EFFECTS OF THE ANNEALING TREATMENT ON THE ELECTRICAL AND DEGRADATION CHARACTERISTICS OF ZINC OXIDE-BASED VARISTOR CERAMICS [1]

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Abstract

The behavior of the leakage current that occurs in varistor ceramics is a central problem of the varistor technology. It limits the performance and the reliability of these surge voltage suppressors by means of the reduction of their electrothermal stability. The aim of this work is to evaluate the effect of the annealing thermal treatment on the electrical characteristics of the zinc oxide (ZnO)-based varistors. Conventional (ZnO-Bi_2O_3-based) and ZnO-Pr_6O_{11}-based varistor ceramics obtained by conventional ceramic processing were heat treated and their electrical and degradation characteristics were analyzed. Electrical characterization from voltage-current and voltage-capacitance data, besides of the heat dissipation factor and X-ray diffraction analysis were used to evaluate the effect of the annealing treatment on the electrical characteristics and the electrothermal stability of the produced varistors. The results of this study denote that the annealing treatment resulted in stabilization of the varistor ceramics studied, by means of the reduction of the leakage current, which is not occasioned by phase changes. The best results were obtained in the 600-700^{0}C temperature range and four hours of annealing time. In general, the rare-earth-ZnO-based varistor ceramics present better electrothermal stabilization level against degradation than conventional varistors when under the same thermal treatment conditions.

Key-words: Varistor ceramics; Eectroceramics; Annealing treatment.

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INTRODUCTION

Zinc oxide (ZnO) varistors (variable resistors) are polycrystalline ceramic devices exhibiting highly nonlinear (nonohmic) electrical behavior and greater energy absorption capabilities and are widely used as devices for overvoltage protection (GUPTA, 1990, p. 1817). The fabrication of ZnO varistors is done by mixing semiconducting ZnO powder with other oxide powders such as Bi, Co, Mn, and Pr, and subjecting the powder mixture to conventional ceramic pressing and liquid-phase sintering techniques (LAGRANGE, 1991, p. 1). The sintering results in a polycrystalline ceramic with a singular grain boundary property which produces the nonlinear current-voltage (I-U) characteristics of the device (SANTOS *et al.*, 1998, p. 1152), shown in Figure 1, which presents three regions: (i) Pre-breakdown region, where the I-U characteristic is ohmic and the ac current is about two orders of magnitude higher than the dc current for a given operating voltage; (ii) Nonlinear region, wherein the device conducts an increasingly large amount of current for a small increase in voltage; and (iii) Upturn region, where the I-U characteristic is again linear.

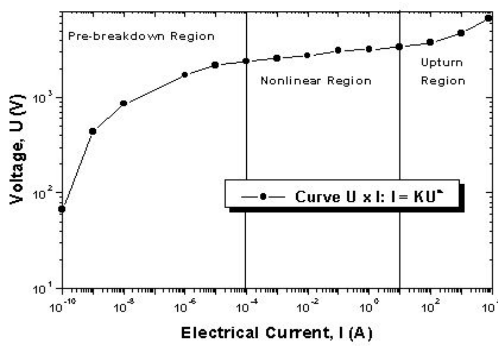


Figure 1. Typical current-voltage (I-U) characteristics of a zinc oxide varistor ceramic device. The curve is separated into three regions: pre-breakdown, nonlinear, and upturn.

The varistors have a tendency of increasing in leakage current (I_L) with increasing time and temperature. This behavior is known as the degradation phenomena and have been studied under ac, dc and pulse electric field. The mechanisms that have been proposed to account for this phenomena include electron trapping, dipole orientation, ion migration and oxygen desorption (CLARKE, 1999, p. 485). Because varistors are operated under voltage, any increase in the leakage current is of great importance. This is especially so for the surge arresters that are used in power distribution systems, because the power dissipated within the varistor represents a loss in distributable power and reduces the protection level of the system (GUPTA & STRAUB, 1990, p. 845).

