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Effect of Particle Size on Ammonia Sensing Response of Zinc Oxide

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Abstract—*Efficient gas sensor was made with zinc oxide nanoparticles synthesized by mechanical alloying using high energy planetary ball mill. As received and milled zinc oxide nanoparticles were characterized with X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and UV-Vis Spectroscopy techniques. Morphological study was carried out by using Transmission Electron Microscopy (TEM). The particle size of ZnO nanoparticles was reduced from micron to nano meter range after milling for 45h. The Ammonia gas sensing response of as received and milled ZnO nanoparticles was studied from I-V characteristics plots obtained for different concentration of ammonia (50 to 600 ppm). The sensitivity of zinc oxide was observed to increase with decrease in particle size from micron to nanometer.*

Keywords*: Ammonia, Nanoparticles, Sensing, Ball Mill.*

1. INTRODUCTION

The environmental safety and the human health is the main concern of scientific research in these days. In most of the industries, the working environment has many harmful and toxic gases such as ammonia, hydrogen sulphide, carbon dioxide, Carbon monoxide etc. [1]. Among these gases ammonia is most widely used gas in industries (agriculture, refrigeration, fire power plant, fertilizer, food and medical etc [2, 3] and R&Ds. Ammonia affects both externally and internally to human body. The exposure of Ammonia can cause lung diseases, skin irritation, skin etching and even burning. Even 25 ppm exposure of ammonia for 8 hours can affect the human health [4]. For the safe working environment proper monitoring system must be there [5]. The monitoring includes the gas sensor and the pre-warning system to detect even the small concentration of ammonia. Metal oxide gas sensing materials are most prominent in the range of gas sensors due to their interesting gas response [6-8] Among the class of transition metal oxides, the nanomaterials like SnO_2, TiO_2, CuO, In_2O_3, Fe_2O_3 and ZnO [9] etc. and their nano composites are under the growing research due to their strong sensitivity upon adsorbed gases to their surface, and other promising properties with the change in particle size [10]. The metal oxide nanoparticles are synthesized with different synthesis methods such as Sol-gel method, Auto combustion method, co-precipitation method, Vapor transport method and mechanical alloying etc [11].

In the present research work as received ZnO particles (0.5-1.0µm) are reduced to nanosize range (20nm) by mechanical alloying. Further these nanoparticles are used for sensing of ammonia with different concentration.

2. MATERIALS AND METHODS:

Mechanical alloying technique was used for the synthesis of ZnO nanoparticles. The as received ZnO powder with ball to powder ratio of 10:1 was used as starting material which further milled for 45 hours at the rotation speed of 150 rpm.

Both the samples of zinc oxide, viz. as received ZnO and as milled ZnO, were ultrasonically prepared by dissolving 30mg of the sample in 8ml of HCl. The solutions of zinc oxide thus obtained were spin coated on porous silicon substrates.

3. CHARACTERIZATION

Phase identification of as received ZnO and milled ZnO nanoparticles was done by X-Ray Diffraction (XRD) using Bruker AXSD8 differactometer with Cu-kα radiation. The average crystallite size of ZnO particles was measured by the X-ray line broadening technique, employing Scherrer's formula. The UV-Vis absorption spectra of as received ZnO and milled ZnO nanoparticles were studied using LAB India UV-3200 spectrophotometer with 10mm path length in the 200-800 nm wavelength range. The Fourier Transform Infrared Spectroscopy (FTIR) spectra was obtained 'milled' zinc oxide nanoparticles in the range 400 to 4000 cm^{-1} was recorded using Thermo Scientific Smart Omni-Transmission Nicolet iS10 spectrometer. The morphology of milled ZnO nanoparticles was carried out by Transmission Electron Microscopy (TEM: JEOL-JEM 2100).

4. RESULT AND DISCUSSIONS

The indexed XRD pattern of as received and milled ZnO nanoparticles are shown in Fig. 1. From the results, it can be inferred that the zinc oxide powder in 'as received' and milled conditions is showing only the peaks corresponding to ZnO phase (JCPDS Reference code: 36-1451) [12]. As expected, the degree of crystallinity is further increased with increase in milling time. The crystallite size of zinc oxide powder is observed to decrease with increase in milling time. It decreases from micron size range to 20 nm size after milling for 45 h [13].

