

## Abstract

Texture transformations in Cu were characterized via XRD. Previous studies have shown that minimization of nanotwin boundaries provides the driving force for texture transformations in Ag thin films. Due to the larger stacking fault energy of Cu, it was expected that Cu films would have fewer nanotwins and, therefore, less transformation. Cu was deposited onto five silicon substrates via e-beam physical vapor deposition. Samples were annealed at either 200°C or 400°C for 2-6 hours. Texture was characterized via XRD before and after annealing. Results show no transformation in any of the samples studied.

## Previous Studies

### Abnormal Grain Growth

Previous unpublished studies on texture transformation performed by Houghton College and the Cornell Center for Materials Research show that, during the deposition process, the film develops a strong (111) texture. Once the sample is annealed, the texture transforms into a strong (100) texture. Figure 1 shows a transformation in a Ag film, where texture is defined by the XRD fractional intensity  $I_{(111)}/[I_{(111)}+I_{(100)}]$ . It was also found that the grains that grew during the annealing process were much larger than the film thickness (Figure 2). These characteristics are consistent with abnormal grain growth, which is growth due to an orientation-specific driving force.

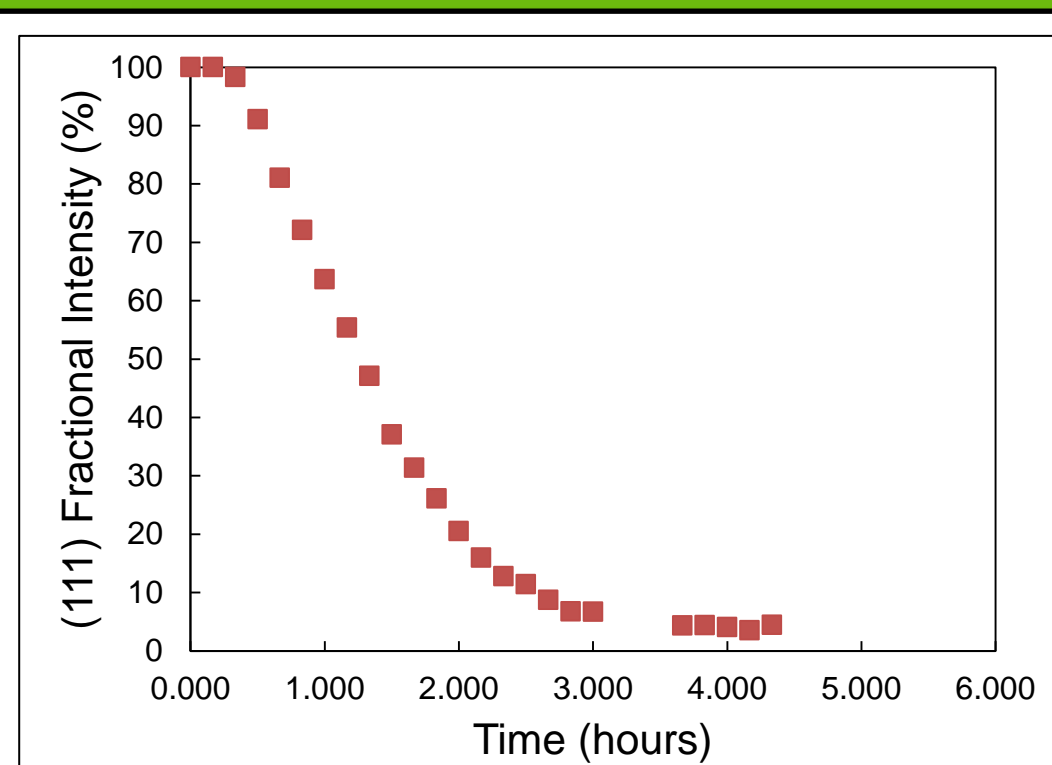


Figure 1. 1800nm Samples Deposited at 40 Å/s; Annealed at 100 C. A graph comparing the fraction of (111)/(100) and the time the sample was annealed. This is based on the XRD scan after each annealing process.

