The American Journal of Applied Sciences
ISSN 2689-0992 | Open Access

TYPE Original Research
PAGE NO. 116-120
DOI 10.37547/tajas/Volume07Issue11-13

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Synthesis And Phase Formation Of Cordierite Ceramics Prepared From High-Purity Oxides

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OPEN ACCESS

SUBMITTED 14 October 2025
ACCEPTED 09 November 2025
PUBLISHED 30 November 2025
VOLUME Vol.07 Issue 11 2025

CITATION
Muminov Samandar Minavar ugli, Kadyrova Zulayho Raimovna, Eminov Azizjon Ashrapovich, & Khomidov Fakhriddin Gafurovich. (2025). Synthesis And Phase Formation Of Cordierite Ceramics Prepared From High-Purity Oxides. The American Journal of Applied Sciences, 7(11), 116–120. https://doi.org/10.37547/tajas/Volume07Issue11-13

Abstract: In this study, cordierite ceramics were synthesized by the solid-state reaction method from a mixture of high-purity oxide powders, including magnesium oxide (MgO), aluminum oxide (Al_2O_3), and silicic acid ($SiO_2 \cdot nH_2O$). The cordierite phase was formed through sintering at 1400 °C and 1430 °C for 1 hour (h). The phase formation and structural transformations during sintering were analyzed using X-ray diffraction (XRD). The XRD analysis results showed that cordierite was a completely formed phase.

Keywords: Cordierite; Solid-state reaction; Phase transformation; α-cordierite; μ-cordierite; Spinel; Cristobalite; XRD analysis; Ceramic synthesis; Thermal stability.

Introduction

Cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$) is a magnesium aluminum silicate ceramic that has attracted significant attention due to its low thermal expansion coefficient, high thermal stability, low dielectric constant, excellent resistance to thermal shock high chemical durability, high refractoriness high mechanical strength. These properties make it a promising material for various applications such as honeycomb-shaped catalyst carriers in automobile exhaust systems, substrate material for integrated circuit boards, thermal shock-resistant tableware, heat exchanger for gas turbines, porous ceramics and refractory materials [1-7].

Cordierite is synthesized through different methods, such as solid-state reaction of MgO, Al2O3 and SiO2 or their precursors [8], wet chemical methods such as sol–gel, hydrothermal, spraypyrolysys, and combustion synthesis [9-11]. Among these, the solid-state reaction method is the most widely used because of its simplicity, cost-effectiveness, and suitability for large-scale production. However, this process generally requires high heating temperatures (above 1300 °C) to achieve complete reactions between the oxide components (MgO, Al_2O_3, and SiO_2) and to form stable α-cordierite.

The formation of cordierite occurs through a series of intermediate phases such as spinel ($MgAl_2O_4$), cristobalite (SiO_2), and μ-cordierite, before the transformation into the stable α-cordierite phase at higher temperatures. Previous studies by El Ouahabi et al. [12], and Maleki et al. [13] have shown that μ-cordierite acts as a metastable intermediate phase between 1250–1350 °C, which subsequently transforms into α-cordierite above 1350 °C.

Despite extensive research, the phase stability, transformation mechanism, and heating behavior of cordierite ceramics derived from pure oxide precursors are still of great scientific interest. Therefore, this study focuses on the synthesis of cordierite ceramics via the solid-state reaction method using analytical grade magnesium oxide (MgO), aluminum oxide (Al_2O_3), and silicic acid ($SiO_2 \cdot nH_2O$) as raw materials. The phase formation and transformation behavior were systematically investigated by X-ray diffraction (XRD) in the temperature range of 1200–1430 °C, with special emphasis on the transition from μ- to α-cordierite.

Methods

High-purity oxide powders were used as starting materials for the synthesis of cordierite ceramics. The raw materials consisted of magnesium oxide (MgO, pure grade), aluminum oxide (Al_2O_3, pure grade), and silicic acid ($SiO_2 \cdot nH_2O$, pure grade). These oxides were selected for their high chemical purity to ensure accurate stoichiometry and minimize the formation of secondary phases during sintering.

