



Effect of Sc_2O_3 addition on densification and microstructure of different spinelized magnesium aluminate spinels

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Abstract: The densification and microstructure of different spinelized magnesium aluminate spinels (MAS) were studied adding Sc_2O_3 as additive. Sintered products were then characterized in terms of densification, phase analysis, quantitative elemental analysis and microstructure. The results show that Sc_2O_3 is found to be beneficial for the densification of MAS. Sc_2O_3 has a more significant effect on the densification of partially spinelized MAS batch than that of fully spinelized MAS batch. At the sintering temperature of 1650 °C, the bulk density of sintered products of partially spinelized powders increases by 0.243 g/cm³ as the Sc_2O_3 content increases from 0 to 4% (mass fraction) and that of fully spinelized powder increases by 0.14 g/cm³. Compared with the sintered samples prepared from the fully spinelized powder, the sintered samples using the partially spinelized powders as raw materials have more compact microstructures.

Key words: magnesium aluminate spinel; Sc_2O_3 ; densification; microstructure; elemental analysis

1 Introduction

Magnesium aluminate spinel (MAS) features a high melting point (2135 °C), high hardness (16 GPa), low thermal expansion, good chemical stability, and good resistance to thermal spalling and corrosion [1–4]. MAS has been used for various applications, such as refractories [5,6], humidity sensors [7] and transparent ceramic materials [8–11]. MAS has also been utilized as anode materials [12] and man-made ledge materials [13] in aluminum electrolytic cells. Nevertheless, MAS cannot be densified through single-stage firing because its volume expands as high as 8% in the solid-state reaction. Thus, MAS is densified through a double-stage firing process, in which alumina and magnesia are initially calcined at above 1500 °C to produce MAS powders with the desired spinel phase (>80%); the powders are then ground, compacted and sintered at above 1900 °C [14]. The formation temperature of the MAS phase is high in the first stage and the agglomeration of the spinel phase is serious [15]. The calcination temperature of the solid-state reaction is controlled to obtain different spinelization degrees and sintering activities of MAS

powders in the first stage, which considerably affects the subsequent sintering process.

The spinelization of the MAS powder is not only related to the formation temperature, but also to the added amount of spinelizer. GANESH et al [16] studied the effect of AlCl_3 on the synthesis and densification of MAS through double-stage firing. The result showed that the spinelization of spinel was approximately 82% without any additive and increased to 100% with the addition of 0.03% AlCl_3 (mole fraction). They also found that AlCl_3 exhibited optimal performance on the densification of partially spinelized MAS powder. These findings indicate that the influence of additive on the sintering behavior varies in different spinelized MAS samples. Over the recent years, researchers have reported the effect of additives on the densification of MAS [17–20]. Adding LiF could enhance the densification of spinel through liquid-phase sintering [17]. The addition of TiO_2 also enhanced the densification of spinel through exsolution of Al_2O_3 and dissolution of TiO_2 [18]. In the case of ZnO addition, the formation of anion vacancy could induce the densification of spinel [19]. Satisfactory densified spinel products were obtained after addition of different rare earth oxides. Dy_2O_3 prevented the

exaggerated grain growth and thereby contributed to the densification process [20]. Nevertheless, these studies did not evaluate the effect of sintering additives on the densification of different spinelized MAS samples.

Rare earth oxides can be easily converted to a solid solution with sintered products and then increase the lattice defects [21]. Hence, these oxides can also promote the densification of MAS. In addition, the difference between rare earth cation ionic radius and the main lattice ionic radius directly affects the densification of MAS. The ion radius of Sc^{3+} is the smallest compared with that of the other rare earths, and Sc^{3+} exhibits similar characteristics to Al^{3+} . In view of these findings, a systematic study was performed to investigate the influence of Sc_2O_3 addition on the densification and microstructure of different spinelized MAS batches prepared at 1200 and 1400 °C from magnesia and alumina (mole ratio of MgO to Al_2O_3 , 1:1). Different spinelized batches were sintered at 1550, 1600 and 1650 °C. The final sintered products were characterized through X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive analysis of X-ray (EDAX) analyses to determine the role of Sc_2O_3 addition in MAS.