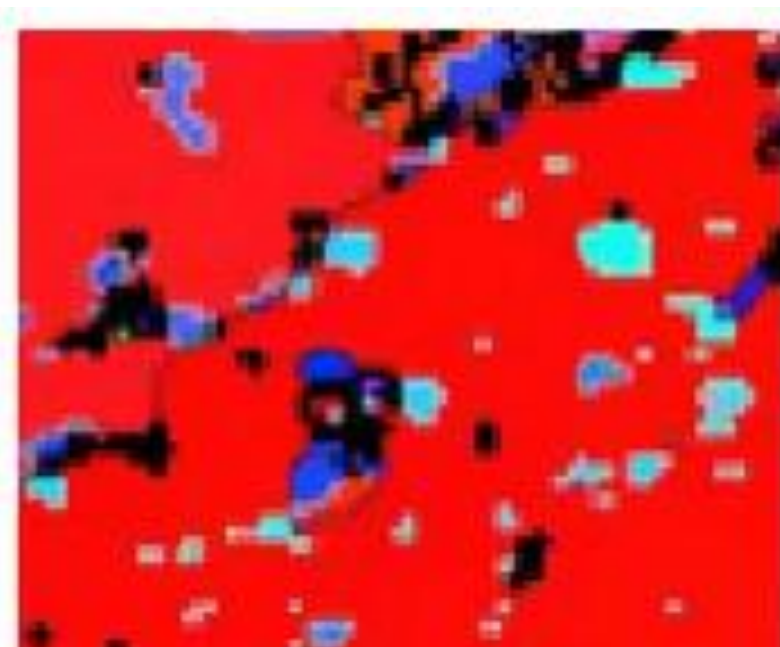


Figure 2. The EBSD Picture of a sample after the annealing process. The red are the grains in the (100) orientation. The grains are large.

### Nanotwins

During the deposition process, atoms land on and stick to the substrate. These atoms form layers and are formed based on what would produce the lowest energy state of the sample. For Ag, the lowest energy state occurs when the atoms form hexagonal

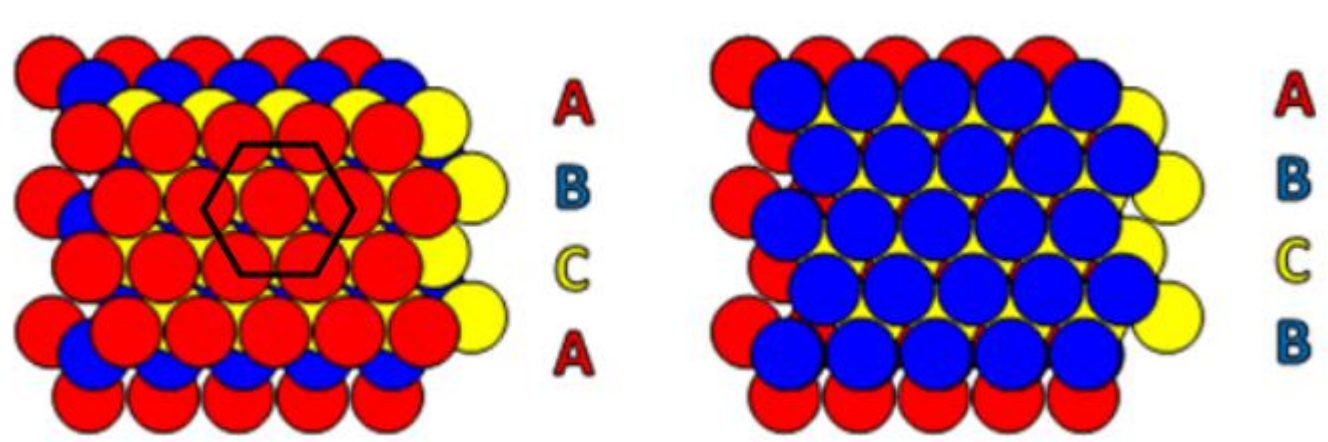


Figure 3. The layers of atoms that are formed and aligned. Each color represents an alignment of the layer of atoms. (A) The hexagonal shape that the atoms form that allows the layer to be formed. (B) The B alignment formed after C which creates a nanotwin layer.

layers as shown in Figure 3a (black lines). The next layer, B in Figure 3, is positioned over the crevasses formed by the previous two layers. As shown in Figure 3a, the lowest energy state the fourth layer would be in the same alignment as the first layer.

