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Effects of Ta2O5 on the microstructure and electrical properties of ZnO linear resistance ceramics

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Abstract

ZnO linear resistance ceramics were synthesized from ZnO-Al2O3-MgO-TiO2-SiO2-Ta2O5 by a conventional ceramics method. Effects of Ta2O5 on the phase composition, microstructures, and electrical properties of ZnO linear resistive ceramics were investigated. The results show that doping with appropriate amount of Ta2O5 can refine the grains of the main crystalline phase ZnO and the secondary crystalline phase ZnAl2O4 in terms of microstructure, and also can reduce the grain boundary barrier and optimize the I–V characteristics in terms of electrical properties. In addition, the doping of Ta2O5 can improve the stability of the resistivity, and the impedance frequency indicates that the doping of Ta2O5 makes the sample suitable for high-frequency electric fields. The resistivity of the sample doped with 0.2 mol% Ta2O5 is 56.2 Ω·cm, and this sample has the best grain boundary barrier height, nonlinear coefficient and temperature coefficient of resistance of 0.054 eV, 1.04 and −3.48 × 10−3 °C−1, respectively.

1. Introduction

As a semiconductor material with wide direct band gap and large excitation binding energy, ZnO has good optical and electrical properties, Which make it widely used in short-wavelength optoelectronics devices, transparent conducting layers for displays, photocatalysts, photoconductors [1–4]. ZnO has been used as varistors [5] and linear resistance ceramics as well. ZnO linear resistance ceramics studied in this paper have special nonlinearity in current and voltage, small resistivity fluctuations, small resistance temperature coefficient and large absorbed energy [6, 7]. Compared with carbon-based resistors [8], ZnO linear resistors exhibit characteristics that are less susceptible to oxidation, easy to control resistivity, good stability, and high flux density. Compared with metal-type resistors, ZnO linear resistors consist mainly of non-ohmic ZnAl2O4 phase and low-ohmic phase ZnO, which have linear I–V characteristics [9], small resistance temperature coefficient, and high serviceable temperature. This makes it more suitable for application in the power-electronics sector, especially in overvoltage protection systems.

In recent years, many reports have discussed the influence of different dopants on the properties of ZnO linear resistive ceramics. According to reports, Al2O3 could play an active role in controlling the grain growth and regulating the resistance [10, 11]. MgO promoted the development of the temperature coefficient of resistance in the positive direction [12], and TiO2 was more friendly to the stability of resistivity [13]. Besides, the properties of ZnO linear resistance ceramics were improved by doping some rare earth oxides (La2O3 [14], Y2O3 [15], Sm2O3 [16]). Based on the characteristics of ZnO linear resistance materials, the Fermi energy level at the grain boundary is smaller than that at the grain. However, a high grain boundary barrier is detrimental to linear I–V characteristics [17].
Nahm [18] mentioned in the report of Ta$_2$O$_5$ doped ZnO–V$_2$O$_5$–MnO$_2$ ceramics that when the doping amount of Ta$_2$O$_5$ was more than 0.05 mol%, its nonlinear coefficient was significantly reduced, and the grain growth could be limited to promote the densification of ceramics. Tominc Sara [19] also mentioned that the addition of Ta$_2$O$_5$ led to the nonlinear collapse. In addition, Ta$^{5+}$ has a large ionic radius and trivalent cation equivalence, which is similar to the ionic radius [20] of Zn$^{2+}$, which make it easy to dissolve into the ZnO lattice and cause donor doping. All this indicated that Ta$_2$O$_5$ may be a good additive for ZnO linear resistance ceramics. However, there are few reports on the doping of Ta$_2$O$_5$ in related aspects. This paper presents a systematic study on the influence of Ta$_2$O$_5$ doping on microstructure, phase composition, and electrical properties of ZnO linear resistance ceramics, and the stability of resistance was also discussed.

2. Experimental

2.1. Raw materials

Samples were prepared with a formulation of (75-x) mol% ZnO-12 mol% Al$_2$O$_3$-5 mol% SiO$_2$-7 mol% MgO-1 mol% TiO$_2$-x mol% Ta$_2$O$_5$(x = 0.00, 0.05, 0.10, 0.15, 0.20, 0.30), ZnO(Nantong Jingqi Zinc Industry Co., Ltd), Al$_2$O$_3$(XiLong Scientific), MgO(XiLong Scientific), SiO$_2$(XiLong Scientific), and TiO$_2$(Sinopharm chemical Reagent Co., Ltd) are analytical reagent(AR), which belong to the second grade. The purity of Ta$_2$O$_5$(Aladdin industrial corporation) is 99.5%.

