Effects of Deposition Temperature on Stacking Fault Density and Texture Transformation in Thin Silver Films

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I. Abstract X-Ray Diffraction analysis was used to measure stacking fault density and texture transformation in silver films created with electron-beam evaporation techniques. Films were heated during the deposition process to obtain samples with different deposition temperatures. Samples were analyzed using X-Ray Diffraction for texture and fault density in as-deposited states and after intervals of annealing. Analysis of scans reveals fewer faults and less (111) to (100) texture transformation in films deposited at higher temperatures.

II. Background Thin metal films are commonly used in microelectronics. Understanding how these films form and change provides valuable information about their properties. Previous unpublished research [1] completed by the Houghton College Physics Department and the Cornell Center for Materials Science has shown a dependence of (111) to (100) texture transformation on the deposition rate of thin silver films. A higher deposition rate was shown to cause more complete texture transformation, occurring more quickly in the material. Current theories [2] predict a correlation between stacking fault density and texture transformation in such films.

Stacking faults occur in films when layers of the crystal structure begin to stack out of sequence with proceeding layers. Layers of the same type stack so that the atoms line up with those in previous layers of that type (Figure 1). Layers will stack according to a pattern (ABCABC) but can also stack in the opposite order (CBACBA). When a new layer is stacked on previous layers out of sequence of the preceding pattern, a stacking fault occurs (Figure 1).

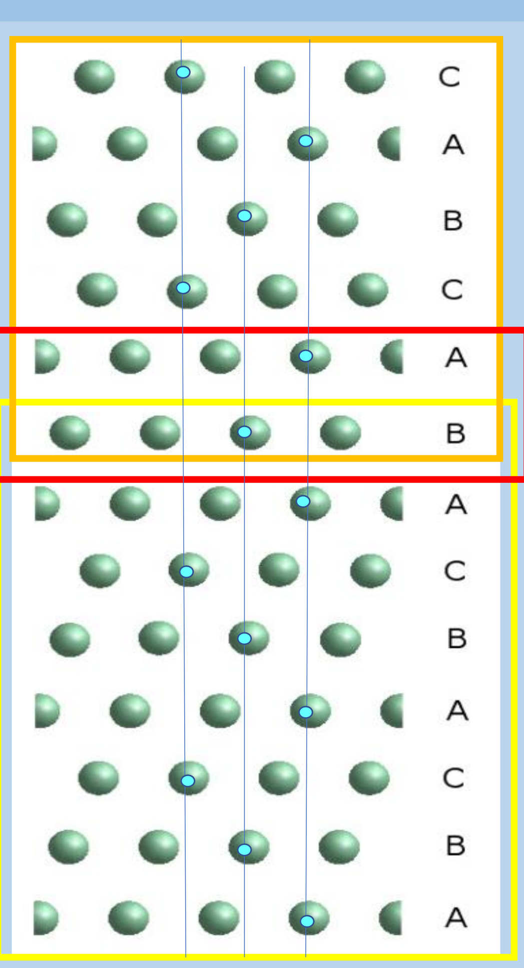

Figure 1. Atomic Layers of silver crystal have different layer types (A,B,C). When layer stacking shifts from ABC (yellow) to CBA (orange) a stacking fault occurs at the layer that breaks the pattern (red).

III. Procedure Films were deposited onto silicon wafers using electron beam evaporation in the apparatus shown in Figure 2. Wafers were heated during the deposition process to a constant temperature for each film. Desired temperatures were achieved through the use of nichrome wire, insulated with fiberglass sleeving and attached to a thermocouple. This wire snaked between plates of aluminum to heat the metal onto which the silicon wafers were mounted.

