TEM Investigation of Thermal Stability of Au Metallization to SrFeO₃/Al₂O₃ Sensing System

Dashan Wang, James J. Tunney, Michael L. Post

Institute for Chemical Process and Environmental Technology, National Research Council of Canada, Ottawa, Ontario, Canada K1A 0R6

 $SrFeO_{2.5+x}$ x>0.4 (hereafter designated as $SrFeO_3$) materials can be used as thin film gas sensors, which typically have high sensitivity and rapid response to the changes of analyzed gas concentration at temperatures ranging from 400 to 600°C [1]. The reversible uptake of oxygen of the SrFeO₃ thin film can readily occur at this temperature range resulting in compositional change accompanied by significant changes in electrical conductivity which can be monitored as the sensor transduction signal. The electrical and gas sensing properties of the film is strongly dependent on the cation and oxygen stoichiometries and film morphology, which implies that the SrFeO₃ thin film sensing system together with the substrate and electrical pads have to withstand and to be durable at the operating temperatures in order to maintain long term sensor functionality. Variations in microstructure and chemical composition of the thin film could result failure in signal production. However, at the elevated operating temperatures, the films may react with the underlying substrate or the thin film contacts of the metallizations. Transmission electron microscopy (TEM) investigations on the thermal stability of the SrFeO₃/SiO₂/Si and SrFeO₃/Al₂O₃ systems [2, 3] indicated that interfacial reactions occurred in the thin film systems during the thermal treatment; therefore, it is essential to take into account in sensor design the thermal stability of the thin film system in order to ensure longer term signal reproducibility. In addition, since the electrical contacts in a sensing system act as connections or electrical communication links during device operation between the external circuit and the active regions of semiconductor devices, as illustrated in figure 1, the contacts are preferred to be ohmic and must be low in electrical resistance; therefore, thermal stability of the contacts is critical in maintaining their small ohmic contact resistance and the functional performance of the sensor system. In the present study, the SrFeO₃ thin films were grown onto single crystal ($1\overline{1}02$) Al₂O₃ by pulsed laser deposition (PLD) and the Au thin film metallization (electrical connection pads) to the SrFeO₃ thin film by e-beam deposition respectively. The thin film systems were subjected to post-deposition thermal treatments at different temperatures. TEM investigation of the interfacial structures and reactions was performed on the Au/SrFeO₃/Al₂O₃ thin film system.

TEM characterization of the interfacial reactions on the thin film system annealed at 700°C for 48 hours indicates that the interfacial structures of the thin film system remain unchanged. High temperature thermal treatment results in significant interfacial reactions in the thin film system. As shown in figure 2, an additional layer containing Sr, O and Al, confirmed by EDS, is present at the SrFeO₃/Al₂O₃ interface in the system annealed at 1000°C for 2.5 hours and down to 500°C for 48 hours, which is believed to be the phase SrAl₂O₄, identified in our previous study [2]. In the original SrFeO₃ layer, two phases have been identified; one is Fe rich phase (SrFe₁₂O₁₉) [2] and the other is the remaining of SrFeO₃. Both phases are adjacent to the phase SrAl₂O₄ implying that they, together with the phase SrAl₂O₄, are the phases in thermal equilibrium at the annealing temperature. The outer most layer Au is no longer a uniform capping layer at this temperature, but separated spheres about one micrometer in diameter. It is seen that the film of

SrFeO₃ in the area surrounding the Au spheres is thicker than other areas, shown in figure 3 (left), and the Au spheres are partially embedded into SrFeO₃. Nano sized Au particles are also observed in the SrFeO₃ layer, as pointed by the arrows in figure 3 (right), suggesting that the Au/SrFeO₃ interface energy be lower than the Au/air surface energy. Although, in general, the Au pads show fair thermal stability, the spherization tendency and diffusion into the SrFeO₃ thin film indicate that the contact resistance might change at lower temperature for long term aging.

References

- [1] M.L. Post et al., Sensors and Actuators B 13-14 (1993) 272.
- [2] D. Wang et al., J. Mater. Res. Vol. 22, No. 1, (2007) 76.
- [3] D. Wang et al., J. Appl. Phys. 104 (2008).023530.



FIG.1. Schematic illustration of a thin film sensor system: (a) gold pads (electrical contacts); (b) SrFeO₃/Al₂O₃; (c) thermal couple; (d) heater.



FIG.2. TEM BF cross-section micrograph of the Au/SrFeO₃/Al₂O₃ system annealed at 1000°C for 2.5 hrs and 500°C for 48hrs.





FIG.3. TEM BF cross-section micrograph of the Au/SrFeO₃/Al₂O₃ thin film system annealed at 1000°C for 2.5hrs and 500°C for 48 hrs: Au sphere on the surface of SrFeO₃ (left); Nano sized Au particles embedded in SrFeO₃ (right).