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Study on the Sintering Behavior of Spinel-Al Composite: Preparation and Microstructure

Qasid A. Saleh, A. Hussein Saleh

Abstract—Al-Spinel composites are of interest in many industrial and electronic fields. So in this research, Al-spinel specimens were prepared by powder technology technique, by mixing Al (matrix material) with different weight percentage of spinel (reinforced material). The prepared composites were characterized through x-ray diffractometer (XRD), scanning electron microscopy (SEM). The effect of spinel content on the phase compositions, density, water absorption, compressive strength, and thermal conductivity was investigated. The result showed that the amount of spinel added to Al matrix material affected the sintering parameters, thus effecting the density, strength, thermal conductivity and microstructure of the composites. With an increase in spinel content, sintered density decreased, water absorption increase. The strength and thermal conductivity decreased. The optimized product is composite specimen with 2 wt.% spinel content which has a high sintered density (2.31, 2.32, 2.33, 2.34, 2.35 g\cm^3), high strength (17 N/ mm^2), high thermal conductivity (1.25 w/m.c), and low water absorption (2.69, 2.6, 2.4, 2.27, 2.16%).

Index Terms—Sintering, behavior, microstructure, Spinel-Al, composite.

1 Introduction

Fabrication and application of aluminum matrix compo sites have received a great deal of attention because of their good wear resistance, high specific strength, and superior thermal properties compared with pure aluminum metal and alloys [1,3]. Pure aluminum has been studied extensively as a matrix material of MMCs due to its good mechanical properties, good formability, and corrosion resistance [4]. Among different kinds of ceramics, Sic [5,6], and Al_2O_3 [7] are mostly used as reinforcing material to improve aluminum matrix properties. As the softening temperature of pure Al is 2.36 × 10^{-6} in the temperature range from 0 to 300 C^0, which limits the application of Al at elevated temperatures. So, $MgAl_2O_4$ spinel is extensively used to reinforce Al metal matrix which has made wide applications of these composites. As a result of low thermal expansion coefficient (CTE) of $MgAl_2O_4$ ($9\times10^{-6}/C^0$) [8], $MgAl_2O_4$ distributed in aluminum matrix are expected to reduce the CTE and enhance the wear resistance of aluminum composites [9]. Therefore, $MgAl_2O_4$ could be very promising in improving the thermal stability and wear resistance of Al matrix composites. According to previous studies and as reported, the addition of ceramic particles as reinforcement, can significantly improve the overall properties of the composites, and also have a direct effect on the sintering behavior. Most of the conventional synthesis methods of aluminum matrix composites are the ex-situ process (10-11), that is, the reinforcements are added in to the matrix and mixed together by mixer. This kind of process inevitably causes inherent problems, such as poor distribution of reinforcements in matrix, weak interference bonding between both, matrix and reinforcement, structural damage of the reinforcement [13]. So the main object for composite synthesis is obtaining a good combination between spinel and aluminum matrix. Shweinfest et al. reported that the misfit of $MgAl_2O_4$ (100)/Al (100) interface is rather small, only 0.25% [14], which means a good combination between the interfaces.

The $MgAl_2O_4$ Spinel can thus be a proper reinforcement in the aluminum matrix. Tateoki et al. investigated the effects of the average size on the properties of $MgAl_2O_4$ reinforced AC4C aluminum composites. Also, they observed that the dislocations in the matrix around the $MgAl_2O_4$ particle are resulting from the mismatch of thermal expansion coefficients between $MgAl_2O_4$ and Al. In this study, aluminum matrix composites reinforced with different synthesize spinel content were prepared conventional solid state reaction (SSR). The microstructural tests and sintering properties of Spinel-Al composite were investigated.

