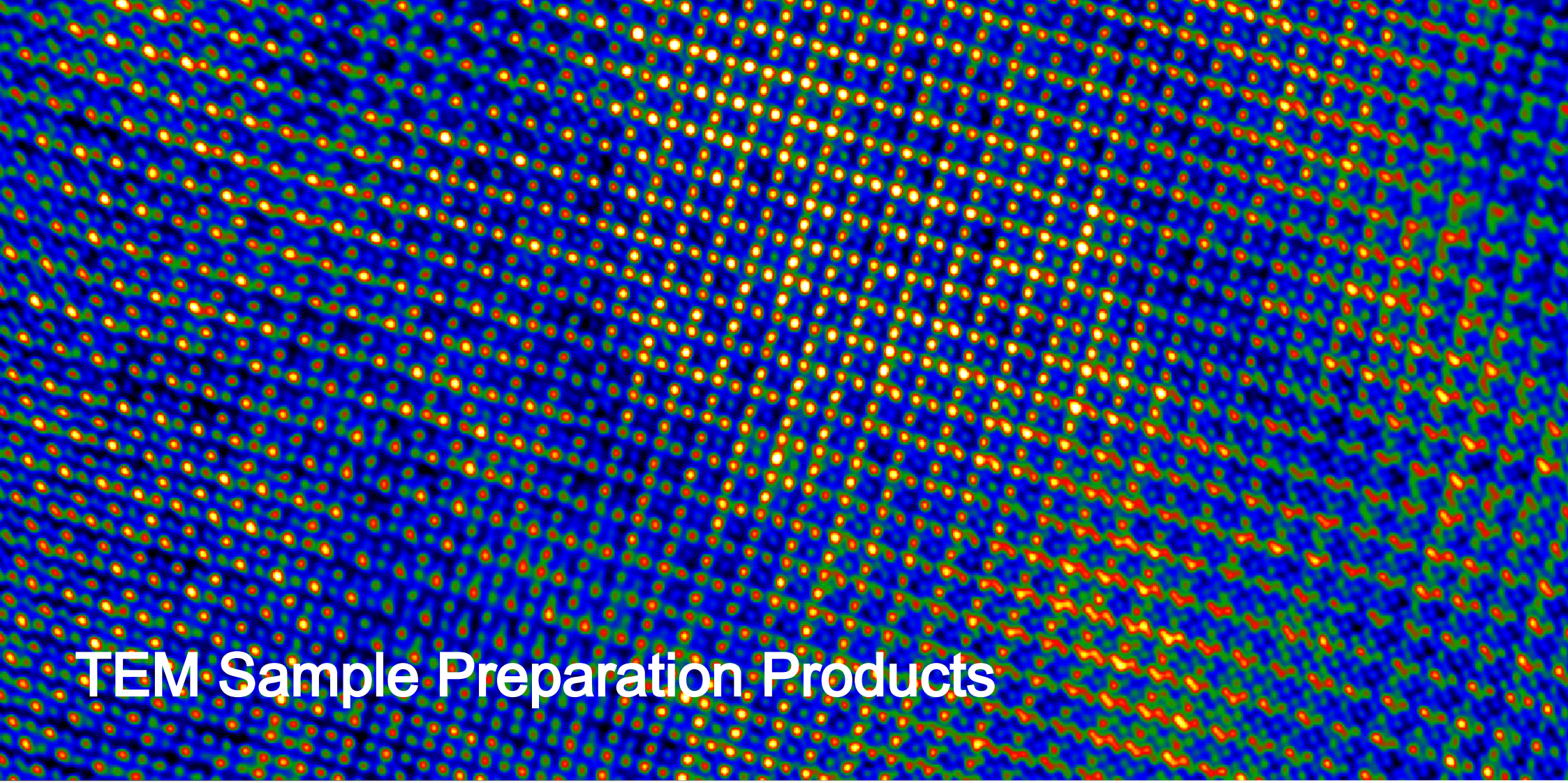

TEM Sample Preparation Products

Redesign > Pursuing Scenario BKM Solutions

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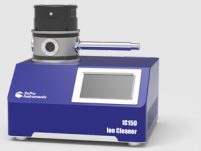
TEM Sample Preparation Faces Greater Challenges

With the rapid development of Transmission Electron Microscope (TEM) technology, its applications in fields such as materials science, nanotechnology, and biology have become increasingly widespread. However, the challenges in TEM sample preparation have also become more prominent, raising higher demands for sample preparation instruments and processes.

