Processing Effects and

Use of Electrochemical Abrasive Jet Polishing for Nb-SRF Cavities

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Introduction

Three critical steps in successfully polishing SRF-cavities:

- Discovering how various cavity manufacturing steps impact surface chemistry and structure
- Linking the surface chemistry and structure to material removal rates by chemical and mechanical methods
- Creating a process to time-effectively create a near-perfect ordered and clean surface on the curved, assembled cavity

Overview

Manufacturing

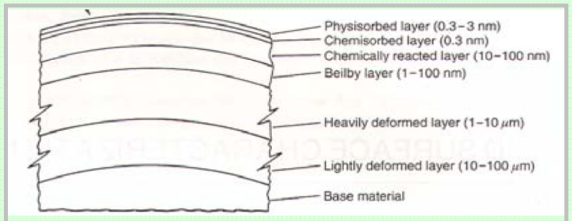
- Bending ⇒ roughness, crystal texture
- Welding ⇒ crystal texture
- Deep-drawing / hydro-forming ⇒ roughness, crystal texture
- Deep-drawing / hydro-forming ⇒ Hardness

Techniques

- Electropolishing
- Buffered Chemical Polishing
- Tumbling
- UHV heat treatment (H, O, C)
- In-situ baking: surface oxides

CMP or eCMP

- Polishing technique primarily for flat wafers,
- May be possible to implement into concave surfaces
- Slurry chemistry, particles, polishing pressure and sliding velocity…

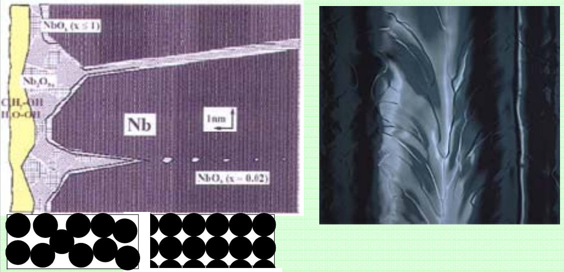


Bhushan, B., Introduction to Tribology, Wiley, 2002

- Crystal texture
- Surface roughness
- Oxides
- Contaminants (C,N, H)

Tribo/chemistry

- Hardness adjustment
- Material removal
- Surface polishing



Halbritter, J., Proceedings of 10th Workshop on RF Superconductivity, 2001, Tsukuba, JP, p.291-307.

Characterization tools

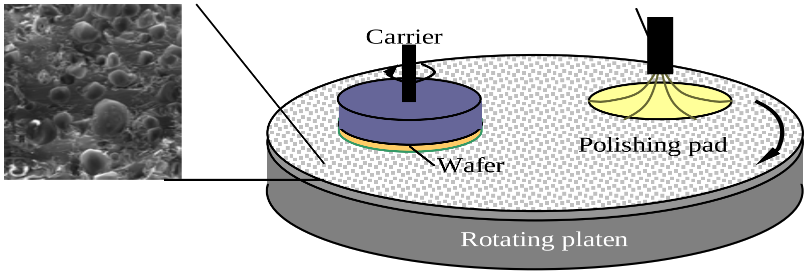
- XPS/ARXPS:
  Niobium, oxide, contaminants, short range bond order, thickness
- Nano-, micro-indenters:
  Hardness
- AFM, stylus and optical profilr:
  roughness
- SEM with EBSD detector:
  Metallographic texture

EFJP (Electrochemical Fluid Jet Polishing)

- Concave surfaces
- Hardness
- Shaping
- Polishing

Niobium Surface Engineering :Chemical Mechanical Polishing

- A *wafer* is pushed against a polymeric *polishing pad*
- (P_a = 1-10 psi)
- Pad and wafer rotate independently (~60 rpm).
- *Slurry*, containing *<u>oxidizing chemicals</u>* and *<u>abrasive particles</u>* is
- **Material removal** occurs due to ***particle abrasion*** of the ***chemically passivated*** *wafer surface.*

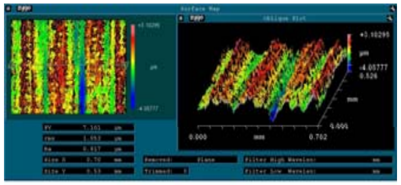


Schematic of CMP operation

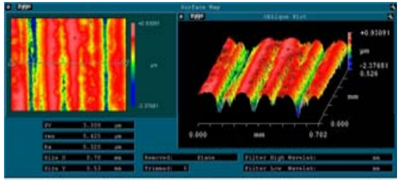
•**The Effects of Interfacial Particles on the Contact of an Elastic Sphere with a Rigid Flat Surface,** D. Bozkaya and S. Müftü, ASME Journal of Tribology,October 2008.