2.2. Preparation of samples

(75-x) mol% ZnO-12 mol% Al$_2$O$_3$-5 mol% SiO$_2$-7 mol% MgO-1 mol% TiO$_2$-x mol% Ta$_2$O$_5$(x = 0.00, 0.05, 0.10, 0.15, 0.20, 0.30) linear resistance ceramics were synthesized by a conventional ceramics method. In the first step, Ta$_2$O$_5$ and ZnO were ball-milled with ZrO$_2$ balls in a planetary ball mill(CX-QM2L) at 180 r/min for 6 h, in which the ratio of ball to mixed powder and water is 2:1:1.2. Then put the ball-milled slurry in Electric Blast Drying Oven(XMTD-8222) to dry and calcine at 800°C for 3 h. In the second step, MgO, Al$_2$O$_3$, TiO$_2$ and SiO$_2$ were ball-milled in the same way, and the slurry was dried and calcined at 1150°C for 3 h to make it uniformly dispersed. In the second step, the Ta$_2$O$_5$–ZnO mixture obtained in the first step and the mixture obtained in the second step are ball-milled, and the slurry was dried. The dried slurry is pelletized with a polyvinyl alcohol binder, and then compressed into a circular sheet (Φ15 mm × 6 mm). In the fourth step, the binder was first excluded from the round pieces and then sintered in the high temperature furnace of silicon molybdenum rod(KSL-1700X-A2) at 1330°C for 3 h in an air atmosphere to get ZnO linear resistor ceramic samples.
2.3. Performance testing and characterization

The microstructure of the sample was examined by backscattered electron (BSE, JSE-6510, Japan) imaging and energy dispersive spectroscopy (EDS). Bulk density and porosity are measured by the Archimedes method. The average size of the crystal grains is measured by Nano-Measurer software. The phase composition was examined by x-ray diffraction (XRD, Rigaku, Japan) using Cu K\textsubscript{α} radiation.

To measure the I–V characteristics, both sides of the sintered sample were covered uniformly with aluminum paste and fired at 650 °C for 40 min. The nonlinear coefficient \( \alpha = \frac{\lg(I_1/I_2)}{\lg(V_1/V_2)} \) is measured by the DC voltage-stabilized source, where the voltage corresponds to the current, \( V_1 \) corresponds to \( I_1 \), and \( V_2 \) corresponds to \( I_2 \).
corresponds to $I_2$. Respectively, the AC impedance spectrum is carried out at a frequency of 0.1 Hz to $10^6$ Hz by using electrochemical stations (CHI760E). The resistance temperature coefficient $\alpha_T$ was obtained from the relation $\alpha_T = (R - R_0)/(T - T_0) R_0$, where $T_0 = 303.15$ K and $T = 433.15$ K. The equation for the standard deviation is $\delta = \left( \sum_{i=1}^{n} (x_i - \bar{x})^2 / (n - 1) \right)^{1/2}$, where $x_i$ is the resistance of any slice in a set of resistive slices, $\bar{x}$ is the average resistivity of the set, and $n$ is the overall number of slices in the set.

3. Results and discussion

Figure 1 depicts the XRD patterns of Ta$_2$O$_5$ doped ZnO linear resistance ceramics sintered at 1330 °C. The results showed that ZnO, ZnAl$_2$O$_4$ and a small amount of Zn$_2$SiO$_4$ phases were present in all samples and no any other addition peak was observed, which indicated that doping Ta$_2$O$_5$ does not change the phase composition. Figure 2 shows the magnified XRD plots of all samples in the $2\theta$ range of 32°–40°. There are two points of interest in the figure. Firstly, the Zn$_2$SiO$_4$ diffraction peak gradually enhances with the increase of Ta$_2$O$_5$. Zn$_2$SiO$_4$ (trigonal system) could inhibit ZnO grain growth by the particle blocking effect at the grain boundaries. Zn$_2$SiO$_4$ also could facilitate the precipitation of excessive conductive impurities at the grain boundaries, generating more carriers, which reduced the barrier height of grain boundary. Secondly, the diffraction peak of
Table 1. The microstructure parameters and electric properties of ZnO linear resistance ceramics with different content of Ta$_2$O$_5$.

