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Research Article

Electrical insulation enhancement of the Al₂O₃ composite films at high temperature by oxygen vacancy suppression

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Abstract: The electrical insulation film is critical for the reliable operation of Ultra-high temperature thin-film sensors (*UTTSs*) at elevated temperatures. However, the resistance of the insulation film easily decline at high temperatures due to the formation of the oxygen vacancy and through-type defects, which will affect the long-term stability and reliability of the *UTTSs*. Here, we report a novel gas-liquid phase composite strategy to fabricate Al_2O_3 composite insulation films. An Al_2O_3 bottom film was first deposited by liquid-phase electrohydrodynamic jet printing, followed by the deposition of a dense Al_2O_3 film by magnetron sputtering. This design not only effectively repairs through-type defects in the liquid-phase film and improves overall densification, but also suppresses the formation of oxygen vacancy in films. The gas-phase Al_2O_3 (G- Al_2O_3)/liquid-phase Al_2O_3 (L- Al_2O_3) composite film exhibits a smooth surface with substantially reduced micro-cracks and porosity. X-ray photoelectron spectroscopy reveals a significantly lower relative oxygen-vacancy level in the composite films compared with the pure L- Al_2O_3 film. Consequently, the G- Al_2O_3 /L- Al_2O_3 film achieves an insulation resistance of 44 k Ω at 1400 °C, an order of magnitude higher than that of the pure L- Al_2O_3 film. In addition, three thermal cycles between 800 and 1200 °C demonstrate excellent repeatability of the insulation resistance within this temperature range. Practically, the Al_2O_3 composite films were applied to the thin-film strain gauges (*TFSGs*), and the *TFSGs* exhibited excellent performance at high temperatures.

Keywords: Al_2O_3 film; gas-liquid phase composite; high-temperature electrical insulation; electrohydrodynamic jet printing; magnetron sputtering

1 Introduction

Ultra-high temperature thin-film sensors (*UTTSs*) are increasingly used for the in-situ monitoring of hot-section components in aerospace, energy, and industrial equipment. Owing to their high sensitivity, rapid thermal response, and minimal influence on the monitored structure, *UTTSs* enable real-time strain monitoring under harsh thermal environments, which is important for ensuring the safe operation of equipment[1-6]. Recent advances in thin-film temperature sensing have shown that the coordinated optimization of sensing materials, electrode–substrate configurations, and structural designs can broaden the measurable temperature range, improve signal stability, and enhance measurement reliability, with the upper operating temperature reaching 1200 °C [7-8]. A typical *UTTS* consists of three films: an insulative film, a sensitive film, and a protective film. Herein, the insulation film can provide reliable electrical isolation to prevent electrical conduction between the sensing film and the conductive substrates at high temperatures, thereby ensuring measurement accuracy and durability of the *UTTSs* [9-10]. However, the resistance of the insulation film easily decline at high temperatures, which will affect the long-term stability and reliability of the *UTTSs* [11]. Therefore, developing insulation film with thermal stability is of great value for advancing high-temperature sensing technologies.

To date, ceramic materials such as Al_2O_3 and YSZ have been widely used as high-temperature insulation films due to their high dielectric strength, excellent thermal stability, and chemical inertness [12–14]. Vapor-phase deposition techniques such as magnetron sputtering can produce dense Al_2O_3 films with low defect densities and good dielectric properties [15–18]. However, such vapor-deposited films are prone to thermomechanical stress at high temperatures, leading to cracking or decreased adhesion, which compromises the long-term stability of the sensor [19]. In contrast, liquid-phase fabrication techniques such as electrohydrodynamic jet printing can offer high process flexibility and deposition efficiency. Nevertheless, the films prepared by these methods are susceptible to the

formation of through-type defects such as microcracks and pores during the deposition process, which can evolve into conductive pathways under high-temperature thermal cycling, resulting in a marked degradation of insulation performance [20]. Therefore, it still remains a key challenge in the development of high-temperature insulation films on effectively balancing structural densification and defect controllability to prevent the generation and evolution of defects at high temperatures. .

