RELIABILITY OF THIN FILM PHASE SHIFTER MATERIALS FOR ELECTRONICALLY STEERED ANTENNAS

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Electronically steered antennas are crucial for the Army to realize the Future Force goals. Low cost phase shifter devices will enable this technology to be fielded on a wide variety of Army platforms. It is essential that any device being incorporated in such a critical technology be reliable. Residual stress, which is known to have a drastic effect on the material, electrical, and dielectric properties, becomes of particular importance in thin film materials, where the residual stress can be several orders of magnitude higher than in bulk materials. Residual stress will be a primary determiner of the long-term reliability. Barium strontium titanate ($Ba_XSr_{1-X}TiO_3$) thin films are the principal materials of interest in phase shifter applications, primarily because of their low loss, high dielectric constant and large tunability. (Chang et al., 2000 and Ban and Alpay, 2003)

The effects of acceptor (3-10% Mg) doping, added to further tune film properties, on the BST thin films were investigated in parallel with the annealing temperature (600 to 950°C), a strong determiner of film crystallinity. Both doped and undoped films were fabricated, and they were deposited on MgO single crystal substrates using metal organic solution deposition (MOSD) (Cole et al., 2003). The residual stress in these films was measured in three ways. A Tencor stress analysis system was employed to measure the change in the substrate curvature due to the film stress (Saha and Nix, 2002), and a nano-indentation method was used to calculate the residual stress in a system by measuring the maximum penetration, the force at maximum penetration, the measured modulus and the slope of the initial unloading curve (Suresh and Giannakopoulos, 1998). These two methods were validated using the third: XRD lattice calculations (Cullity, 1978). Stresses as high as 2 GPa were observed under certain conditions, and it was possible to tune the stress level within the films by varying the dopant concentration and annealing temperature. By combining the results from all three stress measurement techniques, it became apparent that the films had a highly compressive stress at the interface that transitioned to a significantly lower, but still non-zero, stress at the top surface. Atomic force microscopy indicated that different nucleation behavior was observed at different doping levels, and that was confirmed with nano-indentation (Schwartz, 1997). The measurements also demonstrated that the surface region of the films is not stress free at these thicknesses (~200 nm) as originally thought. In this research, we evaluated and reported the effects of residual stress on acceptor doped BST as a function of post deposition annealing conditions, and how it would impact the reliability of these devices.

Barium strontium titanate films doped with 3, 5, 7 and 10 mol-% magnesium were deposited by MOSD on a 2cm by 2cm by 0.5mm magnesium oxide substrate. A total of five coats were applied, followed by a 400°C pyrolization for 30 minutes. The films were pyrolized at 350°C for ten minutes between coats. The films were then annealed in a flowing oxygen environment at temperatures ranging from 600°C to 950°C for 60 minutes. After pyrolization and post-deposition annealing, the consolidated films possessed a nominal thickness of 190-200 nm. Residual stress was evaluated via three different measurement techniques: lattice strain using glancing angle x-ray diffraction (GAXRD) (Cullity, 1978), substrate curvature by reflective laser curvature analysis (RLCA) (Saha and Nix, 2002), and effective film modulus via nano-indentation (Suresh and Giannakopoulos, 1998).

At magnesium concentrations greater than 3%, acceptor doping compensates for residual stress within the materials caused by oxygen vacancies. Annealing in an oxygen rich atmosphere can also accomplish the reduction of residual stress and oxygen vacancies, but it requires the use of annealing temperatures greater than 850°C. At 10% doping a second phase, pure or highly concentrated MgO, is visible in all samples annealed above 850°C. For all dopant concentrations, there is very little non-uniform residual stress observed via GAXRD in the films at annealing temperatures above 800°C. There is a large stress gradient observed via nano-indentation, though, and this supports the hypothesis that the grain size effects measured in GAXRD mask the non-uniform stress effects. The larger effect of annealing temperature masked any effect that Mg doping had on residual stress. A change in the crystallization behavior between 5 and 7% Mg doping was observed by all methods that can be attributed to the thermodynamics effects of the Mg in BST, as shown for the 5 and 10% in Figure 1 A. Similarly, the acceptor doping is likely the source of the drastic changes in stress observed between 800 and 850°C by all methods for the 3, 5, and 10% doped samples, as seen in Figure 1 B. The

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effect of slight film contamination during processing was seen for the 5% Mg doped BST thin films, which exhibited higher stress levels and poorer properties. In Mg doped BST thin films on MgO (001) single crystal substrates, there is a very high residual stress layer concentrated near the interface with the substrate, and this stress quickly reduces towards the surface, and the surface of the film has a slight tensile stress. The high compressive stress at the film substrate interface produces enough driving force to allow condensation and growth to be favorable. This creates a film structure that is very granular, as opposed to columnar. The stress profile for the 5% Mg doped BST films extends into the films indicates that both surface and bulk nucleation occur simultaneously within the films, as seen in Figure 1 C.