For an ac application, the total leakage current in the pre-breakdown region (low current density region) is composed of a resistive current (I_{LR}) and a capacitive current (I_{LC}). The behavior of the I_{LR} (which is responsible for joule heating) with respect to time and temperature is of fundamental importance to the design and specification of a surge suppressor. The parameters which are know to influence the value of I_{LR} are the (i) chemical composition of the ZnO varistor, (ii) applied voltage, (iii) ambient temperature, and (iv) time duration of the applied voltage (GUPTA, 1990, p. 1817). The extent of the degradation process can be quantified in a number of ways. One is to express the resistive component of the leakage current, at a fixed voltage, according to an empirical power-law (GUPTA & STRAUB, 1990, p. 845):

$$I_{LR}(t) = K_R t^n \tag{1},$$

where $I_{LR}(t)$ is the I_{LR} at time t, K_R is an effective rate constant (corresponding to magnitude of the I_{LR} at time zero) and n a time exponent that gives a measure of the degree of stability of the device. In general, for an unstable device, the experimental data indicate that n is equal to 0.5 to 1, depending on preceding history and chemical composition. Commercial varistors present n value about 0.5 (LEITE *et al.*, 1990, p. 48). From studies with conventional varistors (ZnO-Bi_2O_3-based ceramics), it was observed that the value of the exponent n has been found to decrease considerably by subjecting the device to a thermal annealing treatment, resulting in a stabler device (GUPTA & STRAUB, 1990, p. 845; HUANG *et al.*, 1994, p. 1526; KIM *et al.*, 1986, p. 3491). In the present work an evaluation of the effect of the annealing treatment on the electrical and degradation characteristics of zinc oxide-based varistor ceramics was made and applied to the understanding of the varistor degradation behavior. The studied systems, with their chemical composition and sintering temperatures, are shown in Table 1 The aims of this study is to determine whether there is difference in activation energy and electrothermal behavior between unannealed and annealed samples for both varistor ceramic systems analyzed, and to find the optimized annealing temperature for these systems.

Table 1. Chemical composition and sintering temperature of the varistor ceramic investigated systems.

System	Chemical Composition (mol%)	Sint. Temp. (°C)
ZB	$96.5 \cdot ZnO$-$0.5 \cdot Bi_2O_3$-$1.0 \cdot Sb_2O_3$-$1.0 \cdot CoO$-$0.5 \cdot MnO$-$0.5 \cdot Cr_2O_3$	1250
ZPN	$97.5 \cdot ZnO$-$0.5 \cdot Pr_6O_{11}$-$1.0 \cdot CoO$-$1.0 \cdot Nd_2O_3$	1300

EXPERIMENTAL

Appropriate molar reagent grade powders were used to prepare the ZnO-based varistor ceramics. The sample compositions are shown in Table 1. The powders with adequate compositions were ball milled with zirconia balls in isopropyl alcohol media inside of a zirconia jar for 24 h. The resultant mixtures were dried at 110^0C for 12 h and calcined in air at 750^0C for 2 h. The calcined mixtures were granulated in a 200-mesh sieve and pressed into discs of 12.4 mm in diameter and 2.1 mm in thickness at a pressure of (80 $\pm$ 5) MPa. The discs were sintered at 1250^0C (ZB system) and 1300^0C (ZPN system) in air atmosphere for 2 h. The heating and cooling rates were 4^0C/min. The average size of the final samples was 10.2 mm in diameter and 1.1 mm in thickness. The sintered bodies were sanded and polished, and the silver electrode was deposited on both faces of the samples. The electrodes areas were approximately 0.22 cm^2. The post-sintering annealing treatment was made in the same furnace (RHF 1500 Carbolite) used in the sintering step. The samples were annealed at 600, 700 and 800^0C in air for annealing times from 1 to 6 hours.

