EG0100132
Seventh Conference of Nuclear Sciences & Applications
6-10 February 2000, Cairo, Egypt

WM-11

Preparation, Characterization and application of Alumina powder Produced by advanced Preparation Techniques

By

T.Khalil, **J. Bossert,A.H.Ashor and *F. Abou EL-Nour**

***Hot Labs Center, Atomic Energy Authority (AEA), Cairo, Egypt**
***** Physics Department, Radiation Technology Center, (AEA), Egypt**
**** Material Science Department, Friedric Schiller University, Jena, Germany**

ABSTRACT

Aluminum oxide powders were prepared by advanced chemical techniques. The morphology of the produced powders were examined using scanning electron microscopy (SEM). Surface characteristics of the powders were measured through nitrogen gas adsorption and application of the BET equation at 77 K, through the use of nitrogen gas adsorption at liquid nitrogen temperature and application of the Brunauer-Emett-Teller (BET) equation. The total surface area, total pore volume and pore radius of the powders were calculated through the construction of the plots relating the amount of nitrogen gas adsorbed V_l and the thickness of the adsorbed layer t (V_l-t plots). The thermal behaviour of the powders were studied with the help of differential thermal analysis (DTA) and thermogravimetry (TG). Due to the presence of some changes in the DTA base lines, possibly as a result of phase transformations, X-ray diffraction was applied to identify these phases. The sintering behaviour of the compact powders after isostatic pressing was evaluated using dilatometry. The sintering temperature of the studied samples were also determined using heating microscopy. The effect of changing sintering temperature and of applying different isostatic pressures on the density and porosity of the compacts was investigated

Keywords: fabrication of ceramic powders/ alumina/ flame hydrolysis/ polymeric routes/ sol-gel technique/ nano-powders.

INTRODUCTION

The preparation condition as well as the preparation techniques for the fabrication of ceramic powders are receiving new research emphasis because of their recognized relationship with the physical and chemical characteristics of the end products[1-3]. To achieve the desired properties of the ceramic powders, some authors [4-14] all over the world practiced different techniques such as coprecipitation and thermal decomposition of carbonates, vapour phase reactions, laser techniques, flame hydrolysis, polymeric routes, sol-gel ,etc. to obtain ultra pure, ultra fine and homogeneous powders.

Aluminium oxide is considered as one of the most important and most widely studied oxide ceramic powder due to its applications in different fields such as structural, electrical, optical and nuclear technology [15-16]. Al_2O_3 oxide spheres fabricated by different methods can be applied as inorganic ion exchangers for the removal of radioisotopes from thier solutions[15] and fixation of very high concentration of high level radioactive waste elements[17]. However, due to its covalent nature, the pure material require very high sintering temperature (> 1700 °C) to produce dense material (> 95 % of theoretical density). Such high sintering temperature produces porous, coarse grained (>10μm) ceramic material with poor properties[18]. Accordingly, sintering aids [18-20], forming techniques [18,20,21] as well as advanced preparation techniques are to be used to improve the sintering kinetics and to control non-uniform grain growth producing a high dense ceramic materials.

In the present work three wet chemical methods for powder preparation have been studied and applied to produce ultra fine ceramic powders which in turn reduce the sintering temperature of the fabricated materials. Physico-chemical characteristics of the produced powders using scanning electron microscope, BET technique (surface area measurments using <brunauer.Emmitt Tailor equation), x-ray diffraction and heating microscope were studied and the results were discussed.

POWDER METHODOLOGY AND WET CHEMICAL METHODS:

Wet chemical methods became an almost universal techniques for production of all types of ceramic powders with high efficiency. Also the produced oxides by wet methods have several advantages over others which are produced by conventional methods. The three different techniques that applied in the present work are:

1. Flame Hydrolysis

This method is used to prepare oxide powders having nanometeric particle size. This process is based on the generation of aerosol droplets of liquid reactants. The droplets are then exposed to a coreactants in the vapour phase to yield predictable shape and size powders [22-25]. It involve spraying of salt solutions (aluminium chloride) into a furnace having a temperature between 900-1000 °C, where the droplets are solidified and the salt decomposes to oxide. The chemical reaction follows the equation:

$$4\,AlCl_3 + 6\,H_2 + 3\,O_2 \;\text{-----------}\!> \; 2\,Al_2O_3 + 12\,HCl \qquad (1)$$

The used Al_2O_3 powder are produced commercially in a very large quantity by a Degussa Company (Germany) and have the commercial name aluminum oxide C.

