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A Study of the Effectiveness of Particulate Cleaning Protocols on Intentionally Contaminated Niobium Surfaces

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Abstract

Particulate contamination on the surface of SRF cavities is known to limit their performance via the enhanced generation of field-emitted electrons. Considerable efforts are expended to actively clean and avoid such contamination on niobium surfaces.

The protocols in active use have been developed via feedback from cavity testing. This approach has the risk of over-conservatively ratcheting an ever increasing complexity of methods which are found to result in adequate cavity performance in particular circumstances.

A complementary and perhaps helpful approach is to quantitatively assess the effectiveness of candidate methods at removing intentional representative particulate contamination. Toward this end, we developed a standardized contamination protocol using water suspensions of Nb_2O_5 and SS 316 powders applied to BCP'd surfaces of standardized niobium samples which resulted in particle densities of order 100 particles/mm^2.

From these common starting conditions, controlled application of high pressure water rinse, ultrasonic cleaning, or CO_2 snow jet cleaning were applied and the resulting surfaces examined via SEM/scanning EDS with particle recognition software. Preliminary results comparing these methods and selected parametric variations of each are reported.

Ultrasonic cleaning (US)

- Standard small US cleaner used
- Series with varied detergent concentration
- Series with varied US duration

Ultrasonic cleaning

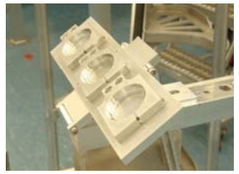
- Cleaning with only water removed 100% of Nb_2O_5 particles > 5 µm.
- US cleaning with only water removed 100% of SS particles > 15 µm, 96% of all SS particles.
- Cleaning with 1% Micro solution was comparable to water only (95%) for removing Nb_2O_5 particles 0.5 - 5 µm.
- US cleaning with 2% Micro solution removed 100% of SS316 particles.
- Cleaning with > 2% Micro solution was worse at removing Nb_2O_5 than water for all sizes sampled >0.5 µm.
- Removal efficiency of 1–8 µm Nb_2O_5 particles with 2% Micro independent of cleaning time was 94–98%.

High Pressure Rinse (HPR)

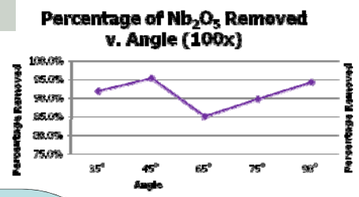
- Samples exposed to standard two-pass rinse, rotating around the spray as cavities do, duration 45 minute.
- Series with varied distance and fixed angle (60°)
- Series with varied angle and fixed distance (9 cm)

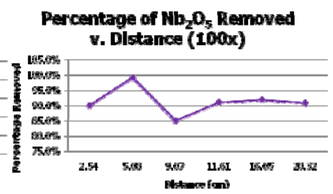
High Pressure Water

- 100% SS316 particles were removed in every HPR run.
- HPR cleaning removed ~90% of Nb_2O_5 particles for all sizes sampled (0.5–10 µm)
- HPR cleaning was more effective at 45° and 90° angle than 65° and 75° at 9 cm spray distance.
- HPR cleaning at 65° removed only 60-90% Nb_2O_5 particles 0.5 – 10 µm at distances 2.5 – 20 cm



Fixture for holding three samples during HPR





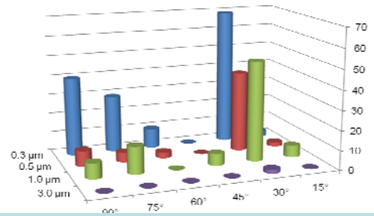
No particularly strong dependence on HPR angle / distance was observed

CO_2 Snow jet – Dry-ice cleaning

- Used hand-held dual-gas system with nitrogen gas shroud to impede condensation
- Standard near-to-far raster pattern and duration
- Series with varied incident angle and fixed distance (2 cm)
- Series with varied distance and fixed angle (60°)