<u>Chemicals:</u>
- Oxidizers
- Buffers
- Surfactants

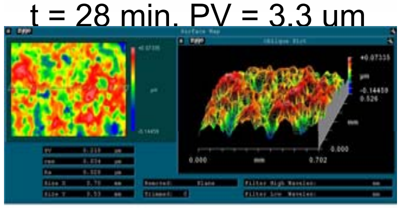
<u>Particles:</u>
- Material: Silica (SiO2), alumina (Al2O3, Ceria (CeO2)
- Size: 50-150 nm
- Shape: Spherical



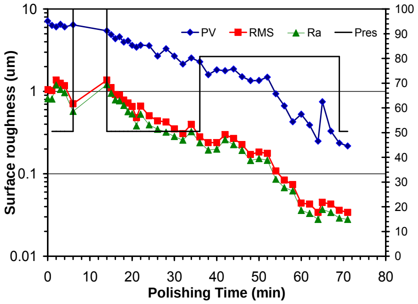
t = 0, PV=7.2 um

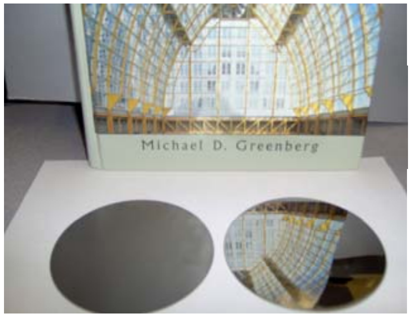


t = 28 min, PV = 3.3 um

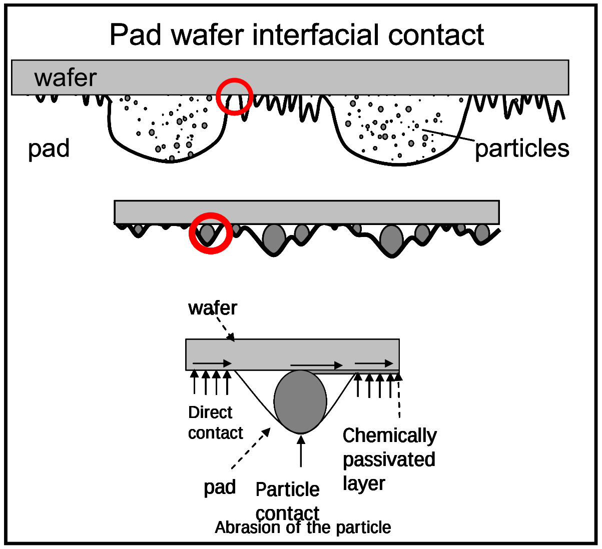


t = 71 min, PV =0.2 um





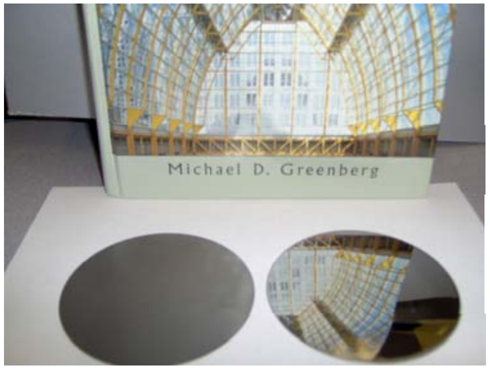
•**Investigation of Chemical/Mechanical Polishing of Niobium,** G. Calota, N. Maximova, K. Ziemer, S. Müftü, STLE Tribology Transactions, Vol 52(4), pp. 447-459, 2009.



