Microfier Silica Challenge Test
Test Results

Introduction

A new ultrapure water (UPW) metrology tool called Microfier significantly shortens particle collection time for scanning electron microscopy (SEM) imaging and allows for elemental analysis of sub-50 nanometer (nm) particles. The device agglomerates sub-50nm particles in 24 hours or less and then releases particles for collection on a standard SEM filter where the established X-ray diffraction (EDS) technique can then be used to identify the elemental characteristics of the agglomerated particles (non-agglomerated particles, smaller than 100 nm, are beyond the sensitivity of the EDS). While traditional SEM analysis measures particle size distribution and elementally analyzes particles greater than 100nm, the Microfier rapidly agglomerates sub-50nm particles for better visual and elemental analysis.

Test Goals

The primary goal of the test is to further document the Microfier's ability to capture and agglomerate sub-50 nm particles by challenging the Microfier with a controlled solution of 12 nm colloidal silica particles. SEM imaging is used to validate the presence of agglomerated particles greater than 1 µm (1,000 nm) and EDS analysis is used to determine the elemental character of observed particles.

Note: the *n*PCD uses electromagnetic force to mobilize and capture charged particles within UPW. Colloidal silica is very weakly charged relative to other ionic and organic constituents found in UPW; as such, the mono-speciation challenge test with buffered colloidal silica is a demanding (worst case) test condition for the technology.

Test Conditions

Analytical Instrument:	Microfier manufactured by Microfier Inc.
General Description:	The Mirofier captures particles from ultrapure water, then releases agglomerated particles for collection on a 100nm pore SEM analytical filter membrane. Collected particles can be evaluated visually by SEM and the elemental constituent(s) determined by EDS.
Capture Flowrate:	375 mL/min.
Release Flowrate:	SEM filter flowrate of 100 mL/min. (approximate) Bypass (PC1) flowrate of 100 mL/min. Total flow of 200 mL/min.

Challenge Test

Material:	Ludox HS-40 (approximately 12 nm SiO_2) Injection Concentration: 13.11 µg/Liter (ppb)
SEM Membrane Filter:	25 mm diameter analytical filter (100 nm pore size)
Test 1 (Background/Control) (SEM Filter 1)	36 hour capture of laboratory UPW Capture voltage - 100 Release voltage – 150
Test 2 (Colloid Silica Challenge) (SEM Filter 3)	30 min. capture of UPW injected with 13 ppb of SiO_2 Capture voltage – 100 Release voltage – 150
Test 4 (Colloid Silica Challenge) (SEM Filter 4)	30 min. capture of UPW injected with 13 ppb of SiO_2 Capture voltage – 200 Release voltage - 250

Results (see Exhibit A for detail SEM images and EDS data)

Test 1: UPW with no SiO2 Background/Control (SEM Filter 1)	**Particle 1-1:** 2 µm smooth transparent particle with Na and K peaks (background Au, C, and O peaks) **Particle 1-2:** 10 µm smooth opaque particle with a F peak (background Au, C, and O peaks) **Particle 1-3:** 5 µm agglomerated opaque particle with Si, Fe, K, and Al peaks and an enlarged O peak (background Au, C, and O peaks). Maximum current of 25 mA during Test 1 particle release Inter-electrode current represents the real time measurement of charge movement during the release of rod particles
Test 2: SiO2 Challenge (SEM Filter 3)	**Particle 3-1**: 2 µm smooth transparent particle with Si and S peaks and an enlarged O peak (background Au, C, and O peaks) **Particle 3-2:** 1 µm smooth transparent particle with Si, and S Peaks and an enlarged O peak (background Au, C, and O peaks) **Particle 3-3:** 1 µm smooth transparent particle with Si and S Peaks and an enlarged O peak (background Au, C, and O peaks) Maximum current of 5 mA during Test 2 particle release
Test 4: SiO_2 Challenge (SEM Filter 4)	**Particle 4-1**: 8 µm solid opaque particle with large Si peak. (smaller/compressed background Au, C, and O peaks) **Particle 4-2:** 1 µm smooth transparent particle with a Si peak (background Au, C, and O peaks) **Particle 4-3**: 5 µm amorphous/transparent particle with Si, Fe, and F peaks (background Au, C, and O peaks) Maximum current of 5 mA during Test 4 particle release

Analytical Summary

- Silica peaks were identified in all six particles
- Particles ranged in size between 1 to 8 μm, generally 1 to 5 μm
- Particles were smooth with transparent edges and denser opaque centers or spots.
- Particles were observed in approximately 1 field per 10 observed fields
- Other elements detected included S, O, Fe, and F

Conclusions

Microfier demonstrated successful capture and agglomeration of 12 nm SiO_2 particles. The observed SEM particles were greater than 1 μm, confirming agglomeration and the EDS element analysis validated that the particles were primarily SiO_2 (silica and oxygen)

- The observed particles ranged in size from 1 to 8 microns. All six particles observed by SEM and analyzed by EDS contained silica and oxygen. Two particles contained only SiO_2 (4-1 and 4-2). Silica and oxygen were consistent and dominant elemental species. The only other species observed were sulfur, fluoride, and iron, indicating only a small amount of cross or background contamination from the Microfier and/or UPW system.

- All six particles had similar morphology – smooth with transparent edges and denser opaque centers or spots. The similar morphology indicates a consistent type of material being introduced from the homogeneous silica challenge.

Comments/Observations

- Some difference should be expected between the current challenge test and naturally occurring UPW particles because of:

    1. High concentration of the silica colloids
    2. Potential saturation of the Microfier capture rod
    3. Stabilization constituents within the colloid test solution (Na counter ions)
    4. Absence of other elemental constituents to facilitate agglomeration

- Qualitatively, particles in Test 4 appeared more solid (see Particle 4-1) than in Test 2, suggesting that the high voltage may have increased the amount of agglomeration.

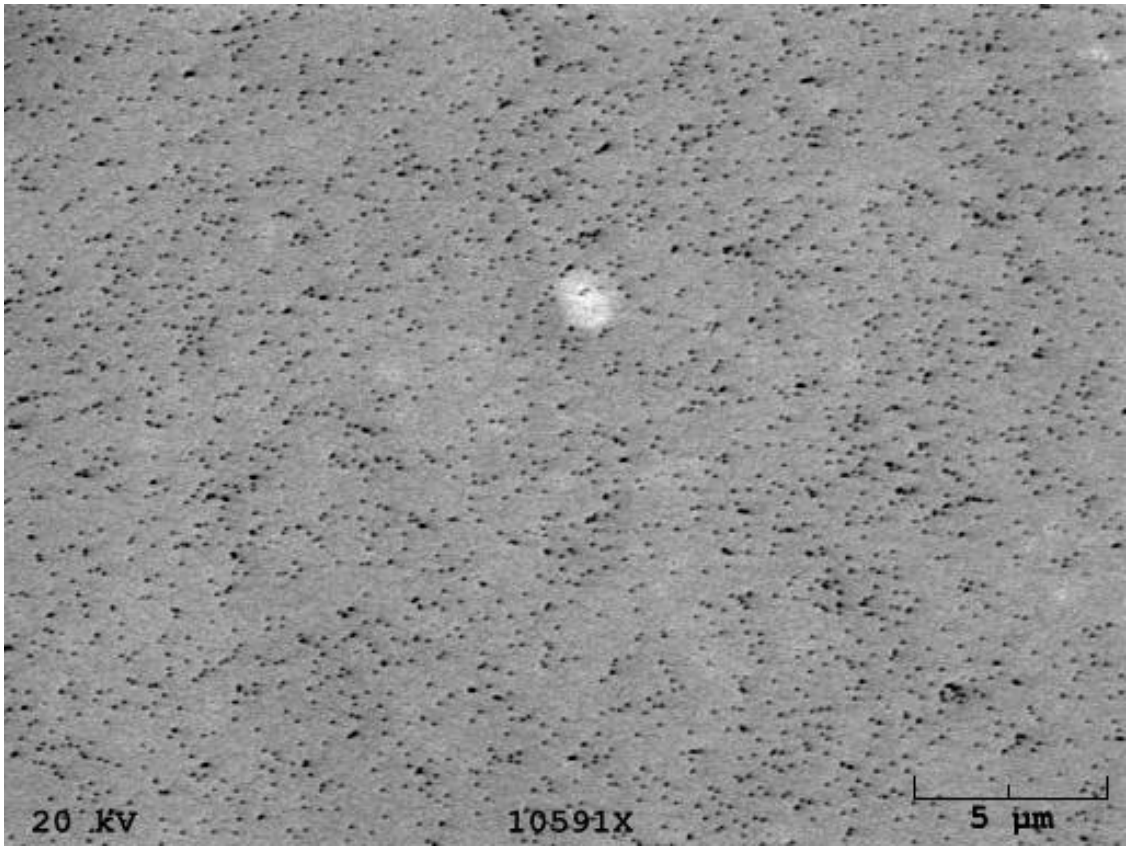
- The release current (5 ma) for the silica challenge test was significantly lower than the UPW background test current (25 ma). This is to be expected because colloidal SiO_2 has low charge potential compared to than natural occurring UPW contamination.

