GLOBAL JOURNAL OF PURE AND APPLIED SCIENCES VOL. 7, NO. 1 JANUARY 2001: 81 - 84
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SYNTHESIS AND X-RAY DIFFRACTION STUDIES OF γ-Al_2O_3 USING ALUMINUM ACETYLACETONATE (AAA) PRECURSOR VIA A NOVEL SOL-GEL ROUTE

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(Received 2 March, 1999; Revision accepted 27 July, 2000)

ABSTRACT

Crystalline powder of γ-Al_2O_3 has been successfully synthesized by employing aluminum acetylacetonate-methanol-ethylene glycol-water system via a novel sol-gel route. The crystalline particles of the powder obtained were examined by X-ray diffraction, scanning electron microscopy and transmission electron microscopy, while the surface area of the oxide powder was obtained by nitrogen adsorption BET surface area measurement. The result obtained indicated that the γ -Al_2O_3 is cubic with a = b = c = 7.5911A^0, the mean particle diameter is 85 nm and the BET surface area is 120 m^2/g. The result showed that the product obtained is a pure γ -Al_2O_3 with a nanocrystalline structure. The oxide powder could be very important in catalysis and gas sensing of hydrocarbons.

Keywords: Synthesis, X-ray diffraction, Acetylacetonate, Gamma Alumina, Sol-gel route

INTRODUCTION

There is increasing interest in Al_2O_3 materials for use as additive in the conversion of magnetite (Fe_2O_3) to γ-Fe_2O_3 for gas sensing purposes (Nakatani et al., 1982). Yamazoe (1991) reported the use of Al_2O_3 as binder in commercial SnO_2 sensor. High-purity alumina is also useful as abrasives, catalysts and structural ceramics (Martin and Weaver, 1993). Sol-gel method is an extremely valuable method for preparing metal oxide based materials. Conventional sol-gel method requires the initial formation of a metal hydroxide or hydrous oxide in the form of a gel obtained by hydrolysis of one of its compounds in solution followed by heating the hydrolysis product to give the oxide (Spiccia et al., 1996). The use of sol-gel method involving aluminum chloride and either aluminum triisopropoxide or diisopropyl ether precursors to prepare crystalline γ -Al_2O_3 was reported by Wafers and Misra (1987). Murugavel et al.(1998) reported the preparation of crystalline κ-Al_2O_3 film via nebulized spray pyrolysis using aluminum acetylacetonate as a precursor. However, a report on the preparation of nanocrystalline γ-Al_2O_3 powder from aluminum acetylacetonate-methanol-ethyleneglycol-water system is lacking in the literature. In view of the numerous importance of Al_2O_3 in science and technology, this paper reports the preparation of γ-Al_2O_3 via a novel sol-gel route method.The prepared nanocrystalline γ -Al_2O_3 was characterized by XRD, SEM and TEM.

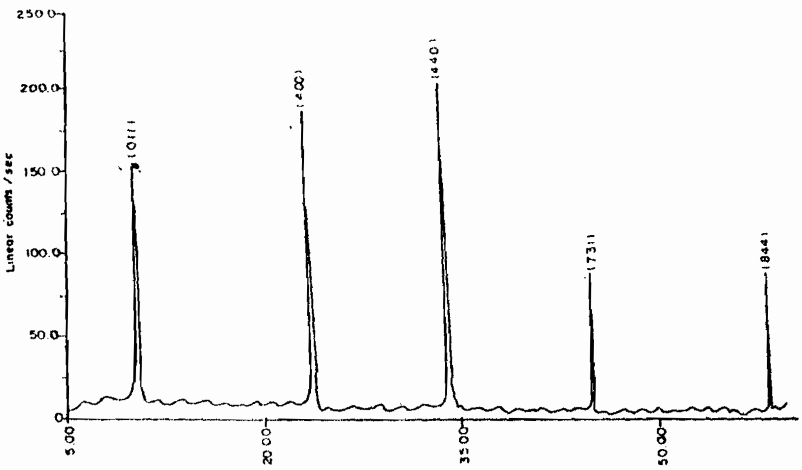
EXPERIMENTAL PROCEDURE

Preparation of γ -Al_2O_3 by sol-gel method

10ml of 0.025M aluminum acetylacetonate solution in methanol was mixed with 10ml of

Table 1. Joint Committee on Powder Diffraction Standard (JCPDS) file number 10-425 for γ-Al_2O_3 (Cubic; a = 7.90 A°; λ= 1.5418).

d (A°)	Int	hkl
4.56	40	011
2.80	20	220
2.39	80	311
2.28	50	222
1.977	100	400
1.520	30	511
1.395	100	440
1.140	20	444
1.027	10	731
0.989	16	800
0.884	10	846
0.806	20	844



The XRD pattern of γ-Al_2O_3 prepared from $Al(acac)_3$ at 800^0C by sol-gel method

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ethyleneglycol previously hydrolysed with 10ml of distilled water in a 250ml beaker. The mixture was stirred thoroughly for 60 minutes using a magnetic stirrer. The sol formed was dried at 80°C for 45 minutes. Gelation was observed to occur at this time. The dried gel obtained after 10 minutes of further drying was then calcined at temperatures of 800°C and 900°C for a period of 20hrs respectively.

