UNIVERSITI PUTRA MALAYSIA

OPTIMIZING NANO-SOLUTION COATING METHOD AND BALL MILLING METHOD TO ACHIEVE MAXIMUM NON-LINEARITY PROPERTY FOR ZnO- BASED LOW-VOLTAGE VARISTOR CERAMICS

MASOUMEH DORRAJ

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By

MASOUMEH DORRAJ

Thesis Submitted to the School of Graduate Studies Universiti Putra Malaysia in Fulfillment of the Requirements for the Degree of Master of Science

July 2014

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DEDICATION

To my beloved family

Thanks for their supports, understanding, love and encouragement.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science

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MASOUMEH DORRAJ

July 2014

Chairman : Professor Azmi Zakaria, PhD
Institute : Advance Technology

In ZnO based low voltage varistor ceramics, the microstructure development is depends on Bi_2O_3, TiO_2 and Sb_2O_3 molar ratio. Thus, the selection of a composition with an appropriate molar ratio is completely important. In this study, the optimal levels of these dopants to achieve maximized nonlinear electrical property (alpha) were quantified by the response surface methodology (RSM) for the nano-solution coating (NSC) and ball milling (BM) methods. Secondly, the electrical and physical properties of optimized samples obtained by both methods were compared with each other. The central composite rotatable design consisting of three variables and alpha as a response, with 20 runs was used to conduct the experiments in each method. To obtain actual responses, the design was performed in laboratory by the NSC and BM methods. For both methods, the actual responses were fitted into a valid second order polynomial model. Then the analysis of variance (ANOVA) showed that the actual responses could be adequately fitted to quadratic polynomial model by several evidences. For the NSC method, these evidences included the high F-value (77.56), very low P-value (<0.0001), R-squared (0.986), adjusted R-squared (0.973) and predicted R-squared (0.950), while for the BM method consisted of the F-value (28.79), very low P-value (<0.0001), R-squared (0.963), adjusted R-squared (0.930) and predicted R-squared (0.780). The optimum values of additives were investigated by graphical and numerical optimization methods for both techniques. Based on these optimization methods, for NSC technique, the optimum values of Bi_2O_3, TiO_2 and Sb_2O_3 in maximum alpha (14.52) were predicted 0.52, 0.50 and 0.30, respectively, while for BM technique in maximum alpha (9.47) were predicted 0.44, 0.40 and 0.29, respectively. Experiments were then carried out under the recommended conditions and resulting responses were compared to the predicted values. The results for both methods were quite close to the alpha values by the equation models. In conclusion, RSM has been successful for modeling and optimizing the additives such as Bi_2O_3, TiO_2 and Sb_2O_3 of ZnO-based low voltage varistor ceramic to achieve maximized non-linearity properties in both methods. The highest value of alpha was obtained by NSC method (14.55) in compare the BM method (9.43). Moreover, the improvement in electrical properties of varistors made by NSC method could be explained by the homogeneous distribution of various dopant in the mixed powder and the more chemically uniform in structures.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk Ijazah Master Sains.

PENGOPTIMUMAN KAEDAH- KAEDAH PENYADURAN LARUTAN-NANO DAN PENGISARAN-BOLA UNTUK MENCAPAI SIFAT TAK-LINEAR MAKSIMA UNTUK SERAMIK VARISTOR BERVOLTAN RENDAH BERASASKAN ZnO

