NL-UHV / DX3 Nanoparticle Deposition Source

Operation Manual

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Contents

Safety Precautions

Please read the following instructions carefully before installing the NL-DX3 and refer to them as needed to ensure the continued safe operation.

Symbols:

= High Voltage

= Warning

= Information / Tip

Follow all warnings and instructions marked on or supplied with the product. Failure to observe these precautions could lead to personal injury or death and/or damage to the equipment.

Health and Safety – General Information

When connected to its power supply, the sputter source is supplied with potentially lethal currents at very high voltages. The source should always be disconnected from its supply during maintenance.

The toxicity of nanoparticles has not been determined in many cases and consequently care should be taken when opening the vacuum system. Always use gloves and the use of a fine-filter mask is also highly recommended as an absolute minimum.

Please note that some materials will burn if you use an IPA wipe for cleaning. Such materials are Platinum, Hafnium and Iron. For cleaning systems which contain these materials, use lint free wipes with a few drops of DI water and immediately drop the used wipes into a bucket of water. This will render the materials safe.

Introduction

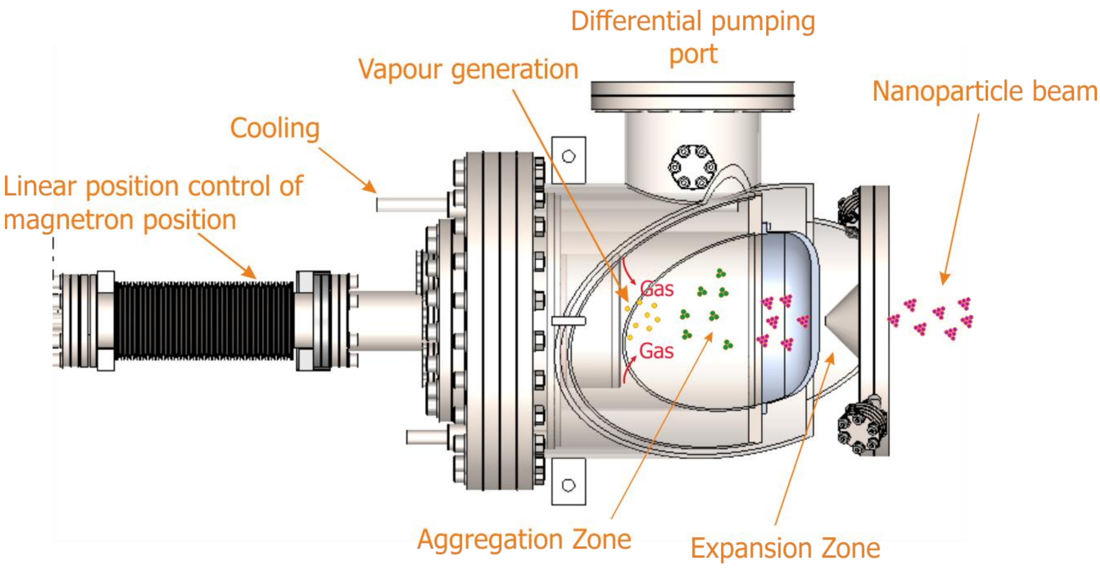
The NL-UHV nanoparticle source consists of a UHV compatible vacuum envelope which houses a range of sputter sources specifically designed to work together to generate nanoparticles of many types of materials.

The sources that are compatible with the NL-UHV include a single 1″ source (NL-D1), a single 2″ source (NL-D2), a single 3″ source (NL-D3) and a triple 1″ source (NL-DX3). This manual covers the operation of the NL-UHV in combination with the NL-DX3.

Each individual sputter source is known as and will be referred to here as a *magnetron*.

Extreme caution is required when working with the magnetron power supplies. Do not switch them on unless all leads are connected and shielded. All supplies, cables and equipment must be suitably earthed.

Nanoparticles are formed when the magnetron(s) are running and sputtering material under a range of conditions. The user can control several parameters to control the formation and size of the nanoparticles. These parameters include gas flow (usually argon) over the magnetron(s), a separate 'carrier' gas flow (usually helium), plasma power, magnetron to aperture distance, and aperture size. The magnetron(s) are typically powered by 630V, dc supplies, but in addition pulsed DC supplies can be used.



The NL-UHV produces nanoparticles by two main processes; 1) generation of atomic and molecular species by magnetron sputtering and 2) subsequent generation of nanoparticles by condensation in an aggregation zone. The NL-UHV consists of two zones, the Aggregation zone and the Expansion zone with apertures between. The Expansion zone is differentially pumped by a 300 l/s turbo pump.

Installation

For detailed information on the installation and the services required, please see the accompanying document NL-Dxx & NL- QMS Installation.

Operation

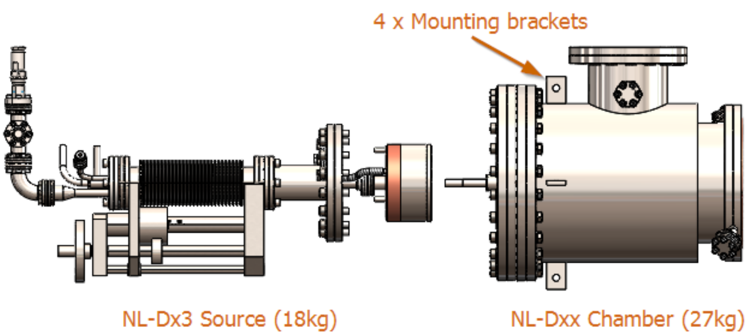
NL-DX3 Sputter Head Targets

The toxicity of nanoparticles has not been determined in many cases, consequently care should be taken when removing the NL-DX3 after use. Always use gloves. The use of a fine-filter mask is also highly recommended. Use a fume hood if you have one.