2 Experimental

The physicochemical properties of the starting oxides are listed in Table 1, which shows that the purity values of alumina and magnesia are 97.286% and 99.899%, respectively.

Batch composition was made from the raw materials in such a way that mole ratio of MgO to Al_2O_3 was 1:1. All batches were milled for 3 h using a planetary ball mill. Milled powders were calcined at a heating rate of 3 °C/min and 1200 and 1400 °C for 3 h, respectively. The spinels prepared at 1200 and 1400 °C were termed as LS batch and HS batch, respectively. The degree of conversion to MAS was determined by the conventional acid dissolution method [22].

Sc_2O_3 (99.99%, $w(\text{SiO}_2)<0.005\%$, $w(\text{CaO})<0.012\%$, $w(\text{Fe}_2\text{O}_3)<0.05\%$) was added at 1%, 2% and 4% (mass fraction) into the LS and HS batches, respectively. All batches were individually milled for 1 h. Afterwards, the milled powders were uniaxially pressed at 200 MPa on a hydraulic press (WE-300C) into $d20\text{ mm} \times 15\text{ mm}$

cylindrical samples. The pressed samples were then sintered at 1550, 1600 and 1650 °C for 3 h. Firing was conducted in a program-controlled electric furnace (KSL-1750X) and the heating rate was maintained at 3 °C/min.

The bulk density and apparent porosity of the sintered products were measured using the conventional liquid displacement method, which applied Archimedes' principle. Phase analysis was conducted via the X-ray diffraction technique. The diffraction patterns of the finely powdered samples were obtained with an X-ray diffractometer using nickel-filtered Cu K_α radiation. The lattice parameter (a) was calculated from the characteristic XRD peaks of the spinel phase using the following equation [23]:

$$a = \frac{\lambda}{2 \sin \theta} \sqrt{(h^2 + k^2 + l^2)} \quad (1)$$

where a is the lattice parameter; λ is the radiation wavelength; θ is the Bragg's angle; and h , k and l are the corresponding Miller indices. The microstructures of the sintered products were observed with a scanning electron microscope (SEM, Nova NanoSEM 230) using sputtered gold coating on the polished surface after thermal etching. Quantitative elemental analysis of the selected grain was done by EDAX.

3 Results and discussion

3.1 Characterization of raw materials

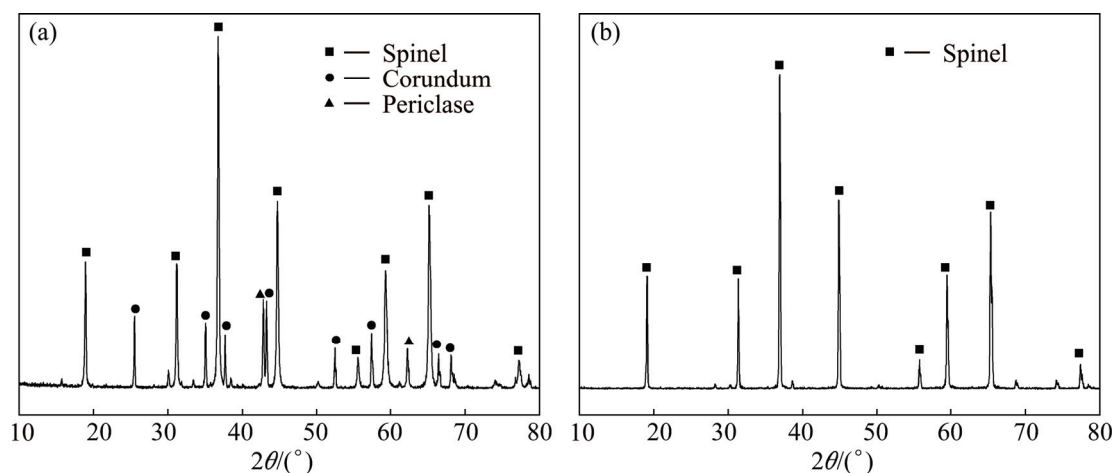
The physicochemical properties of the calcined powders prepared at 1200 and 1400 °C are given in Table 2. The XRD patterns (Fig. 1) of the calcined powders indicate the presence of the spinel, periclase and corundum phases for the LS batch, whereas only the spinel phase is observed for the HS batch. The degree of conversion to MAS calcined at 1200 °C is 93.1%. The starting raw materials were converted completely into MAS for the HS batch, which indicated an increase in the conversion degree to MAS with the increase in calcination temperature. Table 2 shows that the true density of the LS batch is the same as that of the HS batch, which is 3.58 g/cm³. However, the average particle size of the HS batch is 65.79 μm, which is significantly larger compared with that of the LS batch (23.76 μm). The difference may be attributed to the higher calcination temperature of MAS, which results in