2 Experimental Procedures

The $MgAl_2O_4$-Al composite was synthesized through powder metallurgy by using aluminum powder (99% purity, purchased from Central Drug House (P) Ltd. 7/28 Vardaan House, Daryaganj, New Delhi-110002 India) as a raw material. Aluminum, magnesia, and alumina powders have a particle size ranging from 20 to 30 µm. The MgO (magnesia) and alumina powders were mixed in a molar ratio of 1:1. The mixed powders were then compressed in to a cylinder with 12mm in diameter and 40mm height under a pressure of 25Mpa. Subsequently, the obtained compacts were put in to a furnace and sintered at 1200 and 1400 C^0 for 2h. Thus, the stoichiometric $MgAl_2O_4$ Spinel was prepared at each sintering temperature via solid state reaction. After crushing and grinding the sintered spinel specimens, different amount of Spinel powder (0,2,4,6,8 wt.%) were added in to the aluminum powder and mixed in ball milling. Next, mixed powders were compacted in to a bulk with 12 mm in diameter and 50 mm height under a pressure of 40 Mpa. The green compacted specimens of $MgAl_2O_4$-Al composite were heated in a furnace at 450,500,550,600,650 C^0 for 2h. Then the properties of the produced composites with spinel contents ranging from 0,2,4,6,8 wt.% were studied, such as bulk density of the fired specimens which was evaluated by using Archimedes method, the compressive strength of the sintered specimens was also evaluated by Instron device, while the phase transformations were characterized by X-ray diffractometry with Cu Kα radiation (λ= 1.54187 A^0). Also, the microstructures of the specimens were measured by scanning electron microscopy (SEM, FEI, inspect S50, Netherlands).

3 Results and discussion

3.1 Sintered density

The effect of spinel content calcined at 1400 °C on the sintered density of spinel -Al composite as shown in Figure 1. Since the density of $MgAl_2O_4$ Spinel (3.45 g/cm^3) [15] is higher than that of pure Al (2.79 g/cm^3), the theoretical density of the composites increases with increasing spinel content. The resulting densities are much lower than the theoretical value as shown in Fig. 1. Sintered density of the samples increased with short range addition of spinel reaching their maxima at 2 wt.% of spinel addition and their densities decreased with long range addition of spinel content, due to the formation of a number of the pores and voids resulting from the consumption of Al matrix and Mg powder during sintering reaction. On the other hand, the network structure of the $MgAl_2O_4$ prevents the flow of metal and expansion of Al matrix at high temperatures [16].

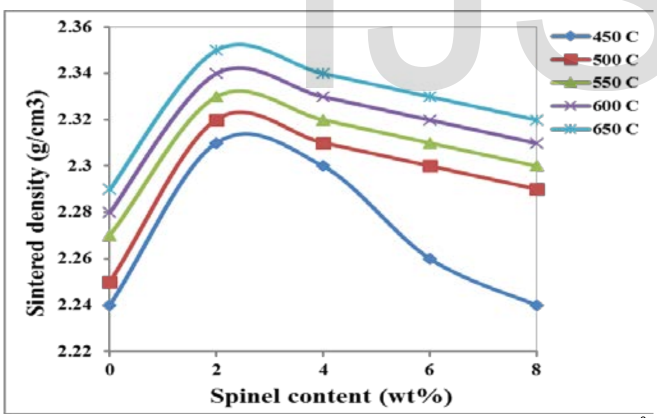

Fig. 2. Effect of spinel content on the sintered density of spinel1400 °C –Al composite.

3.2 Water absorption

Fig. 2 show that effect of the spinel content synthesis at 1400 °C on water absorption of Spinel-Al composite. The figure shows that water absorption of composite increases with increasing spinel content and increasing firing temperature as a result of the un complete densification which increases a large content of pores or voids present in the existence of additives like spinel [17].

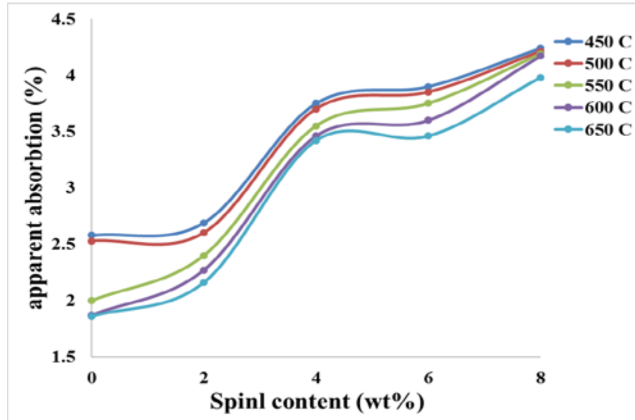

Fig. 2. Effect of spinel content on the water absorption of spinel 1400 ^{0}C –Al composite.