Oleh

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Dalam seramik varistor bervoltan rendah berasaskan ZnO, pembangunan mikrostruktur adalah lebih bersandarkan kepada nisbah molar Bi_2O_3, TiO_2 dan Sb_2O_3. Oleh itu, pemilihan suatu komposisi dengan nisbah molar yang bersesuaian adalah sangat penting. Di dalam kajian ini, paras optimum bagi pendopan-pendopan ini untuk mencapai sifat elektrik tak-linear (alfa) maksimum telah dikira menggunakan kaedah permukaan sambutan (RSM) untuk kaedah penyaduran larutan-nano (NSC) dan pengisaran-bola (BM). Keduanya, sifat-sifat fizikal dan elektrikal bagi sampel-sampel teroptimum dari kedua-dua kaedah ini telah dibandingkan antara satu sama lain. Reka-bentuk berputar komposit pusat terdiri daripada tiga pembolehubah dan alfa sebagai respons, dengan 20 larian telah digunakan untuk menjalankan eksperimen bagi setiap kaedah. Untuk mendapat respons sebenar, reka-bentuk telah dilakukan di dalam makmal menggunakan kaedah NSC dan BM. Bagi kedua-dua kaedah, respons sebenar telah disesuaikan ke dalam model polinomial peringkat kedua yang sah. Kemudian analisis varians (ANOVA) menunjukkan bahawa respons sebenar boleh disesuaikan secukupnya dengan model polinomial kuadratik oleh beberapa bukti. Bagi kaedah NSC, bukti-bukti ini termasuk nilai-F yang tinggi (77.56), nilai-P yang sangat rendah (<0.0001), kuasa-dua-R (0.986), kuasa-dua-R terlaras (0.973) dan R-kuasa dua ramalan (0.950), manakala bagi kaedah BM terdiri daripada nilai-F (28.79), nilai-P yang sangat rendah (<0.0001), kuasa-dua-R (0.963), R-kuasa-dua-R larasan (0.930) dan kuasa dua-R ramalan (0.780). Nilai optimum bahan-bahan penambah telah disiasat menggunakan kaedah grafik dan pengoptimuman angkaan bagi kedua-dua teknik. Berdasarkan kaedah pengoptimuman, untuk teknik NSC, nilai optimum bagi Bi_2O_3, TiO_2 dan Sb_2O_3 untuk alfa maksimum (14.52) telah diramalkan 0.52, 0.50 dan 0.30, masing-masing, manakala untuk teknik BM, untuk alfa maksimum (9.47) telah diramalkan 0.44, 0.40 dan 0.29, masing-masing. Eksperimen seterusnya diteruskan dengan keadaan-keadaan yang telah dicadangkan dan keputusan-keputusan respons telah dibandingkan dengan nilai ramalan. Keputusan-keputusan bagi kedua-dua kaedah adalah hampir sama dengan nilai alfa menggunakan model-model persamaan.

Kesimpulannya, RSM telah berjaya bagi pengoptimuman dan permodelan bahan-bahan penambah seperti Bi_2O_3, TiO_2 dan Sb_2O_3 bagi seramik varistor bervoltan rendah berasaskan ZnO untuk mencapai sifat-sifat ketaklinearan maksimum bagi kedua-dua kaedah. Nilai tertinggi bagi alfa adalah diperolehi menggunakan kaedah NSC (14.55) berbanding dengan kaedah BM (9.43). Selain itu, penambah-baikan dalam sifat-sifat elektrik varistor diperbuat daripada kaedah NSC boleh dijelaskan dengan taburan homogen pelbagai pendopan di dalam serbuk tercampur dan keseragaman kimia yang lebih dalam struktur.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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Declaration by Members of Supervisory committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) were adhered to.

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LIST OF ABBREVIATIONS

V_c	Breakdown Voltage
I-V	Current-Voltage
I	Current
K	Constant
DSB	Double Schottky Barrier
XRD	X-ray Diffraction
FESEM	Field Emission Scanning Electron Microscopy
VPSEM	Variable Pressure Scanning Electron Microscopy
EDAX	Energy Dispersive X-Ray Analysis
µm	Micrometer
RSM	Response Surface Methodology
ANOVA	Analysis of Variance
MLS	Method of Least Square
CCD	Central Composite Design
SMSS	Sequential Model Sum of Sqares
PRESS	Prediction Error Sum Squares

LIST OF SYMBOLS

.	Nonlinear Coefficient
ºC	Degree celsius
Å	Angstrom
	Angle of diffraction
E	Electrical field
J	Current density