| Ta$_2$O$_5$ (mol%) | ZnO average grain size (μm) | ZnAl$_2$O$_4$ average grain size (μm) | Grain resistivity (Ω·cm) | Grain boundary resistivity (Ω·cm) | $\alpha_T \times 10^{-3}$ |
|-------------------|----------------------------|-------------------------------------|--------------------------|----------------------------------|------------------|
| 0.00              | 3.85                       | 1.68                                | 1408.12                  | 4824.73                          | −5.6             |
| 0.05              | 3.55                       | 1.50                                | 1325.11                  | 2962.14                          | −5.3             |
| 0.10              | 3.46                       | 1.33                                | 212.45                   | 42.99                            | −4.9             |
| 0.15              | 2.65                       | 1.14                                | 178.88                   | 26.27                            | −4.8             |
| 0.20              | 2.36                       | 1.14                                | 44.72                    | 6.15                             | −3.48            |
| 0.30              | 2.43                       | 1.05                                | 224.52                   | 31.70                            | −5.3             |
ZnO shifts to a higher angle with the increase of Ta2O5, which indicates that Ta5+ gradually dissolves into the ZnO lattice.

Figure 3 depicts the EDS spectrum of 0.20 mol% Ta2O5-doped ZnO linear resistance ceramics. In order to distinguish the different phases and their distribution, BSE micrographs of this sample were used. The ZnO phase is represented by the white grains marked by 01, the ZnAl2O4 phase is represented by the black colored grains marked by 02, and the Zn2SiO4 phase is represented by the gray grains marked by 03. The Zn2SiO4 phase is mainly mixed in the ZnAl2O4 phase. The element Ta is present in ZnO phase, which indicated that Ta5+ gradually dissolves into the ZnO lattice, and which is consistent with the XRD test results.

Figure 4 depicts the BSE images of ZnO linear resistance ceramics with different content of Ta2O5. It can be seen from the figure that with the increase of Ta2O5, Zn2SiO4 becomes abundant and evenly distributed between ZnO and ZnAl2O4, which inhibits the growth of ZnO grains. The ZnO gradually changes from the large particles in picture a to uniform elongated ‘rods’, and the black holes are gradually reduced, which is beneficial to the homogenization of the microstructure. To further investigate the effect of Ta2O5 doping on the grain size, the Nano-Measurer software was used to measure the average grain size of the primary crystalline phase ZnO and the secondary phase ZnAl2O4. As shown in table 1, the average grain sizes of ZnO and ZnAl2O4 all were reduced with the increase of Ta2O5. However, excessive addition of Ta2O5 leads to agglomeration, which results in abnormal growth of ZnO grains. It shows that there is an optimal value of 0.2 mol% for the doping of Ta2O5.

Figure 5 depicts apparent porosity and bulk density of ZnO linear resistance ceramics with different content of Ta2O5. It can be seen from the figure that with the increase of Ta2O5, Zn2SiO4 becomes abundant and evenly distributed between ZnO and ZnAl2O4, which inhibits the growth of ZnO grains. The ZnO gradually changes from the large particles in picture a to uniform elongated ‘rods’, and the black holes are gradually reduced, which is beneficial to the homogenization of the microstructure. To further investigate the effect of Ta2O5 doping on the grain size, the Nano-Measurer software was used to measure the average grain size of the primary crystalline phase ZnO and the secondary phase ZnAl2O4. As shown in table 1, the average grain sizes of ZnO and ZnAl2O4 all were reduced with the increase of Ta2O5. However, excessive addition of Ta2O5 leads to agglomeration, which results in abnormal growth of ZnO grains. It shows that there is an optimal value of 0.2 mol% for the doping of Ta2O5.

Figure 6 depicts the AC impedance complex plane of the sample by separating the real and imaginary parts of the complex impedance ($Z'$-$Z''$). And the equivalent circuit fitting with Z-view software, as shown in figure 6. The impedance spectra of all samples are arcs, which can be described by the following formula [17]:

Figure 7. The impedance-frequency (0–10 wHz, 10 wHz–100 wHz) characteristics of ZnO linear resistance ceramics doped with 0–0.05 mol% and 0.10–0.30 mol% Ta2O5.
Where $\omega$ refers to the circular frequency, $C_{gb}$ refers to the grain boundary capacitance, $R_{gb}$ and $R_g$ are the resistance of the grain boundary and the grain. The impedance arc fitted by the Z-view software is used for analysis. The grain and grain boundary resistivities ($\rho_g, \rho_{gb}$) shown in table 1 can be simply converted from $R_g$ and $R_{gb}$. The grain boundary resistivity and grain resistivity both decrease and then increase with the doping of $\text{Ta}_2\text{O}_5$, showing a ‘U’ shape trend. In terms of electrical properties of ZnO linear resistor ceramics, the main defects considered are donor-type defects [21]. Ta, which is a VB group element, is suitable for n-type doping of ZnO [22]. The ionic radius of $\text{Ta}^{5+}$ is slightly smaller than that of $\text{Zn}^{2+}$, and the valence state difference between $\text{Ta}^{5+}$ and $\text{Zn}^{2+}$ is 3. Therefore, $\text{Ta}^{5+}$ can easily replace $\text{Zn}^{2+}$, and one dopant atom can provide 3 free electrons.

The equation [18] for the possible defects arising in this period is as follows:

$$\text{Ta}_2\text{O}_5 \xrightarrow{ZnO} 2\text{Ta}^{5+}_{\text{Zn}} + 4\text{O}^{2-} + \frac{1}{2}\text{O}_2(g) + 6e^-$$

Figure 8. The resistivity-temperature (R–T) properties of ZnO linear resistance ceramics doped with 0–0.05 mol% and 0.10–0.30 mol% $\text{Ta}_2\text{O}_5$.
In this equation, Ta$^{5+}$ replaces Zn$^{2+}$ in the zinc oxide lattice as a donor, introducing conductive electrons, which leads to an increase in conductivity. At this time, the resistivity of the sample showed a downward trend. But when the donor concentration exceeds a certain amount, the electronic compensation becomes vacancy compensation, and the carrier concentration decreases with the increase of the Ta$_2$O$_5$ value, which leads to a decrease in conductivity. At this time, the resistivity of the sample shows a rising trend. It is worth mentioning that when the doping amount of Ta$_2$O$_5$ is more than 0.05 mol%, the dominant position changes from grain boundary resistivity to grain resistivity. The significant decrease in the resistivity of the grain boundary makes the difference between the resistance of the grain and the grain boundary smaller, which is beneficial to suppress the interface polarization and promote the sample to be suitable for a high-frequency electric field \[23\].

Figure 7 depicts the impedance-frequency (0–10 WHz, 10 WHz–100 WHz) characteristics of ZnO-based linear resistance ceramics doped with 0–0.05 mol% and 0.10–0.30 mol% Ta$_2$O$_5$. Surprisingly, the resistivity of the samples with 0.00–0.05 mol% Ta$_2$O$_5$ are significantly more strongly affected by the electric field frequency than that of the samples with 0.10–0.30 mol% Ta$_2$O$_5$. Especially under the frequency of 10 WHz–100 WHz, the sample doped with Ta$_2$O$_5$ is more stable. This is mainly because the addition of Ta$_2$O$_5$ significantly reduces the grain boundary resistivity of the sample. The resistivity of the grain boundary is more susceptible to the influence of the electric field, so the decrease of the resistivity of the grain boundary is beneficial to the stability of the sample resistivity. This also shows that Ta$_2$O$_5$ doped ZnO-based linear resistance ceramics are excellent candidates for high-frequency electric field applications.

Figure 8 depicts the resistivity-temperature (R–T) properties of ZnO-based linear resistance ceramics with different content of Ta$_2$O$_5$, and the resistance temperature coefficients ($\alpha_T$) could be calculated as the table 1 listed. As shown in the table, all the values are negative. Because ZnO-based linear resistance ceramics follow the principle of ‘thermal excitation’, which makes them exhibit negative temperature coefficient (NTC) characteristics \[24\]. $\alpha_T$ develops in a positive direction as the mole percentage of Ta$_2$O$_5$ doping increases from 0.00 mol% to 0.20 mol%. Doping Ta$_2$O$_5$ promotes the increase of Zn$_2$SiO$_4$, and the thermal expansion coefficient of Zn$_2$SiO$_4$ (7.0 $\times$ 10$^{-6}$K$^{-1}$) \[25\] is higher than that of ZnO (5.0 $\times$ 10$^{-6}$K$^{-1}$). Therefore, the Zn$_2$SiO$_4$ grains may thermally induce disconnection of ZnO grains, thereby increasing $\alpha_T$, which is consistent with the results of TiO$_2$-doped ZnO ceramics \[13\]. But when the mole percentage of Ta$_2$O$_5$ exceeds 0.20mol%, $\alpha_T$ develops in a negative direction. Because the thermal expansion coefficient of Ta$_2$O$_5$ \[26\] is lower than that of ZnO, excessive Ta$_2$O$_5$ will cause $\alpha_T$ to develop in a negative direction.