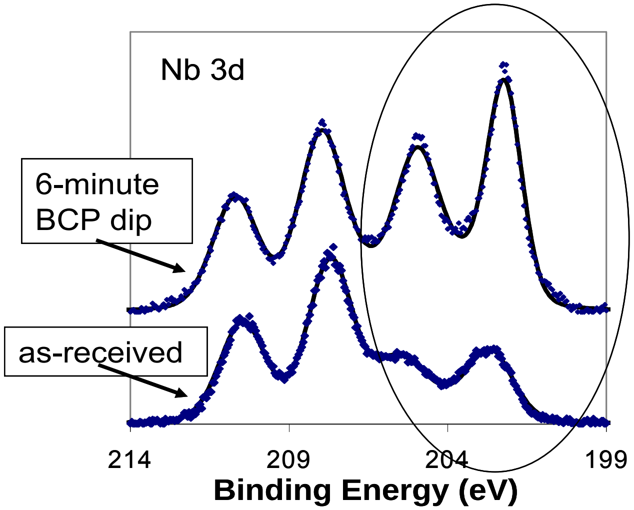
•**Effects of surface forces on material removal rate in chemical mechanical planarization,** D. Bozkaya, S. Müftü, Journal of the Electrochemical Society, Vol. 157(3) H287-296, 2010.

•**A Material Removal Model for CMP based on the contact mechanics of pad, abrasives and wafer,** D. Bozkaya, S. Müftü, Journal of the Electrochemical Society, Vol. 156(12), H890-902, 2009.

Niobium Surface Engineering: XPS of polished Nb



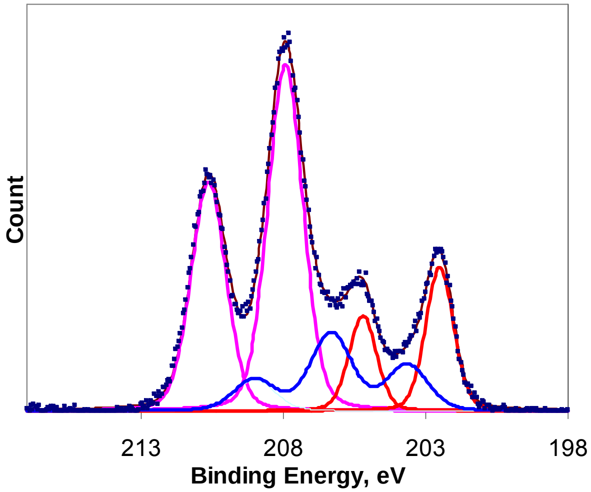
Buffered Chemical Polish



Full width half maximum (FWHM)

- Nb peak = 1.15 eV
- Oxide peak = 1.60 eV
- Thickness, NbxOy = 4.1 nm
- Roughness (PV) = 8-10 um

Chemical Mechanical Polish



Full width half maximum (FWHM)

- Nb peak = 1.20 eV
- Oxide peak = 1.50-55 eV
- Thickness, NbxOy = 4.6 nm
- Roughness (PV) = 230 nm

(RMS) = 39 nm

- Niobium forms a stable Ni2O5 oxide, ~ 4.5 nm thick, and self limiting.
- BCP treatment exposes ordered Nb metal, but prolonged treatment does not improve surface roughness.
- Mechanical polishing can expose ordered Nb metal faster, with a greener approach, and smoother surface

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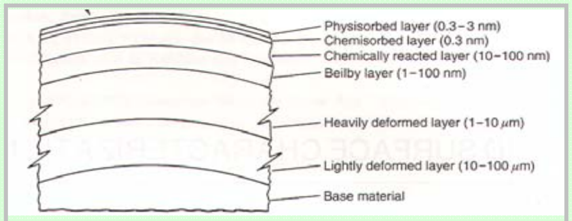
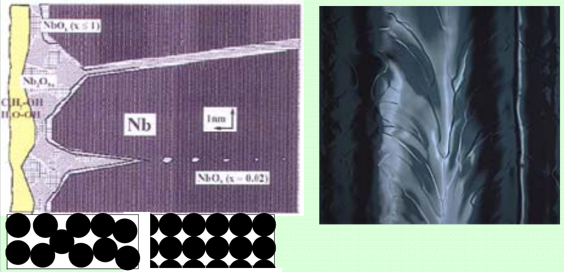
- Deep-drawing / hydro-forming ⇕ Hardness