Sometimes, the fourth layer will be formed in a slightly higher state that

aligns with the second layer (Figure 3b). When this happens, it forms a nanotwin layer. The energy difference between the two choices of layers is called the stacking fault energy.

### Driving Force

The overall energy state of the grain with nanotwins is higher than the one without nanotwins. When the sample is annealed, the atoms move around in such way as to reduce the overall energy state. Reducing the amount of nanotwins will reduce the energy

state. Therefore, the grains will grow in order to reduce the nanotwins. The nanotwin layers of (100) grains are all slanted, whereas (111) can form horizontal nanotwins (Figure 4). As the (100) grows, the high angled nanotwins hit the top or bottom and will stop growing with the grain. That is, any

further growth will be free of nanotwins. By growing the (100) grains, the energy state of the sample reduced. Therefore, reduction of nanotwins is the driving force for the abnormal grain growth of (100) grains and the resulting texture transformation.

### Motivation

Greater stacking fault energy results in a lower probability of nanotwin formation during deposition. This means that, with same thickness, the sample with higher stacking fault energy will have less nanotwins formed than the sample with lower stacking fault energy. Studying the effect of stacking fault energy on the texture transformation and being able to create a model for the relationship is the next step. The previous studies shown are on Ag samples. Cu has a higher stacking fault energy than Ag and is, therefore, the starting point to understanding that relationship.

## Procedure

### General Setup

A Si wafer was used as a substrate in the deposition chamber. A layer of Ta was added to create a diffusion barrier between Cu and Si. Once that was completed, a linear shutter was positioned over the Ta film. Cu was then deposited while the shutter was slowly removed. This resulted in a thickness gradient over the substrate (Figure 5). Once the whole process was completed, the sample was cut into rows and columns. Each row is the same thickness and each column is basically identical to the others. Initial XRD scans were done on one column of the samples. The samples were then annealed in a tube furnace using a forming gas of 95% N<sub>2</sub> and 5% H<sub>2</sub>. The samples were removed from the furnace after it cooled down. The samples were scanned via XRD again for the final texture.

