Sphere polishing and surface properties

with contributions from WG´s:

5.56 "Manufacturing Technology": manufacturing of spheres
5.41 "Interferometry on Spheres": sphericity
5.14 "3D Roughness Metrology": surface roughness
7.11 "X-ray Radiometry": surface roughness
4.33 "X-ray Optics": crystal lattice, orientation and contamination

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OUTLINE:

- manufacturing of spheres
- basic results for sphericity and roughness
- metall contamination analysis by XRF
- crystallographic orientation by Laue method
- subsurface damage by two crystal XRD

nothing about: Oxide growth, stability, stoechiometry, homogeneity, density and thickness
Manufacturing-Chain:
Step 1: Cutting with diamond-plated tool
Manufacturing-Chain:
Step 2: Turning with PCD (poly-crystalline diamond) tool
Manufacturing-Chain:
Step 3: Lapping

- Alumina, suspended in aqueous solution

Several measurements of form and topography for control of processes
Manufacturing-Chain:
Step 4: Polishing

Media:
• Colloidal Alumina
• Colloidal Titanium-dioxide for finish
• in pitch calottes

Several measurements of form and topography for control of processes
Measurement of sphericity, WG 5.41

Topography; Sphere AVO28-S5c, peak-to-valley 98 nm
Silicon surface and analysis methods

<table>
<thead>
<tr>
<th>Surface</th>
<th>Method</th>
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<tr>
<td>Carb. Cont.</td>
<td>XRF, XPS</td>
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<td>Water</td>
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<tr>
<td>Metals</td>
<td>XRF, XPS, XANES</td>
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<tr>
<td>Si oxide</td>
<td>XRR, XRF, Ellipsometry, Gravimetry</td>
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<tr>
<td>Ni/Cu silicide</td>
<td>Interf.-mikroscopy, XRR (Interface !)</td>
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<td>Si crystal</td>
<td>XRD</td>
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</table>
Measurement of surface roughness, WG 5.14

Typical topography; Sphere Si28-S5c, roughness below 0.3 nm

Messposition 1.MMD

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Area: 416.0 x 312.0 µm

Smooth Phase (grün)

Sq: 0.260 µm
Sa: 0.207 µm
St: 5.568 nm

Points: 307200
QUARTIC

WG 5.14 Atos Micromap
e.g. sphere Si28-S5: procedure after metal contamination removal

![Graph showing reflectance vs. grazing incidence angle with points labeled for original reflectance at 10°, 10 min Syton soft polishing, 4 min Syton soft polishing, and 2 min Freckle etch.]
XRF > 1 keV: metal contamination, WG 4.33

Between 4 and 12 keV:
K-shell XRF from K to As
L-shell XRF from Cd to Pb
detection limit about $10^{13}$ atoms/cm²
XRF > 1 keV: metal contamination, WG 4.33

contamination and polishing history of Si28-S5c

XRF-spectra of the Ni and Cu contamination on Si28-S5:
- CSIRO-ACPO polished
- Freckle etch 1 min
- Freckle etch 2 min
- therm. oxidized (7 spectra)
- PTB polished

2×10^{15} \text{ atoms/cm}^2
Laue orientation, WG 4.33

Image plate scanner
Å min. pixel size 12.5 µm
Å max. image plate 30 x 40 cm²
Å 16 bit grey levels
Observation of polish steps, WG’s 5.56 and 4.33

Si (800) rocking curves, Mo Kα

- W22, polished, 1st step (diamond powder)
- W22, polished, 2nd step (Syton, 1 µm removed)
- W22, polished, 3rd step (Syton, ? µm removed)
- W22, etched 100 µm
Rocking curves of Si28-S5c and PTB 12-03

The linear plot of the rocking curve center Ǻ shows no differences to deeply etched WASO 05. Ǻ follows the calculated rocking curve.

The logarithmic plot of the wings ǻ shows no differences to deeply etched WASO 05. ǻ does usually not follow the calculated curve:
- (in)elastic scattering
- thermal diffuse scattering
- diffuse defect scattering
- lattice deformation close to the surface/interface
Three crystal diffractometer: reciprocal space map

Simulated reciprocal space intensity maps from (a) stacking faults, (b) perfect dislocations and (c) Shockley dislocations with radius 7000Å from: X-ray Diffuse Scattering from Dislocation loops in Czochralski Grown Silicon Wafers, by: P. Klang and V. Holý
The new polishing methods of PTB WG 5.56 lead to spheres of very high quality. (sphericity, roughness, metal contamination, subsurface damage) These spheres can now be routinely analysed by the combined XRF, XRD, Laue set-up.

Improvement of the analysis is possible with the goal of a deeper insight into the diffuse scattering:
- by adding a third crystal to the diffractometer
- by measurement of the "Reciprocal-space-map"

• with an enhanced XRF analysis area
  - by a larger detector area 25 → 100 mm²
  - by sphere rotation during measurement

Thank you very much for your attention!