Study of optical constants and dielectric properties of nanocrystalline α-cordierite ceramic

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ABSTRACT

The nanocrystalline 2Mg2Al2O5.5SiO2 as amorphous-type α-cordierite was prepared by the modified sol-gel (or Pechini) technique. After calcination at 800°C, the pressed powder as disk pellets was sintered at 1150°C, 1250°C, and 1350°C. The structural information of α-cordierite was obtained by X-ray diffraction (XRD), scanning electron microscopy (SEM), micro-Raman scattering spectra, and Fourier transform infrared (FTIR) spectra. The XRD and Raman spectra showed that the nanocrystalline α-cordierite phases from pellets of glass-ceramic powders appeared when the temperature increased. The SEM results confirmed the rounded shape of the nanocrystalline α-cordierite. Using Kramers–Kronig (KK) method, the FTIR spectroscopy was evaluated. The increasing synthesized temperature increased the size of nanocrystalline. Furthermore, by increasing the synthesized temperatures, the optical constants of nanocrystalline, both real and imaginary parts of the refractive index and dielectric function decreased. Consequently, the transversal optical (TO) and longitudinal optical (LO) phonon frequencies shifted to red frequencies by increasing reaction temperature.

1. Introduction

The nanocrystalline α-cordierite – one of the glass ceramics phases – is a suitable raw material for industrial and electrical applications due to low dielectric constants in the range of 5.5–7.5, low dielectric losses in the range of 0.015–0.025, and low coefficients of thermal expansion in the range of 1.22–4.32 × 10⁻⁶ K⁻¹ against high chemical and thermal stability [1,2]. Spinel, mullite, sapphire, corundum, and cristobalite are some of the common phases of glass ceramics that were observed with the nanocrystalline α-cordierite phase by different experimental conditions.

The nanocrystalline α-cordierite has the highest melting points among glass ceramics (about 1460°C) and has three polymorphic forms: α-cordierite stable in high temperature with hexagonal structure, β-cordierite stable in low temperature with orthorhombic structure, and µ-cordierite that it is a metastable form [3,4].

There are several methods for synthesizing nanocrystalline α-cordierites such as solid state reaction [5], sol-gel [6], spray pyrolysis [7], co-precipitation method [8], and combustion synthesis method with flame or without flame [9]. The Pechini method is similar to sol-gel technique with some advantages such as being economic and affordable, utility, control of stoichiometry, low processing temperature, and high degree of homogeneity.

In order to accurate explanation of the phenomena, optical theory is important by means of some methodologies. One of the essential methodologies is obtaining the physical quantities, such as complex dielectric constant or complex refractive index of materials from the recorded spectra. Due to obtain these, least-squares refinement calculations, ellipsometry and/or Kramers-Kronig analysis are benefit in the IR region [10]. Among those methods, Kramers–Kronig analysis has been widely used to determine the complex refractive index of materials in the IR region, even though it has some difficulties to record data of samples. However, to our best knowledge, limited attention has been devoted to nanocrystalline α-cordierite based on optical constant and dielectric coefficient by this method.

Herein, the nanocrystalline α-cordierite was produced by Pechini method that investigated the effect of different synthesized temperatures. Accordingly, the nanocrystalline α-cordierite is characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) imaging, micro-Raman scattering, and Fourier transform infrared (FTIR) spectroscopy. Consequently, this study reported the complex refractive index, the complex dielectric constant, the frequency of transverse optical phonon (TO), and the longitudinal optical (LO) phonons of nanocrystalline α-cordierite in IR wave numbers (800–1150 cm⁻¹), based on the different synthesized temperatures via Kramers–Kronig (KK) method.
2. Experimental procedures

The starting raw materials used for synthesizing nanocrystalline α-cordierite gel were magnesium nitrate (Mg(NO$_3$)$_2$.6H$_2$O, 99.0%, Merck), aluminum nitrate (Al(NO$_3$)$_3$.9H$_2$O, 99.0%, Merck), silica (SiO$_2$, 99.99%, Merck), and ethylene glycol (EG, 99.5%, Merck), and citric acid (CA, 99.0%, Sigma) was used for chelating metallic ions. For preparing 10 g of nanocrystalline α-cordierite in this method, we needed 7.3 g Mg(NO$_3$)$_2$.6H$_2$O, 25.6 g of Al(NO$_3$)$_3$.9H$_2$O, 5 g of SiO$_2$, 6.5 g of CA, and 20cc EG.