3.3 Compressive strength

To investigate the effects of spinel content on the compressive strength of the composites, compressive tests at firing temperature 650^{0}C were performed. It can be seen in Fig. 3 that the compressive strength of the composites decreases with an increasing content of $MgAl_2O_4$ especially that synthesis at 1400^{0}C firing temperature, which is mainly due to the decreasing in ductility of composites compared with that of the Al matrix material. Besides, calcined temperature of spinel makes difference in compressive strength of Spinel-Al samples due to the rate of spinel formation. This related to the better sintering (densification) which accompanied by relatively lower contents of pores and voids [18].

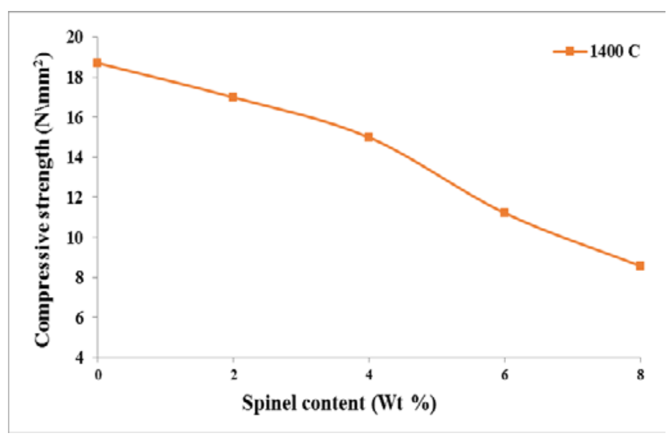

Fig. 3. Effect of spinel content on the compressive strength of Spinel-Al composite fired at 650 ^{0}C

3.4 Thermal conductivity

Fig. 4 shows the effect of spinel content sintering at 1400 ^{0}C on thermal conductivity of composites sintering at 650 ^{0}C for 2h soaking time. The thermal conductivity values of the composites with different spinel content at the temperature 650 ^{0}C decrease with increasing spinel content. This decrease becomes slowly with spinel content of 6% and 8%, which means that with lower spinel content, there is an ob-

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vious decline of the thermal conductivity can be observed. So, increasing spinel content in the Al matrix is beneficial for reducing the thermal conductivity of pure Al.

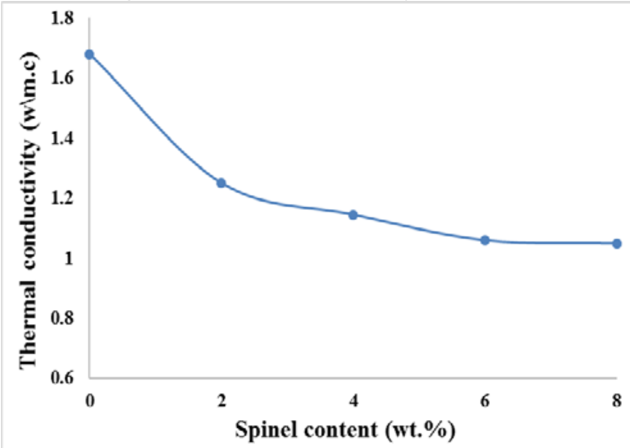


Fig. 4. Effect of spinel content on the thermal conductivity of Spinel 1400 ^{0}C-Al composite at 650^{0}C

3.5 Microstructure of composite

XRD diffraction patterns of the composites with different spinel content are shown in Fig. 5. Only two phases were detected, aluminum and $MgAl_2O_4$ spinel proving that the composite product is single pure $MgAl_2O_4$ spinel with no by-products. It can be seen that the strongest four peaks appeared at 2Θ values of 38.5^0, 44.67^0, 65.1^0 and 78.1^0. These peaks respond to (111), (200), (220) and (311) diffraction planes of Al samples, whereas the strongest four peaks appeared at 2Θ values of (38.65^0, 44.8^0, 65.1^0 and 60.12^0), (38.7^0, 44.9^0, 65.25^0 and 60.27^0), (39.16^0, 45.4^0, 65.71^0, and 60.34^0) and (38.81^0, 44.96^0, 65.45^0 and 59.66^0) corresponding to (111), (200), (220) and (333) for 2,4,6,8wt% of spinel respectively. This confirmed that the growth rates as well as its high crystillanity of the produced Spinel-Al composite depends on the firing temperature of the spinel which complete the spinalization process [19].

