Determination of Thin Film Elastic Modulus by Magnetostrictive Sensor and Finite Element Simulation

Cai Liang, PhD student, Mechanical Engineering, 275 Wilmore Engineering Labs, Auburn University, Auburn, Al-36849
B. C. Prorok, Assistant Professor, Mechanical Engineering, 283 Wilmore Engineering Labs, Auburn University, Auburn, Al-36849

ABSTRACT: This paper reports on the measurement of elastic modulus of thin film materials by a magnetostrictive sensor. Thin film materials of Cu, Cr, Al, Au, Sn, In, Au-rich lead free solder (AuSn) and SiC were sputter deposited onto well defined strips of MetglasTM sensor. The elastic modulus of the thin film materials was determined by measuring the resonant frequency of the sensors before and after film deposition and using the frequency shift. Thin film material's elastic modulus of Cu and Au was also determined by finite element simulation to verify the experimental results. The as sputtered films were examined by X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) to characterize their microstructures. Elastic modulus of Au and Cu films was experimentally determined with the value of 75.9 GPa and 139.2 GPa, respectively. The simulated results show about 2.5% to 4% higher than the experimental ones. This method represents a potentially new, non-destructive method to determine critical material properties of as deposited materials.

INTRODUCTION: Thin film materials have been widely applied in microelectronics and microelectromechanical systems (MEMS) for interconnection and packaging. Such as Al, Cu and Au films are dominant materials in the electric circuit interconnection. Solder materials have played an equal important role for packaging and assembly microelectronic and MEMS devices. Indium and AuSn (80/20 wt %) thin film solders, in particular, are often applied in the assembly of optical fiber and components. A Au-rich, AuSn eutectic solder for example, is the ideal choice for passive assembly photonics devices [1], not only because of its excellent creep and fatigue resistance and self alignment, but also the facts that AuSn solder can be reflowed without using the flux process[2], since flux can easily contaminate the photonics facet. Moreover, AuSn and Indium solders are often used for hermetical packaging for optical device and MEMS[3]. In addition, Pb containing solder materials are limited in use in the European Community Japan and other countries due to environmental contamination issues from disposed electronic products. High temperature, lead free AuSn (80/20 wt %) eutectic solder then becomes one of the most important alternatives for high temperature power device packaging and assembly. The mechanical properties of film materials employed in interconnection and packaging are critical to successful function of the device, and they may differ from their bulk counterparts, since the volume of materials applied in those applications is much less than the volume of bulk materials used for conventional testing. To assess the mechanical property of thin film materials, many available techniques [4-6] require additional fabrication steps to construct the test structures. These steps are not only very cost ineffective, but also may alter the properties of the film material. Nanoindentation is simple approach but it may be influenced by the substrate and the depth of indentation self. All of the techniques employed for measuring materials in bulk form are not applicable for the thin film case[7].

We report on our recent investigation of measuring the elastic modulus of thin film materials via magnetostrictive sensors. Magnetostrictive behavior exists in soft magnetic materials when the applied external magnetic field changes, the magnetostrictive materials will be subjected to compression and tension in longitudinal direction. The oscillation occurs when the applied filed has the same frequency as the material vibrating in its resonant frequency. The 1st resonant frequency of a free standing beam vibrating in longitudinal mode is given by the following equation [8].

$$f_0 = \frac{1}{2L} \sqrt{\frac{E'}{\rho}} \quad (1)$$

Where L, ρ and E' are the length, density and effective elastic modulus in isotropic elasticity of sensor material, respectively. If we take into account the changes of both elastic modulus and mass of the sensor due to a solid
Thin film rigidly and continuously deposited onto the beam surface, the effective resonant frequency and the shift will be expressed in Equations (2) and (3)

\[
f_{\text{eff}} = \frac{1}{2L} \sqrt{\frac{E'_{\text{eff}}}{\rho_{\text{eff}}}}
\]

\[
\Delta f = f_0 \left( \frac{\Delta E}{E} - \frac{\Delta \rho}{\rho} \right)
\]  

(2)  

(3)

Where, \( f_{\text{eff}} \), \( \rho_{\text{eff}} \) and \( E'_{\text{eff}} \) are the effective frequency, density and elastic modulus after thin film materials deposition, respectively. \( \Delta E \) and \( \Delta \rho \) are the elastic modulus and density changes of the sensor due to film deposition respectively.