First, magnesium nitrate was dissolved in deionized water with steady stirring. Following that, aluminum nitrate was added gently since the previous solution is stirring. Then, CA and SiO$_2$ were added to be dissolved in it completely. After adding EG and forming the clear solution, its temperature increased steadily within 2 h at 100°C until the resin was formed. Further calcinations were utilized at 800°C for several hours. For producing fine particles, the calcined powder was milled for 10 h. The pellet-shaped compacts were sintered at 1150ºC, 1250ºC, and 1350ºC.

In this method, the chelating agent (here CA) chelates metal ions. Then, a polyhydroxy alcohol (such as EG) is added and heated to form a polymeric resin. The heating process causes the breakdown of the polymer. The synthesized process is used for several hours for breaking down polymeric resin to obtain the nanocrystalline material. We could produce the final product which remained in high density and purity with other properties of nanocrystalline α-cordierites.

3. Results and discussion

3.1. XRD analysis

The XRD spectra of the synthesized samples are reported by an Xpert X-ray diffractometer model 1480 (Phillips, The Netherlands). The XRD spectra of the nanocrystalline α-cordierite synthesized at 1150ºC, 1250ºC, and 1350ºC for 2 h are shown in Figure 1.

It is clear in the XRD pattern of nanocrystalline α-cordierite, 10 observed diffraction peaks at 10.35º, 18.36º, 19.11º, 21.63º, 26.38º, 28.37º, 29.58º, 33.75º, 38.45º, and 43.12º referred to 10 hkl miller planes of cordierite corresponded to (100), (110), (002), (102), (112), (202), (211), (212), (310), and (312), respectively [5]. The crystalline phase represented in the sample is assigned to α-cordierite (JCPDS card no. 84–1222). Furthermore, there are two peaks near to 31º and 39º that are related to corundum phase of ceramics [11]. Although increasing the temperature causes to increase the intensity of peaks, the broadening of peaks decreased. If the temperature was increased, the extra phases of glass ceramic would be omitted and just the nanocrystalline α-cordierite phase would remain. Moreover, by increasing the temperature, some peaks become stronger and sharper. Following the increase in temperature, the widths of the X-ray diffraction peaks start to decrease approaching the single crystal spectrum and nanostructure of α-cordierite becomes more regular than the previous states. Usually, a single broad peak characteristic of the XRD points to an amorphous type of material. Also, the sharp XRD peaks related to crystallite or nanocrystallite of materials [12]. The crystallite size ($D_{hkl}$) of the identified crystalline phase is calculated using corrected Debye-Scherrer formula as:

$$D_{hkl} = \frac{K\lambda}{\beta \cos \theta}$$  (3.1)

where $\beta' (= B - b)$ is the FWHM (full width at half maximum), $B$ is the line width, $b$ is the instrumental broadening (here is 0.125), $K$ is the shape factor (= 0.9), $\theta$ is the Bragg angle, and $\lambda$ is the wavelength of CuKα$_1$ (1.54056 Å). However, the size of nanocrystalline α-cordierite was calculated and it was found to be in the range of 30–80 nm (Table 1).