Table 1 Physicochemical properties of magnesia and alumina

Starting oxide	Chemical composition, w/%			Physical property		
	Al_2O_3	MgO	Impurities	True density /(g·cm ⁻³)	Average particle size/μm	Phase
Alumina	97.286	–	2.714	3.97	21.7	Corundum
Magnesia	–	99.899	0.101	3.57	5.8	Periclase

Table 2 Physicochemical properties of calcined powders

Calcined powder	Chemical composition, w/%		Physical property		
	MgAl ₂ O ₄	Others	True density /(g·cm ⁻³)	Particle size/μm	Phase
LS	93.1	6.9	3.58	23.76	Spinel, corundum, periclase
HS	100	0	3.58	65.79	Spinel

**Fig. 1** XRD patterns of LS (a) and HS (b) batches

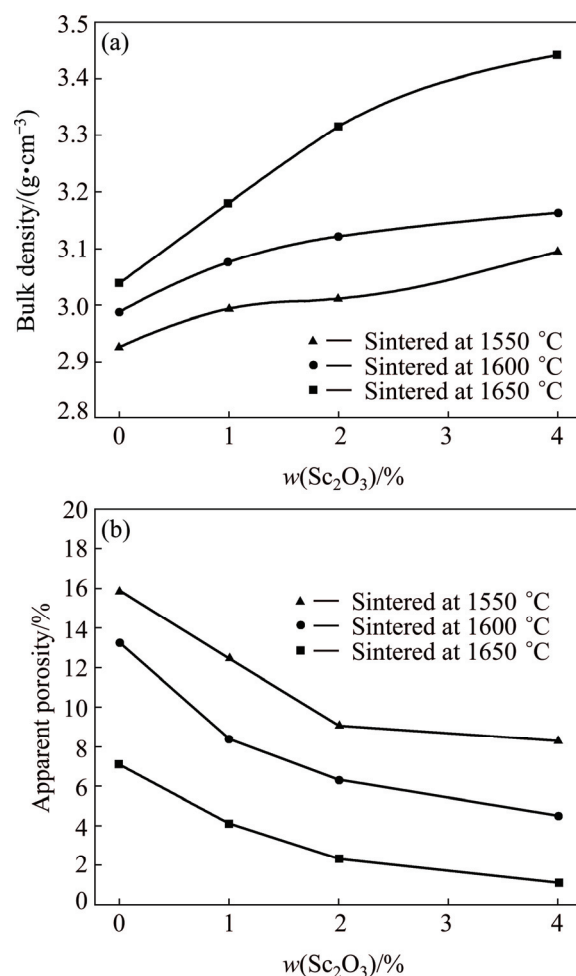
the formation of large agglomerates, thereby inhibiting sintering activity upon subsequent densification operation.

3.2 Densification

The variations in bulk density and apparent porosity of the sintered products of the LS and HS batches (termed as S_{LS} and S_{HS} , respectively) with sintering temperature and Sc_2O_3 content are shown in Figs. 2 and 3, respectively.