2. Polymeric Routes

The polymeric routes for the preparation of reactive and fine oxide ceramic powders using urea formaldehyde resin was applied in this study. The route has been utilized for the production of the powder from their respective salts [26-28]. The alumina powder that used in the present work was prepared through polymerization reaction between urea and formaldehyde to form the respective resin hosting Al^{+3} cation. The clear transparent glassy like gel with no signs of precipitation has been achieved over the full range of pH-values for the composition of urea : formaldehyde of 1: 2 mole.%. Two moles of ethylene glycol (the appropriate alcohol chosen to terminate the polymerization reaction) is added to the mixture of urea formaldehyde resin. The polymeric resin hosting Al^{+3} was synthesized by adding one mole salt solution of aluminium chloride in the final stage of the reaction. The material were left to react under reflux with stirring for about 1 h at a temperature 60-70 °C. Water and other organic by-products were separated using vacuum distillation pump. The prepared resin hosting Al^{+3} was calcined to yield aluminium oxide powders.

3. Sol-Gel Processes

The sol gel process as an example of wet chemical method appears to be particularly promising method for the production of many oxides [29-32]. In this method three operations take place:

1- Preparation of an aqueous sol ,

2- Removing of water from the prepared sol to get the gel ,

3- Thermal treatment of the gel to produce the oxide .

The gelly state of matter has become a source of special interest due to the fact that from gel of certain composition at certain temperature one may get a solid state.

The high surface area of the dried gels results in a very high reactivity which permits a low temperature processing. By starting with low mixed solution or sols, chemical homogeneity can be obtained and high purity can be maintained. The general overview of the sol gel process established by some authors [33] is shown in the fig (1).

In this sol-gel method, aluminium oxide was prepared using aluminium tri sec butylate as precursor. 250 g of this alkoxide was added to 300 ml isopropyl alcohol with strong stirring. Excess amount of distilled water and drops of ammonium hydroxide (pH of the mixture = 7) were added under vigorous stirring to the alkoxide mixture for hydrolysis. The gel solution was then centrifuged for 5 min. (4000U/min.). The separated gel was then slowly dried at a different temperature range 60-120 °C.