The powders were weighed in stoichiometric proportions corresponding to $2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$. The mixture was blended in a ball mill for 3 hours. After milling, the powders were slightly moistened with distilled water for pressing. The resulting mass was pressed into pellets under a pressure of 25 MPa using a hydraulic press.

Pressed samples were fired in an electric furnace at 1200, 1300, 1350, 1400, and 1430 °C for 1 h. The heating rate was 10 $°C\ min^{-1}$.

Phase identification was carried out using a Rigaku MiniFlex 600 diffractometer with Cu-Kα radiation ($\lambda = 1.5406$ Å), operated at 40 kV and 15 mA. Data were collected in the 2θ range of 4–75° at a scanning speed of 2°/min.

Results and Discussion

The XRD patterns of the samples fired at 1200 °C and 1300 °C are presented in Fig. 1 (a) and (b). The results show that the crystalline phases form significantly with increasing firing temperature, indicating the progress of solid-state reactions among MgO, Al_2O_3, and SiO_2 components.

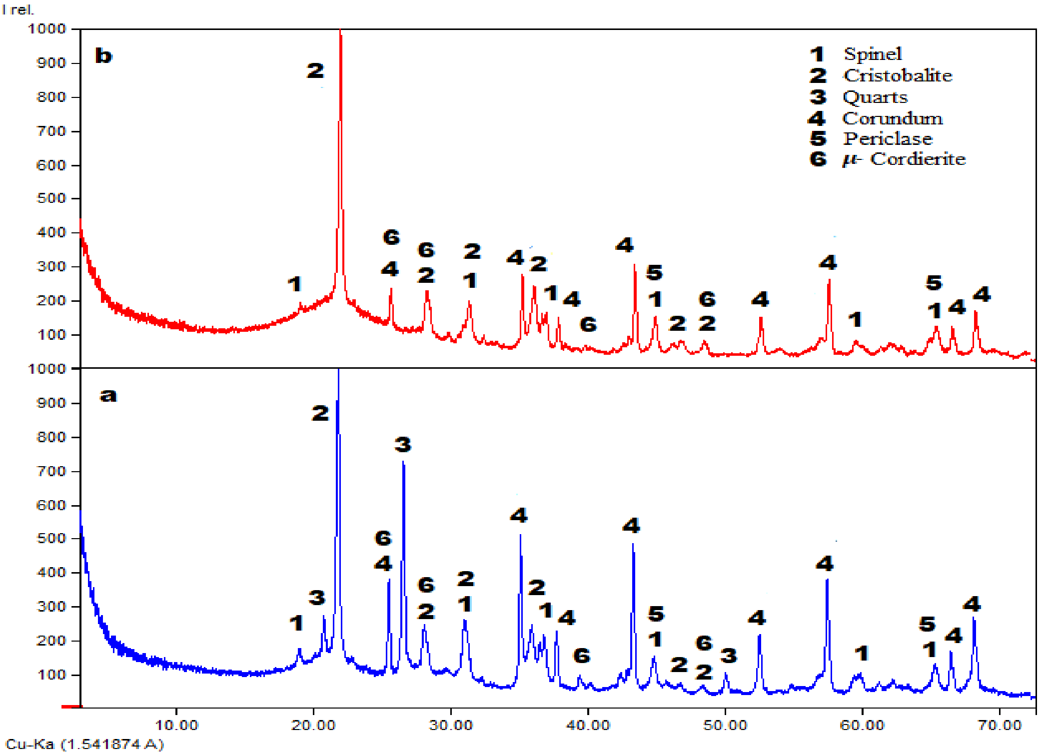

Fig. 1. XRD patterns of samples fired at a) 1200 °C and b) 1300 °C in 1 h.

