Improvement of Non-linear Characteristics of Multicomponent ZnO-based Ceramics Containing Nb_2O_5

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ABSTRACT

The combined effect of minor variations in the Nb_2O_5 content and initial particle size on the current-voltage characteristics and energy handling capability of ZnO based composites is reported. Samples containing 0.2 wt% of Nb_2O_5 along with other additive oxides, prepared as a finely milled mixture were found to posses the best overall properties, characterized by high non-linearity index ($\simeq$60), low leakage current (0.6–1.2 $\mu A/cm^2$), high energy handling capability (>325 J/cm^3), and long life (>150 yr), among the samples studied. Addition of Nb_2O_5 above 0.2 wt%, maintaining other parameters constant, was found to affect the properties drastically.

INTRODUCTION

The gapless surge arresters based on zinc oxide are extensively used in recent years because of their excellent protection characteristics against transients in electrical circuits. The working principle of these ZnO elements is similar to that of a back-to-back diode which enables it to limit an overvoltage of either polarity [1–2]. Extensive studies have been carried out to understand the mechanism of conduction [3–8] and the degradation phenomenon [1,9–13] caused by electric stress in ZnO elements since 1970. It has been observed that the characteristics of the ZnO non-linear resistor elements are significantly influenced by the composition as well as the processing techniques [14]. Because of the increasing use of the device, it is essential to understand how the processing parameters influence the performance. The role of additive oxides and selected processing parameters on the physical and electrical properties are reviewed elsewhere [15–17].

This paper highlights the combined effect of minor variations in the composition and initial particle size on the *V-I* characteristics and energy handling capabiiity of ZnO based composites. Studies on the stability and life time of selected ZnO based non-linear resistor are also discussed. It may be mentioned that studies have

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been carried out on 15 mm diameter and 3 mm thick samples as facilities to prepare larger diameter samples were not readily available.

EXPERIMENTAL PROCEDURE

SAMPLE PREPARATION

ZnO based composites were prepared by the conventional ceramic fabrication procedures as described elsewhere [14]. Reagent grade (99.9%) pure) ZnO and other additive oxides of the type Nb_2O_5 , Bi_2O_3 , Sb_2O_3 , CoO, MnO_2 , Cr_2O_3 , NiO and Al_2O_3 were used for the preparation of the samples. The major steps involved in the preparation of the samples are summarized below.

Step 1: Calcination of ZnO and Nb_2O_5 mixture at 1200°C for 1 h. The Nb_2O_5 content was 0.2, 0.25 or 0.3wt%.

Step 2: The calcined mixture was ball milled for 10 h using deionized water as a grinding medium.

Step 3: Mixing the ball milled charge and other additive oxides in the appropriate proportion mentioned below. 6 wt% Bi_2O_3 , 3.5%Sb_2O_3 , 0.55%CoO, 0.7% MnO_2 , 0.9%Cr_2O_3 , 0.9% NiO and 0.025% $Al(NO_3)_3$

Step 4: Ball milling the mixture for 10, 20 or 30 h. The milled mixture was characterized with respect to particle size using a laser granulometer [Cilas-Alcatel model 715 (France)]

Step 5: Pelletization at a pressure of 30 MPa, followed by sintering at 1250°C for 2 h and post-sintering at 1100°C for 10 h.

The final dimensions of the sintered samples were about 15 mm diameter and 3 mm thickness.

CERAMOGRAPHY

The surface of the sintered samples was lapped and polished to a mirror-like finish using micro-cloth polishing wheel as described in detail elsewhere [16]. The polished surface was mild-etched with 5% glacial acetic acid. The etched surface was examined by scanning electron microscope (SEM). The surface of the samples was metallized with a thin coating of gold to reduce the charging effects and to improve the definition and resolution of the image.