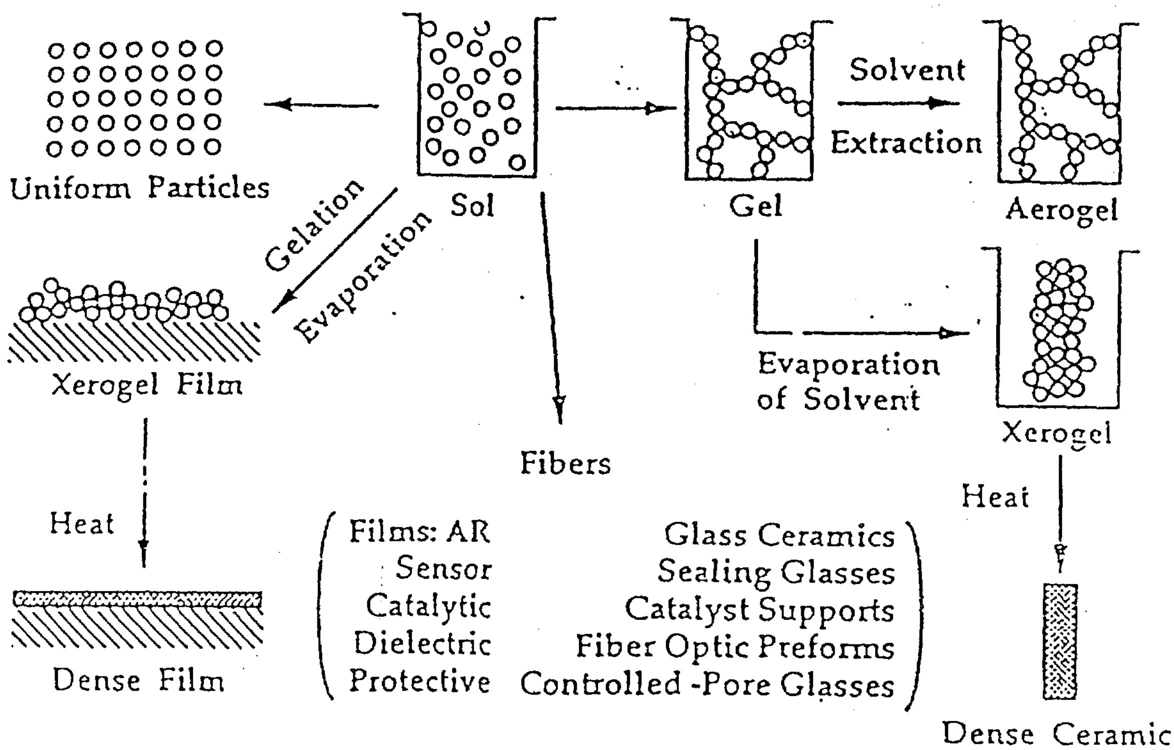

Fig. (1) General overview of the sol-gel process [33].

III. EXPERIMENTAL AND RESULTS

III.1. Physico-chemical Characteristics of the Fabricated Powders

The powders produced by the different techniques were slowly dried till 120 °C. The specific surface area of the different aluminium oxide powders was measured using calibration method by nitrogen adsorption at 77 K and application of the BET equation. The pH value, were followed during preparation. The densities of the dried powders were measured using pechnometer at 120 °C. The particle size of the powders was measured using scanning electron microscope and the present phases were detected using x-ray diffraction technique.

Table (1) shows the effect of preparation method on the specific surface area, pH, density, particle size and the present phases, While Table (2) shows the effect of calcination temperature on the properties of the produced powders.

Table (1): Effect of preparation method on the different properties of the ceramic powder after drying at 120 °C

Measured Properties	Flame Hydrolysis	Polymeric Route	Sol-Gel Process
Sp. surface area [m^2/g]	91,4	38	45
pH value	9	1-2	7
Density [g/cm^3]	2,9	---	----
Particle size [nm]	< 20	< 75	<50
Present phases	gamma	amorphous	amorphous

Table (2): Effect of calcination temperature on the surface area and the present phases for the fabricated powders.

Preparation method	Calcin. temperature ℃	Specific surface area [m^2/g]	present phases
Flame Hydrolysis	500	85	gamma
	700	81	gamma
	900	44,5	gamma + theta+delta
	1150	20.68	alpha
Polymeric route	500	205	amorphous
	700	163	amorphous
	900	96	gamma + theta
	1150	73	alpha
Sol-gel process	500	68	amorphous
	700	79	amorphous
	900	125	gamma + theta
	1150	92	alpha

2. Scanning Electron Microscope Examination :

Fig (2a,b) show the morphology of the prepared aluminium oxide powder produced by polymeric routes and sol-gel process respectively using scanning electron microscope (SEM), while Fig. 2c shows the transmission electron micrographs (TEM) of the powder produced by flame hydrolysis. The use of TEM to examine the morphology of ceramic powders produced by Flame hydrolysis is due to its very fine structure which could not be detected by SEM.

