# Low Temperature Synthesis of $SrAl_2O_4$ and its Characterisation

Ahalya H.G, B.H. Doreswamy and B.M. Nagabhushana

**Abstract---** This paper reports the detailed preparation of strontium aluminate phosphors ($SrAl_2O_4$) by solution combustion method using respective metal nitrates and oxalyline dihydrazine as a fuel. It results in low density, voluminous mass compared to the samples prepared by other conventional methods. The present work reports the changes made in combustion process to achieve homogeneous nano powder in large scale with a short interval of time. The sample has been characterized by X-ray diffraction studies, scanning electron microscopy, TG/ DTA, FTIR and UV-Visible. XRD result shows the sample is in monoclinic phase. Scanning electron microscope reveals the product is highly voluminous and porous in nature. The size of the particles was about 100 nm. In FTIR transmittance peaks observed at 3744 and 1471 $cm^{-1}$. The band gap comes out to be 5.18ev.

**Keywords---** $SrAl_2O_4$, Solution Combustion Synthesis, Powder X-Ray Diffraction, Oxalyline Dihydrazine, Atomic Force Microscopy.

## I. INTRODUCTION

Strontium aluminates are alkaline earth aluminate phosphors. The studies on these compounds started in 1970 and become popular after 1990 because of many attracted properties in dark environment [1, 2]. Strontium aluminate doped with rare earth is useful for many applications such as long lasting phosphorescence, airport, luminous paints, detection of damages in buildings and bridges, ceramic products, textile industries, watch dial glow plates, warning signals, fluorescent lamps display devices, long phosphorescence and more intensive light is emitted. The doped rare earths are sensitive to the lattice environment of the parent structure. When the compound synthesized at nano level shows size dependent properties [3–22]. These are excellent materials for quantum efficiency in visible region, efficient materials for basic science because of no radioactive effect, technical and medical applications. It can also be used for mechano luminescence application in which mechanical action such as elastic deformation, plastic deformation etc can be applied [23]. Mechano luminescence phosphor can be used in the potential market for liquid crystal display devices [25]. Electron hole trap and long after glow can be studied by $SrAl_2O_4$. This can be synthesized by several methods like solid state reaction, sol-gel technique, chemical precipitation, spray pyrolysis, hydrothermal, green synthesis, emulsion method, hard template method and solution (aqueous) combustion synthesis [8, 12]. The oxide phosphors have long time emission of light. The combustion method is one of the fascinating methods to prepare precursor powders and the fuel should have low igniting temperature. This method saves time and energy. Many materials were synthesized using this technique. [22]. In this method the size can be controlled and different shape particles can be synthesized. It is one of the reliable method for the preparation of long after glow phosphors [17 -19]. Solution combustion synthesis proved convenient method for nano sized oxide materials. In the present study we have attempted to prepare $SrAl_2O_4$ phosphor by solution combustion synthesis and the obtained as formed nano powder was characterized.

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## II. EXPERIMENTAL PROCEDURE

### A. Materials Used

Analytical grade aluminium nitrate [$Al(NO_3)_2.9H_2O$], strontium nitrate [$Sr(NO_3)_2.4H_2O$] and fuel oxalynie dihydrazine (ODH) were used as staring materials.

### B. Preparation of as Formed SrAl2O4

Stoichiometric composition of the redox mixture for a solution combustion synthesis were calculated using the total oxidizing (O) and fuel (F) valencies of the components which serve as the numerical coefficients for the balance of the equation so that equivalence ratio $Ø_e$ is equal to unity. That is O/F = 1. The precursors are mixed in a dish with double distilled water. The mixture stirred using magnetic stirrer at 80 revolutions per second for 5 minutes. Then the mixture introduced into 550 $^0$C preheated muffle furnace. Initially the mixture melts and undergoes dehydration followed by decomposition with evolution of large amount of gases (oxides of nitrogen, carbon and ammonia). The energy released by combustion is maximum. Hence this process is exothermic. The mixture catch up fire and glows with more brightness (incandescence). During incandescence the foam further swells to the capacity of container. The entire combustion process completes within 5 minutes. The flame temperature as high as 1400 to 1600˚C, converts the vapour phase oxides into mixed aluminates. The flame persists for nearly 40 seconds. The product is milled to get fine powder.

### C. Instrument Description

Powder XRD data of the phosphor was collected from Rigaku- D X-ray diffractor (40kV, 35mA) using Cu/Kα radiation (λ = 1.5418Å) continuous scan at the rate of 10˚/min. The particle size and morphological investigations of phosphor prepared in the process carried out with a scanning electron microscope (SEM, LEO 440 System). TGA measurements with TG209F3 Tarsus. FTIR spectroscopy recorded with an IR spectrometer Model EQUINOX55, Bruker Co. Germany using KBr discs. AFM studies carried out with non contact mode AF60, the UV-Visible instrument was DU 640 spectrometer.

