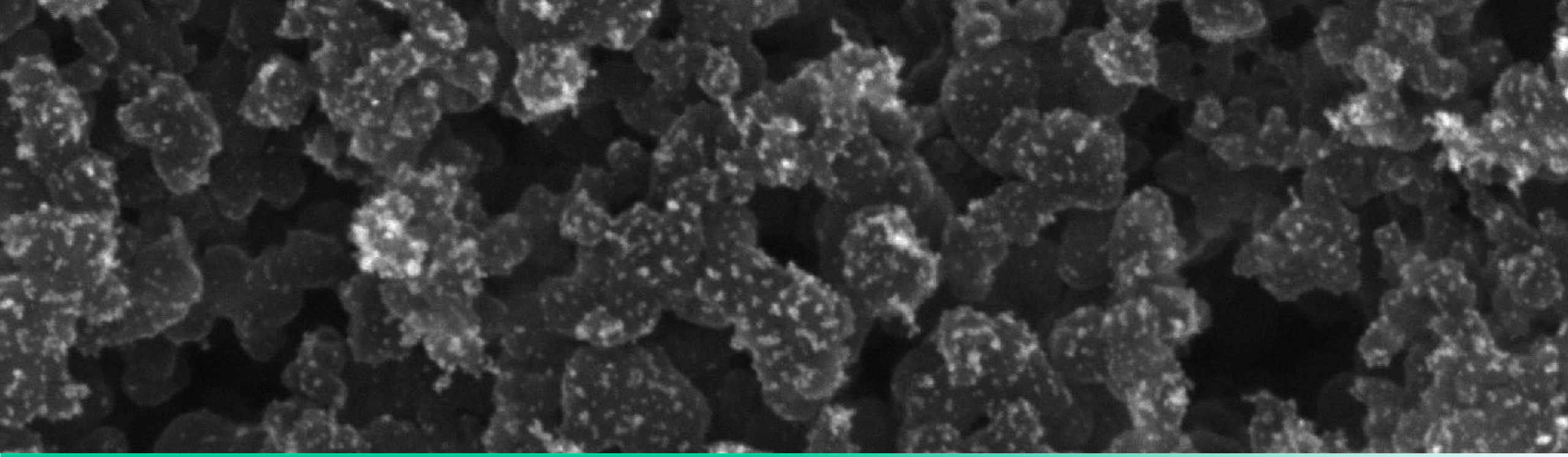

TEM & SEM Gallery

Particles made with the VSP-G1 & Deposition Accessories

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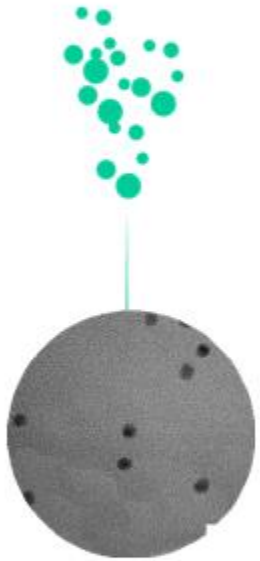
vsparticle

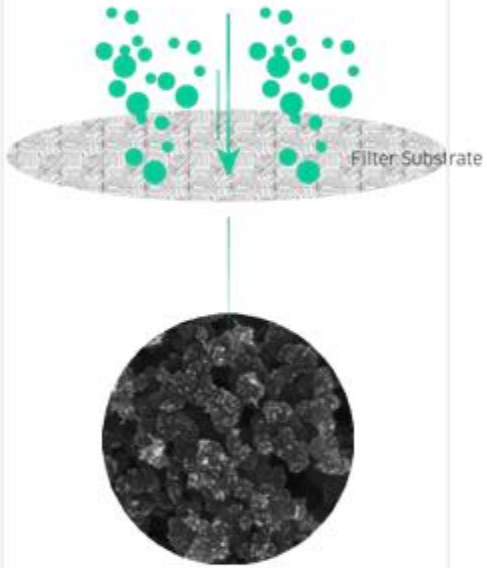
Deposition methods

Aerosol



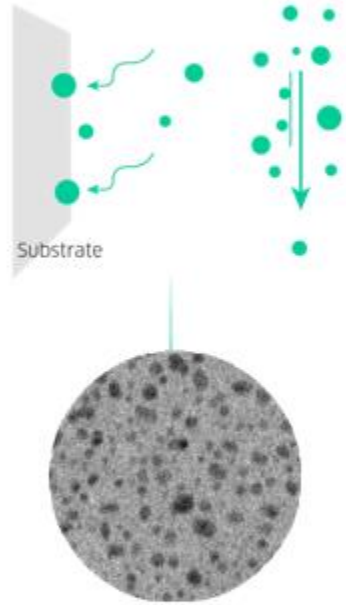
- Deposition possible when for example combined with cell culture exposure systems
- For calibration

Filtration



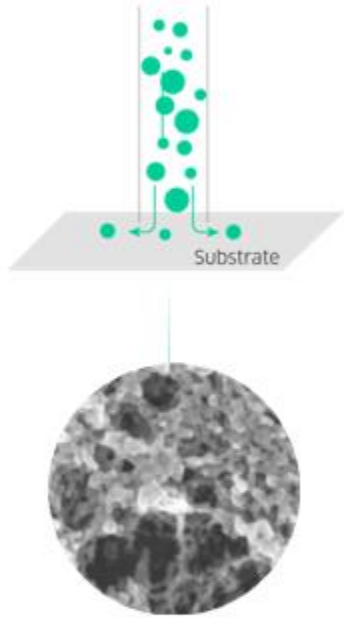
- Porous substrates
- High deposition efficiency

Diffusion



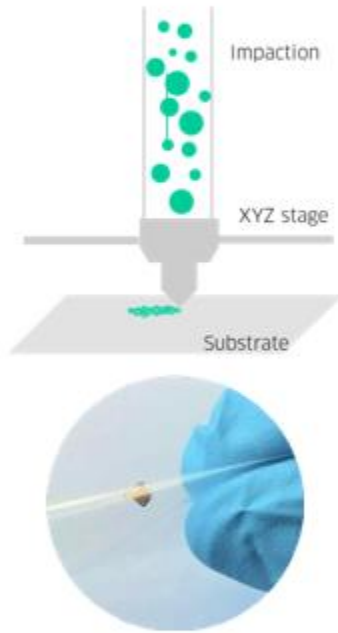
- Low impact, no particle deformation
- Ideal for low surface coverages of nanoparticles

Impaction



- High surface coverages
- Production of porous material

Printing



- Patterning and impact sintering
- High surface coverages
- Production of porous material

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Gallery Contents

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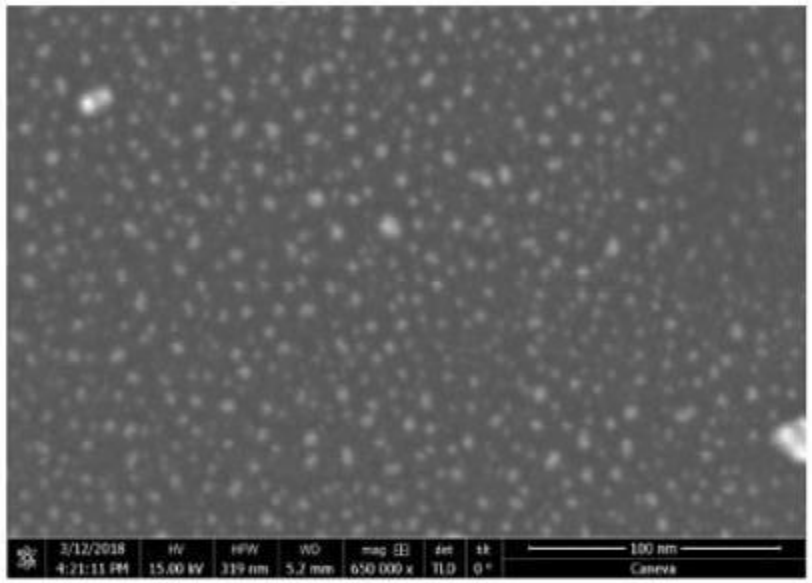
Diffusion

UNAGGLOMERATED PARTICLES

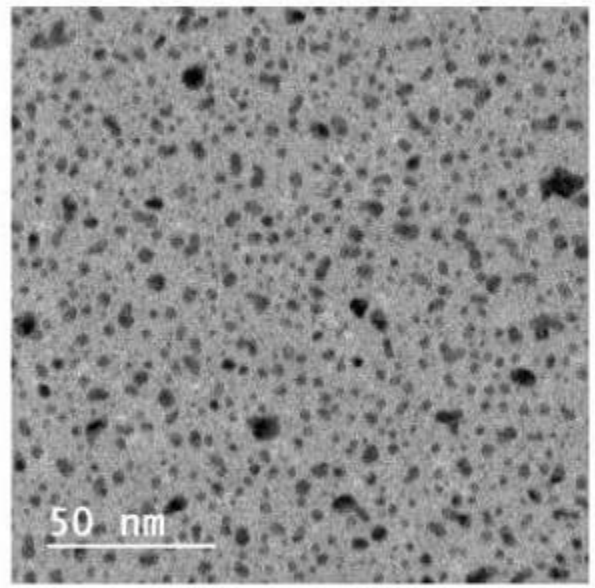
By VSP-G1 plus VSP-A1 diffusion Accessory

Gold nanoparticles

Comparison of SEM and TEM data



(a)



(b)

Example image of SEM and TEM images of the same type of samples. Au on silicon (a) or on carbon TEM grid (b).

Thirty minute deposition gives a 20% surface coverage. Both small (atomic clusters) and large (15 nm) particles are present. The smallest clusters can only be observed with TEM (right).
Coverage can be varied by changing the deposition time. A deposition time of 1-2 minutes gives enough material on the sample for TEM analysis.

Electrode :	Gold
Gas:	Argon
Voltage :	1.3 kV
Current :	8.1 mA
Flowrate :	10 SLM
t_{depo} :	30 minutes

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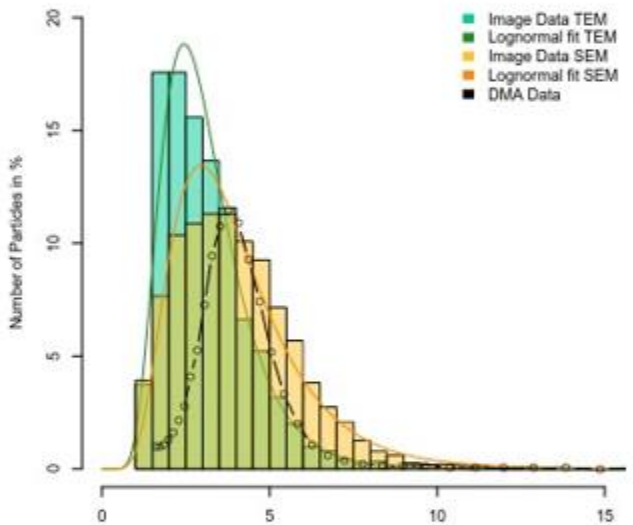
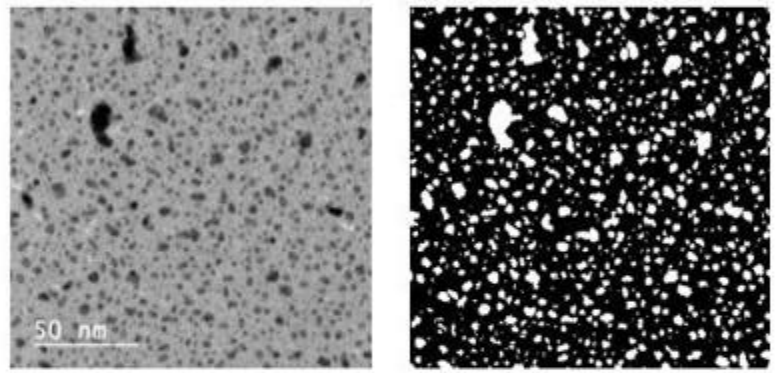
Gold nanoparticles

Comparison of SEM and TEM data



Data set	*Mode in nm*
TEM	2.4
SEM	2.9
DMA	3.8

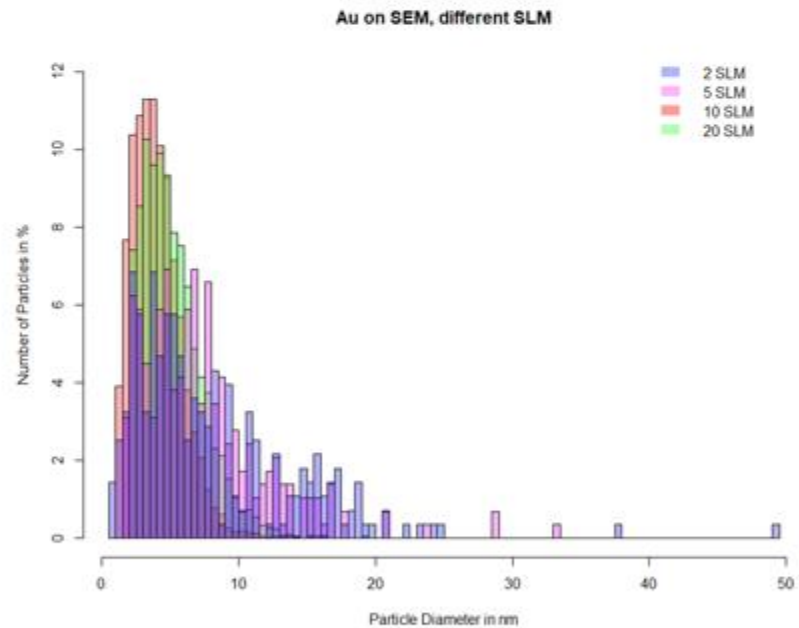


(a) (b)

To analyse the data (a) first a binary image is made. The histogram of particle sizes can be constructed from the binary image.

Particles can be characterized in-line (DMA). The mode is overestimated compared to TEM with 1.4 nm. (left)

Size analysis with SEM (1.3 kV, 8.1 mA, t_{depo} 30 min) for different flow rates.