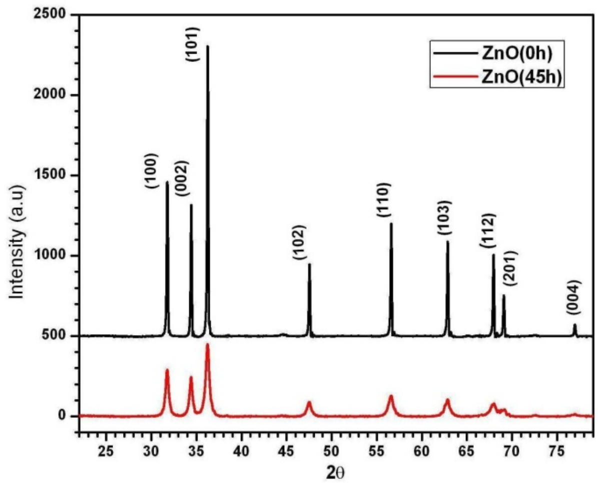

Fig. 1: Showing the effect of milling on the XRD pattern of as received ZnO (0h) and milled ZnO powder (45 h).

Fig. 2 shows the Infrared spectroscopic spectra of as received and milled ZnO nanoparticles. The strong peaks at 470 cm^{-1} and 3417 cm^{-1} are attributed to the vibration of Zn-O bond and hydroxyl group respectively.

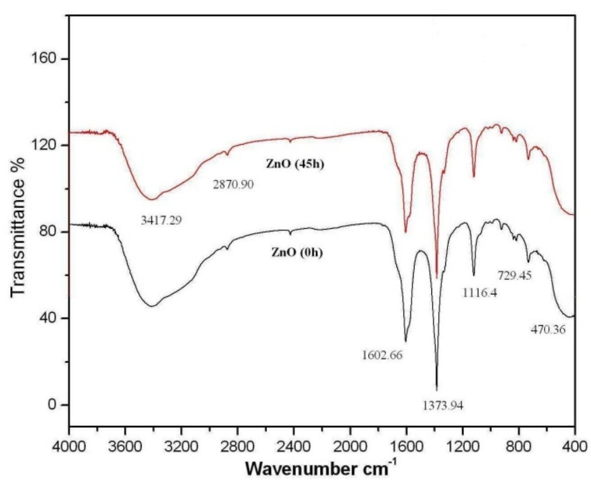

Fig. 2: Typical FTIR Spectra of ZnO nanoparticles

The peaks at 729 cm^{-1} and 1116 cm^{-1} corresponds to C-C-O and C-O stretching vibrations respectively. The peak at 1373 cm^{-1} corresponds to the vibration of COO^- stretching. The peaks at 1602 cm^{-1} and 2870cm^{-1} corresponds to the –C═O and $–CH_3$ vibrations respectively [14].

The UV-visible spectra of 'as received' and 'milled' ZnO nanoparticles are shown in Fig. 3. It is observed from the spectra that the absorption band at 334nm (π-π* transition) has shifted towards right with the milling of zinc oxide. This might be due to the change in particle size and lattice parameters of zinc oxide nanoparticles after milling of 45 h [13].

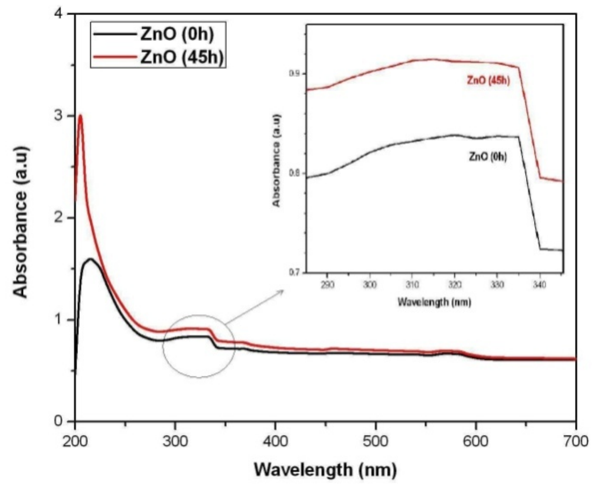

Fig. 3: UV-Vis Spectra as received ZnO (0h) and milled ZnO (45h)

The TEM micrographs of 'as-received' and milled zinc oxide powder are shown in Fig. 4. In the 'as-received' condition, the particles seem to have agglomerated morphology with particle size in the range of 0.1-0.5 µm. After milling for 45 h, the particles size is observed to decrease to nano size (45-50 nm).

