TEM Specimens Preparation Techniques

There are a lot of TEM sample preparation techniques, which have to be selected based on the demands of the particular investigation and the material. For in situ TEM experiments, Nano-Chip design enables researches to prepare TEM samples directly on the Nano-Chip by various methods, such as drop casting, evaporation / sputtering and FIB.

Drop casting method

Drop casting – dropping a dilute solution onto a substrate with subsequent drying or annealing – is particular easy but may produce artefacts through drying process.

Film thickness depends on the volume of dispersion used and the particle concentration, both of which can be easily varied. There are also other variables that affect the film structure such as how well the solvent wets the substrate, evaporation rate, capillary forces associated with drying, etc.

Generally, it is desirable to use solvents that are volatile, wet the substrate, and are not susceptible to thin film instabilities (de-wetting). Water tends to be a poor solvent for drop casting due to the low vapor pressure and large surface tension. In some cases, alcohols can be in place of water, while organic solvents (such as hexane, toluene or halogenated solvents) are often very good choices for nanoparticles with hydrophobic capping ligands.

One drawback of drop casting is that even under near ideal conditions, differences in evaporation rates across the substrate or concentration fluctuations can lead to variations in film thickness or internal structure. However, drop casting does serve a quick and accessible method to generating thin films on relatively small substrates.

Stepwise explains of the drop casting method (*Figure 1*):

- *Prepare an aqueous solutions of the nanoparticles.*
- *Place the Nano-Chip upside down in order to protect the contact pads.*
- *Drop cast solution onto the Nano-Chip.*
- *Wait till evaporation occurs.*
- *Insert Nano-Chip into the DENSsolutions holder.*
- *Perform the experiments.*

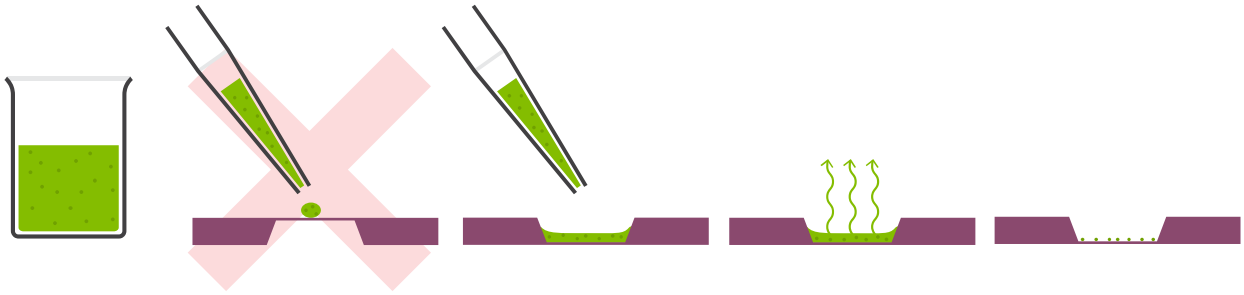

Figure 1. Drop casting method.

Tips and tricks:

Plasma cleaning a Nano-Chip before drop casting.

The Nano-Chips are covered with the Silicon Nitride, which in nature is hydrophilic but gets hydrphobic due to the hydrocarbons present in the environment (in the air, in the Nano-Chip boxes, etc), which physisorb on the surface. Therefore, removing such hydrocarbons with a plasma cleaning will greatly help to prepare a TEM specimen by drop casting method.

Sputtering method

Terminology: The source material is called the *target* and the emitted atoms or molecules are said to be *sputtered* off.

Sputtering – process whereby atoms are ejected from a target and deposited on a substrate as a result of the bombardment of the target by high energy particles.

Stepwise explains of the sputtering method (*Figure 2*):

- *Place target material and the Nano-Chip in a vacuum chamber of sputter coater (in order to protect the contact pads of the Nano-Chip, it should be placed upside down).*
- *Set the parameters of the sputter coater (current, voltage, preferable film thickness etc.) in a range, which you normally use.*
- *Apply the voltage, so the target is the cathode and the Nano-Chip is attached to the anode.*
- *A plasma is created by ionizing gas (usually chemically inert, heavy gas as Ar).*
- *Deposit target material to the Nano-Chip surface by means of bombarding the target with the sputtering gas.*
- *After finishing, take out Nano-Chip from the sputter coater.*
- *Insert Nano-Chip into the DENSsolutions holder.*
- *Perform the experiments.*

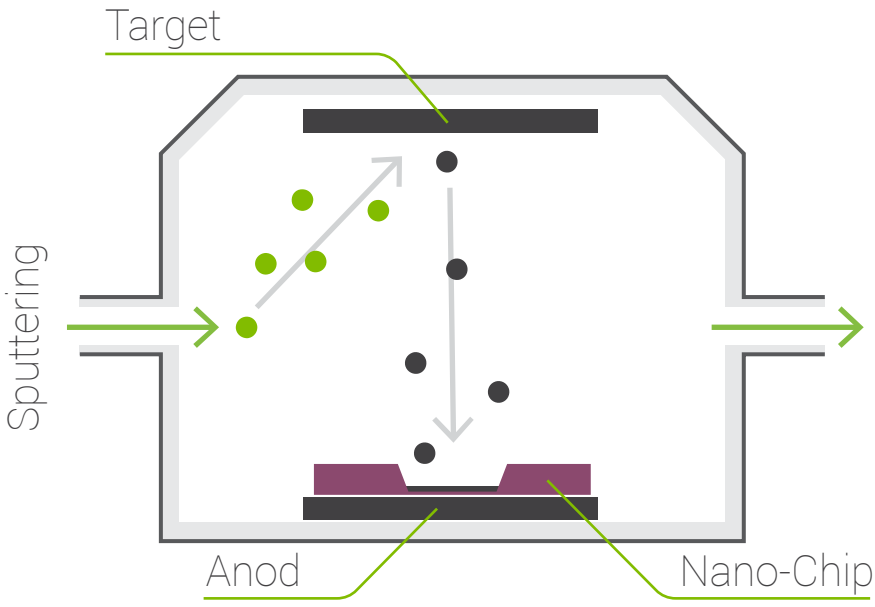

Figure 2. Sputtering method.

Unless there are enough collisions, the plasma will quickly die. In order to have a self-sustaining plasma, each electron has to generate enough secondary emission. Since we want collisions to occur, the pressure cannot be too low. Also, since we want the electrons to gain enough energy between collisions, the pressure cannot be too high.

Film uniformity can be an issue. Alloy evaporation is very complicated and in most cases, not possible.