Nanoparticles can be highly reactive. When venting a chamber for the first time after extended use, rapid oxidation of the nanoparticles may lead to strong exothermic heating. Take particular care when cleaning the aggregation zone. Nanoparticle residues removed with flammable material (paper tissue for example) may heat up and spontaneously combust.

Magnetron Target Installation

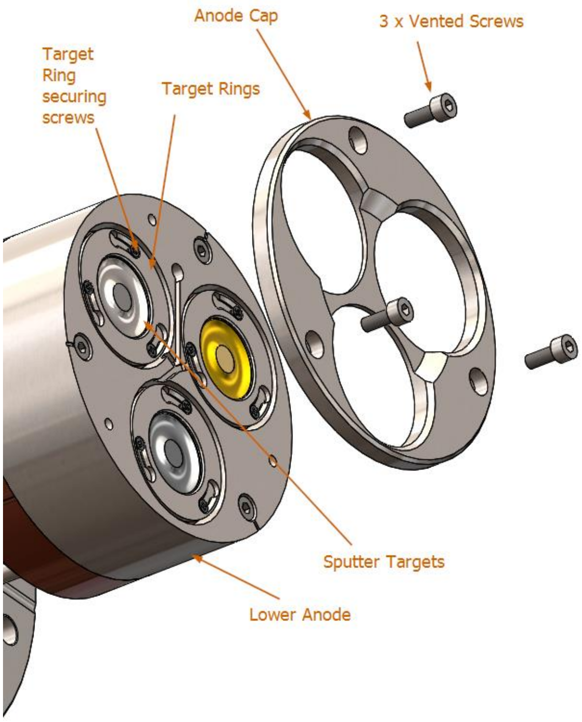
In order to fit a source target, it is necessary to remove the NL-DX3 from the NL-UHV chamber. The NL-UHV chamber can remain connected to the main system chamber if desired.



Use the following procedure:

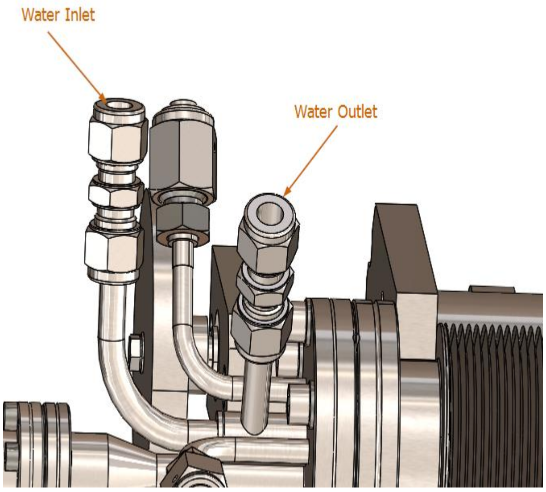
1. Isolate the magnetron power supplies.
2. Vent the vacuum system.
3. Disconnect the water-cooling connections to the NL-DX3. It is a good idea to cap the water tubes on the source to prevent spillage.
4. Disconnect the magnetron power supply cables. The source has three magnetron heads, it is important to make sure you note which wires go to which source. The source feedthroughs are etched 1, 2 and 3.
5. Disconnect the gas connections to the source, note the orientation of the two connections.
6. Remove the 16 cap head bolts connecting the source to the NL-UHV chamber.

7. Carefully remove the source from the chamber.
8. The source has 3 targets, each secured independently via its own target ring. To access the targets, it is easiest to remove the Anode Cap first. Note the orientation of the anode cap to the assembly,



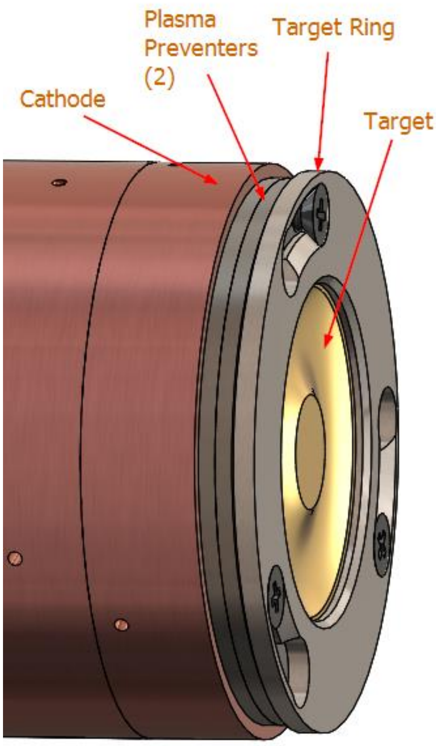
9. Loosen by about 3 turns, but don't remove the three Target Ring securing screws from the head.
10. Rotate the target ring and remove it.
11. The new target will be clamped to the top surface of the source (actually called the cathode) by the target ring.
12. Make sure that the surface of the cathode and the target is free from dust, chips and has a smooth finish.
13. When refitting / replacing the target, make sure that the mating surface of the source (known as the cathode) is clean and that there is no material on it that will prevent the new target making a good thermal contact with the cathode. Good cooling of the rear of the target face is very important.
14. Fit the target and replace the target ring, rotate the ring back into position and tighten the three securing screws **slowly and evenly** to ensure that the target is evenly clamped to the source.
15. Clean and refit the Anode cap to the assembly in the original orientation, referring to the indicators on the side of the Lower Anode and Anode Cap. The Anode Cap has been etched to identify which number magnetron pocket is which.
16. With a multimeter, check that each target is isolated from ground and the anode, and makes contact to the relevant source vacuum feedthrough.
17. Fit a new CF100 copper gasket to the NL-UHV chamber flange.

18. Re-mount the source to the NL-UHV chamber and tighten the 16 cap head bolts evenly.
19. Using a multimeter, double check that each magnetron feedthrough is isolated from ground.
20. Reconnect the gas connections to the source, using new ¼" VCR gaskets.
21. Reconnect the power connections.
22. Reconnect the water pipes in the original positions, note the connections should be as shown.