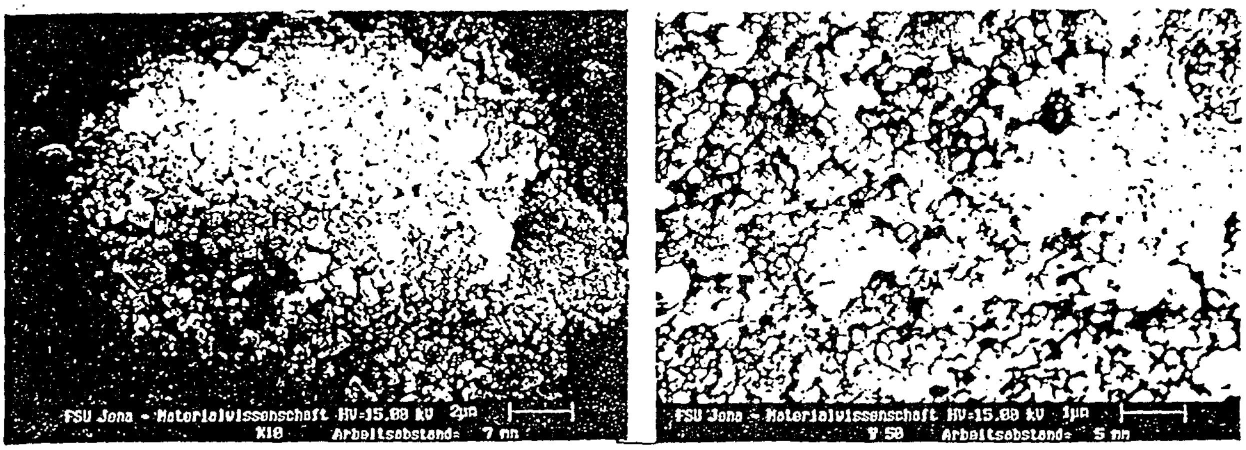

Fig (2):- SEM micrograph of the Al_2O_3 powders prepared by
(a) Polymeric routes (b) Sol-Gel Process

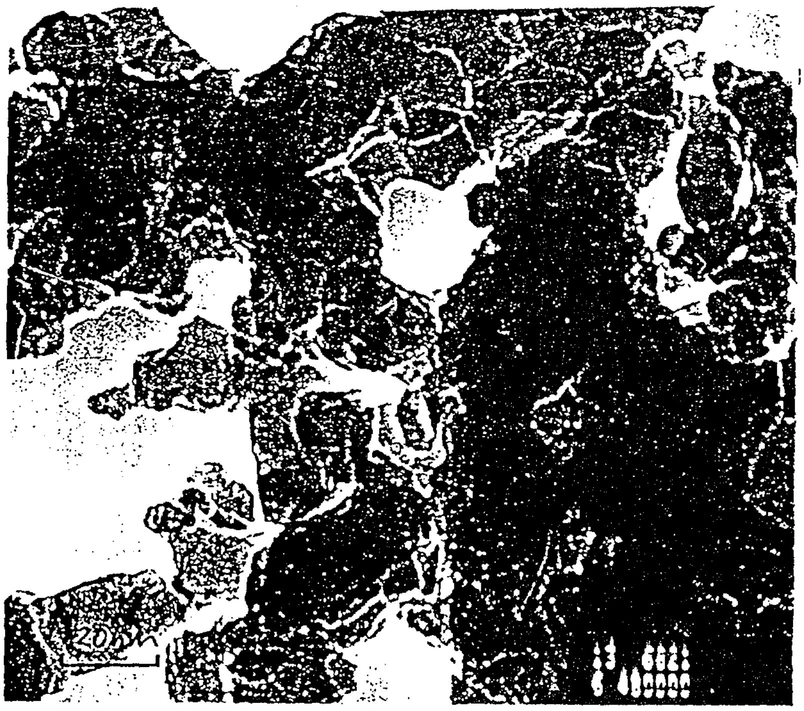

Fig (2c):- TEM micrograph of the Al_2O_3 powders prepared by Flame Hydrolysis

3. Thermal behaviour of the compact samples using LEITZ heating microscope:

Different Al_2O_3 powders produced by the three used preparation methods were pressed biaxially at a pressure of 255 MPa.

The thermal behaviour of pressed powder were examined using LEITZ Heating Microscope Fig (4) shows the axial shrinkage of the three different powders.