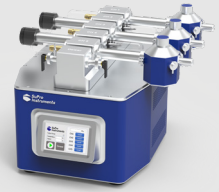
During the process of TEM sample preparation, issues like sample and support membrane rupture, deformation, and contamination are crucial. These factors directly affect the clarity of subsequent TEM imaging and the accuracy of analytical results. Balancing thorough cleaning of samples without compromising their structural integrity or the integrity of support membranes, especially ultra-thin carbon films, presents a significant challenge. Additionally, concerns related to vacuum storage of samples and hydrophilic treatment of grids also need simultaneous consideration.

To address these critical issues in TEM sample preparation, we have developed a series of intelligent and convenient sample preparation instruments. Our goal is to provide reliable Best Known Method (BKM) solutions that assist you in achieving outstanding results in scientific research and engineering applications.

Plasma Cleaning & Hydrophiling



Storage of TEM Hoders

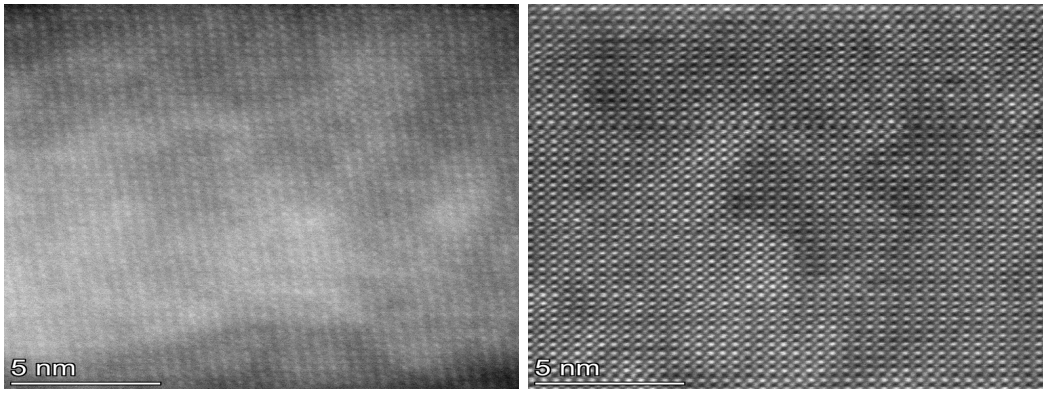


FE-TEM Scenario

TEM Samples & Holders Cleaning

Exposure of TEM holders or samples to air or improper storage can lead to the generating of organic contamination. If introduced into the TEM chamber, this contamination not only affects image quality but also pollutes the TEM chamber.

The IC150 Plasma Cleaner utilizes an RF radio-frequency power supply to ionize incoming air. The resulting oxygen radicals, ozone, and oxygen plasma oxidize organic contaminants on the sample's surface through chemical reactions. This process generates gases like CO_2 and H_2O, which are then pumped out by the vacuum system, effectively achieving the goal of cleaning organic contamination from sample holders or sample surfaces.



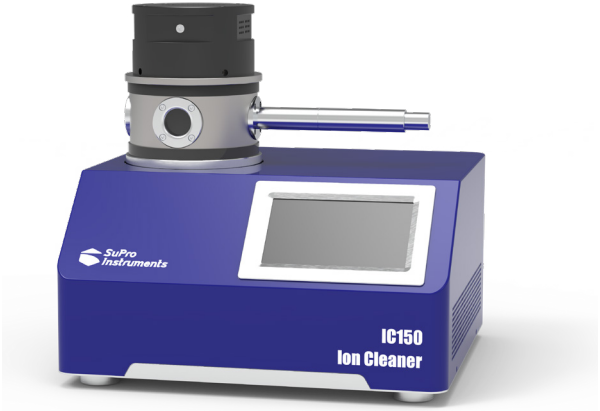
Beofre cleaning　　After cleaning

The $BaFeO_3/SrTiO_3$ sample exhibited blurry imaging due to carbon buildup before being cleaned with IC150. After cleaning, the carbon accumulation was eliminated, leading to improved imaging resolution and contrast.

TEM Grids & Samples Hydrophiling

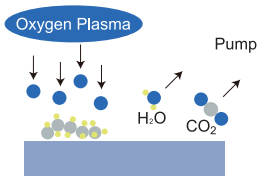
Plasma generated by IC150 will interact with the sample surface, which can increase the surface energy of the sample and change the surface from hydrophobic to hydrophilic.

