

Advanced Materials

Developing a Fundamental Understanding of Gold Spitting During Evaporation

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Abstract

We report on studies focused on the electron-beam (E-Beam) evaporation of gold (Au) that have enabled us to develop a fundamental understanding of the causes of spitting. We have found that the factors that affect spitting can be divided into two broad groups: 1) evaporation process parameters and 2) material quality. Process parameters that have an effect on the heat input or heat loss of the system have the most influence on spitting. If these parameters are not carefully regulated, an out of control process results with significant variation in spitting and the resulting measured particles. For a process that is in control, the quality of the Au plays a significant role in the degree of spitting. The level of purity, cleanliness, and the Au manufacturing methods were all shown to influence the amount of spitting.

Introduction

Spitting during the evaporation of Au is the inadvertent ejection of liquid droplets from the melt pool which cause defects known as nodules or particles in the deposited film. These nodules can significantly affect production yields as they can lead to device malfunction through the breakdown of layered structures. This is especially true of in a metal-insulator-metal (MIM) capacitor structure where nodules on an electrode layer can cause failure during operation [1]. There is thus a compelling need to reduce the number of defects caused by spitting during the evaporation of Au. In order to do that, a thorough understanding of all the factors that contribute to spitting is required.

Method

A Telemark 291 E-beam gun with copper turret containing six 40 cc pockets was used to deposit 2000 Å thick Au layers onto 100

mm diameter silicon (Si) wafers with the aid of titanium (Ti) adhesion layers. A 10 kV power supply set to 7 kV was used. Quartz crystal microbalances were used to determine film thickness and deposition rate in-situ. A double wafer holder shown in Fig. 1 was constructed for these trials. The bottom wafer received the deposition, while the top wafer served to monitor particulate pick-up from the chamber environment. Tungsten (W) and molybdenum (Mo) crucible liners with 30 cc and 40 cc capacities were used and Au levels were kept consistent by controlled additions between runs (Fig. 2).

Figure 1



Fig.1 Double Wafer Holder

Figure 2



Fig.2 Au Replenishment

The Au used for these studies was 99.999% (5N) pure. A programmable thin film controller was used to adjust applied power in order to maintain a rate set point of 10 Å/s. A standard ramp profile was run under shutter before deposition. The beam was set in circular sweep pattern at 3 Hz for both the ramp profile and the deposition time. Following deposition, particles larger or equal to 0.5 μm that were present in the Au layer were quantified with a KLA-Tencor 6420 wafer scanner. A

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gauge repeatability and reproducibility (Gauge R&R) study was conducted to quantify the amount of variance caused by the measuring system and the amount of variation was less than 10 % of the total variation. It was additionally confirmed that the Ti layer did not generate any particles due to spitting.

Process Parameters

Chamber Cleanliness

During initial trials thousands of particles were measured on the deposited Au layers. Scanning of the monitor wafers placed in the top wafer holder (see Fig. 1) produced equivalent counts. This indicated severe particulate pick-up from the chamber environment. In response, the slow rough pumping time and the venting time were significantly increased. These actions resulted in negligible chamber environment particle pick-up.

Heat Transfer

A series of trials were run using a 40 cc W crucible liner placed on top of a W shim in a 40 cc copper pocket. The intention of using a shim is to raise the crucible liner sufficiently to prevent contact with the copper pocket wall, thereby limiting heat loss

from the Au melt to the water cooled copper hearth. In so doing the power required to achieve a given rate can be dramatically reduced. However, it was found that the measured power during deposition varied by up to a factor of 3 from run to run under nominal identical conditions – Fig. 3. It was also found that the amount of Au consumed from run to run showed similar levels of variation. Close inspection of the placement of the crucible liner in the copper pocket revealed occasional point contact between the liner and the pocket. The heat transfer between the Au melt and the copper hearth was therefore inconsistent. This is a good example of process instability. From run to run the Au melt heated up to different temperatures during the programmed power ramp and conditioning cycles (under shutter) because of the varying conditions of heat loss to the copper hearth. When the shutter opened and power was under rate control, large differences in measured power were observed as a direct result of the different starting Au melt temperatures. A higher amount of spitting at higher powers might reasonably be expected and this is observed as shown in Fig. 4.

Figures 3 & 4

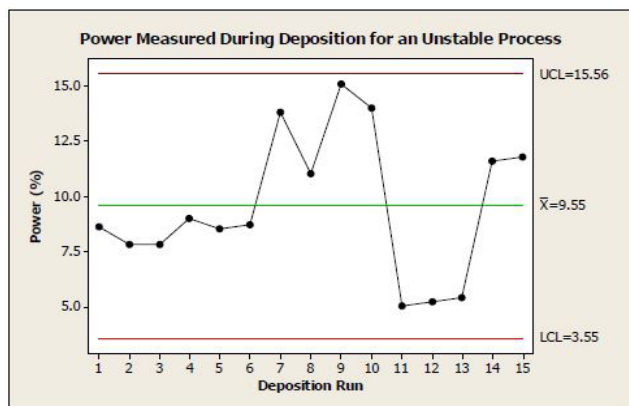


Fig. 3 Large variations in power were observed from run to run due to process instability resulting from inconsistent heat transfer.

