



Electrophysical properties of ceramic–polymer composite films as function of sintering temperature



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Abstract: The effect of changes in sintering temperature on electrical properties and microstructure of ZnO–polyaniline–polyethylene composite ceramics, prepared in disk form at the pressure of 60 MPa and at five different temperatures, has been investigated. Increasing sintering temperature from 30 to 120 °C noticeably reduces breakdown voltage from 830 to 610 V. Further increase in sintering temperature causes breakdown voltage to increase. Interface voltage barrier height behaves differently when sintering temperature increases, which is in contrast to breakdown voltage behavior. These samples have a very low leakage current, a factor which indicates low degradation. Then again, the higher the sintering temperature gets, the less the nonlinear coefficient becomes. In addition, each sample has hysteresis which decreases through increase of sintering temperature up to 120 °C. Further increase in sintering temperature, however, causes the hysteresis loop to spread. Regarding UV spectra of the samples, it is revealed that there are three impurity levels whose behaviors against sintering temperature are subtractive. Analysis of composite samples by scanning electron microscopy indicates that their microstructure consists of grains and grain boundaries. Resistivity of grain boundaries is the main responsible factor for these changes in varistor characteristic as a function of sintering temperature.

Key words: ZnO; electrical properties; sintering temperature; varistor; composite; microstructure

1 Introduction

The need of surge protecting devices for electric power lines and electronic systems has motivated researchers to study ZnO-based electronic ceramic devices [1–3]. These devices are polycrystalline ceramics manufactured by sintering approximately 90% (molar fraction) ZnO powder, mixed with a great variety of cationic oxides such as Bi₂O₃, Sb₂O₃, MnO and CoO [4–10]. Due to their versatility, ZnO-based varistors have been widely applied in various electrical and electronic industries including power systems and semiconductor circuits in AC and DC fields, over a wide range of voltages from a few volts to tens of kilovolts, and a wide range of currents from microamperes to kilo amperes [11]. Highly nonlinear electrical behavior of ZnO varistors at high currents is quite important for applications of these devices [12–18]. It is well-known that nonlinear current–voltage characteristics of ZnO-based varistors are controlled by the well-recognized double Schottky barrier mechanism, which is triggered

by the contacting layer between ZnO grains and the second phase [19]. With the increasing demand for miniaturized devices in the electronic industry, small-sized electronic components including various low-voltage varistors are extensively desired [20]. To date, preparation methods of ZnO-based low-voltage varistors have been principally based on controlling the growth of ZnO grains or the thickness of electronic components [21–23]. In spite of their high performance, sintering temperature of these devices is still too high (more than 950 °C) [24]. Nowadays, surge arresters combine a complex metal oxide varistor technology inside a polymeric housing [25], which makes it possible to manufacture low-voltage protectors at lower sintering temperatures. While considerable research has been conducted on ceramic–polymer composite varistors, the effect of different sintering temperatures is still unavailable. Furthermore, the existing data on temperature dependence of electrical behavior of ZnO varistor ceramics are not only scarce but also contradictory. In order to study the quantitative relationship between sintering temperature and the performance

of a composite varistor, a composition of ZnO–polyaniline–polyethylene tiny particles was pressed at different sintering temperatures and its electrophysical properties were investigated.

2 Experimental

2.1 Sample preparation

Pure zinc oxide, dedoped polyaniline, and high-density polyethylene powders were sifted using a 200 U.S. mesh sieve. Then, 85% ZnO, 15% PANI and 5% HDPE (mass fraction) were weighed with the accuracy of 10^{-4} g. To have a completely homogeneous mixture, they were then admixed in a planetary mill for 8 h. Afterward, ZnO–polymer composite varistors were prepared at a pressure of 60 MPa at five different temperatures of 30, 60, 90, 120 and 130 °C. Final samples were in form of disks with a diameter of 10 mm and a thickness of 250 μ m.