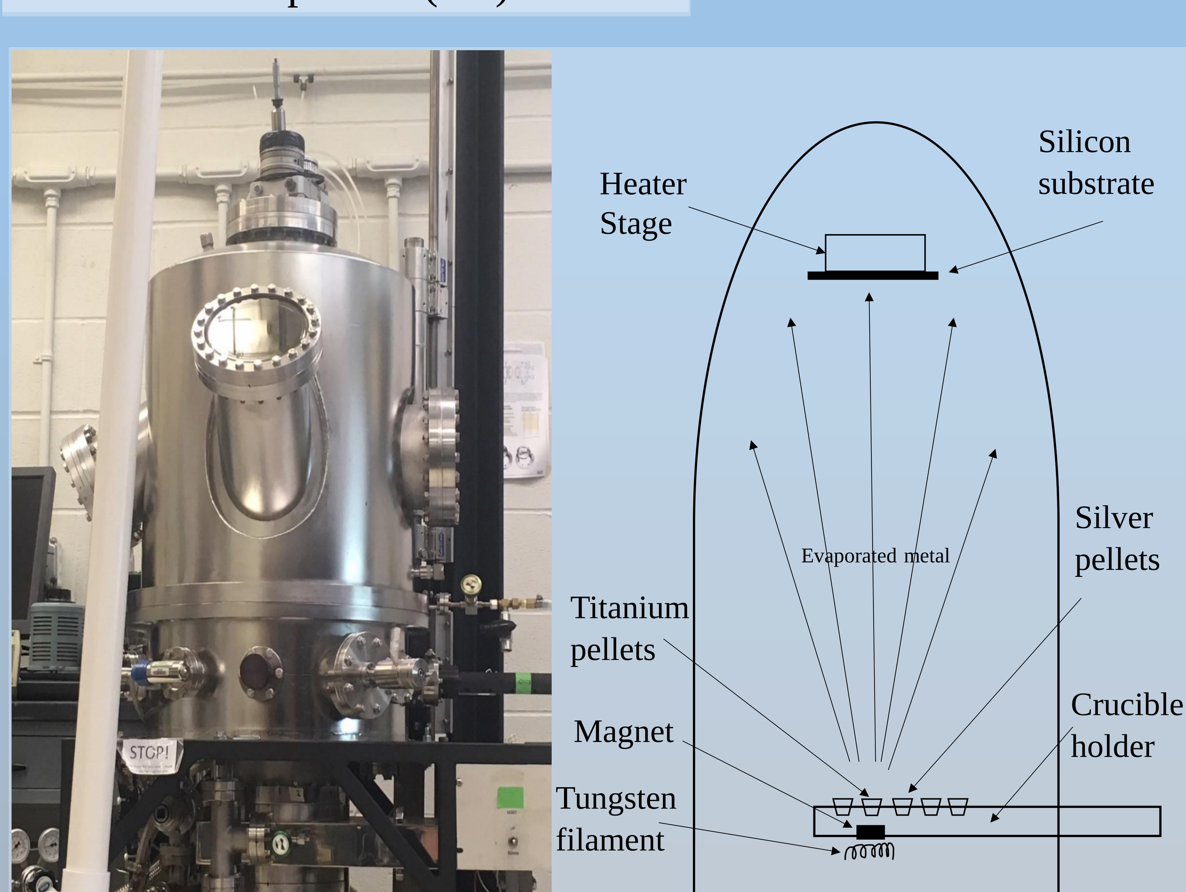

Figure 2. Deposition chamber used to create sample films using electron beam evaporation. Silicon wafers were placed on a heated stage to control temperature of films during deposition. Titanium and silver were evaporated from pellets and deposited on silicon wafers.

A titanium adhesion layer was deposited on the wafers first to prevent the silver films from pealing off of the silicon wafer. Silver was then deposited on top of the titanium layer.

After deposited, films were cut into smaller samples to be analyzed and annealed. XRD scans were taken using the Rigaku SmartLab XRD at the Cornell Center for Materials Research. Symmetric theta-2theta scans and L-scans were taken of each sample after deposition and after each period of annealing. Annealing was carried out in ten minute increments for the sample deposited at room temperature and in two and a half hour increments for all other samples. Annealing occurred at 120°C. Theta-2theta scans reveal the (111) and (100) texture of the samples, and show the texture changed through annealing. L-scans were used to analyze the fault density in (111) oriented grains within the crystal structure of the samples.