CURRENT-VOLTAGE CHARACTERISTICS AND ENERGY HANDLING CAPABILITY

The sintered samples were lapped to remove surface flaws and an ohmic contact was provided on both surfaces by coating with conductive silver paint. These

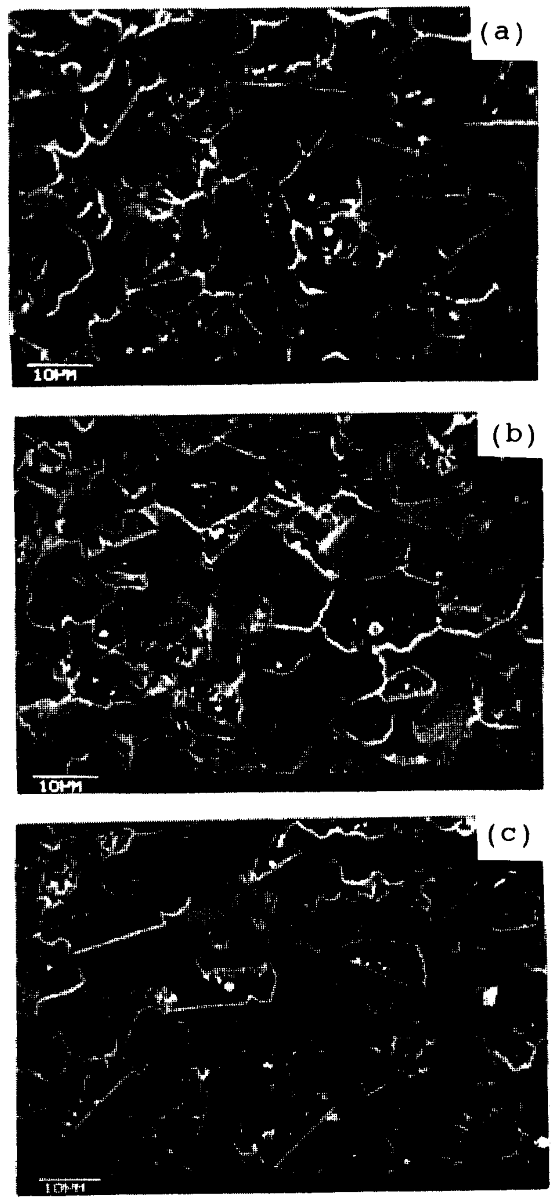

Figure 1.

Scanning electron micrographs of the samples of type A. The milling time of the samples are (a) 10 h, (b) 20 h and (c) 30 h.

samples were characterized for their V-I behavior and energy handling capability test as described earlier [18]. The non-linearity index α was calculated in a current range of 1 mA to 1 A. The leakage current was calculated at 80% $V_{60\mu A}$ of the samples (15 mm dia and 3 mm thick) which is equivalent to 80% V_{1mA} of the standard ZnO non-linear resistor (62 mm dia and 30 mm thick). The protective ratio, also known as voltage ratio was calculated using the voltages corresponding to the currents of 450 A and 60 μA obtained from V-I data.

RESULTS

It has been found that the sample having the composition, 87.325 wt% ZnO + 0.1% Nb_2O_5 + 3.5% Sb_2O_3 + 6% Bi_2O_3 + 0.55% CoO + 0.7% MnO_2 + 0.9% Cr_2O_3 + 0.9% NiO + 0.025% $Al(NO_3)_3$ subjected to calcination at 1200℃ for 1 h, ball milling for 30 h, sintering at 1250℃ for 2 h and post-sintering at 1100℃ for 10 h was found to possess good non-linear characteristics [17]. The samples prepared as described above were found to exhibit non-linearity index $\alpha \simeq 60$, leakage current $\simeq$ 3 to 4 μA and voltage ratio $\simeq$ 1.89. It was also observed that the leakage current increased to 6 μA when the sample was subjected to high energy pulses of $\simeq$ 300 J/cm^3. This level of leakage current is considered too high from the view point of life. Attempts were therefore made to improve the characteristics further by varying the amount of Nb_2O_5 . This is based on the information obtained from the ZnO-Nb_2O_5 binary system [15]. In the present work the amount of Nb_2O_5 in the above sample was increased from 0.1 wt% to 0.2, 0.25 and 0.3wt%, keeping the proportion of other additive oxides constant. The procedure adopted for the preparation of the samples is described already. The samples containing 0.2, 0.25 and 0.3 wt% Nb_2O_5 are designated as types A(10/20/30), B(10/20/30) and C(10/20/30), respectively. The numbers 10, 20, and 30 indicate the time of ball milling in hours (Step 4).