To address this challenge, two main factors need to be focused including oxygen vacancy and through-type defects. Oxygen vacancies in insulation films act as deep donor states, releasing two localized electrons into the conduction band upon thermal excitation, thereby significantly increasing the carrier concentration[21]. On the other hand, the existing through-type defects in insulation films will provide the conductive pathway[22]. Thus, the insulation performance will be significantly enhanced after the two problems being addressed. Researchers have explored multilayer composite structures combining the advantages of different processes to control the through-type defects. For instance, Liu and his co-workers fabricated YSZ/ Al_2O_3 multilayer films by RF magnetron sputtering, achieving an insulation resistance of 200 k Ω at 800 °C [23]. Wang and his colleagues used dual-ion-beam sputtering to prepare $\text{HfO}_2/\text{Al}_2\text{O}_3$ multilayer heterostructures, increasing the insulation resistance by two orders of magnitude at 1100 °C [24]. Although these vapor-phase composite structures have improved insulation performance to some extent, they suffer from low fabrication efficiency and complex processing. In recent years, liquid-phase methods have gained attention due to their high deposition rates and process flexibility [25–26]. In our previous work, we prepared Al_2O_3 composite films by electrohydrodynamic jet printing, partially repairing defects by optimizing the ink rheology, and obtained insulation resistances of 3 M Ω and 38 k Ω at 800 °C and 1200 °C, respectively [27]. More recently, we developed an M- Al_2O_3 /S- Al_2O_3 composite film in which both layers were fabricated by electrohydrodynamic jet printing [28]. The M- Al_2O_3 bottom layer was prepared from an Al_2O_3 mixture, whereas a more fluid Al_2O_3 sol was used to form the S- Al_2O_3 top layer and repair defects in the underlying film. Although this entirely liquid-phase route improved the surface morphology and

insulation performance, its defect-control mechanism still relied mainly on the fluidity and spreading of the liquid precursor. However, due to the poor rheological properties of the Al_2O_3 hybrid ink, the defect repair capability was limited, leading to a rapid decline in insulation performance at 1400 °C due to the formation of the through-type defects, as shown in Fig. S2. Notably, few researchers have focused on the impact of oxygen vacancies on the high-temperature insulation performance of films, and the underlying mechanism remains unclear. Hence, a composite film fabrication strategy that can effectively repair through-type defects and oxygen vacancy to enhance high temperature insulation property is urgently needed.

In this study, we proposed a novel gas-liquid phase composite process to prepare high-performance Al_2O_3 insulation films. In this strategy, an Al_2O_3 bottom film was first deposited by liquid-phase electrohydrodynamic jet printing, followed by the deposition of a dense Al_2O_3 or YSZ top film by magnetron sputtering. This design improves the overall densification of the composite film by covering and reducing surface-connected pores and microcracks in the liquid-phase layer, thereby reducing the probability of forming through-type conductive pathways at elevated temperatures. Results show that the G- Al_2O_3 /L- Al_2O_3 composite film achieved an insulation resistance as high as 44 k Ω at 1400 °C, representing an order of magnitude improvement over the pure liquid-phase film. After three thermal cycles from 800 °C to 1200 °C, the composite film still exhibited excellent insulation stability. Furthermore, the surface roughness of the composite film was significantly reduced to 0.776 μm , effectively suppressing defect formation. O 1s XPS deconvolution analysis revealed a marked reduction in the relative oxygen-vacancy level of the G- Al_2O_3 /L- Al_2O_3 composite film, thereby decreasing the thermal carrier concentration and further enhancing high-temperature insulation performance. Overall, this work will provide an effective fabrication paradigm for developing the insulation films with both superior high-temperature performance and long-term stability, potentially overcoming the performance limitations of high-temperature thin-film sensors in extreme application environments.

