



Microstructure and Electrical Properties of Low-Voltage Barium Titanate Doped Zinc Oxide Varistor Ceramics

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Abstract: In the present, varistor ceramics through the combination of zinc oxide (ZnO) with a perovskite material have become widespread because of their unique properties for a wide range of applications in electronic protection devices. Low-voltage zinc oxide (ZnO) varistors with fast response and highly nonlinear electrical properties for overvoltage protection in an integrated circuit are increasingly significant in the application of low-voltage electronics. The present study highlights the interaction between barium titanate ($BaTiO_3$) and ZnO varistors through the employment of solid-state reaction method in the production of low-voltage varistors. The effects of $BaTiO_3$ on the microstructure of ZnO varistors were analyzed through scanning electron microscopy (SEM), energy dispersive X-ray analysis spectroscopy (EDS) and X-ray diffraction (XRD). The EDS analysis and XRD measurements suggest the presence of ZnO and $BaTiO_3$ phases. The electrical properties of $BaTiO_3$ -doped ZnO varistors were examined based on the current density-electric field (J-E) characteristics measurement. The varistor properties showed the nonlinear coefficient (α) from 1.8 to 4.8 with the barrier height (ϕ_B) ranged from 0.70 to 0.88 eV. The used of $BaTiO_3$ additive in ZnO varistors produced varistor voltages of 4.7 to 14.1 V/mm with the voltage per grain boundary (V_{gb}) was measured in the ranges 0.03 to 0.05 V. The lowest leakage current density was 348 $\mu A/cm^2$, obtained at the samples containing 12 wt.% $BaTiO_3$ with high barrier height. The reduction in barrier height with increasing $BaTiO_3$ content was associated with the excessive amount of $BaTiO_3$ phase, hence cause the deterioration

of active grain boundary due to the variation of oxygen (O) vacancies in the grain boundary.

Keywords: Low-voltage, barium titanate, varistors, zinc oxide

I. INTRODUCTION

The used of low-voltage varistors for overvoltage protection in integrated circuits has increased significantly due to high demands in the application of low-voltage electronics. Due to these reasons, it is necessary for the continuous development of ZnO varistors with highly nonlinear electrical properties and energy absorption capabilities at low voltage [1]. In developing this electronic component, the additive plays an important part to modify the defect concentration in the grain boundary layers within the ZnO microstructures. Such notion is due to the varistor performance is very sensitive to the presence of some dopants even though their content is very low [2]. The additive plays a vital role in inhibit grain growth in the application of high voltage varistor and exhibit grain growth for low voltage varistor [3].

Previously, the varistor composition has been developed by Matsuoka [4], with five additive oxides to improve the nonlinear electrical characteristics of Bi_2O_3 -based ZnO varistors. The output of the research is significant, and hence it is still being used as a reference to any other manufacturing technology nowadays. However, the fabrication of varistor with multiple additive oxides caused an increase in the initial cost of production. Moreover, the progress in its development continuously increases the complexity in varistor compositions. In the desire to simplify manufacturing technology, a simple varistor composition with only a single $BaTiO_3$ additive was used as varistor former. The study conducted by Kharchouche [5] shows that the used of $BaTiO_3$ as an additive in ZnO ceramics produced a high nonlinear coefficient (α). When the ceramic materials have a higher value of nonlinear electrical properties, then the varistor is said to approach the ideal varistor [6].

$BaTiO_3$ is a dielectric ceramic and widely used as a capacitor, positive temperature coefficient of resistivity (PTCR) thermistors and electro-optic devices [7]. The resistivity of PTCR thermistors is affected by the temperature, which exhibits a variation in resistance with heat treatment due to the presence of a potential barrier at the grain boundary [8].

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BaTiO₃ as perovskite materials display various interesting properties that make these materials one of today's most attractive and studied dielectric ceramic. Meanwhile, the results acquired from BaTiO₃-doped ZnO ceramic combination are important for capacitor to varistor integration [9]. In the production of the low-voltage varistor, TiO₂ doping is usually applied to increase the grain growth of ZnO. Low-voltage varistors can be obtained by either reducing the thickness of the samples or enhance the size of ZnO grains [10]. Varistor behavior has been studied to occur in ZnO ceramics doped with additives of the large ionic radius where Ba ions showed the largest ionic radius among alkali earth metal oxides which can induced the nonlinearity of ZnO varistors [11], [12]. Therefore, the presence of BaO and TiO₂ within BaTiO₃ is suitable in the fabrication of low-voltage varistors with high nonlinearity.