The I-U characteristics of ceramics were measured using curve tracer source-measurement units (Tektronix 576, Wayne Kerr 3245) for unannealed (as sintered) and annealed samples. The breakdown electric field (E_B) was measured at 1.0 mA/cm^2 and the leakage current density (J_L) was measured at 80% of breakdown electric field. In addition, the nonlinear coefficient (α) was estimated for current-density range of 1.0 – 10.0 mA/cm^2. Adittionally, the sample microstructures were examined by scanning electron microscopy (SEM, ZEISS DSM 960) applied on the polished and 6M-NaOH aqueous solution-etched (5 min) surface of samples, and the crystalline phases were identified by powder X-ray diffraction (XRD, Diano XRD-8545, $\lambda CuK\alpha$ radiation). The activation energies for the electrothermal behavior of the I_{LR} were estimated from a plot of the resistive current versus temperature according to Arrhenius equation:

$$I_{LR}(T) = K_0 \cdot \exp\left(- \frac{E}{kT} \right) \quad (2),$$

where $I_{LR}(T)$ is the resistive current at temperature T (absolute temperature, in Kelvin), K_0 is a pre-exponential factor corresponding to I_{LR} at the initial temperature, E is the activation energy, and k is the Boltzmann constant.

RESULTS AND DISCUSSION

The Figure 2 presents the results obtained (varistor characteristics) from electrical measurements of the ZB and ZPN varistor ceramic systems in function of the time and temperature of annealing. It was verified that, in general, the increment of the annealing time did not affect significatively the values of the nonlinear coefficient, mainly up to four hours of treatment. The α values obtained for the annealing times equal to 5 and 6 hours are about 1% less than the original values (56.1 and 44.2 for the ZPN and ZB systems, respectively). The greatest reductions occurred for the annealing at 800^{0}C, for both systems. On the other hand, the decreasing of the E_B values is easily observed, mainly for annealing times greater than four hours, and, specially for the annealing treatment at 800^{0}C. In addition, it was verified that the leakage current density decreases for the annealing treatment done at 600 and 700^{0}C, but increases for the annealing at 800^{0}C, mainly for annealing time greater than three hours. Based on the precedent considerations, it was observed that, in general, annealing times greater than four hours are deleterious to varistor properties of the studied systems. Thus, the subsequent analysis are based on an annealing time equal to four hours for each temperature set.

The electrothermal behavior (in the interval to 25 to 175^{0}C) according to the Arrhenius characteristics of ln[$I_{LR}(T)$] versus the reciprocal of the absolute temperature are illustrated in Figure 3(a) for unannealed and annealed samples of the both varistor ceramic systems (ZB and ZPN). It was clearly noted that there are two temperature ranges that I_{LR} presents distinct behaviors: a low temperature range (25-75^{0}C) and a high temperature range (100-175^{0}C). The values of the activation energies and pre-exponential factor, for each annealing temperature and for each varistor system analyzed, were obtained from the linear regression fit of the Arrhenius plots shown in Figure 3(a). These results are presented in Table II.

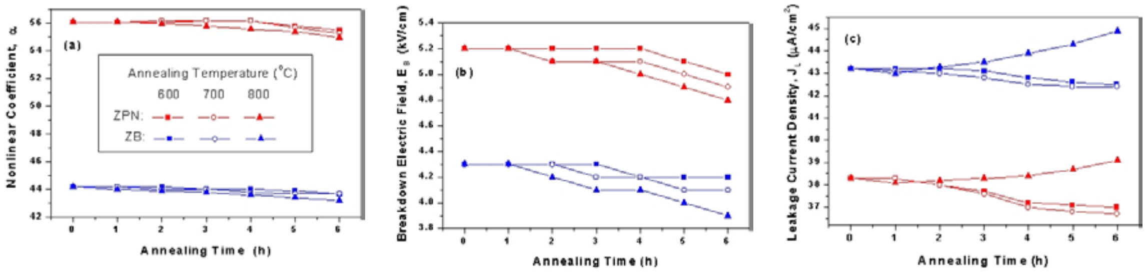


Figure 2. Varistor Characteristics of Investigated Samples (ZB and ZPN Systems) in function of the time and temperature of annealing: (a) Nonlinear coefficient, (b) Breakdown electric field, and (c) Leakage current density. Time equal to zero indicates unannealed (as sintered) samples.

