X-Ray mirror carbon contamination removal tests at the ESRF

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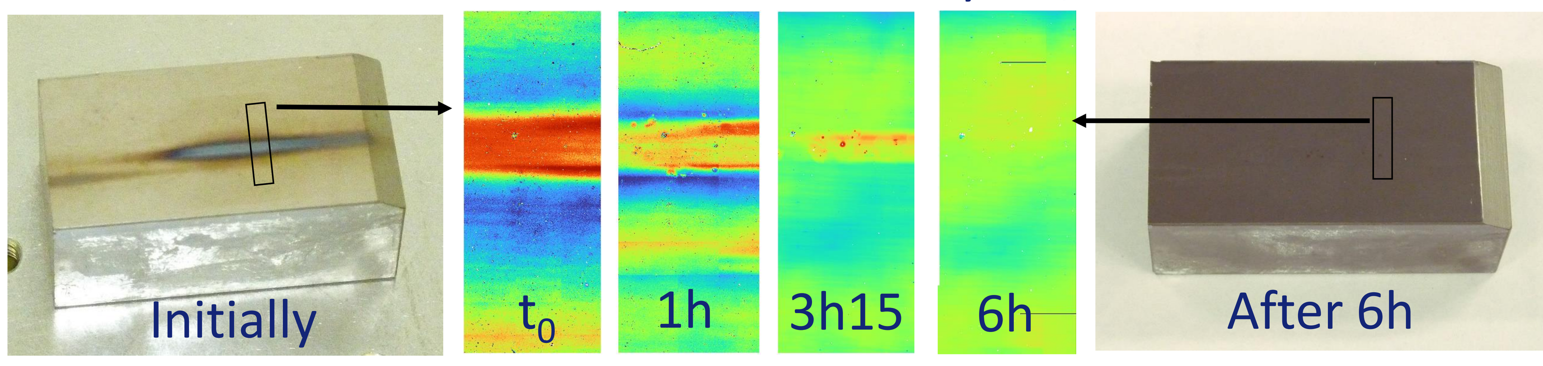
The performance of reflective optics such as X-ray mirrors or diffraction gratings generally degrades following exposure to high intensity X-ray or EUV beams. The most common degradation phenomenon is beam-induced contamination with the formation of inhomogeneous carbonaceous films on the optical surface. Various light sources (e.g. NSLS/BNL[1], SSRL[2], SRC[3], APS[4], SOLEIL[5], ALS[6], INDUS[7], ALBA[8], SPring-8[9]) have investigated strategies to mitigate or remediate such contamination in- or ex- situ. In order to build in-house expertise for the refurbishment of contaminated mirrors we have performed tests on several cleaning and remediation methods: UV-Ozone exposure, oxygen plasma treatment and stripping of various different coating materials. The impact of such treatments on micro-roughness and X-ray reflectivity measurements is presented.

UV Ozone exposure

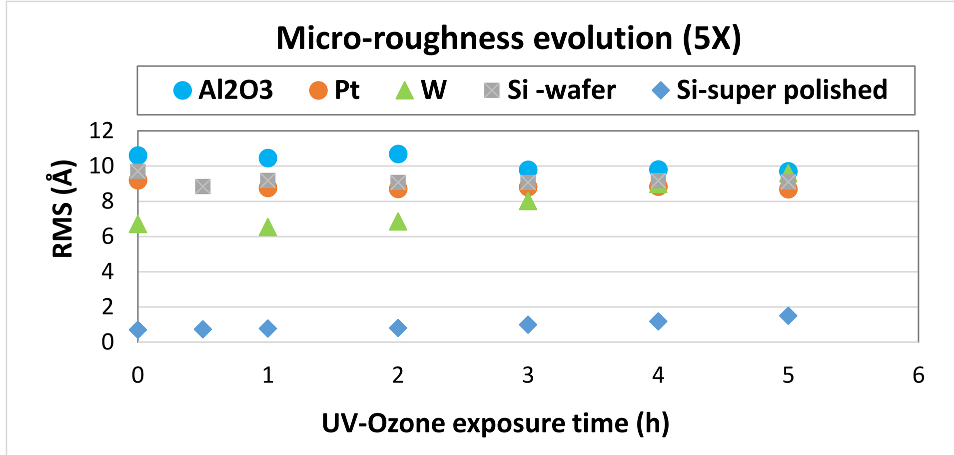
Low-pressure quartz Hg-vapour lamp, generates UV emissions in 254 and 185 nm range.