CHAPTER 1

INTRODUCTION

1.1 Background

Zinc oxide based varistors exhibit highly nonlinear current–voltage characteristics which have been widely applied in the field of protection against transient voltage surges in electronic devices (Abdollahi et al., 2013). Along with the growing demands on very-large-scale integration electronics, application of low-voltage ZnO varistors are now being attracted more attention (Abdullah et al., 2012). The non-linearity properties of ZnO varistor is expressed by $I = KV^{\alpha}$ where K is a constant, and 'α' is nonlinear coefficient (Balzer et al., 2004). The non-linear characteristic is a phenomenon attributed to the formation of double Schottky barriers at the ZnO grain boundaries (Wang et al., 2008). ZnO based varistors are made by sintering mixture of ZnO grains with small amounts of other metal oxides, such as Bi_2O_3, TiO_2 and Sb_2O_3, at a given temperature (Peiteado et al., 2007). Microstructurally, the sintered material is made of highly conductive ZnO grains with two major secondary phases: a spinal-type phase and a Bi-rich phase which are located at the grain boundaries which have strictly effect on nonlinear current-voltage characteristic (Zhang et al., 2002). The microstructure of an ideal varistor has uniform grain size, shape and composition; minimal porosity and a uniform distribution of secondary phases (Puyane et al., 1996). These properties and in particular compositional homogeneity, are difficult to achieve by conventional routes. Thus, enhanced electrical properties could be achieved by using a more advanced processing technique such as nano-technology to approach the ideal ceramic detailed above. Nano-origin varistor powders with high degree of homogeneity in dopant distribution have been attempted through a variety of chemical techniques like coprecipitation, sol–gel , microemulsion and polymerized complex method (Anas et al., 2010; Wang et al., 2008). However most of these methods are complicated and costly, or not suitable to the production of low-voltage ZnO varistors (Wang et al., 2008). In the present work, the low-voltage ZnO varistors were prepared by a novel solution nano-coating technique.

Furthermore, in the microstructure, each of the dopants have a distinctive role in forming the electrical characteristics of the varistor ceramics. The Bi_2O_3 is the basic dopant, which creates the nonohmic behavior of ZnO-based varistor ceramics by forming the electrostatic barriers at the grain boundaries. Other dopants are added to enhance the nonlinear characteristics and control the microstructure development. During the firing process, dopants react with the ZnO and the microstructure of the varistor is formed in the presence of a Bi_2O_3-rich liquid phase (Bernik et al., 2011). The usual classical approach of making low-voltage varistors is through grain coarsening techniques by adding grain-growth-enhancing additive such as TiO_2 (Daneu et al., 2013). Sb_2O_3 is a standard spinel-forming dopant to produce fine-grained high-voltage varistor ceramics. It enhances the nonlinearity of varistor ceramics and reduces the evaporation of Bi_2O_3 during sintering. However, the major role of Sb_2O_3 is to control the growth of the ZnO grains. The inhibition of ZnO grain growth in Sb_2O_3-doped samples is generally explained by a reduction in the mobility of grain boundaries by a pinning effect, caused either by secondary spinel particles or a fine Sb-rich film on the surface of the ZnO grains (Bernik et al., 2004). As a multivariate case, Bernik et al. (2004) have demonstrated that the microstructure development is strongly influenced by the TiO_2/Bi_2O_3 in low-voltage varstor ceramics and the Sb_2O_3/Bi_2O_3 ratio in high-voltage varistor ceramics. This report reveals the synergistic interaction of dopant in order to achieve a desired microstructure with specific electrical properties. On the other hand, the traditional one-factor-at-a-time method to optimization is taking a long time and unable to find an accurate optimum due to having no concern for interaction between factors. To determine the influence of the interactions of doppend on the electrical characteristics of varistors, the molar ratios of the additives have to simultaneously be considered. Opposite of what has been stated, statistical methods can take into account the interaction of variables in creating the process response. Consequently, a statistically designed experiment with minimum experimental runs is greatly desired. Response surface methods (RSM) contain a group of empirical methods specified for the development of relations existing between a cluster of controlled experimental factors and the measured responses, suitable for improving, developing, and optimizing processes by carrying out a limited number of experiments based on experimental design (Li et al., 2013). In this work, the experiments were designed by central composite design (CCD). The composition of the additives such as Bi_2O_3, TiO_2 and Sb_2O_3 were considered as effective variables. The design was performed in laboratory to obtain the non-linearity coefficient (alpha) as actual response. The responses were used for fitting process by using least squares regression analysis. The process proposed a provisional model. Adequacy of a proposed model is revealed by diagnostic checking provided by analysis of variance (ANOVA). The validated model was used to determine the optimum values of the variables.

