AC 2012-4257: PROCEDURE DEVELOPMENT OF THERMAL EVAPORATION PROCESS FOR INCORPORATION INTO UNDERGRADUATE CURRICULUM

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Procedure Development of Thermal Evaporation Process for Incorporation into Undergraduate Curriculum

Abstract
The physical vapor deposition (PVD) of metal thin films by means of evaporation at low pressure was investigated as part of an undergraduate independent study to streamline the process and improve its efficiency, with the ultimate goal of incorporating the process into an undergraduate senior engineering course. The study would result in the development of an optimized and streamlined procedure for use by students in a laboratory based course focused on the design and production of Micro and Nano devices, like solid state devices. Based on known issues related to this process, three specific objectives were targeted: optimizing the thermal evaporation procedure that would result in a >4000Å aluminum thin film layer, exploring the effects of the process parameters on deposition rate, and investigating ways to extend the usable life of the metal source holders. Several process variations were explored and conclusions were drawn as to the process parameters which best streamline the process for use in a time limited undergraduate course. Based on the work of the independent study, a complete process procedure was developed which was successfully adopted in the lab activity of the Nano/micro Systems Engineering course.

Keywords: Micro and Nano education, Thermal Evaporation, Nanotechnology

Introduction
The study of micro- and nano-scale devices and materials continues to provide promising new avenues of research and application across a wide spectrum of fields [1]. The broad multidisciplinary nature of the subject prompts recognition of the need for expanded treatment in university level curricula for undergraduate students. In a specific case like solid state micro-electronics, students’ exposure to experiences related to equipment and processes used for the fabrication of functional devices greatly impact their education and understanding of the field. Moreover, it provides them the opportunity to develop a skill set and an advantage that helps them as they start their careers in this field.

The production of completed devices often entails a considerable investment of time and resources owing to the number of processing steps associated with these devices as well as the complex nature of the processing technology. Successful device fabrications necessitate a certain level of procedural optimization, which is not suitable for integration into a semester long undergraduate course. This is in addition to inherent challenges related to working in the clean room and the diversified background needed to be covered before even starting this process. The involvement of undergraduate students into the development of processing procedures allows the students to gain a deeper level of understanding and experience in focused areas of study. It also allows the instructor to assess the experience and produce a frame of reference when attempting to integrate the fabrication part into an undergraduate curriculum. A well-defined processing
sequence is crucial for the successful, and reproducible, fabrication of small scale devices like integrated circuit [2]. This is particularly important in undergraduate laboratory coursework to maintain focus on the educational objectives and avoid distractions related to the optimization of the process.

Fabrication processes for devices typically involve a combination of layer additions, modifications, and selective material removals; each layer change being the result of a distinct processing step. In the specific case of integrated circuit (IC) fabrication on silicon wafers, all processing steps must be executed within narrowly specified ranges in order to obtain functional final products. One of the last steps in IC fabrication is the formation of a top, conducting layer, patterned into surface traces and contacts used to interface with the outside world. A number of materials can be used for a top contact layer, with aluminum being one of the more common materials due to its relatively low cost and high conductivity [3].

One of the laboratory activities in the newly developed senior undergraduate course EGR457 Nano/Micro-Systems Engineering at GVSU is to deposit a 4000Å thick aluminum layer using a thermal evaporation system. In order to obtain the optimized deposition procedure and process parameters, an independent study was arranged for an undergraduate student to investigate the effects of deposition parameters on the quality of deposited aluminum thin film layers and produce results indicating an optimized process with a stream-lines procedure, prior to the offering of the course. This study included using the thermal evaporator to deposit aluminum thin film layers through a process called thermal evaporation which is one form of Physical Vapor Deposition (PVD). Inside the vacuum chamber of the evaporator, high currents are passed through tungsten resistive filaments, heating attached aluminum clips which act as the evaporation source [3]. As the temperature of the tungsten increases the aluminum would first melt and then evaporate. Material deposition occurs through the line-of-sight travel of the evaporated aluminum from the source to the substrate. A wide range of tungsten filament configurations are available for various types of evaporant.