Figure 1 shows the scanning electron micrographs of samples of type A. The Figure reveals that the grain size of the sintered products increases with the decrease in initial particle size. The V-I characteristics of the as-sintered and post-sintered samples containing 0.2 wt% Nb_2O_5 (Type A) are shown in Figures 2 and 3. Table 1 summarizes the electrical characteristics of type A samples obtained from their V-I data. The as-sintered samples of type A(30) are found to possess the highest value of non-linearity index, α = 67, compared to that of all the other combinations studied in the present work. The value of α for post-sintered sample of type A(30) being the next highest, α = 60. However, the post-sintered sample of type A(30) is significantly better from the consideration of a low leakage current ($\simeq$ 1 to 2 μA) and a low voltage ratio ($\simeq$ 1.67) compared to other samples. Further, the post-sintered samples withstood high energy pulses of about 325 J/cm^3. This energy level being the highest available in the laboratory, a number of pulses were given at the same level and the effect on leakage current was studied. Prior to the first pulse, the leakage current was in the range of 1 to 2 μA and the voltage ratio was $\simeq$ 1.67. After the first high energy pulse (at 325 J/cm^3) was applied, the leakage current and the voltage ratio were 3 μA and 1.69 respectively. This was followed by nine pulses (at the same energy level) at 1 min intervals. The leakage current and the voltage ratio remained sensibly constant. It is quite possible that this sample may withstand higher energy pulses than the highest available energy level of 325 J/cm^3. However, it is believed that this energy han-

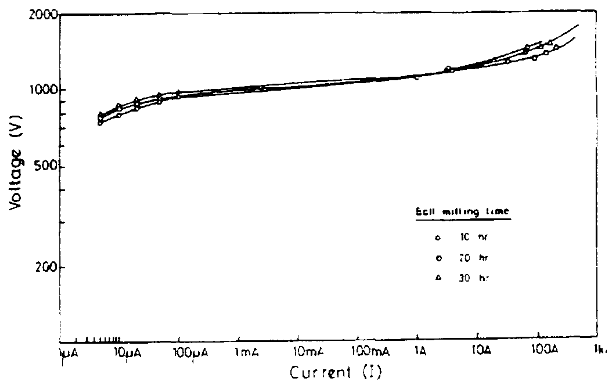

Figure 2.

V-I Characteristics of as-sintered samples of Type A.

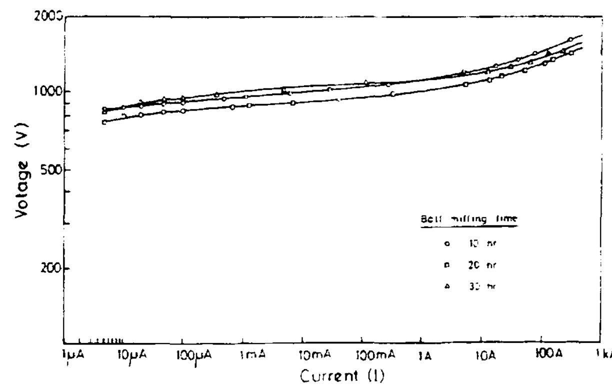

Figure 3.

V-I Characteristics of post-sintered samples of Type A.

Table 1.
Influence of initial particle size on non-linearity index(α),leakage current and voltage ratio of samples of Type A, B and C.

Ball milling time (hours)	Average Particle size (µm)	Non-linearity index (α) (60 µA to 1 A)	Leakage Current (µA)	Voltage ratio (45 µA - 450A)
TYPE A (as-sintered)				
10	3.7	48	4.5	1.88
20	1.6	53	3-4	1.75
30	1.0	67	4	1.77
TYPE A (post-sintered)				
10	3.7	47	1	1.86
20	1.6	54	1	1.80
30	1.0	61	1-2	1.67
TYPE B (as-sintered)				
10	3.8	40	20	2.02
20	1.8	41	18	1.985
30	1.0	43	15	1.972
TYPE C (as-sintered)				
10	4.0	38	22	2.63
20	2.0	38	18-19	2.54
30	1.1	42	16	2.43

dling capability is adequate for EHV systems. Higher energy handling capabilities can be obtained by using parallel stacks of the ZnO non-linear resistor elements. Present experience has shown that several samples of a batch have extremely close *V-I* characteristics, enabling easy formation of parallel stacks.