2.2 Measurements of electrical properties

After observable qualities such as uniform thickness and lack of any cracks have been checked, samples were studied for their current–voltage characteristics using the two-probe method at direct current mode, after applying two copper electrodes with the effective diameter of 6 mm. By increasing the voltage to a degree which was higher than the breakdown voltage and subsequently, decreasing it to zero, it was possible to plot the hysteresis loops of the samples. UV absorption spectra of the varistors were recorded using a PHARMA spectrophotometer, SHIMADZU model 1700. UV absorption spectra, in the wavelength range of 300–1000 nm, were used to estimate the optical gap of each sample using $(ah\nu)^2$ versus $h\nu$ diagram. Finally, microstructures of samples were analyzed by investigating SEM micrographs which were obtained using MIRA3 TESCAN system.

3 Results and discussion

3.1 Current–voltage characteristics

Investigating the I – V characteristic of the prepared composite reveals that its current increases as a nonlinear function of voltage. At this type of nonlinearity, called varistor nonlinearity, I – V characteristic includes three different regions: pre-breakdown region at which the sample has high resistance, breakdown region at which resistance of the sample decreases dramatically, and upturn region at which the sample has low resistance (Fig. 1). The importance of pre-breakdown zone is due to its current, called leakage current, which is an important factor in determining sample degradation. Since current through varistor is mainly controlled by grain resistance,

an important aspect of grain resistivity in varistor ceramics is the irreversible electrical breakdown at high currents [26,27]. The nature of such electrical breakdown in varistors has not been sufficiently investigated yet. If the irreversible breakdown has a thermal nature, then it could occur due to the current instability provided that the temperature coefficient of the grain resistivity is negative [28]. It can be concluded that high leakage current results in temperature increment as a consequence of Joule heating ($Q=RI^2t$), which facilitates the development of current elements between the two electrodes. These elements let electrons flow between two conductive plates, a process which yields a perforated sample. Leakage current of this sample is very low (about 0.1 μ A), a factor which indicates low degradation. It is noteworthy that in case the applied voltage is further increased, increment of current will no longer be linear. The voltage at which nonlinear behavior starts is called breakdown voltage. At this region, the current varies as a function of voltage as follows: $I=kV^\alpha$, where k is a constant and α is the nonlinear coefficient. Greater voltage increase causes I – V characteristic to become linear again. The interesting point about this composite is that its resistance is infinite with very high accuracy (about $10^9 \Omega$) at the pre-breakdown zone, which reaches about $10^5 \Omega$ at upturn zone. In this stage, the sample could be considered an ideal voltage protector to be used in microelectronic industry.

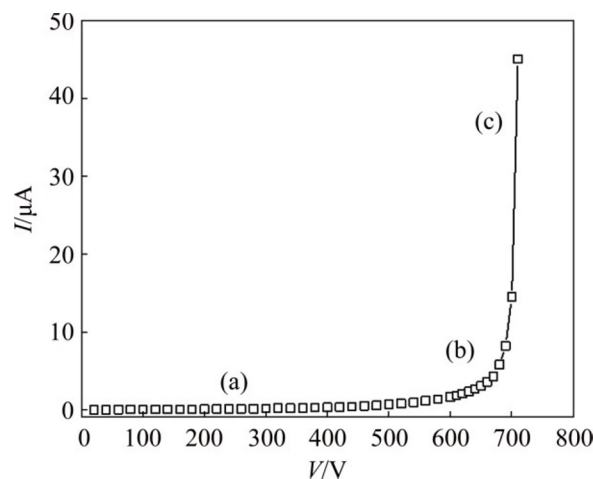


Fig. 1 I – V characteristic of composite varistor: (a) Pre-breakdown region; (b) Breakdown region; (c) Upturn region

Comparison of I – V characteristics of samples prepared at different temperatures shows that by increasing sintering temperature, breakdown voltage of samples decreases whereas their leakage current remains almost constant. Due to low leakage current (about 0.1 μ A), degradation probability is low. Breakdown voltage decrement stops once sintering temperature reaches an optimum and then, increase in sintering

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