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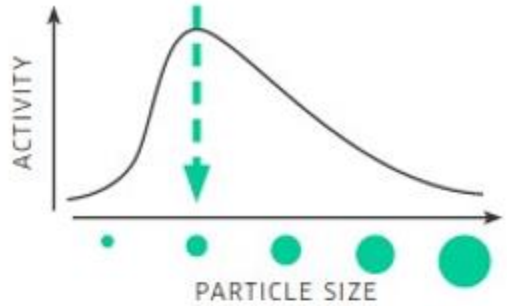
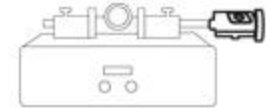
In-situ TEM analysis

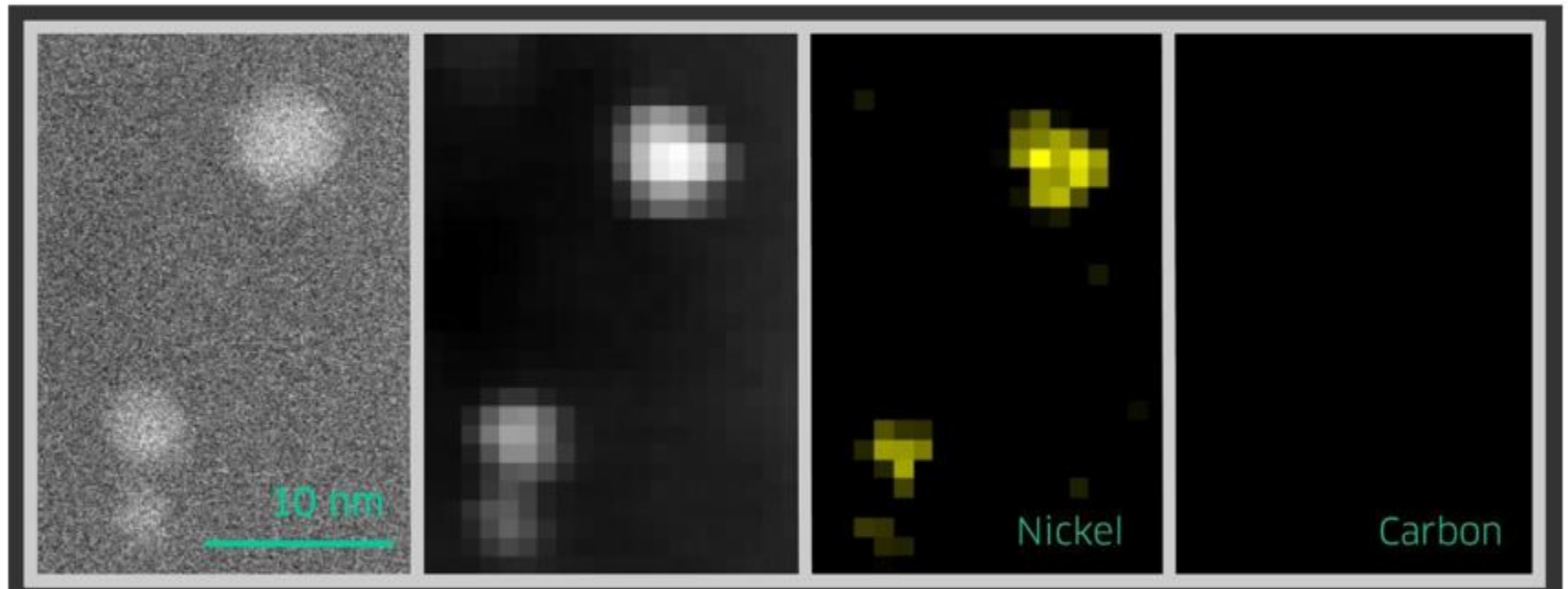




Deposition of Ni on Protochip

TEM images of Ni NPs deposited by diffusion. Analysis with (in-situ) TEM at Oak Ridge National Laboratory. Paper on STEM-EELS analysis and lattice fringes will come out Q2 2019.

Electrode :	Ni
Gas:	Argon
Voltage :	1.3 kV
Current :	8.1 mA
Flowrate :	10 SLM
t_{depo} :	5 minutes



TEM image of spark ablation sample (Charlotte Vogt, University of Utrecht)

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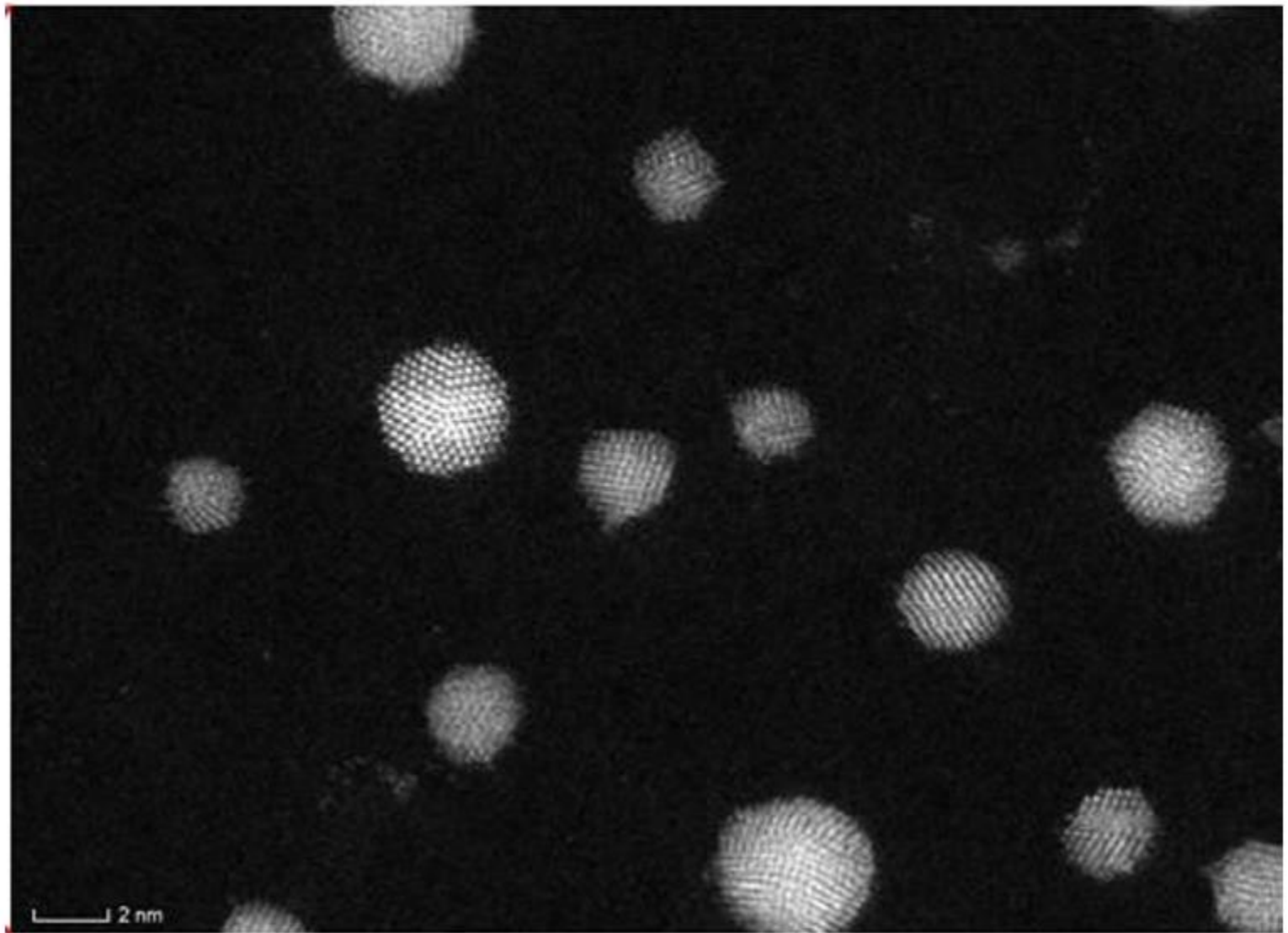
High-res TEM of Au

Analysis of gold Lattice

High-res TEM images in collaboration with TU Vienna (Rupprechter group)

Gold particles are crystalline and bimodal twins appear, even when producing at atmospheric conditions.

Electrode :	Au
Gas:	Argon
Voltage :	1.3 kV
Current :	8.1 mA
Flowrate :	10 SLM
t_{depo} :	5 minutes



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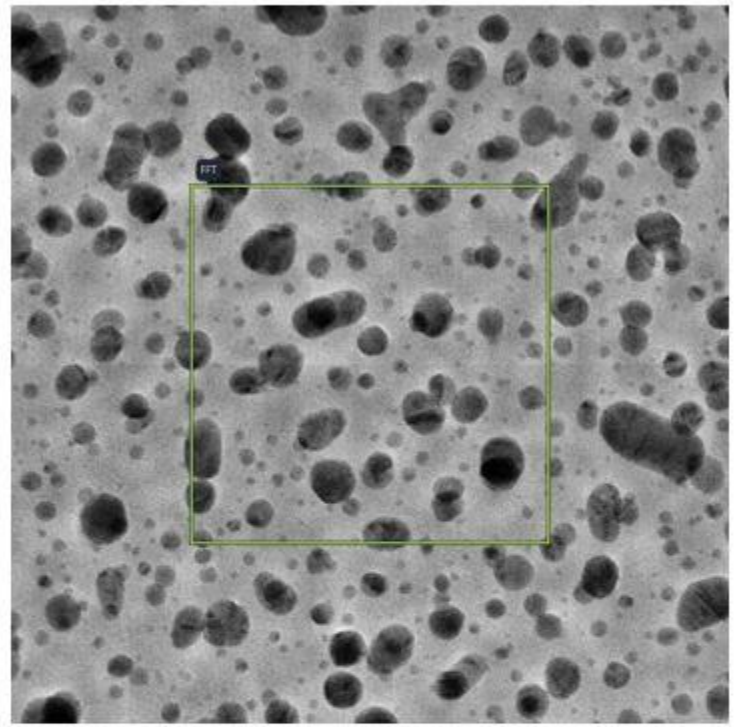
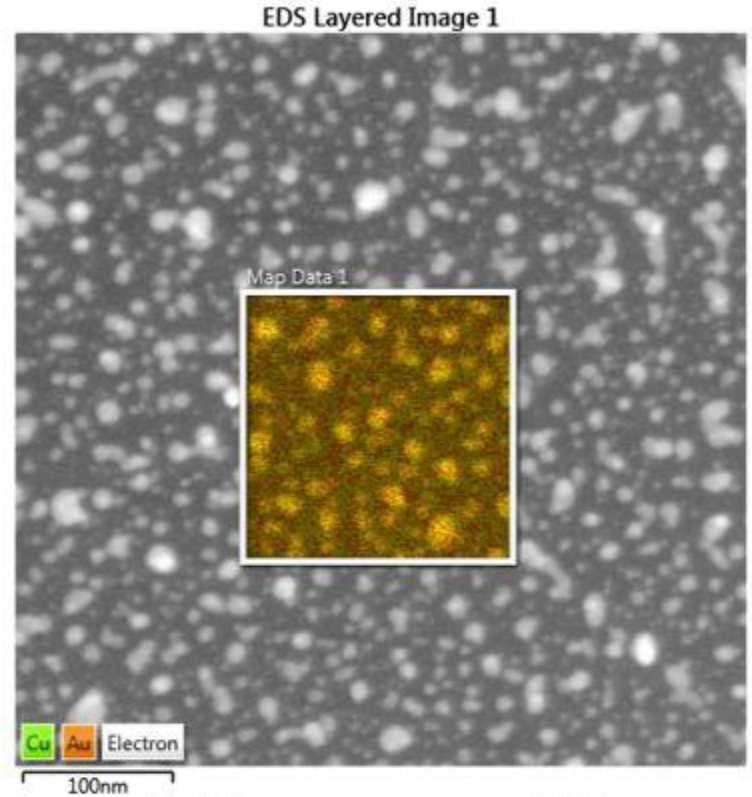
Au-Cu alloy NPs

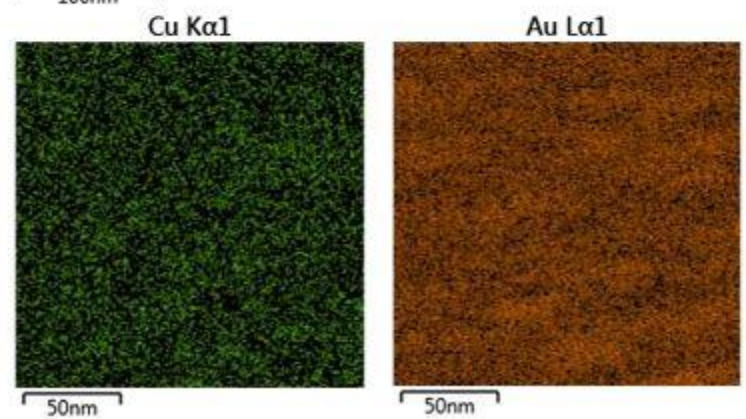
Using Au and Cu electrode

Substrate; DENS climate chip

Electrode : Au & Cu
Gas: Argon
Voltage : 1.3 kV
Current : 8.1 mA
Flowrate : 10 SLM
t_{depo} : 1-4hrs







Successful gas-phase synthesis of Au-Cu nanoparticles using 2 different electrodes. The high gas flow meant the materials are well mixed in the gas phase, which produces alloyed nanoparticles from pure metal electrodes.

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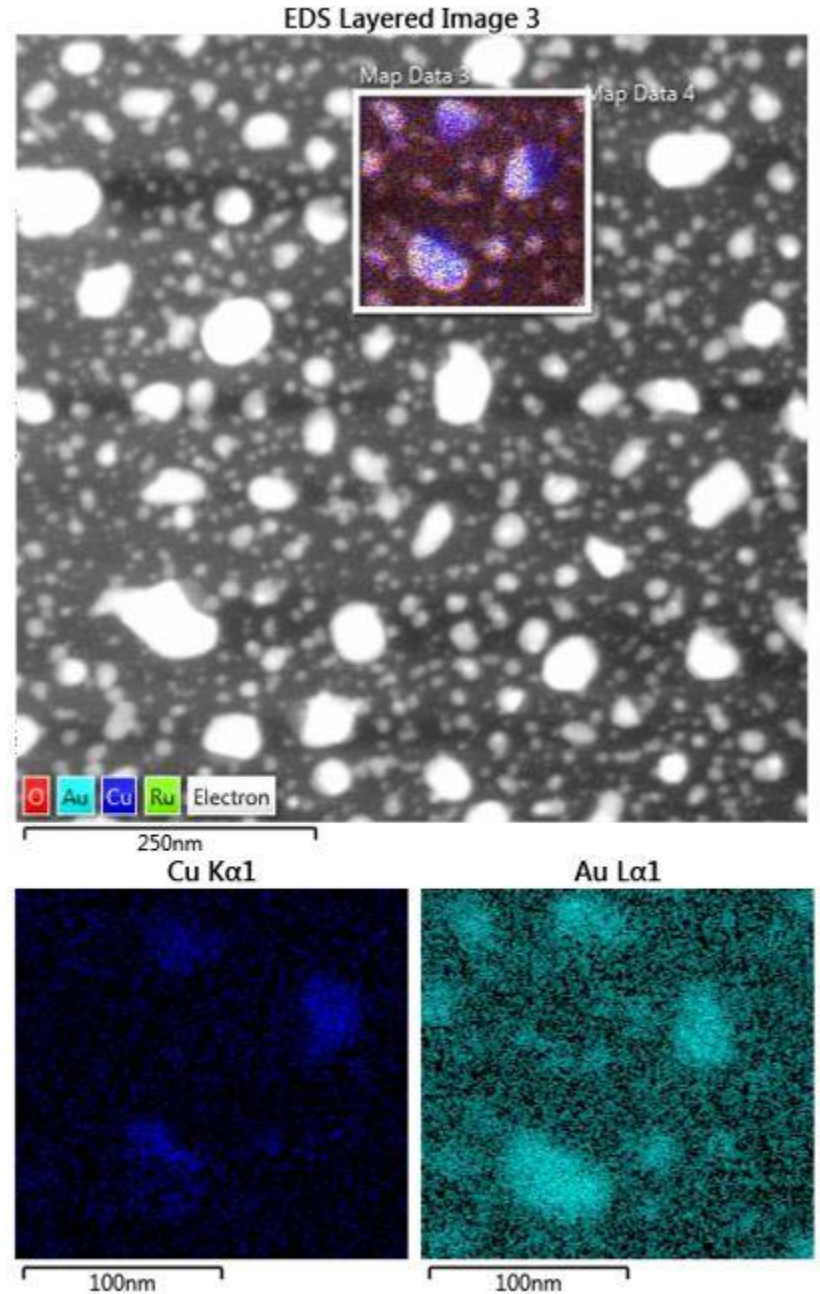
Au-Cu alloy NPs

Using Au and Cu electrode

Substrate; DENS climate chip

Electrode : Au & Cu
Gas: Argon
Voltage : 1.3 kV
Current : 8.1 mA
Flowrate : 10 SLM
t_{depo} : 1-4hrs

Successful gas-phase synthesis of Au-Cu nanoparticles using 2 different electrodes. The high gas flow meant the materials are well mixed in the gas phase, which produces alloyed nanoparticles from pure metal electrodes. After in-situ heating segregation into an Au and Cu phase can be observed.



