial Al powder (S.D. Fine-Chem. Limited, Mumbai, India, average particle size – 25–30 μm, purity – 99.7%) was used in the present study. The Al powder was heated for 45 min in air inside a domestic microwave oven (800 W, 2.45 GHz, BPL, India) with a specially designed in-built applicator [14]. Pt–Pt 13% Rh thermocouple was employed to measure the sample temperature. The measurement accuracy was ±10 °C. The phase composition of the as received and microwave heated Al powders was examined by X-ray diffractometry (PW 1710, Philips Research Laboratory, Eindhoven, The Netherlands) using Cu Kα radiation (45 kV, 35 mA). The chemical structure of the microwave heated Al powder was determined by Fourier transform infrared spectroscopy (Varian 3600 FTIR, USA). FTIR study of pure α-Al_2O_3 powder (S.D. Fine-Chem. Limited, Mumbai, India, average particle size – 22–28 μm, purity – 99.7%) was also conducted

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for comparison. KBr pellet technique was used to prepare the samples for the FTIR studies. The morphology of the as received and microwave heated Al powders was investigated by scanning electron microscopy (s430i, Leo, UK) and transmission electron microscopy (Tecnai 30 G^2 S-T, FEI, The Netherlands) using an accelerating voltage of 300 kV. The elemental composition analysis of the microwave heated Al powder particle was conducted by energy dispersive X-ray analysis (s430i, Leo, UK, SiLi detector).

3. Results and discussion

3.1. XRD analysis

Fig. 1(a and b) shows the XRD data of the as received Al powder and the Al powder heated in microwave, respectively. The temperature measured was ~1000 ± 10 °C after 45 min microwave heating of the Al powder. Al phase was only identified in the as received Al powder (Fig. 1(a)) while α-Al_2O_3 and Al phases were detected in the microwave treated Al powder (Fig. 1(b)). XRD data confirmed the formation of the Al–α-Al_2O_3 composite after the exposure of Al powder to microwave radiation.

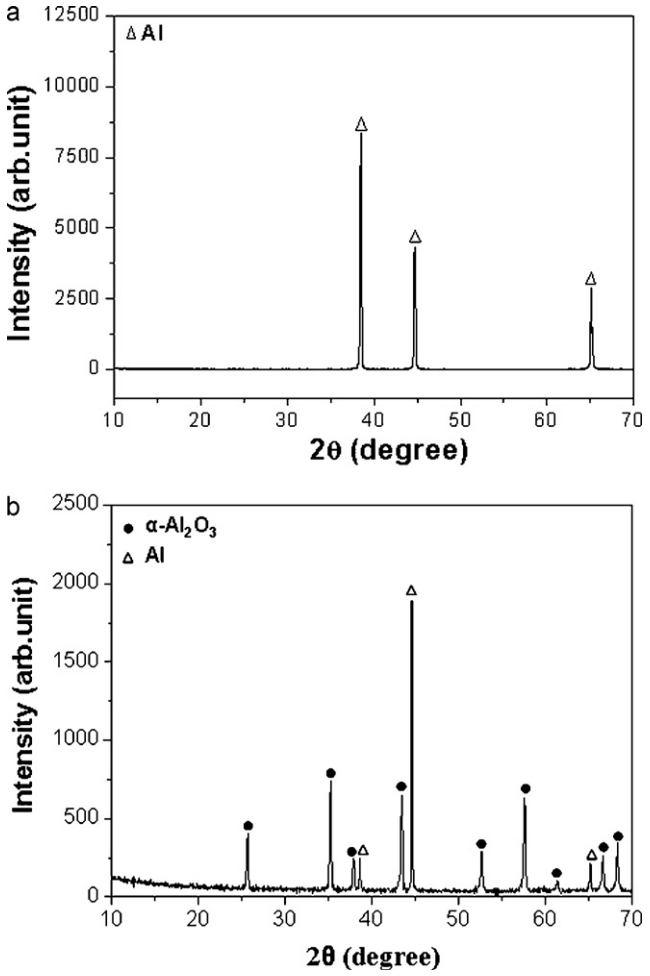

Fig. 1. XRD patterns of: (a) as-received Al powder and (b) Al powder microwave heated for 45 min.