There are a number of generally recognized challenges associated with PVD including issues of layer thickness uniformity, low rates of source utilization and a lack of processing control of film properties [4]. The tungsten filaments are also known to have a high degree of temperature sensitivity, particularly in relation to the rate of temperature increase. Rapid thermal expansion can cause the filament to break prior to the completion of the evaporation. Manufacturers recommend single uses as the reliability of the filaments decreases significantly with any level of usage. As the temperature of the source cannot be controlled in a uniform or repeatable fashion, the end results of evaporations can have wide variations. Using shutters to block deposition to the substrates during source pre-melting, filament wetting, and rate accumulation provide important means to both reduce impurities in the deposited layer and increase the layer uniformity [5].
Developed Approach and Methodology
The objectives of the project included:

1. Investigation of a thermal evaporation process that would result in a >4000Å aluminum thin film layer and optimization of the procedure;
2. Exploration of the effects of the process parameters on deposition rate to arrive at an optimized set for incorporation in an undergraduate course;
3. Investigation of the best ways to extend the usable life of the source holders;

Experimental Setup and Equipment
A Trovato 1830-A thermal evaporation system provides a high vacuum environment, power supplies for source holders, and the monitoring systems necessary to initiate and assess the progress of an evaporation trial. Figure 1 shows the Trovato 1830-A system with major elements highlighted. Two control systems, the Inficon IC-5 Deposition Controller and the Sycon STC-200A Thickness Controller, are available to program, initiate, and monitor a deposition sequence. The Sycon controller was chosen for this study to maintain consistency.

![The Trovato 1830-A thermal evaporator system](image)

Figure 1: The Trovato 1830-A thermal evaporator system

The evaporation chamber can accommodate six substrates which are shielded by a shutter system during rate accumulation. The deposition rate and the thickness of the thin film are determined by the deposition controllers. The power supplied to the tungsten filaments can be controlled with the deposition controller either manually or automatically.

Parameters that affect the deposition process and the quality of the thin films include the types of tungsten filament source holders, the method of powering the source holder, the method of affixing the evaporant to the holder, and the vacuum conditions of the chamber.
Two types of tungsten filament source holders were tested: baskets and coils with variations in the number and thickness of the tungsten wires stranded together. A representative image of the single wire basket is shown in Figure 2 and an example of a coil is shown in Figure 3. Table 1 shows the various types of these filaments that were used for this experiment.

![Figure 2: A single wire Tungsten basket](image)

![Figure 3: General shape of the tungsten filament coils used in evaporation trials](image)

Table 1: Various types of filaments used for PVD optimization.

<table>
<thead>
<tr>
<th>Type</th>
<th>Variation</th>
<th>Current Rating (A)</th>
<th>Power Rating (W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basket</td>
<td>Three-wire</td>
<td>52</td>
<td>272</td>
</tr>
<tr>
<td></td>
<td>Single-wire</td>
<td>59</td>
<td>237</td>
</tr>
<tr>
<td>Coil</td>
<td>Three-wire</td>
<td>87</td>
<td>496</td>
</tr>
<tr>
<td></td>
<td>Four-wire</td>
<td>70</td>
<td>448</td>
</tr>
</tbody>
</table>

In all evaporations, the evaporants used were aluminum clips, approximately 0.5” in length and pre-bent into a v-shape.

**Methodology**

Following a broad review of available processing schemes including many specifically tailored for vastly different configurations of evaporation equipment, two approaches to power sequencing were investigated [6] [7]. These approaches are explained in Table 2. Power levels were specified as a percentage of the user-defined maximum input into the Sycon controller.

**Observations and Experimental Findings**

**Method 1: Manual Linear Increase**

With this method, the general trend was long deposition times of 30 minutes or more, low deposition rates (typically below 5 Å/s), and poor reproducibility. Evaporations from a single filament were unable to obtain the required 2000 Å thicknesses (thus 4000 Å from two filaments), and the usual results were thicknesses between 400 to 1500 Å. Single-wire and 3-wire tungsten baskets were used. Based on multiple experiments, it was found that high deposition rates using the tungsten baskets were not reliable for power schemes held within rated maximums. Subsequent attempts with the 3-wire baskets using this method while keeping
current and power levels within rated tolerances produced unsatisfactory results like: exceedingly long deposition times (30+ minutes), low maximum deposition rates (2-3 Å/s were typical), and low overall layer thicknesses between 400 to 800 Å. For the single-wire baskets cases where the trials ended with a current loss due to breakage, un-evaporated aluminum pooled at the bottom of the basket with little or no wetting of the rest of the wire. The breakage point for the baskets was just below the upper wire bend or near the point of contact of the central wire with the top of the coalesced aluminum.