A general trend of increasing bulk density and decreasing apparent porosity was observed with the increase in sintering temperature at the same Sc_2O_3 content. This trend can be explained by the great driving force for sintering at the high sintering temperature. A general trend of increasing bulk density and decreasing apparent porosity was also observed in Figs. 2 and 3 with the increase in Sc_2O_3 content at the same sintering temperature. Sintering at 1650 °C was not sufficient to obtain high densification in the samples without an additive; however, increasing the Sc_2O_3 content to 4% (mass fraction) results in a densification of 96.17%. This result may be attributed to the fact that Sc^{3+} with characteristics similar to Al^{3+} can substitute Al^{3+} in the spinel. Moreover, incorporating Sc^{3+} into the spinel structure causes strain in the lattice (because of ionic size variation), which improves diffusion, mass transfer and densification.

The S_{LS} samples exhibit better sintering characteristics than the S_{HS} samples at the same sintering temperature and Sc_2O_3 content. This result may be

**Fig. 2** Variations in bulk density (a) and apparent porosity (b) of S_{LS} samples with sintering temperature and Sc_2O_3 content

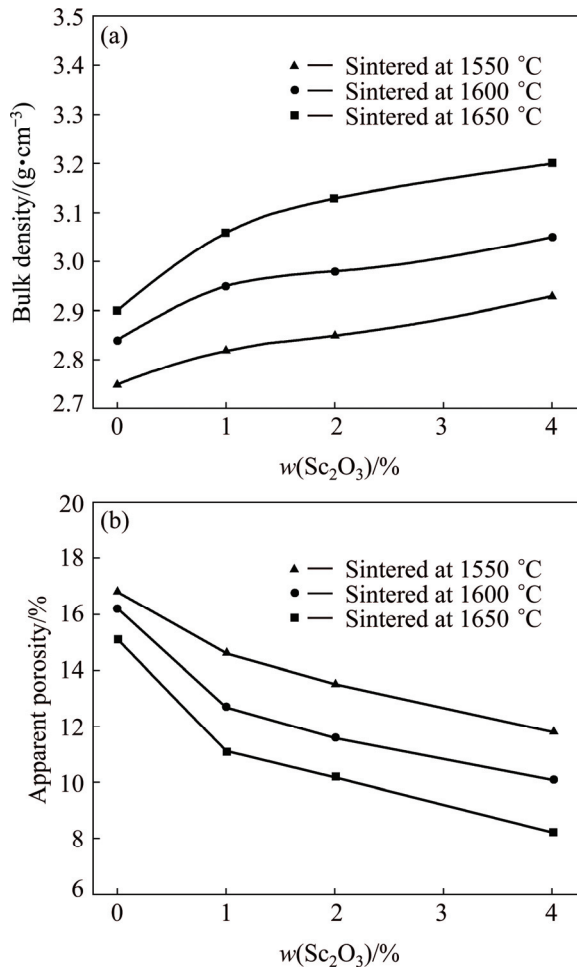


Fig. 3 Variations in bulk density (a) and apparent porosity (b) of S_{HS} samples with sintering temperature and Sc_2O_3 content

attributed to the fact that the average grain size of the LS batch ($23.76\ \mu\text{m}$) is smaller than that of the HS batch ($65.79\ \mu\text{m}$). In addition, free magnesia and alumina of the LS batch can react to produce MAS during sintering. The formation of a new spinel can be considered a seed that acts as a core for the grain growth of the spinel.

Upon sintering at $1650\ ^\circ\text{C}$, the increasing amplitude of the bulk density of S_{LS} , with the increase in Sc_2O_3 content is greater than that of S_{HS} . Moreover, the decreasing amplitude of apparent porosity of S_{LS} is also larger than that of S_{HS} . With the increase in Sc_2O_3 content from 0 to 4%, the bulk density of S_{HS} increases by $0.14\ \text{g}/\text{cm}^3$, whereas its apparent porosity decreases by 3.6%. However, the bulk density of S_{LS} increases by $0.243\ \text{g}/\text{cm}^3$ and its apparent porosity decreases by 7.2%. The higher densification of the S_{LS} sample sintered at $1650\ ^\circ\text{C}$ may be largely determined by the combined effects of the newly formed spinel and Sc_2O_3 addition.

