Sol – gel Synthesis and Characterization of Sodium beta Alumina Powders

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1. INTRODUCTION

Sodium-ion conductivity is a well-known feature of sodium beta alumina ceramics. Sodium beta-alumina electrolyte has been widely employed in large-scale energy-storage systems, such as ZEBRA batteries [1] and high-temperature Na–S [2], due to its strong sodium ion conductivity and outstanding thermal characteristics. The compounds with the formula $Na_2OnAl_2O_3$, where n is typically between 5 and 11, are commonly recognised as sodium beta-alumina. These compounds have high sodium mobility along the conductive planes due to their layered structure. Based on the changeable ratio of sodium to aluminium, there are two structural variations for beta-alumina: β-Al_2O_3 is a rhombo hedral structure, and β''-Al_2O_3 is a hexagonal structure with a stoichiometry of $Na_2O_{(8-11)}Al_2O_3$ and $Na_2O_{(5-7)}Al_2O_3$, respectively[3,4]. The two crystal structures consist of conductive planes stacked in various directions and spinel blocks. The two differ in that β''-Al_2O_3 has two conducting planes and three spinel blocks, while β -Al_2O_3 has one conductive plane and two spinel blocks. Because of its more conductive planes and higher sodium content in conductive planes, the β'' -Al_2O_3 phase has 3-5 times stronger ionic conductivity than b-Al_2O_3 [5-7]. Consequently, a high β'' -Al_2O_3 content is preferred in beta-alumina electrolyte applications in energy-storage devices. The homogeneous microstructure, densification, β'' -Al_2O_3 content, and other factors are among the many variables that regulate the electrical properties of beta-alumina electrolyte [1, 8–10]. However, high content β'' -Al_2O_3 is challenging to synthesize from because of its poor thermodynamic stability. It is usually impossible to avoid unwanted concomitant phases like b-Al_2O_3 and $NaAlO_2$ (around the boundaries) for the standard solid-state reaction synthesis. Suitable stabilizers such as Li_2O, MgO, or their mixture were often used to raise the β'' -Al_2O_3 concentration [11–13].

Zhu et al. observed that the β'' -phase percentage of beta-alumina sinter stabilized by Li_2CO_3 additive was 88 wt%, in contrast to the 5 wt% of beta-alumina sinter obtained without the addition of stabilizer [7, 14]. This proportion is quite high. By encouraging densification and raising the proportion of β'' -Al_2O_3, a small quantity of magnesium oxide stabilizer can aid to improve the electrical performance of the beta-alumina electrolyte, according to Chen et al. [15]. It has also been demonstrated that the addition of other stabilizers, such as TiO_2 [16], NiO [17], Y_2O_3 [18], and their composites [18, 19], can raise the β'' -Al_2O_3 content and enhance the beta-alumina electrolyte's electrical properties.

Moreover, it was found that the β''-Al_2O_3 content of the beta-alumina electrolyte was significantly impacted by the utilization of alumina sources [20]. Alumina sources such as bayerite, pseudo-boehmite, c-alumina, and a-alumina are commonly used to manufacture beta-alumina electrolyte. Barison et al. [21] conducted a thorough investigation into the impacts of various alumina sources on the β'' -Al_2O_3 concentration and densification for beta-alumina electrolyte, as seen in Table 1. It was shown that c-alumina was superior for obtaining higher content β'' -Al_2O_3, even though alpha-alumina assisted in achieving higher densification.

In this work, boehmite was used as an alumina source to prepare highly ionic-conductive sodium beta alumina electrolytes. We looked into how the Na_2O content affected the volume density, microstructure, β'' -Al_2O_3 content, and the electrical characteristics that resulted.

Keywords : Sol-gel,low temperature synthesis, impedance analysis

2. SYNTHESIS PROCEDURE

Sodium beta-alumina powder stabilised by magnesium oxide, with 77.3 mol% Al_2O_3, 12.7 mol% Na_2O, and 10.0 mol% Using the sol-gel combustion process, MgO was created. Analytically pure aluminium nitrate, magnesium nitrate, sodium nitrate, and citric acid were the starting components. A minimum of distilled water was used to dissolve the appropriate proportions of the aforementioned materials. The molar ratio of metal nitrates to citric acid was 1:1. As water was evaporated by heating, the solution formed an extremely thick brown gel. The gel ignited when the temperature was raised to roughly 220^0C. A loose powder was formed by the self-propagating combustion of the dry gel. The burned powder underwent air calcination at 700 ◦C to produce β''-alumina.

3. Material characterization

To characterise the synthesised ZnO-TiO_2 nano composites, a Bruker powder diffractometer (Shimadzu-7000) equipped with monochromatized Cu-K (1.5406) radiation was utilised. Using the Scherer formula, the nano composites' crystal size was ascertained. A Zeiss electron microscope was used to perform scanning electron microscopy (SEM), a method for examining the morphology of nano composites. Impedance analysis was used to examine electrical properties.