Figure 9 depicts the nonlinear coefficient and grain boundary barrier height of Ta$_2$O$_5$ doped ZnO-based linear resistance ceramics. The height of the grain boundary barrier is calculated by Arrehenius fitting and thermal expansion theory. Arrehenius plots of numerically fitted ZnO compound conductive ceramics dc resistivity is shown in figure 10. It can be seen that the changing trends for the nonlinear coefficient and grain boundary barrier height are similar, which decrease first and then increase with the increase of Ta$_2$O$_5$. Previous studies have shown that the nonlinear coefficient ($\alpha$) is positively associated with grain boundary barrier height ($\varphi_0$). The relevant expression for the grain boundary barriers height is as follows \[7\]:

\[ \varphi_0 = \frac{\alpha}{\tau} \]
where \( N_d \) and \( N_a \) represent the donor concentration and the acceptor concentration. As mentioned above, \( \text{Ta}^{5+} \) can replace \( \text{Zn}^{2+} \) for donor doping. When the content of \( \text{Ta}_2\text{O}_5 \) is not more than 0.2 mol\%, the donor concentration is in the dominant position. Therefore, the donor concentration also gradually increases with the increase of \( \text{Ta}_2\text{O}_5 \), which reduces \( j_0 \) and \( \alpha \). However, excessive doping of \( \text{Ta}_2\text{O}_5 \) will cause the excess \( \text{Ta}^{5+} \) to segregate to the grain boundary, which will increase \( j_0 \). Of course, the refinement of crystal grains, the decrease of porosity, and the improvement of resistance stability also affect the internal electric field, and all of those optimize the I-V characteristics.

Figure 11 depicts the standard deviation of resistivity and nonlinear coefficient of \( \text{Ta}_2\text{O}_5 \) doped ZnO-based linear resistance ceramics. As shown in the figure, with the increase of \( \text{Ta}_2\text{O}_5 \) content, the standard deviation of resistivity and nonlinear coefficient shows a trend of decreasing first and then increasing. This shows that the proper doping of \( \text{Ta}_2\text{O}_5 \) provides great help for the stability of resistivity and nonlinear coefficient. The main reason may be the significant reduction of the grain boundary resistivity and the optimization of the microstructure.

\[
\varphi_0 = \frac{e^2N_a^2}{2N_d\varepsilon_0 \varepsilon}\tag{5}
\]
4. Conclusions

ZnO-based linear resistance ceramics with different content of Ta$_2$O$_5$ were prepared by a conventional method. The main results and conclusions obtained in this study are summarized as follows.

(1) Ta$_2$O$_5$ doping optimized the microstructure of the samples, reducing the particle size of ZnO (from 3.85 $\mu$m to 2.36 $\mu$m) and the ZnAl$_2$O$_4$ (from 1.68 $\mu$m to 1.05 $\mu$m) and reducing the porosity. It is very beneficial to the application of ZnO linear resistance ceramics in high voltage and high current transmission grids.

(2) Ta$^{5+}$, which has a slightly smaller ionic radius and a valence difference of 3 than Zn$^{2+}$, can easily enter the ZnO lattice to replace Zn$^{2+}$. A small amount of doping can achieve higher carrier concentration, which lowers the grain boundary barrier height and optimizes the I–V characteristics.

(3) The doping of Ta$_2$O$_5$ improves the stability of the resistance and nonlinear coefficient of the sample. The grain boundary resistivity is significantly reduced, making the electrical properties of the sample more stable in high-frequency electric fields. ZnO linear resistor ceramics have the potential to be an excellent candidate material for high-frequency electric fields in the future.

(4) The sample doped with 0.2 mol% Ta$_2$O$_5$ has the best electrical properties. The resistivity, grain boundary barrier height, nonlinear coefficient and temperature resistivity are 56.2 $\Omega$·cm, 0.054 eV, 1.04 and $-3.48 \times 10^{-3}$ °C$^{-1}$, respectively.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
Declaration of interest statement

No conflict of interest exits in the submission of this manuscript, and manuscript is approved by all authors for publication. I would like to declare on behalf of my co-authors that the work described is original research that has not been published previously.

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