Characterization of γ-Al_2O_3 powder obtained

The phase composition of the powders obtained at the different temperatures were determined by employing a Seifert 3000 TT X-ray powder diffractometer with Cu-Kα radiation (θ-θ geometry). A Leica S 440i scanning electron microscope and a Quantimet Q500MC image analyser enabled the morphology of the crystalline γ-Al_2O_3 to be obtained. Transmission electron microscope images were recorded with a JEOL 3010 microscope. BET surface area measuresment by nitrogen adsorption was done using micromeritics accusorb 2100E instrument.

RESULTS AND DISCUSSION

The XRD pattern of the prepared γ-Al_2O_3 at 800°C is shown in Fig.1. The appearance of peaks in Fig.1 illustrates tl crystalline nature of the prepared γ-Al_2O_3 Absence of peaks is usually associated wi amorphous γ-Al_2O_3 . Fig.1 also reveals th the prepared γ-Al_2O_3 is pure. This report consistent with JCPDS file number 10-42 (Table 1). Proszki computer program fo lattice parameter refinement reveals that tl prepared γ-Al_2O_3 is cubic wit a=b=c=7.5911A°. Fig.2 shows the spheric nature of the particles of size less than abo 100nm. The mean-particle size diamet obtained from TEM micrograph (Fig.3) 85nm. The result shows that the prepared Al_2O_3 is a nanostructured material. Tl crystalline nature of the γ-Al_2O_3 is confirm from the Electron Diffraction (ED) patte shown in Fig.4. The presence of white spots Fig.4 shows the crystalline nature of the Al_2O_3. Amorphous materials do not exhi white spots on ED pattern. It is observed tl heat-treatment of the γ-Al_2O_3 up to 900 results in the phase transformation of γ-Al_2 to α-Al_2O_3 which crystallizes in trigor geometry as shown in Fig.5. This conclusi about geometry is consistent with JCPDS fi number 10-173 (Table2). The latti

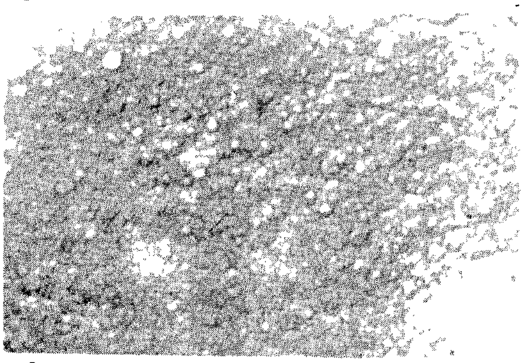

Fig. 2

The SEM micrograph of γ-Al_2O_3 prepared from Al(acac)$_3$ at 800°C by sol-gel method

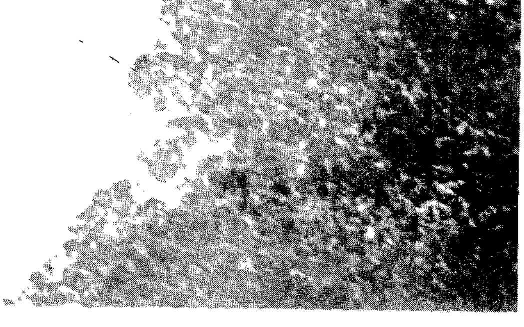

Fig. 3

The TEM micrograph of γ-Al_2O_3 prepared from Al(acac)$_3$ at 800°C by sol-gel method

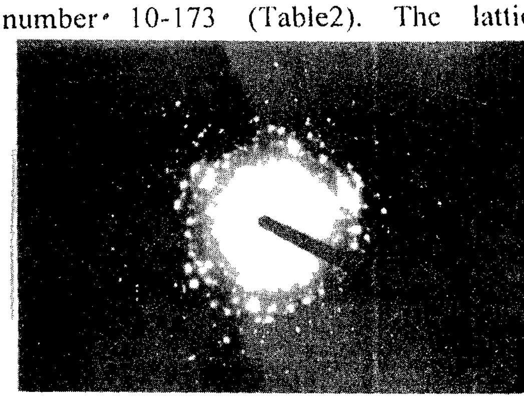

Fig. 4

The ED pattern of of γ-Al_2O_3 prepared from Al(acac)$_3$ at 800°C b sol-gel method

parameters of α-Al_2O_3 are obtained as a 4.7665A°, c = 12.987A°. The mean-partic size diameter of the α-Al_2O_3 is obtained fro the TEM micrograph (Fig.6) to be 120nm. T larger mean-size of the particles obtain for α-Al_2O_3 as compared with those of γ-Al_2 is strongly related to the coagulating nature the alumina particles at higher temperature 900°C. However, α-Al_2O_3 remains crystalli at 900°C (Fig.7). γ-Al_2O_3 with smal crystallites has a higher BET surface area $120m^2/g$ than α-Al_2O_3 ($104m^2/g$). The B

