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APPROACH AND THEIR DENSIFICATION
BEHAVIOUR AND VARISTOR PROPERTIES**

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Functionalit of ZNO Based Nanopowders Using a Non-Hydrolytic Sol Gel Approach and Their Densification Behaviour and Varistor Properties

Prasanta Kumar Nayak¹ Salil Kumar Malla²

¹Research Scholar, CMJ University, Shillong, Meghalaya

²Research Scholar, CMJ University, Shillong, Meghalaya

Abstract – Hexagonal nanocrystalline varistor grade Zno particles with size 50 nm and the particular surface territory of 28 m²/g have been ready by non-fluid gelation procedure including diethylene glycol and triethanolamine. The as-arranged varistor nanopowders were dissected with the backing of XRD, TG/DTA, FTIR, TMA, SEM what's more TEM. Varistor plates were manufactured by pressing and their densification was examined at 850, 950, 1050 and 1150°C. The advancement of varistor microstructures, degree of grain development and the impact of microstructure on the I-V properties were investigated and exhibited.

INTRODUCTION

The interest for electrical vitality expands relentlessly in creating countries like India which has distinct arrangements to set up more nuclear and hydroelectric power plants and immense networking of power dissemination lines alongside small scale power stations in not so distant future. This will bring the varistor business to the spotlight furthermore the need to create superior, high vitality varistors.

The business has settled the vitality taking care of capacity of 600 V/cm³ as focus for the new-era ZnO-Bi₂O₃ varistor hinders inside the confined volume. The execution of ecological security in the mechanical part has likewise called for a suitable 'eco-design' for the new varistor items. These issues get the greater part of the consideration in the design and improvement of state-of-the-craft varistors.

The quick prerequisites for this worry were little estimated, light weight, sintered ZnO varistors holding fine-grained dynamic ZnO with homogeneous appropriation of protecting grain limits. Late reports demonstrate that the utilization of nano-grained ZnO varistors can tackle the current confinements in varistor field.

Henceforth, varistors improvement including nanoparticles was taken up by numerous material researchers and combination of varistors evaluation nanoparticles by novel chemical strategies like sol-gel, aqueous, citrate gel decomposition, also microwave supported blaze ignition what's more urea

decomposition procedures have been variously endeavored. Homogeneous blending of low level dopants and added substances, low temperature densification, diminishment in ZnO grain size and three to four times expansion in the varistors execution were a couple of favorable circumstances figured it out at the point when nano size crude material was utilized.

Since sol-gel is a basic strategy for nanoparticles blend, we utilized the same for get ready varistor nanoparticles. Notwithstanding, when a watery sol-gel union was requisitioned getting multi-segment varistor grade nanopowder, the most paramount antecedent added substances, in the same way as bismuth and antimony salts, get hydrolyzed in a flash which brings about favored precipitation what's more non-homogeneous powders.

Thusly, a non-hydrolytic sol gel blend was performed in this work including glycol and amine forerunners. The densification of varistor evaluation nanopowders at diverse temperatures what's more the relating microstructures and their impact on the breakdown voltage and non-linearity coefficient were examined and reported.

EXPERIMENTAL

Non-hydrolytic blend of nanopowders -Varistor organization comprising of 94 mol% ZnO, 3 mol% Bi₂O₃ and 1 mol% each of Cr₂O₃, CoO and Sb₂O₃ each, was ready in this work. The favored sythesis was discovered to be useful for comprehension the

fundamental varistor properties. Thermogelation was the system utilized for making varistor gel. In a three necked reflux carafe fitted with a condenser, 1.0 M zinc nitrate (99 %, Merck) forerunner result ready in 100 ml iso-propanol (99.9 %, Ranbaxy) was refluxed utilizing a lab warming mantle.

Bismuth nitrate (1.52 g) (99 %, CDH) result, ready in diethylene glycol (DEG) medium (25 ml), was all the while included. While refluxing, triethanolamine (50 ml) (99 %, Ranbaxy) was gradually brought into the forerunner nitrates. The volume degree of the DEG, triethanolamine and isopropanol was kept up as 1 : 2 : 6. The forerunner result turned to a transparent gooey gel in the wake of refluxing for 105 minutes. The resultant forerunner gel was gathered furthermore consolidated further at 100°C in an electrical stove until a thick gooey inflexible mass was acquired.