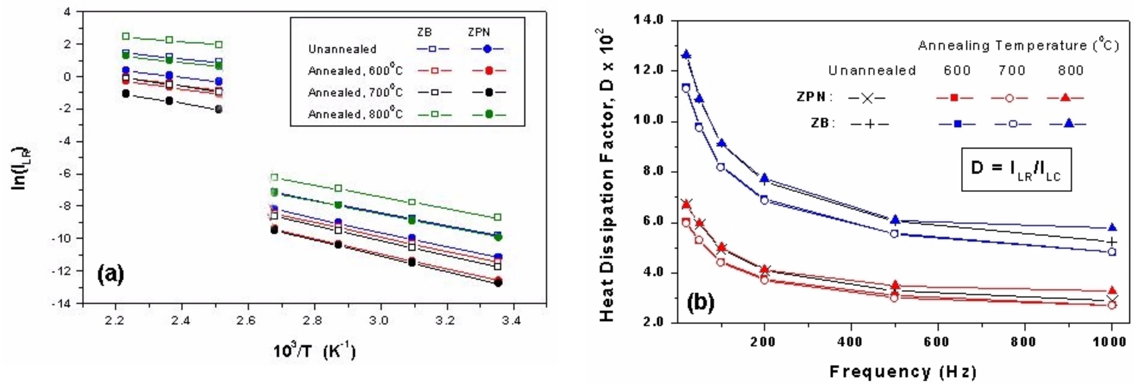


Figure 3. (a) Electrothermal behavior according to Arrhenius plots of I_{LR} for ZB and ZPN varistor ceramic systems unannealed and annealed at 600, 700, and 800^{0}C; (b) Heat dissipation factor analysis in function of the frequency of the applied alternate current (I_{LR} and I_{LC} are, respectively, the resistive and capacitive components of the leakage current).

Table 2. Activation energy (E) and pre-exponential factor (K_0) for the varistor ceramic systems ZB and ZPN.

Temp. Range	25-75^{0}C				100-175^{0}C			
Systems	**ZB**		**ZPN**		**ZB**		**ZPN**	
	Parameters [1, 2, 3]							
Condition	E	K_0	E	K_0	E	K_0	E	K_0
Unannealed [4]	0.341	31	0.373	29	0.173	372	0.215	380
Anneal, 600^{0}C	0.384	32	0.409	27	0.234	389	0.241	360
Anneal, 700^{0}C	0.397	40	0.422	38	0.251	590	0.288	582
Anneal, 800^{0}C	0.320	41	0.350	40	0.151	580	0.199	608

[1] *E (eV) e K_0 (μA).*
[2] *Mean of the three samples of each type and condition.*
[3] *Average discrepancy (δ) (all samples): $\delta E \cong 0.3$ %; $\delta K_0 \cong 0.8$ %.*
[4] *Without thermal annealing treatment (as sintered).*

The results shown in Table 2 indicate that the annealing treatments done at 600 and 700^{0}C promote the electrothermal stabilization of the varistor ceramic systems studied, since that result in increment in the activation energy values associated with the thermal evolution of I_{LR}. However, the annealing at 800^{0}C does not result in stabilization, causing decreasing in E values, also when compared with original values of the unannealed samples. These observations are in agreement with the results shown in Figure 2, which show the degenerescence of the varistor properties, mainly for the annealing at 800^{0}C. Corroborating these results, is the heat dissipation factor (D, ratio between the resistive and capacitive components of the leakage current) analysis in function of the frequency of the applied alternate current shown in Figure 3(b). In fact, it was verified that the highest values of D are associated to the unannealed and 800^{0}C-annealed samples. In addition, it was verified that the decrease in D was obtained by means of annealing treatments at 600 and 700^{0}C for both systems evaluated. In general, the D values to ZPN system were lower than for the ZB system, mainly in the low frequency region, which typically varistors are used in overvoltage protection systems. These results denote that ZPN system is more stable and present greater microstructural homogeneity than the ZB system, since that the heat dissipation factor analysis (dielectric losses) is an indication about of the microstructural homogeneity, as long as the presence of porosities, precipitaties, larger grain boundary phases segregated and polyphase microstructures which some phases do not play any role in the electrical conduction process, provide the existence of microstructural zones which present different temperature and current density profiles, in which the degradation process begin (SATO *et al.*, 1982, p. 8819; HUANG *et al.*, 1994, p. 1526; HE *et al.*, 2004, p. 138).