- Ozone and atomic oxygen are generated.
- Organic contaminant molecules are excited or dissociated by the absorption of 254 nm wavelength UV.
- Excited organic contaminants react with the atomic oxygen to form volatile products such as CO_2, H_2O, etc.
- Essentially room temperature process

➢ Si monochromator decontamination by UV/Ozone



➢ Evaluate effect of extended ozone exposure upon optical micro-roughness of various coatings and Si substrates (spatial periods from 0.004 to 1.3 mm)



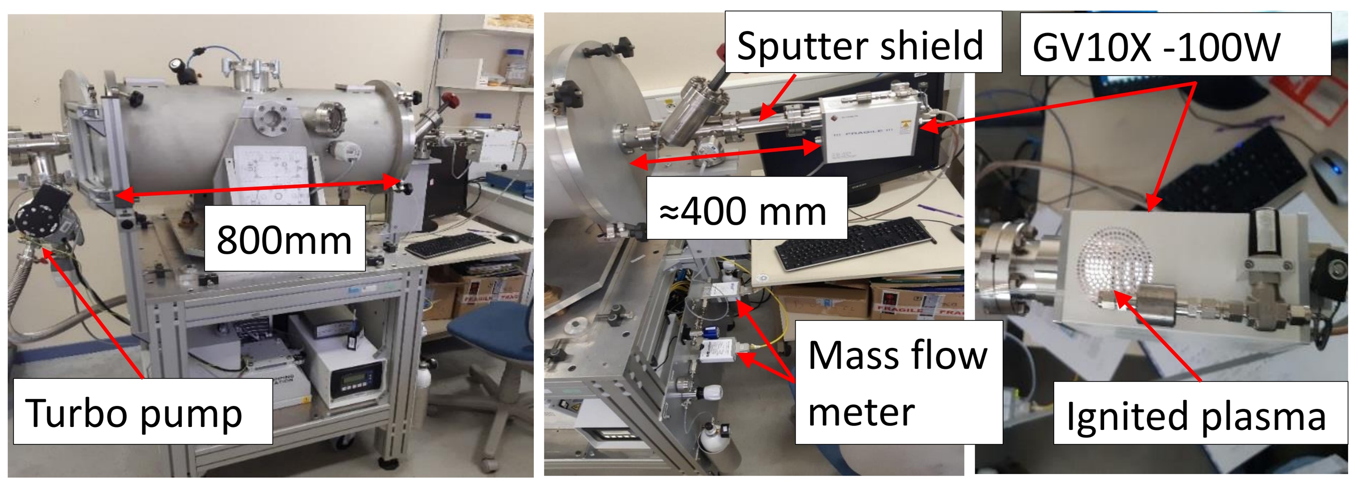
Oxygen Plasma treatment

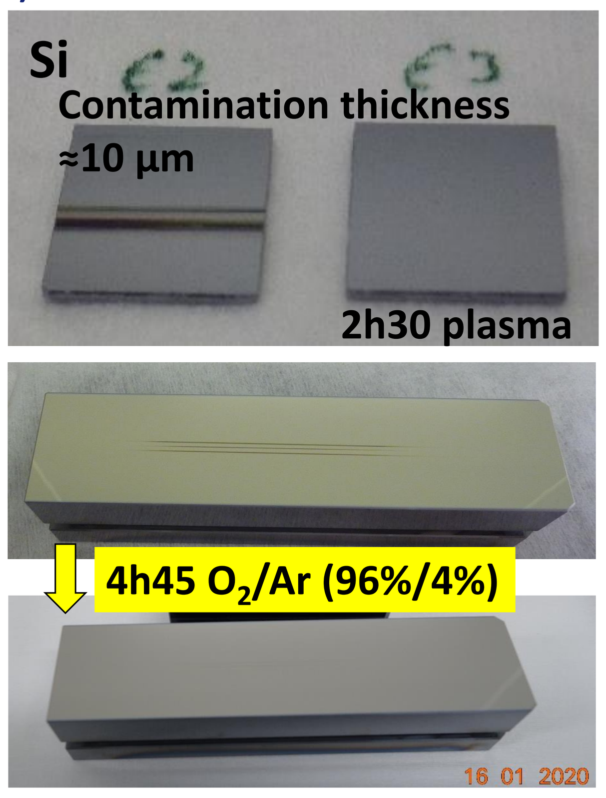
Ex-situ equipment for cleaning mirrors up to 750 mm based on the system developed at ALBA consisting in a low-pressure RF oxygen plasma fed into a vacuum chamber.