- Particles observed in the background/control UPW sample were typical of particles previously observed in UPW testing.

- Iron was documented in particle 4-3. Importantly, the Microfier was operating above recommended capture and release voltage (200/250v).

EXHIBIT A
Microfier Silica Challenge Test

TABLE OF CONTENTS



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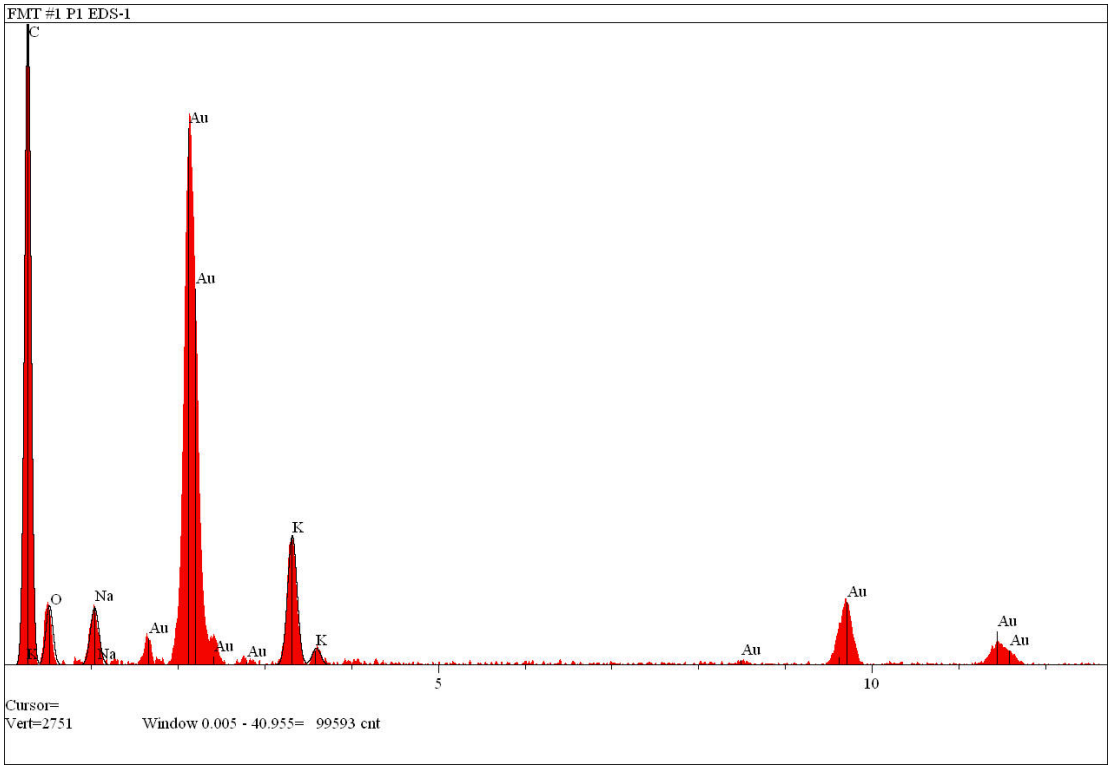

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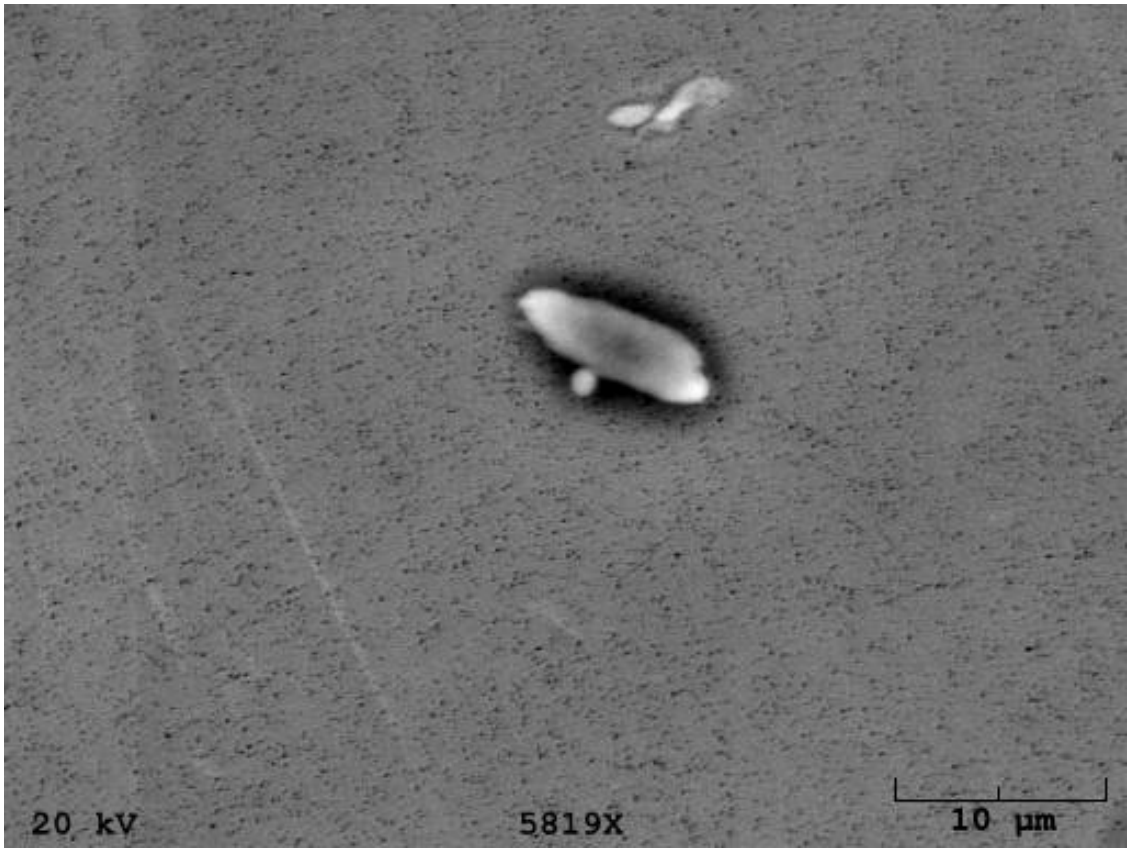