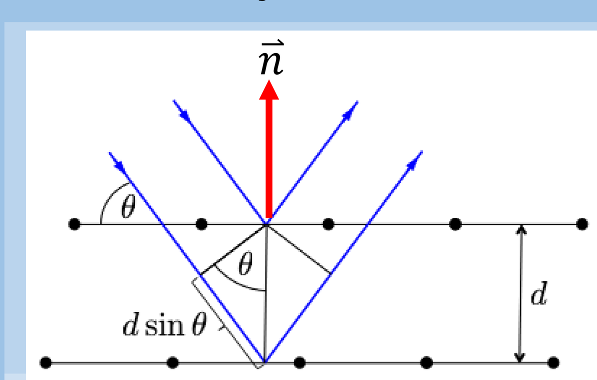

Figure 3. Diagram of incoming and diffracted x-rays on crystal layers of silver samples. Vector $\vec{n}$ represents the scattering vector used in L scans.

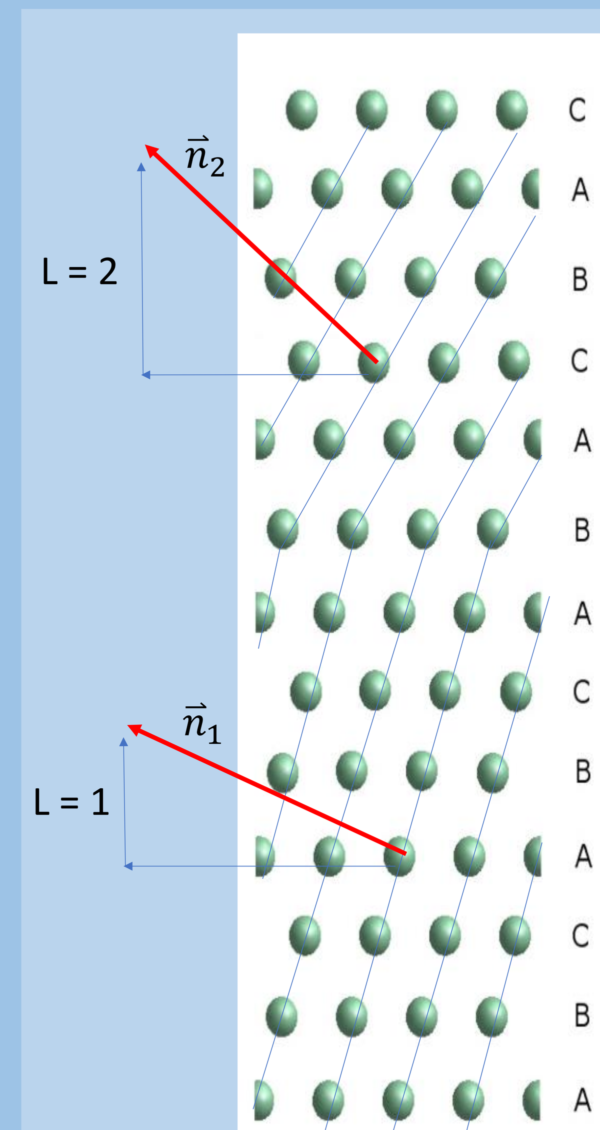

Figure 4. The planes formed by different stacking sequences have scattering vectors with different L components. Rotating the sample allows the x-ray beam to be incident on different planes.

L-scans were designed to measure intensity of scattered x-rays based on the scattering vector of the incident x-rays (see Figure 3) which is normal to the plane of the crystal. This was achieved by rotating the sample and scanning different angles. Each angle corresponds to a scattering vector for a plane oriented in a different direction (see Figure 4). Intensities could then be plotted against the L-component of the scattering vector (see Figure 4). The full width at half maximum (FWHM) can then be compared. Larger FWHM correlates to a larger stacking fault density. When more stacking faults are present in a sample, the thickness of planes oriented in the same direction is smaller, leading to less interference in the outgoing x-rays. This causes a bigger FWHM to occur.

