FTIR STUDIES ON REACTIVE PLASMA SYNTHESIZED ZrO2 NANOPARTICLES

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Abstract

Nano-size ZrO_2 powder was synthesized via reactive plasma processing from micron size zirconium hydride powder. The synthesized ZrO_2 powder was characterized by FTIR for phase analysis. The synthesized powder consists of a mixture of tetragonal and monoclinic phases of zirconia. The characteristic vibrational bands for tetragonal phase and monoclinic phase were identified by FTIR analysis.

Keywords: *Nano-crystalline material, Reactive plasma processing, Zirconium oxide*

Introduction

Zirconium oxide is a refractory material, which offers high strength, high fracture toughness, excellent chemical resistance and low thermal conductivity. These characteristics of ZrO_2 make it highly functional in the field of structural, mechanical and high temperature applications [1-5]. Pure zirconia exists in three polymorphic modifications-namely, monoclinic, tetragonal and cubic phases [6-10]. Monoclinic form of zirconia is the thermodynamically stable phase at room temperature, whereas tetragonal and cubic are stable at high temperatures. Monoclinic zirconia transforms reversibly to the tetragonal phase when heated to about 1170°C and as the temperature exceeds 2370°C, the tetragonal phase transforms into the cubic phase [11 – 15].

The current paper reports synthesis of nano-crystalline ZrO_2 by reactive plasma processing (RPP) and its characterization by FTIR spectroscopy. Reactive plasma processing (RPP) is a novel technique, which takes the advantage of the high temperature and high enthalpy of the thermal plasma jet to effect 'in-flight' chemical reactions in the presence of reactive gas to synthesize nano-sized powders of advanced ceramics, novel coatings and convert minerals and industrial wastes to value added materials. The technique can also be used to produce spherical powders of metals and ceramics for special applications. The high quench rate, which is characteristic of the process, favours homogeneous nucleation resulting in nano-sized particles. The major advantages of the reactive plasma processing includes versatility, short processing time, large throughput, adaptability to process thin films and coatings [16 – 20].

The goal of the present work is to synthesize nano-crystalline ZrO_2 using ZrH_2 powder by reactive plasma processing and carryout phase analysis using FTIR spectroscopic technique.

Materials and Methods

A combination of Ar and N_2 was used as the plasma gas. The DC arc was struck between the cathode and anode. Input power to the plasma torch was varied from 10-16 kW by controlling gas flow rate and arc current. The range of operating parameters is provided in Table 1. The precursor material used was ZrH_2 powder with 99.8% purity from CERAC, USA. ZrH_2 powder (38-53µm size). The precursor powder was injected into the plasma jet by using argon as the carrier gas. Oxygen gas was introduced 10mm downstream of the exit of the plasma torch. ZrH_2 dissociates to form Zr particles and hydrogen gas in the plasma jet that are subsequently converted to ZrO_2 and water vapor, which escapes along with the exhaust gas stream [21 – 25]. The nano-crystalline ZrO_2 formed settles on the walls of the reactor and collection chamber as fine powder. Powders collected from various regions of plasma reactor were characterized by FTIR spectroscopy.

Characterization

The zirconia powder samples collected from different locations of the plasma reactor were characterized by FTIR technique. Fourier transform infrared spectroscopy (Spectrum one: FTIR spectrometer, Perkin Elmer, 450-4000 cm^{-1}, KBr pellet technique) was used to identify the vibrational features of the samples.

Results and Discussion

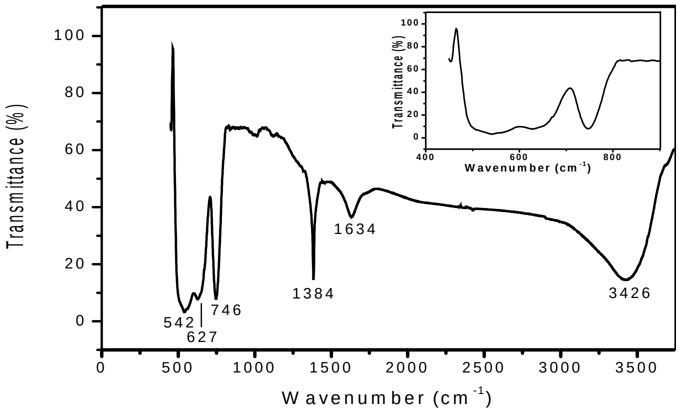

Fig.1 FT-IR spectra of reactive plasma synthesized nanocrystalline ZrO_2- sample C

Figure 1 shows the FTIR spectrum of sample synthesized at 16 kW. Bands observed at 3428 cm^{-1} and 1634 cm^{-1} are assigned to the bending and stretching vibrations of the O-H bond due to absorbed water molecules [26 – 30]. The band at 1384 cm^{-1} is attributed to the absorption of non-bridging OH groups. The sharp band at 746 cm^{-1} is the characteristic of m-ZrO_2. A broad band with a peak at 542cm^{-1} and a shoulder at 627cm^{-1} is ascribed to Zr-O vibrations of t-ZrO_2 [31 – 35] . However, weak peaks in the range 460 cm^{-1} to 690 cm^{-1} corresponding to m-ZrO_2 are not observed due to dilution effect (presence of tetragonal (44%) phase). A similar observation was reported by Chen at al for pure zirconia nanoparticles [36 – 40].

Conclusion

Nano-crystalline zirconium oxide particles have been successfully synthesized by reactive plasma processing and reported in this article. The synthesized sample was characterized by FTIR technique for phase analysis.. It is found that the sample contains higher amount of monoclinic phase compared to tetragonal phase due to more amount of larger-size particles.

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