Plasma Cleaner IC150

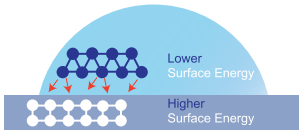


★ TEM samples and holders cleaning
★ TEM grids and sample surface hydrophiling

★ Air source, no need to introduce additional gas
★ High-performance oil-free vacuum pump, clean and efficient
★ Compact design, high cost-effectiveness



Plasma cleaning



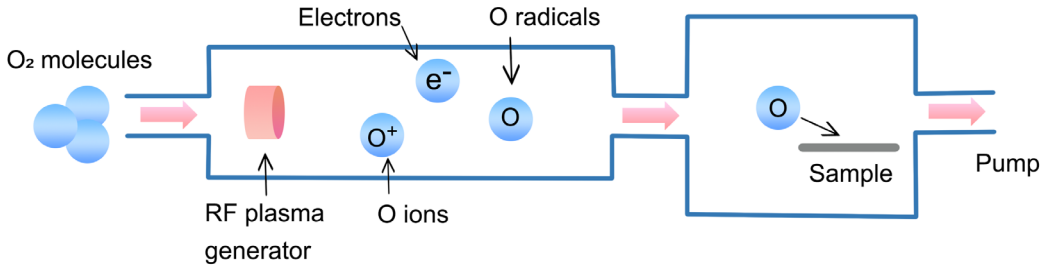
Plasma hydrophiling

AC-TEM Scenario

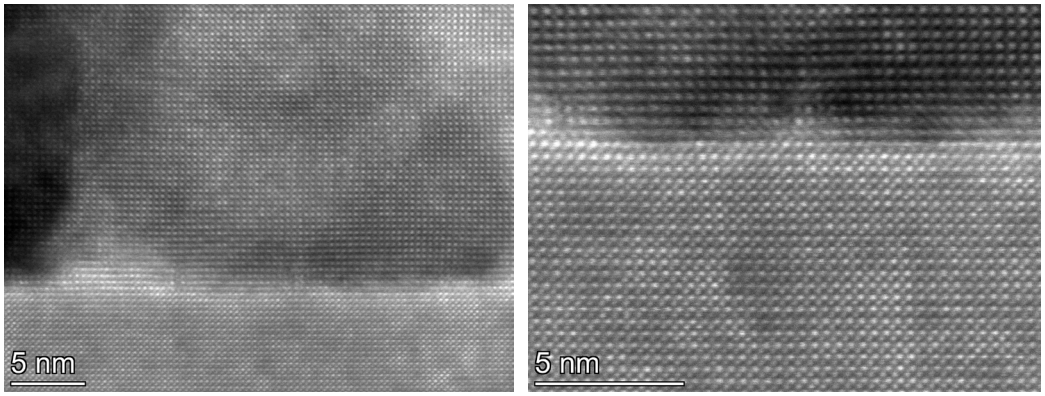
Hydrocarbon Contaminants Cleaning of Ultra-Thin Carbon Films

For well-dispersed nanomaterials, utilizing ultra-thin carbon films as support membranes in aberration-corrected TEM yields better imaging results. However, cleaning the hydrocarbon contamination from ultra-thin carbon films presents a challenge in the sample preparation process.

The SPC150 is a high vacuum plasma cleaner equipped with three gas paths controlled by mass flow controllers (MFCs). With a grid placed between the ion source and the sample chamber, it offers a gentler cleaning effect, making it particularly suitable for removing hydrocarbon contamination from ultra-thin carbon films.



SPC oxygen cleaning schematic diagram



After cleaning

After cleaning

After undergoing SPC150 plasma cleaning, the $BaFeO_3/SrTiO_3$ sample exhibited a clearer membrane interface, greatly improving the quality of STEM-HAADF images.

HV Plasma Cleaner SPC150

★ In-situ TEM holders vacuum leak testing

★ Remote RF ion source: gentle plasma cleaning

★ Plasma cleaning with segmented processing zones: strong treatment zone / weak treatment zone

★ Three workstations, compatible with various TEM holders

★ Precise control of 3 MFCs for flow rates, compatible with Ar, O_2, H_2, etc., offering process flexibility

