Effect of Deposition Rate & Temperature on the Microstructure of Copper Films

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Motivation: Mechanical testing of thin films offers the possibility of exploring phenomena that are difficult or impossible to investigate using bulk materials. Controlling microstructure and surface morphology is critical for creating specimens suitable for testing. It is known that film deposition rate, temperature, and underlying substrate all influence film structure.

Goal: Determine the range of possible microstructures and surface morphologies achievable with sputter deposition of copper . In particular, it is desirable to create smooth films consisting of either large grains (10's of microns) or that are single crystals.

Method: 1 µm thick copper films were grown by sputtering onto a freshly cleaved NaCl substrates. The substrates were single crystals with the [100] direction normal to the surface. Some surfaces were used in the as-cleaved condition while others were polished with water. The copper morphology was evaluated using a scanning electron microscope (SEM). Film microstructure was determined using electron backscatter diffraction (EBSD).

Sputter Coating: Sputter coating is a method of physical vapor deposition in which a 'target' of source material is bombarded by ions, knocking atoms off the target. The ejected atoms land on and adhere to the substrate, growing a film.

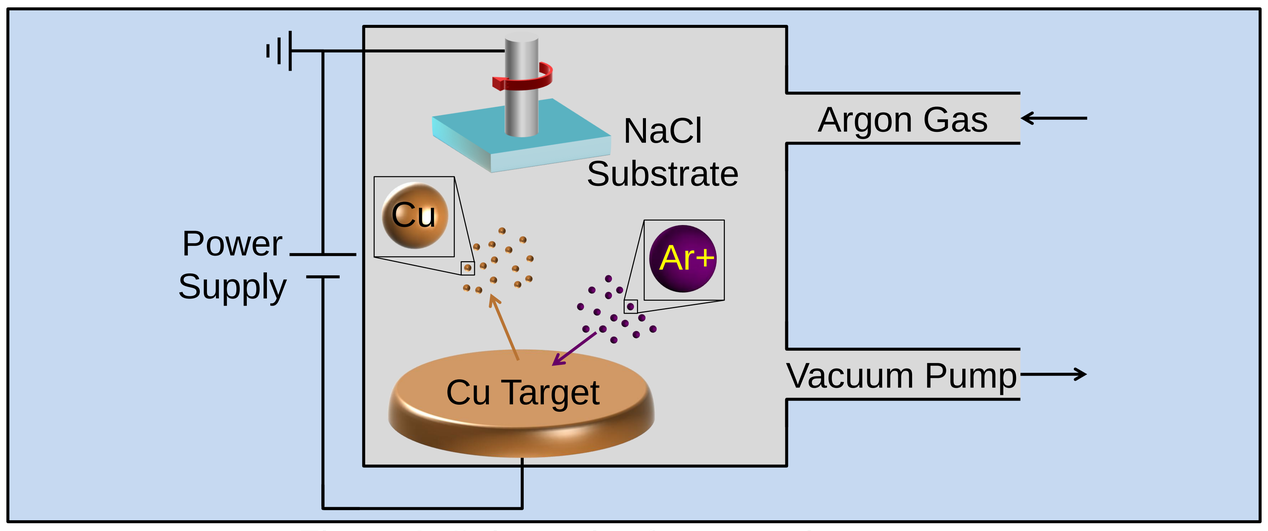

Figure 1: Schematic of a sputtering system

Deposition rate of the source material varies approximately linearly with the wattage applied, allowing thickness to by controlled through power and process time. A quartz lamp allows the substrate to be heated, increasing the surface diffusion.

Electron Backscatter Diffraction: Electron Backscatter Diffraction (EBSD) is a technique used to index the orientation of a crystalline material. When electrons strike a specimen they are either elastically or inelastically scattered. Elastic scattering reflects some portion of high energy electrons back out of the material. As they travel out of the sample some of these 'backscattered' electrons experience Bragg diffraction resulting in patterns of constructive and deconstructive interference. The backscatter diffraction pattern produced is a function of both the crystal structure and the material's orientation. A detector is used to capture the patterns. If the structure is known, the orientation can be indexed automatically. By rastering across the sample and indexing the resulting patterns, the orientation of each grain can be determined.