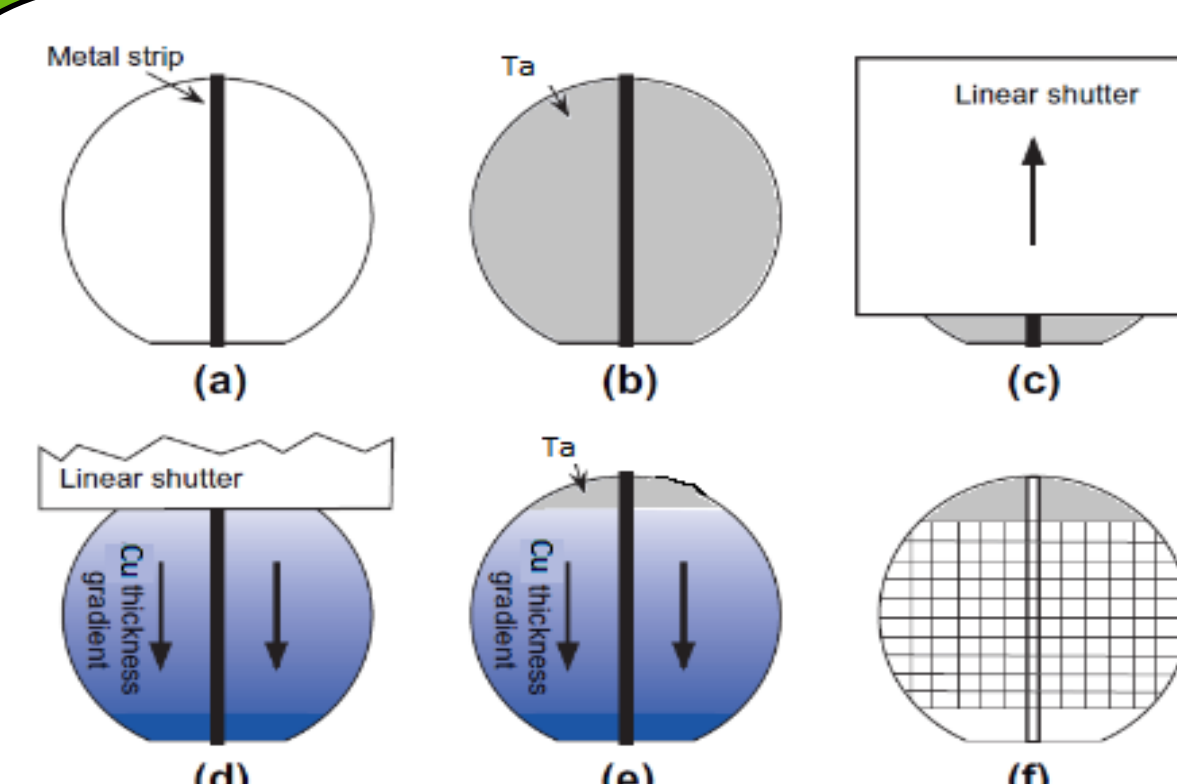


Figure 5. The deposition process for creating the samples. (a) shows the metal strip added to act as a thickness reference. A layer of Ta was added (b). The linear shutter covers the sample (c) and slowly uncovered the sample (d). This resulted in a thickness gradient (e). The sample was cut into rows and columns (f). Fig. taken from Ref [1].

### The Five Samples

Five samples were created with different deposition rates, thickness ranges, annealing times, and annealing temperatures. Some samples did not have a Ta layer. Table 1 shows the experimental parameters of all five samples.

Table 1. Comparing the differences in procedure process for each of the five samples. Each of the samples were processed similarly.

Sample #	Deposition Rate (Å/s)	Thickness Range (nm)	Annealing Time (hrs)	Annealing Temperature (°C)	Ta Layer (nm)
1	40	252 to 1399	2	200	None
2	100	487 to 1364	2	400	10
3	200	742 to 1658	2	400	10
4	200	610 to 1940	2	400	10
5	200	3860 to 4940	6	200	10

### X-Ray Diffractometer (XRD)

The XRD uses Bragg's law to determine the texture of the sample (Figure 6). Using Bragg's law, the distance between the layers can be determined by the angle of constructive interference. There is a constructive interference between two layers if  $2d\sin\theta=n\lambda$ , where the  $\lambda$