Table 2. Joint Committee on Powder Diffraction Standard (JCPDS) file number 10-173 for α-Al_2O_3 (Trigonal; a = 4.758 A° ; c = 12.991; λ = 1.5405).

d (A°)	Int	hkl
3.479	75	012
2.552	90	104
2.379	40	110
2.165	<1	006
2.085	100	113
1.964	2	202
1.740	45	024
1.601	80	116
1.546	4	211
1.514	6	122
1.510	8	018
1.404	30	124
1.374	50	030
1.337	2	125
1.276	4	208
1.239	16	1,0,10
1.2343	8	119
1.1898	8	220
1.600	<1	306
1.147	6	223
1.3282	2	311
1.1255	6	312
1.1246	4	128

surface area results show that the surface area of the particles of alumina depends on the temperature of preparation as well as nature of the particles contained therein. It is also significant to show that poor quality alumina is obtained when water is added to the aluminum acetylacetonate-ethyleneglycol-methanol system at 800°C (Fig.8). Fig.8 also shows very large particles due to coagulation of the particles arising probably from the easy diffusion of the particles into one another. The presence of water tends to create room for

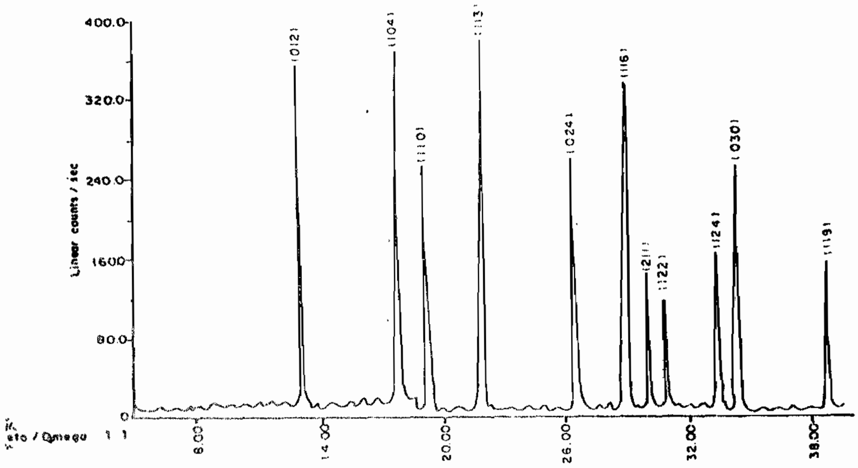

Fig. 5 The XRD pattern of of α-Al_2O_3 prepared from $Al(acac)_3$ at 900°C by sol-gel method

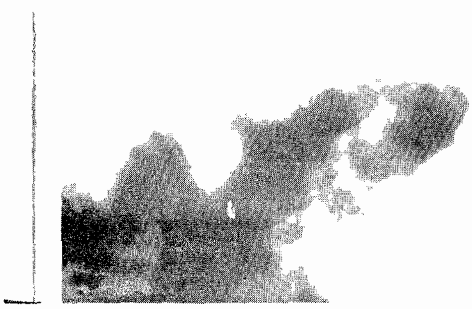

Fig. 6
The TEM micrograph of α-Al_2O_3 prepared from $Al(acac)_3$ at 900°C by sol-gel method

Fig. 7
The ED pattern of α-Al_2O_3 prepared from $Al(acac)_3$ at 900°C by sol-gel method

Fig. 8
The SEM micrograph of γ-Al_2O_3 prepared from $Al(acac)_3$ in the presence of water at 800°C by sol-gel method.

such a diffusion resulting in very large po size of particles as seen in Fig.8.

CONCLUSION

γ-Al_2O_3 has been prepared fro aluminum acetylacetonate-methano ethyleneglycol system for the first time. T prepared γ-Al_2O_3 has nanocrystalline partic and is cubic at 800°C. Poor quality γ-Al_2O_3 obtained when water is present in the syste

ACKNOWLEDGMENTS

The author is thankful to TWAS, Ita for a fellowship to India. He al acknowledges Professor C.N.R. Rao hosting him and for the provision of faciliti

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