Electron Radiation as an Indicator of Gold Nodule Defect during E-beam Evaporation

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Keywords: … Back scattered electron, E-beam evaporation, Gold nodules, Gold spitting

Abstract

Gold (Au) nodule defects are a common problem during metal deposition by evaporation. Small Au spheres, often referred to as spits in the semiconductor industry, can be ejected from the molten source under certain conditions. If the nodules land on the bottom electrode of a metal-insulator-metal (MIM) structure, the capacitor can fail under operating stress and is a reliability concern. Larger particles can cause electrical short circuits and even damage expensive test probes. Although rarely published, there are several known causes for this problem. We have identified several factors that directly influence Au nodule defect density and different mechanisms that lead to spitting. This paper will focus on the material aspect of the spitting phenomenon. The mechanism by which the carbon in the Au source promotes spitting will be explained. We will provide results from our experiments that confirm carbon level in the source material is directly proportional to the spit count on an evaporated film. We will also show that by fabricating an electrode in the evaporator chamber, we are able to determine the cleanliness of the source by the measured electrical potential and therefore prompting us when a melt needs to be replaced before spitting occurs.

INTRODUCTION

Gold spitting or nodule defects have been observed on evaporated films for as long as the semiconductor industry has been evaporating Au for device fabrication. The defects are typically spherical with sizes ranging from sub-half micron to over 10 microns in diameter. These particulates, if deposited on the bottom electrode of a MIM structure, will create a high electric field region causing the capacitor to fail under bias. Even though spitting is a very common problem, there has not been a lot of published work on this subject. Studies had shown that carbon in the gold pool was the main contributor [1] but the spitting mechanism was not well understood.

Crucible and Tantalum

Tantalum (Ta) has been shown to have carbon gettering ability and it is commonly used in the Au source to control spitting. However, in our experience this approach is not always effective and the addition of Ta can lead to other process issues. Using Ta in the gold source [2] can cause the molten gold to wick out of the liner due to a change in the wetting action causing power loss, tool alarms and interruptions to production. See figure 1.

Figure 1 Addition of Tantalum to the gold source can cause the gold to wick out of the crucible

Furthermore, the use of a liner will significantly reduce the charge volume resulting in increased replenishment frequency and lowered throughput.

Source of Carbon

To understand the source of the carbon one needs to look at the manufacturing processes of the Au evaporation material. A common form of Au evaporation material is in slug form, which is basically a cylindrical shape of 1/8” to 1/4” diameter and cut to different lengths per customers’ specifications. The manufacturing process starts with a gold ingot cast from material refined from bank bars or gold sponge. The material has to go through many swaging and drawing steps from an original ingot size of about 2” diameter to reach the final diameter of the slug. Lubricants and sometimes heat are used to draw the Au rod through many successively smaller dies, reducing the diameter in each pass. This drawing process reduces the diameter of the Au wire as well as creates tiny scale-like folds and cavities on the surface. Lubricants and contaminants can easily lodge in the surface scratches and crevices making complete cleaning of the material difficult.
Figure 2 SEM image of the surface of a gold slug revealed features created during the swaging process and carbon particles lodged in the cavities.

Figure 2 is a Scanning Electron Microscopy image of the surface of a gold slug. The dark spots are carbon particles confirmed by energy dispersive X-ray (EDX). See figure 3. Also visible are scale-like features formed by the drawing process. From the image, it is easy to understand that once the lubricant has turned into carbon, solvent cleaning will no longer be effective.

Figure 3 EDX survey confirmed black specks are carbon

Previously we have demonstrated that carbon in the gold charge reflects electrons and the backscattered electrons (BSE) have enough energy to cross link photoresist [3]. Carbon, having a much lower density than gold, will be released from the slugs and floats to the surface. When the beam hits the carbon, a portion of the electrons are reflected. In order to compensate for the loss in coupled energy, the crystal deposition controller responds by raising the power to maintain the deposition rate. We can look at the beam cross section and power in terms of power density. Free carbon floating on the surface of a gold pool effectively masks some of the gold surface available for evaporation; thereby increasing the power density needed for the same deposition rate. The increase in power can cause the molten gold on or near the surface to vaporize rapidly (localized boiling of the gold). As a result, Au particles are ejected from the molten pool as spits. The higher the C level the higher the power requirement the more violent the eruption.

EXPERIMENTAL PROCEDURE

A Temescal 2800 evaporator was used for this study. To detect the level of BSE generated from the gold source, we fabricated and installed an electrode in the product chamber. Since our last experiments [3] we have improved the electrode signal by moving it to a different location in the chamber. An important consideration of the design in the electrode is that no part of the flux should be blocked or the final thickness and uniformity will be affected.

A Keithley 2400 source measure unit (SMU) was connected to the electrode to detect the electrical potential. The output of the 2400 is connected to a computer where the signal is logged. The potential of the electrode during the entire recipe run is captured at a sampling interval of one second.