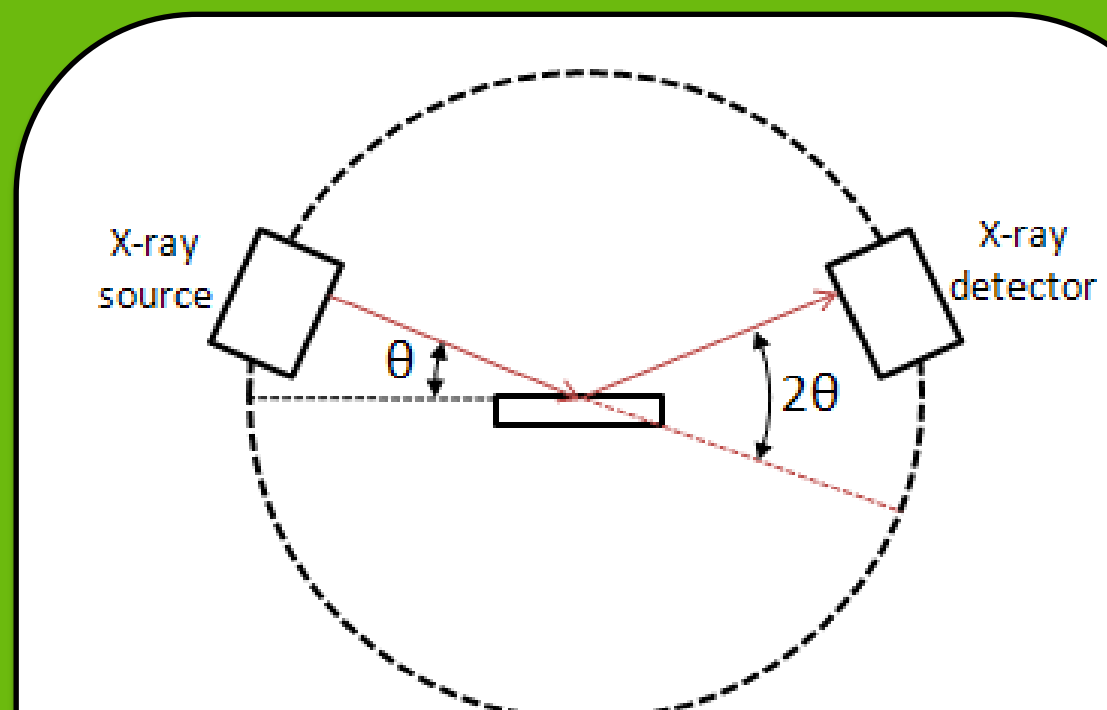


Figure 6. The set up idea of the XRD. The red lines are the x-ray travel path. The angles ( $\theta$ ) are used in Bragg's law. It is used to determine the orientation fraction.

is the wavelength of the X-ray source,  $\theta$  is the angle,  $d$  is the distance between layers, and  $n$  is any integer. Each orientation has a certain distance between layers. The crystal lattice length is known for Ag and Cu. Using that distance, the distance between atomic layers for any orientation can be calculated. Using Bragg's law, the angle of constructive interference can be determined. The fraction of (111) is defined as area of the (111) peak divided by the combined area of the (111) and (100) peaks.

## Results

The initial and final texture of each samples were determined by XRD analysis. Figure 7 shows the plot of fraction of (111) vs. thickness for the first 4 samples (Sample 5 was a much