IV. Results The symmetric theta-2theta scans completed on the sample deposited at room temperature before, during, and after annealing show an increase in (100) oriented grains and a decrease in (111) oriented grains as shown in Figure 5.

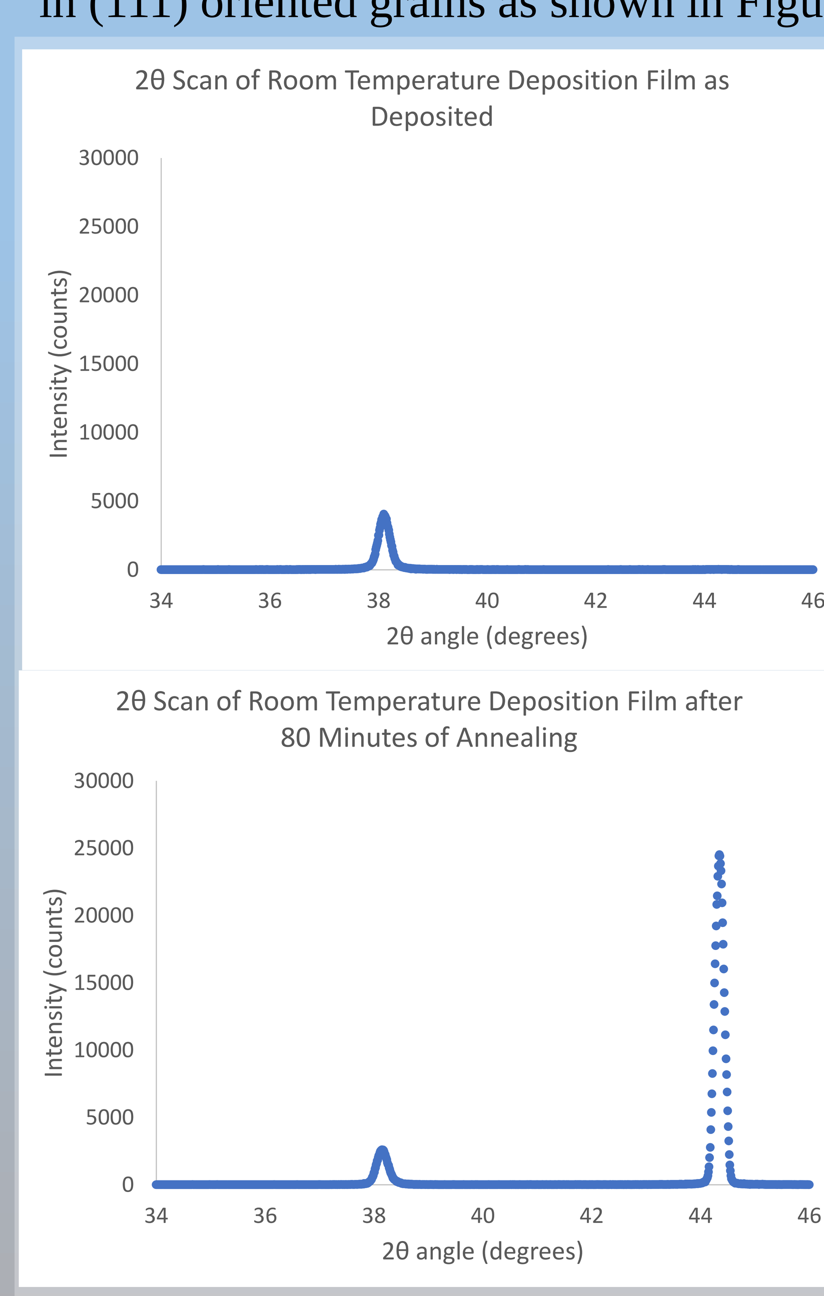

Figure 5. Symmetric theta-2theta scans of the room temperature deposition sample as deposited (left) and after 80 minutes of annealing at 120°C (right) show a change in texture. The as-deposited scans exhibit a (111) peak around 38° and no significant (100) peak indicating that the structure is mainly (111). After 80 mins of annealing, the sample shows a significant (100) peak around 44° and a smaller (111) peak indicating that the texture has transformed to mostly (100).