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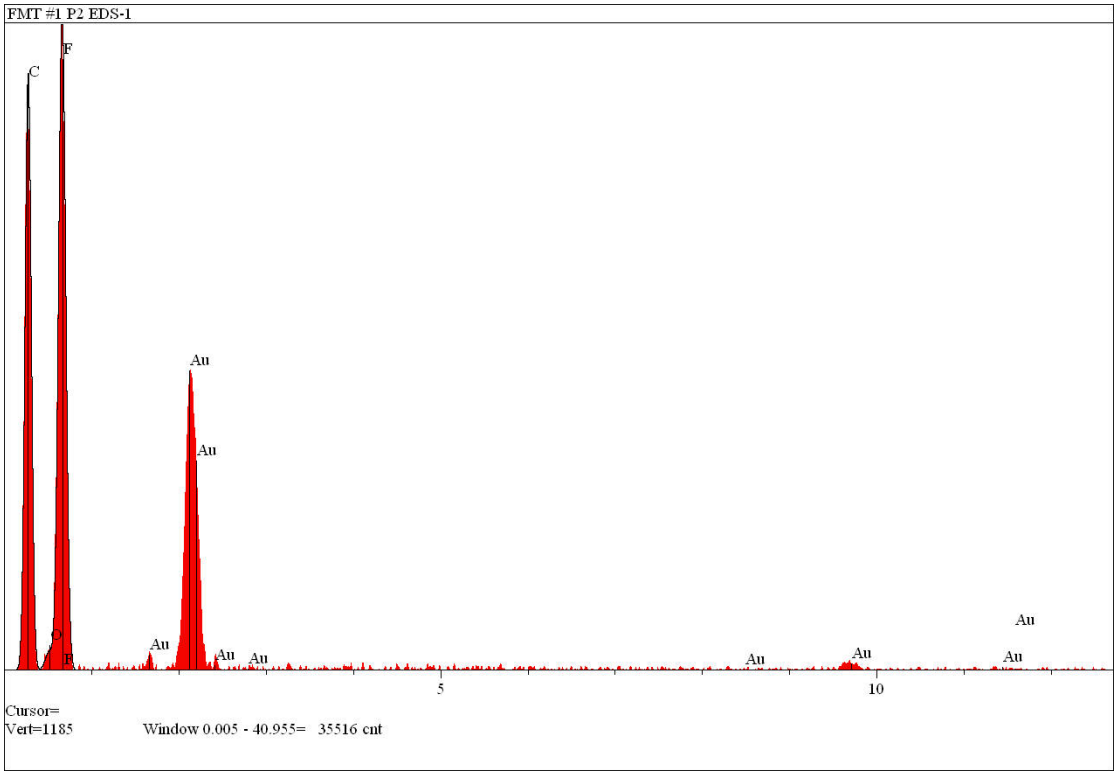

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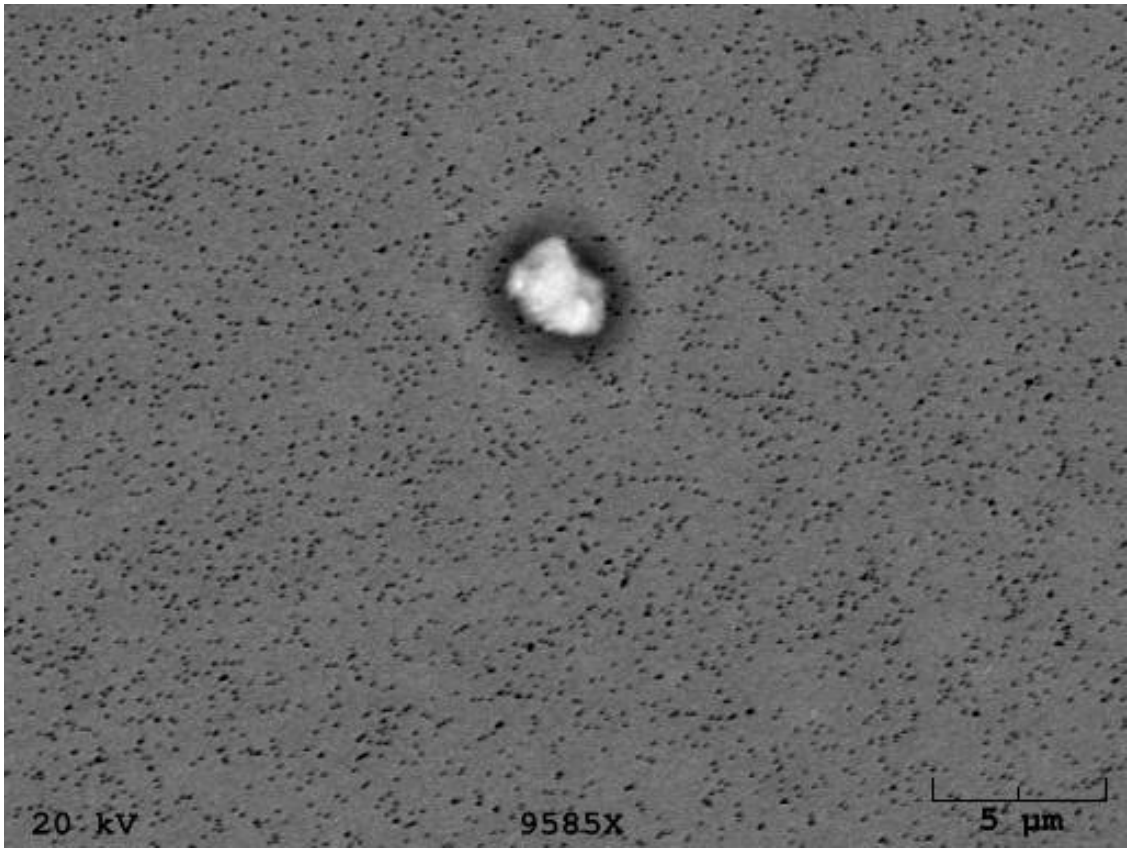