It has been shown (KIM *et al.*, 1986, p. 3491; GUPTA, 1990, p. 1817) that the stabilization by annealing can be caused by phase transformations or formation and annihilation of chemical-structural defects that result in removal of zinc interstitial species by chemical reactions at the grain boundary regions. According to grain boundary defect model for varistors (GUPTA, 1990, p. 1817), there is a grain boundary diffusion of oxygen during thermal annealing treatment in air or oxygen-rich atmosphere, followed by a sequence of reactions that result in the elimination of zinc interstitial species in the depletion layer and in the formation of a ZnO regular lattice site at the grain boundary region. This model supposes that the migration of zinc interstitial species is the main responsible for degradation phenomena (GUPTA, 1990, p. 1817; MANTAS *et al.*, 1995, p. 605; GARCIA *et al.*, 1999, p. 2123).

The varistor properties of ZnO-based ceramics have origin in Schottky potential barriers at grain boundaries, whose height (φ) is given by

$$\varphi = \frac{e^2 N_S^2}{2\varepsilon_r \varepsilon_0 N_D} \qquad (3),$$

where N_S is the number of energy state in the conduction band (trap density), ε_r is the relative permittivity of the ZnO, ε_0 is the permittivity of free space, e is the charge of the electron, and N_D is the donor density (carrier concentration in the ZnO grains). Thus, the Schottky potential barriers parameters, shown in the Table 3, were determined from capacitance-voltage measurements (MUKAE *et al.*, 1979, p. 4475), according to the equation below:

$$\left(\frac{1}{C} - \frac{1}{2C_0}\right)^2 = \frac{2N_G^2}{e\varepsilon_r \varepsilon_0 N_D}(\varphi + U) \qquad (4),$$

where C_0 and C are the capacitance per unit area of a grain boundary without and with bias, respectively, N_G is the average number of grains in sample thickness (determined by SEM analysis), and U the applied voltage per grain boundary. N_S and ν were obtained by adjusting equation:

$$N_S = 2\nu N_D = \left(\frac{2\,N_D\varepsilon_r\varepsilon_0\varphi}{e}\right)^{1/2} \quad (5).$$

Table 3. Electrostatic potential barrier characteristics [1, 2] in function of the thermal annealing conditions employed.

Annealing Treatment	N_D ($10^{18}/cm^3$)	N_S ($10^{12}/cm^2$)	φ (eV)	ν (nm)
ZB System				
Unannealed [3]	0.64	2.19	3.21	17.08
Anneal, 600^0C	0.69	2.06	2.67	14.90
Anneal, 700^0C	0.67	2.09	2.72	15.57
Anneal, 800^0C	0.64	2.12	2.98	16.54
ZPN System				
Unannealed [3]	0.66	2.25	3.38	17.03
Anneal, 600^0C	0.68	2.13	2.93	15.61
Anneal, 700^0C	0.69	2.17	3.03	15.75
Anneal, 800^0C	0.66	2.23	3.22	16.93

[1] *Mean of the three samples of each type and temperature annealing (4 h).*
[2] *Average discrepancy (δ): $\delta N_D \cong 2.5$ %; $\delta N_S \cong 2.2$ %; $\delta\varphi \cong 2.5$ %; $\delta\nu \cong 2.6$ %.*
[3] *Without thermal annealing treatment (as sintered).*