3.3 XRD patterns

Figure 4 shows the XRD patterns of the S_{LS} and S_{HS}

samples with different Sc_2O_3 contents sintered at $1650\ ^\circ\text{C}$. Sc_2O_3 contents were 0, 1%, 2% and 4%, respectively. These samples are termed as $\text{S}_{\text{LS}0}$, $\text{S}_{\text{LS}1}$, $\text{S}_{\text{LS}2}$, $\text{S}_{\text{LS}4}$ and $\text{S}_{\text{HS}0}$, $\text{S}_{\text{HS}1}$, $\text{S}_{\text{HS}2}$, $\text{S}_{\text{HS}4}$, respectively. The phase analysis study of all the sintered products, with and without additives, shows only the spinel phase. No periclase and corundum phases are observed in all the samples, which confirms the consumption of Al_2O_3 and MgO in spinel phase formation.

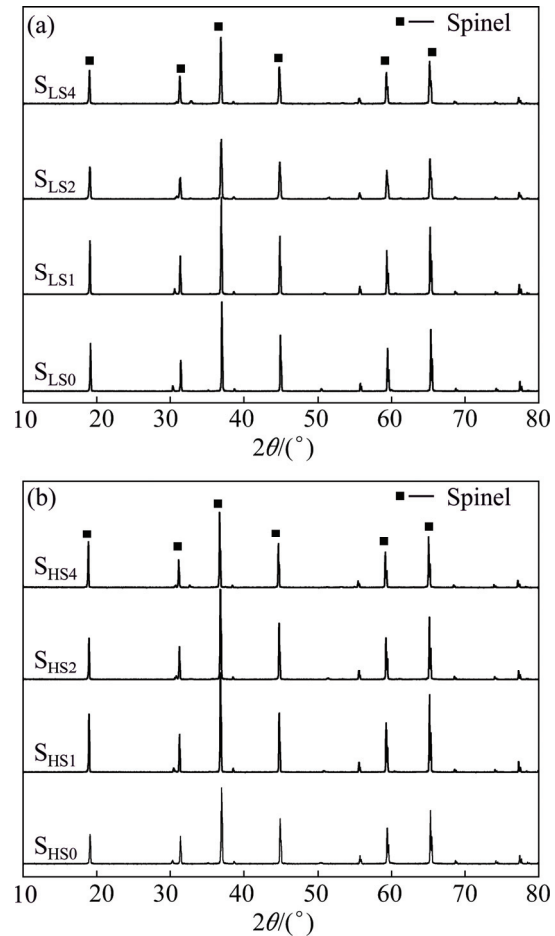


Fig. 4 XRD patterns of S_{LS} (a) and S_{HS} (b) samples with different Sc_2O_3 contents sintered at $1650\ ^\circ\text{C}$

Figure 5 shows the XRD 2θ scans of S_{LS} and S_{HS} with different Sc_2O_3 contents sintered at $1650\ ^\circ\text{C}$. The 2θ values of the peak from the (311) plane of the spinel in the sintered products of S_{LS} and S_{HS} decrease with the increase in Sc_2O_3 content. These values are 36.98° , 36.90° , 36.90° , 36.84° and 36.96° , 36.82° , 36.82° , 36.72° for batches $\text{S}_{\text{LS}0}$, $\text{S}_{\text{LS}1}$, $\text{S}_{\text{LS}2}$, $\text{S}_{\text{LS}4}$ and $\text{S}_{\text{HS}0}$, $\text{S}_{\text{HS}1}$, $\text{S}_{\text{HS}2}$, $\text{S}_{\text{HS}4}$, respectively.