Plasma Preventers

Plasma preventers are thin rings which fit between the target ring and top of the cathode. They surround the target itself.



The purpose of the Plasma Preventers is to reduce the gap that would exist between the outside of the target and the anode. A gap in this position would cause a plasma to form in that area which could cause etching of the surrounding surfaces.

There should be a small gap between the Plasma Preventers and the target ring, so that the rings are free to "float" when the target ring is clamping the target down to the cathode.

When using a 3mm thick target, two Plasma Preventer rings should be used which reduces the gap to less than 0.5mm when the target is fitted. If thinner targets used then the number of plasma preventer can be reduced.

Start-up

Before start-up ensure that:

The Power cable(s) is (are) attached to the source.

All power supplies and the chamber are suitably earthed.

Water cooling is flowing at a minimum of 1.5 l/min to the sputter head. Lack of water cooling to the sputter head may cause overheating and damage.

It is advisable to interlock the power supplies such that if the water and / or vacuum fails, the supplies are disabled so no damage can be caused to the magnetrons.

Suitable gases are connected, the gases are pure and the gas lines have been purged.

The vacuum chamber has pumped down to a pressure of at least 1 x 10^{-5} mbar, though a pressure of 1 x 10^{-6} mbar or better is preferable. The differential turbo and backing pump should be run at the same time as the deposition chamber pump.

Striking a plasma

The magnetron plasma is started by introducing gas into the source and switching on the DC power supply.

Introduce Argon gas flow. Typically, 10 sccm of Argon is sufficient. When working with a new target, or after the system has been vented, it is useful to reduce the gas flow after a plasma has been struck to a value of 2 or 3 sccm for a period of 10 minutes to half an hour to allow the target to "clean up" and the plasma voltage to settle to a stable value. This is essential for targets that tend to oxidise quickly, for example titanium and aluminium targets.

The lower limit of gas flow required to maintain a stable plasma will be to some extent target dependent but for most target materials a value of 2 to 3 sccm should be sufficient.

Switch on the high voltage supply. Before enabling the high voltage set the maximum output voltage to 630V (following the manufacturer's instructions for the supply) and the output current to 100mA using the preview function on the front panel of the DC supply (if the supply is equipped with a preview facility).

Once the HV and current values have been set, enable the high voltage. If the target is new or the system has been vented to air there will be a period of arcing before a stable plasma is achieved. Once the plasma has been struck (as evidenced by a stable current reading) the voltage will normally trend downwards (or sometimes upwards and then downwards whilst the target surface is cleaned) before stabilising at a given current and gas flow. After the plasma has struck the power of the sputtering process can be controlled by varying the current.

Care must be taken not to sputter through the target material as this will cause damage to the sputter head. When developing a new process or working with a new target material regular inspection of the target should be made to monitor target usage. The operating voltage will drop continuously over the lifetime of a target and so can be a useful way to monitor the target usage.

Some target materials (such as ITO) are very brittle and can crack due to rapid changes in temperature. In such cases ramp the power up and down slowly to prevent rapid temperature changes. It can also be helpful to use a thin copper backing plate between the cathode and the target to reduce the thermal stress.

Nanoparticle Generation

Introduction

The operation of the NL-DX3 is straightforward and it is relatively simple to generate a nanoparticle beam. However, the formation of nanoparticles is a complex process which depends on gas flow, pressure, material properties, density and plasma conditions. As a consequence, it is difficult to recommend an absolute set of parameter values which will lead to predictable nanoparticle properties for each material type, although broad guidelines will generally hold true.

Here are some practical tips which will help the user become accustomed to the parameters which influence the formation of the nanoparticles.

The source consists of three DC magnetron heads which are enclosed within a high-pressure Aggregation zone and a differentially pumped Expansion zone. The parameters and conditions which control the formation and size of the nanoparticles are described below. Together with experimental data these should act as a starting point to the user.

Each individual target should be cleaned using the low argon flow plasma (as described above) to ensure a clean, pure surface to generate nanoparticles. This process should be repeated when switching from one magnetron head to another to remove any cross-contaminated sputtered material from the adjacent sources.

It is beneficial to be able to measure the nanoparticle flux, or deposition rate produced by the NL-UHV. The most practical way to do this is to use a quartz crystal monitor (QCM) as is common practice for sputter deposition. It is ideal to place the QCM at a distance of around 150mm from the exit aperture. Copper is an ideal material to use to characterise the source. The NL-DX3 should be able to produce a measured deposition rate of 0.1 - 1 Angstrom per second dependent upon the target material, distance and position in relation to the particle beam.

It is important to note that below a critical flow value of argon there may be no nanoparticle flux generated!

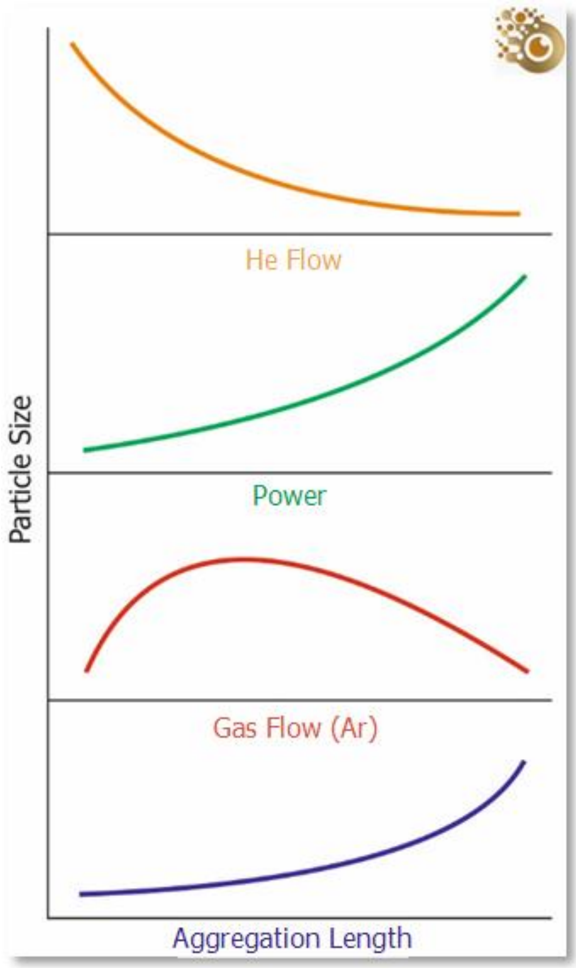
Once a deposition rate has been measured by the QCM it is useful to change some of the parameters to see how they affect the deposition rate. If a Nikalyte NL-QMS has been placed in line then the nanoparticle size distribution may also be measured and selected.