3.2. FTIR study

FTIR spectra of the as received α-Al_2O_3 powder particles and the microwave processed Al powder particles showed the chemical structures of the powders (Fig. 2(a and b)). The spectrum of pure α-Al_2O_3 powder demonstrates the characteristic absorption peaks at 459, 615, 646, 1654, 2339, 2362 and 3448 cm^{-1} (Fig. 2(a)). This spectrum agrees well with those obtained by others [16,17]. The characteristic of aluminium oxide is represented by the absorption peaks in the range of 450–1000 cm^{-1} [16]. Al–O stretching vibrations led to sharp absorption band at 459 cm^{-1} and the broad absorption band in the range of 600–800 cm^{-1}. The absorption band appeared at 1654 cm^{-1} because of the presence of moisture in the powder. The weak bands at 2339 cm^{-1} and 2362 cm^{-1} might be formed due to –OH stretching vibrations. The broad band at 3448 cm^{-1} might be attributed to hydrogen-bonded surface –OH groups [17].

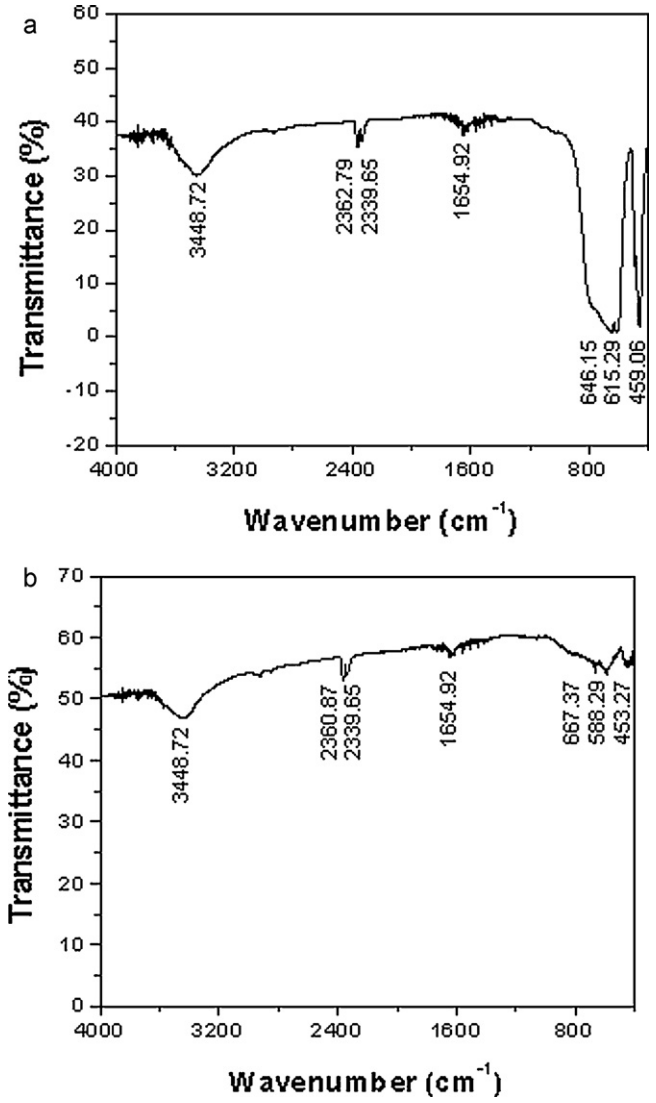

Fig. 2. FTIR analysis of: (a) as received α-Al_2O_3 powder particles and (b) microwave processed Al powder particles.

On the other hand, in the spectrum of the microwave processed Al powder, the absorption peaks appeared at 453, 588, 667, 1654, 2339, 2360 and 3448 cm^{-1} (Fig. 2(b)). Majority of the absorption peaks in the spectrum were in the nearly similar position as shown in the case of pure α-Al_2O_3 powder. However, few absorption peaks of the Al–α-Al_2O_3 composite powder were very slightly shifted in terms of wavelength as compared to corresponding peaks of the pure α-Al_2O_3 powder. Thus, it can be said that the behavior of the FTIR spectrum of the Al–α-Al_2O_3 composite powder was almost similar to that of the pure α-Al_2O_3 powder. Therefore, XRD and FTIR results suggested the formation of Al–α-Al_2O_3 core–shell