The L-scans (Figure 6) conducted on the same sample show a slightly higher density of stacking faults in the as-deposited sample than in the sample after annealing. As anneal time increases, the stacking fault density decreases. This change occurs at the same time as the (111) to (100) texture change is happening.

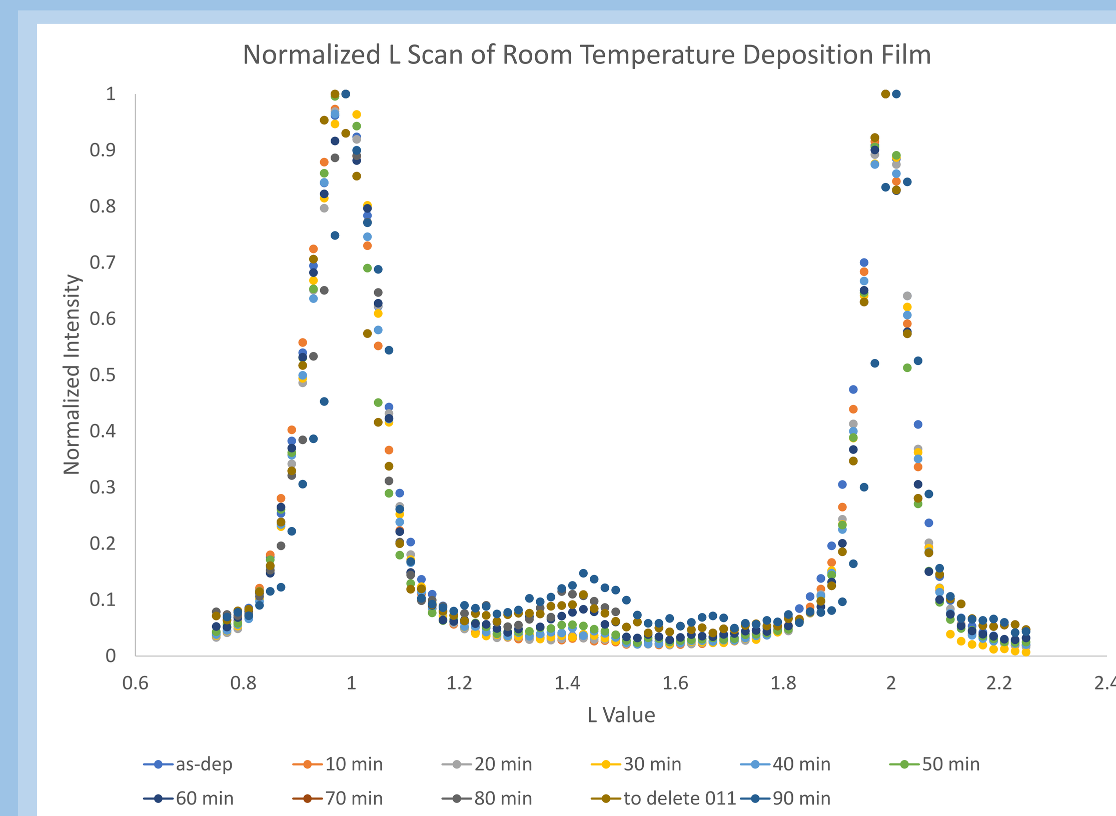

Figure 6. Normalized plot of intensity of diffracted x-rays vs L-values after every ten minutes of annealing for sample deposited at room temperature. FHWM decreases as total anneal time increases, showing that stacking fault density decreases as the sample is annealed.

As-deposited symmetric theta-2theta scans of samples deposited at 80°C, 130°C, and 180°C showed an initial (111) texture as well. L-scans of these films also showed a decreasing density of stacking faults with increasing deposition temperature (Figure 7). The symmetric theta-2theta scans of samples deposited at 130°C and 180°C showed no significant texture transformation after a 2.5 hour anneal time.