2 Experimental details

2.1 Preparation of the insulation films

A 10×10 mm alumina (Al_2O_3) substrate was ultrasonically cleaned with acetone, anhydrous ethanol, and deionized water. An indium tin oxide (ITO) bottom electrode was first deposited on the substrate by magnetron sputtering (LAB1803060307, Kurt J. Lesker Company, USA) using an ITO target ($\text{In}_2\text{O}_3:\text{SnO}_2 = 90:10$ wt%) to enable subsequent high-temperature insulation resistance measurements. Fig. 1 (a) shows a schematic diagram of the electrohydrodynamic jet printing system, which consisted of a precision syringe with an inner diameter of 0.7 mm (TECDIA, Shanghai, China), a high-voltage power supply (DW-P303-1ACD1, Dongwen High Voltage, China), a syringe pump (LSP01-2A, Longer, China), a programmable X–Y motion platform (WNMC400, Beijing Winner Optical Instrument Group Co., Ltd., China), and a micro-image acquisition module (CSFU15BCE18-B, Toshiba Teli Corporation, Japan). As shown in Fig. 1(b), effective atomization of the Al_2O_3 ink was achieved using the electrohydrodynamic jet printing technique, which was a prerequisite for the uniform deposition of the L- Al_2O_3 film. The formation of the electrohydrodynamic jet was governed by the combined effects of the electric field force, ink viscosity, surface tension, and body force [29–32], and the force analysis of the Taylor cone region is shown in Fig. 1(c). Under the applied electrostatic field, the charged jet may undergo several modes, including burst, micro-drip, cone-jet, multi-jet, and ramified-jet modes. The key parameters controlling stable electrohydrodynamic jetting included the applied voltage, ink flow rate, and nozzle-to-substrate distance, and the corresponding operating domains of the different printing modes are shown in Fig. 1(d). The Al_2O_3 printing ink was prepared by mixing a laboratory-prepared Al_2O_3 sol with a commercial $\alpha\text{-Al}_2\text{O}_3$ nanoparticle suspension (average particle size: 100 nm). Detailed preparation procedures can be found in our previous works [27–28]. The electrohydrodynamic jet printing parameters were optimized to maintain stable cone-jet operation. During deposition, the ink flow rate, applied voltage, and nozzle-to-substrate distance were maintained at 6 $\mu\text{L}/\text{min}$, 7.8 kV, and 3 mm, respectively. The nozzle-to-substrate

distance was maintained using a closed-loop gap-control system. The X/Y moving platform was programmed to follow alternating deposition paths to ensure uniform film coverage. To prevent cracking and promote defect healing, each deposited film was sequentially baked on a hotplate at 150 °C for 15 minutes followed by 400 °C for 5 minutes. After every six layers, the film was sintered in a muffle furnace (TCXQ-1700, TongKe, China) at 600 °C for 1 hour. For the single-layer L-Al₂O₃ control film, the deposition process was continued until a thickness of approximately 10 μm was obtained. For the composite films, the L-Al₂O₃ underlayer was approximately 8 μm thick, followed by the deposition of an approximately 2 μm sputtered top layer, giving a total composite-film thickness of approximately 10 μm. The details were also can be found in our previous works[27-28].

To further eliminate through-type defects and enhance the compactness, a dense top film was deposited on the L-Al₂O₃ films by radio-frequency (RF) magnetron sputtering (LAB1803060307, Kurt J. Lesker Company, USA), as illustrated in Fig. 1(e). This method produced films with high density and uniform thickness, effectively filling the microcracks that may have formed in the liquid-phase-deposited Al₂O₃ film after sintering, thereby further improving the high-temperature insulation performance of the Al₂O₃ film. Herein, two different ceramic targets were used for this purpose: an Al₂O₃ target and a yttria-stabilized zirconia (YSZ, 92 wt% ZrO₂ + 8 wt% Y₂O₃) target. The sputtering process was carried out under the following conditions: a base pressure of 3×10⁻³ Pa, a working pressure of 1.3 Pa, an RF sputtering power of 300 W, a substrate temperature at room temperature, an Ar gas flow of 20 sccm, and a target-to-substrate distance of 7 cm. The thickness of the sputtered top film was controlled to be approximately 2 μm. The final composite films were designated as G-Al₂O₃/L-Al₂O₃ for the Al₂O₃ top film and G-YSZ/L-Al₂O₃ for the YSZ top film. The schematic diagram of the overall structure of the gas-liquid phase insulation film was shown in Fig. 1(f).