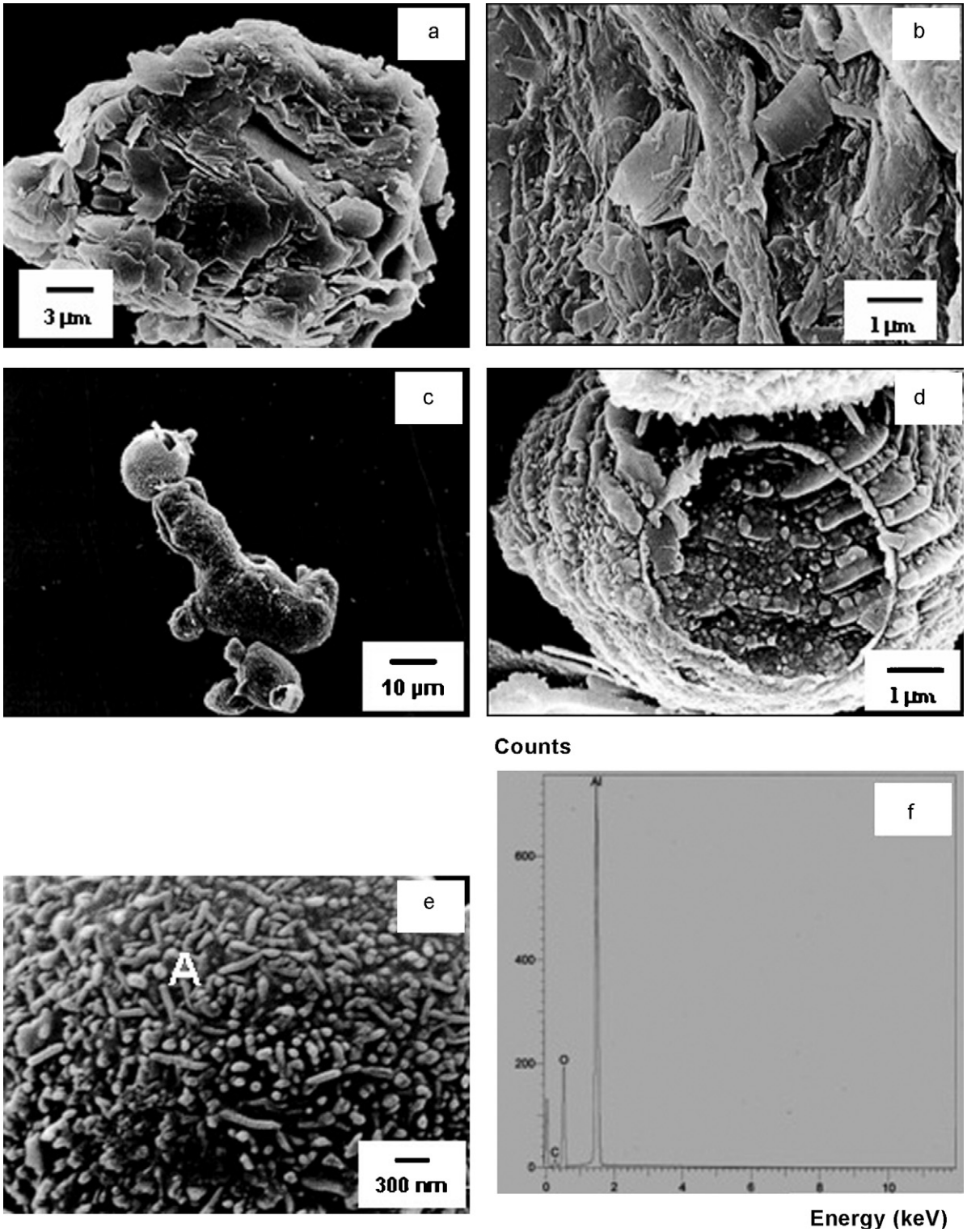

Fig. 3. (a) As-received Al powder agglomerate; (b) microstructure of as-received Al powder agglomerate; (c and d) microwave heated Al powder agglomerate; (e) microstructure of microwave heated Al powder agglomerate shown in (d) and (f) EDX analysis of one microwave heated Al powder particle marked as A in (e).

composite structure after exposure of Al powder to microwave radiation. The low intensities of the absorption peaks in the range of 450–1000 cm^{-1} (Fig. 2(b)) indicated that the thickness of the α-Al_2O_3 films grown on the Al particles was not very high [16]. The formation of core–shell composite has been indicated by others using FTIR analysis [18,19].