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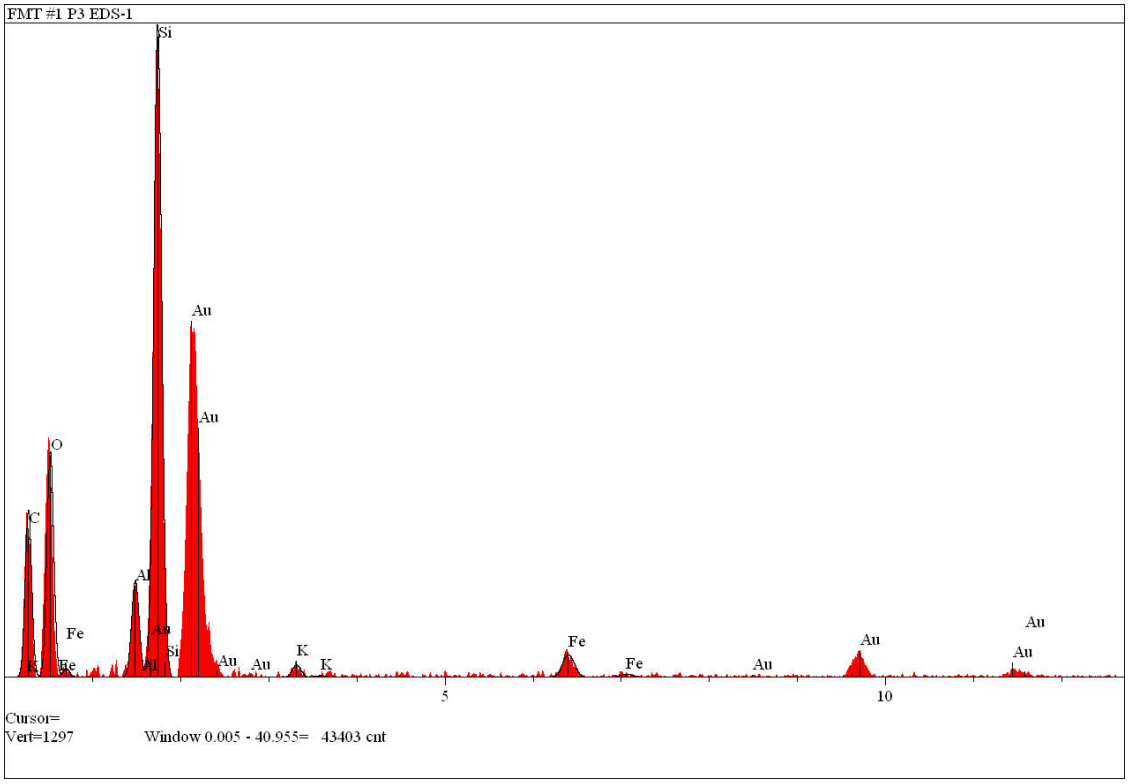

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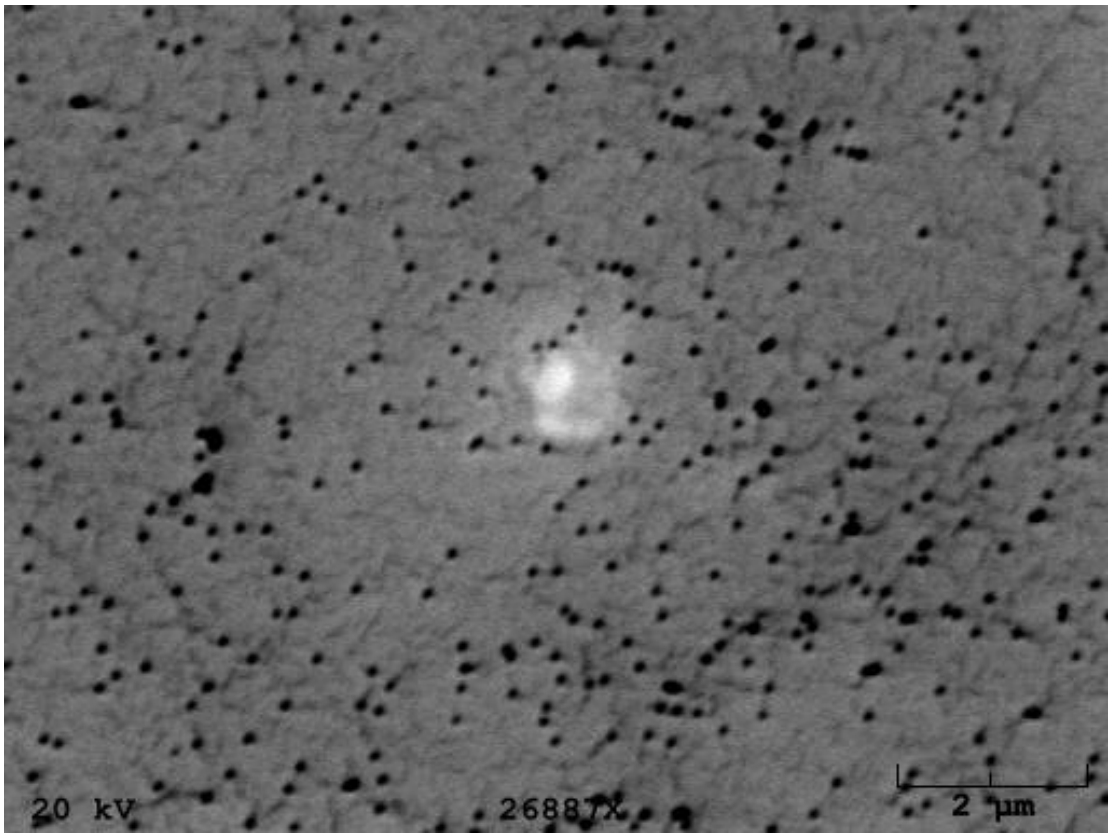
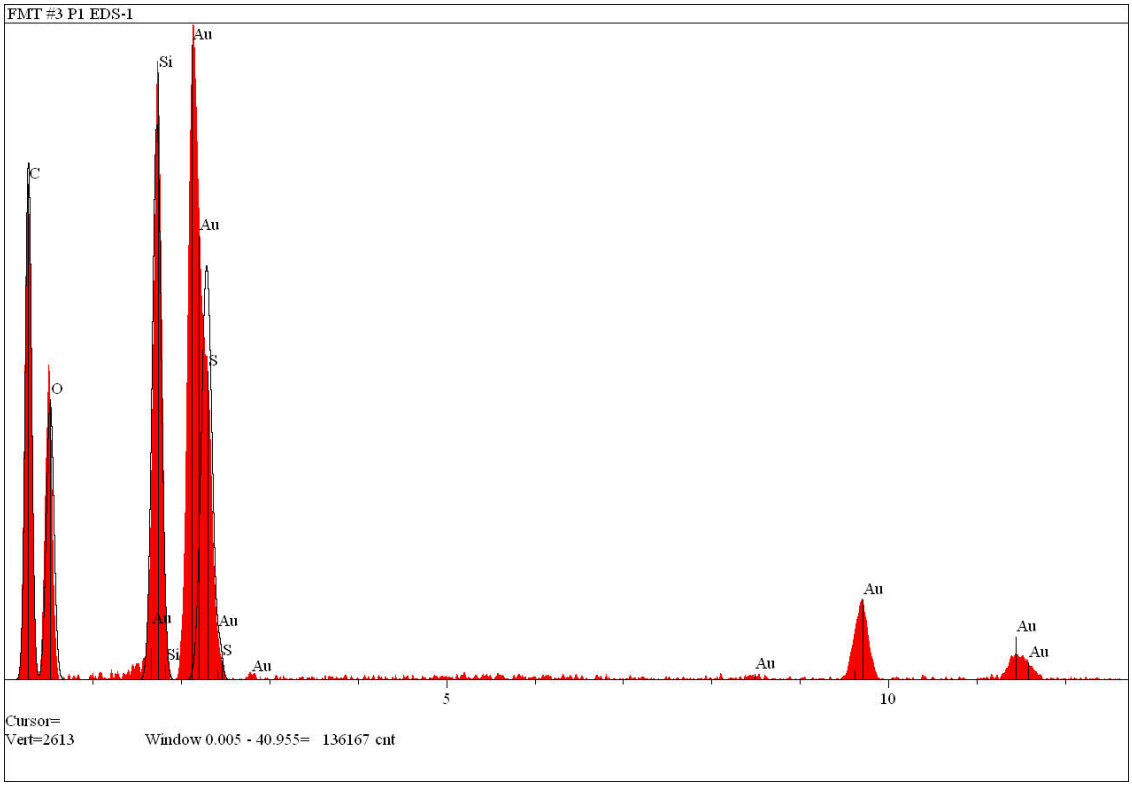