In order to further investigate the crystal structure of the spinel, the lattice parameters were calculated using Eq. (1). The results are presented in Table 3. The lattice parameter of MAS increases with the increase in Sc_2O_3 content at the same sintering temperature. The lattice

parameter of the MAS for S_{HS} samples increases from 0.806 to 0.811 nm with the increase in Sc_2O_3 content from 0 to 4% (mass fraction). This increase may be attributed to the fact that Sc_2O_3 makes a solid solution with the spinel. The ionic radius of Sc^{3+} is 0.083 nm [24] and that of aluminum ion is 0.05 nm [25]. Given its higher ionic radius, incorporating Sc^{3+} into the spinel structure expands the lattice, which increases lattice parameters.

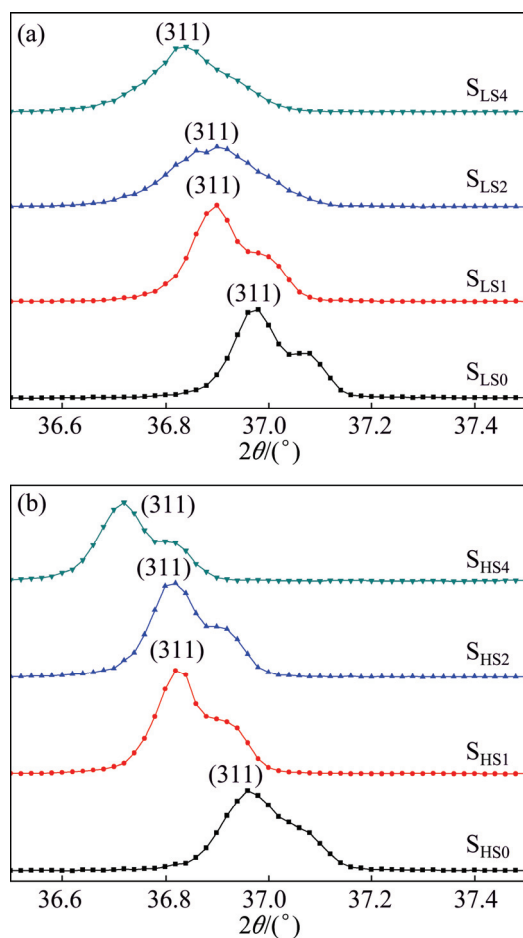


Fig. 5 XRD 2θ scans of S_{LS} (a) and S_{HS} (b) samples with different Sc_2O_3 contents sintered at 1650 °C

Table 3 Lattice parameters (a) of S_{LS} and S_{HS} samples with different Sc_2O_3 contents sintered at 1650 °C

Sample	S_{LS0}	S_{LS1}	S_{LS2}	S_{LS4}	S_{HS0}	S_{HS1}	S_{HS2}	S_{HS4}
a/nm	0.805	0.807	0.807	0.808	0.806	0.809	0.809	0.811

3.4 SEM images and EDAX patterns

The SEM images and the EDAX patterns of the polished and thermally etched samples (S_{LS0} , S_{LS4} , S_{HS0} and S_{HS4}) are shown in Fig. 6. Figures 6(a₁) and (c₁) show an inhomogeneous grain growth for the S_{LS0} and S_{HS0} samples. The spinel grain shape is angular with a clear grain profile. The average grain size of S_{HS0} sample is considerably smaller than that of S_{LS0} sample. Thus,

the formation of a new spinel favors grain growth during sintering. Figure 6 shows that the microstructures of S_{LS4} and S_{HS4} are more compact with a minimal amount of pores, which is responsible for the higher densification of 4% Sc_2O_3 -containing samples.

Figure 6 also shows that the major elements of all the sintered products include Mg, Al, and O. In addition, S_{LS4} and S_{HS4} contain Sc. The quantitative elemental analysis results of various samples by EDAX were converted into oxide contents and presented in Table 4. S_{LS0} and S_{HS0} have a nearly stoichiometric spinel grain without Sc_2O_3 , as shown in Table 4. The Sc_2O_3 additive is mainly presented in the spinel grain, which indicates the solid solubility of Sc_2O_3 in the spinel and verifies that Sc^{3+} ions have replaced Al^{3+} ions from the spinel grains.