The studies were also extended to the samples containing 0.25 wt% (type B) and 0.3 wt% Nb_2O_5 (type C). Figure 4 shows selected scanning electron micrographs of samples of types B and C. It is observed that the grain size increases marginally with decrease in initial particle size. The grain shape of type C (20/30) samples are different from other types of samples as evidenced by the microstructural studies (Figure 4). The non-linear properties of the samples of types B and C are summarized in Table 1. It can be inferred from the results that the characteristics of as-sintered samples of types B and C are inferior to those of type A. These samples exhibit lower non-linearity index and higher leakage current and voltage ratio compared to that of type A(30).

The increase of Nb_2O_5 content resulting in excessive formation of $Zn_3Nb_2O_8$ spinel phase is believed to be responsible for the poor electrical characteristics of type B and C samples. Similar results have been observed for the ZnO-Nb_2O_5 binary system [15]. Attempts have been made to improve the characteristics of these samples by post-sintering heat treatment at 1100℃ for 10 h. It was observed that though the post-sintering heat treatment improved the properties, the level of leakage current and voltage ratio was high compared to that of type A sample. Figure 5 shows typical *V-I* characteristics of types B and C. Further, it was observed that the samples containing 0.25 and 0.3 wt% Nb_2O_5 (i.e. types B and C) withstood energy pulses of about 230 and 150 J/cm^3 respectively. These samples were found to be degraded considerably after subjecting to higher energy pulses. Hence, they are considered unsuitable for field applications.

From the above results, it is evident that the sample (type A) containing 0.2 wt% Nb_2O_5 and ball milled for 30 h (initial particle size $\simeq 1 \mu m$) exhibits superior characteristics compared to the other types of samples studied in the present work. Further the characteristics of these samples are found to be reproducible within the limits of experimental error.

DISCUSSION

It has been reported by many investigators [1–6] that multi-component ZnO based ceramics containing additive oxides of Bi, Sb, Co, Mn, Cr and Al exhibit good non-linear characteristics, $\alpha \simeq 50$ and energy handling capability of about 250 J/cm^3. These samples were prepared via calcination at lower temperatures, < 800℃. It is generally known that ZnO samples possessing high non-linearity index ($\alpha > 50$) tend to have relatively poor energy handling capability. On the contrary, in the present investigation, samples containing 0.2 wt% Nb_2O_5 in addition to other additives mentioned above, prepared via calcination of ZnO and Nb_2O_5 mixture at a higher temperature (1200℃) and ball milling the calcined powder and other additive oxides for 30 h (particle size $\simeq 1$ μm), were found to exhibit high non-linearity index ($\alpha > 60$) coupled with high energy handling capability (> 325 J/cm^3). It is therefore reasonable to expect that addition of Nb_2O_5 , calcination at higher temperatures and ball milling for longer duration to reduce the initial particle size are responsible for the improvement in the non-linearity index as well as the energy handling capability. In order to understand the effect of Nb_2O_5 , studies have been made of the non-ohmic behavior of ZnO-Nb_2O_5 binary system as a function of composition in the range of 0.1 to 0.5 wt% of Nb_2O_5 and sintering temperature varying from 900 to 1300℃ [15] It was found that the non-linearity index (α) varies with