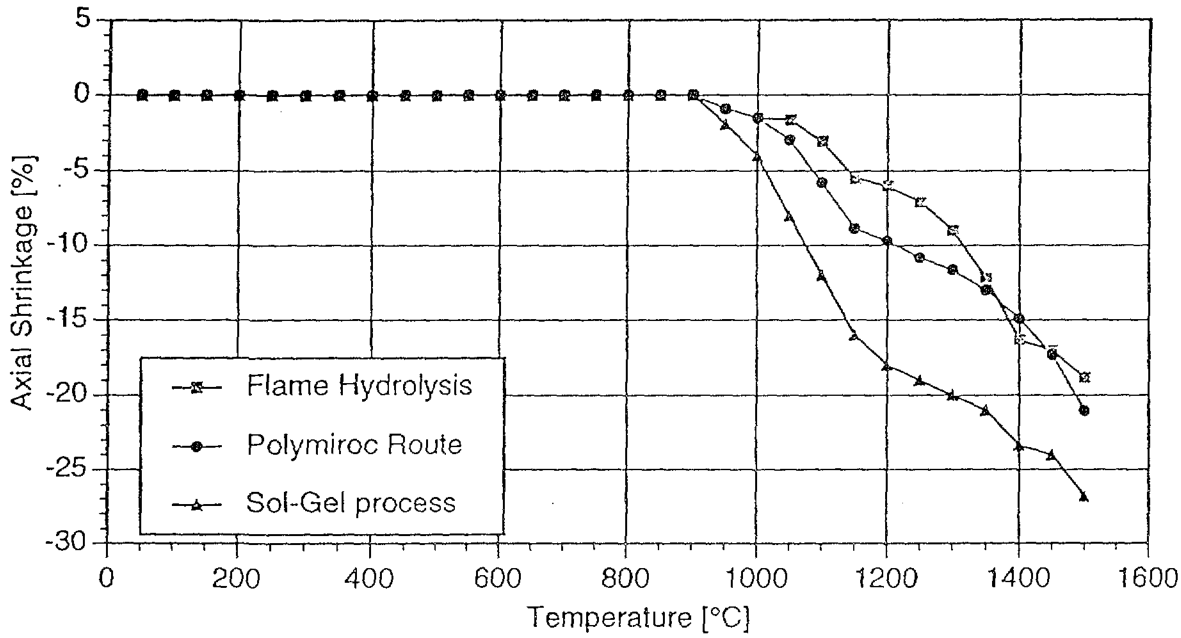

Fig (4):- Axial shrinkage of Alumina powders prepared by different techniques.

4. Sintering behaviour of Al_2O_3 samples:

The different Al_2O_3 powders were pressed isostatically at 600 MPa pressure after its calcination at 900 °C/3h. The compacts were sintered at the following conditions:

a- Heating rate of 10 °C/min from room temperature to 900 °C

b- 10 min. socking time at 900 °C then

c- 5 °C/min from 900 °C and up to the different sintering temperatures (1100, 1200, 1300, 1400, 1500, 1600, 1650, 1700 °C). All samples were soaked for 2 h. at the selected sintering temperature. The relation between sintering temperature and density is shown in Fig (5).

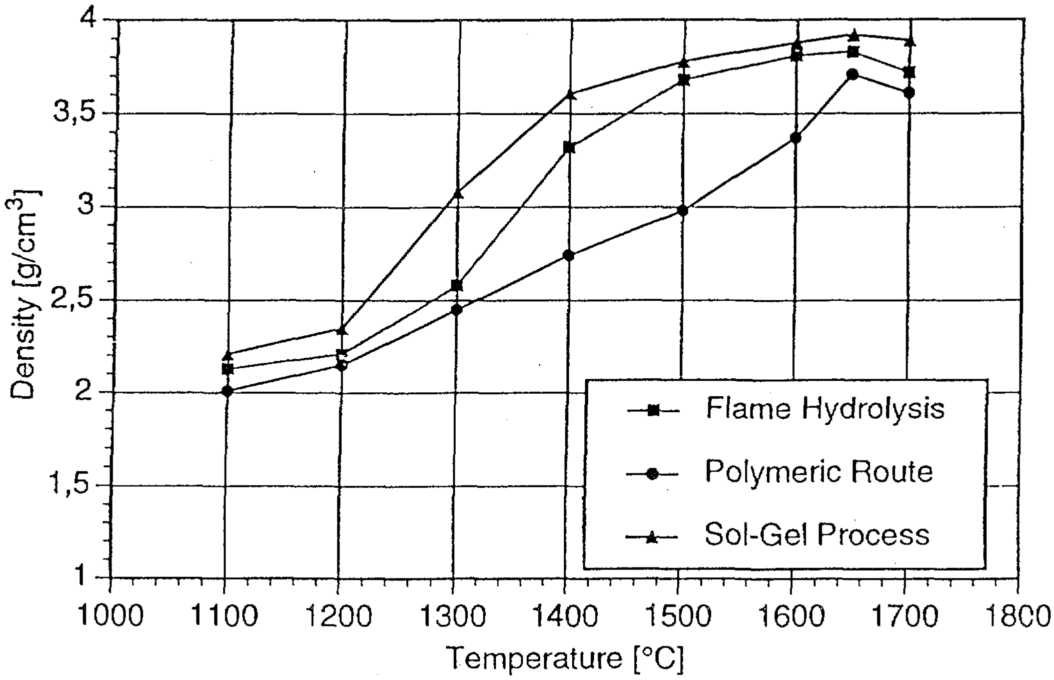

Fig (5):- Densification behaviour of the fabricated Alumina powders using different wet chemical techniques.