Fig.1 shows the XRD patterns of the sample fired at a) 1200 °C, the main crystalline phases identified were spinel ($MgAl_2O_4$), cristobalite (SiO_2), and α-corundum (Al_2O_3), along with small amounts of quartz (SiO_2) and periclase (MgO). The spinel phase, corresponding to PDF card No. 21-1152, showed its characteristic diffraction peaks at approximately $2\theta = 31.3°$, 36.9°, 45.0°, 60.0° and 65.5°, which are attributed to the (111), (220), (311), (400), (511), and (440) planes, respectively. Cristobalite (PDF 39-1425) exhibited reflections at about 21.8°, 28.4°, 31.3°, 36.1°, 46.8° and 48.5°, confirming the partial transformation of quartz into the high-temperature SiO_2 polymorph. α-Corundum (PDF 10-0173) peaks appeared at 25.6°, 35.3°, 37.8°, 43.5°, 52.6°, 57.6°, 66.6°, and 68.3°, which correspond to typical diffraction planes of Al_2O_3. The quartz phase (PDF 46-1045) was identified by weak peaks at around 21.0°, 26.6°, and 50.0°, indicating that part of the silica remained unreacted at this stage. Additionally, weak reflections at 42.9°, 45°, 62.3° and 65.5° confirmed the presence of periclase (PDF 45-0946), suggesting a small amount of unreacted MgO. The coexistence of spinel, cristobalite, corundum, and quartz implies that solid-state reactions between the oxide components had begun but were not yet completed at 1200 °C. These phases represent the early intermediates in the cordierite formation process.

Fig. 1 shows the XRD patterns of the sample fired at b) 1300 °C. The phase composition changed significantly. The quartz peaks observed at 26.6° disappeared completely, while spinel, cristobalite, corundum, and a small amount of μ-cordierite were detected. The spinel phase maintained its peaks at approximately 19°, 31.3°, 37°, 45°, 60°, and 65.5°, indicating continued crystallization of $MgAl_2O_4$. Cristobalite displayed sharper and more numerous reflections at 21.8°, 28.4°, 31.3°, 36.2°, 46.8°, and 48.5°, confirming the complete transformation of SiO_2 into its high-temperature modification. Corundum remained stable with peaks at 25.6°, 35.3°, 37.8°, 43.5°, 52.6°, 57.6°, 66.6°, and 68.3°, although their intensity slightly decreased compared to 1200 °C. New weak peaks at around 25.7°, 28.4° and 48.6° correspond to μ-cordierite (PDF 82-1538), indicating the initial formation of cordierite through reactions among spinel, cristobalite, and corundum phases. The disappearance of the 26.6° quartz peak and the appearance of μ-cordierite reflections confirm that SiO_2 has been fully consumed in the solid-state reaction.

The phase formation during sintering can therefore be described by the following sequence: quartz transforms into cristobalite at intermediate temperatures, and then spinel and cristobalite react to form μ-cordierite according to the reaction:

Spinel ($MgAl_2O_4$) + Cristobalite (SiO_2) $\rightarrow$ μ-Cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$). This reaction sequence agrees well with previous studies by Gören et al., El Ouahabi et al., and Maleki et al., who reported that μ-cordierite appears as a metastable intermediate before transforming into α-cordierite at higher temperatures.

