Research Article

High-Temperature Conductive Stability of ITO/Pt Bilayer-Film Electrode for Applications in High-Temperature SAW Devices

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Surface-acoustic-wave (SAW) devices have been widely investigated over many years as demand has grown for information sensing above 1000°C. At such high temperatures, metal electrode creep occurs, thereby causing the transformation of an electrode from a continuous uniform film to a discontinuous film with hollows or isolated grains. In this study, 100 nm thick ITO conductive oxide film is deposited on 100 nm thick Pt film to form an ITO/Pt bilayer-film composite electrode for high-temperature SAW devices. According to this way, the conductive stability of the Pt film electrode at high temperature can be greatly improved. Compared with the initial value, the electrical conductivity of single-layer 100 nm thick Pt film decreases to 65.4% with the area fraction of Pt at 64.1%. Meanwhile, the electrical conductivity of ITO/Pt bilayer-film decreases to only 95% with the area fraction of Pt at 63.1%. Then, the enhancement of high-temperature conductive stability can be attributed to the added ITO conduction channels. These findings demonstrate a potential route to design multilayer electrodes that can operate above 1000°C with promising applications in SAW devices needing to operate at high temperatures.

1. Introduction

Great efforts have been made to develop high-temperature surface-acoustic-wave (SAW) devices for many promising applications [1–7]. The propagation of acoustic waves is realized by the vibration of a piezoelectric substrate; thus, the performance of SAW devices is determined to a great extent by the mass loading effect of interdigital transducer electrodes (ITDs)—that is, too thick an electrode will decay the propagation of SAW and influence the performance of the device [8, 9]. When environmental temperatures are lower than 700°C, conventional monolayer metal films such as those of Au and Pt can be exploited as ITDs for high-temperature SAW devices without considering the deterioration of electrodes [10–13]. For SAW devices operating at high temperatures (above 700°C and especially above 1000°C), thin metal electrodes exhibit poor conductive stability because creep-induced discontinuous holes in the metal films significantly increase their resistance [14, 15]. In contrast, La3Ga5SiO14 (LGS), as a mature piezoelectric substrate, can maintain piezoelectric behavior up to its melting point at 1470°C [16, 17]. Therefore, finding electrodes that can work continuously and steadily is the most crucial factor to ensure the normal operation of LGS-based SAW devices at extremely high temperatures over long periods of time.

In order to enhance the conductive stability of film electrodes for SAW devices at high temperatures, much research has been done over many decades. Except for their important roles in nanocatalyst, [18, 19] the multilayer metal films...
and alloys present their effect on high-temperature film electrodes. For example, alloys of different compositions have been found to have more stable conductivity at high temperatures [20–24]. In addition, Ti or Ta adhesion layers, [25–30] capping layers, [31] buffer layers, [15] and diffusion barriers [32–34] have also been applied to promote the high-temperature stability of electrode resistivity. Thus, as is known, creep occurs in metal films and forms hollows at sufficiently high temperatures, leading to an increase in resistance [14]. Up to now, almost all the previous investigations about high-temperature film electrodes are concentrated on the effect of the adhesion layer, capping layer, buffer layer, or diffusion barrier, which have proved that materials of varying composition can present better high-temperature performance [35–38]. But in these ways, the creep phenomenon just can be delayed in some extent and the Pt holes appear during long enough high-temperature process, leading to the failure of electrode finally. In this paper, a novel ITO/Pt bilayer-film composite electrode is proposed to improve the high-temperature performance stability of a SAW by introducing ITO conduction channel. By this means, the creep-induced (at 1000°C) Pt holes are filled by an upper ITO film, forming ITO-surrounded Pt grains. Under this condition, the ITO works as a conductive channel and connects the isolated Pt, making the resistance more stable. By exploiting this ITO/Pt electrode, we successfully fabricate SAW devices that can operate stably at 1000°C. Our results may provide a novel possibility for designing multilayer electrodes operating in various electronic devices needing to operate above 1000°C.

2. Materials and Experiments

The ITO/Pt bilayer electrode film was deposited on LGS substrates by using pulsed laser deposition (PLD). Before growth, the LGS substrates were ultrasonically cleaned with deionized water, acetone, and ethanol for 5 min each. Then, the LGS substrates were loaded into a deposition chamber, and the deposition parameters were as follows: the frequency of the pulsed laser was fixed at 3 Hz, the energy density was set at approximately 4 J/cm², the depositing temperature was kept at 500°C, 100 nm of Pt film was deposited in high vacuum (4 × 10⁻⁵ Pa), and 100 nm of ITO film was deposited under a flowing oxygen pressure of 20 Pa. A three-dimensional schematic of the ITO/Pt/LGS structure is shown in Figure 1(a). The ITO/Pt films were dry-etched to form rectangular electrodes for measuring resistivity, with the length and width fixed at 9 mm and 4 mm, respectively, as shown in Figure 1(b). Figure 1(c) shows a schematic of an ITD, which was also fabricated by dry etching and was characterized by an interdigital width and spacing of 10 μm.