Table 2: Approaches to PVD by power variation utilized to investigate the process.

<table>
<thead>
<tr>
<th>Approach</th>
<th>Power increase rate (Amp/second)</th>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Manual, gradual, linear, controlled increase of power levels.</td>
<td>0.5 – 1</td>
<td>- Allows for an increased temperature acclimation period for the filaments.</td>
<td>- Collection of melted aluminum at the bottom of baskets or on the low points of the coils when power increase rate is low.</td>
</tr>
<tr>
<td>Programmable semi-automatic routine using the controller and a pre-set profile.</td>
<td>Ramp profile provided by figure 4.</td>
<td>- Prevents the melted Al from falling from the source holder. - Shortens the overall deposition time. - Increases the deposition rate.</td>
<td>- Complex power scheme.</td>
</tr>
</tbody>
</table>

Higher deposition rates, with a comparable quantity of evaporant, were achieved with greater ease using the 3-wire and 4-wire coils. Deposition rates and total deposited layer thicknesses obtained using tungsten coils showed a high sensitivity to the rate of power increase. With low rates of power increase, the result was typically a thin layer and low maximum deposition rates (< 2 Å/s). A fast rate of power increase tended to achieve higher deposition rates but also produced thin layer thicknesses due to the majority of the evaporant sliding off the coil prior to evaporation.

![Figure 4 Ramped power scheme used with Sycon controller. Actual currents and voltages delivered to the source holders varied.](image_url)
Method 2: Semi-Automated Ramped Increase Approach

Using this method, several evaporation runs were conducted with single-wire baskets and three- and four-wire coils. In comparison to the linear method, total deposition times were much shorter (less than 15 minutes) with this method. Maximum current levels were kept at or below the rated values in order to prolong the source holder life. As shown in Table 3, with the three- and four-wire coils, substantially higher loading levels are possible in comparison to the baskets. Depending on the technique used to place the source clips, 60 or more clips can potentially be loaded.

Table 3: Evaporation runs using the programmed power increase scheme followed by manual control.

<table>
<thead>
<tr>
<th>Source Holder</th>
<th>Loading</th>
<th>Holder Usage</th>
<th>Maximum Deposition Rate</th>
<th>Run Time</th>
<th>Deposited Layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-Wire Basket</td>
<td>15 Al Clips</td>
<td>2nd Use</td>
<td>4.3 Å/s</td>
<td>18:30</td>
<td>1368 Å</td>
</tr>
<tr>
<td>3-Wire Coil</td>
<td>33 Al Clips</td>
<td>1st Use</td>
<td>10.5 Å/s</td>
<td>11:00</td>
<td>1945 Å</td>
</tr>
<tr>
<td>3-Wire Coil</td>
<td>40 Al Clips</td>
<td>2nd Use</td>
<td>11.3 Å/s</td>
<td>9:13</td>
<td>2762 Å</td>
</tr>
<tr>
<td>3-Wire Coil</td>
<td>50 Al Clips</td>
<td>3rd Use</td>
<td>16 Å/s</td>
<td>12:02</td>
<td>2859 Å</td>
</tr>
<tr>
<td>4-Wire Coil</td>
<td>55 Al Clips</td>
<td>1st Use</td>
<td>9.3 Å/s</td>
<td>7:20</td>
<td>1638 Å</td>
</tr>
<tr>
<td>4-Wire Coil</td>
<td>55 Al Clips</td>
<td>2nd Use</td>
<td>11.7 Å/s</td>
<td>8:25</td>
<td>2114 Å</td>
</tr>
<tr>
<td>4-Wire Coil</td>
<td>55 Al Clips</td>
<td>1st Use</td>
<td>19 Å/s</td>
<td>9:27</td>
<td>3131 Å</td>
</tr>
</tbody>
</table>

With this method, the deposition rates obtained from either type of coil were typically near 5 Å/s for the majority of the deposition time with peak deposition rates between 10 and 20 Å/s. Increasing the number of affixed aluminum clips did not automatically lead to an increase in maximum deposition rates or in the deposited layer thicknesses. With basket filaments, a rapid rate of power increase during part of the process would usually lead to a failure as a breakage in the wire would occur. The coils performed much better under these conditions. The thickest layer achieved, with a single coil, was 3131 Å which was obtained with a very rapid increase up to the maximum current rating following the programmed power scheme. Process times were typically under 10 minutes. The overall performance of the coils was seen to degrade with each usage with the total thickness of the deposited layer dropping by as much as 500 Å with each additional evaporation performed.