Moreover, most commercial ZnO varistors were fabricated by using Bi₂O₃ as a varistor former with the addition of various metal oxide to improve their nonlinearity [13]. But, Bi₂O₃ has a few drawbacks due to its volatility at high sintering temperature, which degrades the stability of varistor properties [14]. In recent years, BaTiO₃-doped ZnO varistors are being studied, particularly focusing in finding a substitution for Bi₂O₃-based ZnO varistors. BaTiO₃ is dielectric ceramics with low loss characteristics, and as such is suitable to be used in non-volatile materials [15].

In this study, we investigated the effect of BaTiO₃ additive on the microstructure and electrical properties of low-voltage ZnO varistors. The present work displayed that is possible to obtain low-voltage varistors through the combination of a perovskite structure BaTiO₃ with a wurtzite structure ZnO.

II. EXPERIMENTAL DETAILS

A. Materials and Sample Preparation

The composition of varistor samples was fabricated based on the following proportion: (100 - *x*) wt.% ZnO and *x* wt.% BaTiO₃, where *x* = 4, 8, 12, 16, 20 and 24. ZnO powder with the particle size less than 1 μm and 99% purity (Merck) was selected as the host material. Barium titanate (BaTiO₃) with 99.9% purity (Merck) was used as an additive. The high purity powders of ZnO and BaTiO₃ were weighed in a stoichiometric ratio using a digital analytical balance of accuracy ±0.0001g. The powder mixtures were mixed for 3 h using a planetary ball mill. During the ball milling, the energy provided by constant collisions between particles, balls, and bowl lead to a considerable reduction of the particle size. Then, the mixture was added with the solution containing 1.75 wt.% polyvinyl alcohol (PVA), and dried at 80 °C for 2 h. This PVA will acts as a binder to give mechanical strength to the pressed compact pellets to avoid micro-cracks. After drying, the powders were granulated and sieved to produce starting powders. Next, the dried powders were pressed into disc-shaped with 10.0 mm in diameter and 1.0 mm in thickness at a pressure of 2.6 ton/5min. The pressed discs were sintered at 1300 °C for 90 min in a box furnace with the heating and cooling rate at 3°C/min. Finally, the sintered samples were polished and silver conductive paint are coated on both sides of the samples for further characterizations.

B. Characterizations

The crystalline phases of the samples were identified by X-ray diffraction (XRD, Rigaku MiniFlex II). The microstructure of sintered samples was observed by using a scanning electron microscope (Model JSM-6360LA, JEOL) that had an energy dispersive spectroscopy facility. The current density-electric field (*J-E*) property of varistor samples were measured by using a source meter (Keithley 2400, USA). The varistor voltage (*E_b*) was measured at a standard current density of 1.0 mA/cm². The leakage current density (*J_L*) was determined at 0.80*E_{1mA}* with the nonlinear coefficient (*α*) was calculated from Equation 1 [16]:

$$\alpha = \frac{\log J_2 - \log J_1}{\log E_2 - \log E_1} \quad (1)$$

where *J₁* and *J₂* are the current density corresponding to the electrical field *E₁* and *E₂* in the range of 1.0 mA/cm² to 10 mA/cm². The current density in the linear region can be determined by the following formula, Equation 2 [17]:

$$J = AT^2 \text{EXP} \left[\frac{\beta E^2 - \phi_B}{kT} \right] \quad (2)$$

where *A* is Richardson's constant, *β* is a constant, *k* is the Boltzmann constant, and *φ_B* is the barrier height. Voltage per grain boundary (*V_{gb}*) was calculated from *E_b* ($\frac{d}{D}$), where *d* is the average grain size obtained by the lineal intercept method and *D* is the thickness of the varistor [18].

III. RESULT AND DISCUSSION

A. Scanning Electron Microscopy (SEM)

Fig. 1 shows the SEM images of the samples with different BaTiO₃ additive contents. The sample has a morphology comprising of ZnO grains and covered with particles of BaTiO₃ phase located near the grain boundary. It was observed that the microstructure of the low-voltage varistors with various BaTiO₃ contents consists of two kinds of grains, which are large grains as main phases and small grains as intergranular phases. The average grain size ranges from 2.8 to 11.1 μm with the increase of BaTiO₃ content from 4 to 24 wt.%, respectively as indicated in Fig. 2.