Resistance measurements of the prepared film electrodes were carried out at high temperatures within a box furnace.
(KSL-1200X-J) using a resistance meter. A four-point measurement was performed using four Pt0.9Ir0.1 wires to acquire real-time resistance data. During the entire heating process, the heating rate was maintained at 4°C per minute. The microstructures of the electrodes were determined by transmission electron microscopy (TEM) and scanning electron microscopy (SEM). Area fractions of Pt were obtained from the SEM images using ImageJ software.

3. Results and Discussions

Figure 2 shows the resistance change of the as-deposited ITO, Pt, and ITO/Pt films as the function of temperature from room temperature to 1000°C. It can be found that the resistance of ITO film decreases slowly from room temperature to 700°C and decreases obviously from 700°C to 1000°C. This phenomenon is mainly attributed to the ITO grain growth under high temperature, which results in the lower density of grain boundaries and weaker boundary scattering effect for carriers [39–41]. At the same time, we can see that the resistance of Pt film increases slowly from about 2.5Ω (at room temperature) to 7Ω (at 1000°C). Since the conductivity of Pt is much higher than that of the ITO film, the resistance of ITO/Pt film is mainly dominated by the resistance of Pt. That is also the reason why the resistance curves of the Pt and ITO/Pt films almost coincide.

To accurately investigate the influence of the heating process on the surface topography of the films, the heat preservation time (at 1000°C) was set to as short as one second. That is to say, the heat preservation time could be totally ignored. Under this condition, Figure 3(a) shows the surface topography of the Pt film after annealing at 1000°C, where obvious outlines of Pt grains appeared, indicating that the creep of the Pt film occurred after annealing. In addition, the surface topography of the ITO and ITO/Pt films after annealing are shown in Figures 3(b) and 3(c), where the creep of the ITO film can barely be seen. These results suggest that creep did not occur in ITO during the heating process.

Figure 4 plots the resistance of the ITO, Pt, and ITO/Pt film electrodes as the function of dwell time at 1000°C. The resistance of the ITO film remained stable for approximately 150 min. Then, the resistance began to increase quickly at longer dwell times. This obvious increase in the resistance of the ITO film is attributed to the decomposition of SnO₂, which
decreased the number of oxygen vacancies and the carrier concentration at high temperatures [38]. Further, the resistance of the Pt film increased throughout the entire annealing time and became infinite after annealing at 1000°C for 90 min. Meanwhile, it is worth noting that the resistance of the ITO/Pt film remained stable until 120 min and then increased slowly from 120 min to 180 min. When the dwell time was greater than 180 min, the resistance of the ITO/Pt film increased sharply, which is attributed to an increase in the resistance of ITO.

To determine the effect of additional ITO on enhancing the conductive stability of the electrodes at high temperatures, the surface topographies of Pt and ITO/Pt samples annealed at 1000°C for different times were characterized by SEM, as shown in Figures 5 and 6, respectively. According to the SEM images, the area fraction of Pt can be extracted, which represents the intensity of the Pt creep.

In this way, the area fraction of Pt after annealing for 5 min was determined to be 0.926 according to Figure 5(a) by exploiting the ImageJ software. Similarly, the area fraction...
The area fraction of Pt decreased by increasing the dwell time, as longer dwell times resulted in more drastic Pt creep.

The surface topographies of ITO/Pt samples annealed at 1000°C from 0.5 to 4 h are presented in Figure 6. Figure 6(a) shows a sample surface topography with obvious Pt hollows, indicating that the Pt agglomerated and Pt hollows formed before annealing at 1000°C for 0.5 h. However, compared to the surface topography of the Pt film that was also
annealed at 1000°C for 0.5 hours, the area fraction of Pt for the ITO/Pt sample was much larger. The reason for this phenomenon is that the ITO film played a role in inhibiting the agglomeration of Pt as a capping layer [37]. As the dwell time increased, the area of the Pt discontinuous voids increased further. Finally, insular Pt grains formed, which were embedded in the ITO film. Although Pt voids obviously formed before annealing at 1000°C for approximately 2 h, it was interesting to find that the resistance of the ITO/Pt composite film electrode maintained stable conductivity at this stage, as shown in Figure 3. This phenomenon was thought to be due to ITO providing extra conductive channels between the Pt grains, which decreased the effective resistance of the sample.