Thin film elastic modulus can be found by Equation (4)

\[
\frac{\Delta f}{f_0} = \frac{1}{2} \left( \frac{\Delta t}{t + \Delta t} \right) \left( \frac{E_{\text{film}}}{E_{\text{sensor}}} - \frac{\rho_{\text{film}}}{\rho_{\text{sensor}}} \right)
\]  

(4)

Where \( \Delta f = f_0 - f_{\text{eff}} \), the linear relationship of relative frequency shift with thickness change is built if the elastic modulus and density are constants for film and sensor substrate.

**EXPERIMENTAL DETAILS:** Sensors were made from MetglasTM 2826MB strips, the details of the processes for measurement of sensor resonant frequency can be found elsewhere [9]. Copper, Aluminum, Chromium, Gold, Tin and Indium single phase thin films were sputtering-deposited onto the sensor by a Denton Discovery 18 sputter system. A Au-rich eutectic solder of AuSn (80/20 wt %) thin film was obtained by co-sputtering of Sn and Au targets simultaneously, where Sn and Au deposited by RF and DC sputtering respectively. A deliberated experiment study was preformed to obtain the correct composition of AuSn solder, which was examined by EDX. While SiC was deposited by directly sputtering SiC target by RF magnetron. The surface morphology of the deposited films was characterized by JEOL JSM 7000 Field-Emission SEM with the capability of EDX. And the crystal structure of the films was examined by Rigaku Powder Diffractometer that was equipped with Cu K X-ray radiator.

Simulation of resonant frequency shift due to the thin film materials of Au ad Cu deposition onto the sensor surface has been carried out by using Coventor Ware® software, and the elastic modulus of the film was extracted by the same manner as described above for the experimental data. The material properties of sensor, Au and Cu films used for simulation are listed in table 1, where the Au and Cu film elastic modulus was determined by the experimental testing.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cc)</th>
<th>Elastic modulus (Gpa)</th>
<th>Poisson ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>MetglasTM</td>
<td>7.9</td>
<td>105</td>
<td>0.35</td>
</tr>
<tr>
<td>Au</td>
<td>19.3</td>
<td>73.5*</td>
<td>0.36</td>
</tr>
<tr>
<td>Cu</td>
<td>8.90</td>
<td>139.2</td>
<td>0.35</td>
</tr>
</tbody>
</table>

* value used is the average of two methods described in reference[10]

**RESULTS and DISCUSSIONS:** SEM Micrographs revealed continuous, uniform film deposition on the sensors surface, see Figure 1. Much larger particle sizes and rougher surface for In, Sn and Al films than Au, Cu and AuSn solder were observed, which is commonly observed by other researchers due to their low melting temperatures [11]. Whiskers were only observed on the Sn film surface [12]. In the case of AuSn solder, the surface morphology was not as rough as the Sn film, and no whiskers were observed. These were suppressed by alloying with Au atoms. This is another approach of elimination of Tin whiskers by adding proper element such as gold to reduce the shorting circuit risk.
The as deposited films were examined by XRD and the results indicated that all films were polycrystalline in microstructure except SiC, which was amorphous. As expected the Au film exhibited a strong (111) preferred orientation. The as deposited Sn film developed in the $\beta$ structure, while XRD spectra for the Au-rich AuSn solder revealed a mixture of AuSn, Au5Sn and Sn. More details of the crystal structure of these film materials can be found in the references [9, 10].