Note that the beam from the front of the NL-QMS is collimated at around 40mm diameter, so the QMS should be located in the beam during measurement.

Nanoparticle size variation

A number of parameters can be varied to alter the size of the nanoparticles. In general, to achieve large shifts in mean nanoparticle size, two or more parameters should be varied together. As a general rule, for very small particles, use *low* power, *low* Ar flow, *high* He flow and *high* aggregation length.

However, the effect of these parameters on the nanoparticle size distribution depends very strongly on the nucleation temperature (the optimum temperature for nanoparticle formation) of the material.



This figure shows the approximate relationship between the various parameters and particle size.

Ar is the discharge gas. Increasing Ar pressure allows more rapid thermalisation and hence more rapid particle formation but will also sweep the particles through the aggregation zone more rapidly thus reducing the time for particle growth.

He is a carrier gas which sweeps the particles out of the aggregation zone.

Power changes the density of the target vapour and hence the particle size.

Aggregation length changes the time the particles spend in the aggregation zone and hence the size.

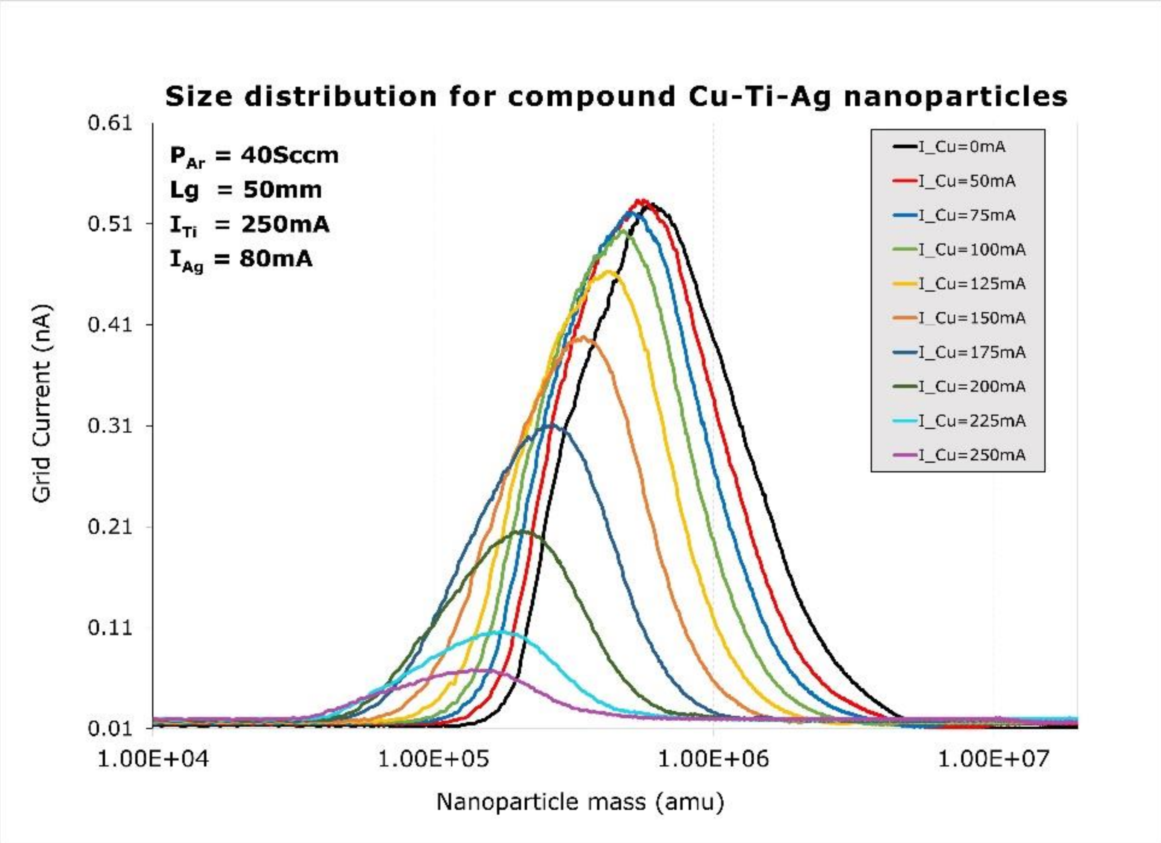
Since the NL-DX3 has three sources which can be run independently in terms of power, but share common gas supplies there will always be some interplay between the sources. In addition, some materials behave differently to others. This can cause particle size to not follow these trends. You are encouraged to explore a wide parameter space to find the optimum conditions for your process.

Indicative data

The NL-DX3 enables the user to sputter 3 different materials together and explore the formation of binary, ternary and hybrid nanoparticle structures. By varying the power to each magnetron, the user can explore different compound or alloy compositions.