Particle 3-1: Silica Colloid Challenge at 100 Volts Capture and 150 Volts Release



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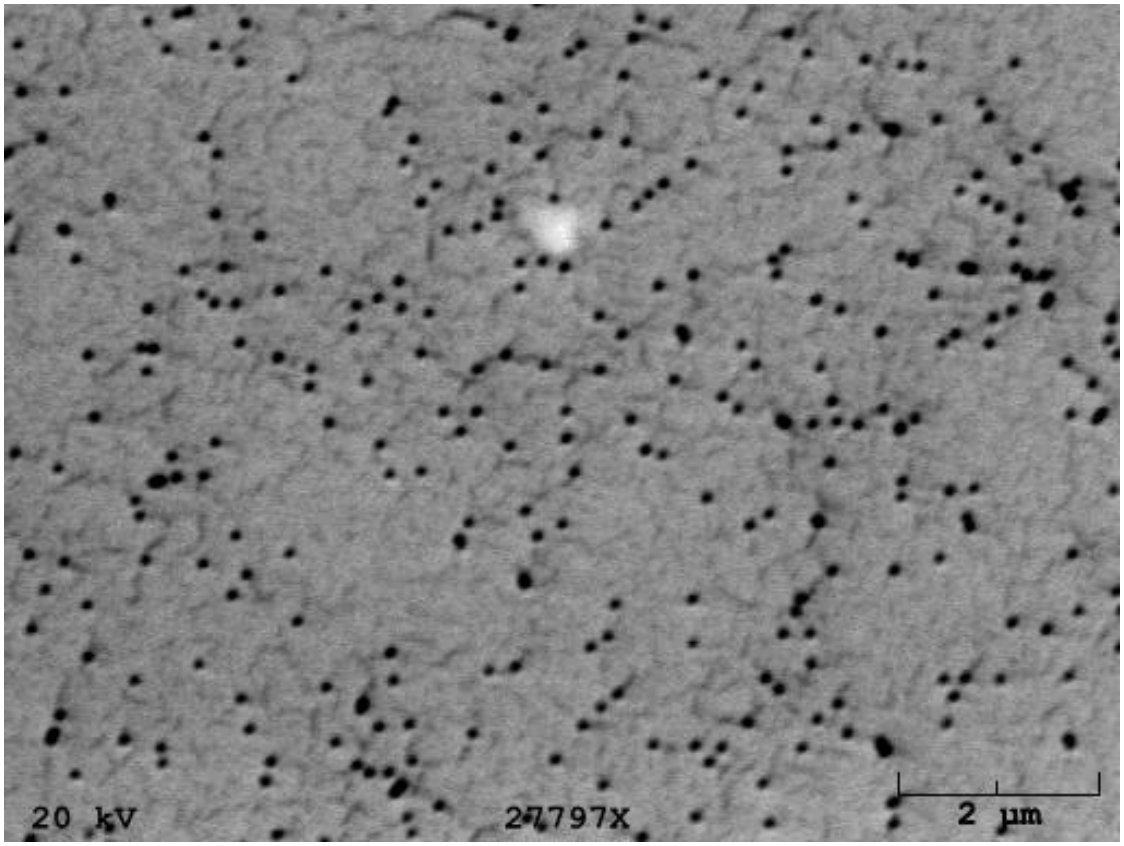

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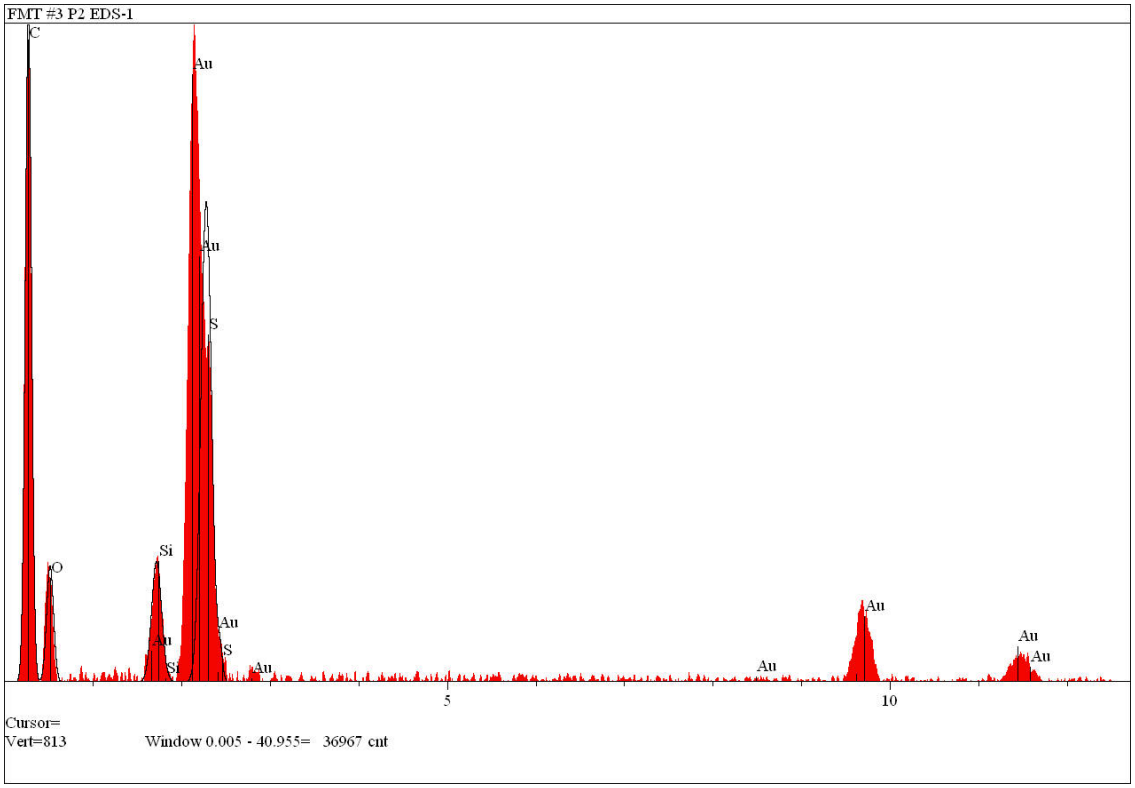