★ Oil-free high-vacuum system, achieving vacuum levels less than $5*10^{-5}$ Pa

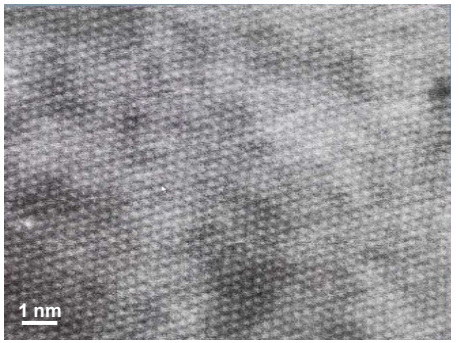
SPC150 Applications

Plasma Treatment of TEM Samples

Using the plasma generated by air to clean TEM samples that are susceptible to oxidation will undoubtedly cause sample modification. The SPC150 can ionize the introduced H_2 and utilize the hydrogen reactive radicals and plasma to chemically react with hydrocarbon contaminants on the sample surface, ultimately achieving the goal of cleaning hydrocarbon contaminants on the sample surface.

Hydrogen plasma treatment is not only suitable for cleaning contaminants on TEM samples that are susceptible to oxidation, but also capable of achieving surface hydrogen doping modification of materials.

SPC can also achieve in-situ plasma treatment of TEM holders or cells, as well as in-situ vacuum leak detection of TEM holders.



STEM image of MoS_2 sample after plasma cleaning.

After plasma cleaning, the MoS_2 sample experiences defocus and long-time imaging without "carbon accumulation" and the problem of white appearance. Using a normal transmission electron microscope with no aberration correction, atomic images can be obtained.

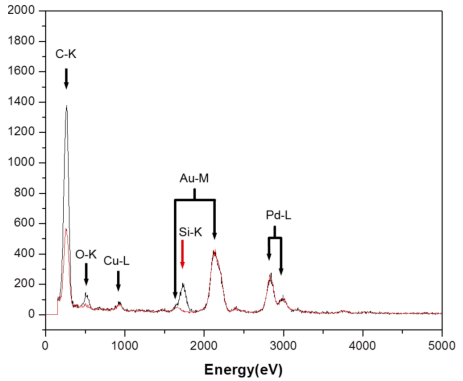
Specialized Silicon Contamination Cleaning

There is often a common silicon contamination in TEM samples that may come from various stages of sample synthesis, preparation, storage, and transfer, which could interfere with TEM imaging, detection and analysis. Conventional plasma cleaning cannot easily remove it.

Our partner* has proposed a new method that uses SF_6 and O_2 as a good mixed process gas to effectively remove surface silicon contamination and hydrocarbon contamination in TEM samples. This technique is essential for STEM with aberration correction.

The SPC150 has 3 gas paths, with MFC able to accurately control gas flow and RF remote plasma design providing strong and weak partition treatment functions. This can make the plasma gentler while ensuring that the carbon film is not damaged, thus achieving a dual cleaning effect for silicon and hydrocarbon contamination.

* Dong Sheng He, Removal of silicon-containing contaminants from TEM specimens. Ultramicroscopy 253（2023）113797.



EDS spectra before and after plasma cleaning.

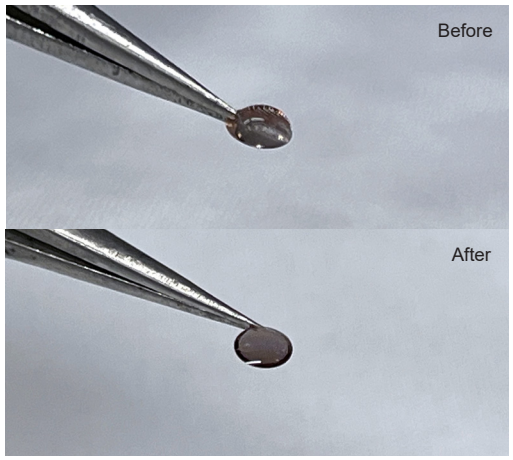
After 1 min of SF_6/O_2 plasma cleaning, both Si and C contaminants on the Au-Pd alloy sample have been eliminated, and the carbon support film remains intact.

CRYO-TEM Scenario

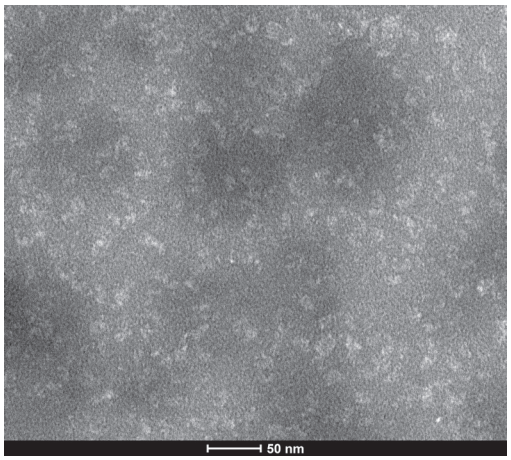
Cryo-TEM Grid Hydrophilic Treatment

With the increasing demand for imaging applications, it is more important than ever to use clean and consistently high-quality TEM grids.