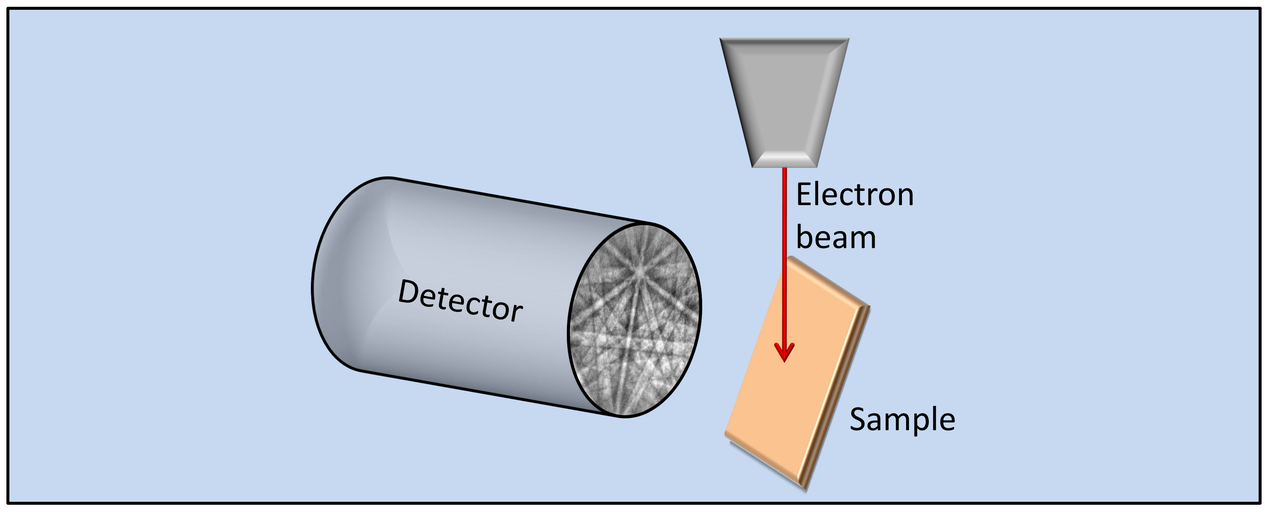

Figure 2: Schematic of an EBSD system

Results: Increasing temperature at a constant power of 150 W increases grain size from ~10-20 nm at 300ºK to ~1 µm at 700ºK. Grain size is constant above ½ the homologous temperature. Higher temperatures increase surface mobility of deposited atoms providing them a greater opportunity for diffusion to a low energy site. Increasing power resulted in the growth of a single crystal whose orientation is matched to the NaCl substrate. Higher powers impart greater kinetic energy to the film, increase deposition rate, and may result in a 'peening' effect, eroding protruding features and densifying the film.

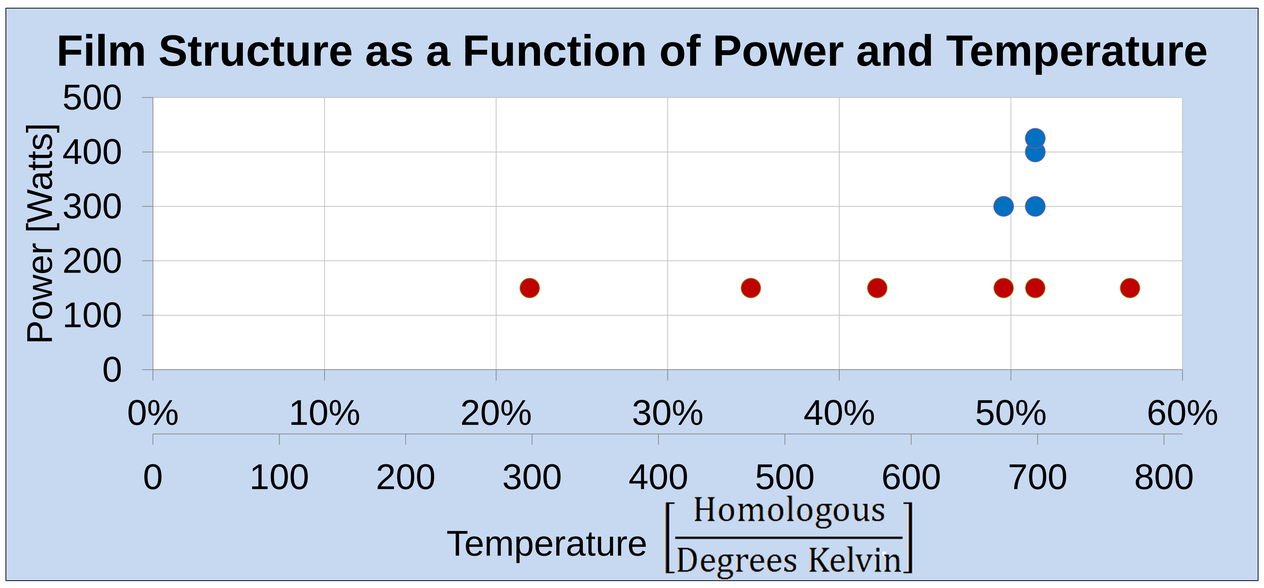

*Figure 3: Effect of power and temperature on microstructure. **Red** points are polycrystalline and **blue** are single crystals*

Surface Treatment: Water polishing radically changes the morphology, generally resulting in a rougher surface with smaller features. Water polishing introduces chlorine ion interstitials which serve as copper nucleation sites.