3.3. SEM and EDX analyses

Fig. 3(a) shows the morphology of the as received Al powder agglomerate while the surface microstructure of same powder agglomerate has been demonstrated in Fig. 3(b). The morphology and surface microstructure of the microwave heated Al powder agglomerate have been shown in Fig. 3(c and d). Fig. 3(e) illustrates the surface microstructure of the microwave heated Al powder agglomerate at higher magnification. It can be noticed that the morphology of the microwave heated Al powder agglomerate was totally different from that of the as-received Al powder agglomerate. The Al powder looked like flaky shaped agglomeration (Fig. 3(a)). After microwave treatment, it changed to an agglomerate of particulate matters (Fig. 3(c and d)). This was more distinct from the surface microstructures of the as received Al powder (Fig. 3(b)) and the microwave treated Al powder (Fig. 3(e)).

The particle morphology of the as received and microwave heated Al powders can be observed from Fig. 3(b and d). The particle size of the microwave heated Al powder was larger (Fig. 3(d)) than the size of the as received Al powder particles (Fig. 3(b)). This was due to the melting of Al powder particles and coalescence during microwave heating and then re-solidification after cooling to room temperature. Furthermore, the particle growth might be ascribed to oxidation of Al powder by microwave heating and subsequently formation of an oxide layer on Al powder particles.

EDX data of the microwave-treated Al powder particle (marked as A in Fig. 3(e)) is shown in Fig. 3(f). EDX analysis identified the presence of Al and O, suggesting the formation of Al_2O_3 and thus, corroborated the XRD results (Fig. 1(b)).

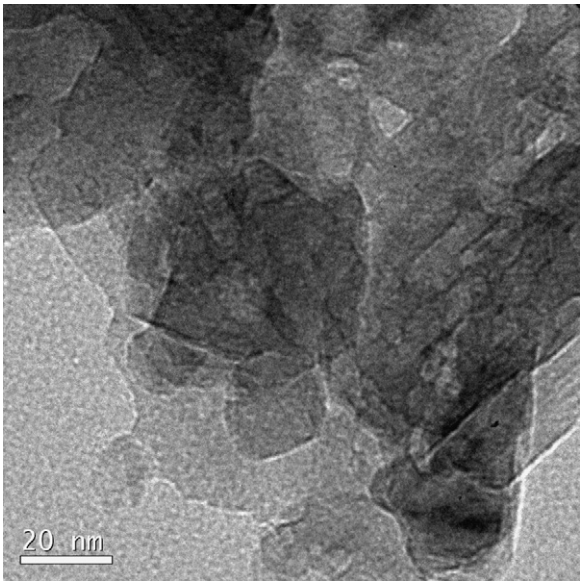

Fig. 4. TEM image of microwave heated Al powder.

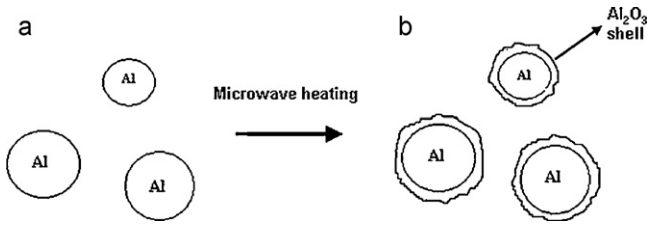

Fig. 5. Schematic representation showing (a) as received aluminium particles before microwave heating and (b) aluminium particles with Al_2O_3 shell after microwave exposure.

3.4. TEM study

Fig. 4 demonstrates the TEM image of the microwave heated Al powder. The TEM micrograph revealed that a coating layer was formed on the surfaces of Al particles. FTIR and EDX data indicated the formation of α-Al_2O_3 coating on Al particles. Therefore, TEM observation further corroborated the FTIR and EDX analysis. In the similar manner, Wang et al. had shown the formation of amorphous silica coating on ultrafine α-Al_2O_3 particles by TEM micrograph [20].

3.5. Mechanism

XRD of the microwave treated Al powder demonstrated that the Al–Al_2O_3 composite powder was formed by microwave heating of the Al powder. FTIR study and EDX analysis indicated the formation of core–shell type Al–Al_2O_3 composite. This can only happen because of the fact that every Al particle absorbs the microwave power that results in heating in ambient atmosphere and thus, it gets oxidized. Therefore, it may be plausible to assume that finely divided Al metal powder was heated due to some kind of energy loss mechanisms in the microwave field. Thus, an oxide shell was formed around the Al core particle as shown in Fig. 5.

4. Conclusions

Al–α-Al_2O_3 composite was formed by the microwave heating of commercial Al powder for 45 min. FTIR study, EDX analysis and TEM observation of the microwave processed Al powder indicated the formation of Al–α-Al_2O_3 core–shell composite. The present method of microwave heating may provide an easier means to achieve the Al–Al_2O_3 composite formation.

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