As illustrated in the SEM images, the presence of defects at the grain boundary during the liquid phase sintering denotes the complete dissolution of BaTiO₃ particles into a liquid phase. Due to the high concentration of BaTiO₃, the undissolved BaTiO₃ particles started to grow during the solution-reprecipitation process, which indicates a strong interaction between ZnO and BaTiO₃ additive [19]. Additionally, the presence of TiO₂ as the varistor enhancer can contribute to the grain growth of ZnO [20]. But, the smaller ionic radius of Ti⁴⁺ (0.061 nm) compared to Zn²⁺ (0.074 nm) and Ba²⁺ (0.135nm) lead to the substitution of Zn at the Ti site in the BaTiO₃ structure [21].

Therefore, the presence of Ti ions at the grain boundary enhances the grain growth of ZnO in the first segregation to the grain boundary. The existence of Ba ions at the grain boundary reduces the growth of ZnO grains due to their large ionic radius compared to Zn ions. Thus, the high concentration of BaTiO₃ additive inhibits the grain growth of ZnO microstructures.

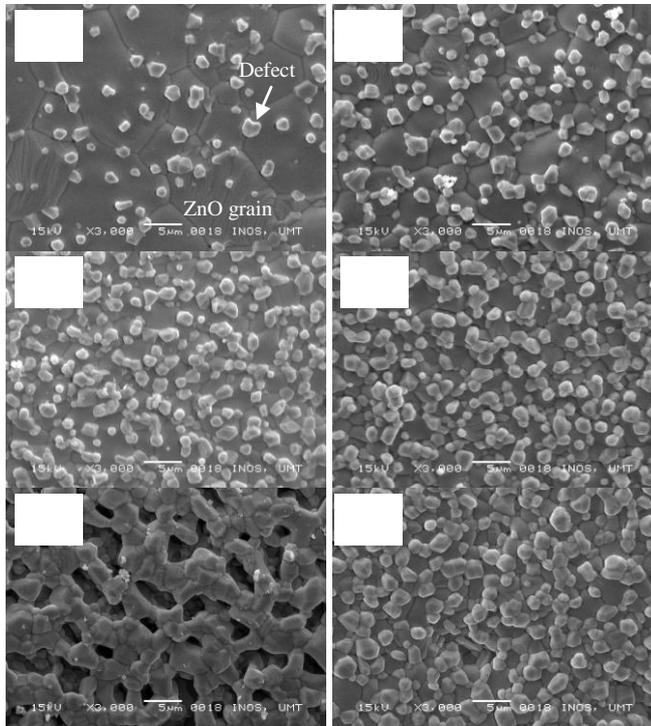


Fig. 1. SEM images of ZnO varistors prepared at different amount of BaTiO₃ additive at (a) 4, (b) 8, (c) 12, (d) 16, (e) 20 and (f) 24 wt.%.

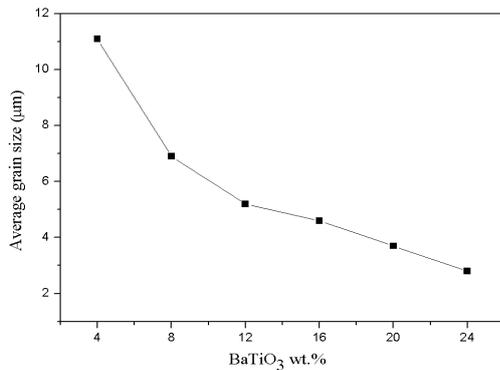


Fig. 2. Variation in average grain size of BaTiO₃ based ZnO varistor ceramics.

B. Energy Dispersive X-ray Analysis Spectroscopy (EDS)

The identified elements distribution on the sample surface obtained from EDS analysis in Fig. 3 and the presence of Zn, Ba, O and Ti elements were confirmed by EDS mapping in Tab. 1. The result showed a high concentration of additive such as Ba and Ti, which means that BaTiO₃ is well dispersed in the ZnO microstructure. The high concentration of additive reduced the concentration of Zn and O which suggested that the aims for Ba and Ti to be segregated into the grain boundary was achieved. The presence of this additive at the

grain boundary is important for the formation of barrier height that leads to the improved nonlinear properties of varistors.