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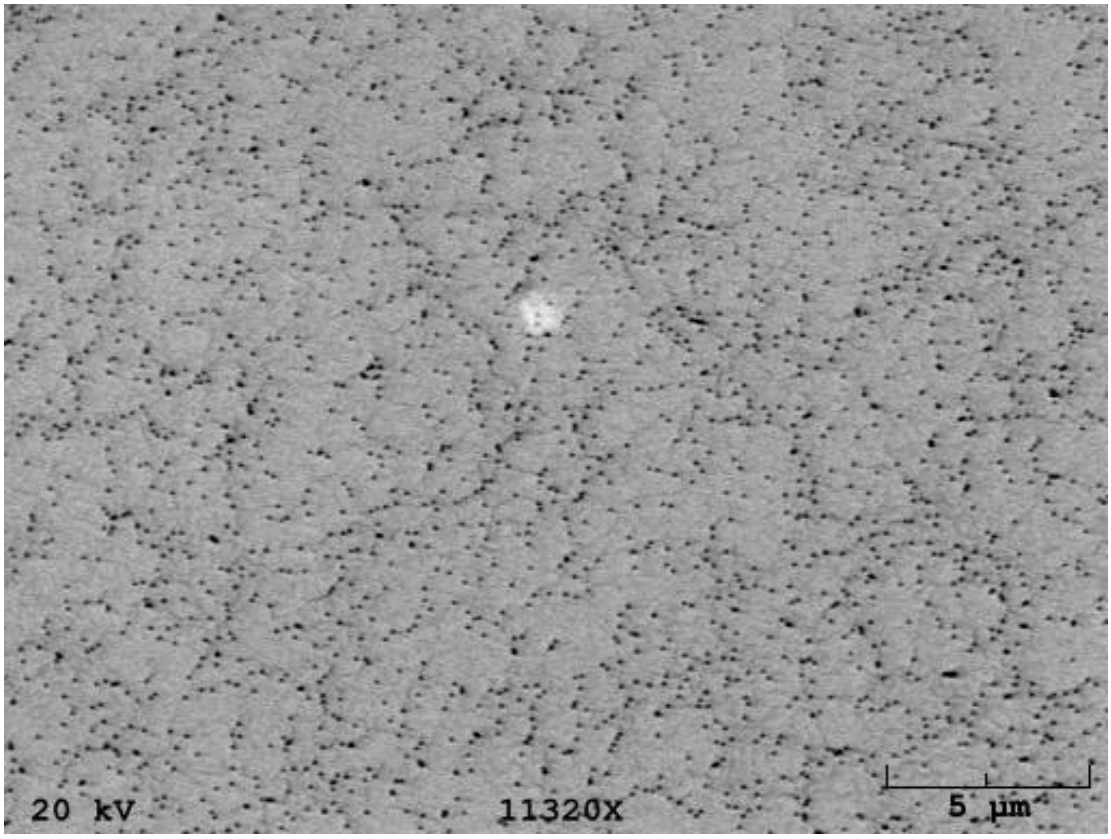

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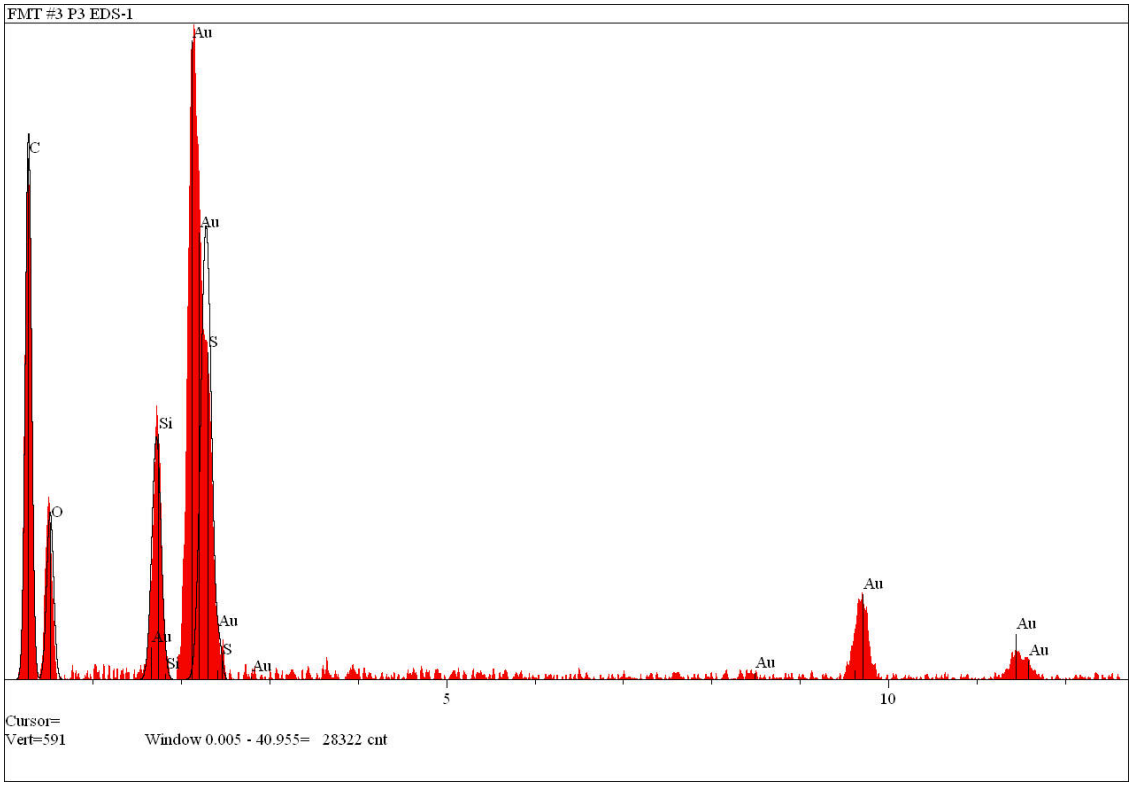