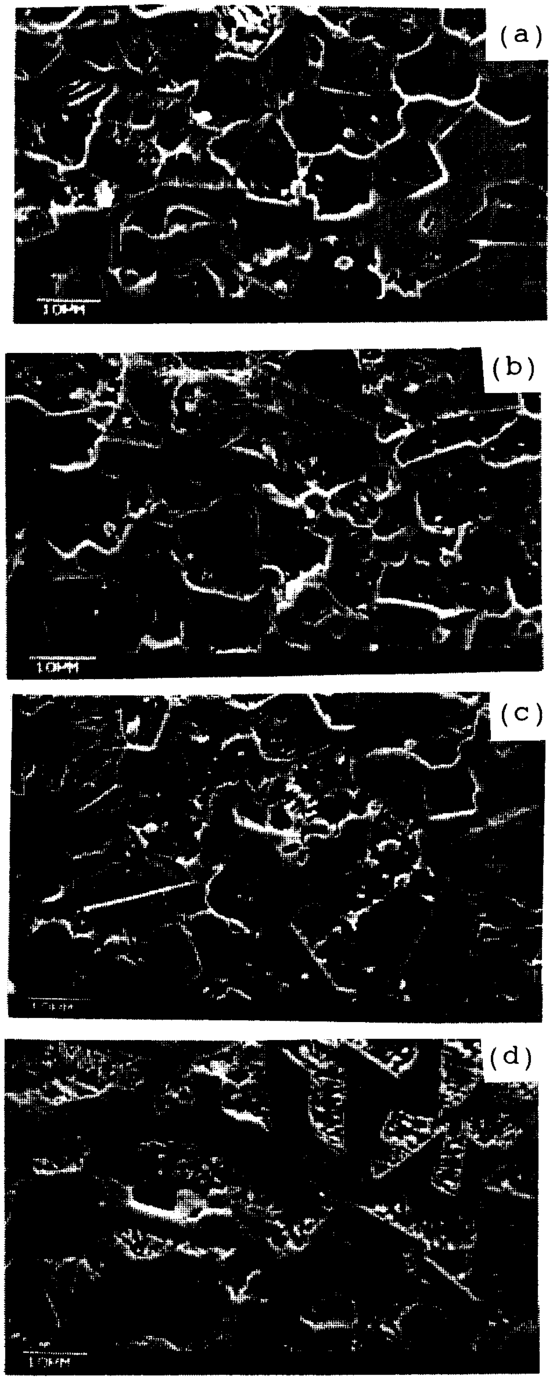

Figure 4.

Scanning electron micrographs of the samples of the Types B and C: (a) B(20), (b) B(30), (c) C(20), (d) C(30). The numbers in the bracket indicate the ball milling time in h.

Nb_2O_5 content and sintering temperature. It was observed that at a constant sintering temperature, the α value increases with Nb_2O_5 content ≤ 0.2 wt% and further addition of Nb_2O_5 decreases the α value. Similarly at constant composition, the α value increases with sintering temperature $\leq$ 1100℃ and then it decreases beyond this temperature. The sample containing 0.2 wt% Nb_2O_5 , sintered at 1100℃ was found to possess the maximum α value of $\simeq 8$. Such a behavior can be attributed to the precipitation of $Zn_3Nb_2O_8$ spinel phase as evidenced by x-ray studies [15]. The spinel phase is presumed to possess different electrical resistivity compared to the ZnO matrix thereby imparting non-ohmic behavior. Further, extensive investigation on ternary and higher component ZnO based systems containing Nb_2O_5 as a special additive have shown that, in general, samples containing Nb_2O_5 posess better electrical characteristics compared to that of the systems without Nb_2O_5 as reported earlier [14,16] These studies confirm that the formation of $Zn_3Nb_2O_8$ spinel phase is primarily responsible for the impovement in the non-linear characteristics of the multi-component ZnO based ceramics, as this phase is expected to form during calcination of ZnO-Nb_2O_5 mixture as explained earlier.

The improvement in the characteristics due to the formation of $Zn_3Nb_2O_8$ is confirmed by studying the samples prepared by mixing the additive oxides (including Nb_2O_5 with calcined pure ZnO powder. It is interesting to note that these samples posess inferior non-linear characteristics compared to that of the samples prepared via calcination of ZnO-Nb_2O_5 mixture. The x-ray diffractogram of the former sample does not provide any evidence for the fomation of $Zn_3Nb_2O_8$ spinel phase as was observed in the latter sample. Despite this, it is presumed that the number of free electrons available for conduction is increased by the addition of Nb_2O_5 thereby resulting in higher non-linearity and lower voltage ratio (Table 1).

Studies were extended to the samples prepared by calcining ZnO + Nb_2O_5 mixture at lower temperatures (< 1200℃) to confirm the importance of calcination at 1200℃. These samples were found to exhibit relatively low non-lineaity index ($\simeq$ 45) and poor energy handling capability ($\simeq$ 250 J/cm^3) compared to that.of the sample calcined at 1200℃. It is presumed that calcination treatment at higher temperatures reduce the density of imperfections of the material. Besides this, the degree of crystallinity and the particle size of the powder increases with the calcined temperature [19]. Since both these parameters are, in general, inversely related to the reactivity of the calcined powder, the calcining temperature is very important.