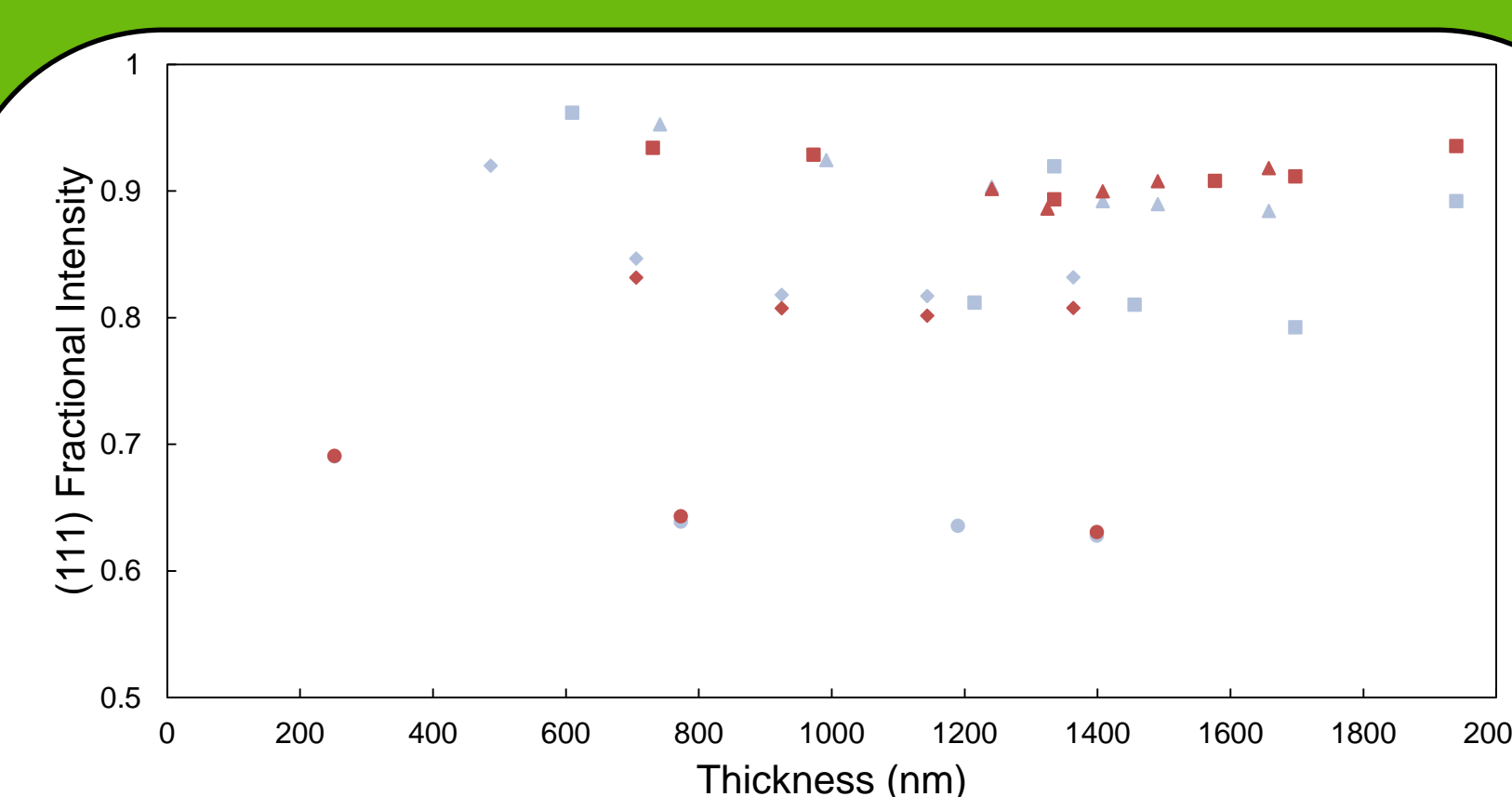


Figure 7. Plot of fraction of (111) vs. thickness for each of the first four samples. The blue dots are the XRD initial scans. The orange dots are final scans done after the annealing process. Round markers are from Sample 1, diamonds are from Sample 2, triangles are from Sample 3, and squares are from Sample 4. The (111) fractional intensity is defined as the ratio of integrated area intensities of XRD peaks:  $I_{(111)}/[I_{(111)}+I_{(100)}]$ .

higher thickness). Unlike the strong as-deposited texture of Ag samples in the past, this plot shows a thickness dependence for the initial texture of Cu. This may be due to undesired heated of the substrate during deposition. For all the samples, the final texture was within 5% of the initial scan. This indicates that there was no transformation in any of the samples. It is presumed that stacking fault energy had an effect on the texture transformation.

### Future Studies

Based on what was observed in Ag and Cu, there are several steps that are needed to understand more about the stacking fault effect.

1. Study other materials that have a stacking fault energy between those of Cu and Ag. This would allow a range of stacking fault energy to be compared to the texture transformation.
2. Use other equipment such as TEM and EBSD to observe nanotwin density and grain size.
3. Develop a model for the production of nanotwins during deposition.

### References

1. Baker, Shefford P., Brandon Hoffman, Lindsay Timian, Adam Sivernail, and Elizabeth A. Ellis. "Texture transformations in Ag thin films." *Acta Materialia* 61.19 (2013): 7121-132.