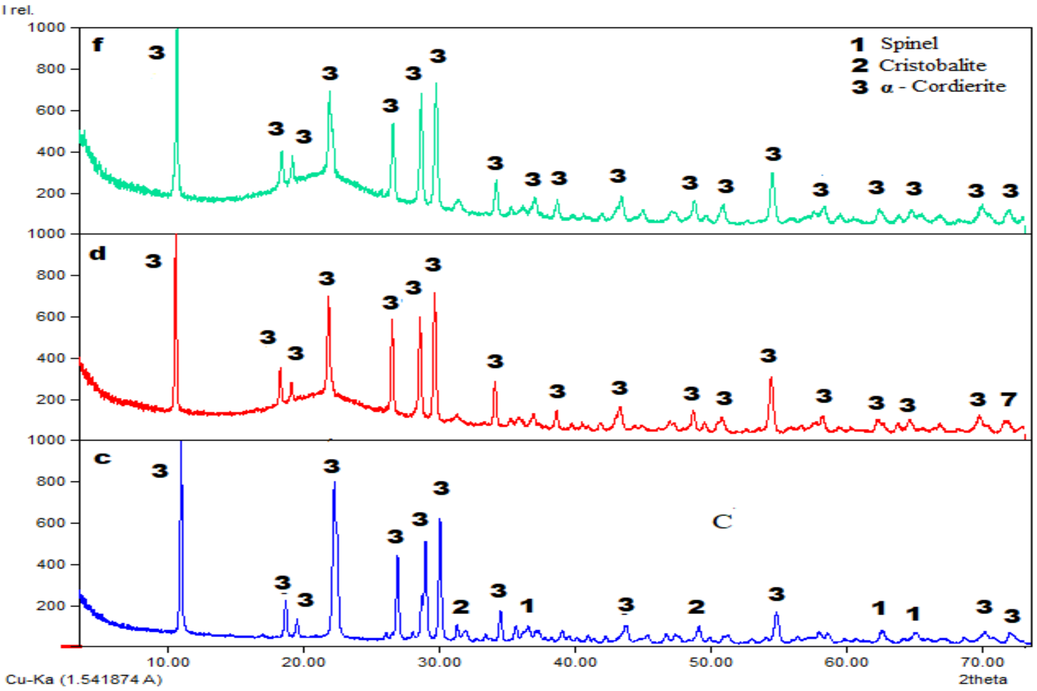

Fig. 2. The XRD patterns of the samples fired at c) 1350 °C d) 1400 °C f) 1430 °C in 1 h

The XRD patterns of the samples fired at 1350 °C, 1400 °C, and 1430 °C are shown in Fig. 2. The results clearly demonstrate the phase formation of the MgO–Al_2O_3–SiO_2 system during sintering, indicating the progressive formation and crystallization of α-cordierite as the temperature increases. At 1350 °C (c), the predominant crystalline phase was identified as α-cordierite, with its characteristic reflections appearing at approximately 2θ = 11°, 18.6°, 19.5°, 22.2°, 26.8°, 28.9°, 30°, 34.4°, 43.7°, 49°, 54.8°, 70.2°, and 72°, which match well with the standard data for α-cordierite (PDF 13-0293). In addition, weak peaks corresponding to spinel ($MgAl_2O_4$) and cristobalite (SiO_2) were also observed at 31.3°, 36.9°, 45°, 60°, 65.5° and 28.4°, 31.3°, 36.2°, 46.8°, 48.5°, respectively. The coexistence of these minor phases suggests that the solid-state reaction was nearly completed but some unreacted intermediates still remained at this temperature.

Fig.2 shows the XRD pattern of the sample fired at d)1400 °C. The pattern exhibited only α-cordierite peaks, indicating a fully developed crystalline structure. The characteristic reflections appeared at 2θ = 10.5°, 18.0°, 20.9°, 26.6°, 31.3°, 34.9°, 36.8°, 43.5°, 45.4°, 56.0°, 62.0°, and 71.0°, confirming the formation of stable and well-crystallized α-cordierite. The disappearance of spinel and cristobalite reflections confirms that the solid-state reaction has been completely accomplished through the transformation:

Spinel + Cristobalite → α-Cordierite.

Fig.2 shows the XRD pattern of the sample fired at f) 1430 °C, which exhibited only strong and sharp peaks corresponding to α-cordierite, with no evidence of residual or secondary phases such as spinel, cristobalite, or periclase. The increased peak sharpness and reduced background intensity indicate complete crystallization and thermal stability of the α-cordierite phase at this temperature.

The formation of α-cordierite begins at 1350 °C, becomes dominant and fully crystallized at 1400 °C, and remains thermally stable at 1430 °C, confirming the completion of the solid-state reaction.

Fig. 2. XRD patterns of samples fired at 1350 °C (c), 1400 °C (d), and 1430 °C (f). The stabilization of α-cordierite peaks at higher temperatures (1400–1430 °C) indicates the complete crystallization and phase stability of the ceramic material.

Conclusions

Cordierite ceramics with the stoichiometric composition $2MgO·2Al_2O_3·5SiO_2$ were successfully synthesized by the solid-state reaction method using high-purity oxide powders. The XRD results showed that the synthesized material sintered at 1430 °C for 1 h is formed of the cordierite phase only.

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