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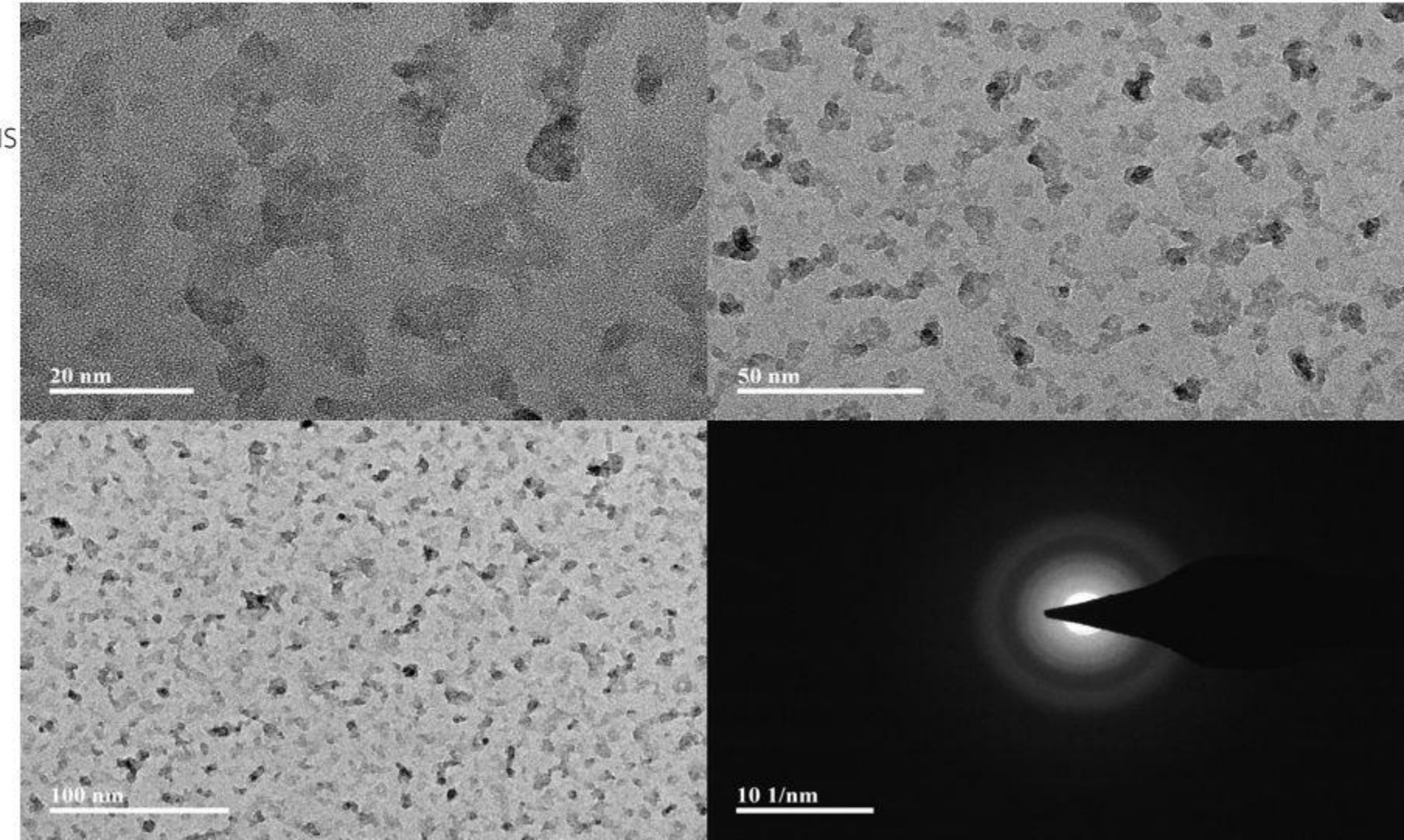
Co nanoclusters

Research by Zhejiang University (China)

Synthesis of Cobalt Clusters using the VSP-G1 and diffusion accessory. The particles are directly deposited on a DENS wildfire chip.

Electrode: Co
Substrate: TEM Chip
Atmosphere: Argon
Voltage: 1.3kV
Current: 10mA
Flow: 8L/min
Time: 1h
Throughflow
Supplier: Zhejiang University



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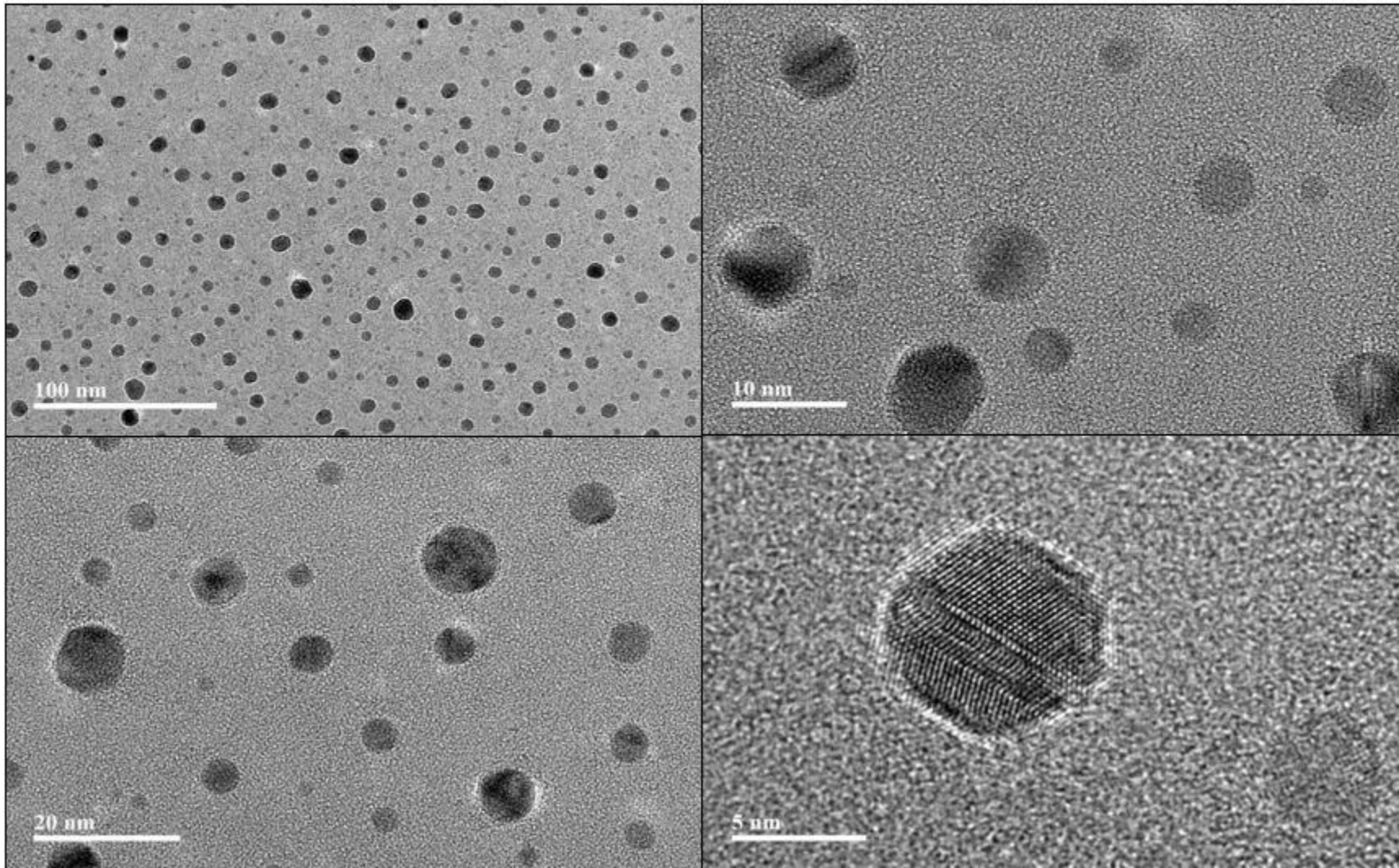
Co nanoclusters

Research by Zhejiang University (China)

After in-situ heating, the clusters have formed spherical particles.

Electrodes: Co
Substrate: TEM Chip
Atmosphere: Argon
Voltage: 1.3kV
Current: 10mA
Flow: 8L/min
Time: 1h
Throughflow
Supplier: Zhejiang University



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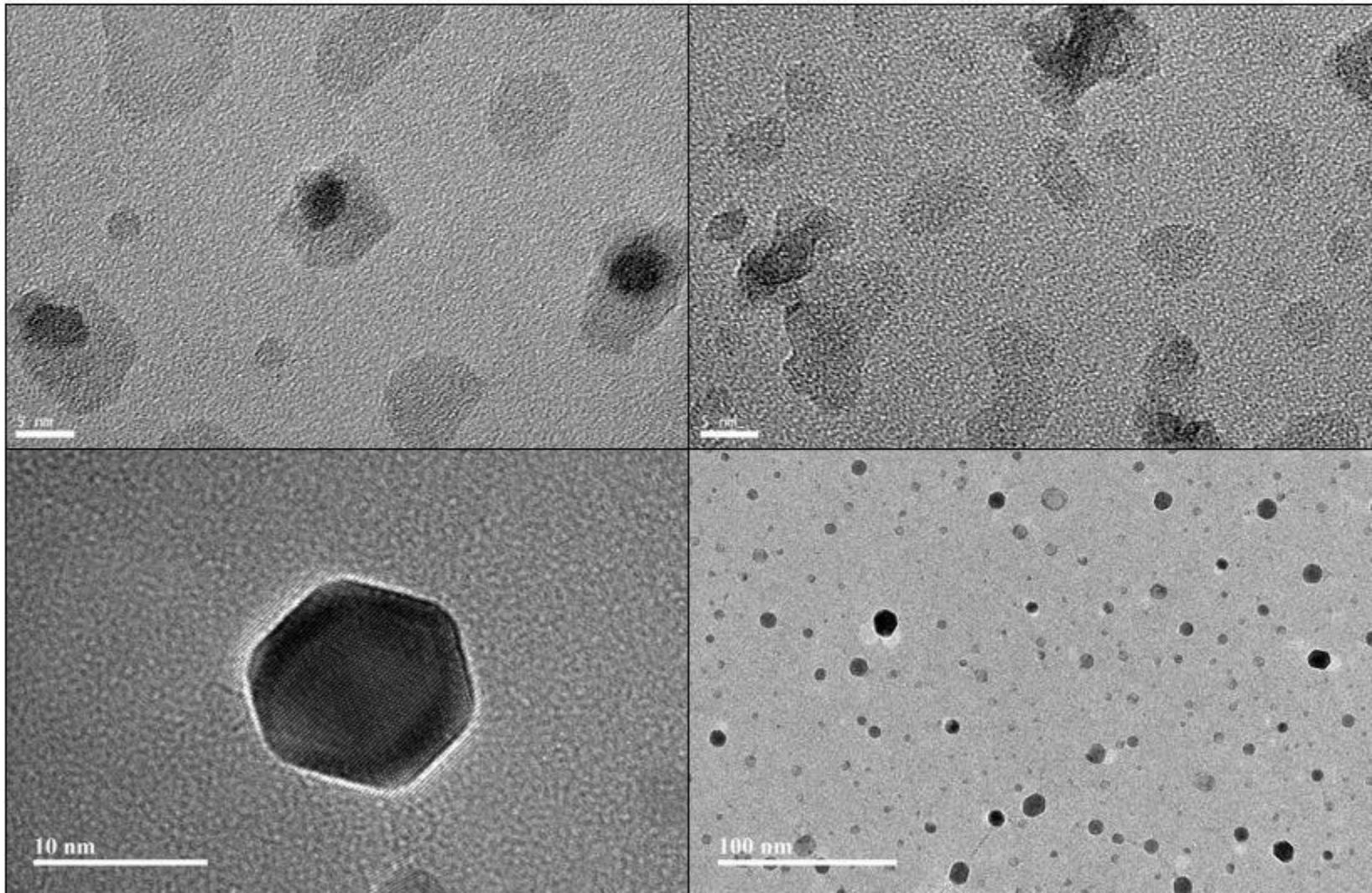
Fe nanoparticles

Research by Zhejiang University (China)

Particles are deposited directly on a TEM grid. Both spherical and hexagonal particles are found.