Table 4 Chemical compositions of spinel grain in various samples by EDAX

Spinel grain in samples	Chemical composition, w/%		
	MgO	Al_2O_3	Sc_2O_3
S_{LS0}	27.09	72.91	—
S_{LS4}	27.15	70.70	2.15
S_{HS0}	26.21	73.79	—
S_{HS4}	15.57	61.17	22.69

S_{LS0} , S_{LS4} , S_{HS0} and S_{HS4} samples were characterized in terms of chemical compositions by XRF and shown in Table 5. 2.37% Sc_2O_3 measured by XRF is nearly the same as 2.15% Sc_2O_3 measured by EDAX for S_{LS4} . This finding indicates that Sc_2O_3 is uniformly distributed in the spinel grains of S_{LS4} . Sc_2O_3 is inhomogeneously distributed in the spinel grains of S_{HS4} according to the difference in Sc_2O_3 content between 22.69% (by EDAX) and 5.21% (by XRF). This result may be ascribed to the fact that Sc_2O_3 dissolves firstly in MgO and then precipitates gradually from MgO during the formation of the spinel [26]. Thus, Sc_2O_3 is uniformly distributed throughout the spinel grains of the S_{LS} sample. This distribution may be the reason why the bulk density of S_{LS4} is greater than that of S_{HS4} .

Table 5 Chemical compositions of S_{LS0} , S_{LS4} , S_{HS0} and S_{HS4} samples by XRF

Sample	Chemical composition, w/%			
	MgO	Al_2O_3	Sc_2O_3	Others
S_{LS0}	27.53	67.89	—	4.58
S_{LS4}	27.44	67.71	2.37	2.48
S_{HS0}	28.21	67.99	—	3.80
S_{HS4}	26.19	62.99	5.21	5.61