To further confirm that the creep of the Pt layer had occurred during the heat treatment process, cross-sectional TEM morphologies of the ITO/Pt bilayer-film before and after annealing (1000°C, 4 h) were obtained as shown in Figure 7. Specifically, Figure 7(a) shows the cross-sectional TEM morphology of the ITO/Pt film before annealing, and Figure 7(b) shows the morphology after annealing. From Figure 7(a), we can clearly determine that the thicknesses of the interface and the Pt and ITO layers were 100 nm. However, from Figure 7(b), we find that the ITO/Pt interface disappeared after annealing at 1000°C for 4 h. This phenomenon can also be attributed to the high-temperature creep of Pt, which led to the melting of Pt into the ITO film. On this basis, it is reasonable that the ITO/Pt electrode could keep its...
conductivity at 1000°C. The creep-induced Pt grains were embedded into the ITO films, and the ITO provided conductive channels between the isolated Pt grains. Therefore, the ITO/Pt films maintained their conductivity at 1000°C even after approximately 3 h. The increase in the resistance of the ITO/Pt films with increasing temperature, as shown in Figure 1, can be interpreted as the resistivity of Pt increasing due to the enhanced scattering of free electrons at high temperatures.

As mentioned above, the area fraction of Pt was chosen as a quantifiable physical characteristic to present its degree of agglomeration, which is proposed in three-dimensional effective media theory by referring to the volume fraction [39]. In this case, the area fabrications of Pt are calculated according to the SEM results in Figure 5 (Pt film) and Figure 6 (ITO/Pt film). Further, the conductivities of all these samples were measured before and after annealing. By combining the abovementioned SEM and conductivity results, the variation of $\sigma_{\text{eff}}/\sigma_1$ with the area fraction of Pt is plotted in Figure 8(a), where $\sigma_1$ represents the as-deposited conductivity measured at room temperature, and $\sigma_{\text{eff}}$ represents the measured conductivity at room temperature after annealing. The Pt area fractions of single-layer Pt film can be determined as 88.4%, 76.7%, 64.1%, 58.0%, 54.1%, 50.2%, 46.5%, 38.4%, and 33.0% from SEM results in Figure 5 corresponding to the measured $\sigma_{\text{eff}}/\sigma_1$ of 0.926, 0.811, 0.654, 0.532, 0.436, 0.389, 0.150, 0, and 0. As a contrast, the Pt area fractions of ITO/Pt bilayer-film can be determined as 85.0%, 74.7%, 63.1%, 58.0%, 53.1%, 43.5%, 37.5%, 30.0%, and 25.5% from SEM results in Figure 6 corresponding to the measured $\sigma_{\text{eff}}/\sigma_1$ of 0.990, 0.981, 0.951, 0.933, 0.840, 0.294, 0.205, 0.105, and 0.040. In this figure, it can be seen that the $\sigma_{\text{eff}}/\sigma_1$ of the Pt film decreased sharply as the Pt area fraction decreased. In contrast, the $\sigma_{\text{eff}}/\sigma_1$ of the ITO/Pt film almost remained stable before the Pt area fraction decreased to below 0.5. It is worth noting that the resistance of ITO began to increase sharply when the area fraction of Pt became lower than 0.45 as shown in Figures 3 and 6(f). Thus, the sharp decrease in $\sigma_{\text{eff}}/\sigma_1$ for the ITO/Pt film is considered to be caused by the increased resistance of the ITO film. The destroyed conductivity of ITO shows that this proposed (conductive oxides) Pt granular film system still requires further optimization in the future.

Figure 8(b) shows the fabricated SAW device, and Figure 8(c) shows its performance after annealing at 1000°C for different times. The as-fabricated SAW device exhibited a resonant frequency of 68.2 MHz, which was controlled by the width and spacing of the interdigital transducer electrode. Note that the signal intensity of S11 did not vary even after annealing at 1000°C for at least 9 times.

4. Conclusions

In this paper, an ITO/Pt bilayer-film electrode was prepared for high-temperature SAW devices by PLD. According to the SEM and high-temperature resistance measurements, we found that the coated ITO layer indeed improved the conductive stability at 1000°C. This improvement is thought to be due to the ability of the ITO conductive channel to fill the Pt hollows and connect them, forming a conductive channel for Pt. These results show the great potential of ITO/Pt bilayer-film electrodes in preparing composite film electrodes that can work stably at high temperatures. In conclusion, the introduction of ITO conduction channel improves the high-temperature conductive stability of the Pt film electrode, which paves the path for the development of SAW sensors applied at high temperature above 1000°C.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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