Bhushan, B., Introduction to Tribology, Wiley, 2002



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EFJP (Electrochemical Fluid Jet Polishing)

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- Shaping
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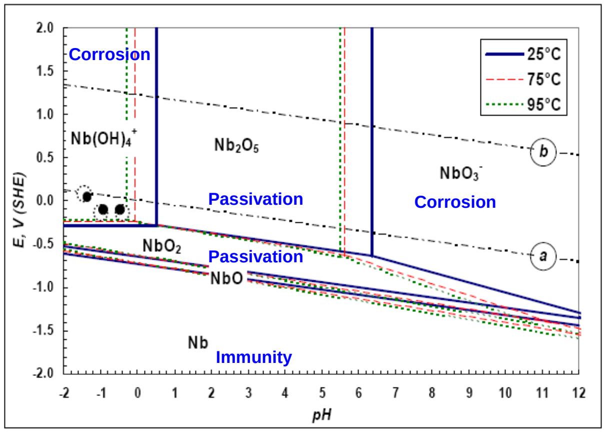
Electrochemistry

- The composition of the solution and applied field can indicate the stability of a particular metal in a specific environment.
  - **Immunity** indicates that the metal is not attacked
  - **Corrosion** shows that general attack will occur.
  - **Passivation** occurs when the metal forms a stable oxide coating
- Electrochemical polishing processes use energy from an applied electric field to stimulate or suppress certain oxidation or reduction chemical reactions with metals.
- eCMP combines electrochemistry with CMP.
- In the case of SRF cavity manufacture, however, it is the final curved surface that must be polished, and the standard ECMP tools are useless.



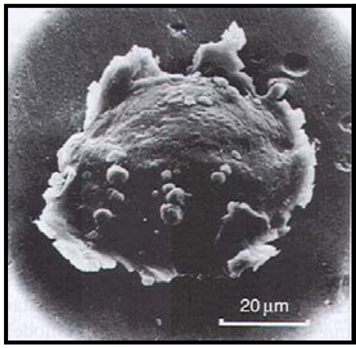
Pourbaix diagram for Nb-H2O system at 25, 75, and 95°C. Lines a and b represent hydrogen evolution and oxygen reduction, respectively. Modified from Asselin *et al.* .

Particle Impact Based Manufacturing Processes

High velocity impact of particles in manufacturing:

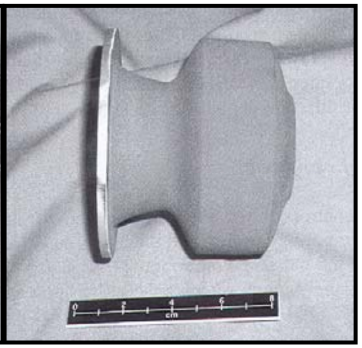
- Abrasive blasting: surface cleaning
- Cold-spray: surface coating
- <u>Fluid Jet Polishing of glass</u>:
  - The material removal rate from glass at a stationary spot was measured to be in the range of 28 – 5000 nm/min.
  - Resulting surface roughness of the glass was in the range of 1 – 500 nm, depending on the particles.
  - There appears to be no work in the published literature on using FJP for polishing/shaping of a ductile surface.