2.2 Characterization and test

The morphology and crystal structure of the composite films were characterized using scanning electron microscopy (SEM, FEI NOVA Nano SEM450, USA), a 3D optical surface profiler (ZYGO, NV50005022S, USA), and X-ray diffraction (XRD, Smart Lab 9, Japan). The chemical states of the elements were analyzed by X-ray photoelectron spectroscopy (XPS, K-Alpha+, USA) using a monochromated Al K α $h\nu = 1486.6$ eV X-ray source. The base pressure of the spectrometer was approximately 5×10^{-9} mbar, the energy resolution was 0.82 eV, and the X-ray spot diameter was 400 μm . A flood gun was used for surface charge neutralization during the measurements. The binding energy scale was calibrated using the C 1s line at 284.8 eV. All XPS peak contributions were fitted using mixed Gaussian–Lorentzian product functions following Shirley background subtraction. The high-temperature insulation resistance of the films was measured from 400 °C to 1400 °C using a two-wire method with an insulation resistance meter (HPS2683, Helpass, China) and a muffle furnace, and the details were followed by our previous works[27]. The heating and cooling rates were both set at 20 °C/min. For the insulation resistance, surface roughness, and XPS O 1s analysis, the corresponding statistical analyses are provided in the Supporting Information.

3 Results and discussion

3.1 Failure mechanisms of the insulation films

Al_2O_3 and YSZ are two typical insulative materials widely used for the *UTTSs*, and the inner defects are critically important for their insulation properties. A deep understanding of the crystal structures, oxygen vacancy formation mechanisms, and their effects on carrier concentration in these two materials is of great significance for improving their high-temperature insulation performance. Fig. 2(a) shows the lattice structure of an Al_2O_3 film, which typically exhibits a hexagonal close-packed (hcp) crystal structure of $\alpha\text{-Al}_2\text{O}_3$ (corundum phase). In this structure, aluminum (Al) atoms occupy octahedral interstitial sites with sixfold coordination, while oxygen (O) atoms form the hcp oxygen sublattice with fourfold coordination. Oxygen vacancies are easily formed during the Al_2O_3 films preparation process, as shown in Fig. 2(b). This will act as electron and hole traps and lead to the

decrease of their high temperature insulation performance [33]. Fig. 2(c) shows the lattice structure of a YSZ film, which typically adopts a face-centered cubic (fcc) fluorite structure. Zr^{4+} ions are located at the fcc lattice points, while O^{2-} ions occupy the tetrahedral interstitial sites. Similarly, the schematic diagram of the oxygen vacancies formed in the film deposition can be seen in Fig. 2(d). The influence mechanisms of oxygen vacancies in Al_2O_3 and YSZ films are shown in Figs. 2(e) and 2(f), respectively. Density functional theory (DFT) calculations have indicated that oxygen vacancies in Al_2O_3 act as deep donor states, releasing two localized electrons into the conduction band upon thermal excitation, thereby significantly increasing the carrier concentration [21]. Oxygen vacancies in YSZ are mainly introduced through aliovalent doping. When trivalent yttrium (Y^{3+}) substitutes for tetravalent zirconium (Zr^{4+}), each Y^{3+} ion replacing a Zr^{4+} ion generates an effective negative charge to maintain charge neutrality [34]. In addition, microstructural defects in the film, such as microcracks and pores (Fig. 2g), can synergistically accelerate the degradation of insulation performance [35].

3.2 Microstructure of the insulation films

The microstructure of the prepared films is important for their high-temperature insulation performance. To examine the defect-repairing effect of the gas-phase films on the L- Al_2O_3 films, the microstructures of different composite insulation films prepared by the gas-liquid phase method were characterized using SEM and a 3D optical surface profiler. Fig. 3(a) shows the cross-sectional SEM micrograph of the L- Al_2O_3 film. Figs. 3(b) and 3(c) present cross-sectional SEM images of different composite insulation films. All the films consisted of the insulation film, ITO electrode film, and the substrate from top to bottom. The different gas-phase insulation films with no obvious defects were bonded to the L- Al_2O_3 film, which was beneficial for suppressing the formation of through-type conductive pathways at high temperatures. Figs. 3(d–f) show surface SEM images of the different insulation films. The surface of the L- Al_2O_3 film still exhibited obvious pores and defects, which could provide conductive pathways for the insulation film in high-temperature environments above 1200 °C,