CO_2 snow jet

- 100% SS316 particle removal for all angles and distances tested.
- Near 100% removal of Nb_2O_5 particles.
- N_2 blow-off into laser particle counter technique used to sample the entire surface for further relative measurements:
- More effective cleaning Nb_2O_5 at 45° angle than 30°, 60°, or 90° for particles < 3 µm.
- Equally effective cleaning Nb_2O_5 particles at distance 1.0 – 2.5 cm, but degraded quickly with further increase in distance.
- Significant redeposition of Nb_2O_5 particles from a contaminated sample to a clean witness sample 3 cm distant via CO_2 snow jet cleaning of the former was easily demonstrated.



45° incident angle was most effective at removing submicron Nb_2O_5 particles

Controlled Initial Contamination

The standardized particulates

Commercially available Nb_2O_5 and SS316 powders

Nb_2O_5 powder grade HPO 400 from H.C. Stark Inc.

Particle size: **0.3 to 31 µm**

SS316 powder from Goodfellow Inc.

Particle size: **8 to 50 µm**

Controlled distribution of particulates onto sample surface

Standardized suspension in water

Aim to maximize density and particle aggregation

Nb_2O_5 – 8.2 mg/liter

SS316 – 698 mg/liter

0.5 ml of the suspension metered onto Nb sample surface

Allowed to air dry in cleanroom

Standardized particle counting using scanning SEM/EDX system

Sampled five areas on each sample

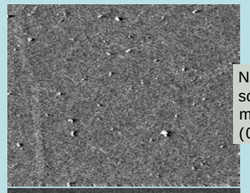
Elemental composition used to identify relevant particles

Starting contaminated conditions

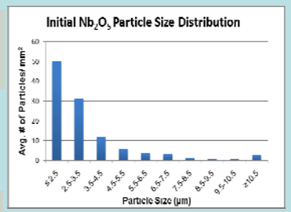
~110 particles/mm^2 - Nb_2O_5

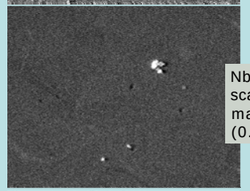
~13 particles/mm^2 - SS 316

Five niobium samples with 0.5 ml particulate/water suspension drying in cleanroom air

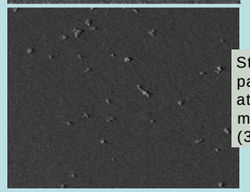


Nb_2O_5 particles scanned at 100x magnification (0.841 mm^2).

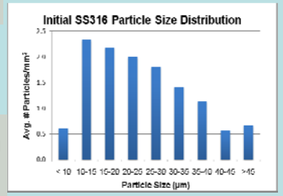




Nb_2O_5 particles scanned at 750x magnification (0.01mm^2)



Stainless steel particles scanned at 50x magnification (3.37 mm^2)



Comparisons

- Particles of Nb_2O_5 and SS316 > 5 µm were thoroughly cleaned by all techniques except HPR, which left some Nb_2O_5 particles on the surface.
- Particles of SS316 < 5 µm were not tested.
- No clear dependence on nozzle-sample distance was found for HPR, although tests at the most effective angles, 45° and 90°, have not yet been performed.
- CO2 snow jet cleaning of these Nb_2O_5 particles appeared to be significantly more effective than the other two techniques tested.

Lessons for future work

- Consider revisiting the starting suspension concentrations with a view to increasing by factor of >10 to improve the counting statistics.
- Continue the HPR study of effectiveness with distance using 45° and 90° angles.
- Repeat the Nb_2O_5 study with optimal EP-treated Nb samples for comparison with BCP surfaces
- As redeposition is a distinct possibility with both US and CO_2 snow jet cleaning, investigate the effectiveness of a sequence of fresh cleaning solutions, to approximate use of a flowing, filtered solution for cavity applications, and a purge gas flow in the CO_2 snow jet cleaning application to carry away removed particles.

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