## III. RESULTS AND DISCUSSION

### A. XRD, SEM, TG-DTA and AFM Studies

To study the crystalline structures of the sample, XRD measurement were carried out at room temperature. Fig 1 shows the XRD patterns of the $SrAl_2O_4$ as-formed. All the reflections can be indexed to $SrAl_2O_4$ monoclinic phase. One impurity peak is observed at 35˚ which might be due to $Sr_3Al_2O_6$ [27]. This is due to the release of heat during combustion reaction. Which shows the fuel is not enough to form the pure $SrAl_2O_4$ phase due to low adiabatic temperature. [3]. The XRD pattern matched with JCPDS 34-0379. The lattice parameters are a = 8.442 Å, b = 8.822 Å, c = 5.160 Å and β = 93.415 $^0$ [1]. The structure of low temperature phase has three dimensional network of corner sharing $AlO_4$[24]. The average particle size found to be 50nm calculated from Debye-Schriffer formula. Fig 2 shows SEM micrograph of as formed strontium aluminate prepared by solution combustion synthesis. In case of combustion synthesis release of large volume of gases from mixed solution result in the production of fluffy form nano particles. The grain boundaries with complete morphology can be seen [6].The wide particle size distribution as well as irregular shapes of the particles probably due to non uniform distribution of temperature and mass flow in the combustion wave [26]. The behaviour of solution combustion synthesised solid nano powder was studied by thermal analysis. The TG and DTA plot as shown in Fig 3. Thermo gravimetric Analysis (TGA) measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Measurements are used primarily to determine the composition of materials and to predict their thermal stability at temperatures up to 900°C. The technique can characterize materials that exhibit weight loss or gain due to decomposition or oxidation or dehydration.

In the present study TG curve exhibits 4 distinct weight loss steps (Fig3). The first weight loss step with 2.4% took place in the temperature region 30 $^0$C to 200 $^0$C due to dehydration In sol gel method weight loss is 5% due to dehydration [8]. The second weight loss step 200 $^0$C to 300$^0$C corresponds to the decomposition. As expected, the sharp and intense exothermic peak was observed at 295$^0$C in DTA curve. The weight loss associated with this step is 7.7% due to decomposition. A weight loss of nearly 2.9% observed at 700$^0$C. A loss of 3.1% observed 900$^0$C both are due to oxidation. No other peak observed in DTA it proves no residue organics in ashes hence the prepared sample is pure enough. From the graph we can deduce that during combustion process of the compound decomposition and auto combustion of sample takes place. In these two steps more amounts of gases such as nitrogen dioxide released. In combustion weight loss is less when compared to other methods. Atomic force microscopy results reveal that as formed strontium aluminates the average particle size observed to be from 50 nm to 100 nm. This is as shown Fig 4.

### *B. FTIR and UV – Visible Studies*

The FT-IR spectrum of $SrAl_2O_4$ powder sample is shown in Fig 5. The bands between 350 and 1000 cm$^{-1}$ can all be assigned to IR active vibration modes of as formed sample. The symmetric bonding of O-Al-O appears below 500 cm$^{-1}$ the anti symmetric stretching bands range from 588-845 cm$^{-1}$ is ascribed to the Sr-O vibrations. The band positioned at 782 and 900 cm$^{-1}$ originates from aluminates group ($AlO_4$). The band at 1471 cm$^{-1}$ is C-O vibration band. The band located at 3744 cm$^{-1}$ is –OH group symmetric vibration.

The optical absorption spectra is an important characterization to know the behaviour of nano crystals. Fig 6shows the absorption spectra in the range 90nm to 500nm. From this band gap between filled valance band and empty conduction band can be calculated. An abrupt increase in absorption can be observed at 240nm. This is due to the energy gap. No absorption occurs when $\lambda > 400$nm. The band gap was calculated corresponding to 240nm using Beer Lamberts law which comes out to be 5.18ev.