1.2 The Electrical Properties of ZnO Varistor

Zinc oxide varistor are ceramic semiconductors devices that have great nonlinearity in their current–voltage behavior, and therefore, are broadly utilized for electronic devices (Wang et al., 2008). In doped ZnO varistor the nonlinear current-voltage properties can can be defined by the experimental relationship $J=KE^{\alpha}$, where J is the current density, E is the applied electric field and α is the coefficient of nonlinearity and K is the persistent of proportionality. Good varistors are described by high α in the non-ohmic region (Banerjee et al., 2001). The non-linear response originates on its polycrystalline microstructure and more precisely in detail thorough procedures happening at the grain/grain interfaces (Peiteado et al., 2007). Varistors are manufactured by mixing a number of different metal oxides in powder form and the mixture is treated by liquid phase sintering to form the final varistor. ZnO is the major ingredient in the oxide mix and Bi_2O_3 and Sb_2O_3 are common additives which enhance the performance of the varistors. The process is called liquid phase sintering because the metal oxide mixture is heated at temperatures in the series of 1000-1300 ^{o}C for a long time in a furnace. The additives melt at these temperatures while the ZnO grains stay crystalline which means that the ZnO grains are floating in a melt of metal oxides during the sintering. It is believed that conduction electrons close to the grain boundaries are attracted towards, and trapped in particular electron states (Leach, 2005; Peiteado et al., 2007). These are called interface states and are located in the grain boundaries as is indicated in Figure 1.1.

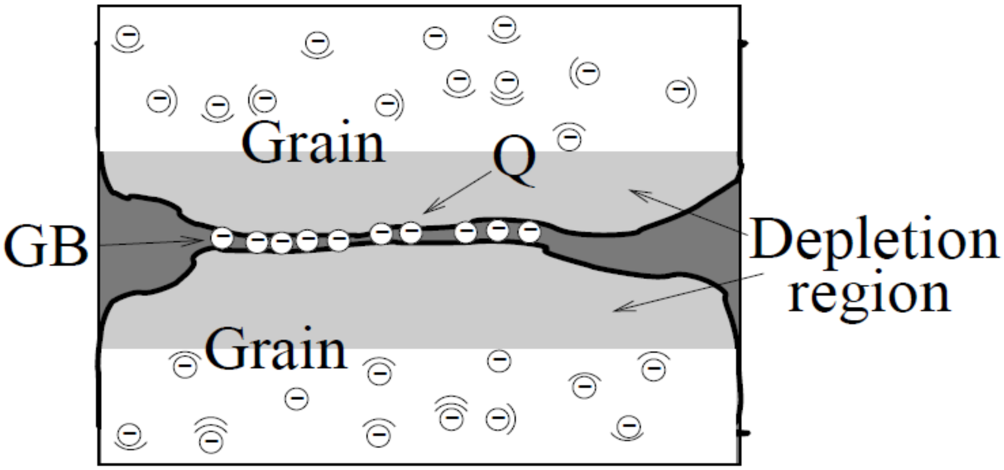

Figure 1.1. Sketch of the charge distribution in the vicinity of a grain boundary. Q is the charge trapped in the localized states in the grain boundary and the shaded area is the depletion region. (Carlsson, 2002)

This creates a depletion region on both parts of the grain boundaries. The depletion region acts as a barrier for the electron current across the grain boundaries according to the Double Schottky Barrier (DSB) model, because there are no free conduction electrons which can sustain the electron flow from one grain to the next. The trapping of electrons at the grain boundary is voltage dependent since the electrons start to get enough energy from the applied field to leave the interface states at a critical voltage. The barrier disappears and the conduction electrons can maintain an electron flow also in the grain boundary region (Carlsson, 2002). The existence of potential barriers in turn creates critical voltages for breakdown per boundary and the total breakdown voltage of the device becomes proportionate to the number of such grain boundaries in between two electrodes (Banerjee et al., 2001). From the schematic of Figure 1.2, it is obvious that the electrical features of ZnO varistors are associated to the bulk of material. This inherently multijunction feature of varistor cause its action shared between the different ZnO grain boundaries (Levinson and Philipp, 1986).