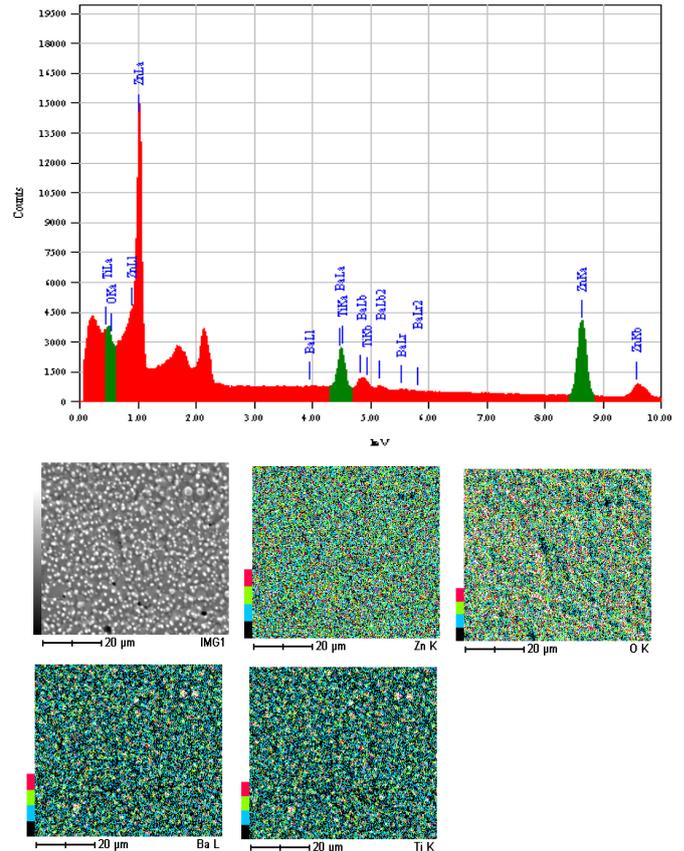


Fig. 3. The element distribution of Zinc, Oxygen, Barium and Titanium.

Table 1. EDS analysis of ZnO varistors containing BaTiO₃ ceramics.

Element	keV	mass%
O K	0.525	0.43
Ti K	4.508	3.53
Zn K	8.630	86.90
Ba L	4.464	9.14
Total		100.00

C. X-ray Diffraction (XRD)

Fig. 4 shows the XRD measurement of pure ZnO powder and its combination with different BaTiO₃ content. The presence of the BaTiO₃ phase was also verified by XRD analysis. It was observed that all peaks of the XRD pattern matched with the single phase polycrystalline hexagonal close-pack of pure ZnO per the ICSD No. 067849. Traces of BaTiO₃ was confirmed by ICSD No. 028851 with a cubic perovskite structure. None of the extra peaks appeared on the XRD patterns of ZnO ceramic until the addition of 4 wt.% BaTiO₃. The XRD results revealed the presence of main ZnO phase and the cubic BaTiO₃ phase at the planes of (100), (110) and (200), signifying the interaction between these ceramics. The formation of large BaTiO₃ particles on the ZnO microstructure due to the dissolution of many BaTiO₃ particles that segregated near the grain boundaries and triple points can be seen in the SEM images from Fig. 1.



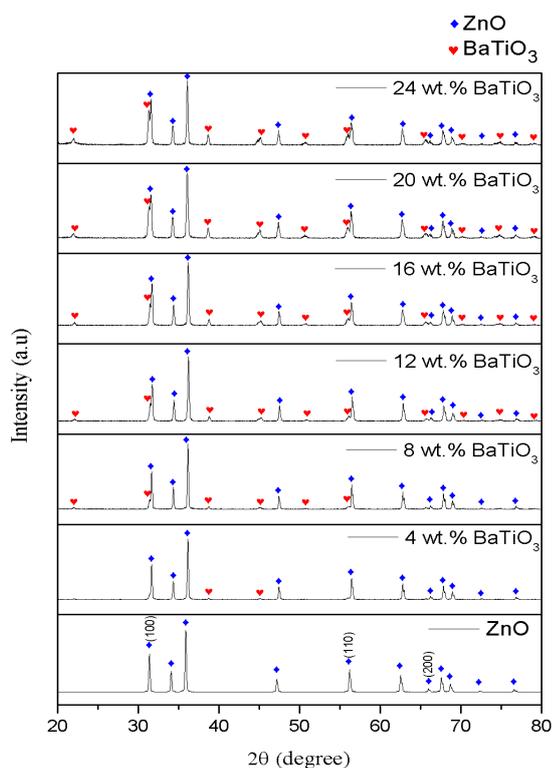


Fig. 4. XRD spectra of sintered ZnO varistors with BaTiO₃ content.