It was then exchanged to a 1000 ml measuring glass and warmed at 270°C for 2 h at a rate of 3°C/min. Exceptionally permeable dark Zno based froth was at long last acquired. The dark froth was then ball processed in iso-propanol medium (300 ml) for a time of 2 h. So acquired dark powder was washed with iso-propanol, dried at 70°C and after that subjected to control calcination at 500°C for 2 h at a warming rate of 3°C/min. Nanocrystalline varistor evaluation powder was at last gotten. It was saved for the creation of varistor plates.

Creation of varistors - 1.5 g of varistor evaluation Zno based nanopowder was weighed and compacted at a weight of 80 Mpa into a plate with measurements 12 mm diameter and 2 mm thickness utilizing a water powered press (Lawrence & Mayo, India). The plates were sintered at temperatures going between 850°C and 1150°C in an electrically warmed silicon carbide heater. The warming rate was controlled by Librathern temperature software engineer. In all cases, after sintering, the heater was cooled at typical heater cooling rates. The as created varistor circles were cleaned also electroded with silver and warmed up to 600°C for 10 min, before completing the electrical estimations.

Characterization - The as ready varistor grade Zno based antecedent gel and the gel calcined at 270°C and 500°C were portrayed for the structural gimmicks, warm security, stage examination, mass surface region and morphology utilizing FTIR, TG/DT, XRD, BET, SEM and TEM expository instruments individually.

FTIR spectra were recorded on a Nicolet Magna 560 FTIR (USA) spectrophotometer over the ghostly scope of 4000–400 cm⁻¹ by Kbr pellet system. Warm decomposition conduct was broke down at a steady high temperature stream of 10°C/min in air climate up to 1200°C utilizing Shimadzu, TG/DTA-50h (Japan) instrument.

The varistor circles created from the nanopowders were described for the densification, thickness estimations, sintered microstructural gimmicks and current voltage (I-V) properties. Densification bends were acquired utilizing thermo-mechanical analyzer (TMA-60h Shimadzu, Japan). Round and hollow examples of size 6 mm stature and 5 mm diameter were utilized for the TMA investigation.

The test was performed up to 1300°C at a warming rate of 10°C/min. A dead heap of 0.3 g was given for guaranteeing the contact between the measuring test and the test. The direct shrinkage degree ($\Delta L/L_0$) of the example as for temperature was measured. The green thickness was dead set from the dimensional estimations. Densities of the sintered varistor pellets were measured by Archimedes method.

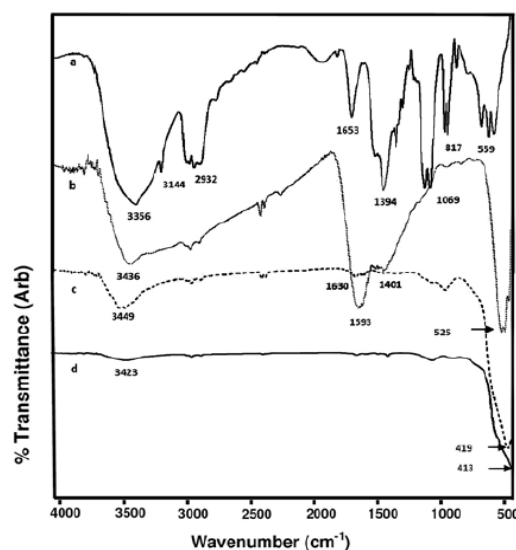


Figure: FTIR transmittance spectra of: varistor grade precursor gel (a), gel calcined at 270°C (b), gel calcined at 500°C (c) and ZnO powder (d)

I-V estimations Electrical execution of the sintered varistor was measured utilizing a beat mode D.c. power supply having an inherent power source (600 Volts, Digitronics, India) with a current farthest point of 100 mama. The measure of current passing through the cross segment of a specimen was checked for each 5 volts and the current thickness vs. electric field bends were plotted. From the I-V bends the breakdown voltage (Vb) and the nonlinearity coefficient (α) were resolved.