The Optimized Evaporation Process

Tungsten coils have the distinct advantage over the baskets in that they can support increased source loading and higher current levels. Significantly higher deposition rates with corresponding reductions in process times and increased overall thicknesses are also characteristics of the deposition runs with the coils. For both types of filament configurations, the number of breakages was significantly reduced by maintaining applied currents and voltages
within rated tolerances. The implementation of the automatic power ramping for pre-melting
and wetting of the filaments further aided in the extension of the usable life of the filaments. In
the case of the three- and four-wire baskets, typical usages extended to four or five evaporations
at the expense of slightly diminishing layer thicknesses. The critical component of the vacuum
evaporation proved to be the manual control of the power supplied to the tungsten filament.
Attempting to maintain a balance between the rates of power increases and the overall level of
supplied power proved to be most effective in maintaining a stable deposition rate and achieving
optimal layer thicknesses.

Based on the results of the detailed evaporation study, the optimized evaporation process was
formulated. The required aluminum layer thicknesses could be achieved most reliably using 3-
wire coils with a loading of 40-50 aluminum clips. The baskets were unable to provide a
sufficiently thick aluminum layer due to their lower carrying capacity. Using the
preprogrammed power sequence to pre-heat and wet the filament holder helped to stabilize the
process and increase the reusability of the source holders. Manual control of the power
following the automated sequence provided a means of changing deposition rates.

**Implementation and Assessment**

Following the design of the process procedures for thermal evaporation, the evaporation process
was adopted in the Nano/micro Systems Engineering course offered during Fall 2011 as part of a
laboratory fabrication sequence. The result of a batch evaporation process, where the metal layer
for silicon integrated circuits was deposited, successfully produced an aluminum layer of the
required thickness using the developed evaporation process from two source holders. The
student involved in the procedure development was available to explain the details of the
equipment operation as well as demonstrate the actual process. 87.5% students received 75% or
higher lab grades. During the end of term evaluation, 80% of students indicated that the course
was taught well. One of the comments regarding the lab activity was: “The lab did well to
reinforce what we learned in lecture and I enjoyed the lab.” A major advantage gained from the
independent study is the streamlined procedure for PVD which was carried out during the regular
course without delays relating to process parameters optimization.

From the independent-study participating student’s perspective, the study of the problem of
thermal evaporation as an independent study was valuable for two reasons. First, it offered an
opportunity to study in depth an integral process involved in solid state device fabrication, with
direct supervision from the professor. The scope of the study covered a number of new areas for
the student in terms of equipment operation, material properties, and process execution. Second,
it provided an opportunity to build experience with experimental methodology and experimental
design. In order to effectively compare the results of processing changes, a large data set had to
be accumulated and assessed. Efficiency of the optimization study depended on the student
being able to identify and track the key variables that required focused attention.
Conclusion
A lab activity of thermal evaporation was developed for the senior undergraduate course EGR457 Nano/micro Systems Engineering through an independent study. Involving an undergraduate student to carry out narrowly focused optimization projects provides an opportunity to develop individual student’s skill sets. It also prepares the student for further academic endeavors and allows an opportunity where the student can share acquired knowledge with peers. The selection of evaporant source holder and the design of the evaporation process vary considerably depending on individual requirements. The tungsten coils proved superior to the basket design to reliably and consistently achieve the stated objectives. The process procedures, with an initial coil warming followed by a rapid power increase to evaporation conditions, proved the most effective way to provide the necessary layer thicknesses without exceeding current ratings. Manual power control combined with the developed procedures for the pre-melting of the evaporant allowed for the most consistent results given the constraints of the equipment.

References