- 100W plasma source GV10X (ibss Group Inc)
- Source power, gas flow and pressure controlled ($P \approx 10^{-3}$ mbar)
- UV/ViS spectrometer for plasma characterization
- Process gas: O_2 (96%)-Ar (4%) possibly H_2 in the future

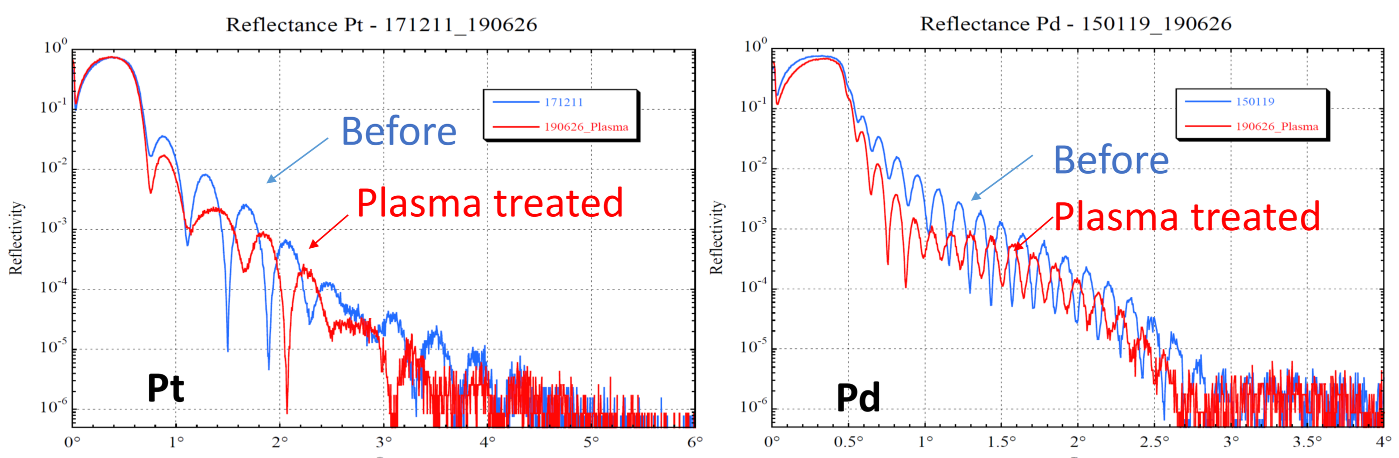
➢ First conclusions:

- Oxygen plasma allows removal of surface carbon contamination on Si mirrors without degrading micro-roughness.
- Pt, Pd, Rh, Ni, W/B4C micro-roughness not affected.
- Removal rate increase with Ar (4%) - 10 times faster.
- Removal rate higher when closer to plasma source (for long mirrors - flip to reduce overall exposure time).





➢ X-ray reflectivity spectra show changes in the surface whatever the coating. Cleaned surfaces are well simulated by slight adjustments of an oxide top/main layer density/ thickness. Impact of cleaning on critical angle remains small.