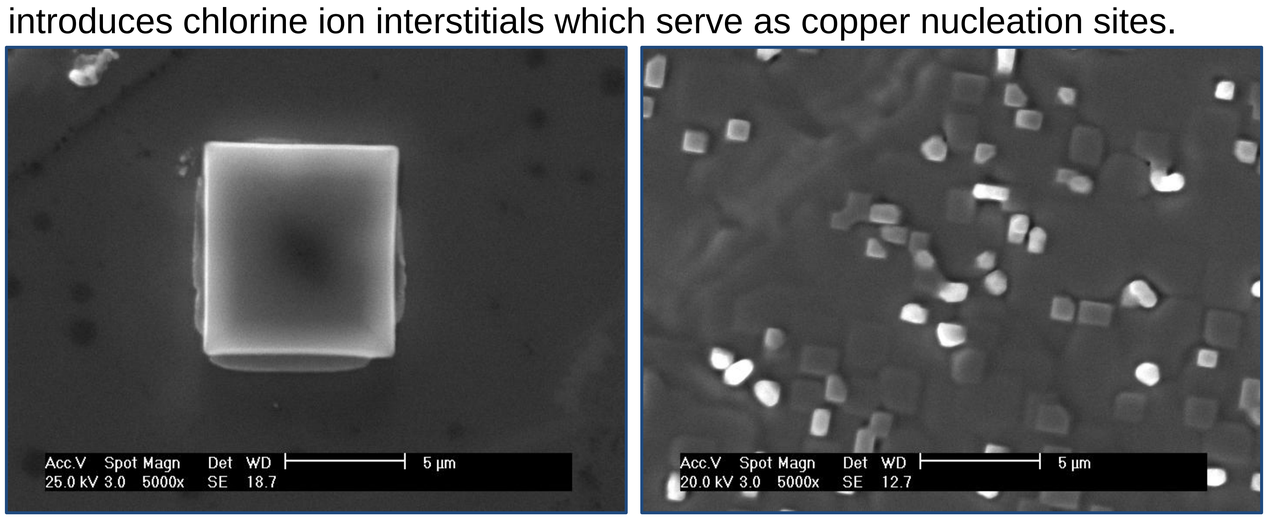

Figure 4: Cu films grown at 700ºK and 300 W. The left film is on 'as cleaved' NaCl while the right film is on 'water polished' NaCl

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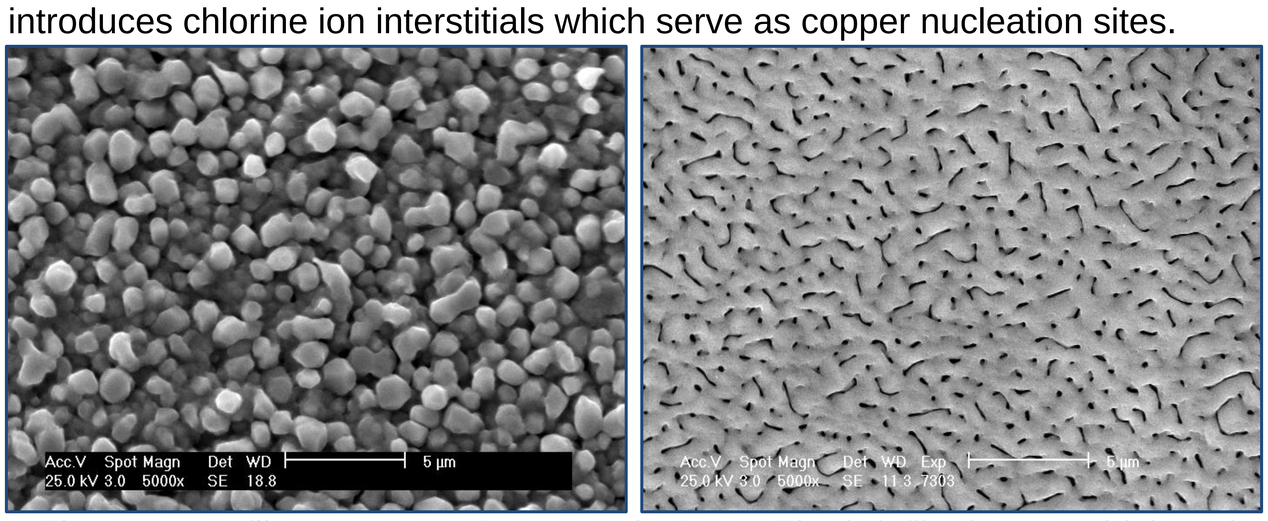

Figure 4: Cu films grown at 700ºK and 150 W. The left film is 1 µm thick and the film on the right film is ½ µm thick.

Conclusions:

- Increasing temperature increases grain size of polycrystalline films
- Increasing power causes a transition from polycrystalline to single crystal films
- Water polishing changes surface morphology but does not influence whether the film will be a single crystal or polycrystalline

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