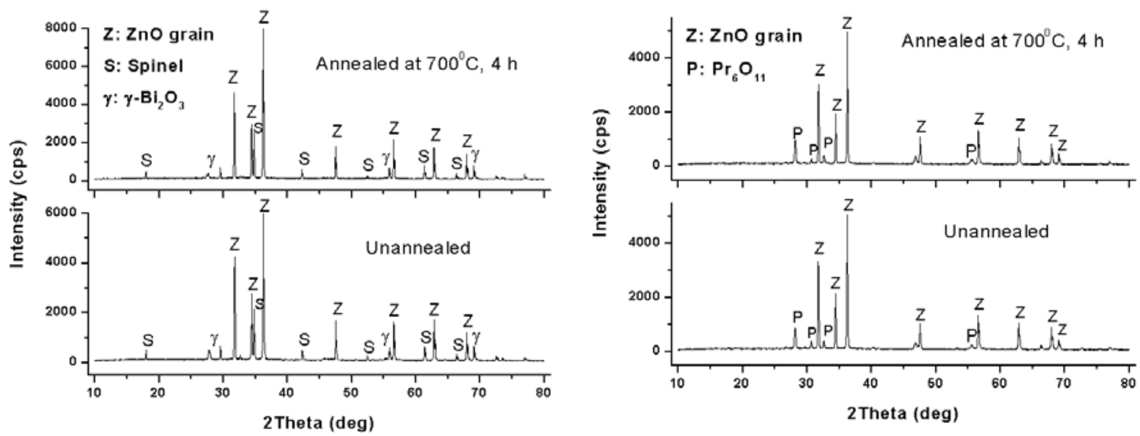
From the results shown in Table 3, it can be seen that the annealing treatments at 600 and 700^0C provided decrease in φ values by means of increase in N_D and diminution in N_S values. This can be due to migration of ions in depletion layer or through the grain boundary interfaces. In fact, the first hypothesis can be provoked by oxygen adsorption in the grain boundaries, resulting in the annihilation of zinc interstitial species; and the second hypothesis can be provoked by desorption of oxygen anions in the interface, reduction the N_S value and, consequently, decrease the height of the potential barrier (LEITE *et al.*, 1990, p. 48). Although, in general, these processes result in small reduction of the varistor properties, likewise results in stabilization of the varistors against the electrothermal degradation. Additionally, the XRD analysis shown in Figure 4 provide evidence that there is not phase transformations in analyzed systems. These results contrast with those obtained by Kim *et al.* (1986, p. 3491) for ZnO-Bi_2O_3-Sb_2O_3-based varistor ceramics.

The Figure 5 presents the results obtained about the I_{LR} evolution with respect the time, according to the equation:

$$\Delta I_{LR} = I_{LR}(t) - I_{LR}(0) = K_R t^n \quad (6),$$

where ΔI_{LR} is the time-dependent increase in I_{LR}, and $I_{LR}(0)$ is I_{LR} at time zero. It can be verified that for experiment at 50^0C practically there is not differentiation of behavior between unannealed and annealed samples at all annealing temperatures studied and for both varistor ceramic systems evaluated. On the other hand, the test at 100^0C indicates a difference in I_{LR} behavior. It is noted that the annealing treatment at 600^0C resulted in reduction in ΔI_{LR}, in relation to original (unannealed) and others temperature annealed samples. Furthermore, this difference was more significative for ZPN system as can be seen in the Figure 5(b). It is interesting to notice that the first test temperature corresponds to the average value of the low temperature range

(Figure 3(a)), showing that both systems are practically stable at this temperature (50^0C) during the test time interval. In contrast, the second test (at 100^0C, the initial temperature of the high temperature range) evidenced a greater growth of ΔI_{LR}. As a matter of fact, the continuity of this process results in that the power generated by the device can be bigger that the dissipated power leading to the electrothermal breakdown of the varistor ceramic. These observations are in accord to the previous results shown in Table II about the thermal behavior of I_{LR}.



