Thermal Plasma Processing of Spherical ZnO Nano Powders

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Abstract. Nano Zinc oxide (ZnO) is gaining more importance and its demand is keep increasing in recent decades because of its potential applications in energy, optoelectronic and sensor fields. To address this issue, the bulk synthesis of nano ZnO is demonstrated in the present article by the thermal plasma processing. Nano sized ZnO spherical particles of size 3 – 25 nm evidenced by transmission electron microscopy (TEM) have been synthesized from micron size zinc metal powder precursor. Surface chemical composition and presence of oxygen vacancies are analyzed by XPS and reported for future application.

INTRODUCTION

Zinc oxide is a direct band gap semiconductor of wide band gap (~3.37 eV) and larger excitation energy of 60 meV [1]. In nano dimension ZnO exhibits good electrical, electronic, electrochemical, and photo catalytic properties. These inherent properties makes ZnO as a useful material to design field emission displays, supercapacitors, solar cells, gas sensors, nano-photonic devices, gas sensors, piezoelectric transducers, varistors, phosphors, and transparent conducting films [2 – 4]. ZnO has been reported as an alternate photocatalyst for TiO_2 to treat wastewater contaminated with organic and inorganic pollutants due to low cost and non- toxicity [5]. These potential applications of nano structured ZnO demanded materials scientists to synthesize nano ZnO by variety of methods including precipitation, facile synthesis, spray-pyrolysis, hydrothermal, sol-gel, thermal evaporation and mechanochemical synthesis [6 – 13].

The present work reports the synthesis of nano ZnO particles by thermal plasma processing and its structural, morphological and X-ray photoelectron microscopic characterization. Thermal plasma processing 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 [14].The high quench rate, which is characteristic of the process, favors 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.

EXPERIMENTAL PROCEDURE

Thermal Plasma Synthesis of Nano ZnO Powder

The nano sized Zno powder is synthesized using a thermal plasma reactor. The main component of the plasma reactor is a 40 kW DC non-transferred arc plasma torch, which generates high temperature and high velocity plasma jet. It also has provision to feed plasma forming gas and precursor powder. The Zn metal powder (38-53 μm size, 99.8% purity, CERAC, USA) is stored in a powder feeder and is injected into the plasma jet through a side port provided at the anode of the plasma torch. Reactive gas (oxygen) is injected downstream the plasma jet, by means of a gas injector ring fitted on the torch head section. The product can be collected from various segments of the plasma reactor. The description and working of the plasma reactor is well documented in our earlier work [15]. The operating parameters of the plasma reactor are given in table 1.

TABLE 1.Operating Parameters of the Plasma Reactor

Operating Parameter (with Unit)	Values
Torch Input Power (KW)	10
Arc Voltage (V)	40
Arc Current (A)	250
Primary Plasma Gas(Ar) Flow Rate (LPM)	30
Secondary Gas(N_2) Flow Rate (LPM)	2
Carrier Gas (Ar) Flow Rate (LPM)	10
Reactive Gas (O_2) Flow Rate (LPM)	20
Precursor Powder (Zn) Feed Rate (g/min)	5

Powder Characterization

X-ray diffraction (XRD) patterns of the synthesized samples were recorded on a Bruker D8 advanced X-ray powder diffractometer. Particle size and morphology of the samples were carried out using JEOL transmission electron microscope (JEM 2100F, Japan) operated at 200 KV. 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 sample. The BET surface area measurement was carried out using a Micromeritics TriStar 3000 at 77 K with N_2 as adsorbate. The optical properties of ZnO nanoparticles were studied using UV-Vis spectroscopy (Perkin Elmer, LambdaIs USA). The x-ray photoemission spectra were measured on VSW x-ray photoelectron spectroscopy (XPS) set-up using Al K_α radiation with a spectral resolution of ~0.6 eV at 530 eV.

RESULTS AND DISCUSSION

XRD, TEM, FTIR, UV - Visible and XPS Studies

The XRD pattern of the plasma synthesized sample is shown in Fig. 1(a). The XRD pattern coincides with the JCPDS card no 361451 and confirms the formation of ZnO particles with wurtzite structure. The average crystallite size has been calculated using Scherrer's formula and is found to be 20 nm. The broadening of the XRD peak and crystallite size determined showed the formation of nano sized particles. The FTIR transmittance spectrum of the reactive plasma synthesized ZnO nano particle is show in Fig.1 (b). The spectrum clearly depicts the characteristic vibrational bands of Zn – O at 457 cm^{-1} and 545 cm^{-1}. This result further confirms the formation of ZnO particles.

The TEM image of the plasma synthesized ZnO powders are shown in Fig. 1(c). The image shows that the ZnO particles have spherical morphology and nano size. The synthesized ZnO particles size varied from 3 – 25 nm. The TEM result confirms the findings of XRD result. The Brunauer–Emmet–Teller (BET) nitrogen adsorption–desorption analysis showed that the specific surface area of the synthesized nano ZnO powder is (24 m^2 / g), which is good enough for photocatalytic and other chemical reactions at the surface of the particle. The UV – visible absorption spectrum of the reactive plasma synthesized nano ZnO powder is shown in Fig. 1(d). It showed that the ZnO powder has good absorption in UV region and absorption edge at 400 nm.

