Synthesis And Characterization Of Al_2TiO_5 By Sol-Gel Method

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Abstract

Al_2TiO_5 has been obtained by the sol-gel technique. Aluminum nitrate and titanium isopropoxide in required concentrations will dissolved in ethanol with citric acid as a nucleating reagent. The mixtures were reflux for 2h under continuous heating and stirring. The precursor obtained was then sintered in air at 1050 °C for 2h. The sintered products were characterized by X-ray diffractometer and Electrochemical Impedance Spectroscopy. To the highest room temperature is 4.02×10^{-9} S cm^{-1}, which is exhibited by the sample containing 0.08 moles of aluminum nitrate and 0.02 moles of titanium isopropoxide.

Keywords: aluminum titanate, conductivity, xrd

1. INTRODUCTION

Initial interest in aluminum titanate, Al_2TiO_5 was due to its low thermal expansion coefficient (typically of the order of 1 – 2 x 10^{-6} K^{-1}), but further research was soon discouraged following the discovery of the expansion anisotropy, leading to extensive microcracking during cooling, and the instability of the compound over the temperature range of ~800-1200 °C. Aluminum titanate belongs to the pseudobrookite family of compounds with the general formula A_2BO_5, represented by the homologous Fe_2TiO_5. In the Al_2TiO_5 structure, each Al^{3+} or Ti^{4+} cation is surrounded by six oxygen ions forming distorted oxygen octahedra. These AlO_6 and TiO_6 octahedra form (001) oriented double chains weakly bonded by shared edges. Such structure is resposible for the strong thermal anisotropy, which creates a complicated system of localised internal stresses during cooling from the firing temperature. These stresses can, and frequently do, exceed the intrinsic fracture strength of the material, resulting in severe microcracking. This microcracking is responsible for the mechanical weakness of the ceramic and explains the quoted low thermal expansion coefficient. These microcracks also contributed to a low thermal conductivity and an excellent thermal shock resistance [1].

2. EXPERIMENTAL

2.1 Sample Preparation

The method of preparation employed in the present study is similar to that reported by Sobhani et al [2]. The raw materials for synthesis were aluminum nitrate ($Al(NO_3)_3.9H_2O$), titanium isopropoxide ($C_{12}H_{28}O_4Ti$), ethanol, and citric acid. x mole of aluminum nitrate was dissolved in 40 cm^3 ethanol where $x = 0.01$, 0.02, 0.04, 0.06, 0.08 and 0.09 and then (0.1-x mole) titanium isopropoxide was added dropwise under magnetic stirring at room temperature (sol 1). After 10 minutes of stirring, citric acid (0.1-x mole) was introduced into the sol 1, and was refluxed under magnetic stirring at 80 °C. After 2 hours, a gel was formed (sol 2). The honey-like sol 2 was dried on a hot plate. After drying, the mixed powder was placed in a crucible and sintered in air at 1050 °C for 2h in an electrical furnace in order to release volatile products coming from the starting materials and then cooled overnight. The resulting powder was then examined using X-ray diffraction (XRD) to confirm the formation of Al_2TiO_5. The sintered samples obtained from the final step were then pelletized at 500 bars.

2.2 Characterizations

2.2.1 Electrochemical Impedance Spectroscopy (EIS)

HIOKI 3531 Z-HiTester electrochemical impedance spectroscopy was used to measure the impedance of the pelletized sample. The thickness of the pellets was measured using a micrometer gauge. The sample holder was kept in a furnace and the conductivity measurements were carried out from 25 °C to 120 °C. Frequency ranging from 50 Hz to 1 MHz was used. The conductivity of the pellets was calculated using the equation below:

$$\sigma = \frac{t}{R_b A} \tag{1}$$

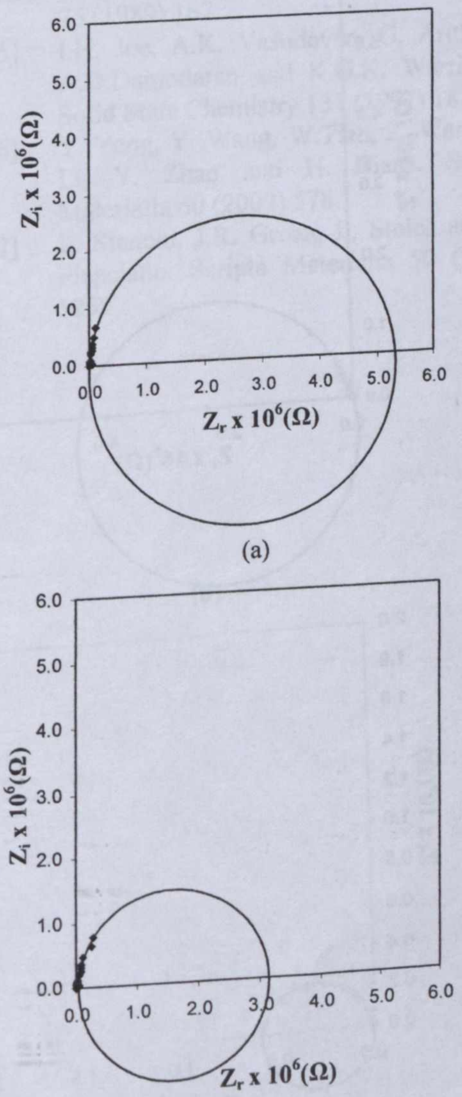
Here t is thickness of sample, A is area of contact between electrode and sample and R_b is bulk impedance of the sample.