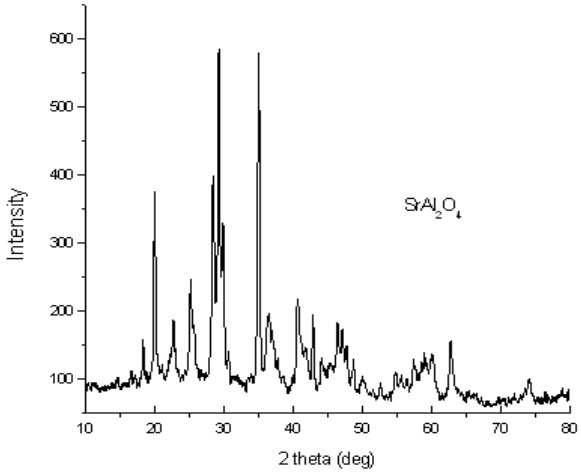


Fig. 1: XRD of $SrAl_2O_4$

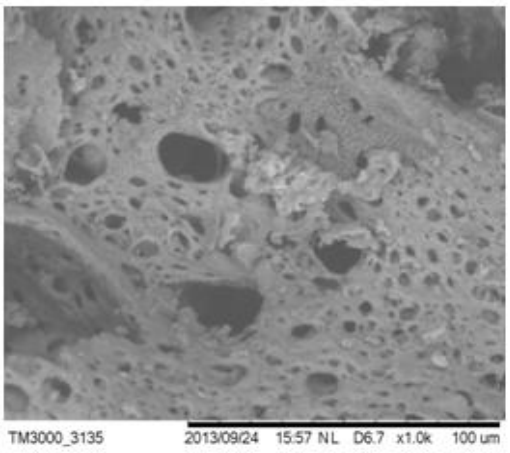


Fig. 2: SEM of $SrAl_2O_4$ Nanopowder

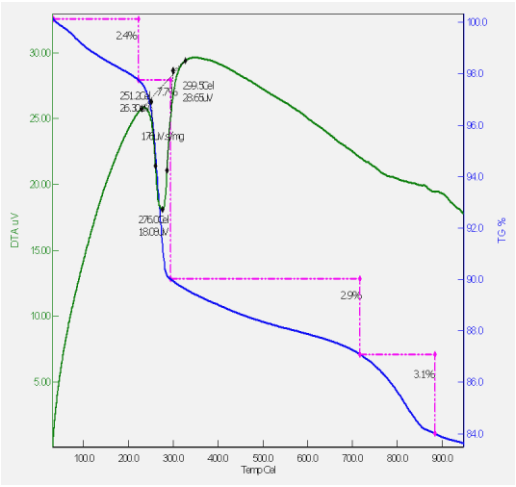


Fig. 3: TG/DTA of SrAl2O4

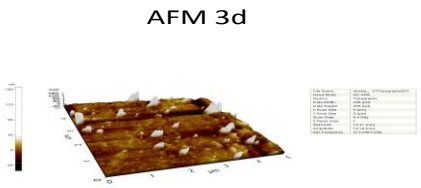


Fig. 4: AFM of $SrAl_2O_4$

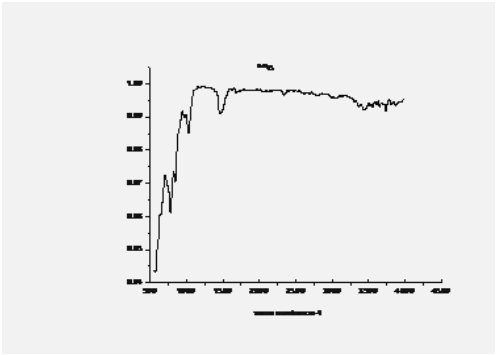

Fig. 5: FTIR of $SrAl_2O_4$

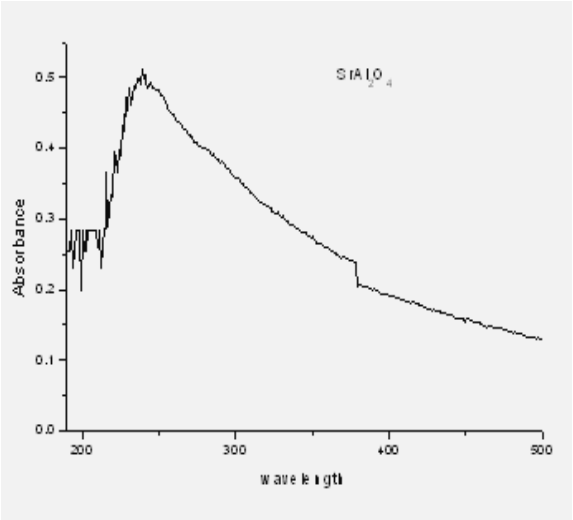


**Fig. 6: UV-Visible of $SrAl_2O_4$**

## IV. CONCLUSION

On the basis of above research $SrAl_2O_4$ nano powder was successfully synthesized by solution combustion method. This process enables fast and energy efficient production of well crystallized powder particles. XRD results implied that phosphor powders have monoclinic phase with an average particle size of about 50nm. AFM analysis which supports the same results. TG/DTA shows that in combustion the energy loss is less when compared to other methods, UV-Visible shows the band gap about 5.18 ev.

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