Electrodes: Co
Substrate: TEM Chip
Atmosphere:Argon
Voltage: 1.3kV
Current: 10mA
Flow: 8L/min
Time: 1h
Throughflow



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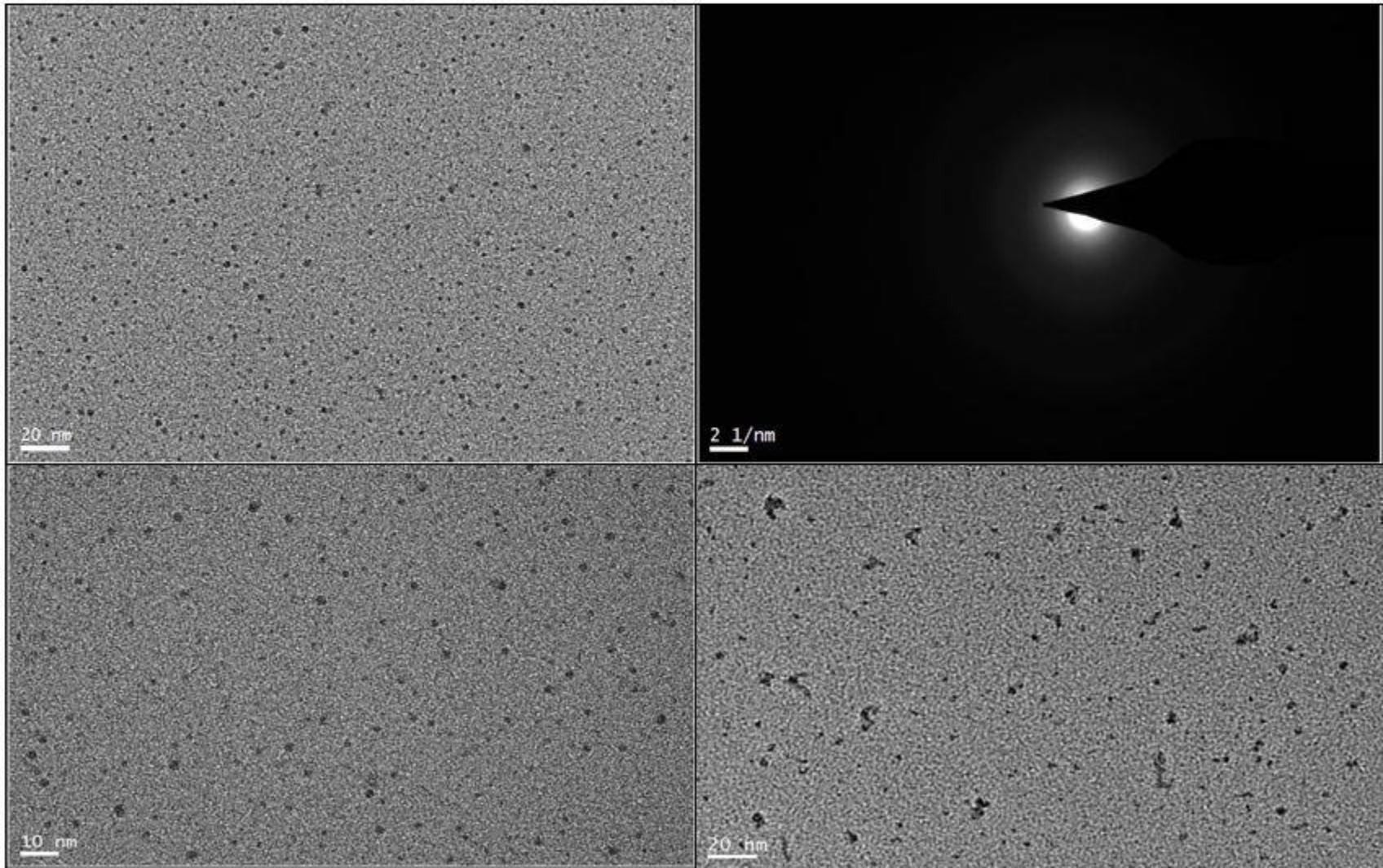
Cu nanoparticles

Research by Zhejiang University (China)

Electrodes: Cu
Substrate: TEM Grid
Atmosphere: Argon/Nitrogen
Voltage: 1.0kV
Current: 6mA
Flow: 10L/min
Time: 30min
Throughflow
Supplier: Zhejiang University



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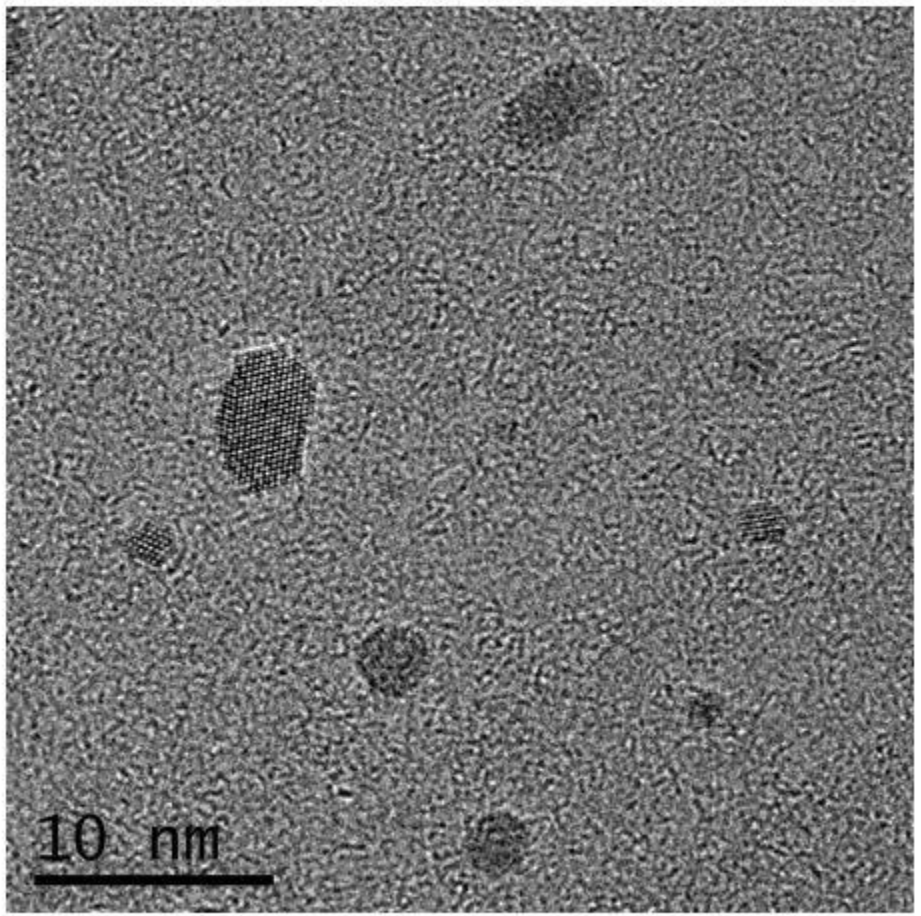
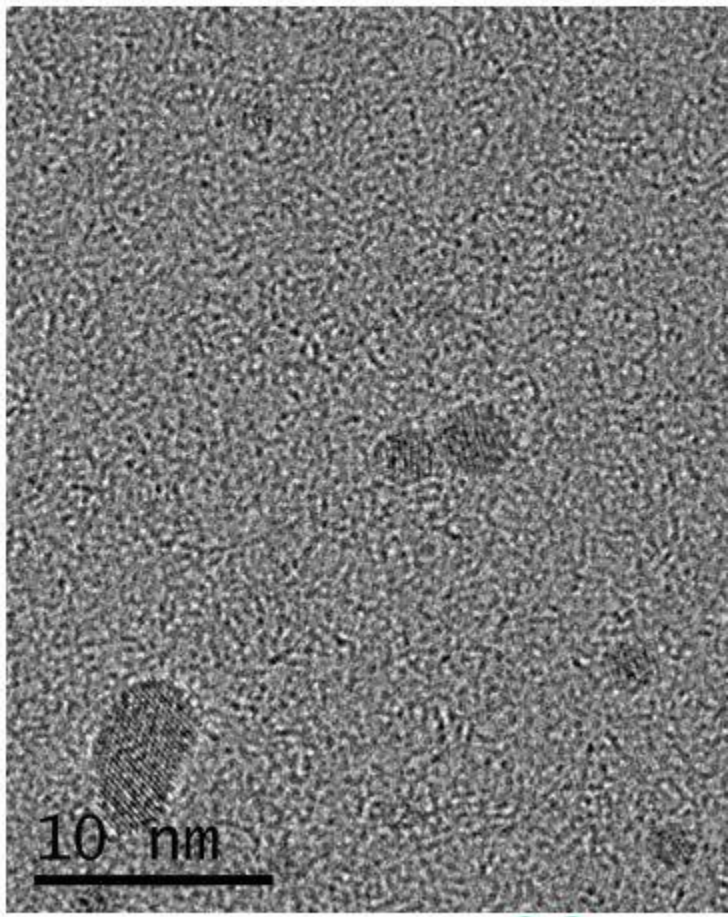
Pt nanoparticles

Research by Beijing Tech University (China)

Electrodes: Pt
Substrate: TEM Grid
Atmosphere: Argon
Voltage: 1kV
Current: 6mA
Flow: 10L/min
Time: 30min
Crossflow
Supplier: Beijing Tech University





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Pd nanoparticles

Research by Zhejiang University (China)