However, it should be noted that the influence of calcining temperature on sinterability is not precisely understood [19]. The samples prepared without calcination treatment or calcination at lower temperatures results in poor sinterability due to the absorbed moisture and the void formation during compaction [19]. On the other hand, samples prepared via calcination at higher temperatures exhibit only marginal improvement in the non-linear characteristics. This is because the particle size of the powder increases with calcining temperature. To overcome this problem, the powder calcined at higher temperatures was ball milled for longer duration to reduce the particle size of the mixture. Milling after calcination is therefore required to improve the reactivity of the powder.

It has been observed that the energy handling capability of ZnO-based ceramics depends strongly on the uniformity in the composite, including chemical and granular homogeneity [20,21]. It is also known that the microstructure of sintered products depend primarily on the additive oxides and the initial powder characteristics such as particle size and its distribution and absorbed moisture. In the present work, calcination of ZnO + Nb_2O_5 mixture not only leads to the formation of $Zn_3Nb_2O_8$ spinel phase and also aids to remove the absorbed moisture. The particle size distribution is controlled by milling operation as already discussed. The milling was carried out in an agate ball mill to avoid impurities. Care was taken to get a narrow particle size distribution so that large particles do not seed secondary grain growth during sintering. Thus the chemical and microstructural uniformity was achieved to improve the energy handling capability.

It may be concluded from the foregoing discussion that addition of Nb_2O_5 , calcination at higher temperature and ball milling for longer duration results in good non-linearity coupled with high energy handling capability.

STABILITY OF ZnO-BASED NLR

The voltage-current characteristics of ZnO-based NLRs presented until now pertain to room temperature (30℃). All types of ZnO based NLRs exhibit a negative temperature coefficient of resistance in the leakage current region. That is, at a given voltage, the current increases with temperature. This characteristic is of great importance in field application. If the temperature of the NLR rises beyond certain level, the increase in leakage current would cause a further rise in temperature which in turn increases the leakage current. This constitutes 'thermal runaway' leading to failure of the arrester. This occurs when the thermal energy generated within the element exceeds the rate of heat dissipation. In practice, the temperature rise is due to several factors.

(1) Steady state power dissipation (given by the product of steady state voltage and steady state current).

(2) Increase in the ambient temperature: The temperature of NLRs operating in a hot climate with long exposure to direct sun light (as experienced in locations such as some parts of Africa and USA) would be high. In some parts of India such as Rajasthan, Andhra Pradesh and Tamil Nadu, the ambient temperature can go as high as 45℃ in summer.

(3) Temporary overvoltages: These voltages are relatively of low magnitude (about 1.1 to 1.2 per unit) but are of a long duration ($\simeq$ 2 ms to $\simeq$ 20 s), and

(4) Transient overvoltages caused by lightning and system switching operations: Lightning can cause flow of very high currents ($\simeq$ 2 kA to $\simeq$ 30 kA) for a very short duration ($\simeq$ 30 μs). Switching overvoltages are, by far, the most severe as it can involve $\simeq 100 to 1000$ A flowing for ms duration.

Furthermore, the weighted average temperature of the porcelain housing of ZnO arresters has been reported to be 45℃ in a high temperature location (Phoenix, Arizona). The temperature of the NLR elements may be higher by about 15℃ and therefore the temperature of the element would be around 60℃. It is believed that temperature rise due to temporary overvoltages or transient surges could be about 30 to 40℃. Thus, the *V-I* characteristics from room temperature to $\leq$ 100℃ are of practical interest.

In view of the above, it is imperative to study the influence of temperature on voltage-current characteristics, in the leakage current region. The sample Type A(30), possessing the best non-linear characteristics among the samples studied, was chosen for the above study. The current-voltage measurements were made on this sample at different temperatures ranging from room temperature (30℃) to 100℃ in a current range of 1 μA to 1 mA. Figure 6 shows the *V-I* characteristics of the sample Type A(30) at different temperatures. It can be inferred from the figure that the leakage current region is affected considerably by the change in temperature. However, the non-linear region is not affected. The leakage current of the sample is around 18 μA at 100℃ (at the continuous operating voltage). It was also found that the current was quite stable under these conditions. Further, the voltage was raised to the rated

