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Prepration of Ceramic Material Dopped with Cr^{3+}

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Abstract: **Zinc aluminate ($ZnAl_2O_4$) being of spinal shaped has drawn considerable attention as a catalytic material. It can be improves proprieties like high thermal stability, high mechanical resistance, crystal structure and the optical spectra [1-4]. More over zinc aluminate tends to prevent sintering of starting materials due to a strong material-support interaction. Nano sized zinc aluminate powder was prepared by heating method at 600^0C sintering temperatures. Zinc sulphate ($ZnSO_4$), alum (Na_2SO_4-Al_2O_3-24 H_2O) potassium dichromate ($K_2Cr_2O_7$) and urea (NH_2 CO NH_2) were used as starting materials. Here urea is used as a chelating agent.**

Keywords: **spinal zinc aluminate, picture of fired sample and SEM of samples.**

I. INTRODUCTION

Nano sized zinc aluminate powders were prepared by sintering method. Zinc aluminate ($ZnAl_2O_4$), naturally occurring as the mineral. Zinc aluminate has drawn considerable attention as a catalytic material with improved properties due to high thermal stability, high mechanical resistance, and low surface activity. Moreover, zinc aluminate tends to prevent sintering of noble metals due to a strong metal–support interaction [5-7].Zinc aluminate (ZnAl2O4) is a member of the class of inorganic materials called spinals. These spinal oxides are wide-band-gap semiconductors; the optical band gaps are reported [2] to be about 3.8eV for ($ZnAl_2O_4$) Zinc aluminate spinal ($ZnAl_2O_4$) has been widely used as catalyst, ceramic and electro-conductive materials. In high temperature processes sintering resistance and chemical stability are the properties of the most significance for the catalytically active phases. Beside catalytic purposes zinc aluminate has been used as ceramic and electro conductive material because of its high thermal stability, high mechanical resistance and excellent optical properties. Optical properties make it material of interest for ceramic pigments and coatings. [8]

Zinc aluminate is normally prepared by a solid state reaction of zinc oxide and aluminum oxide however, for ensuring complete reaction; a temperature above 1000^0 C has to be maintained for several days [9]. On the theoretical front, we are not aware of any studies except that leading to the prediction of the stability of the normal spinal structure over the inverse structure for $ZnAl_2O_4$ [10]. We have therefore embarked upon a detailed theoretical study of these zinc-based spinal oxides. In an earlier work, we have reported the results of an atomistic simulation study [11] that included derivation of inter atomic potentials, and determination of the equation of state and compressibility behavior at the octahedral and tetrahedral sites in the spinal lattice, and the energetic of point defects in $ZnAl_2O_4$.

In the present paper, the synthesis of $ZnAl_2O_4$ nano powders, we use two different methods. The first method is sintering method and second one is combustion synthesis. For achieving combustion at low temp we use a few techniques like use of flux, eletation or sol-gel method because nano sized materials, in comparison with bulk materials, display improved properties viz. lowered sintering temperatures, increased hardness, stability, diffusion etc. which can be overcome by above said special techniques. The disadvantages of solid-state reaction are in homogeneity, high sintering temperature requirement and low surface area.

II. SINTERING METHOD

Sintering is a method for making objects from powder, by heating the material in a sintering furnace below its melting point (solid state sintering) until its particles attain sufficient energy to adhere to each other. Sintering is traditionally used for manufacturing ceramic objects, and has also found uses in such fields as powder metallurgy.

Advantages and requirements of sintering method:

Peculiar advantages of above referred powder technology are:

Very high levels of purity and uniformity in starting materials

1. Preservation of impurity, due to the simpler subsequent fabrication process (fewer steps) that it makes possible
2. Stabilization of the details of repetitive operations, by control of grain size during the input stages
3. Absence of binding contact between segregated powder particles – or "inclusions" (called stringering) – as often occurs in melt processes
4. No deformation needed to produce directional elongation of grains
5. Capability to produce materials of controlled, uniform porosity.

Sintering is effective when the process reduces the porosity and enhances properties such as strength, translucency and thermal conductivity; yet, in other cases, it may be useful to increase its strength but keep its gas absorbency constant. During the firing process and as it continues; grain size becomes smaller and more spherical because the particle's surface tends to flow into the pores within it based on the difference between vapor-pressure and cross-sectional area of the pore's neck.

III. WORKING

sintering is a effective process which reduces the porosity and enhance the strength, translucency and thermal conductivity of the material. By keeping the gas absorbance constant, material strength can be increased. Particle’s surface tends to flow in to the pores with it.

The method of making zinc aluminate nano material comprise of following steps:-

Firstly, provide a growing substrate and growing device, comprising a heating apparatus with reacting chamber. Secondly, placing growing substrate and a quantity of reacting materials into the reaction chamber. Lastly, heating the reaction chamber to a temp of 600 ^{0} C. This paper presents the comparison between doped and undoped zinc aluminate spinal.

The method of using solid-state reaction for making zinc aluminate nano material includes the following steps.

1. Take a quantity of zinc sulphate ($ZnSO_4$) powder (750 mg), alum (Na_2SO_4 Al_2O_3 $.24H_2O$) powder (1500 mg) and urea (NH_2 CO NH_2) powder (750 mg).
2. Grind the zinc sulphate powder, alum powder and urea. Now putting the mixture into a furnace.
3. Heating the mixture to a temperature at 600^0C for 15 minutes to obtain a zinc aluminate nano material.
4. Keep the material (A) into the furnace when the temp decreases gradually.
5. Again take different quantity of alum powder (1500mg), zinc sulphate powder (750mg), urea powder (750mg) and an impurity potassium dichromate (10mg).
6. Now grind it and put the mixture in to furnace at 600^oC then keep material (B) into the furnace and let cool it down.