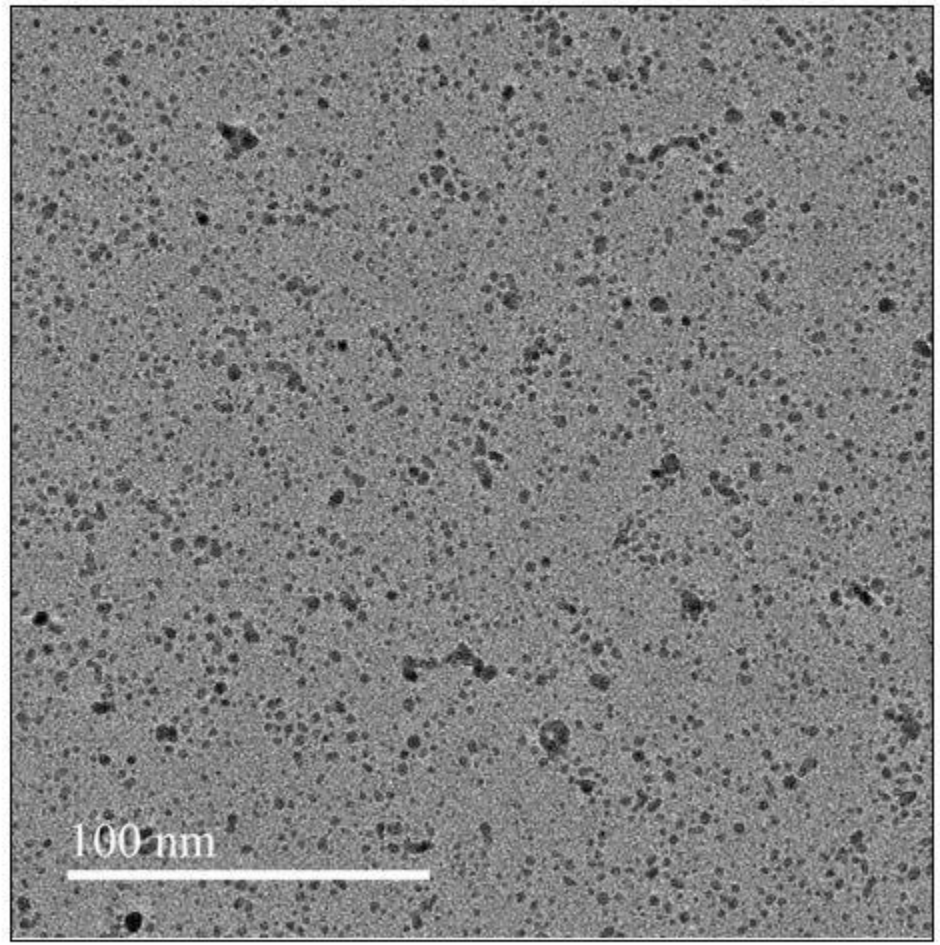
Electrodes: Pd

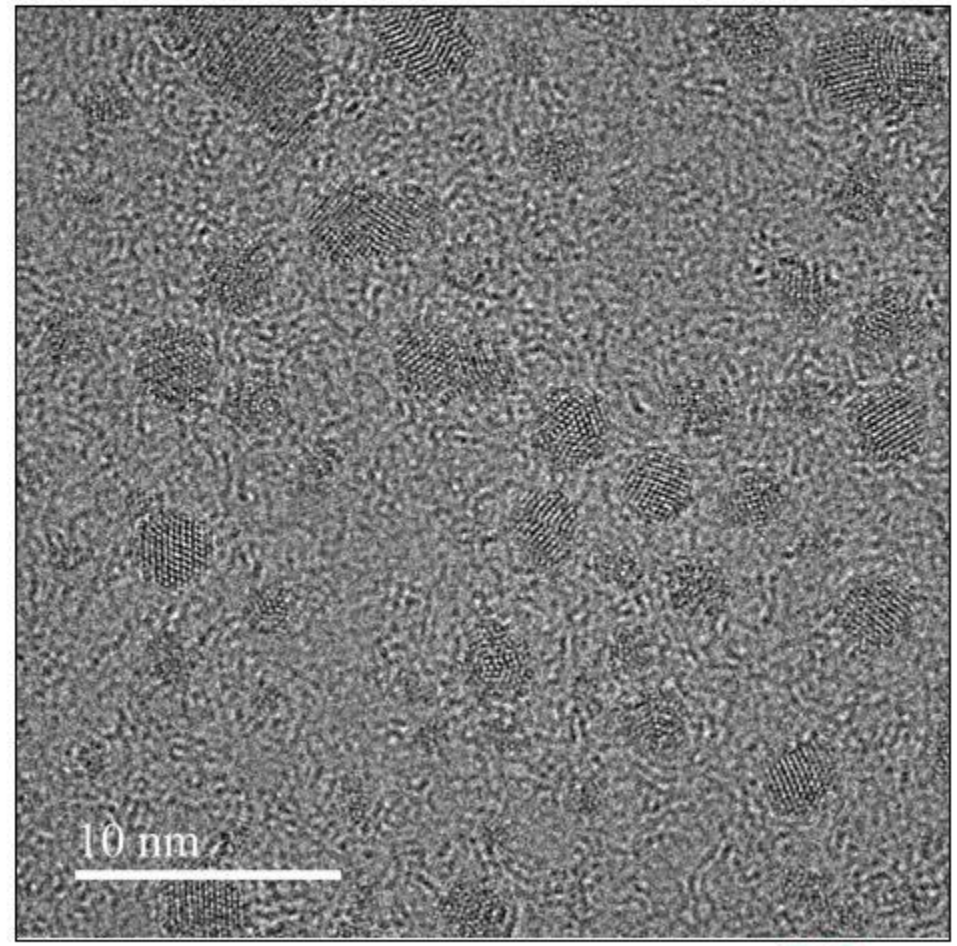
Substrate: TEM Grid

Atmosphere: Argon

Throughflow

Supplier: Zhejiang University





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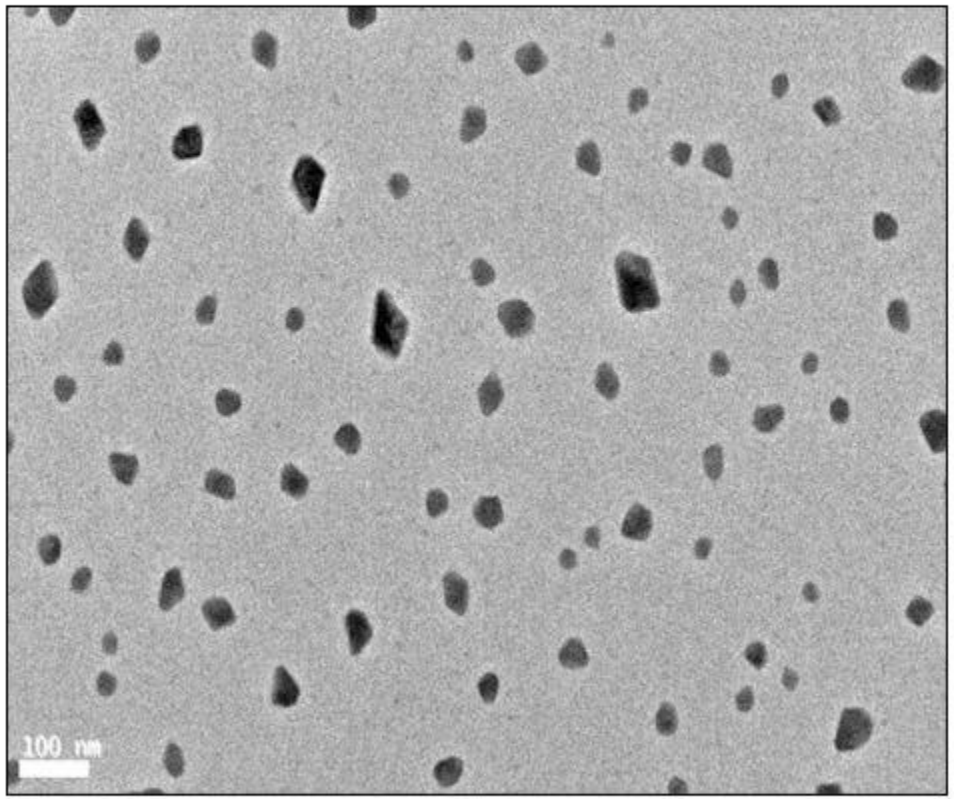
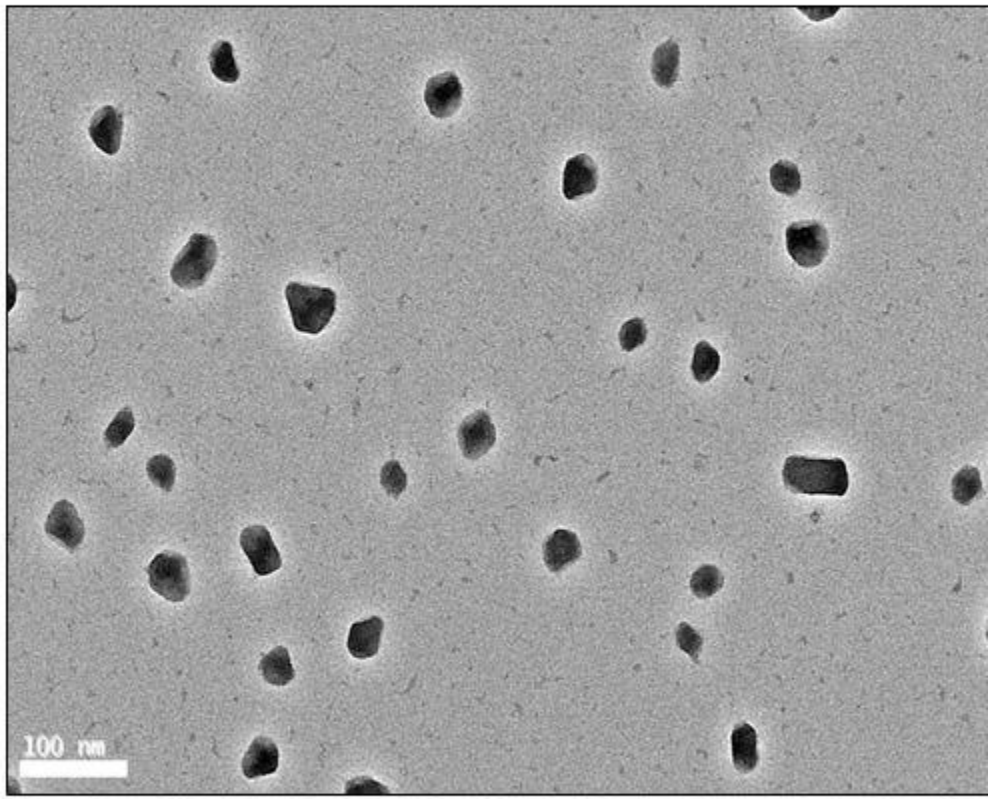
Re nanoparticles

Research by Zhejiang University (China)

After heating
Substrate: TEM Grid
Atmosphere: Argon
Voltage: 1.36kV
Current: 9mA
Flow: 5L/min
Time: 1h
Crossflow
Supplier: Zhejiang University





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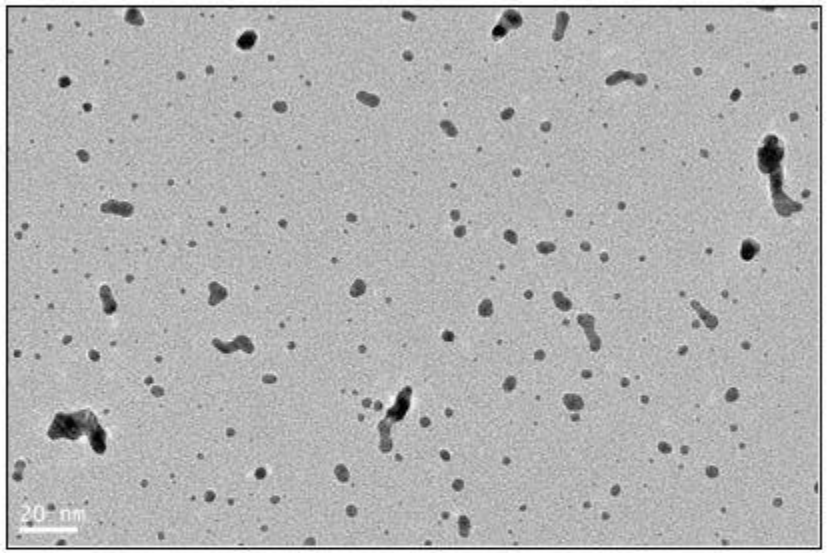
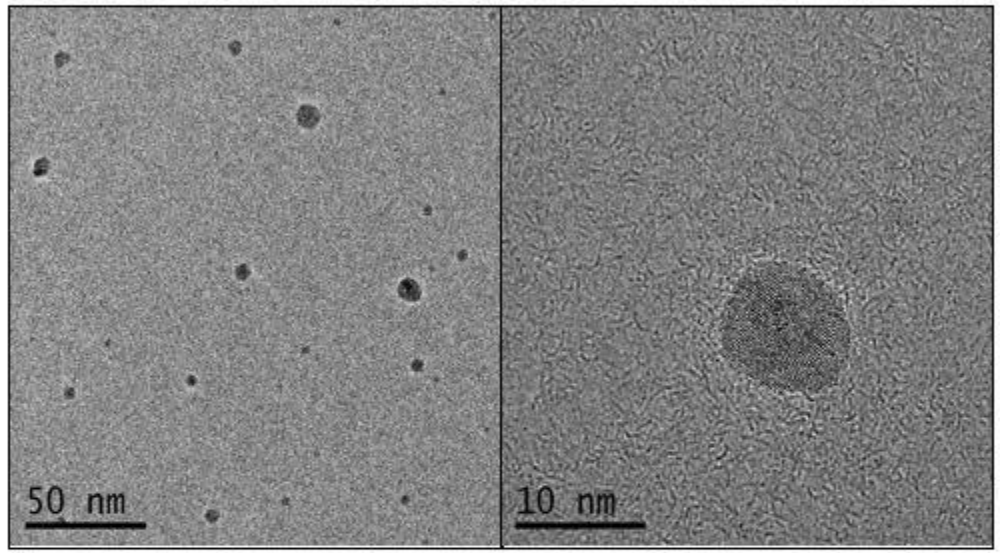
Alloyed nanoparticles

Cu-Au & Cu-Ag



Substrate: TEM Grid
Electrodes: Cu-Au
Carrier gas: Nitrogen
Flowrate : 5L/min
Voltage: 1.36kV

Time: 1 hour
Current: 9mA
Crossflow
Zhejiang University



Substrate: TEM Grid
Cu-Ag NPs
Carrier gas: Argon
Flowrate: 10L/min
Voltage: 1.36kV

Time: 1 hour
Current: 9mA
Crossflow
Dongnan University

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Filtration

DEPOSITION ON POROUS MATERIAL

By VSP-G1 plus VSP-A2 filtration Accessory

Catalyst fabrication

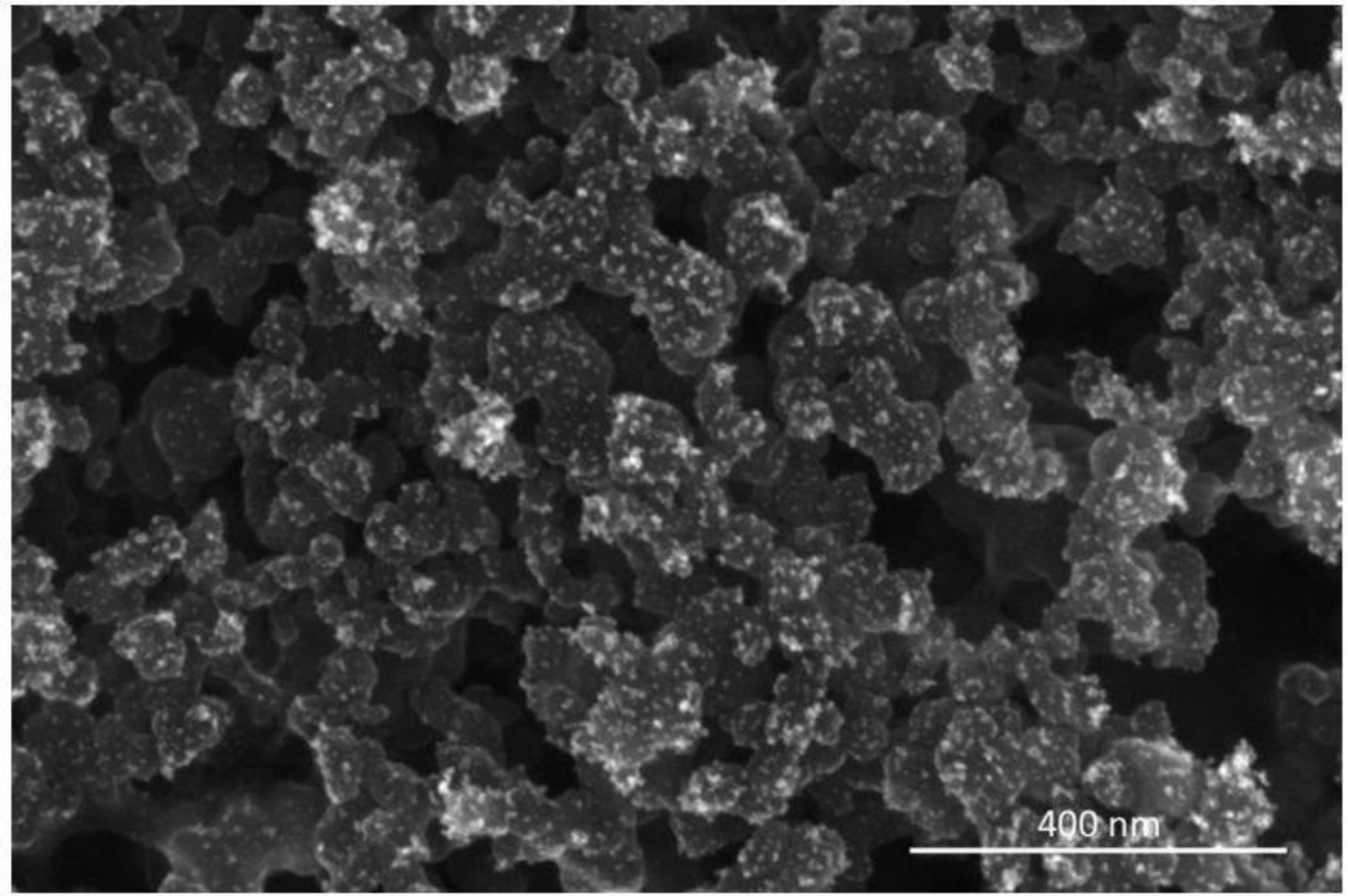
On Carbon Cloth substrate

Using the filtration accessory, particles can be deposited on porous substrates.

Examples of substrates are: Membrane filters, fiber filters and carbon cloth.

On the right a SEM image of gold deposited on porous carbon using this method. The carbon acts as a support and the gold nanoparticles as the catalyst. This sample preparation can be used in electrocatalysis studies.

Electrode :	Gold
Gas:	Argon
Voltage :	1.3 kV
Current :	8.1 mA
Flowrate :	3 SLM
t_{depo} :	30 minutes



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Catalyst fabrication

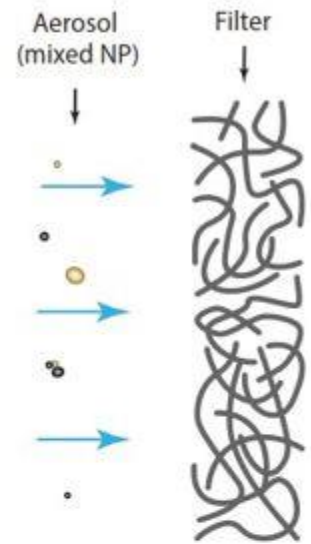
On Carbon Cloth substrate

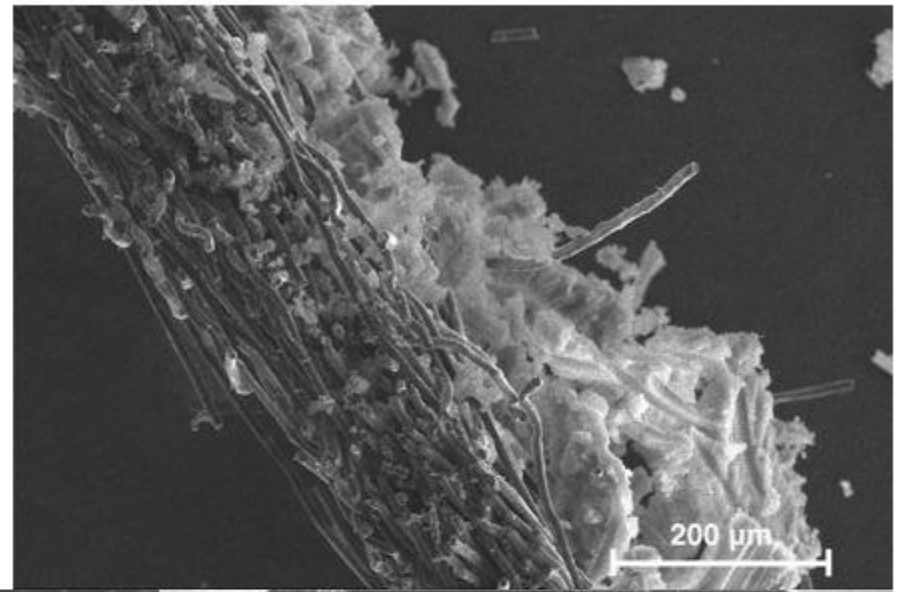
Using the filtration accessory, particles can be deposited on porous substrates.