severely compromising its high-temperature insulation performance. Compared with the L-Al₂O₃ film, the surfaces of the composite films with YSZ and Al₂O₃ films were much denser, with no obvious pores, cracks, or other defects. This indicates that the dense gas-phase film was very effective in repairing defects in the L-Al₂O₃ film. Furthermore, the surface of the G-Al₂O₃/L-Al₂O₃ film (Fig. 3f) was smoother and denser than that of the G-YSZ/L-Al₂O₃ film (Fig. 3e). This suggests that the G-Al₂O₃ film can repair the defects in the L-Al₂O₃ film more effectively. To intuitively describe the defect-repairing effect of the gas-phase films on the L-Al₂O₃ film, the surface roughness of the different composite insulation films was measured using a 3D optical surface profiler, and the results are shown in Figs. 3(g–i). Compared with the L-Al₂O₃ film, the surface roughness of the composite insulation films after gas-phase deposition decreased to varying degrees. This was because the dense gas-phase films will effectively fill the pores, microcracks, and other defects on the surface of the L-Al₂O₃ film, making the surface of the gas-liquid-phase composite film smoother and denser. In addition, the surface roughness of the G-Al₂O₃/L-Al₂O₃ composite film (0.776 μm) was lower than that of the G-YSZ/L-Al₂O₃ composite film (0.957 μm). This was attributed to the better interfacial compatibility between the G-Al₂O₃ and the L-Al₂O₃, which had the same crystal orientation. This can facilitate the formation of an α -phase solid solution film after high-temperature sintering, resulting in a better defect-repairing effect and lower surface roughness. This was also consistent with the observations in Figs. 3(e) and 3(f). Overall, compared with the L-Al₂O₃ film, the density of the gas-liquid-phase composite insulation films was effectively improved, and defects were further repaired. In consequence, this will effectively avoid the formation of through-type defects and greatly reduce the probability of forming conductive pathways at elevated temperatures. Eventually, this is beneficial for the enhancement of the insulation performance of the films at higher temperatures.

The composition of the insulation film plays a key role in its high-temperature insulative performance [36–37]. Fig. 4(a) shows the XRD patterns of the different insulation films. Both the G-Al₂O₃/L-Al₂O₃ film and the L-Al₂O₃ film exhibited polycrystalline structures with the same diffraction peaks, namely (012), (104), (011), (113), (024), (116), (214), and (300), indicating that both films were α -Al₂O₃ [38]. In addition, the G-YSZ/L-Al₂O₃ film also exhibited a polycrystalline structure with diffraction peaks at (101), (104), and (202). Obviously, the (101) peak is the strongest, which suggests that the prepared YSZ film had a preferred orientation along this crystal plane [39–40]. By comparison with the standard PDF card (#37-1307), the G-YSZ film prepared by magnetron sputtering was identified as the Zr_{0.82}Y_{0.18}O_{1.91} phase [41]. In addition, Fig. 4(b) shows the XPS survey spectra, and the peaks for O, C, Zr, Y, and Al were identified [42]. Figs. 4(c–f) present the high-resolution XPS spectra of Al 2p, Zr 3d, and Y 3d. The Al 2p XPS spectra of both the G-Al₂O₃ and the L-Al₂O₃ showed a peak at 74.4 eV, confirming the presence of Al₂O₃ in both cases [43]. Due to spin-orbit splitting, the high-resolution Zr 3d and Y 3d spectra were deconvoluted into two peaks. The binding energies of the Zr 3d_{3/2} and Zr 3d_{5/2} peaks were 184.4 eV and 182.0 eV, respectively. While those of the Y 3d_{3/2} and Y 3d_{5/2} peaks were 158.9 eV and 156.8 eV, respectively, indicating the presence of ZrO₂ and Y₂O₃ [44–45].

3.3 Analysis of the oxygen vacancies and electronic structure

Oxygen vacancies in the L-Al₂O₃, G-Al₂O₃/L-Al₂O₃, and G-YSZ/L-Al₂O₃ films were analyzed by their O 1s XPS spectra, and the results were shown in Figs. 5(a–c). The O 1s spectra of all three films could be fitted into three characteristic peaks, designated as the O_I, O_{II}, and the O_{III} peaks. The assignment of the three peaks has clear physicochemical meaning. According to previous studies, the O_I peak is assigned to adsorbed oxygen species, the O_{II} peak corresponds to oxygen vacancies, while the O_{III} peak represents lattice oxygen [46–48]. The relative oxygen vacancy level in the L-Al₂O₃, G-