The resonant frequency of a sensor after deposition of a thin layer coating can shift either up or down according to the equation (3), which is dependant on the film’s material properties, e.g. elastic modulus and density. The relative resonant frequency changes as a function of the relative thickness ratios for the tested materials are plotted in Figure 2. By assuming the thin film elastic modulus is independent on the film thickness, and the film density is the same as its bulk material counterpart. Calculated elastic modulus of these tested films is listed in Table 2. Bulk Young’s modulus implies that the test materials are in bulk scale. The value of Young’s modulus of these tested thin film materials are fell in the range of values obtained by other techniques, the details of comparison can be found in the published reference[9]. The measured Young’s modulus of indium thin film was much higher than its bulk counterpart value of 12.7GPa. This is because indium is soft that the film was scratched by the profilometer during measurement of film thickness, which underweighted the thickness ratio and resulted in higher Young’s modulus measurement. All solder materials in this study were not reflowed, thus the measured properties in this study may differ from the reflowed material.
Simulation of thin film deposition onto sensor was conducted only for Au and Cu materials. Using the same sensor geometry and thin film thickness as for the experimental test, the relative resonant frequency shifts with the relative thickness changes are plotted with the comparison of experimental data in Figure 3.

Table 2. Young’s modulus of tested thin film materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Al</th>
<th>Au</th>
<th>Cr</th>
<th>Cu</th>
<th>In</th>
<th>Sn</th>
<th>AuSn</th>
<th>SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk Young’s modulus</td>
<td>70</td>
<td>78</td>
<td>140</td>
<td>128</td>
<td>12.7</td>
<td>41.4</td>
<td>59.2</td>
<td>*</td>
</tr>
<tr>
<td>Bulk density</td>
<td>2.70</td>
<td>19.30</td>
<td>7.19</td>
<td>8.96</td>
<td>7.29</td>
<td>7.31</td>
<td>14.52</td>
<td>3.20</td>
</tr>
<tr>
<td>Thin film Young’s modulus</td>
<td>55.4</td>
<td>75.9</td>
<td>130.8</td>
<td>139.2</td>
<td>23.9</td>
<td>47.4</td>
<td>51.7</td>
<td>160.4</td>
</tr>
</tbody>
</table>

Notice: * elastic modulus of SiC varies in a wide range.
From Figure 3, it is clearly shown that 1) well defined linear relationship of relative frequency shift and relative thickness ratio observed for both Cu and Au materials by simulation 2) Frequency shift down more for Au film and shift up less for Cu film for experimentation data than simulation data, this is because the damping effect is not avoidable in experimental test. 3) Determined Young’s modulus of Au and Cu by both experimentation and simulation is in good agreement.
The relative Error of measuring the elastic modulus caused by the method used in this technique can be expressed by Equation (5), which is directly derived from Equation (4)

\[
\frac{\Delta E_{\text{film}}}{E_{\text{film}}} \leq \left| \frac{d\Delta t}{\Delta t} \right| + \left| \frac{df}{f_0} \right| + \left| \frac{df}{f_{\text{eff}}} \right| + \left| \frac{d\rho_{\text{film}}}{\rho_{\text{film}}} \right|
\]

(5)

Where \(d\Delta t\), and \(df\), are the instrument resolutions for measuring the film thickness, and resonant frequency, which were 5nm, 2µg and 25Hz, respectively. \(d\rho\) was the density diversity of the film used for calculation from its actual value, 0.1 g/cc was used for estimation. The film density was used as its counterpart bulk density, e.g. Au is 19.3 g/cc, Cu is 8.96 g/cc.

![Figure 4: Relative error of Young's modulus vs. thin film thickness](image)

Figure 4 is the relative error of elastic modulus (\(\Delta E/E\)) varies with the thickness of some of the tested films by calculation from the method applied. The accuracy of measuring the film thickness and the error of the film density dominated the error by this method. Larger error occurred when the film thickness was very small. This is attributed to error in thin film thickness measurement. It is noteworthy that the relative error resulting from this technique was less than 6% for the Au film, for less dense materials, this error could be larger, e.g. less than 7% for Al [13], which was lower than that reported by other techniques [14, 15].

CONCLUSIONS: The technique of measuring the thin film elastic modulus by employing a magnetostrictive sensor has been well demonstrated. Advantages of such technique are convenient, inexpensive and directly measuring the film material as it is. Using the density of bulk material to determine the thin film modulus would not result in considerable errors.

Acknowledgement: This work was sponsored by the National Science Foundation, Directorate for Engineering, Civil & Mechanical Systems Division under award number CMS-0528265. The authors would like also like to thank to Conventor Ware, Inc. for providing software to complete the simulation work.

REFERENCES