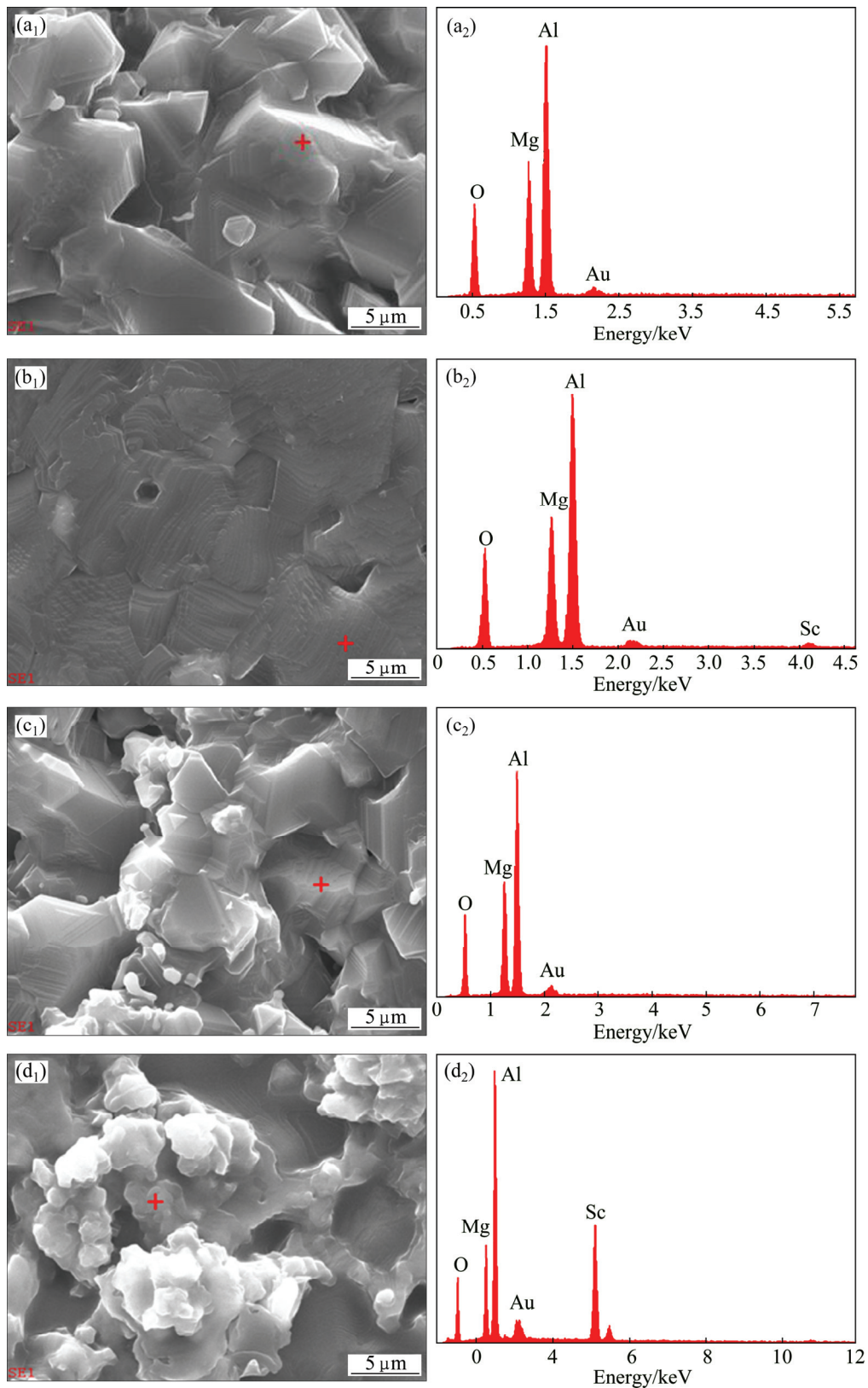


Fig. 6 SEM images and EDAX patterns of S_{LS0} (a₁, a₂), S_{LS4} (b₁, b₂), S_{HS0} (c₁, c₂) and S_{HS4} (d₁, d₂) samples

4 Conclusions

1) Incorporating Sc_2O_3 into magnesium aluminate spinel favors the densification and up to 96.17% of true

density can be achieved at 1650 °C. The Sc^{3+} of Sc_2O_3 can substitute the trivalent Al^{3+} of the spinel structure at a high sintering temperature. Incorporating of Sc^{3+} into the spinel structure generates the lattice strain and improves mass transfer and densification.

2) The densification of partially spinelized powder is higher than that of the fully spinelized powder at the same sintering temperature and Sc_2O_3 content.

3) Quantitative elemental analysis by EDAX shows that the Sc_2O_3 additive is mainly present in the spinel grains, which confirms the solid solubility of Sc_2O_3 in the spinel. The combined SEM and XRF analysis results show that Sc_2O_3 is uniformly distributed throughout the spinel grains of the S_{LS} sample, but inhomogeneously distributed throughout the spinel grains of the S_{HS} sample.

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Sc₂O₃ 对不同尖晶石化镁铝尖晶石的致密化和显微组织的影响

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摘 要: 探讨添加 Sc₂O₃ 对不同尖晶石化镁铝尖晶石的致密化和显微组织的影响。通过致密化研究、物相分析、元素定量分析和显微组织研究对烧结产物的性能进行表征。结果表明: 添加 Sc₂O₃ 有利于镁铝尖晶石的致密化, 且相对于完全尖晶石化的镁铝尖晶石, Sc₂O₃ 对促进部分尖晶石化的镁铝尖晶石的烧结作用更显著。在 1650 °C 烧结, Sc₂O₃ 添加量从 0 增加到 4% (质量分数) 时, 部分尖晶石化原料的烧结产物的体积密度增加了 0.243 g/cm³, 完全尖晶石化原料的烧结产物的体积密度增加了 0.14 g/cm³。与用完全尖晶石化粉末制备的烧结试样相比, 以部分尖晶石化粉末为原料的烧结试样具有更加致密的显微组织。

关键词: 镁铝尖晶石; Sc₂O₃; 致密化; 显微组织; 元素分析

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