The plot below shows typical mass distribution spectra for Ti, Ag and Cu deposited together using the NL-DX3. The data was collected using the inline NL-QMS mass filter. In the plot the gas flow (P_{Ar}), aggregation length (Lg) and the current to the Ag (I_{Ag})and Ti (I_{Ti}) magnetrons are fixed.

The data shows the change in the mass distribution as the current to the copper magnetron (I_Cu) is increased. The strongest signal is observed for currents of 50mA for copper, 80mA for silver and 250mA for titanium. The peak position is also affected by the changing currents to the individual sputter sources. Note that for the case of alloy nanoparticle deposition the QMS size distribution is plotted as mass rather than diameter, as the true mass of the nanoparticles generated is dependent on the alloy fractions and is not yet known.



The plots below show examples of nanoparticle distributions of copper for varying process parameters of Argon gas flow, magnetron current and aggregation length. The data was collected by placing the Nikalyte NL-QMS mass filter in line with the NL-DX3. In all three plots the peak position, distribution width and the peak intensity vary with gas flow, aggregation length and current. Thus, the peak size, range of sizes and the number of nanoparticles generated can be varied by changing the process parameters.

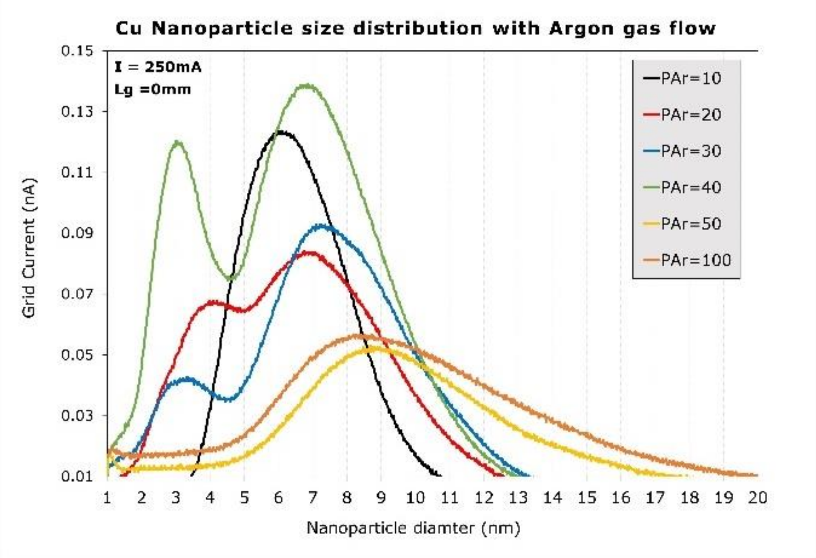
When comparing the real data with predicted trends, see section Nanoparticle size variation, it is clear that the real data follows some but not all the general trends. This is normal and reflects the complex interplay of thermodynamics, kinetics and surface chemistry that occurs inside the aggregation zone.

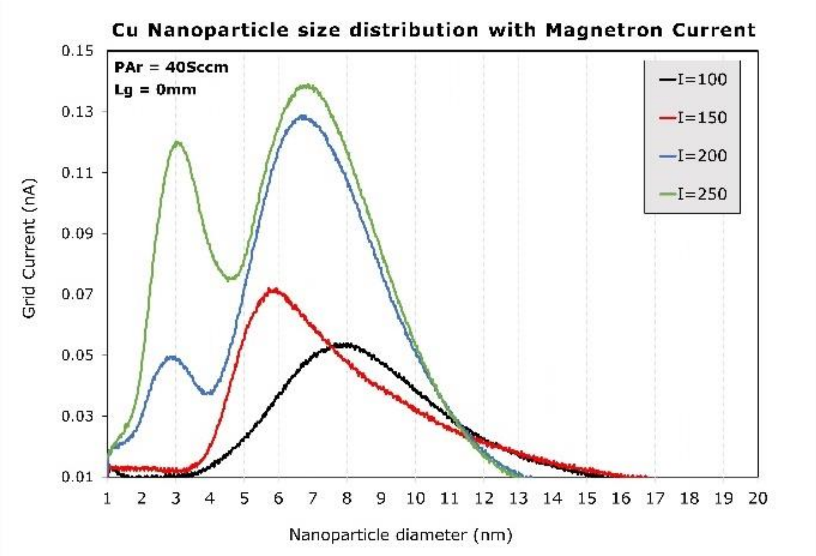
You are encouraged to explore parameter space for their specific materials and use the predicted trends as a starting point. Knowledge of the sputtering parameters of the material is also useful to note, as heavy materials such as gold and platinum with high sputter yields provide higher nanoparticle deposition rates than lower density materials such as aluminium.

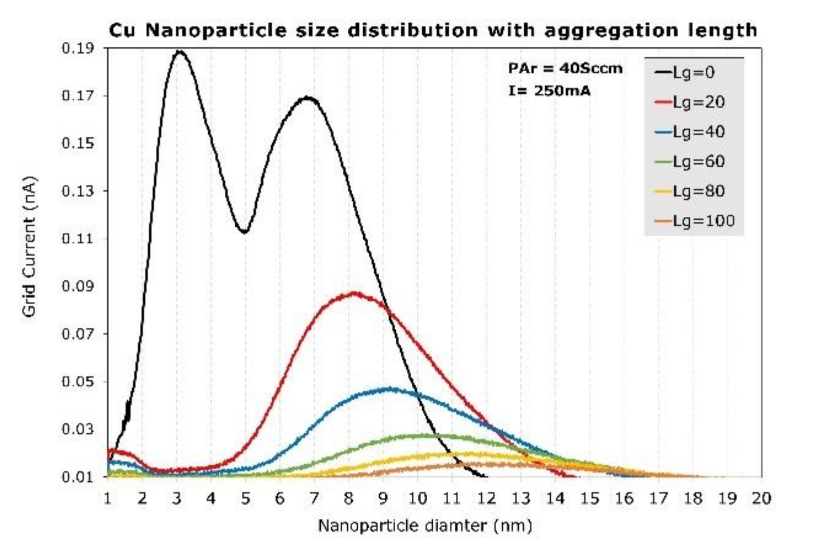
Another important parameter to understand is the nucleation temperature of the material, which is the plasma temperature at which the materials readily form nanoparticles. This temperature may be calculated, and you can also experiment with varying the plasma temperature by using different power supplies such as pulsed dc, adding helium gas or using different coolants, such as LN2 in the aggregation zone cooling jacket.