4. Characterization Results

By using SEM and EDAX, the morphologies and composition of the powder were examined. Shafts are porous or hollow, as demonstrated by a closure study. An aggregation of sphere-shaped particles makes up the calcined powder. EDS elemental analysis that corresponds to it. The beta-alumina electrolyte sinter findings that were synthesised were shown in the insets of Figure 1.

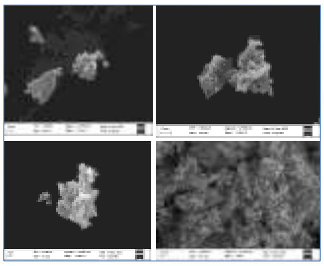

Figure 1: SEM Images of Sodium beta Alumina Beta

EDAX

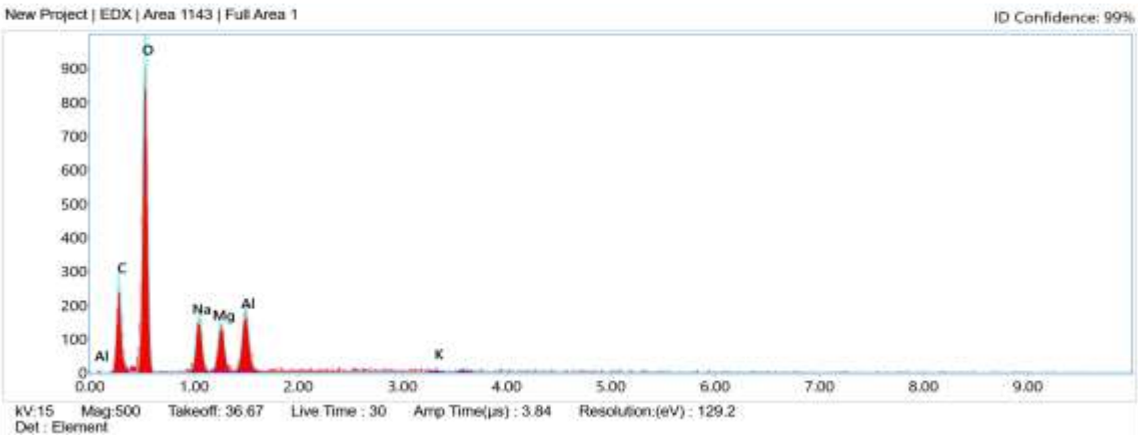

Figure 2: EDAX results of Sodium beta Alumina

XRD Analysis

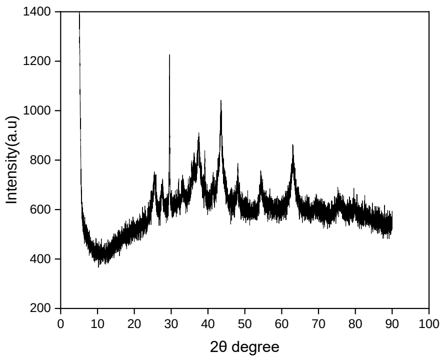

Figure 3: Structural study of Sodium beta Alumina

The powder's crystal structure as synthesized was determined by X-ray diffraction (XRD). All of the diffracted peaks were intense and narrow, indicating that the powder obtained by sol-gel reaction was of good crystal quality. All of the diffracted peaks can be perfectly indexed into a crystal phase, indicating a pure β'' alumina. The XRD depiction of the produced ceramic particles matches well with the diffraction properties of standard β''-Al_2O_3 (JCPDS 19–1173). The graph makes it evident that the diffraction patterns of hexagonal β- Al_2O_3 (JCPDS 19–1174) and rhombo hedral β''-Al_2O_3 (JCPDS 19–1173) are strikingly similar. By using the Scherer's method, the crystallite size of β''-alumina was computed and found to be between 10nm and 34nm.

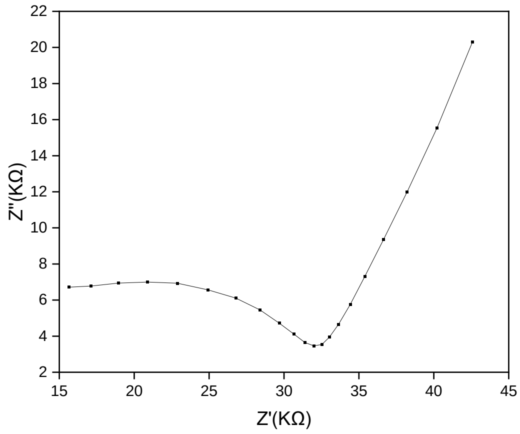

Figure 4: Impedance analysis of Sodium beta Alumina

The electrochemical characteristics of the electrolytes are investigated using the electrochemical impedance spectroscopy (EIS) method. The AC impedance spectra of ceramic pellets made of sodium beta-alumina at 700^0C are shown in Figure 4. The sol-gel method was used to create the sodium beta-alumina nano powder product. The sodium beta-alumina ceramic pellets' ohmic resistance is represented by the semicircle's intercept with the real axis. The outcomes demonstrated that the synthetic samples could improve this phase's conductivity.

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