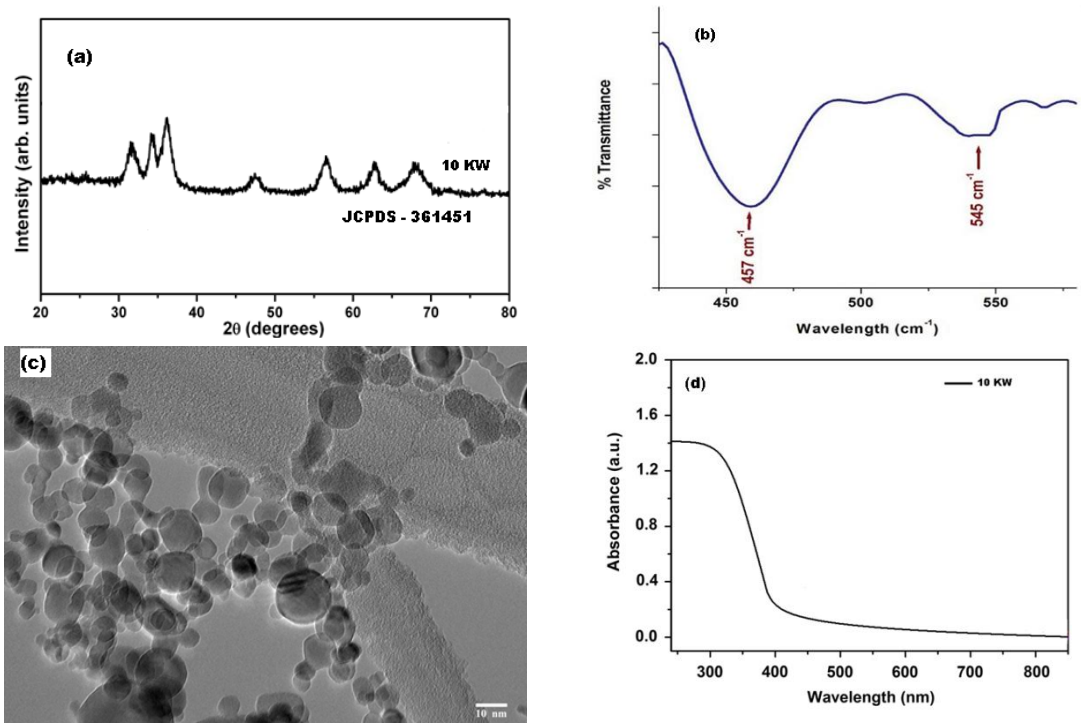

FIGURE 1. XRD, FTIR, TEM and UV- Visible Studies of Thermal Plasma Synthesized Nano Zno Powder. (a)XRD pattern, (b) FTIR Spectrum, (c) TEM image and (d) UV – Visible spectrum

The survey scan XPS spectrum of the plasma synthesized and annealed ZnO powders are shown in Fig. 2 (a). The high resolution spectrum of O1s peak of ZnO powder is shown in Fig. 2(b) and is de-convoluted into two peaks. The peaks centered at 530 eV and 531 eV are attributed respectively to Zn – O and Zn – O vacancy [16]. The concentration of oxygen vacancy in the samples is determined by calculating the ratio of intensity of the peaks that corresponds to Zn – O (530 eV) and Zn – O vacancy (531 eV). For the synthesized ZnO powders this ratio was found be to 0.98. This shows that the plasma synthesized sample has considerable amount of oxygen vacancy at the surface. These oxygen vacancies are attributed to the inherent nature of reactive plasma processing [17].

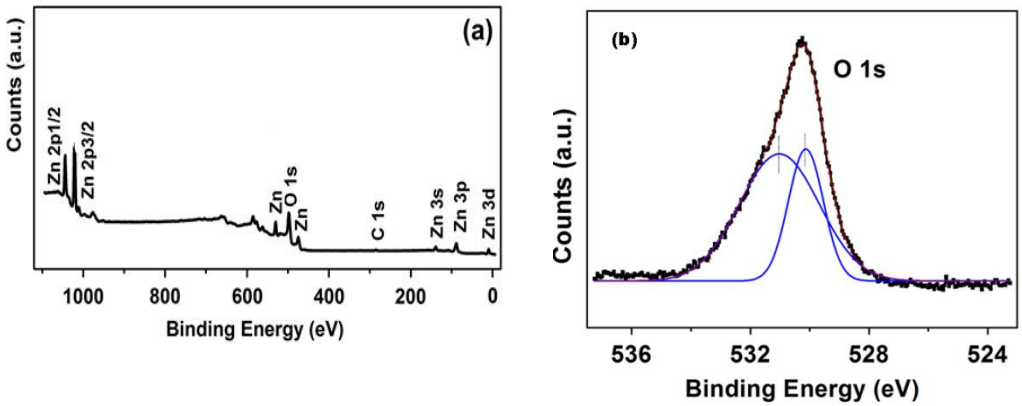

FIGURE 2. XPS Spectra of Thermal Plasma Synthesized Zno Nao Powder: (a) Survey Scan XPS Spectrum of ZnO, (b) High Resultion Spectrum of O1s Spectrum.

CONCLUSION

This paper demonstrates the formation of nano sized ZnO particle from Zn metal particles of micron size. The crystallite structure of the thermal plasma synthesized ZnO particles was characterized by powder XRD and FTIR spectroscopy. The TEM studies clearly revealed the spherical morphology of the particles and size varied from 3 – 25 nm. Further XPS spectroscopy showed the presence of surface oxygen vacancies. The BET measurements showed that the synthesized ZnO particle have high surface area (24 m^2/g).

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