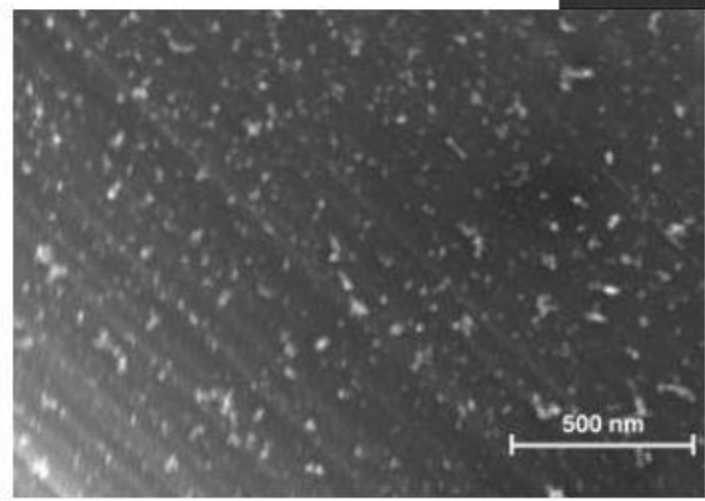
Ni nanoparticles can be deposited in the same way. After deposition for a short while, the particles build up a filter cake.

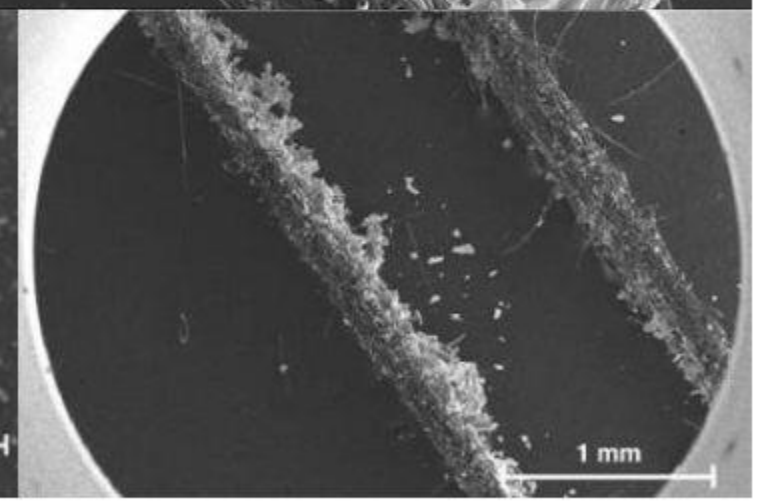
Left, down: Deposition of single nanoparticles on the fiber. All images taken with SEM.

Electrode :	Gold
Gas:	Argon
Voltage :	1.3 kV
Current :	8.1 mA
Flowrate :	3 SLM
t_{depo} :	30 minutes









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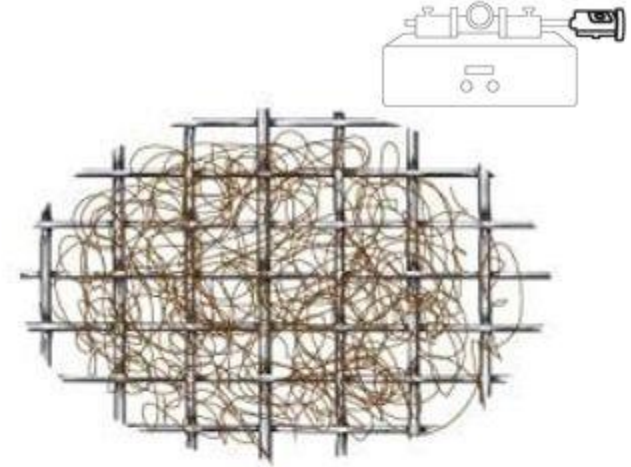
Catalyst fabrication

On electrospinned wires (right)

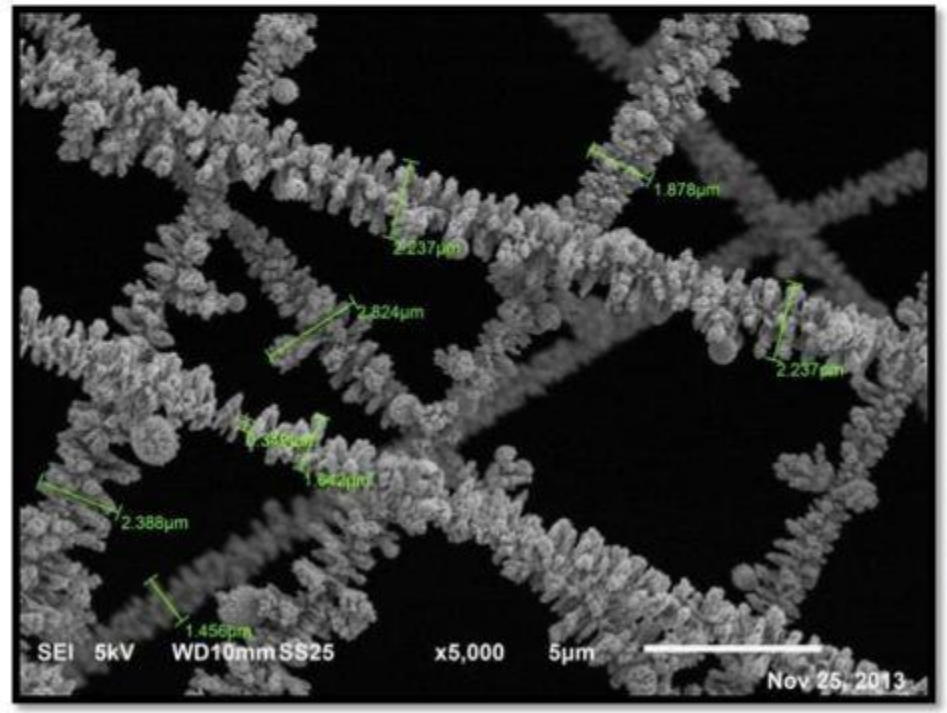


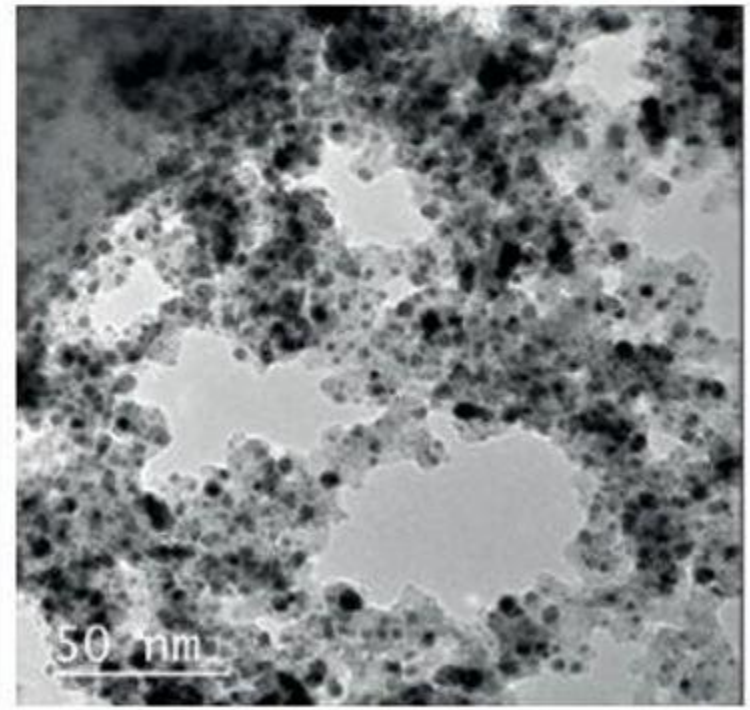
Using the filtration accessory, particles can be deposited on porous substrates.

Left (SEM): Particles are deposited on the electrospun wires. They grow porous structures.

Right (TEM): Aluminum oxide nanoparticles are observed (grey) as well as gold nanoparticles (black) on the right image. The fiber is in the top left of the image.

Electrode :	Au-Al
Gas:	Argon
Voltage :	1.3 kV
Current :	8.1 mA
Flowrate :	3 SLM
t_{depo} :	30 minutes





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Powders

DEPOSITION ON POWDERS

By VSP-G1 plus Powder Coater Accessory

(in development)

Beta G1 accessory: Powder coater

Gas-Phase deposition on powders

Specifications

Minimum powder size: 0.1 µm

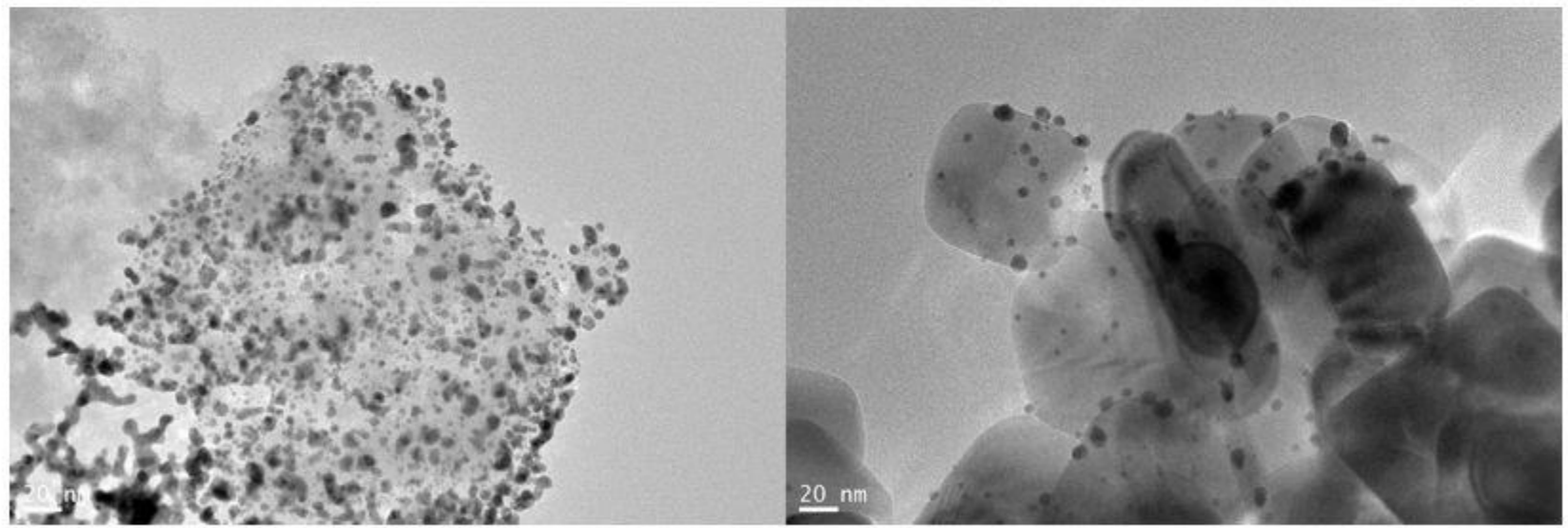
Maximum powder size: 400 µm

Target loading: 0.1-10wt%

Coating speed : Maximum 100 mg/hr of powder

Demonstrations possible from Q1 2020

Right : Au on C
Left: Au NP on TiO2



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Impaction

POROUS LAYERS

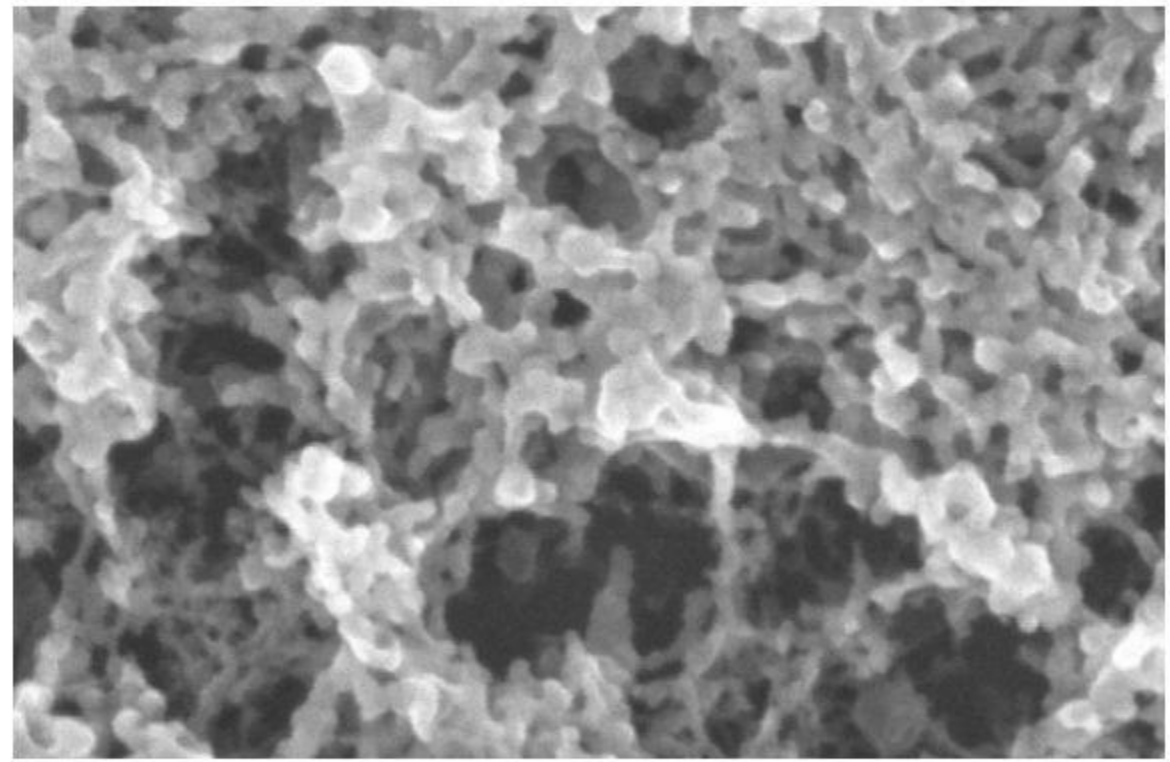
By VSP-G1 plus VSP-A3 Impaction Accessory

Impaction

Nanoporous structures

When multiple layers of particles are deposited we see the growth of a nanoporous structure.