3.2. SEM imaging analysis

The SEM images of the thin layer of gold coated samples are provided under a SEM Seron Technology-AIS2100. The SEM was used to investigate the morphology of the nanocrystalline α-cordierite under different synthesized temperatures. The SEM images of the synthesized samples are presented in Figure 2. It demonstrated that the size of nanocrystalline powders formed were almost a rounded shape in the range of 20–150 nm. The average size of nanocrystalline α-cordierite is 100, 70, and 40 nm for 1350°C, 1250°C, and 1150°C synthesized temperature, respectively. The XRD analysis also confirmed the obtained results. Furthermore, the uniform distribution and the number of grains of the nanocrystalline powders
were more observable by increasing the synthesized temperature.

The size distribution histogram of the α-cordierite nanocrystalline powder is also presented in Figure 3. Besides, it was found that more than 80% of nanocrystalline α-cordierite is in the size range from 60 to 120 nm, 30 to 50 nm, and 20 to 40 nm at 1350°C, 1250°C, and 1150°C synthesized temperatures, respectively.

3.3. Micro-Raman scattering spectra analysis

Raman spectra of the nanocrystalline α-cordierite were collected by using a Thermo Nicolet Almega dispersive micro-Raman scattering spectrometer operating by a 532 nm laser line as the second harmonic of an Nd:YLF laser.

The Raman spectrum of synthesized nanocrystalline α-cordierite and its thermal treatment is illustrated in Figure 4. The Raman spectra reveal the presence of broad band vibrations due to the deformation of cyclosilicate rings (bands between

| Angel (2θ) (degree) | FWHM (β) (Rad.) | D (nm)* | Temperature (ºC) |
|--------------------|-----------------|--------|------------------|
| 10.34              | 0.243           | 66.05  | 1350             |
| 18.04              | 0.227           | 76.87  |                  |
| 21.65              | 0.230           | 74.96  |                  |
| 26.31              | 0.236           | 71.29  |                  |
| 28.38              | 0.244           | 66.92  |                  |
| 29.41              | 0.281           | 51.72  |                  |
| 33.86              | 0.233           | 63.28  |                  |
| 10.36              | 0.244           | 65.24  | 1250             |
| 18.05              | 0.229           | 75.35  |                  |
| 21.65              | 0.230           | 74.93  |                  |
| 26.28              | 0.240           | 69.20  |                  |
| 28.35              | 0.250           | 64.01  |                  |
| 29.38              | 0.288           | 49.59  |                  |
| 33.83              | 0.264           | 58.31  |                  |
| 10.31              | 0.268           | 54.47  | 1150             |
| 18.02              | 0.235           | 71.35  |                  |
| 21.63              | 0.239           | 69.16  |                  |
| 26.29              | 0.257           | 60.63  |                  |
| 28.35              | 0.267           | 56.62  |                  |
| 29.37              | 0.305           | 44.91  |                  |
| 33.82              | 0.273           | 54.96  |                  |
| 54.22              | 0.377           | 35.06  |                  |

* Use Equation (3.1).
550 cm$^{-1}$ and 750 cm$^{-1}$), and by raising the temperature, the number of these bands increased [13,14]. It seems that by increasing temperature, the peaks become sharper. The bands around 950−1200 cm$^{-1}$ are related to antisymmetric stretching Si-O band [15,16]. The other bands correspond to the vibration of Al-O (615, 870, 925 cm$^{-1}$) and Mg-O (682 cm$^{-1}$) [17]. It is obvious that increasing temperature in synthesized processes affects the Raman spectra. According to common Raman spectra, by increasing the synthesized temperature, the intensity of peaks increased and the peaks became sharper.