RESULTS

Characterization of antecedent gel and varistor grade Zno nanoparticles - FTIR examination the complexation of the antecedent metal nitrates with TEA and DEG. The wide extending groups at 3356, 3436 and 3449 cm⁻¹ in the as-arranged varistor grade antecedent gel and the specimens calcined at 270°C and at 500°C, individually, demonstrate the vicinity of reinforced and free hydroxyl bunches. The arrangement of transmittance tops at 1653 cm⁻¹, 1630

cm^{-1} and 1593 cm^{-1} are demonstrating the bowing modes of -OH groupings. The antecedent gel displays the trademark extending groups of nitrate grouping at 1394 cm^{-1} and 817 cm^{-1} . The nitrates decompose amid calcinations and accordingly these crests are getting vanished in the calcined specimens.

To affirm the creation of crystalline ZnO, the FTIR spectra of the antecedent gel and the calcined powders were contrasted and the standard ZnO powders. In stage unadulterated ZnO, extremely serious tops are watched in the reach $600\text{--}400\text{ cm}^{-1}$. Such serious trademark tops are nonattendant in the forerunner gel demonstrating that there is no immediate structuring of ZnO cores amid refluxing. It is as opposed to the prior distributed reports where immediate structuring of ZnO was accounted for in reflux responses directed in the basic media.

Nonetheless, it is watched that the spectra taken for the gel has a red movement in the scope of $600\text{--}400\text{ cm}^{-1}$ showing the complex establishment in the middle of zinc and the TEA-DEG complexing operators. The various low extreme crests in the scope of $500\text{--}400\text{ cm}^{-1}$ for 270°C calcined gels might speak to the ZnO essential seed cores establishment. Calcination at 500°C results in the development of a solid top at 419 cm^{-1} comparing to the trademark crest of ZnO.

Sintering conduct of nanopowder compacts - The dilatometric bend demonstrates the on-set densification temperature as 780°C affirming the sintering effectiveness of the nano size varistor particles. The densification was advancing up to 1024°C at a speedier rate. A little venture at 830°C is additionally watched showing the framing of polished fluid stage because of Bi_2O_3 . We have prior reported the sintering conduct of spread granulated mechanical evaluation varistor powder where the on-set densification temperature was watched just at 846°C . Leeway of no less than 100°C is evidently seen in the on-set densification temperature when the nanopowder is utilized.

Electrical properties of sintered compacts In ZnO- Bi_2O_3 varistors, the electrical execution change determinedly with the amount of mass ZnO grains and the thickness of the grain limit polished stage. The change in breakdown voltage and nonlinearity coefficient of the varistors arranged by means of gel inferred nanopowder as for the sintered thickness and grain size is abridged in Table 2. The straight build in the grain size from 950 to 1150°C demonstrates a diminishing pattern in the ostensible breakdown voltage. This is as per the exact relationship reported for the breakdown voltage and ZnO grain size, $V_b = m d/G V_{gb}$, where V_b is the breakdown field, d is the specimen thickness, G is the normal grain size and V_{gb} is the voltage over a solitary potential obstruction

and the coefficient m is averaging the potential obstruction conveyance.

CONCLUSIONS

High surface region nano crystalline varistor grade ZnO based particles with platelet morphology and a normal particle size of 50 nm have been attained through a non-hydrolytic sol-gel method. The varistor nanoparticles have preferences as on-set densification at 746°C , one dimensional ZnO grains at 850°C , a normal grain size $<4\text{ }\mu\text{m}$ at 1050°C and $<8\text{ }\mu\text{m}$ at 1150°C individually with $>95\%$ t.d. The nano outline varistors with thin grain size conveyance have higher breakdown voltage and non-linearity when contrasted with the reported traditional varistors, which were ready from the splash dried varistor granules.

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