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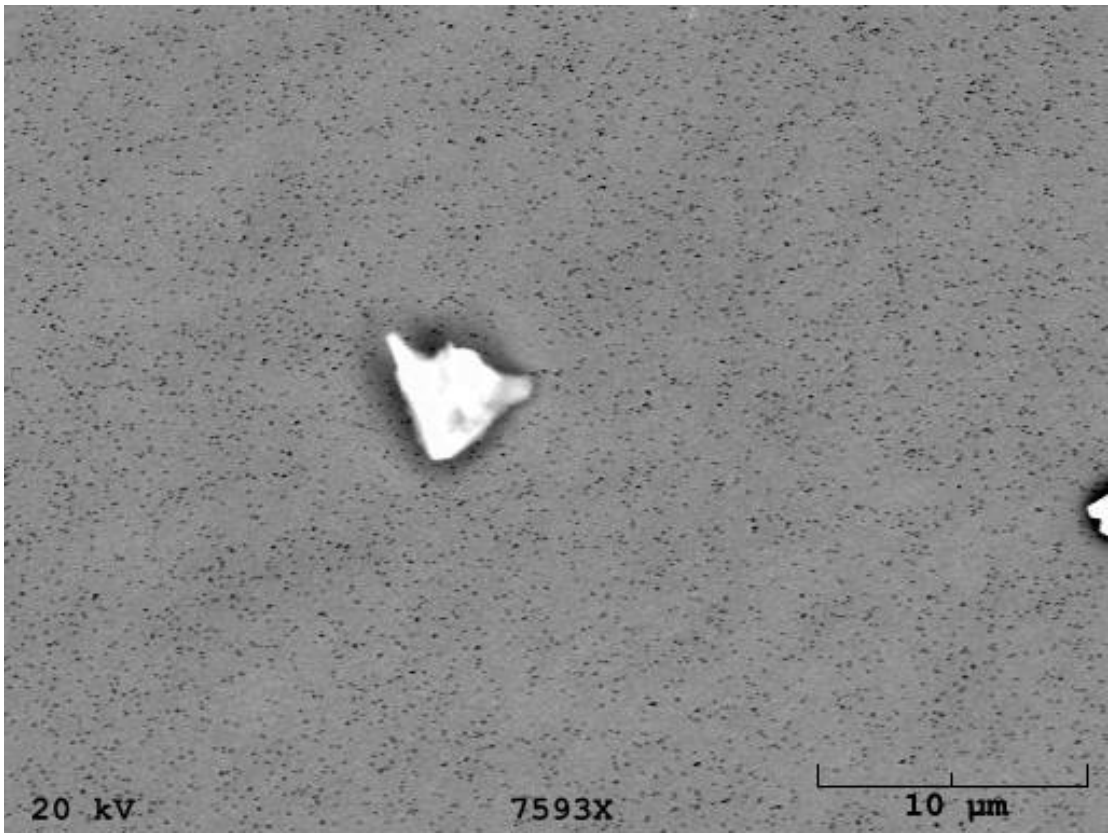
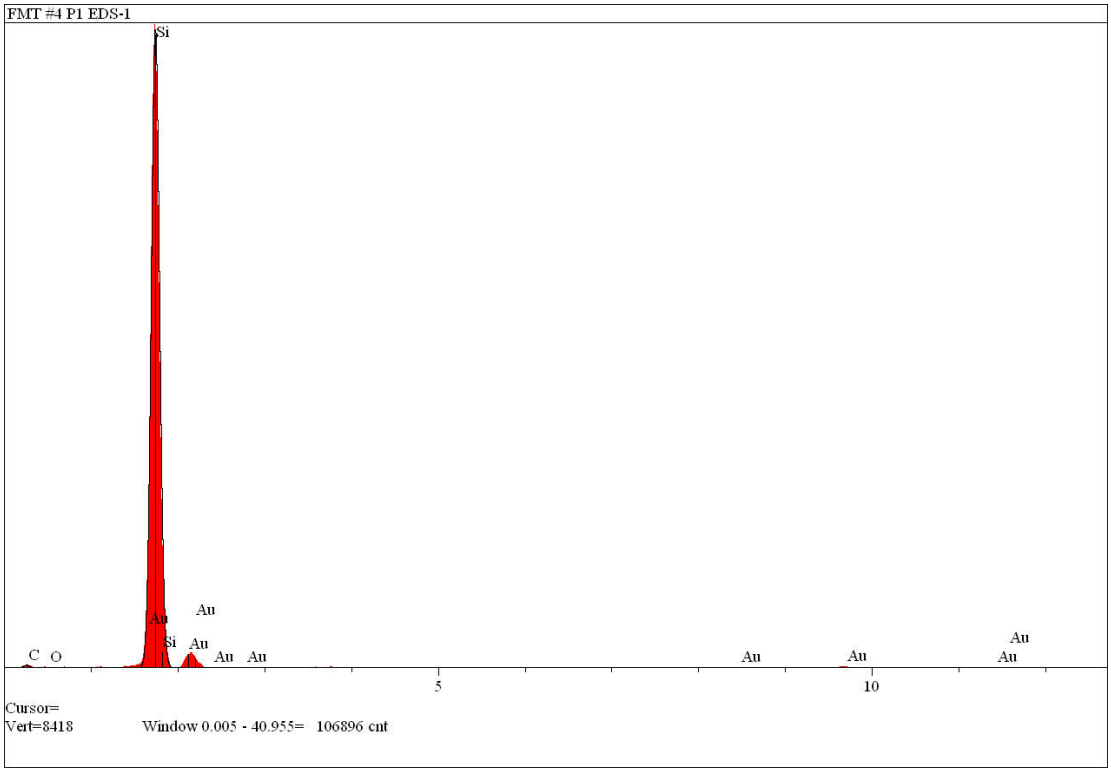


Particle 4-1: Silica Colloid Challenge at 200 Volt Capture and 250 Volt Release



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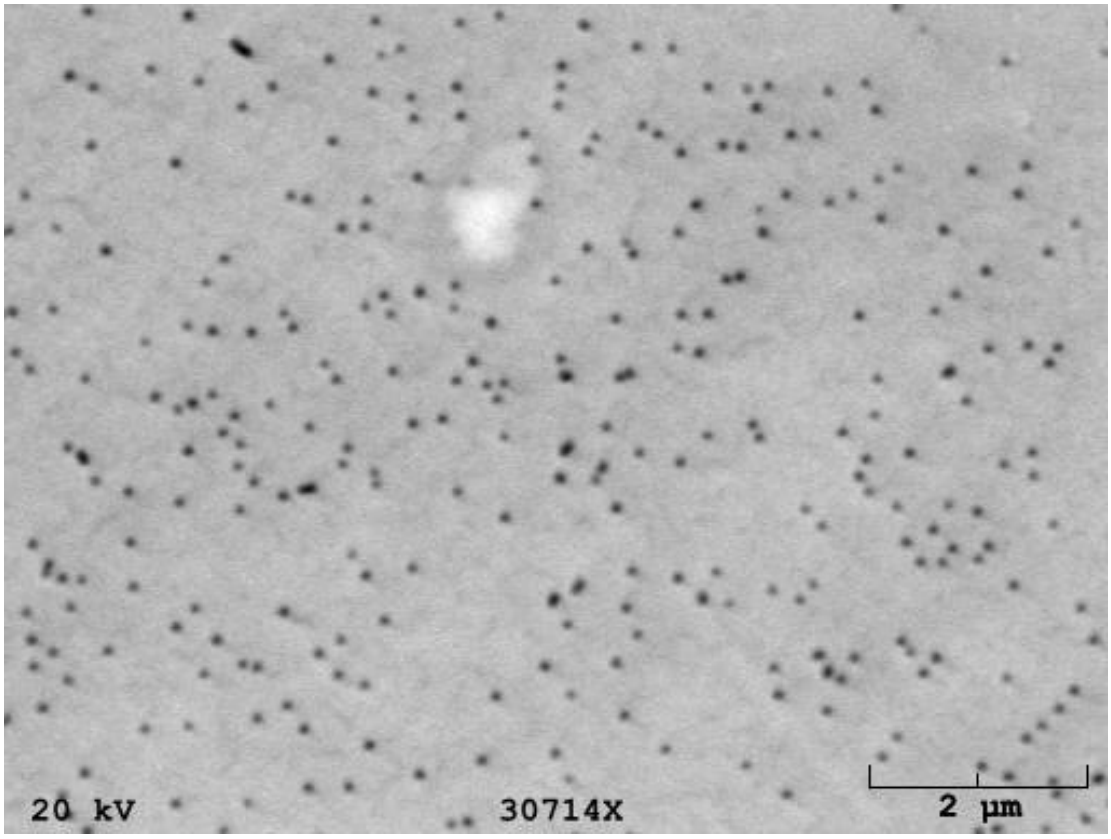

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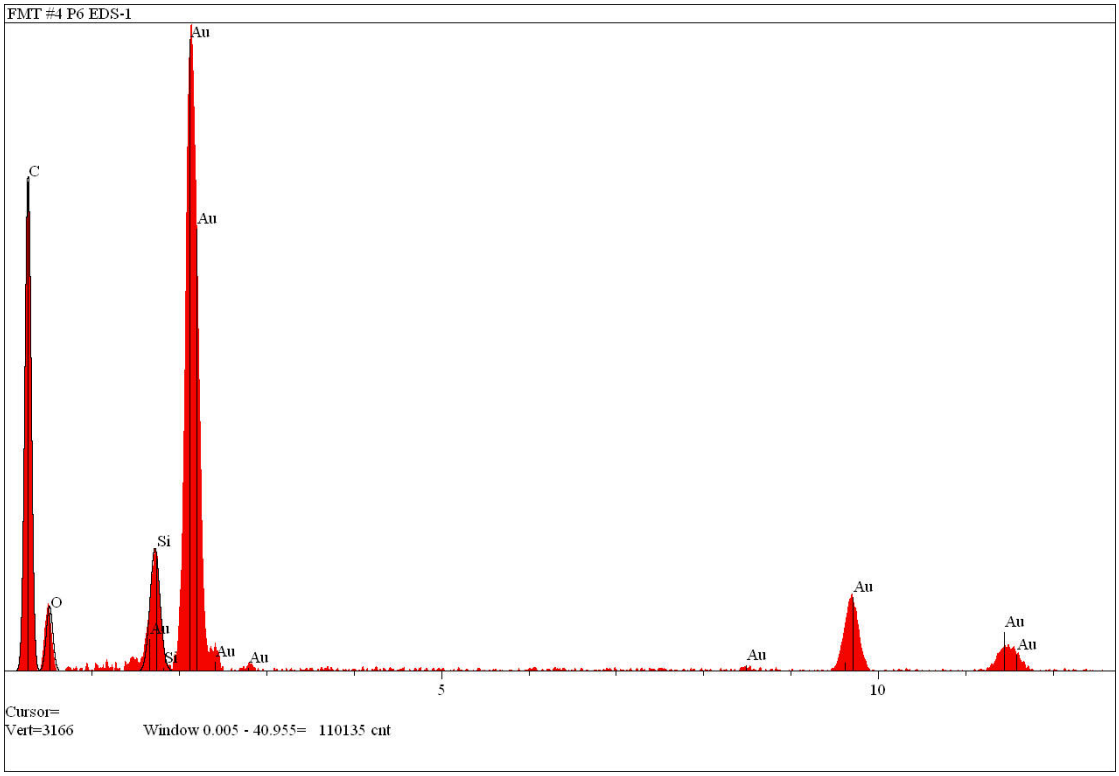