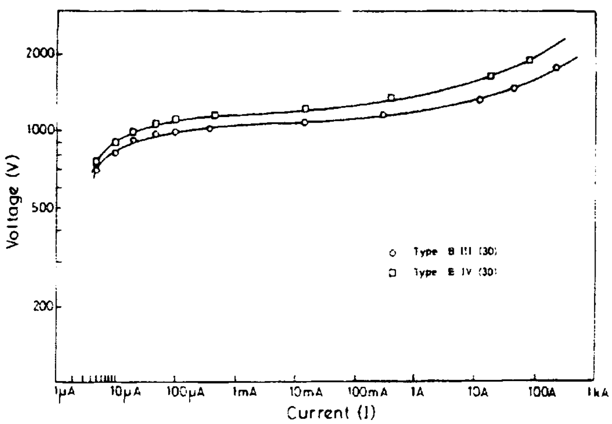

Figure 5.

V-I Characteristics of the post-sintered samples of the types B and C (ball milling time - 30 h).

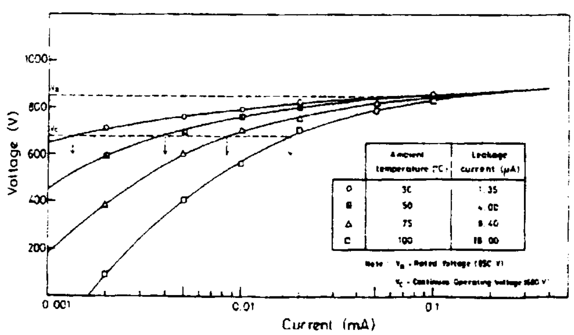

Figure 6.

V-I Characteristics of Type A(30) sample at different ambient temperatures.

voltage V_R and maintained for one minute and the current was found to be quite stable. It may therefore be concluded that the characteristics of the sample, Type A(30), developed in the present work are quite stable.

LIFE ESTIMATION

The *V-I* characteristics of the ZnO based NLRs are known to undergo a gradual degradation in service due to the continuous power dissipation. It is therefore very important to assess its 'life'. This has been widely done on the basis of the Arrhenius law [22–23]. This law states that the rate of any thermally activated reaction is governed by the relation

$$R' = R'_o \exp[-Q/RT] \quad (1)$$

where R' is the rate of the reaction, R'_0 is a rate constant, Q the activation energy, T the temperature, and R the universal gas constant. The 'life' of any device subject to degradation as per the above relationship may therefore be defined as the time taken for a certain specified change in a property related to the reaction. In the case of ZnO-based NLR, the time taken (at a given temperature) for the power loss to double (from its initial value) is currently accepted as its life at that temperature. As the applied voltage is sensibly a constant, doubling of power loss implies doubling of the leakage current. In the present investigation, samples pepared with and without Nb_2O_5 (keeping all other parameters constant as explained above) were subjected to life estimation on the basis of the Arrhenius law. The time for doubling of the leakage current was measured in the temperature range of 120 to 160℃. Plots of $1/T$ against time to double the leakage current were extrapolated to determine the life of NLR elements at 50℃. Several samples of each kind were subjected to the above test. The estimated life of the samples containing Nb_2O_5 [A(30)] was found to be in the range 160 to 200 yr while the samples without Nb_2O_5 had a lifespan of 90 to 120 yr. It is evident that addition of Nb_2O_5 has significantly improved life of the varistor.

CONCLUSIONS

1. Minor variations in the initial particle size (in the order of a few μm) and Nb_2O_5 content (in the order of 0.1 wt%) modify the overall properties of ZnO based non-linear resistor singificantly.

2. The samples (Type A), containing 0.2 wt% Nb_2O_5 along with other additives, ball milled for 30 h (initial particle size $\simeq$ 1 μm), sintered at 1250℃ for 2 h and post-sintered at 1100℃ for 10 h possess the best overall properties, characterized by high non-linearity index ($\alpha \simeq 60$), low leakage current ($\simeq$ 1 to 2 μA) and high energy handling capability ($\simeq$ 325 J/cm^3).

3. Increase of Nb_2O_5 content beyond 0.2 wt% results in poor electrical characteristics.

4. The characteristics of type A samples are found to be highly stable.

5. The life-time of the sample type A is estimated to be around 160 yr.

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