The evaporation material used is 5N purity Au slugs are ¼” in diameter and ½” in length. A standard production recipe melts the pellets into a source. After our standard conditioning process, the melt is used as the clean source. A gold source near the end of its useful production service was used as the bad source for comparison. We run the gold source directly in the hearth. No crucible was used in the experiments.

A Tencor Surfscan 6220 particle counter was used to summarize the number and size of particles on the monitors. We evaporated 2000A of Au film on clean Si wafers using the two different sources. A standard recipe consisted of a 30 second ramp step and 30 seconds of soak before opening the shutter and deposited the Au film at 5 A/sec. Deposition rate is controlled by an Inficon XTC-2 crystal monitor, which adjusts the beam power using signal feedback from the crystal to maintain the deposition rate. This is a typical deposition control system used in an E-beam evaporator. Deposition power was recorded for each source along with the electrode potential. To ensure consistency and repeatability, three runs were done using both the clean source and the bad one.

RESULTS AND DISCUSSION

The recipe had a simple 30 second ramp to Soak 1 power of 55 percent power and another 30 second ramp to Soak 2 power of 45 percent before deposition began. When the shutter opened, the XTC-2 took over the control with feedback from the crystal monitor to keep the rate at 5A/sec. The clean melt delivered over 9A/sec of flux upon opening the shutter and the tool responded by cutting the power to
the gun until the deposition rate stabilized at 5A/sec. The electrode picked up a potential of -0.3V during the deposition step.

![Figure 4 Deposition rate and power of a clean gold source](image)

The bad melt run had a different response. When the shutter opened, the deposition rate at 45 percent was below 3A/sec and the tool responded by delivering more power to increase the deposition rate. The deposition power eventually settled at about 55 percent. During the deposition step, the SMU measured -2.2V at the electrode.

![Figure 5 Deposition rate and power of a gold source with 30PPM of carbon.](image)

Results of the experiment are summarized in Table 1. The Surfscan data showed a clear difference in spit count between the runs. The clean source averaged about 50 particles from a starting particle count of about 20. The melt with 30ppm of C generated on average about 1,000 particles in the 2KA Au film.

### Table 1

<table>
<thead>
<tr>
<th>Carbon level</th>
<th>Spit count</th>
<th>Deposition Power</th>
<th>Electrode Potential</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 1 ppm</td>
<td>50 / 4” wafer</td>
<td>38%</td>
<td>-0.3 V</td>
</tr>
<tr>
<td>30 ppm</td>
<td>984 / 4” wafer</td>
<td>55%</td>
<td>-2.5 V</td>
</tr>
</tbody>
</table>

![Figure 6 Gold spit in a MIM structure can cause delayed failure.](image)

**THEORY**

Spitting is the result of rapid evaporation of the material near the surface of a molten pool and the phase change to gas causes the molten material to erupt. Carbon creates a partial mask to the gold source, reducing the surface area in the melt, resulting in the need for a higher deposition power for any given deposition rate. The higher deposition power coupled with a reduced clean area effectively raises the power density of the beam. When the power density reaches a threshold value, Au spitting will result.

As much as the material vendors try to remove all the oil after the manufacturing process, a lot of the slugs can still have residual carbon on them. As the Au slugs melt, the carbon floats to the surface of the source. Since we make the source from Au pellets and we add the materials in multiple runs, the carbon is trapped in layers. This explained why the carbon keeps returning even though we have not introduced any new material to the pool. As we deplete the source, we are exposing new layers of carbon trapped underneath rather than introducing more carbon into the pool.

**CONCLUSION**

We have demonstrated that for the same deposition rate, spit count increases with carbon level in the source. Au nodule defect is the result of rapid evaporation (boiling) of the material near the surface. Carbon in the Au source reduces the effective beam area while it raises the power requirement for any given deposition rate. The relationship can be viewed as power density. When the power density exceeds a certain threshold value, spitting will begin.

Carbon level in the Au source can be detected by installing an electrode in the chamber of the evaporator. Results of our experiments showed that the electrode potential is a reliable indicator of the carbon level in the gold source.
Electrons from the gun are back scattered when the beam hits the carbon on the gold source. The BSE are collected by the electrode and detected by the SMU as a negative potential. The higher the carbon level, the higher the electrode potential.

The relationship between carbon level and gold spitting can be explained by power density. High carbon level in the gold source has the same effect as increased power density. The carbon reduces the available source for evaporation and therefore reduces the beam area. Higher power is therefore needed to compensate for the loss. The net effect is increased power density. When the power density exceeds a certain threshold value, gold spitting will commence.

Proper conditioning of the gold source coupled with careful handling and preparation of the evaporation materials can reduce the amount of carbon in the gold source. The use of an electrode described in this paper is a reliable indicator of the condition of the source, therefore prompting us when a source needs to be replaced.

ACKNOWLEDGEMENTS

The author would like to thank Larry Hanes for his support and the Skyworks’ Newbury Park process engineering group for helpful discussions.

REFERENCES


ACRONYMS

pHEMT: Pseudomorphic High Electron Mobility Transistor
NMP: N-Methyl Pyrrolidone
MIM: Metal-insulator-metal