Microwave Studies of Thermally Oxidized Vacuum Evaporated Bismuth Thin Films on Alumina

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Abstract: The bismuth thin films of various thicknesses (100nm-550nm) were deposited on alumina substrate by vacuum evaporation technique. These films were thermally oxidized in air at three different temperatures (125°C, 150°C, and 175°C). The films were characterized by XRD and SEM. X-ray diffraction studies shows dominant -Bi₂O₃ crystal structure. Surface morphology shows granular shaped particles of Bi₂O₃ on alumina substrate. Microwave properties were studied in the X band (8-12 GHz) using waveguide setup. The Bismuth oxide thin film showed higher microwave transmittance in the 10 to 10.8GHz range and lower reflectance. The dielectric constant and impedance at 12 GHz was measured using waveguide slotted section. The microwave dielectric constant of these Bi₂O₃ thin films varied from 17 to 46 at 12 GHz and was dependent on thickness and oxidation temperature.

1. Introduction
Bismuth oxide is a very useful dielectric material because it exhibits considerable resistivity, permittivity and thermal stability. However, it is used only in ceramic form [1]. Bismuth oxide exists in five main polymorphic forms such as , , , , and -Bi₂O₃. There is great interest in -Bi₂O₃ due to its high oxide ion mobility arising from numerous oxygen vacancies in the crystal structure [2]. It is well known that many oxides when heated in air at high temperature dissociates to their lower oxides. Similar dissociations and the formation of new species have also been observed during the vacuum deposition of several oxides by Goswami et al. [3].

Recently, Bismuth oxide has been investigated extensively due to its optical and electrical properties [2] as well as for microwave components such as phase shifter, tunable filters and in phased array antennas [4]. Bismuth oxide is characterized by the values of dielectric permittivity, impedance and losses in microwave region. There are various reports about electrical and optical properties of bismuth oxide thin film [5, 6] but very few reports on the microwave properties of these films [4].

In the present paper, crystalline bismuth oxide thin films have been obtained by thermal oxidation of vacuum evaporated bismuth thin films. Structural, surface morphological and microwave properties have been carried out.

2. Experimental
Bismuth oxide thin films were prepared by thermal oxidation of bismuth thin films, deposited on alumina substrate by thermal evaporation under vacuum (resistive heating under a vacuum of 5 x 10⁻⁵ mbar). High purity element bismuth was used as a source material. Prior to vacuum deposition the alumina substrate were cleaned using chromic acid and soap solution, finally rinsed with distilled
water. The bismuth was deposited onto a cleaned alumina substrate by vacuum deposition technique. The films were thermally oxidized in air at different temperatures of 125°C, 150°C, and 175°C. Thickness of bismuth oxide thin films was varied from 100nm-550nm which was measured by surface profiler. The structural analysis was performed by X-ray diffraction technique (Philips PW 3710). The surface morphology was studied by SEM (JSM-6360 JEOL, Japan). Microwave transmission and reflection were studied in the X-band (8-12 GHz) for different temperature with different thickness using waveguide reflectometer setup which is shown in fig.1.

The dielectric constant and impedance of bismuth oxide thin film at microwave frequency was measured by Voltage Standing Wave Ratio (VSWR) slotted section method. The measurement of the perturbation in the position of the maxima and minima gives the permittivity and impedance of the medium. The real and imaginary parts of the permittivity and impedance can be calculated by using smith chart.

Fig. 1 Microwave reflectometer setup

3. Results and Discussion

3.1 X-ray Diffraction studies

Figure 2 shows XRD pattern of bismuth oxide thin film. The X-ray diffraction patterns of Bi₂O₃ thin films obtained for different oxidation temperature indicate predominance of -Bi₂O₃ (222) mixed with - Bi₂O₃ phase. For all temperatures polycrystalline and multiphase structure of Bi₂O₃ was obtained. The low temperature oxidation below 200°C results in -phase of Bi₂O₃. The substrate temperature has a great influence on the crystal structure of the as grown thin films.[2]

3.2 Surface Morphological studies

Figure 3 shows SEM image of bismuth oxide thin film deposited on alumina using vacuum deposition technique. The SEM graph shows the granular structure. Similar granular structure was observed by Fan et al. [2]. The diameter of the grain is approximately 2 to 3 m.

3.3 Microwave Transmission and Reflection studies

Figure 4 shows microwave transmission and reflection of bismuth oxide thin films oxidized in air at different temperature. From the figure, it is seen that, due to higher oxidation temperature, the transmittance increases by ~ 25% for all thicknesses of the bismuth oxide thin film. At lower oxidation temperature bismuth oxide of 100nm thickness shows lower transmittance than alumina. The film of 550nm thickness shows transmittance independent of oxidation temperature.
Fig. 2 XRD patterns of bismuth oxide thin film

Fig. 3 SEM image of bismuth oxide thin film.
Figure 4: Microwave Transmittance and Reflectance of Bi₂O₃ Thin Film.

The reflectance does not show any thickness or oxidation temperature dependent effect. For higher oxidation temperature high band gap was observed [6] which might be responsible for high transmittance and low reflectance.

3.4 Dielectric constant measurement

The graph of real (') and imaginary part (") of dielectric constant versus thickness is shown in fig. 5.
Dielectric constant measurement was carried out by VSWR slotted section method. The real and imaginary part of dielectric constant was calculated using the following relation [7]

\[
\varepsilon' = \left(1 + \frac{\Delta \phi \times \lambda_0}{360 \times d}\right)^2 \\
\varepsilon'' = \frac{\Delta A \lambda_0 \sqrt{\varepsilon'}}{8.686 \pi d}
\]

Where, 
\( \Delta \phi \) = Phase angle in degree
\( \lambda_0 \) = Guided wavelength
\( d \) = Thickness
\( \Delta A \) = Difference in attenuation in dB

\( \varepsilon' \) of bismuth oxide thin film at 12 GHz varies from 17 to 46. Low dielectric constant was observed for low oxidation temperature and for high oxidation temperature high dielectric constant was observed. Similar results were obtained by Valant at al. [4] for bulk material. Imaginary part of dielectric constant (\( \varepsilon'' \)) varies between 0.021 and 0.172. Imaginary part indicates the extent dielectric loss in the film. As temperature of oxidation increases the dielectric loss decreases and dielectric constant increases.

Impedance of bismuth oxide thin films varied from 47.5 + j0.5 to 51 + j1 for all temperature. It was inductive in nature.

4. Conclusion
Bismuth thin films deposited by vacuum evaporation followed by thermal oxidation are polycrystalline in nature and multiphase with predominance of -Bi₂O₃. These Bismuth oxide thin films have more transmitting nature and it was observed that transmittance increases with oxidation temperature. The reflectance in the X-band is very low. The permittivity is in the range suitable for miniaturized antenna fabrication.

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