(a) **(b)**

Figure 4. XRD patterns of unannealed (as sintered) and annealed (700^0C, 4 h) systems: (a) ZB, and (b) ZPN.

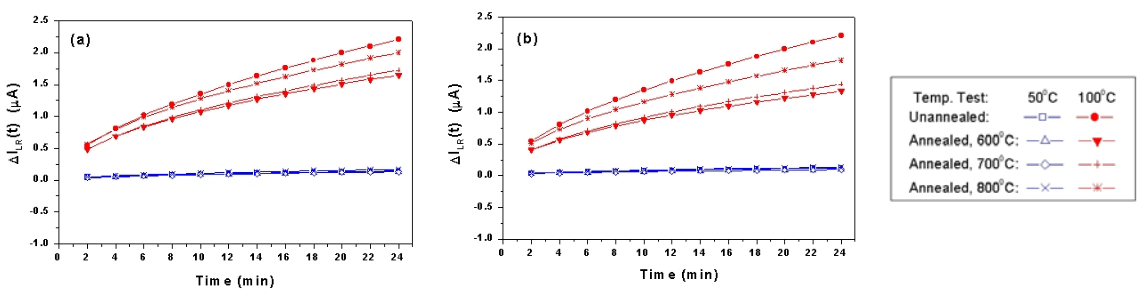


Figure 5. ΔI_{LR} curves for (a) ZB and (b) ZPN varistor ceramic systems unannealed and annealed at 600, 700, and 800^0C.

CONCLUSIONS

Based on the experimental results, it was verified that the resistive component of the leakage current, for the varistor ceramic systems analyzed, presented two different behaviors with respect to the temperature. One between 25 and 75^0C (more stable behavior interval), and other between 100 and 175^0C (where I_{LR} is more sensitive to temperature increment). Additionaly, it was demonstrated that the thermal annealing treatment provided the electrothermal stabilization of the varistor devices studied, by means of the decrease of the leakage current. The best results were obtained to annealing time equal to four hours and temperature range of 600-700^0C.

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EFEITOS DO TRATAMENTO DE RECOZIMENTO SOBRE AS CARACTERÍSTICAS ELÉTRICAS E NA DEGRADAÇÃO DE CERÂMICAS VARISTORAS À BASE DE ÓXIDO DE ZINCO [1]

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Resumo

Um problema central da tecnologia de varistores diz respeito ao comportamento da corrente de fuga que ocorre nestes dispositivos, estando relacionado às limitações no desempenho e na confiabilidade destes supressores de sobretensões. Este trabalho tem por objetivo avaliar os efeitos do tratamento de recozimento sobre as características elétricas de varistores à base de óxido de zinco (ZnO), tanto para varistores baseados no sistema $ZnO\text{-}Bi_2O_3$, quanto para o caso de varistores de ZnO dopados com terras-raras ($ZnO\text{-}Pr_6O_{11}$), os quais foram produzidos através do método cerâmico convencional. O tratamento térmico de recozimento foi efetuado em temperaturas na faixa de 600 a 800^0C durante intervalos de tempo compreendidos entre uma e seis horas. Caracterização elétrica consistindo na determinação das curvas tensão versus corrente, tensão versus capacitância e avaliação do comportamento da componente resistiva da corrente de fuga em função do tempo e da temperatura foi efetuada para avaliar os efeitos do tratamento de recozimento sobre as características elétricas e a estabilidade eletrotérmica dos varistores produzidos. Os resultados evidenciam que o tratamento de recozimento promove a estabilização das cerâmicas varistoras estudadas, mediante redução da corrente de fuga. Os melhores resultados foram obtidos na faixa de temperatura de 600 a 700^0C e com um tempo de recozimento igual a quatro horas. Em geral, os varistores à base de ZnO dopados com terras-raras apresentaram maior grau de estabilização que os varistores convencionais quando submetidos às mesmas condições de tratamento térmico.

Palavras-chave: Varistores; Eletrocerâmicas; Tratamento de recozimento.

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