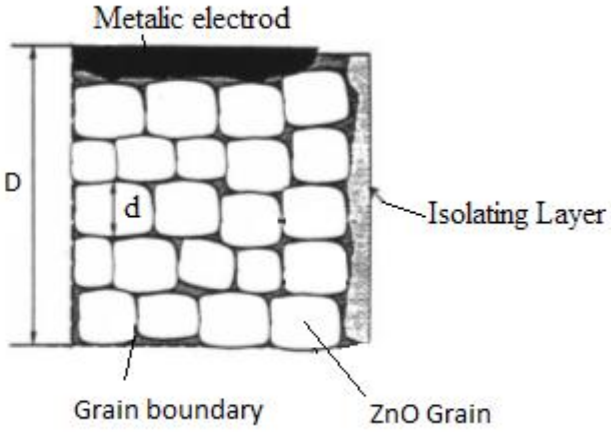

Figure 1.2. Schematic of the microstructure of ZnO varistor

(Meshkatoddini, 2011)

This then suggests that the breakdown voltage, V_B is a matter of constructing a varistor related to the number of grains, n, in sequence between the electrodes through the relation:

$$V_B = n\, v_g = D\, v_g / d \qquad (1.1)$$

where d is the ZnO grain size D the electrode spacing and v_g is the breakdown voltage per grain boundary. Therefore, to attain a given breakdown voltage one could alter the varistor thickness (for fixed grain size) or another one could change the grain size (for the device thickness constant).

1.2.1 ZnO Low-voltage Varistor

Applying of low-voltage varistors for circuit supporting are progressively important owing to rising requests on low-voltage electronics. For example, mobile appliances and battery powered necessitate support from transient dc voltage of between 4 to 20 V. (Abdullah et al., 2012). The electrical features of low-voltage ZnO varistors are thoroughly connected to their microstructure and composition, specially the ZnO grain size and the construction at the grain boundaries. As the breakdown voltage (V_B) of the varistor is proportional to the number of ZnO grains in sequence between the electrodes, two major procedures have been utilized for production of low-voltage ZnO varistors . One method is thinning the devices. This may either be from a screen printed paste of dopants (Schwing and Hoffmann, 1981) or a solid dopant layer sandwiched between two ZnO substrates (Selim et al., 1980). However, the thin ZnO varistors are challenging to make and suitable for breaking. The other is the classical method of choosing additives that motivate grain growth. The most significant additives are TiO_2, which can significantly increase the grain growth of ZnO, therefore is frequently utilized as a grain growth enhancing additive to create low-voltage ZnO varistors (Trontelj et al., 1986).

1.3 Fabrication of Polycrystalline Ceramics From Powders

The fabrication of ceramics from powders is illustrated in the flow chart shown in Figure 1.3.

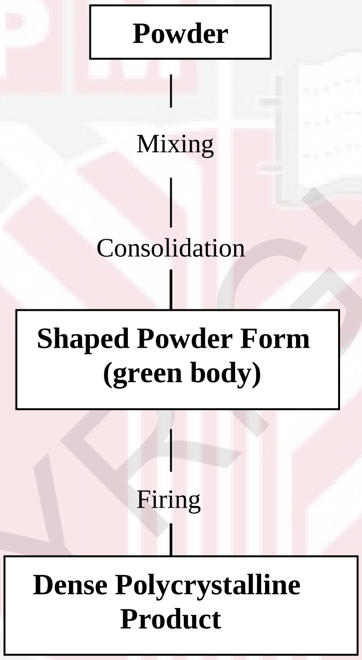

Figure 1.3. Basic flow chart for the production of polycrystalline ceramics by firing of consolidated powders. (Rahaman, 2007)

In most cases, the fabrication process starts from a mass of powder obtained from commercial sources. Nevertheless, knowledge of powder synthesis methods is very important. Equally important are methods that can be used to determine the physical, chemical, and surface characteristics of the powder. The characteristics of the powder rely intensely on the technique utilized to synthesize it, and these, in turn, affect the subsequent processing of the ceramic. The more details are explained in section 1.4. The consolidation of ceramic powders to produce a green body is commonly referred to as forming. The main forming methods include: dry or semidry pressing of the powder (e.g., in a die). It is found that the green body microstructure has importance effects on the subsequent firing stage. If severe variations in packing density occur in the green body, then under

conventional firing conditions, the fabricated body will usually have a heterogeneous microstructure that limits the properties and reliability.
After consolidation, the green body is heated to yield the preferred microstructure. The alterations happening through out this stage might be quite complicated, relying on the difficulty of the initiating materials. (Lee, 1994; Rahaman, 2007).