Coolglow is a compact and user-friendly glow discharger that generates a vacuum pressure of <50Pa using a high-performance mechanical pump. It then ionizes the air using an RF power supply, causing a transition of the grid surface from hydrophobic to hydrophilic. This allows for more uniform dispersion of polar suspensions on the carbon film. The RF power supply is efficient, producing a plasma that is gentler (<15eV) and has good reproducibility, resulting in reliable and repeatable results.



before and after plasma treatment of the TEM grid



TEM image of sample after plasma treatment of the grid

After plasma treatment by Coolglow, the TEM grid changes from hydrophobic to hydrophilic, and the negatively stained sample of protein complex is uniformly dispersed, resulting in clear TEM imaging.

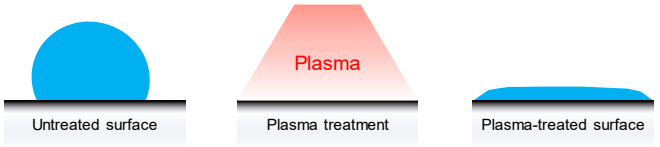
Plasma Cleaning of Sample Surface

Coolglow also offers plasma cleaning of the sample surface to eliminate carbon accumulation during the imaging process.

Glow Discharger Coolglow

★ Room temperature treatment without damage
★ Good uniformity and repeatability
★ Convenient operation

★ Air source, no need to introduce additional gas
★ RF ion source, gentle plasma treatment



TEM Holder Vacuum Storage

LV TEM Holder Storage Station THS 05

If the TEM holder is exposed to the atmosphere for an extended period, water vapor and residual contaminants in the air can adhere to the surface of the holder. When it is reinstalled with the sample and inserted into the TEM, it significantly prolongs the pumping time of the TEM. Furthermore, the residual contaminants can pollute the observed sample and TEM chamber.

THS 05 utilizes a dry pump to rapidly generate a clean vacuum at the order of 100 Pa. Storing the sample and holder within the vacuum storage station ensures maximum isolation from atmospheric water vapor and pollutants. This consequently enhances the vacuum level and cleanliness of the TEM chamber, reducing the pumping time for introducing samples.

★ 5 independent storage stations with no interference in vacuum retention
★ Automatic pressure maintenance to ensure constant vacuum degree
★ Fast speed, cost-effective, and compact structure.

HV TEM Holder Storage Station THS 05T

THS 05T is a high-vacuum TEM holder storage station that uses an oil-free molecular pump system to quickly generate a clean <10^{-4} Pa-level high vacuum. It is particularly suitable for degassing Dewar cans for cryo-TEM holders and the routine vacuum storage and removal of water vapor for TEM holders

It is compatible with holders from various brands of TEM, ensuring that the holders remain in a vacuum state for extended periods and reducing the pumping time for TEM sample loading.

★ 5 independent storage stations with no interference in vacuum retention
★ Automatic pressure maintenance to ensure constant vacuum degree
★ High-vacuum heating and degassing of TEM cryogenic holders

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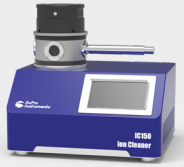
TEM Sample Preparation Product Series

SPC150



IC150

THS 05

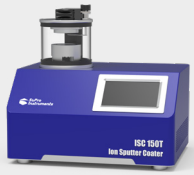
THS 05T

SEM Sample Preparation Product Series

Mini Coater

ISC150

J20



ISC150T

J20T

RPS50

Coolglow

Supro Instruments is headquartered in the Nanshan District of Shenzhen and has offices in Beijing and Suzhou. The company has obtained certifications such as ISO9001 production system certification, CE certification, national high-tech enterprise certification, and specialized and new certifications. The company has a team of members who are passionate about product design and instrument development, with the core team coming from the Chinese Academy of Sciences system.

Supro Instruments is committed to providing lean-level preparation, measurement, and control instruments for electron microscopy sample preparation and micro-nano thin film fields, assisting customers in improving research and development and production efficiency, and providing customers with a better user experience.

For more information, please visit our website: www.suproinst.com

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