2.2.2 X-Ray Diffractometer (XRD)

The identifications of the sintered samples were carried out using Siemens D5000 diffractometer. Scans were taken over the 2θ angular range of 5 to 80° with a step size of 0.05°. The peak positions were watched with JCPDS 841641.

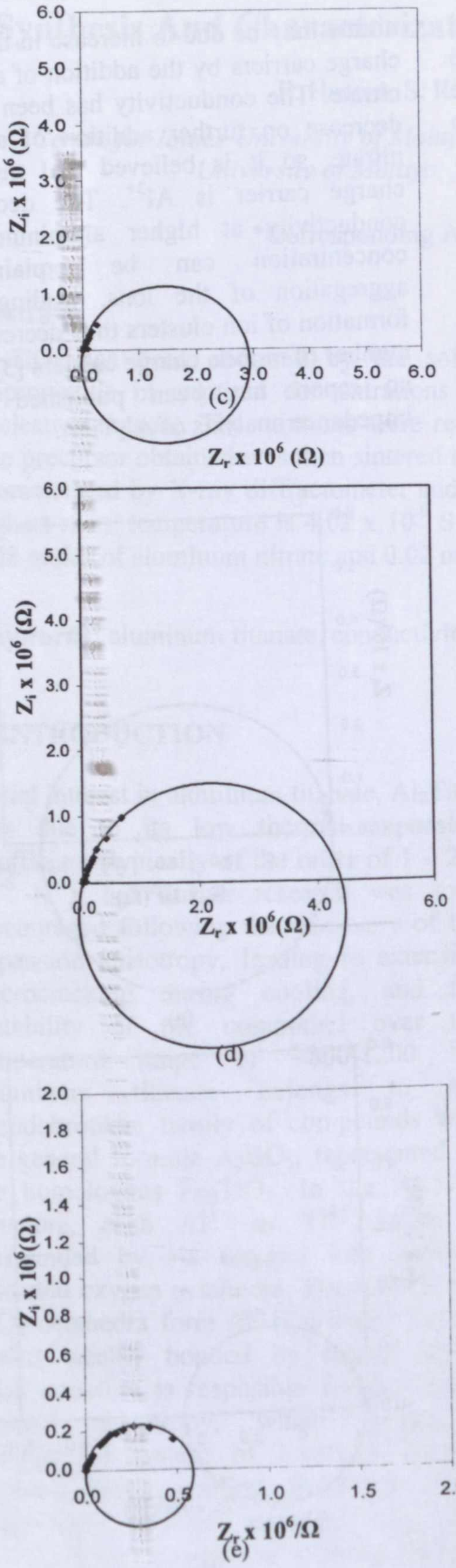
3. RESULTS AND DISCUSSION

Fig. 1 shows the Cole-Cole plot at room temperature of Al_2TiO_5 for different number of mole. From Fig. 2, it is obvious that 0.08 mole of $Al(NO_3)_3.9H_2O$ has the highest conductivity at room temperature which is 4.02×10^{-9} S cm^{-1}. The conductivity seems to increase with increase in mole concentration of aluminum nitrate from 0.01 to 0.08 moles. The increase in the conductivity with increasing aluminum nitrate may be due to increase in the mobile charge carriers by the addition of aluminum nitrate. The conductivity has been found to decrease on further addition of aluminum nitrate, so it is believed that the mobile charge carrier is Al^{3+}. The decrease in conductivity at higher aluminum nitrate concentration can be explained by aggregation of the ions, leading to the formation of ion clusters thus decreasing the number of mobile charge carriers [3]. So far, no report has been published on the impedance analysis of Al_2TiO_5.



(a)

(b)



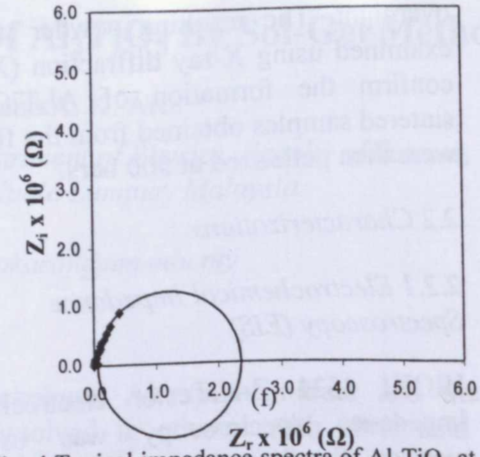


Fig. 4 Typical impedance spectra of Al_2TiO_5 at 25 °C (a) $0.01Al_2O_3$-$0.09TiO_2$, (b) $0.02Al_2O_3$-$0.08TiO_2$, (c) $0.04Al_2O_3$-$0.06TiO_2$, (d) $0.06Al_2O_3$-$0.04TiO_2$, (e) $0.08Al_2O_3$-$0.02TiO_2$ and (f) $0.09Al_2O_3$-$0.01TiO$