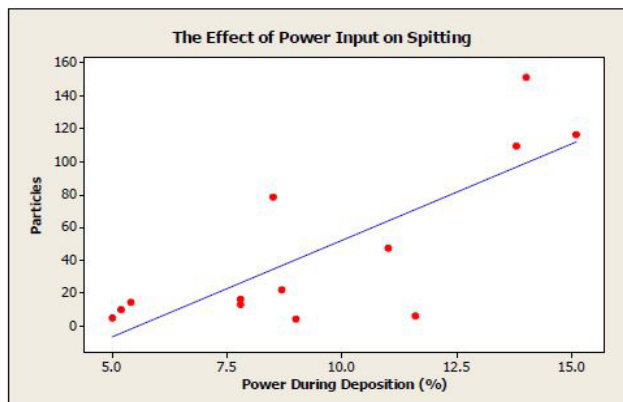


Fig. 4 Particles due to spitting showed a positive correlation ($r = 0.79$) with power during deposition.

Crucible Liner Weight

Another process parameter which, if not controlled, can lead to process instability is crucible liner weight. It was found that crucible liner weights can vary by as much as 20 % even when they have the same nominal dimensions, are made from the same material and are purchased from the same vendor. Fig. 5 shows the measured power from a series of deposition runs from two Au melts made in 30 cc W crucible liners. Since these were 30 cc liners placed on W shims in a 40 cc copper pocket there was a consistent gap created between the liner and the pocket and the process instabilities associated with point contact referred to in the previous section were avoided. There was however a significant difference in the weights of the two W liners: 175 g and 200 g. Fig. 5 shows quite clearly that the measured powers during deposition were in distinctive ranges. Although this difference is

not as extreme as that shown in Fig. 3, it nevertheless can be expected to influence the degree of spitting during the deposition of Au.

Figure 5

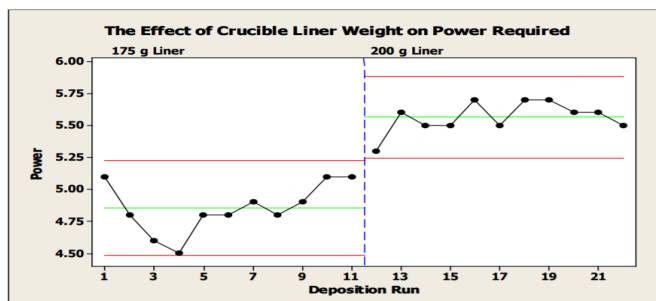


Fig. 5 Different ranges of power during deposition were measured for two Au melts in 30 cc W crucible liners with different weights

Crucible Liner Material

In terms of Au spitting, a more significant process parameter than crucible liner weight was the material type of the crucible liner. Two distinct batches / lots of Au were used to create four Au melts in 30 cc crucible liners. Two melts were in W crucible liners and two melts were in Mo crucible liners. Separate ramp profiles were created for the W and Mo crucible liner melts to account for material density differences. In both cases the ramp profiles were optimized to produce the same rate of deposition (10 Å/s). The particles due to spitting from the melts in the Mo crucible liners were measured to be significantly higher compared to those from the melts in the Mo crucible liners. Fig. 6 illustrates this observation with average particles counts of 46 and 68 for the two melts in W compared to average particle counts of 11 and 4 for the two melts in Mo.

Figure 6

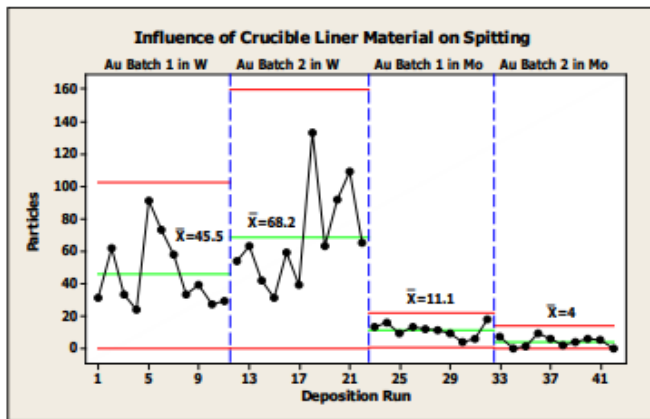


Fig. 6 Particles due to spitting were observed to be much lower for Au melts in Mo crucible liners compared to Au melts in W crucible liners

Material Quality

Purity

All the Au used in this study had a purity of at least 99.999% as measured by Glow Discharge Mass Spectroscopy (GDMS). This is considered the minimum purity required in order to prevent excessive spitting during evaporation since volatile elements with higher vapor pressures (compared to Au) are kept to a minimum [2].