- Noble Metal (Au) and metal oxides (e.g. Cu_xO)
- Room-temperature / 0.1 mbar operating pressure.
- Stable porous structure at ambient conditions
- Sample preparation time: 5 minutes



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Impaction

Porous layers of nanoparticles

Deposition of porous layers of Cu_xO (upper) and Au (lower) fabricated with the G1 plus impaction accessory.

- Applications include (but not limited to)
  - Gas sensor fabrication
  - Materials research
  - Printing of Catalyst support

Analysis with SEM. It is possible to alter the deposition parameters such as spot size (0.1um – 5mm), porosity, layer thickness (50nm – several microns)

Electrode :	Cu/Au	Current :	4 mA
Carrier Gas:	Argon	Flowrate :	1 SLM
Voltage :	1 kV	t_{depo} :	1 minute

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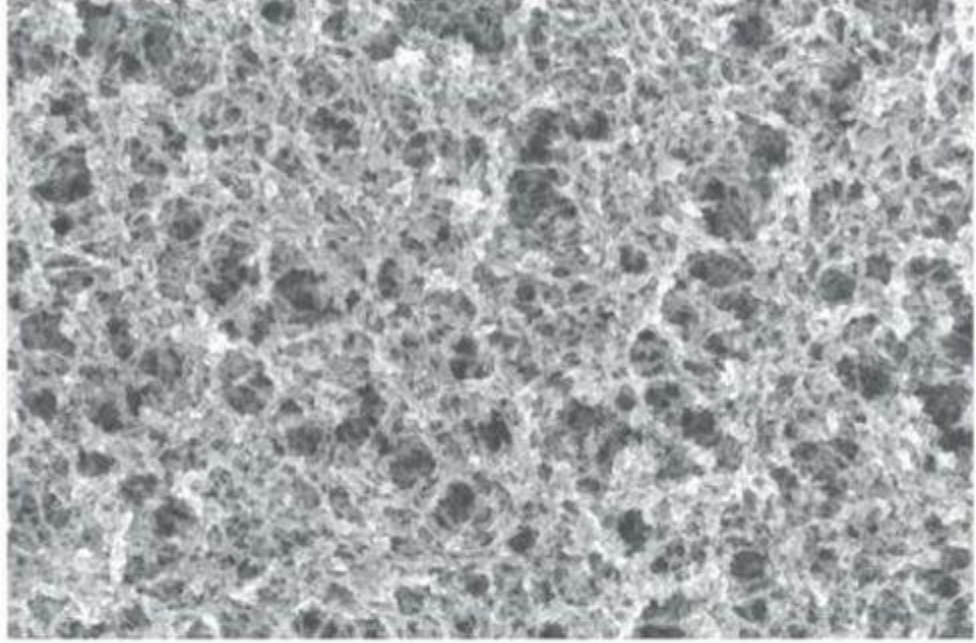
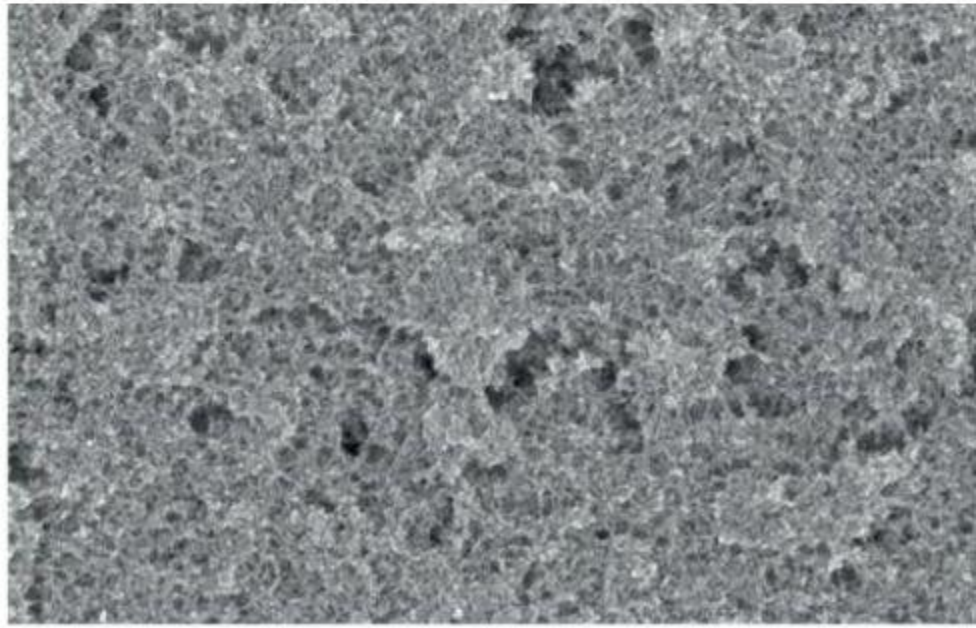


Cu_xO nanoporous film — 500 nm

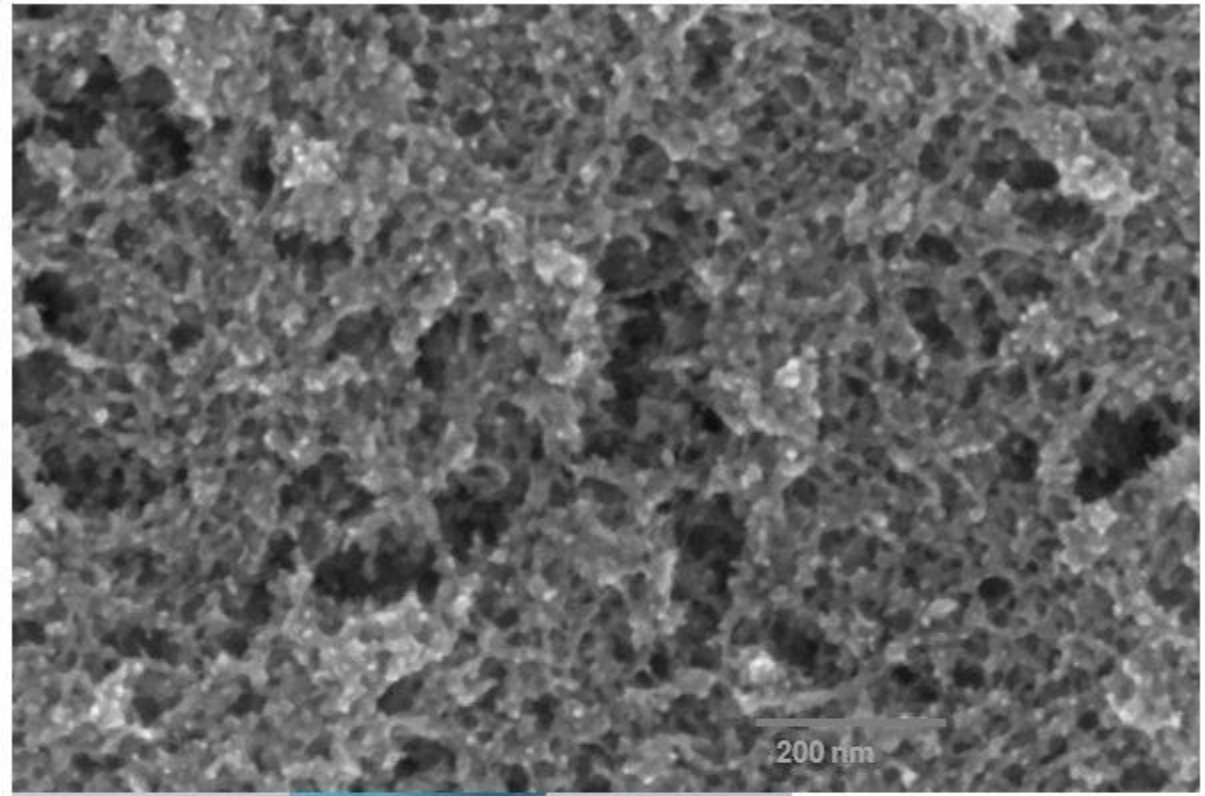


Au nanoporous film — 500 nm

Printing

When multiple layers of particles are deposited, we see the growth of a nanoporous structure.

- Metal (Au) and metal oxides (Cu_xO)
- Room-temperature / 0.1 mbar operating pressure.
- Stable porous structure at ambient conditions
- Spatial control (0.1 mm resolution)
- Beta program printer in 2020



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Size Selection

UNAGGLOMERATED SIZE SELECTED PARTICLES

By VSP-G1 plus Size Selection module

(available from 2019)

Size selection

Deposit monodisperse particles for TEM analysis

With our new size selection module, particles can be selected in-line and deposited on e.g. (in-situ) TEM substrates and electrodes.

Monodisperse gold particles of D= 3nm were deposited successfully.

Electrode : Au

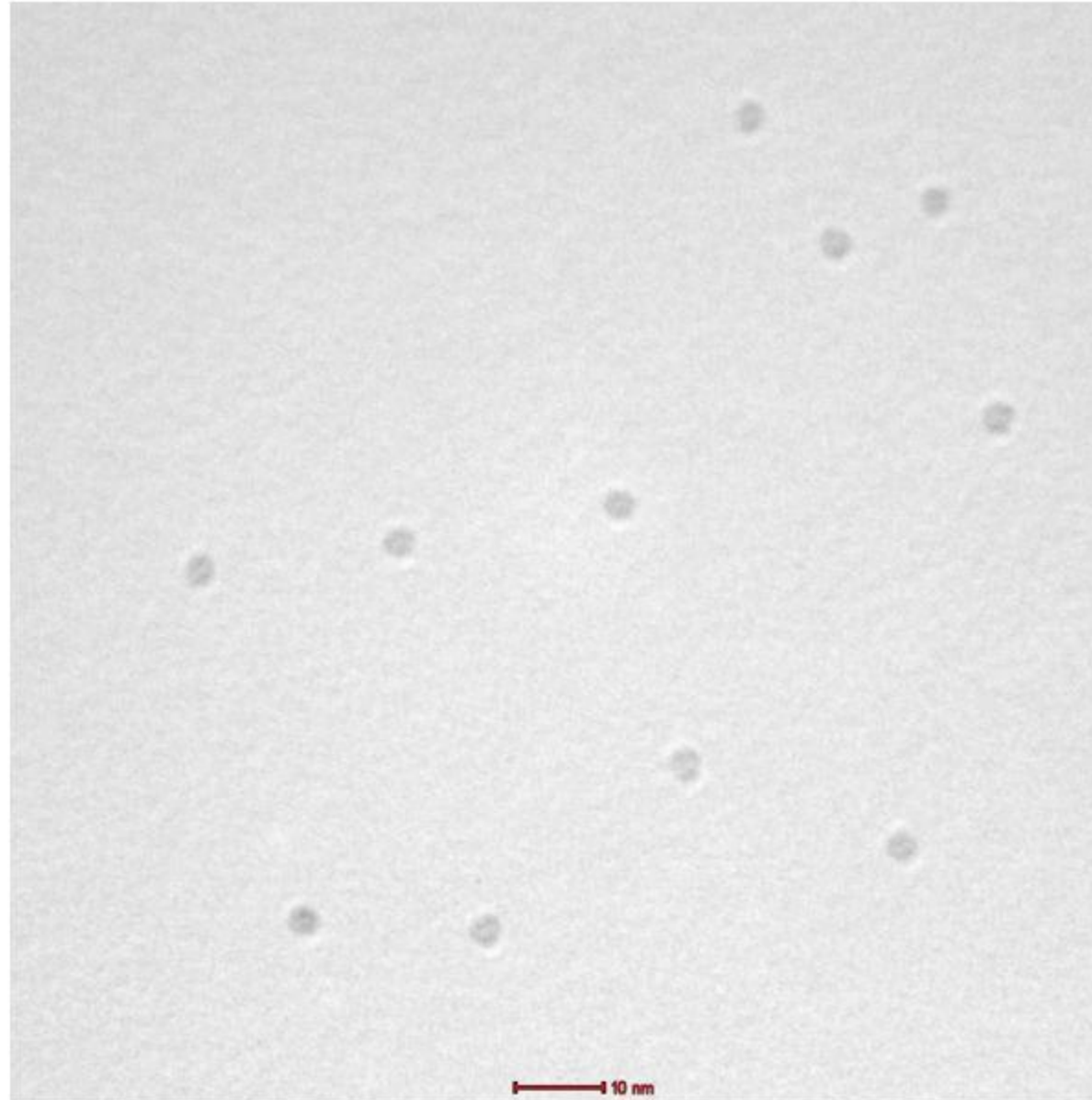
Carrier Gas: Argon

Voltage : 1 kV

Current : 4 mA

Flowrate : 10 SLM

t_{depo} : 5 minutes



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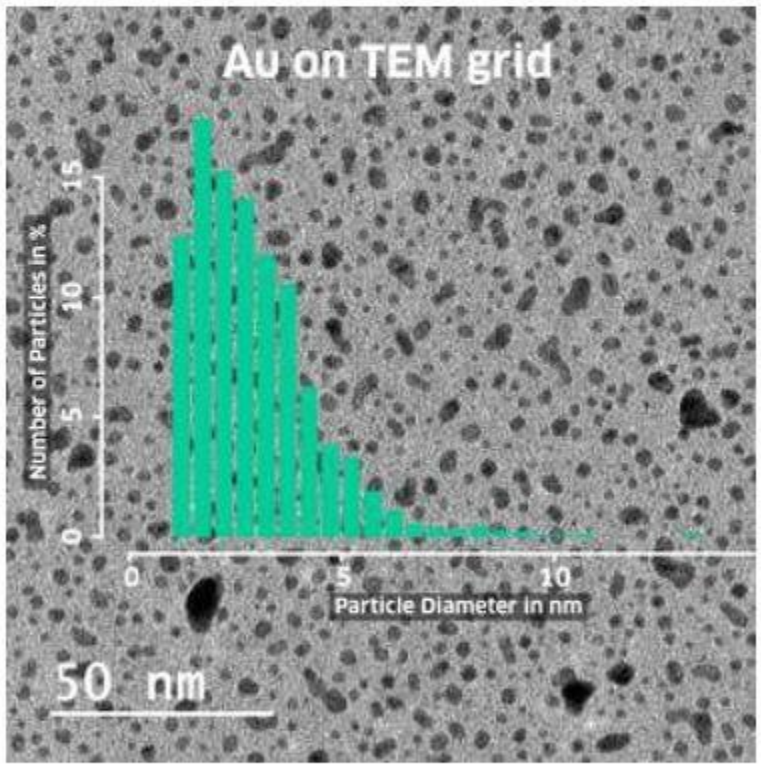
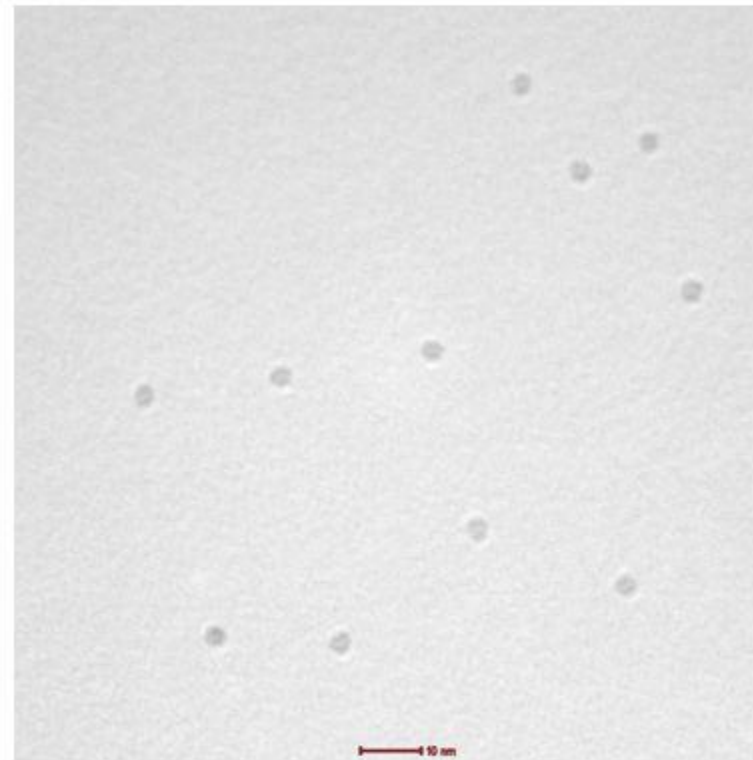
Comparison

Non-size selected Au (left), Size-selected Au (right)



Left: Non-size selected Au, optimized for 3nm, generated with VSP-G1 and deposited by diffusion accessory

Right: Size-selected 3nm Au generated with the VSP-G1 and deposited with the Size Selector (S1)



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Deposition Examples

Screening of Pt catalyst for Fuel Cell research

With our new size selection module, particles can be selected in-line and deposited on e.g. (in-situ) TEM substrates and electrodes.