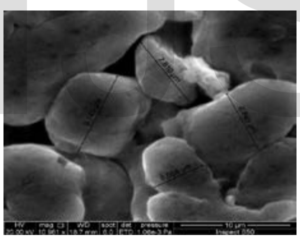
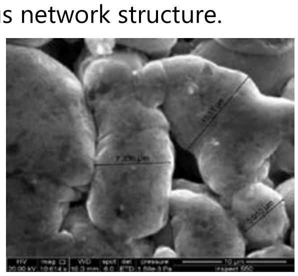


Figure 6 shows the morphology of different $MgAl_2O_4$ spinel content in the Al matrix at 650 ^{0}C. Spinel with different contents are dispersed uniformly in the matrix and form a continuous network structure.

Also, it will be notice a good growth and combination between spinel reinforced material and aluminum matrix material, which significantly improved the homogeneity and binding ability of spinel with aluminum matrix compared with other reinforcement materials. As a result of appearing the voids in composites Fig. 6 a, b, c, d and e no higher density could be obtained in spite of the spinel particles were pushed in to the matrix during sintering and combined with neighboring Al particles.



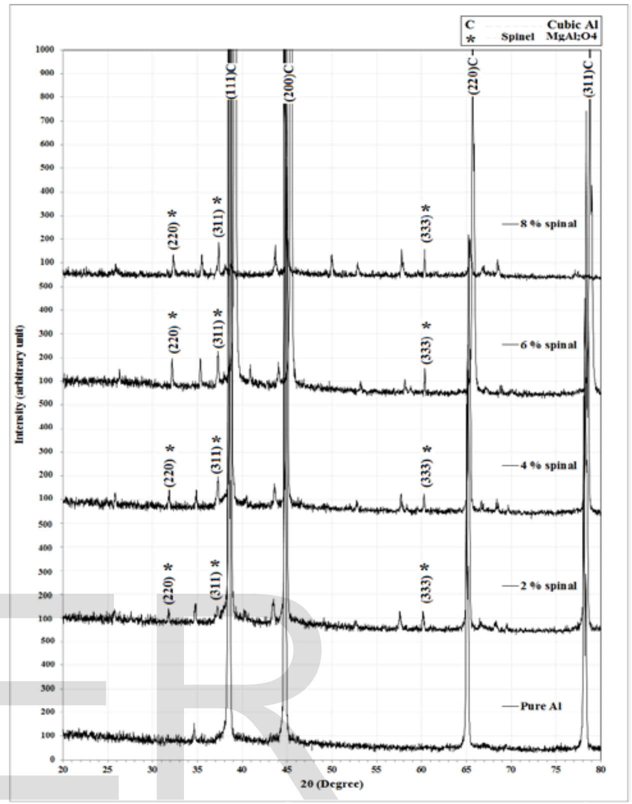
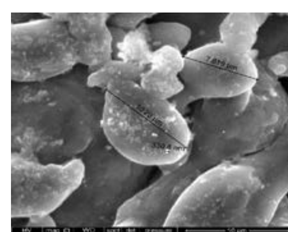
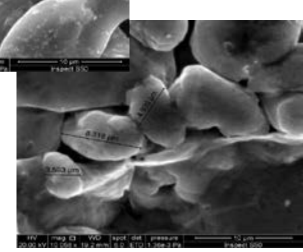


Fig. 5. XRD plot of Spinel$_{1400}$ ^{0}$_C$-Al composite samples fired at 650^{0}C with different spinel content.

(a) (b) (c) (d)

(e)

Fig. 6. Effect of Spinel content on microstructure of Spinel 1400 ^{0}C-Al composite sintered at 6500C (a) 0wt%, (b) 2 wt.%, (c) 4 wt.%, (d) 6 wt.%, (e) 8 wt.% Spinel content.

4 Conclusion

Based on the microstructure, sintering, mechanical and refractory properties studies, it can be concluded that:

1. Different compositions of synthesized $MgAl_2O_4$ spinel reinforced Al composites could be attained at firing temperature 450,500,550,600,650 ^{0}C via powder metallurgy technique.

2. $MgAl_2O_4$-Al composites are characterized with better sintering, mechanical and refractory properties that the pure Al matrix material. Ideal yield of $MgAl_2O_4$-Al composites was obtained at 2wt.% of spinel content which formed at 450,500,550,600,650 ^{0}C.

3. The resistance to the matrix softening and the generation of voids in the Al matrix by the spinel particles played the most important role in the reinforcing effect of these particles at elevated temperatures.

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