UNIVERSITI PUTRA MALAYSIA

RESPONSE SURFACE METHODOLOGY FOR OPTIMIZATION OF SINTERING PROCESS FOR PREPARATION OF ZnO LOW VOLTAGE VARISTOR

SEYEDEHMARYAM MOOSAVI

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By

SEYEDEHMARYAM MOOSAVI

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

June 2014

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DEDICATION

This thesis is dedicated to

my beloved husband

and

parents

who support me with their

endless love to carry out my education.

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

RESPONSE SURFACE METHODOLOGY FOR OPTIMIZATION OF SINTERING PROCESS FOR PREPARATION OF ZnO LOW VOLTAGE VARISTOR

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June 2014

Chairman : Prof. Azmi Zakaria, PhD
Faculty : Science

Optimization of variables in order to maximize an electrical non-linear coefficient (α) of varistor ceramics by traditional method is a daunting and time-consuming task. The objectives of the present work are optimization of sintering temperature and holding time as input variables, and secondly the cooling and heating rates of the sintering process in order to maximize an electrical non-linear coefficient (α) of varistor ceramics. It was achieved by designing the varistor ceramic fabrication using the central composite design (CCD) of four levels of variables, sintering temperature, holding time, cooling rate and heating rate, and a response. Then the varistor ceramics was fabricated in laboratory according to the design to achive actual responses, α. The responses were used for modeling in optimizing the above factors by response surface methodology (RSM). The actual responses were fitted into a valid second order algebraic polynomial equation as quadratic model. The quadratic model was suggested to investigate by analysis of variance (ANOVA) based on some statistical conformation such as lowest standard deviation, P-value, PRESS and the highest R_{adj}, R_{pred} values. The validated model optimized the above factors by either canonical equation or by 3D plots as optimum point on the surface area. Results indicated that the maximum electric property of the varistor was at temperature 1260°C and holding time of 60 minutes that maximized the nonlinear value around 13. Also, the optimum area is around 5.5 for both cooling rate and heating rate which the maximum α was around 12. Moreover, the model suggested a high desirable solution in special condition to predict the optimum amount of the factors. The condition was including minimum standard error and maximum nonlinearity. The predicted solution included standard error, 0.096, sintering temperature, 1253°C, holding time, 53 min, and α, 11.2. The optimized sample tested by further experiments confirmed that the obtained α value (11) was very close the model predicted value. Then this information was used in optimizing the cooling and heating rates and the predicted solution has standard error of 0.038, cooling rate of 4.65, heating rates of 4.8, and α value of 10.96. The optimized sample tested by experiments, confirmed that the obtained α value is 11.4 was very close the model predicted value. Therefore, RSM was succeeded in modeling of the sintering profile in fabrication of zinc oxide based low voltage varistor.

Abstrak tesis dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENGOPTIMUMAN PROSES PERSINTERAN SERAMIK VARISTOR BERVOLTAN RENDAH DENGAN KAEDAH TINDAK-BALAS PERMUKAAN

Oleh

SEYEDEHMARYAM MOOSAVI

Jun 2014

Pengerusi : Prof. Azmi Zakaria, PhD
Fakulti : Sains

Pengoptimuman pembolehubah-pembolehubah dalam usaha memaksimumkan pekali tak-linear elektrik (α) seramik varistor secara kaedah tradisional adalah suatu tugas sukar dan memerlukan masa. objektif karjian ini adalah Pengoptimuman pensinteran suhu dan memegang masa sebagai pembolehubah input, dan kedua penyejukan dan pemanasan kadar proses pensinteran untuk memaksimumkan pekali bukan linear elektrik (α) seramik varistor. Ianya tercapai melalui reka-bentuk fabrikasi seramik varistor menggunakan central composite design (CCD) dari empat jenis pembolehubah, suhu persinteran, masa pegangan, kadar penyejukan dan kadar pemanasan dan tindak-balas. Kemudian, seramik varistor telah dilakukan di dalam makmal mengikut reka-bentuk untuk mencapai tindak-balas sebenar, α. Respons-respons ini digunakan untuk model bagi mengoptimumkan faktor-faktor dengan kaedah tindak-balas permukaan (RSM). Tindak-balas sebenar telah dipadankan ke dalam persamaan polinomial aljibra tertib kedua yang sah sebagai model kuadratik. Model kuadratik telah dicadangkan untuk diselidiki dengan menggunakan analisa varians (ANOVA) berdasarkan beberapa pengesahan statistik seperti sisihan piawai terendah, nilai-P, PRESS, dan nilai tertinggi R_{adj}, R_{pred}. Model optimum menetusah faktor-faktor samada melalui persamaan kanonikal atau plot 3D sebagai titik optimum kawasan permukaan. Keputusan menunjukkan ciri elektrik maksima varistor adalah pada suhu 1260°C dan masa pegangan 60 minit yang memaksimakan nilai tak-linear disekitar 13. Juga, kawasan optimum adalah disekitar 5.5 untuk kedua-dua kadar penyejukan dan kadar pemanasan α maksima adalah di sekitar 12. Tambahan pula, model mencadangkan suatu penyelesaian wajar yang tinggi untuk keadaan khas untuk meramal amaun optima faktor-faktor tersebut. Syarat ini adalah mengambil-kira kesalahan piawai minima dan tak-linearan maksima. Penyelesaian ramalan adalah termasuk ketidakpastian piawai, 0.096, suhu persinteran, 1253 °C, masa pegangan, 53 min dan α, 11.2. Pengoptimuman sampel diuji dengan ujikaji selanjutnya mengesahkan bahawa nilai α (11) sangat hampir dengan nilai model jangkaan. Kemudian maklumat ini telah digunakan dalam pengoptimuman kadar penyejukan dan pemanasan, dan penyelesaian jangkaan mempunyai ketidakpastian piawai sebanyak 0.038, kadar penyejukan sebanyak 4.65, kadar pemanasan sebanyak 4.8 dan nilai α ialah 10.96. Pengoptimuman sampel telah diuji dengan ujikaji,

mengesahkan nilai yang diperoleh ialah 11.4 yang mana tersangat hampir dengan nilai jangkaan model. Oleh itu, RSM telah berjaya dalam memodelkan profail persinteran dalam memfabrikasi varistor bervoltan rendah berasaskan zink oksida.

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Seyedehmaryam Moosavi

2014

I certify that a Thesis Examination Committee has met on 26 June 2014 to conduct the final examination of Seyedehmaryam Moosavi on her thesis entitled "Response Surface Methodology for Optimization of Sintering Process for Preparation of ZnO Low Voltage Varistor" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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Faculty of Science
Universiti Putra Malaysia
(Chairman)

Zulkifly bin Abbas, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Halimah binti Mohamed Kamar, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Supian bin Samat, PhD
Professor
Universiti Kebangsaan Malaysia
Malaysia
(External Examiner)

NORITAH OMAR, PhD
Associate Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 19 September 2014

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

Azmi Zakaria, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Raba 'ah Syahidah Azis, PhD
Researcher
Faculty of Science
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