1.4 Synthesis of Powders

The characteristics of the powder have a remarkable effect on subsequent processing, such as consolidation of the powder into a greenbody and firing to produce the desired microstructure. The significant relations between atomic structure, chemical composition, microstructure, fabrication, and properties of polycrystalline ceramics are illustrated in Figure 1.4. As a result, powder synthesis is very important to the overall fabrication of ceramics. The desirable characteristics that a powder should possess for the production of successful ceramics are explained in the next topic (Rahaman, 2007).

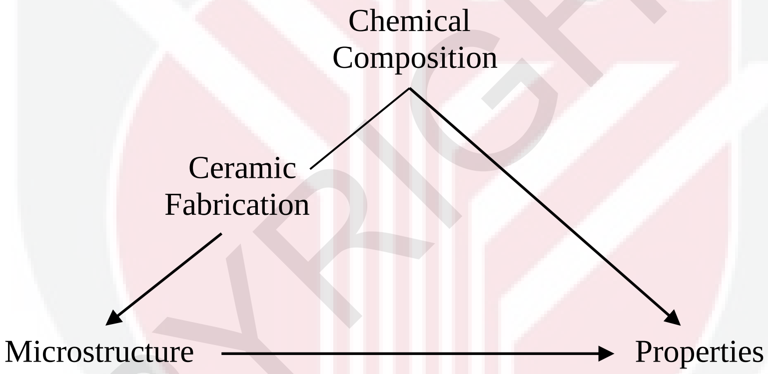

Figure 1.4. The important relationships in ceramic fabrication

1.4.1 Desirable Powder Characteristic

Advanced ceramics must meet very specific property requirements and therefore their chemical composition and microstructure must be well controlled. Advanced ceramics include ceramics for electrical, magnetic, electronic, and optical applications. Careful attention must be paid to the quality of the starting powders. For advanced ceramics, the important powder features are the size, size distribution, shape, state of agglomeration, chemical composition. These properties have an significant influence on both the powder consolidation stage and the microstructure of the fired body. The most profound effect of the particle size, however, is on the sintering. The rate at which the body densifies increases strongly with a decrease in particle size. Normally, if other factors do not cause severe difficulties during firing, a particle size of less than ~ 1μm permits the attainment of great density in a sensible time (e.g., few hours). Homogeneous packing of a narrow size distribution powder generally allows greater control of the microstructure. Agglomerates result in heterogeneous packing in the green body that, in turn, result in differential sintering during the firing stage. Differential sintering occurs when diverse areas of the body shrink at diverse rates (Ma and Lim, 2002; Rahaman, 2007). This can lead to serious problems such as the development of large pores and crack like voids in the fired body. Surface impurities may have a significant influence on the dispersion of the powder in a liquid, but the most serious effects of variations in chemical composition are encountered in the firing stage. Impurities may lead to the formation of a small quantity of liquid phase at the sintering temperature, which causes selected growth of large individual grains. In such a case, the achievement of a fine uniform grain size would be impossible (Rahaman, 2007). To summarize, the desirable powder characteristics for the fabrication of advanced ceramics are listed in Table 1.1.

Table 1.1. Desirable Powder Characteristics for Advanced Ceramics

Powder characteristic	Desired property
Particle size	Fine(< ~ 1 m)
Particle size distribution	Narrow or monodisperse
State of agglomeration	No agglomeration or soft agglomerate
Chemical composition	High purity

1.5 Optimization

Optimization refers to developing the performance of a system, a procedure, or a product in order to obtain the highest benefit from it. Optimization has been commonly utilized in analytical chemistry as a means of exploring circumstances at which to apply a process which creates the greatest possible response (Araujo and Brereton, 1996). Traditionally, optimization in analytical chemistry has been carried out by observing the effect of one factor at a moment on an experimental response. Whereas just one parameter is altered, others are maintained at a constant level. This optimization method is named one-variable-at-a-time. Its main disadvantage is that it does not involve the interactive effects amongst the variables considered. As a result, this method does not represent the whole effects of the parameter on the response (Lundstedt et al., 1998). Another detriment of the one-factor optimization is the growth in the quantity of experimentations necessary to conduct the study, that results in a raise of expenses and time and also a raise in the consumption of materials and reagents. In order for overcoming this difficulty, the optimization of analytical processes has been carried out through utilizing multivariate statistic techniques. Amongst the most pertinent multivariate methods used in analytical optimization is response surface methodology (RSM). Response surface methodology is a collection of statistical and mathematical methods based on the fit of a polynomial equation to the experimental data, that must describe the behavior of a data set with the objective of making statistical previsions. It could be well applied when a response or a set of responses of interest are affected by numerous variables. The objective is to simultaneously optimize the levels of these variables to attain the best system performance