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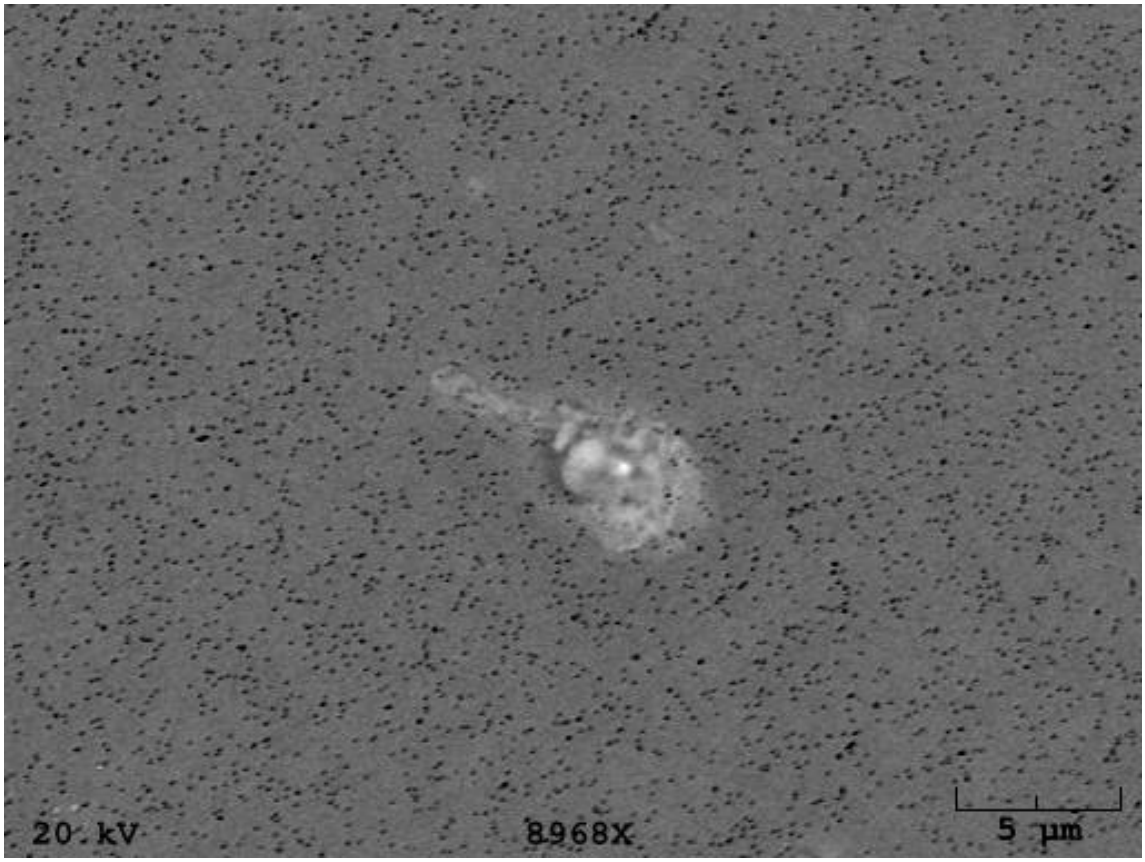

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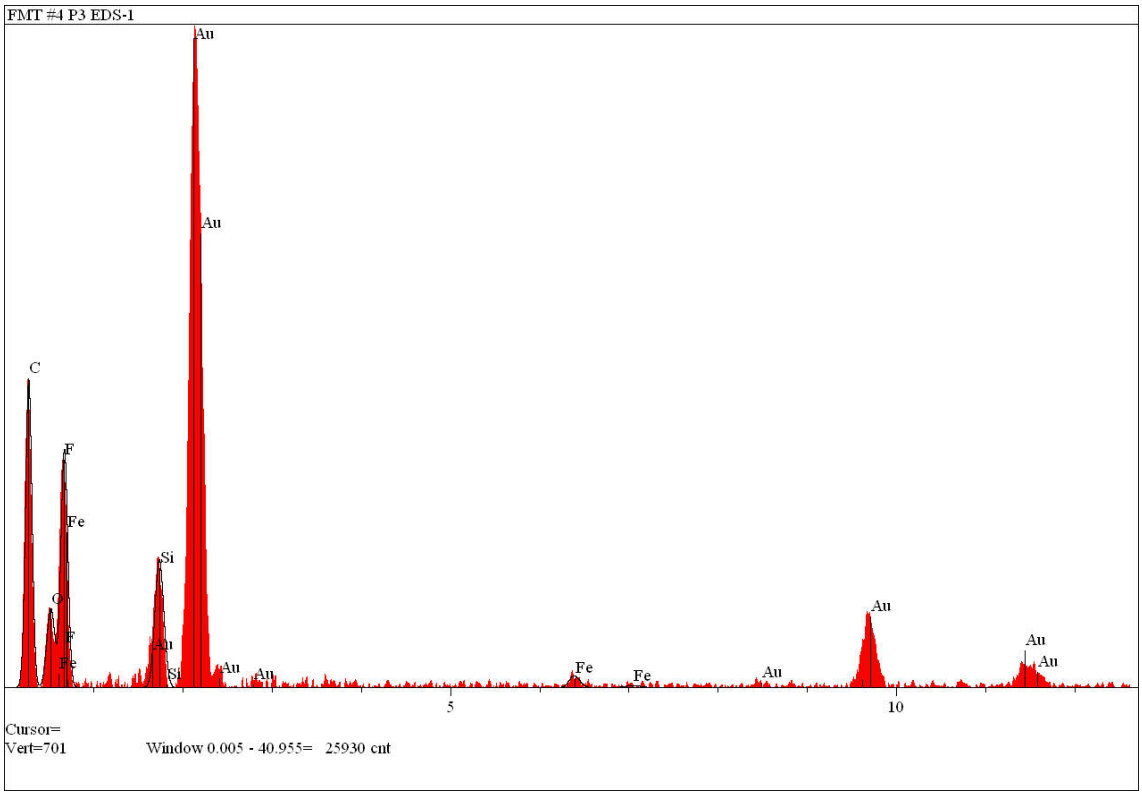

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