Cold Spray



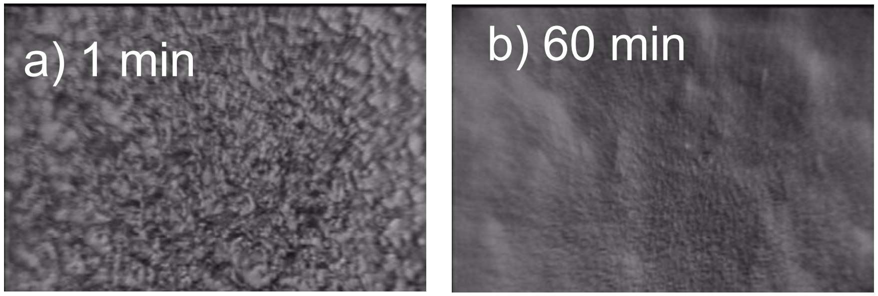
Al particle on Cu-substrate

V_p = 1100 m/s



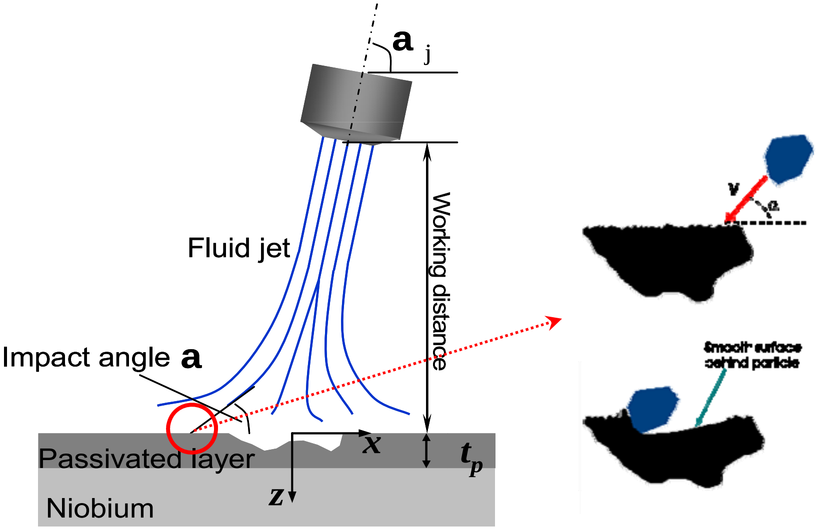
Ti-6Al-4V on Al substrate

FJP of #400 SiC Glass



Nomarski pictures of the #400 SiC pre-ground surface. The *Ra*-values are 625 nm and 225 nm, resp. from Booij, S.M., *Fluid Jet Polishing, 2003.*

FJP has been used for both polishing and shaping surfaces



Schematic representation of FJP.

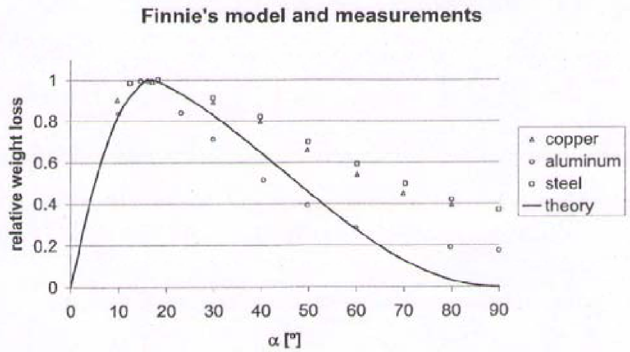
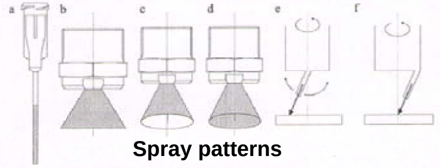


Fig. 4.17 Normalised relative weight loss as a function of the angle of impact of the particle (not of the jet), according to Finnie's model, and some measurement data according to Finnie of SiC grains on copper, aluminum and steel [fin58].

Booij, S.M., *Fluid Jet Polishing, 2003.*



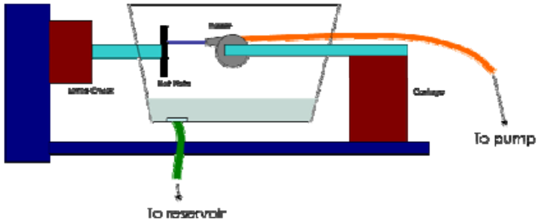
Spray patterns

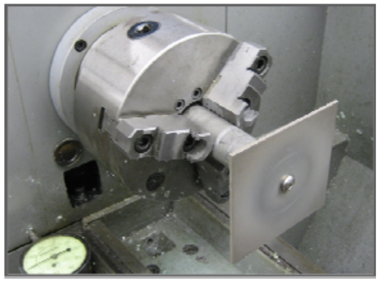
Booij, S.M., *Fluid Jet Polishing, 2003*.

$$\dot{V} = \begin{cases} \left(\dfrac{\rho V^2}{2\psi p_f}\right)\left[\sin 2\alpha - 3\sin^2\alpha\right] & \text{if } \tan\alpha \le 1/3 \\ \left(\dfrac{\rho V^2}{6\psi p_f}\right)\cos^2\alpha & \text{if } \tan\alpha \ge 1/3 \end{cases}$$