(Bezerra et al., 2008). This method consists of changing or manipulating levels or amount of selected independent variables to investigate their effect on the dependent variables (Brown and Melamed, 1990). RSM was applied by Box and collaborators in the 50s (Gilmour, 2006). This term was initiated from the graphical perspective produced after fitness of the mathematical model (Teófilo and Ferreira, 2006).

1.6 Research Problem and Hypothesis

In fabrication of the varistors, homogeneity of the additive is the critical point for producing good varistor microstructure. The first problem is that varistor ceramic with inhomogeneous microstructure has negative effect on the current–voltage characteristics due to high local currents. In other words, to achieve a uniform sintered microstructure, uniformly packed particles with a narrow size distribution are needed. As is well known, nanoparticles yield a narrow grain size distribution. In order to get such uniform nanoparticles with a narrow size distribution, the nano-solution coating process for doped ZnO synthesis provides an efficient way to increase the microstructural homogeneity. In addition in this method, more additive atoms could be accommodated at the boundary regions to better control and improve the electrical properties of resulting materials. The hypothesis is that nano-solution coating method can be more efficient than the conventional method because it provides the desired microstructures and consequently improved electrical properties. The second problem is that the additives have been optimized by the traditional methods such as one variable at a time. The method is changing one parameter at a time while the other parameters are kept constant that provides the information related to that particular parameter only. This method of optimization is time consuming and cannot take the mutual interactions of the parameters during performance. The statistical procedures such as RSM provides an alternative technique to optimize the productive process by considering the interactions between the variables and gives an estimate of the combined effect of these variables on final result. The other hypothesis is that RSM is able to optimize the input variables such as the mole fraction of Bi_2O_3, TiO_2 and Sb_2O_3 to achieve the maximum homogeneity and consequently non-linearity property.

1.7 Objectives

In this study two fabrication methods of low-voltage varistor have been performed according to CCD experimental design. The performance was carried out to achieve the following objectives:

a) To optimize the additives of ceramic (Bi_2O_3, TiO_2 and Sb_2O_3) in ZnO based low voltage varistor via nano-solution coating and ball milling methods by Response surface methodology (RSM).

b) To compare the physical properties and electrical characteristics of the optimized valistors that are fabricated by nano-solution coating and ball milling methods.

1.8 Scope of Study

The present study reports the results of modeling and optimizing of molar ratio of Bi_2O_3, TiO_2 and Sb_2O_3 as additives for the maximization of alpha in nano-solution coating and ball milling methods. CCD of 20 experiments based on RSM by the assistance of Design-Expert software version 8.0.7.1, Stat-Ease Inc., USA has been used in each method. A model to predict the response (alpha) has been formulated and validated by ANOVA. The model optimized the molar ratio of input additives and after that maximized the alpha as output. The model also predicted the desired situation involving minimum standard error and the maximum alpha that are validated by additional experimentations. The predicted samples were considered by Filed Emission Scanning Electron Microscopy (FESEM), variable pressure scanning electron microscope (VPSEM), Energy-dispersive X-ray (EDX) and X-ray diffractometer (XRD).

1.9 Chapter Organization

Chapter 2 presents the previous research on non-ohmic devices. In this chapter, the effects of additives/dopants selection and reviews related to ceramic varistors prepared from solution processes also were briefly discussed. In chapter 3, two methods were introduced for fabrication of ZnO based low-voltage ceramic according experimental design. Moreover, detailed discussions on sample preparation for each method were explained and followed by characterization techniques in this chapter. Finally, it should be noted that the software procedures were defined at the end of this chapter. The results and discussion begin by data collection according experimental design in the laboratory and then continue with analysis of the resulting data by software for both method. Data analysis include standard error, result of fitting process, ANOVA, checking adequacy of model by different diagnostic plot, model presentation, optimization, prediction and validation. Finally, the comparison between methods was performed by the characterization of the validated samples. It includes chemical, morphological and electrical analyses. Last but not least, chapter 5 summarizes the present study and expresses some recommendations for future works.

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