Cleanliness

A further source of material contamination that is not easily detected by bulk measurements of purity is organic surface residue. If the Au source material has appreciable surface organic residue then during melt formation and subsequent additions, the organic material is transformed into a thin carbon film that floats over the top of the Au melt. The carbon film has the effect of reducing the effective area of the Au melt from which evaporation can occur. Under deposition rate controlled

conditions, power is increased to compensate for the reduced evaporation area and the level of spitting subsequently increases [3, 4]. In this study we monitored the surface cleanliness of Au source material with a Scanning Electron Microscope (SEM) using a back scattered electron detector that produces excellent contrast between organic contaminants and the Au background. Fig. 7 shows two such SEM micrographs: one corresponding to a clean Au surface and one corresponding to a contaminated Au surface.

Figure 7

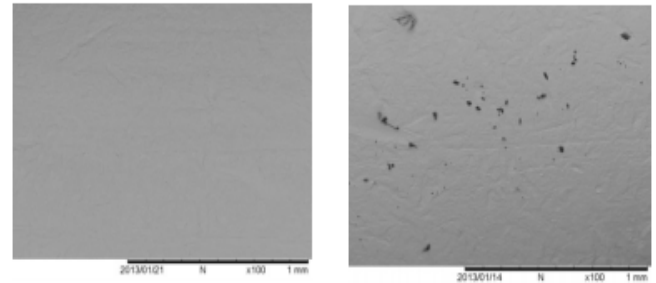


Fig. 7 SEM micrographs of Au source material showing an example of a clean surface (left) and an organic contaminated surface (right).

The effect of organic surface contamination on spitting was confirmed by E-Beam evaporation trials and subsequent measurement of particles. Fig. 8 is a plot of particles corresponding to two distinct Au material lots used to create two Au melts in 30 cc W crucible liners. The first series plotted is that for a known clean Au material lot. Particle levels were seen to be consistently low with an average count of 45. The second series plotted corresponds to a dirty Au material lot with an average count of 637 particles after 7 runs. Following these 7 runs, the surface of the Au melt was wiped using a lint free wipe soaked in isopropyl alcohol. A significant amount of carbon residue was detected on the wipe (Fig. 9). Further deposition runs were then conducted on this melt and the particles counts from these runs demonstrated an immediate drop to an average of 58.

Figure 8

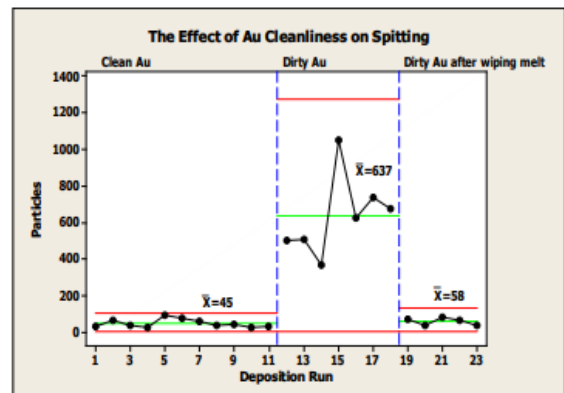


Fig. 8 Particles due to spitting from a clean Au material lot (1st series) compared to those from a dirty Au material lot (2nd and 3rd series).

Figure 9



Fig. 9 Carbon residue after wiping a Au melt made from organic contaminated Au source material

Conclusion

We have found that there are many factors that influence the amount of spitting during the evaporation of Au. First there are many process conditions and parameters, which if left uncontrolled, can have an overwhelming influence on spitting. These include the chamber cleanliness, the degree of heat transfer between the Au melt and the water cooled copper, the consistency of the crucible liner weight, and the material of the crucible liner. Once these process parameters are held consistent and a controlled process results, it is then possible to discern influences of the Au source material. Au that is of very high purity and has an extremely clean surface free of organic contamination results in the least amount of spitting.

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References

- [1] J. Cotronakis, M. Clarke, R. Lawrence, J. Campbell, C. Shaw, Continuous Defectivity Improvements and Impact on High Density Metal-Insulator-Metal (HDMIM) Capacitor Yields, 2004 CS MANTECH Conference.
- [2] D. Mattox, Handbook of Physical Vapor Deposition (PVD) Processing, 1998, Noyes Publications.
- [3] K. Cheng, M. Le, D. Mitchell, L. Hanes, Effects of Electron Radiation Generated during E-Beam Evaporation on Photoresist Liftoff Process, 2010 CS MANTECH Conference.
- [4] K. Cheng, Electron Radiation as an Indicator of Gold Nodule Defect during E-Beam Evaporation, 2011 CS MANTECH Conference.

ACRONYMS

E-Beam: Electron Beam
Au: Gold
MIM: Metal-Insulator-Metal
Ti: Titanium
Si: Silicon
W: Tungsten
Mo: Molybdenum
GDMS: Glow Discharge Mass Spectroscopy
SEM: Scanning Electron Microscope

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