3.4. FTIR analysis

The reported FT-IR spectra of the synthesized samples are presented in Figure 5. The IR active vibrational modes can be easily revealed. The peaks at 853 and 1179 cm$^{-1}$ corresponded to Si-O and AlO$_4$ tetrahedral bonds, respectively [18,19]. On the other hand, the peaks at 1080−1088 cm$^{-1}$ relied on the existence of Si-O bands of amorphous SiO$_2$ powder. These peaks shift to the lower values showing the formation of Si-O-Al or Si-O-Mg bonds in the structure of nanocrystalline α-cordierite [18,20,21]. The reaction between the spinel phase and the absorption bands of MgAl$_2$O$_4$ would have appeared the new band at about 750 cm$^{-1}$ [22–24]. The two peaks at about 525 cm$^{-1}$ are assigned to MgO$_6$ octahedral bands [18]. The stretching modes of TiO$_2$ are at about 455 cm$^{-1}$ [25]. If the synthesized temperature increased, all the observed peaks would be sharper in intensity.

3.5. Optical properties characterizations

The aim of this section is to derive the spectral functions for the complex refractive index and the complex dielectric coefficient. By putting emphasis on the IR spectroscopy using KK method, our study can give insights into the nature of phase transitions and the optical phonon modes.

3.5.1. Theory of KK method

The optical constants of the synthesized nanocrystalline α-cordierite in 1150ºC, 1250ºC, and 1350ºC were assessed by the KK method using FTIR spectra data. The ratio intensity of the light transmitted through the sample ($I$) to the incident light intensity ($I_0$) through the sample has discussed the transmittance ($T$) as a function of the wavenumber. By using the percent transmittance ($T\% = 100 \cdot T$), the recorded absorption ($A(\nu)$) and the reflectance ($R(\nu)$) can be calculated the complex refractive index $N$ at particular wavenumber. The complex refractive index $N$ is discussed as:

$$N(\nu) = n(\nu) + ik(\nu),$$

where $n(\nu)$ and $k(\nu)$ are the real and imaginary parts of the refractive index, respectively, and $\phi(\nu)$ is the phase change for a frequency $\nu$ between the incident signal and reflected signal can be evaluated by KK dispersion relation explained by Prathap and his coworkers [26] or by Maclaurin’s method [27] as follows:

$$n(\nu) = \frac{1 - R(\nu)}{1 + R(\nu) - 2 \sqrt{R(\nu)} \cos \phi(\nu)}$$

$$k(\nu) = \frac{-2 \sqrt{R(\nu)} \sin \phi(\nu)}{1 + R(\nu) - 2 \sqrt{R(\nu)} \cos \phi(\nu)}$$

Since the dielectric function is the square of the refractive index, both real and imaginary parts of dielectric function, ($\varepsilon, \varepsilon'$), can be derived by following [28]:

Figure 4. Raman spectra of the synthesized α-cordierite synthesized at (a) 1150°C, (b) 1250°C, and (c) 1350°C.

Figure 5. FTIR spectra of α-cordierite synthesized at (a) 1350°C, (b) 1250°C, and (c) 1150°C.
\[
\varepsilon(u) = \varepsilon'(u) + i\varepsilon''(u)
\]

where

\[
\begin{align*}
\varepsilon'(u) &= n^2(u) - k^2(u) \\
\varepsilon''(u) &= 2n(u)k(u)
\end{align*}
\]

(3.4)

3.5.2. Optical phonon mode

In the KK method, one of the factors that can be deduced are the optical phonon modes related to the optical interactions with the lattice. Generally, these interactions assigne assigned to optical characteristics – the TO and LO resonances. One way to estimate LO and TO modes is the detection of two crossed points in the extinction and refraction traces for n and k graphs \[29\]. The observed lower and higher wavenumbers of these two interaction points refer to TO and LO optical modes, respectively.

Besides, the intermediate wavenumber of the positive to negative and negative to positive real dielectric function is assigned to TO and LO frequency modes, respectively \[27\]. Furthermore, the maximum position of \(\varepsilon''\) and \(\text{Im}\{-1/(\varepsilon = \varepsilon' + i\varepsilon'')\}\) curves is TO and LO modes, respectively \[30,31\]. Herein, the calculated LO and TO modes are constant by both methods.