D. Current density-Electric field (J-E) Measurement

Table 2 shows the electrical properties of BaTiO₃ based ZnO varistors characterized by nonlinearity in the *J-E* characteristics. *J-E* measurement indicated that α was in the range of 1.8 to 4.8. The nonlinearity of the ZnO varistor increases with the increase of BaTiO₃ content up to 12 wt.%. ZnO varistor containing BaTiO₃ additive above 12 wt.% reduces the α indicating that the ceramics have reached their optimum value. The decrement value of α with BaTiO₃ content could be assigned to the existence of pores or O vacancies at the grain boundaries. The presence of oxygen ions can reduce the formation of other vacancies by increasing the barrier height at the interface states in the grain boundary [22], [23]. The formation of the Schottky barrier height is due to the presence of defect ions with the capability of restricting the electrons flow at the grain boundary [24]. It is suggested that the agglomeration of excessive Ba ions reduced the oxygen ions and induced the formation of pores or O vacancies. The reduction in barrier height also was affected by the excessive used of dopants, which deteriorate the varistor properties due to the variation of electronic state at the grain boundary [25].

Varistor containing BaTiO₃ exhibited low-voltage characteristic, where the varistor voltage significantly increased from 4.7 to 14.1 V/mm. The increased in varistor voltage is attributed to the decrease in average grain size. The average grain size was approximately 2.8 μm , with the voltage per grain boundary (V_{gb}) varies between 0.03 to 0.05 V as the BaTiO₃ amount was increased from 4 to 24 wt.%, respectively. The prepared low-voltage varistor showed a good agreement with several low-voltage varistor ceramics fabricated in previous studies [26], [27]. In addition, the reduction in barrier height with further increased of BaTiO₃ content up to 24 wt.% is caused by the increment of leakage

current density. The presence of defect gives rise to a potential barrier at the grain boundary, which reduces the leakage current. The barrier height dropped from 0.88 to 0.70 eV with the increase of BaTiO₃ from 12 to 24 wt.%, where this indicates that the presence of a high concentration of defects deteriorates the microstructure properties of low-voltage varistors. These high concentrations of defects induced the formation of vacancies in the ZnO microstructure with non-uniform average grain size distribution [28]. Consequently, the variation of vacancies occurred and increased the leakage current density due to the reduction in barrier height.

Table 2. *J-E* characteristics parameters of the ZnO varistor sample with different amounts of BaTiO₃ additive.

BaTiO ₃ (wt.%)	α	E_b (V/mm)	V_{gb} (V)	J_L ($\mu\text{A}/\text{cm}^2$)	ϕ_B (eV)
4.0	4.3	4.7	0.0	426	0.81
8.0	4.2	4.9	0.0	483	0.78
12.0	4.8	6.1	0.0	348	0.88
16.0	2.5	6.2	0.0	635	0.68
20.0	2.3	10.2	0.0	606	0.65
24.0	1.8	14.1	0.0	650	0.70

Based on this electrical performance, the highest value of α equal to 4.83 was measured on the sample made with 12 wt.% of BaTiO₃, where it also exhibited lower leakage current density with higher value of barrier height. Thus, the composition can be considered as the best for all samples. Meanwhile, the high leakage current may heat up the varistor device and can affect the other electrical characteristics. Due to the high leakage current, the generated heat is not transferred to the outside environment and often leading to a thermal runaway of the device. The lowest leakage current density of ZnO varistors was 348 $\mu\text{A}/\text{cm}^2$, which was obtained from 12 wt.% of BaTiO₃ content. This indicates that a high resistivity of the ceramics, where the electrical conduction mechanism is controlled by the barrier height present at the grain boundaries [29]. Hence, it is important to reduce the leakage current to as low levels as possible. Industrially, the most preferable properties of a varistor include a high value of nonlinearity coefficient (α), an acceptable rating of varistor voltage (E_b), and a small value of leakage current density (J_L) [30].

IV. CONCLUSION

Low-voltage BaTiO₃ doped ZnO varistor ceramics have been successfully prepared via a solid-state reaction method.

BaTiO₃ doped ZnO varistors obtained after sintering at 1300 °C for 1.5 h had compact microstructures with a grain size in the range of 2.8 to 11.1 μm. The highest α value of 4.8 obtained with the 12 wt.% BaTiO₃ additive is considered as the optimum output for the study. Consequently, the ceramics exhibited poor nonlinearity with further addition of BaTiO₃ content up to 24 wt.% with the range of barrier height reduced from 0.88 to 0.70 eV. The reduction of these interface barrier height is due to the increase in J_L value from 348 to 650 μA/cm². Furthermore, the prepared varistor showed a relatively low-voltage in the range of 4.7 to 14.1 V/mm with the V_{gb} decreased from 0.05 to 0.03 V for 4 to 24 wt.% of BaTiO₃ additive, respectively. The α value lowering with increasing BaTiO₃ content, which suggests the high concentration of defects and reduced the barrier height caused by the existence of pores and other vacancies at the grain boundaries.

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