original resistance in air. The relation for S is as: S = Rg-Ra/Ra, where Ra and Rg are the resistance of sensing materials in air and in a target gas medium (ammonia), respectively. Fig. 5 shows the variation of sensitivity of zinc oxide powder in the as received and milled conditions for various concentration of ammonia gas (50, 100, 200, 300, 400, 500 and 600 ppm). It can be seen that in both the materials, sensitivity tends to saturate with increase in concentration of ammonia gas. The response of a sensor depends on removal of adsorbed oxygen species in reaction with a testing gas and generation of electrons. For small concentrations of gas, there is a lower surface reaction occurred due to lower coverage of gas molecules on the grain surface. An increase in gas concentration increases the number of the surface reactions and sensitivity. At higher concentrations, saturation is reached due to full surface coverage with test gas molecules. The best sensitivity was observed at room temperature for milled zinc oxide nanoparticles than as received micron sized zinc oxide powder. Sensing material that had smaller particle size and larger specific surface area was observed to had larger gas sensitivity and vice –versa [15].

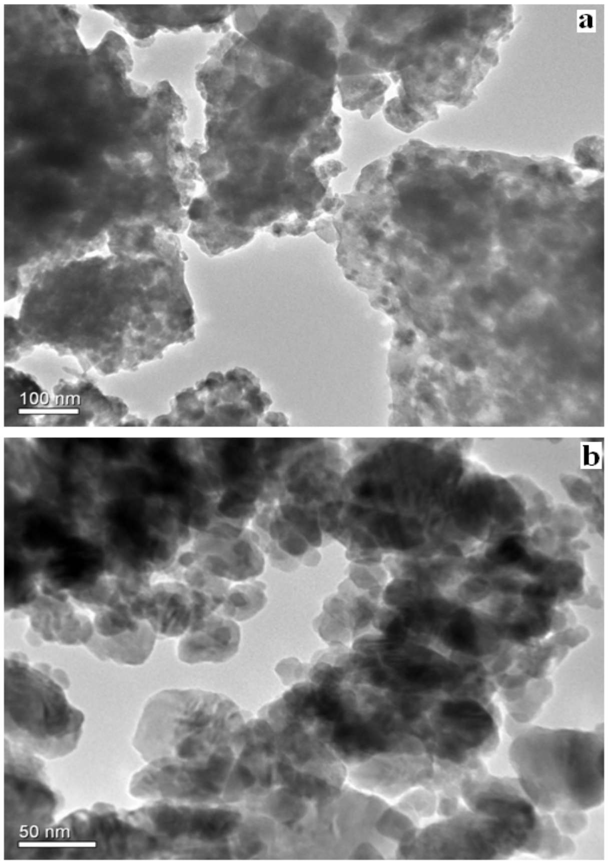

Fig. 4: Typical TEM Micrograph of a) As Recived ZnO b)'Milled' ZnO Nanoparticles.

Gas response (S) is defined as the ratio of the change in resistance of the sensor on exposure to the target gas to the

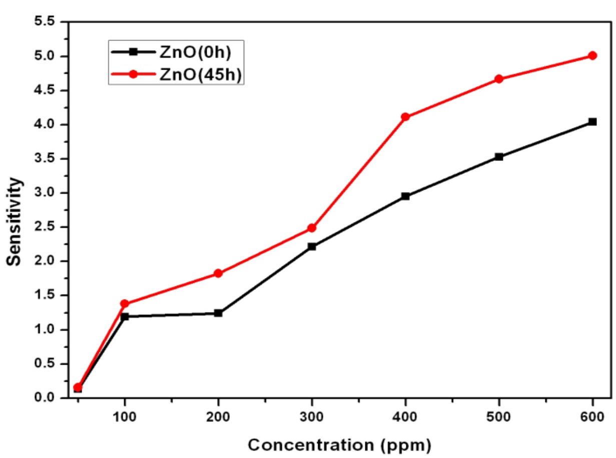

Fig. 5: The Correlation between Gas Concentration and Sensitivity of As Received (0h) and Milled (45h) Zinc Oxide Powder in Air Atmosphere at Room Temperature.

5. CONCLUSIONS

Zinc oxide nanoparticles having crystallite size of about 20 nm were obtained by mechanical alloying of micron size as received zinc oxide powder. From TEM study, zinc oxide powder is observed to have multiple shape morphology after milling for 45 h. The XRD pattern confirmed the purity and crystallinity of zinc oxide powder in both before and after milling the powder. From the sensitivity data of both the samples, sensitivity is observed to increase with increase in ammonia concentration. The minimum concentration of 50 ppm is detected successfully at room temperature.

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