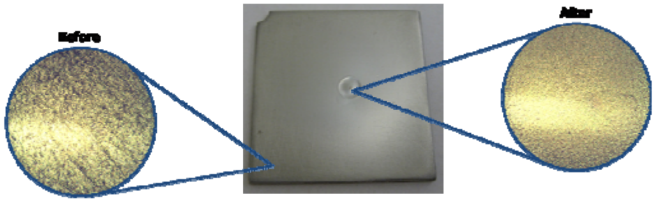
Finnie, I., *The mechanism of erosion of ductile metals*, Proc. of the third U.S. Int. Cong. of Appl. Mech., pp. 527-532 (1958)

Preliminary FJP experiments on Al plate

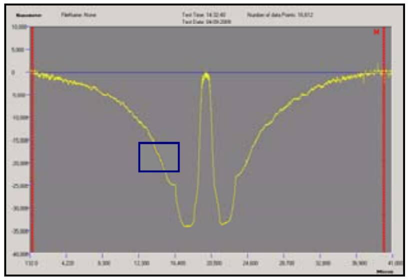




Stationary Flat plate: 100 psi, 20 min

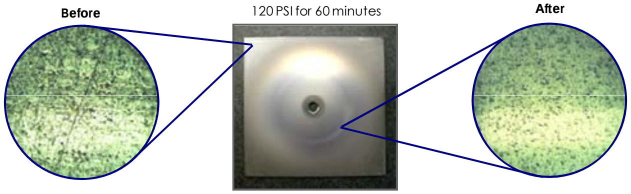


Roughness improved: 100 – 200 nm



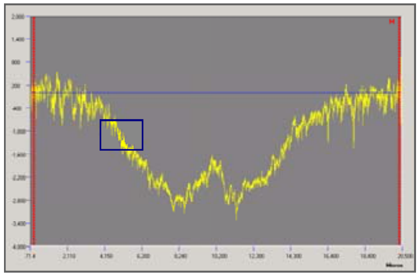
Material removed: 20 – 70 µm

Rotating Flat plate: 120 psi, 60 min



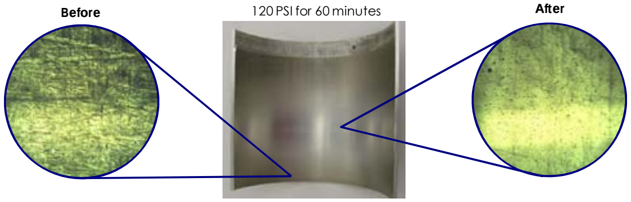
Images magnified 20x

Roughness improved: 150 nm



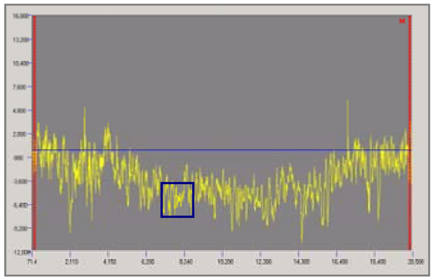
Material removed: 3 µm

Rotating cylinder: 120 psi, 60 min



Images magnified 20x

Roughness improved: 250 nm



Material removed: 9 µm

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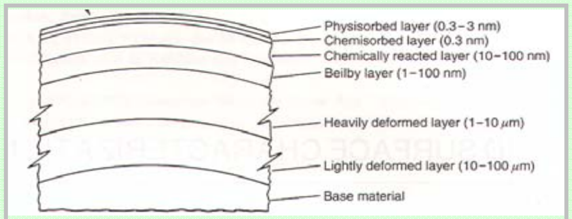
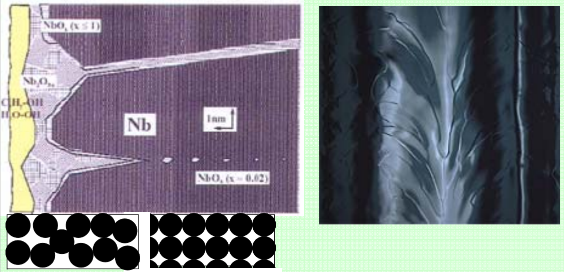