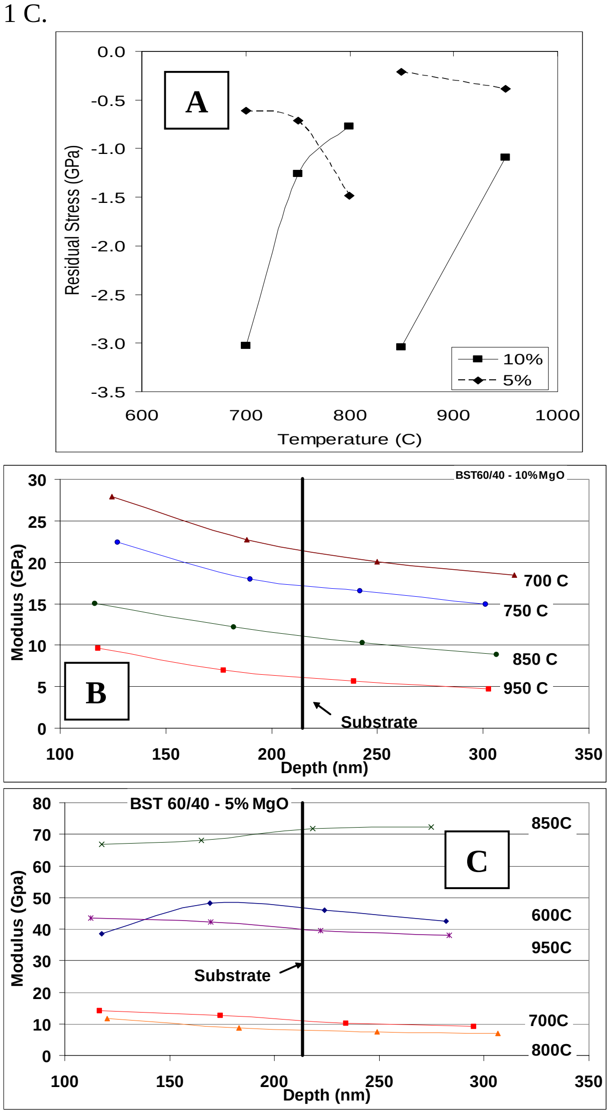

Figure 1: The residual stress measurements for 5 and 10% Mg doped BST thing films are shown in (A) for measurement taken by Tencor. Nano-indentation measurements and the modulus as a function of depth for doped BST thin films are shown in (B) 10% Mg, and (C) 5% Mg.

It is apparent that multiple measurement methods must be employed to thoroughly understand how residual stress manifests and affects thin film materials. In polycrystalline films without significant epitaxy, GAXRD, which illuminates the influence stress within the crystalline grains of the films, can only play a partial, though important, role. Nano-indentation, until now rarely used to measure residual stress in electronic thin film materials, quantifies the effects of stress on the modulus through the thickness of the film, but still requires significant effort to standardize all of the procedures and account for the numerous factors. Reflective curvature analysis, which is a standard semiconductor tool, captures the interaction between the heterostructure and substrate, but it is designed to give a gross view of entire wafers, and will require some exertion to adequately adapt the technique to the fine detail of small samples. No one technique gives the entire picture of the stress formation and distribution, and dissipation, but through a combination of these techniques a better picture of the residual stress emerges. Residual stress is a very difficult property to measure in bulk materials, and in thin films accurate determination is exceedingly difficult. For a material, where ferroelectricity can be induced with moderate levels of residual stress, it is increasingly important to fully understand the level and mechanisms of stress formation and relief, which is why it is so important to use a full battery of tests and measurements. Proper mitigation of the effects of residual stress in BST thin films for phase shifter applications is critical for the success of the technology. By varying the annealing temperature and doping of the materials, it was possible to tune the residual stress, such that long term reliability and film properties are ensured.

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