3.5.3. Optical coefficients

Figure 6 shows real and imaginary parts of the complex refractive index n and k, as optical coefficients of nanocrystalline \(\alpha\)-cordierite for different synthesized temperatures. Considering the pictures earlier, increasing synthesized temperatures causes the decrease in the dependence of optical coefficients to wavenumber for nanocrystalline \(\alpha\)-cordierite. It is clear that the optical coefficients in highest temperature tend to be steadier than in the temperatures of 1150°C or 1250°C. By increasing synthesized thermal treatment temperature for nanocrystalline \(\alpha\)-cordierite, both the refractive and extinction indices decreased. The fundamental band-gap absorption led to decreasing the refractive index and extinction coefficient \[32\]. Similar results in different ranges of wavenumber were also obtained by other researchers \[33,34\].

The real and imaginary parts of dielectric coefficients of nanocrystalline \(\alpha\)-cordierite were observed via a function of wavenumber for different synthesized temperatures at 1150°C, 1250°C, and 1350°C in Figure 7. There is a relationship between the refraction index and dielectric coefficient of nanocrystalline \(\alpha\)-cordierite obtained from the fitted value of \(\varepsilon_n = 1.527\) in the visible region as confirmed by the measurements conducted by other researchers \[33,34\].

In Figure 7, \(\varepsilon'\) and \(\varepsilon''\) shown in the range of 800–1150 cm\(^{-1}\) are almost flat in the long wavenumber region. It is evident that increasing the synthesized...
temperature causes to decrease both $\varepsilon'$ and $\varepsilon''$ of dielectric function of nanocrystalline $\alpha$-cordierite. The maximum position of $\text{Im}\{1/(\varepsilon = \varepsilon' + i\varepsilon'')\}$ curve is LO mode (Figure 8). As mentioned, TO and LO frequencies were obtained by Figures 6 and 8 tabulated in Table 2. The red shifts were visible as the results of increasing

Figure 7. The real (solid line) and imaginary (dash line) parts of dielectric coefficients of $\alpha$-cordierite synthesized at (a) 1150°C, (b) 1250°C, and (c) 1350°C.

Figure 8. The $\text{Im}\{1/\varepsilon\}$ of $\alpha$-cordierite synthesized at (a) 1150°C, (b) 1250°C, and (c) 1350°C.
the synthesized temperatures. Finally, the high level of purity of nanocrystalline α-cordierite phase is related to the low dielectric constant [34].

4. Conclusion

Nanocrystalline α-cordierite is synthesized by Pechini method using different synthesized temperatures. The synthesized sample was characterized via XRD diffraction, SEM imaging, micro-Raman scattering, and FTIR spectroscopy. The Debye-Scherrer nanocrystalline size calculated through the X-ray diffraction patterns agreed with the SEM results. The micro-Raman scattering and FTIR spectra used to assign the nanocrystalline α-cordierite phase of ceramics were more visible by increasing the synthesized temperature. Furthermore, the optical constants of nanocrystalline α-cordierite were evaluated by the KK method using FTIR transmission spectra data in different synthesized temperatures. By increasing the synthesized temperatures, the following results could be attained:

- The size of nanocrystalline α-cordierite increased in the range of 40 to 100 nm.
- The structure of nanocrystalline α-cordierite was more regular, and the intensity of Raman, FTIR, or XRD peaks increased.
- The number of nanocrystalline α-cordierite increased.
- Both real/imaginary parts of the refractive index and dielectric function decreased.
- The resonance frequency of samples (TO and LO) experienced red shifts by increasing the synthesized temperatures.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Table 2. Longitudinal and transverse optical phonon modes (LO and TO) of α-cordierite treated at different temperatures.

| Temperature [ºC] | Transvers optical phonon (TO) [cm⁻¹] | Longitudinal optical phonon (LO) [cm⁻¹] |
|------------------|-------------------------------------|----------------------------------------|
| 1150             | 1064                                | 1046                                   |
| 1250             | 957                                 | 941                                    |
| 1350             | 899                                 | 870                                    |
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