Al₂O₃/L-Al₂O₃, and G-YSZ/L-Al₂O₃ films was described by calculating the relative area ratio $R_{A(II)}$, defined as $A(II)/[A(I) + A(II) + A(III)]$. Similar O 1s XPS peak-area analyses have been used to evaluate the relative variation of oxygen-vacancy content in oxide thin films [49]. As shown in Fig. 5(d), the $R_{A(II)}$ values for the three films were 0.53, 0.25, and 0.34, respectively. This indicated that the L-Al₂O₃ film had the highest oxygen vacancy concentration. The result is consistent with defect chemistry theory: pure L-Al₂O₃ tends to form intrinsic oxygen vacancies during preparation to maintain charge balance [50], whereas in the G-Al₂O₃/L-Al₂O₃ film, the lower relative oxygen-vacancy level may be associated with the denser microstructure and the different defect states produced by the sputtered top layer. In G-YSZ/L-Al₂O₃, Y³⁺ substitution and the associated charge-compensation mechanism additionally affect the oxygen-vacancy population [51–53].

To further understand the effect of oxygen vacancies on electronic transport in Al₂O₃, spin-polarized first-principles calculations were performed based on the variation in oxygen vacancies revealed by O 1s XPS analysis. Two oxygen-deficient α -Al₂O₃[54] configurations containing one and six oxygen vacancies were constructed[55]. The corresponding vacancy concentrations in the oxygen sublattice were 1.39% and 8.33%, respectively. The high-vacancy supercell containing six oxygen vacancies was generated using the special quasirandom structure (SQS) method. This approach was used to reproduce the ideal random values of the selected short-range O-O pair-correlation functions as closely as possible. The two configurations enabled a relative comparison between low- and high-vacancy conditions. They were also used to examine the evolution of the electronic structure with increasing oxygen-vacancy concentration. All calculations employed the projector augmented-wave (PAW) method implemented in the VASP package[56] and the GGA-PBE exchange-correlation functional[57]. The DFT-D3 dispersion correction[58] and Monkhorst–Pack k-point sampling[59] were also applied. Detailed computational parameters are provided in the Supporting Information.

As shown in Fig. 6(a) and (b), the high-vacancy configuration exhibits six occupied vacancy-derived in-gap bands[60–62]. These bands are distributed from approximately -1.56 to -0.19 eV. The corresponding density of states (DOS) shows a broad distribution of defect states below the Fermi level. These vacancy-induced states provide more energetically accessible electronic states within the band gap. They may therefore facilitate the thermal excitation of charge carriers and defect-assisted electrical transport at high temperatures[63–65]. The calculated indirect PBE band gap of the high-vacancy configuration is approximately 3.014 eV. These results indicate that a higher oxygen-vacancy concentration produces a larger number and broadening of in-gap defect states. This effect is unfavorable for maintaining high electrical insulation at elevated temperatures.

In contrast, the low-vacancy configuration exhibits only one occupied and weakly dispersive in-gap state, as shown in Fig. 6(c). This state lies within a narrow energy range of approximately -0.28 to -0.18 eV. The corresponding DOS shows an isolated defect-state peak approximately 0.15 eV below the Fermi level (Fig. 6(d)). This feature differs from the broad defect-state distribution observed in the high-vacancy configuration. In addition, the indirect PBE band gap increases to approximately 3.692 eV. This value is approximately 0.68 eV larger than that of the high-vacancy configuration. Therefore, reducing the oxygen-vacancy concentration decreases the number of vacancy-derived states and makes the remaining defect state more localized. These changes may suppress thermal excitation and defect-assisted carrier transport.

Combined with the XPS results, the DFT calculations provide an electronic-structure interpretation of the improved high-temperature insulation of the G-Al₂O₃/L-Al₂O₃ composite film. A lower oxygen-vacancy concentration is associated with fewer and more localized vacancy-derived states, together with a larger calculated band gap. These features may reduce the contributions of thermally excited and defect-assisted carriers to electrical conduction. Suppressing oxygen-vacancy-related carrier transport may therefore be an important factor in improving the high-temperature insulation resistance of the G-Al₂O₃/L-Al₂O₃ composite film.