⚠ Do not use LN2 in the NL-DX3 Source, or damage to the source will result!

The NL-UHV has an additional level of flexibility which is the ability to change the diameter of the orifice in the front of the aggregation zone. This will affect the environment in the aggregation zone in terms of pressure, gas flow and dynamics, and hence nanoparticle size etc. See the section Changing the Primary Orifice. The orifice that is fitted as standard is 3mm diameter. The installation pack includes orifices of 2, 4 and 5mm diameter.


Cu Nanoparticle size distribution with Argon gas flow
I = 250mA
Lg =0mm
PAr=10
PAr=20
PAr=30
PAr=40
PAr=50
PAr=100
Grid Current (nA)
Nanoparticle diamter (nm)


Cu Nanoparticle size distribution with Magnetron Current
PAr = 40Sccm
Lg = 0mm
I=100
I=150
I=200
I=250
Grid Current (nA)
Nanoparticle diameter (nm)


Cu Nanoparticle size distribution with aggregation length
PAr = 40Sccm
I= 250mA
Lg=0
Lg=20
Lg=40
Lg=60
Lg=80
Lg=100
Grid Current (nA)
Nanoparticle diamter (nm)

Shut-down

The source is switched off by either reducing the gas flow, or the output on the DC power supply. The source may be quickly re-started by the reverse of this process. Care should be taken if you have been using a fragile target material to reduce the magnetron power slowly to avoid thermal shock which may fracture a delicate target.

Maintenance

The NL-DX3 source is designed to require little maintenance. The main areas which require periodic maintenance and inspection are the target mounting areas of the source. As the source is used, the deposited material will build up around the target, the target ring and around the surrounding anode.

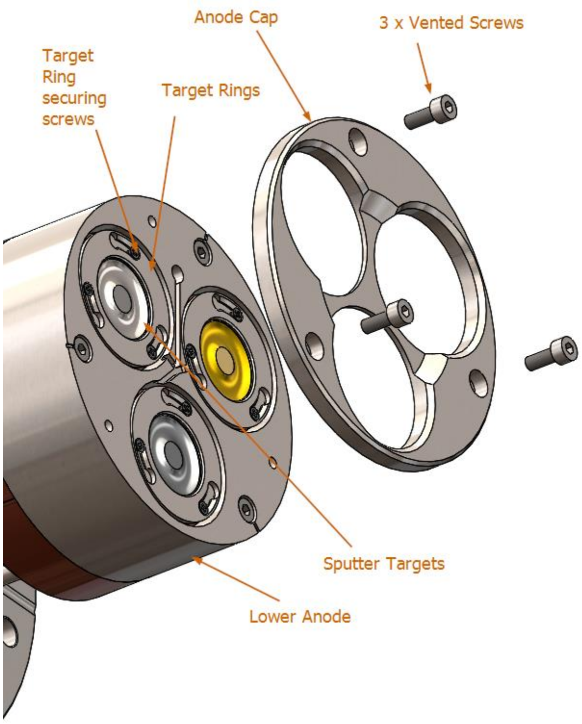
The toxicity of nanoparticles has not been determined in many cases, consequently care should be taken when removing the NL-DX3 after use. Always use gloves. The use of a fine-filter mask is also highly recommended. Use a fume hood if you have one.

Nanoparticles can be highly reactive. When venting a chamber for the first time after extended use, rapid oxidation of the nanoparticles may lead to strong exothermic heating. Take particular care when cleaning the aggregation zone. Nanoparticle residues removed with flammable material (paper tissue for example) may heat up and spontaneously combust.

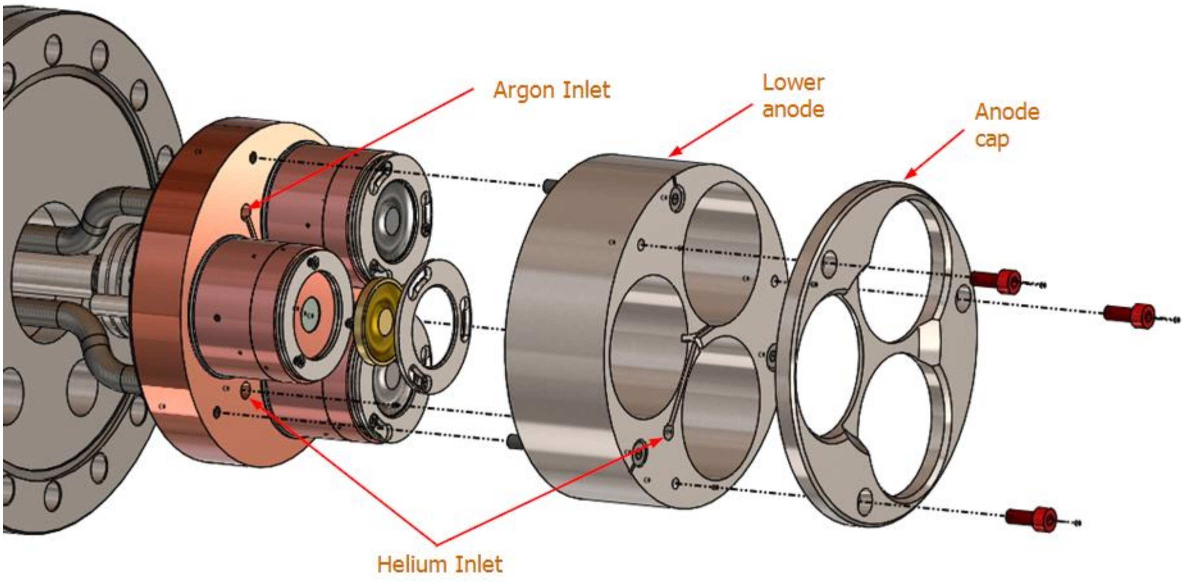
Use the following procedure to clean the source head:

1. Isolate the magnetron power supplies.
2. Vent the vacuum system.
3. Disconnect the water-cooling connections to the NL-DX3. It is a good idea to cap the water tubes on the source to prevent spillage.
4. Disconnect the magnetron power supply cables. The source has three magnetron heads, it is important to make sure you note which wires go to which source. The Source feedthroughs are etched 1, 2 and 3.

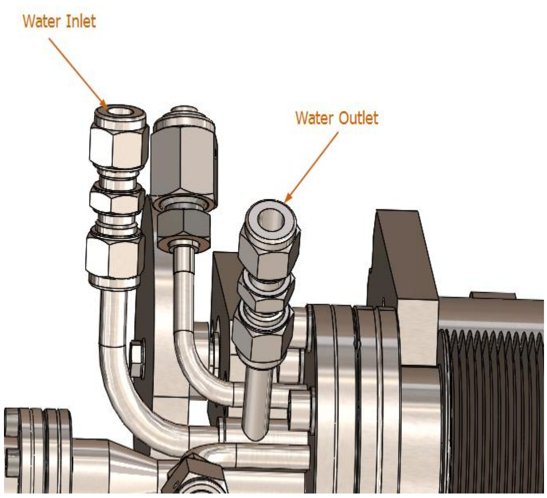
5. Disconnect the gas connections to the source, note the orientation of the two connections.
6. Remove the 16 cap head bolts connecting the source to the NL-UHV Chamber.
7. Carefully remove the source from the chamber.
8. The source has 3 targets, each secured independently via its own target ring. To access the targets, it is easiest to remove the Anode Cap first. Note the orientation of the anode cap to the assembly.
9. Loosen by about 3 turns, but don’t remove the three Target Ring securing screws from the target you are changing.
10. Rotate the target ring and remove it.



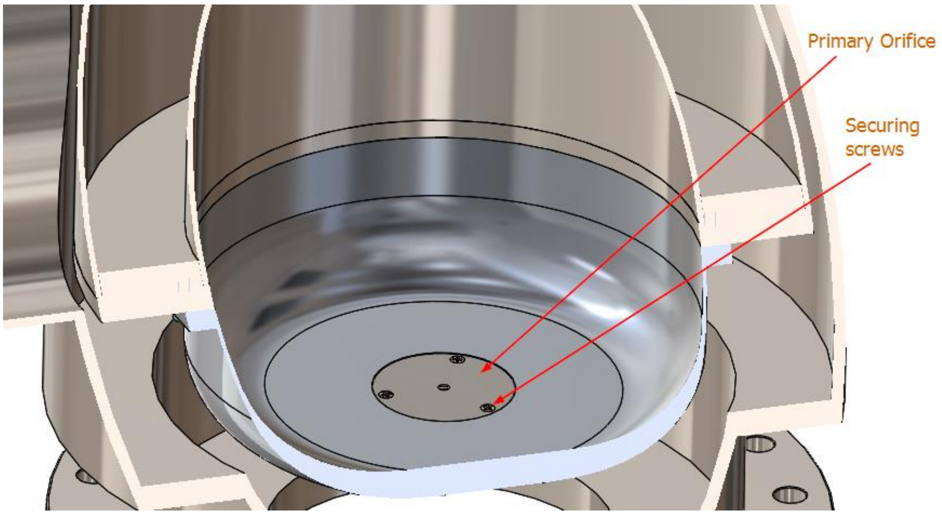
11. The target should then come free from the source below. It may need a light tap to allow it to float free.
12. When replacing the target material with a different material it is good practice to clean the target ring and the anode cap before replacement.
13. After prolonged use it will be necessary to remove and clean the Lower Anode, which is achieved by removing the three long cap head screws on the top of the lower anode.
14. It is **MOST IMPORTANT** to ensure that the lower cathode and anode cap are replaced in the original orientation, there are alignment arrows on the both parts to make this easy. Please refer to the diagram below.
15. When refitting / replacing the target, make sure that the mating surface of the source (known as the cathode) is clean and that there is no material on it that will prevent the new target making a good thermal contact with the cathode. Good cooling of the rear of the target face is very important.



16. Refit the target and replace the target ring, rotate the ring back into position and tighten the three securing screws **slowly and evenly** to ensure that the target is evenly clamped to the source.
17. Clean and refit the Anode cap to the assembly in the original orientation, referring to the indicators on the side of the Lower Anode and Anode cap. The Anode cap has been etched to identify which number magnetron pocket is which.
18. With a multimeter, check that each target is isolated from ground and makes contact to the relevant source vacuum feedthrough.
19. Fit a new CF100 copper gasket to the NL-UHV chamber flange.
20. Re-mount the source to the NL-UHV chamber and tighten the 16 cap head bolts evenly.
21. Using a multimeter, double check that each magnetron feedthrough is isolated from ground.
22. Reconnect the gas connections to the source, using new ¼" VCR gaskets.
23. Reconnect the power connections.
24. Reconnect the water pipes in the original positions, note the connections should be as shown.