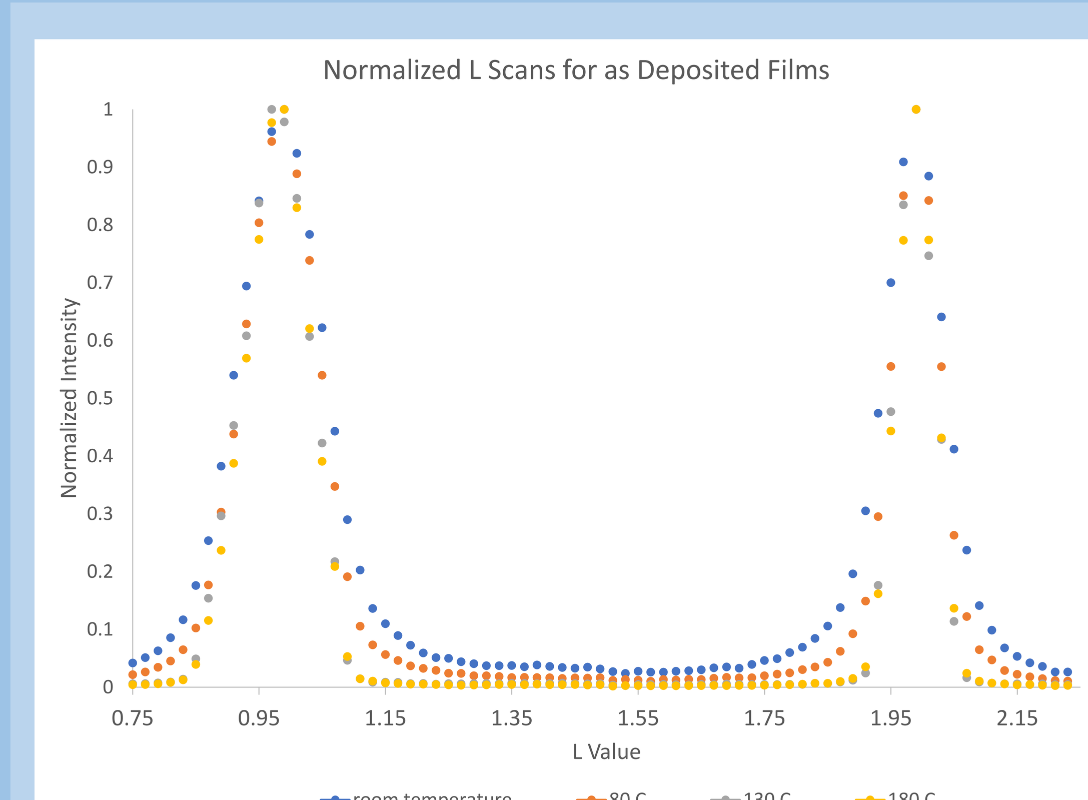

Figure 7. Normalized plot of intensity at each L value for as-deposited films shows peaks for films deposited at different temperatures. FHWM decreases as deposition temperature increases. Peaks for room temperature deposition films show the largest amount of stacking faults. Fault density decreases in films deposited at higher temperatures.

V. Conclusion These results show that thin silver films deposited at higher temperatures exhibit fewer stacking faults in their as deposited states and do not transform as thoroughly to a (100) texture. It is likely that stacking faults are a driving force for texture transformation as: films with higher stacking fault densities transformed more than films with lower stacking fault densities; and fault density was observed to decrease as the texture transformed from (111) oriented grains to (100) oriented grains. This is consistent with the preferential removal of stacking faults in (111) grains leading to the growth of (100) grains.

VI. References

[1] J. Yuly, K. Flemington, P. Lashomb, B. Hoffman, (Unpublished).
[2] E.A. Ellis, M. Chmielus, M. Lin, H. Joress, K. Visser, A. Woll, R.P. Vinci, W.L. Brown, S.P. Baker, Acta Mater. **105**, 495 (2016).