3.4 High-temperature insulation performance

The insulation performance of the L-Al₂O₃, G-Al₂O₃/L-Al₂O₃, and G-YSZ/L-Al₂O₃ films was evaluated. Figs. 7(a–b) show the insulation performance for the different films in the temperature range of 400~1400 °C. All the films exhibited high insulation resistance values, indicating good insulation performance below 1200 °C. Notably, The insulation resistance values of the G-Al₂O₃/L-Al₂O₃ film and the L-Al₂O₃ film were very similar at 1200 °C. This suggests that the L-Al₂O₃ film was the main factor influencing the insulation performance and was sufficient to meet the high-temperature insulation requirements below 1200 °C. However, the insulation resistance of the G-YSZ/L-Al₂O₃ film in the range of 400–1200 °C was lower than that of the L-Al₂O₃ film. This was mainly attributed to the relatively high oxygen-ion conductivity of YSZ at elevated temperatures. In YSZ, substitution of Zr⁴⁺ by Y³⁺ generates oxygen vacancies for charge compensation. At high temperatures, O²⁻ ions can migrate through vacancy-mediated hopping, thereby increasing the ionic conductivity and decreasing the electrical resistance of the YSZ film [66]. Notably, the resistance values of gas-liquid-phase composite films were higher than that of the L-Al₂O₃ film in the high-temperature range of 1200~1400 °C. This demonstrated the repair of defects in the L-Al₂O₃ film by the gas-phase insulation film became the main factor affecting the insulation performance at higher temperature above 1200 °C. This also confirmed the feasibility of improving the insulation performance of the film by repairing defects in the L-Al₂O₃ film via gas-phase deposition of an insulation film. Among them, the G-Al₂O₃/L-Al₂O₃ film exhibited the best insulation performance with an insulation resistance of 44 kΩ at 1400 °C due to its more stable states including less defects (Fig. 3), less oxygen vacancies (Fig. 5) and relatively high bandgap gap (Fig. 6). Compared with the insulation resistance of the L-Al₂O₃ film at 1400 °C, the insulation resistance of the G-Al₂O₃/L-Al₂O₃ film was improved by approximately one order of magnitude.

In polycrystalline Al_2O_3 , the grain interiors and grain-boundary/interfacial regions play different roles in high-temperature electrical transport. Within the grain interiors, oxygen-vacancy-related defect states introduce localized electronic states and thermally activated carriers, whose contribution to conduction increases with temperature. In contrast, grain boundaries and interfaces can introduce carrier-trapping sites and transport barriers. Structurally intact boundaries may hinder carrier migration, whereas grain-boundary regions containing pores, microcracks, impurity segregation, or locally reduced barriers may facilitate local carrier transport [67]. For the G- Al_2O_3 /L- Al_2O_3 composite film, the dense sputtered G- Al_2O_3 layer improves the structural integrity of the grain-boundary/interfacial regions and restricts the effective migration of oxygen-vacancy-related carriers. Together with the reduced oxygen-vacancy-related defect level, this contributes to the improved high-temperature insulation performance of the composite film.

The thermal stability of the insulation film is also one of the important indicators for evaluating its high-temperature performance. The G- Al_2O_3 /L- Al_2O_3 film was subjected to three thermal cycles in the temperature range of 800–1200 °C, and the changes in its insulation resistance were recorded, and the results were shown in Fig. 7(c). The resistance change curves of the G- Al_2O_3 /L- Al_2O_3 film exhibited excellent repeatability during the three thermal cycles from 800 °C to 1200 °C, indicating that the composite film had outstanding insulation stability at high temperatures. This is because the G- Al_2O_3 film repaired the defects in the L- Al_2O_3 film, stabilizing its insulation performance at high temperatures. On the other hand, the deposited G- Al_2O_3 film also had the α phase, which was consistent with the crystal structure of the L- Al_2O_3 film, resulting in good thermal compatibility at the film interface. Therefore, these factors contributed to the improved high-temperature stability of the film. Fig. 7(d) shows the Arrhenius-type plots of the G- Al_2O_3 /L- Al_2O_3 film during the three thermal cycles (800–1200 °C). The apparent activation energies derived from the linear fits of the three heating–cooling cycles ranged from 2.1165 to 2.1631 eV, with corresponding R^2 values ranging from 0.99699 to 0.99989. The narrow variation in activation energy indicates good consistency of the thermally

activated electrical transport during repeated high-temperature cycling, which may be associated with the stable phase composition of the G-Al₂O₃/L-Al₂O₃ film [68]. Furthermore, the insulation resistance of the film still reached 123 MΩ at 800 °C and 247 kΩ at 1200 °C after three thermal cycles. Finally, Fig. 7(e) showed a comparison of the high-temperature insulation performance of various films prepared by other methods. The insulation resistance of the G-Al₂O₃/L-Al₂O₃ film was higher than that in the existing literature at high temperatures[22, 27, 68-71]. This highlights the advantages of the G-Al₂O₃/L-Al₂O₃ film prepared in this study.