Monodisperse Platinum particles of D= 3nm were deposited successfully.

All Pt particles are crystalline.

- Electrode : Pt
- Carrier Gas: Argon
- Voltage : 1 kV
- Current : 4 mA
- Flowrate : 10 SLM
- t_{depo} : 45 minutes

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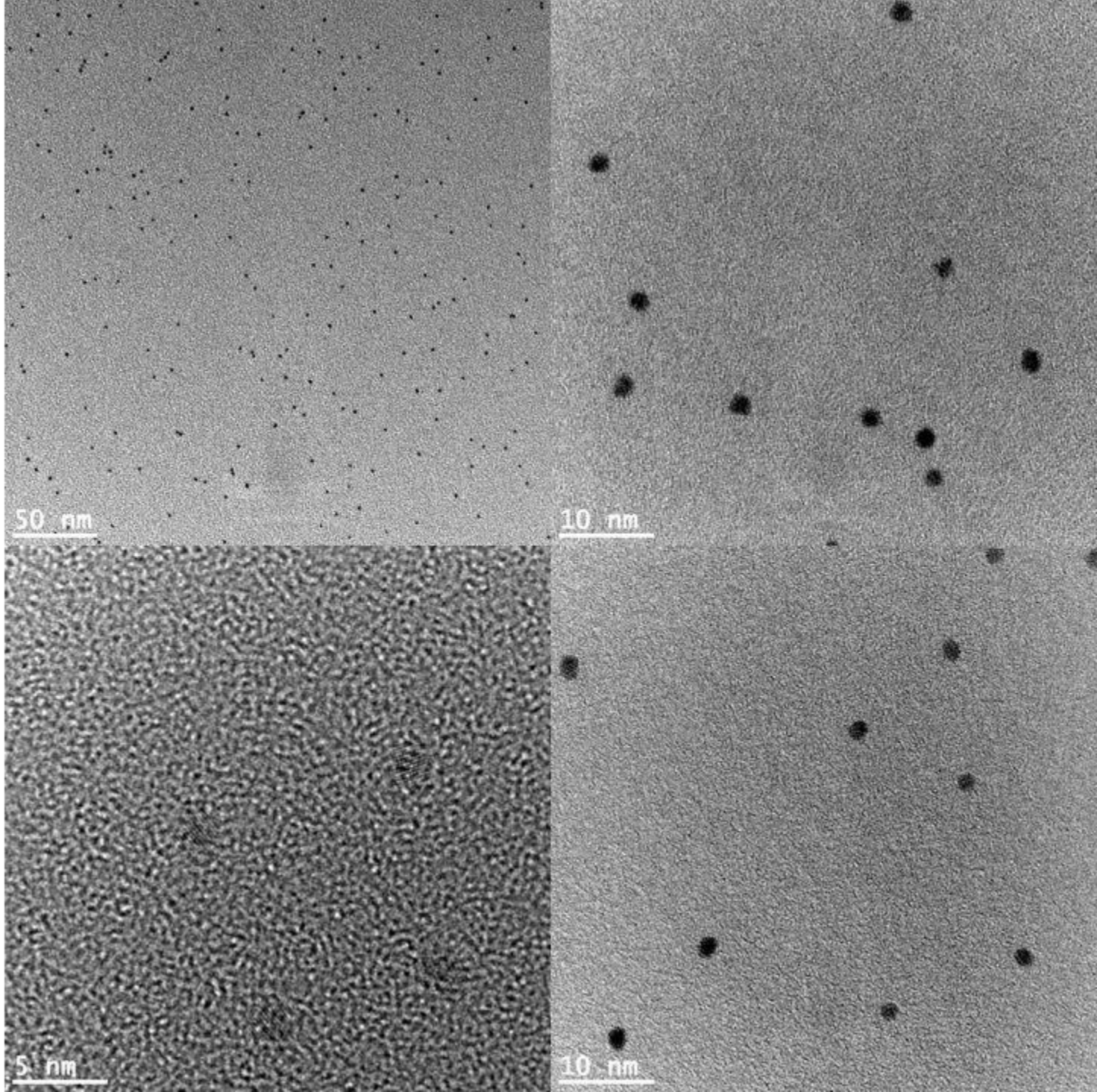

Deposition Examples

Screening of Pt catalyst for Fuel Cell research

With our new size selection module, particles can be selected in-line and deposited on e.g. (in-situ) TEM substrates and electrodes.

Monodisperse Platinum particles of D= 4nm were deposited successfully.

All Pt particles are crystalline.

Electrode : Pt
Carrier Gas: Argon
Voltage : 1 kV
Current : 4 mA
Flowrate : 10 SLM
t_{depo} : 45 minutes

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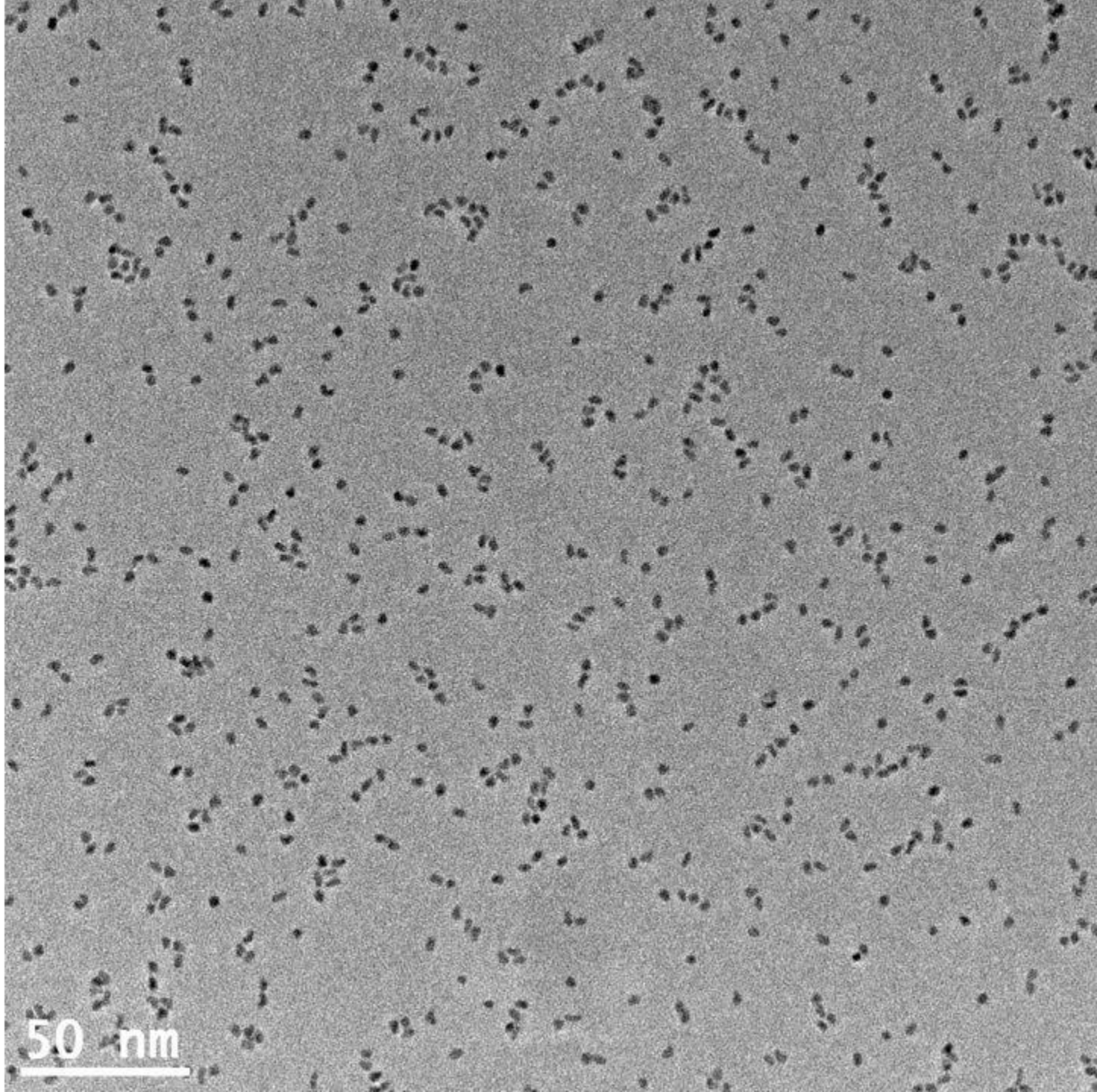

Size selection

Deposit monodisperse particles for TEM analysis

With our new size selection module, particles can be selected in-line and deposited on e.g. (in-situ) TEM substrates and electrodes.

Monodisperse cobalt particles of D= 5nm were deposited successfully.

Electrode : Co
Carrier Gas: Argon
Voltage : 1 kV
Current : 4 mA
Flowrate : 10 SLM
t_{depo} : 30 minutes

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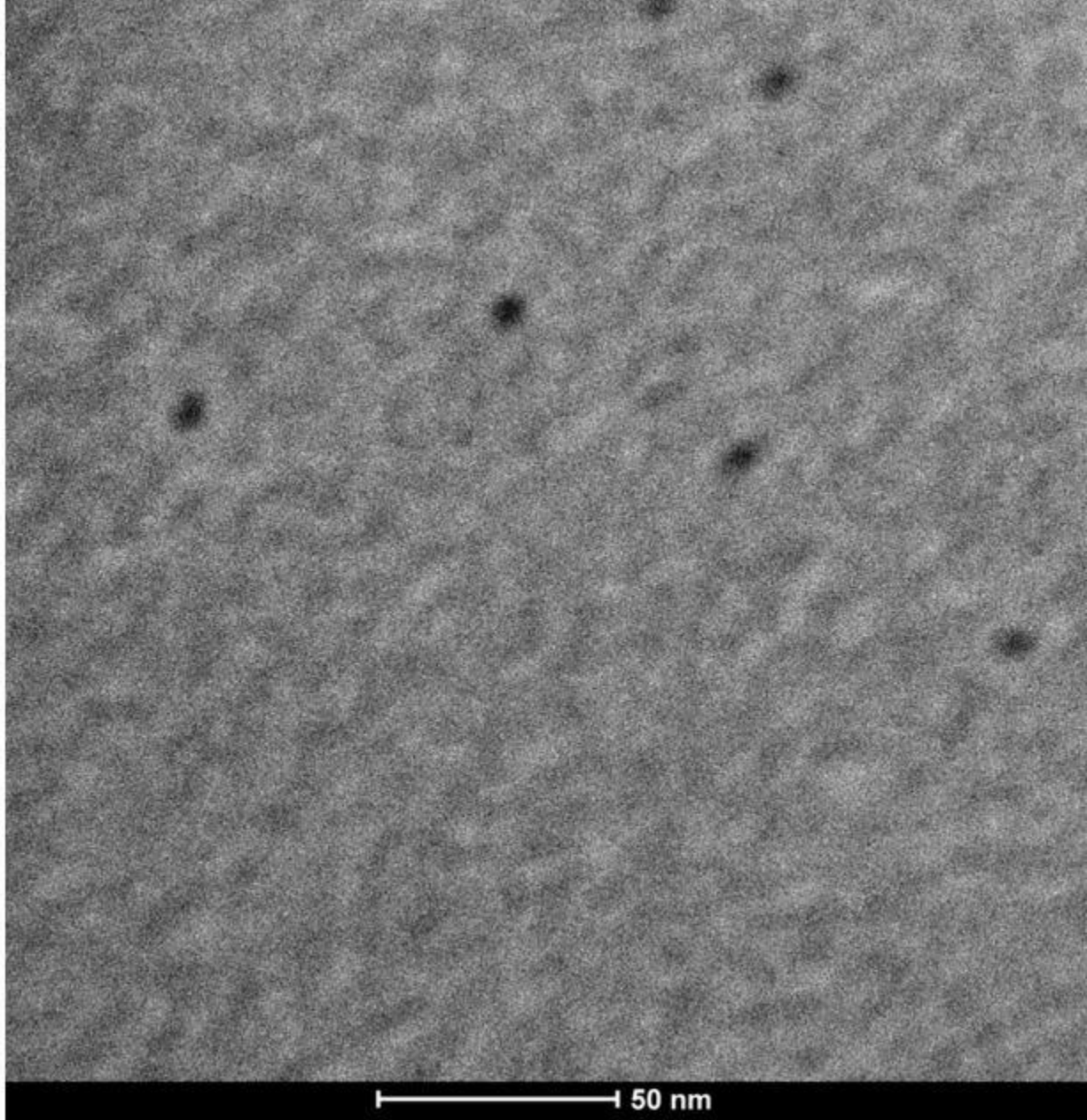

Courtesy of Dr. Matteo Monai (ARC-CBCC Lab, NL)
Size-selected 5 nm Cobalt particles by spark ablation

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