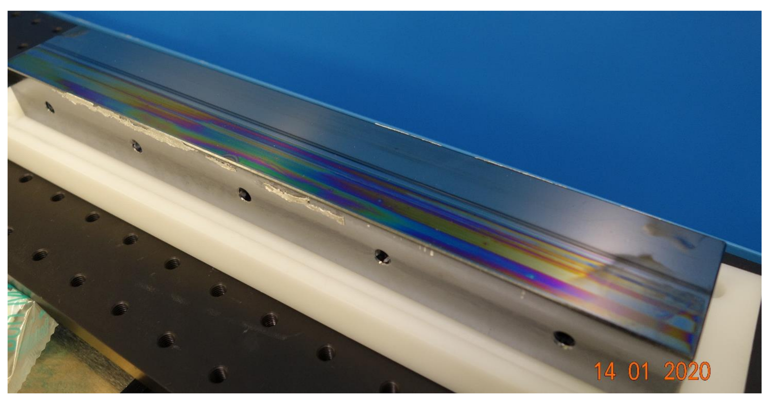


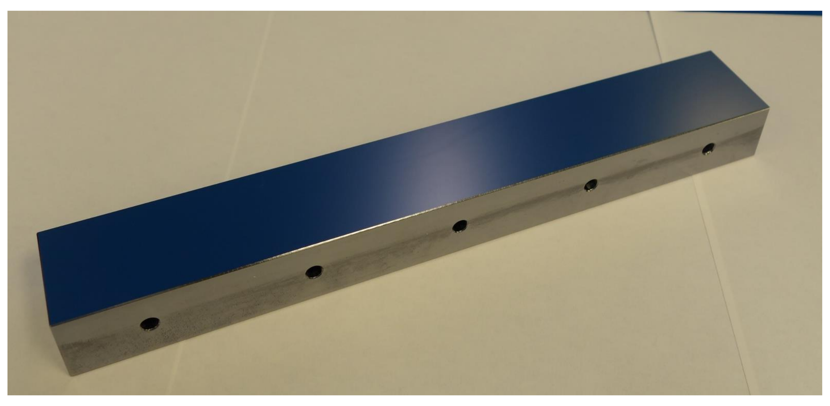
Stripping coatings

Following APS recipe based on Chromium etchant solution.

Chromium Etch 1020: dilute nitric acid and cerium ammonium nitrate
Rinsing with Transene 100 solution

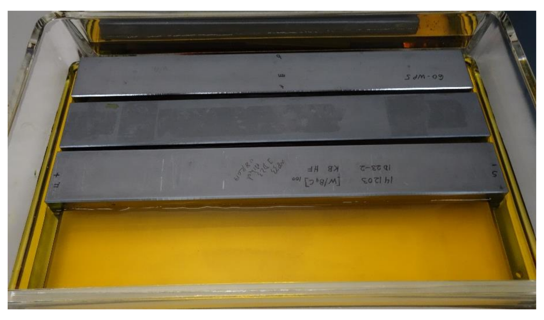
The solution can also etch other metals like Al, Ni, Ru, Ti, … [10]

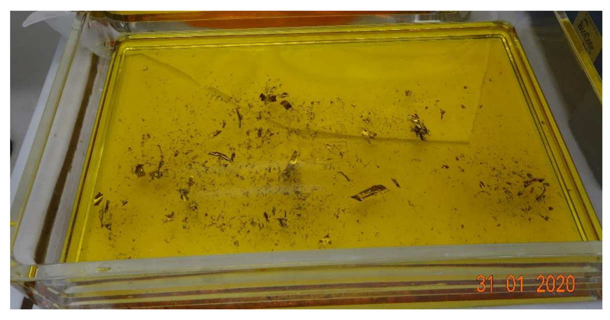




➢ About 40 mirrors treated, some without Cr binder layer:

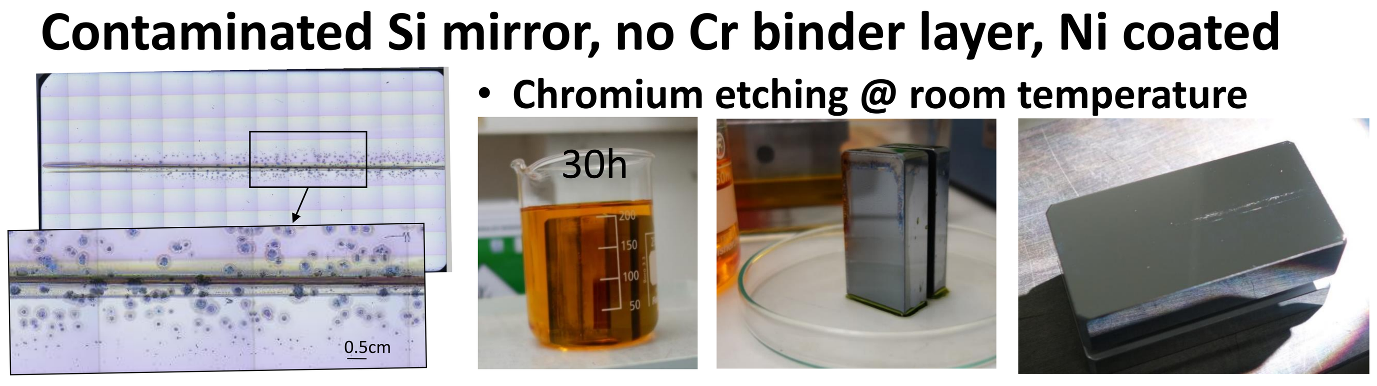
- Initial micro-roughness appears to be recovered after stripping except at coating boundaries
- In some instances coating residuals may remain stuck to the surface, even for mirrors that have never seen X-rays independently of Cr binder layer presence. Optimal cleaning recipe still to be determined.
- Etching solution loses efficiency with age (expiry date ≈1 year)