3.5 Application of the insulation film in sensor

To verify the practical applicability of the composite insulation film in the *UTTSs*, an indium tin oxide (ITO) thin-film strain gauge (ITO-*TFSG*) was fabricated on a superalloy substrate, as shown in Fig. 8(a). The *TFSGs* consist of the G-Al₂O₃/L-Al₂O₃ insulation film and the ITO sensitive film from bottom up. The G-Al₂O₃/L-Al₂O₃ composite film electrically isolated the conductive superalloy substrate from the ITO sensitive film, while the ITO film served as the piezoresistive sensing element and converted the applied strain into a resistance change. The piezoresistive performance of the ITO *TFSGs* were tested at 600 °C and 800 °C according to the method mentioned in our previous work[72]. The *TFSG* exhibits excellent stability of its piezoresistive response during several loading and unloading cycles at 600 °C and 800 °C, as shown in Figs. 8(b)-(c). The drift rates at 600 °C, 800 °C were only 0.005 h⁻¹, 0.006 h⁻¹, respectively. In addition, the *TFSG* of the temperature coefficient of resistance (*TCR*) was also monitored during three thermal cycles from 600 °C to 1250 °C according to the method mentioned in our previous work [73]. As shown in Fig. 8(d), the resistance-change rates consistently decreased with increasing temperature during the three thermal cycles, and the *TCRs* are -899.8 ppm/°C, -925.3 ppm/°C and -937.2 ppm/°C, respectively. The maximum *TCR* change rate during the three thermal cycles is only 2.1 %, which shows excellent repeatability. The detailed calculation methods and statistical analyses for the quantitative strain-sensing parameters are provided

in the Supporting Information. Thus, these results can fully indicate that the *TFSGs* based on the G-Al₂O₃/L-Al₂O₃ films show stable properties at high temperatures. It should be noted that the maximum test temperature of the ITO *TFSG* was limited to approximately 1250 °C by the thermal stability of the ITO sensing layer, rather than by the G-Al₂O₃/L-Al₂O₃ film. Overall, the sensor-level verification up to 1250°C and the independent insulation-resistance measurements of the G-Al₂O₃/L-Al₂O₃ film up to 1400°C (Figs. 7(a–b)) demonstrate the effectiveness of the composite film as an electrical insulation layer under extreme-temperature conditions.

4 Conclusion

In this study, Al₂O₃ composite films were prepared using gas-liquid phase fabrication strategy. A L-Al₂O₃ bottom film was first deposited by liquid-phase electrohydrodynamic jet printing, followed by the deposition of a dense G-Al₂O₃ film by magnetron sputtering. The advantage of the Al₂O₃ composite films lies in the G-Al₂O₃ film can effectively repair through-type defects and suppress the formation of oxygen vacancy in films. It reduced the surface roughness of the insulation film from 1.097 μm to 0.776 μm and finally repaired the through defects. Consequently, the G-Al₂O₃/L-Al₂O₃ film achieves an insulation resistance of 44 kΩ at 1400 °C, an order of magnitude higher than that of the pure L-Al₂O₃ film due to its more stable states including fewer defects, fewer oxygen vacancies and relatively high bandgap width. Moreover, the insulation resistance remains 123 MΩ at 800 °C and 247 kΩ at 1200 °C after three thermal cycles between 800 °C and 1200 °C. Practically, the Al₂O₃ composite films were applied to the thin-film strain gauges (*TFSGs*). The drift rates of the *TFSGs* at 600 °C, 800 °C were only 0.005/h, 0.006/h, respectively. In addition, the *TFSG* of the temperature coefficient of resistance (*TCR*) was also monitored during three thermal cycles from 600°C to 1250°C, and showed excellent repeatability. Overall, the Al₂O₃ composite films have significant advantages in enhancing the insulation properties and show good potential for application in various *UTTSS* at high temperatures.

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