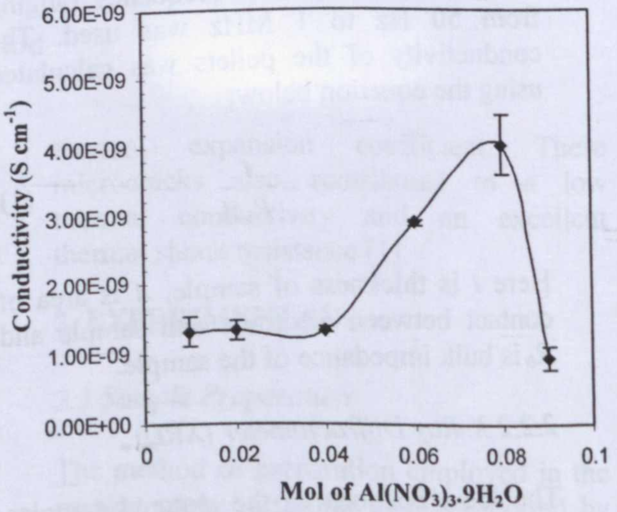


Fig. 2 The plot of conductivity versus aluminum nitrate content (in moles) at room temperature sintered at 1050 °C (2h)

Fig. 3 presents the XRD patterns of the Al_2TiO_5 glass-ceramics sample at 1050 °C with different moles of aluminum nitrate. After the sintering runs, the XRD patterns show complete formation of Al_2TiO_5 with peaks at, 2θ =35.15°, 36.15° [4], 27.5° [5], 37.8°, 52.6°, 54.45°, 57.55° [2], 25.6°, 41.3° [6], 43.4°, 64.05°, 66.55°, 68.3° and 77.2° [7].

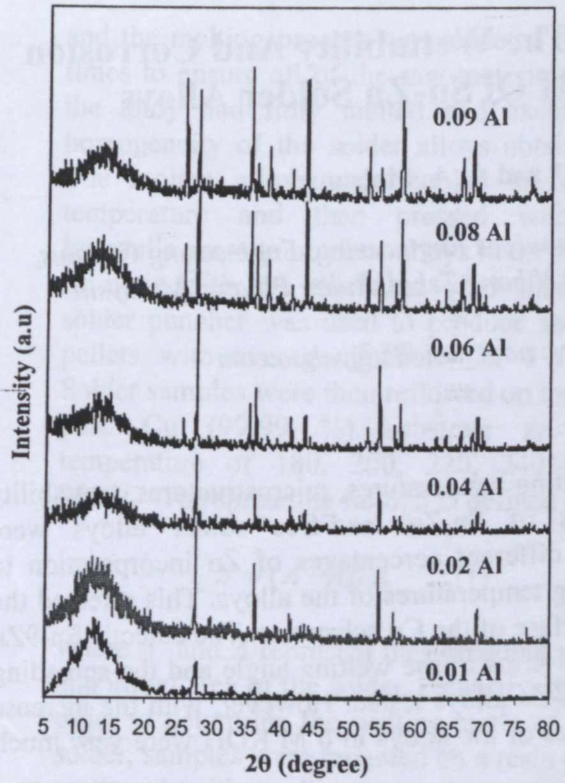


Fig. 3 The XRD pattern of 0.08 mol of Al_2O_3-0.02 mol of TiO_2 system at sintering temperature of 1050 °C for two hours

4. CONCLUSIONS

The Al_2TiO_5 compound was successfully prepared by the sol-gel method. The highest conductivity obtained for this work is 4.02×10^{-9} S cm^{-1} for sample synthesized using 0.08 mole Al_2O_3 and 0.02 mole TiO_2. Corresponding peaks for Al_2TiO_5 are shown from X-ray diffactogram.

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REFERENCES

[1] A.M. Seagades, M.R. Morelli and R.G.A. Kiminami. J. European Ceramic Society (1997)

[2] M. Sobhani, H.R. Rezaie and R. Naghizadeh. J. Mater. Process. Tech. (2008)doi:10.1016/j.jmatprotec.2007.12.023

[3] C.S. Ramya, S. Selvasekarapandian, T. Savitha, G. Hirankumar, R. Baskaran, M.S. Bhuvaneswari and P.C. Angelo. European Polymer Journal 42 (2006) 2672

[4] F. Remy, O. Monnereau, A. Casalot, F. Dahan and J. Galy. J. Solid State Chem. 76 (1989) 167

[5] I.H. Joe, A.K. Vasudevan, G. Aruldhas, A.D.Damodaran and K.G.K. Warrier. J. Solid State Chemistry 131 (1997) 181

[6] Y. Yang, Y. Wang, W.Tian, Z. Wang, C. Li, Y. Zhao and H. Biana. Scripta Materialia 60 (2009) 578

[7] L. Stanciu, J.R. Groza, L. Stoica and C. Plapcianu. Scripta Materialia 50 (2004) 1259