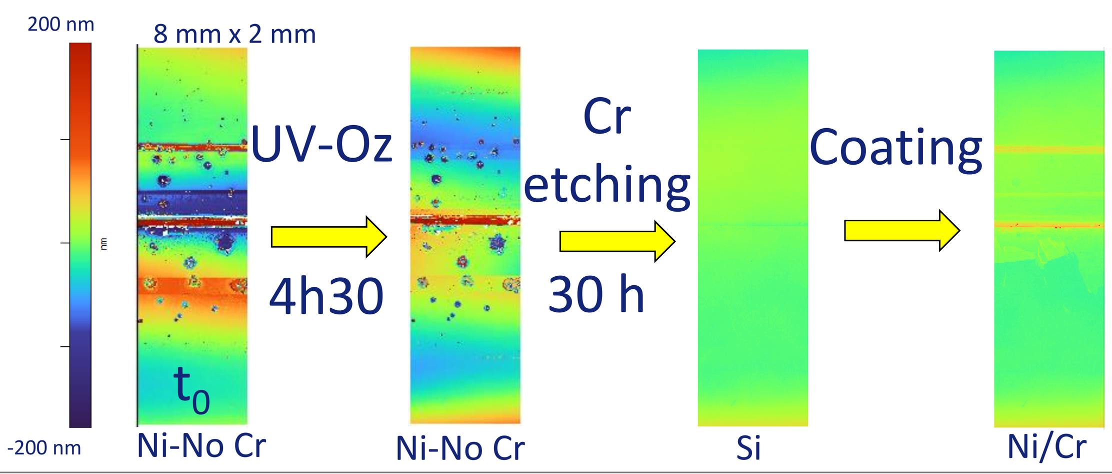


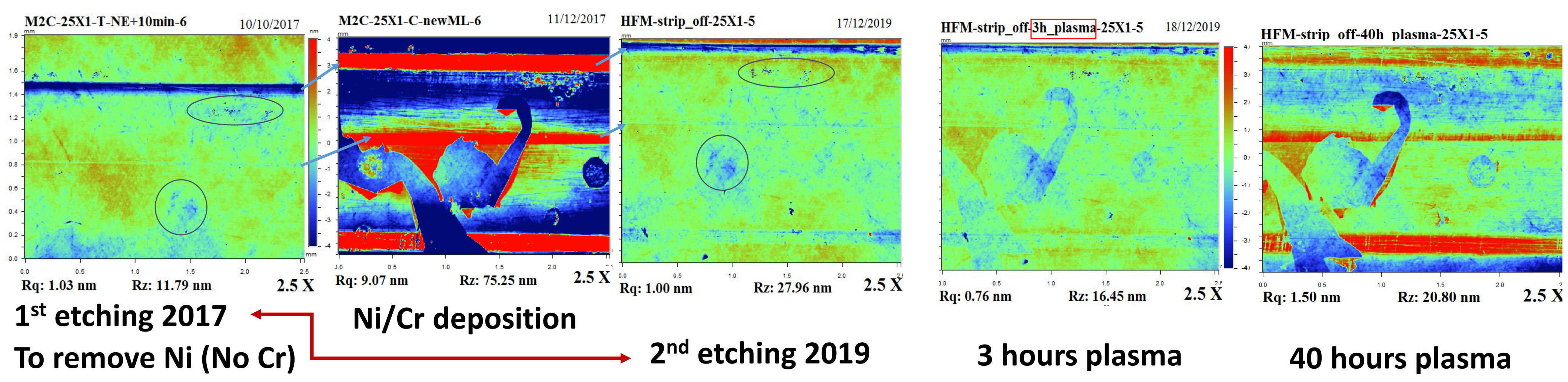


Contaminated Si mirror, no Cr binder layer, Ni coated

- **Chromium etching @ room temperature**







➢ New coating reveals residual surface deposits, that are transparent to micro-interferometer measurements.
These transparent remaining layers are also revealed by O_2 plasma exposure…
Our etching process needs further investigation.

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