Changing the Primary Orifice



To change the Primary Orifice:

1. Remove the source from the NL-UHV chamber as detailed in the section Magnetron Target Installation.
2. The Primary Orifice is then visible at the far end of the aggregation zone.
3. Clean the aggregation zone internally with reference to the warnings in the section Cleaning.
4. Using a long Pozidrive screwdriver, remove the orifice securing screws being careful not to drop the screws into the hole in the orifice or into the zone below the aggregation zone.
5. The orifice will now be removable.
6. When installing the replacement orifice, again be very careful not to drop the screws.
7. Do not overtighten the securing screws.

Cleaning

The toxicity of nanoparticles has not been determined in many cases and consequently care should be taken when removing the source after use. Always use gloves and the use of a fine-filter mask is also highly recommended.

NOTE: Some nanoparticle deposits can be highly reactive. Avoid using volatile solvents when removing the initial deposits as these can spontaneously combust.

The NL-DX3 Anode cap may require periodic cleaning from material build-up. Mostly the material will not be strongly adherent and can be removed by wiping or gentle abrasion. It is not necessary to regularly clean the inside of the aggregation unless there is a build-up of flakes obscuring the apertures or you are experiencing difficulties generating or maintaining a deposition rate of nanoparticles (see troubleshooting section). A well coated aggregation zone is often helpful for nanoparticle formation. However if you do decide to clean the aggregation zone the material inside will normally be powdered and can be readily removed with a wipe Periodic visual checks to ensure the apertures are free of flakes is advised.
Sputtered material, i.e., material that has not formed nanoparticles can be harder to remove. To remove this an abrasive pad or Dremel is often used. We do not recommend doing this unless necessary to avoid masking or if the sputtered material is causing short circuits.

Consumables

The only consumables required in the NL-DX3 range are the sputter targets. The condition of the targets should be inspected on a regular basis to avoid sputter damage to the cathode in the event of the target being fully used. Typically, a sputter target will last up to 10 hours at a sputter current of around 200mA (although this is dependent on the target material, thickness of the target etc). After a while the user will be able to determine optimum sputter rates for their particular process.

Troubleshooting

Unable to strike a plasma

Note that a fresh target may arc for a period lasting up to a minute as it cleans up. This is quite normal.

Arcing can last longer than this for very old targets which have a thick oxide. In this case it is worth persevering with cleaning at low gas flow for up to 10mins.

If the plasma still hasn't struck after this time perform the following checks: -

- With the cable disconnected and the DC supply turned off - check that the HV connection hasn't shorted. A flake of sputtered materials may have become caught between the cathode and anode.
- With the DC supply turned off - check that the HV cable is wired into the DC supply correctly. The sputter head requires a **negative** bias to operate so the core of the co-axial HV cable should be connected to the **negative** output of the DC supply.
- The gas pressure may not be high enough to strike the plasma. Some target materials require a high gas pressure to strike the plasma. After the plasma has struck the gas flow and chamber pressure may be lowered if desired.
- A higher power may be required to strike the plasma. Increase the current up to a limit of 150mA. It should not be necessary to apply more than 150mA to strike a plasma.
- The target material may be of too low a purity grade and contain too many contaminants to strike a stable plasma.
- Check the continuity of all external cables.
- The base pressure may be too high. For some materials such as Ti and Si a base pressure of <1e-6 mbar is required to strike a plasma. Other materials such as Cu will generate nanoparticles with a base pressure in e-5mbar range.
- The target may be too thick. For magnetic materials a thin target of 0.5mm is required to retain sufficient magnetic field at the target surface to enable sputtering. For magnetic materials use a backing plate to enable the target retaining ring to tighten fully.

The deposition rate is low or dropping

The nanoparticle generation process is highly repeatable but is also very sensitive to small changes in the environment. If you notice that the deposition rate is lower than you expect:

- The supply gas cylinders may be running out. A close to empty gas bottle will draw in air but will still generate a plasma. If this is the case switch to a new bottle to avoid oxidising the sputter target.

- You sputter power may be dropping due to target usage. For a constant current the dropping voltage associated with target usage will result in a natural fall in the sputter power. Adjust the current to maintain the required sputter power.
- Your QCM quartz crystal may be reaching the end of its life. Check that the QCM crystal still has a good lifetime left.
- The humidity or temperature in the room may have changed. The nanoparticle generation process is sensitive to changes in the temperature and humidity of the room. This is particularly true if you are using plastic gas tubing. Ideally place the nanoparticle chamber in a temperature and humidity-controlled environment.
- There may be a blockage to the apertures in the NL-UHV vacuum chamber. Over time nanoparticles or sputtered material flakes can block the aggregation zone aperture. Remove the NL-DX3 (as described <u>above</u>) and inspect the inside of the NL-UHV chamber. Follow the advice for cleaning the aggregation zone given <u>above</u>.
- The material may not easily form nanoparticles at or above room temperature. For some materials generating any nanoparticles is difficult at room temperature. For example, Silicon and carbon have a very low nucleation temperature. To generate nanoparticles such as these try adding helium to the aggregation zone. The use of Liquid Nitrogen cooling is highly effective for materials like these.

No Nanoparticles produced

- Carry out the check above.
- If you have just starting up after pumping down and you have previously been depositing an oxidising material, for example Ti, Zn etc., you may see a decreased rate of nanoparticle production due to the insulating oxide materials inside the aggregation zone.
- Deposit a layer of copper with the NL-DX3 fully retracted for 30 minutes at a low gas flow and low deposition rate to make the surface conduct again.

If this is a new installation, it is worth looking at the connecting gas pipework to the source. Experience has shown that using stainless steel pipework to the source can prevent heterogeneous nucleation in the aggregation zone. Always use plastic pipework for the last 2-3m.

Source Materials

The three sputter sources are designed for 1" / 25mm targets with a maximum thickness of 3mm.

⚠ Note that if magnetic material targets are used, the maximum thickness should be 0.5mm.

Contacting us

Please contact us if you need more information, help or advice installing your Instrument, through your local Nikalyte representative if you have one.

We are always happy to help, and welcome any comments you may have.