IV. RESULT AND DISCUSSION

the temperature of the formation of zinc aluminate was found to be 600^oC at a reaction time 15 minutes. At short reaction time the reaction rate is obviously depend upon the no of nucleons sites or content point of alumina or zinc. If we increase the reaction time the rate of reaction time increases and the rate of reaction becomes increasingly dependant upon the rate of diffusion of zinc.

In the SEM images and picture of a material “A” (undoped) we found that the surface of the material is porous and upper surface is look like the moon surface which has small hole like structure internal surface is of the spinal form.

When the doped sample “B” with $k_2Cr_2O_7$, we found that the material becomes hard and their surface has spinal form. Material also has porosity on the surface. Photographs of material are shown in the fig.

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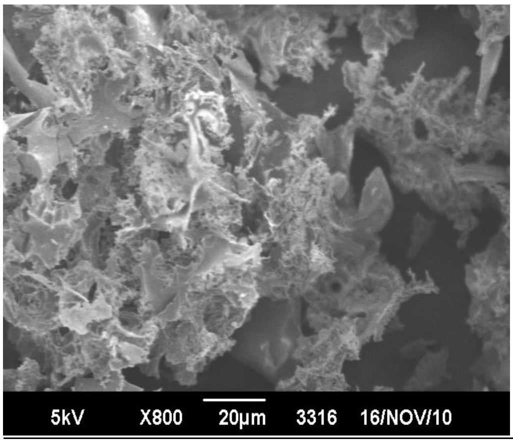
Comparison between doped and undoped zinc aluminate:

Sample	temperature	type	Surface	shape	color
A (Undoped)	600	soft	Porous and upper surface look like moon Internal surface- spinal form	Regular shape	White
B (doped)	600	hard	Porous and spinal form	Irregular shape	Light pink

SEM images and picture of material (A):



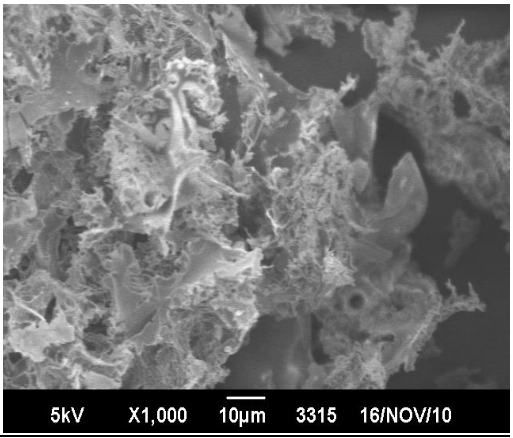
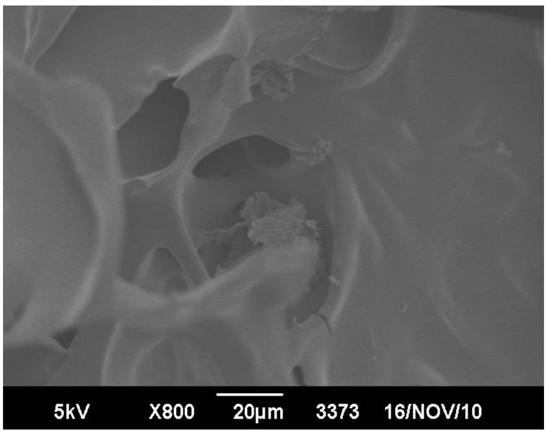
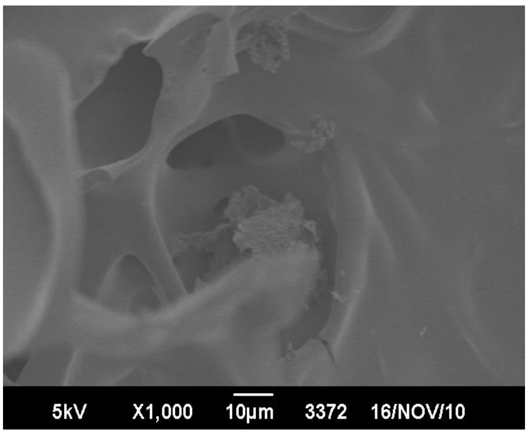


Fig.1 **fig.2**

SEM images and picture of material (B):





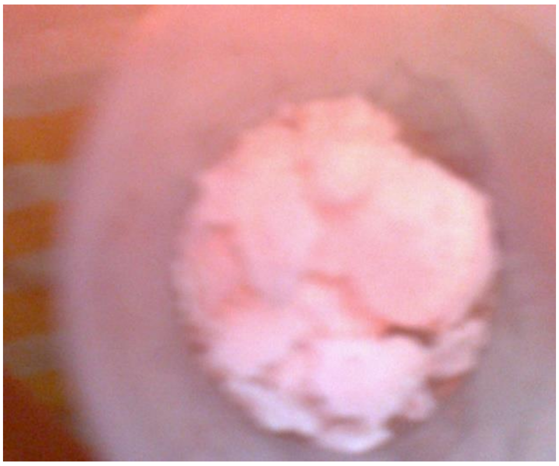


Fig.3

fig.4

V. CONCLUSION

The relative reactivity of the formation of zinc by the powder method was investigated by the SEM image. The images suggested that diffusing spices in $ZnAl_2O_4$ is a one way transfer of ZnO through zinc aluminate layer to the alumina particle. Its conductivity study is to be done. So that material can be used for good thermal conductivity.

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