

Low temperature method for synthesis of MgNb_2O_6 and its characterization

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Abstract

There are various methods for synthesizing luminescent semi-conductor nano-particles. A sample at chemical root of low temperature method was used to synthesize the magnesium niobate (MgNb_2O_6) nano-particles by using Niobium pentoxide (Nb_2O_5), Magnesium nitrate, Ammonium carbonate were used. All chemicals was taken A.R. Grade (99.99%). Ammonium carbonate and Ammonium hydroxide was used to precipitate magnesium carbonate as Mg^{2+} cation and Niobium hydroxide as a Nb^{5+} cations under basic conditions. This precipitate was heating at 800°C it produced MgNb_2O_6 fine powders. The lattice parameters were studied by least square method and obtained $a = 5.687 \text{ \AA}$, $b = 13.975 \text{ \AA}$ and $c = 4.938 \text{ \AA}$. The average particle size of MgNb_2O_6 were studied by Scanning Electron Microscope (SEM) and found to be 45 nm. The crystallite size calculated from Scherrer's formula.

Keywords: A ceramics chemical synthesis, XRD, SEM.

INTRODUCTION

The discipline of material science involves investigating the relationships that exist between the structure and properties of materials. Material Science plays a decisive role in the entire technological breakthrough. One of the most extensively studied materials in the field of material science is the ceramic ferroelectric material. The ceramic dielectric resonators used at microwave frequencies are high dielectric constant high Q. values (low dielectric loss) and near zero temperature coefficients at resonance frequency.⁽¹⁻⁷⁾ Magnesium niobate and its solid solution with magnesium tantalite found applications as microwave dielectric materials due to their low dielectric loss and high dielectric constant. MgNb_2O_6 is also well known as a key precursor material for the successful preparation of single phase relax or ferroelectric material which is becoming more important for transducer, electro-strictor and actuator applications. MgNb_2O_6 has orthorhombic columbite structure and its unit cell parameters are $a = 5.700 \text{ \AA}$, $b = 14.193 \text{ \AA}$ & $c = 5.032 \text{ \AA}$

Low temperature method is one of the more successful techniques for synthesizing ultrafine ceramic powders having narrow particle size distribution.⁽⁸⁻¹⁰⁾

EXPERIMENTAL

For preparing MgNb_2O_6 , niobium pentoxide (Nb_2O_5), Magnesium Nitrate, ammonium Carbonate were used as starting materials and they were of AR grade. A stoichiometric amount of magnesium nitrate (6.22 gm) was dissolved in double distilled water (60 ml) in beaker and Nb_2O_5 (6.43 gm) was dissolved in HF (40% solution) in plastic container placed on water glass beaker. Now placed it on the hot plate of magnetic stirrer. Now continuous stirring for long time till the solution become colourless for this we add more HF (40% solution). Ammonium carbonate crystals (8.90 gm) were dissolved in Ammonium hydroxide solution and added dropwise to the above solution from burate containing Nb^{5+} and Mg^{2+} ions. This leads to precipitation of Mg^{2+} as a carbonate and Nb^{5+} as hydroxide under basic condition (pH-9). Then the precipitate was washed

thoroughly with double distilled water to remove anions, filtered and dried at 100°C from oven for 10 hours. The oven dried precursor was calcined at various temperature like 400°C , 600°C & 800°C for 8 hours XRD were recorded for oven dried and samples calcined at various temperature by using philips PW 1710 model X-ray diffractometer using CuK α . A least square method was employed to determine the lattice parameter.

RESULTS AND DISCUSSIONS

Fig. 1 show the XRD at room temperature for samples heated at various temperatures for 8 hrs. After heating at 400°C the sample is X-ray amorphous no distinct peak are observed. At 600°C only the peaks corresponding to Nb_2O_5 are seen and MgO probably was present as fine amorphous particles. Only at 800°C , does the Magnesium Niobate (MN) phase appears. The calculated unit cell parameters are $a=5.692 \text{ \AA}$, $b=14.203 \text{ \AA}$ and $c=5.039 \text{ \AA}$ (standard deviation= 0.005 \AA)

Fig.2 shows the particle size and morphology of the calcined powders were examined by scanning electron microscope (SEM). Particle morphology of the calcined powder (800°C for 6 hours) prepared by the co-precipitation process was irregular in shape, with an average primary particle size are of around 40 nm. The crystalline size calculated from Scherrer's formula ($t = \frac{KX}{B \cos \theta_B}$) where t is the average size of particles, $K = 0.9$, X is the X-ray radiation, B is the full width at half maximum of the strongest peak and θ_B is the angle of diffraction are 45 nm.

Nano phosphors have been extensively investigated during the last decade due to their application potential for various high performance displays and devices. These act as a strategic component in almost all displays. Synthesis of nano particles can be accomplished in two ways, i.e. through chemical and physical method. In chemical route the low temperature method is widely used for synthesis of small size of nano particles. Nano particle have been used to elimination of pollutants. Nano crystalline

materials possess extremely large grain boundaries relative to their grain size. Hence nano materials are very active in terms of their

chemical, physical and mechanical properties.

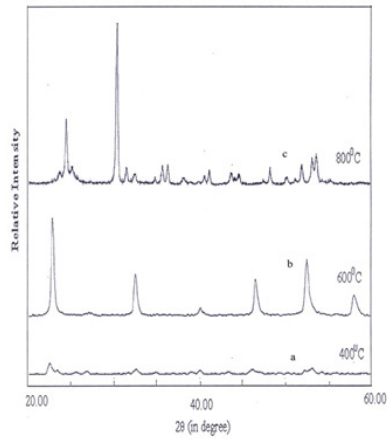


Fig. - 1(a,b,c) X-ray diffraction pattern of MgNb₂O₆ calcined at different temperature.

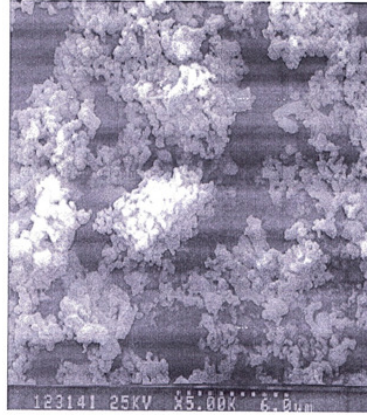


Fig. - 2 Scanning Electron Micrograph (SEM) of MgNb₂O₆.

CONCLUSIONS

For the preparation of Magnesium niobate (MgNb₂O₆) prepared by low temperature method. The MgNb₂O₆ phase was found to be formed at 800°C with average particle size of 45 nm. The crystal system for Magnesium niobate is Orthorhombic and the entire observed at line match with the reported value (JCPDS 15-252). The calculated unit cell parameters are a=5.687Å, b=13.975Å and c=4.938Å.

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