Bhushan, B., Introduction to Tribology, Wiley, 2002

- Crystal texture
- Surface roughness
- Oxides
- Contaminants (C,N, H)

Tribo/chemistry

- Hardness adjustment
- Material removal
- Surface polishing



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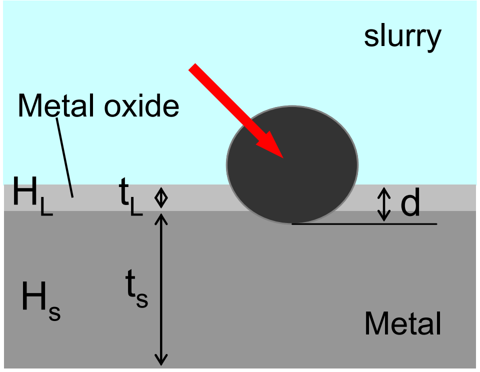
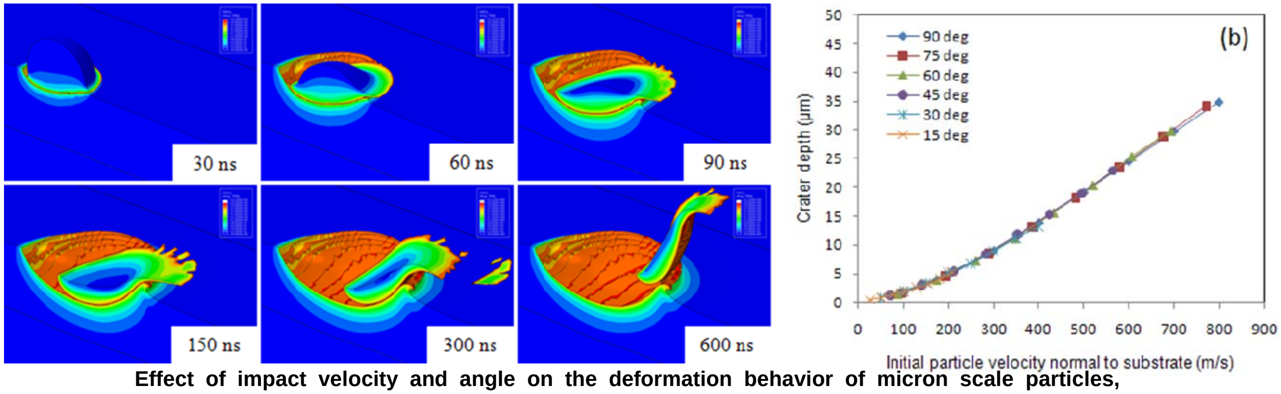
- Polishing

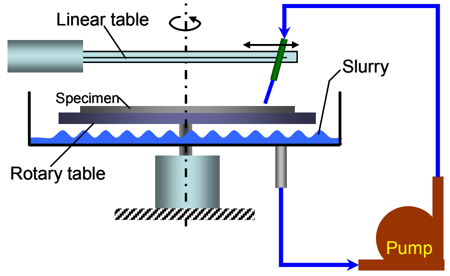
Tribochemistry

Impact on a surface with variable (or bi-layer) hardness

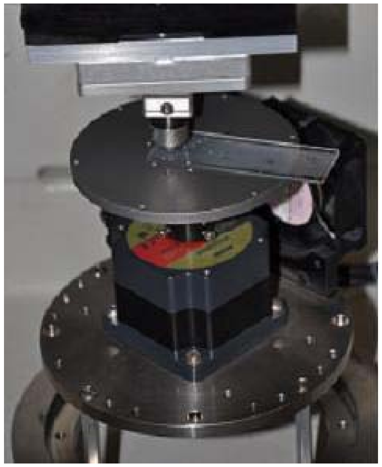




Effect of impact velocity and angle on the deformation behavior of micron scale particles, Yildirim, B. Gouldstone, A., Muftu, S. Proceedings of the 14th International Conference on Machine Design and Production, July 2010.



The schematics of the experimental setup for developing EFJP process.



- Need for tribo-chemistry experiments using a combined tribo-tester tester and electrochemical cell.
- Wear rate should be determined

$$\dot{W} = f(\vec{V}, D, H_L, H_s, pH, v)$$

- Results to benefit EFJP and tumbling

Processing time

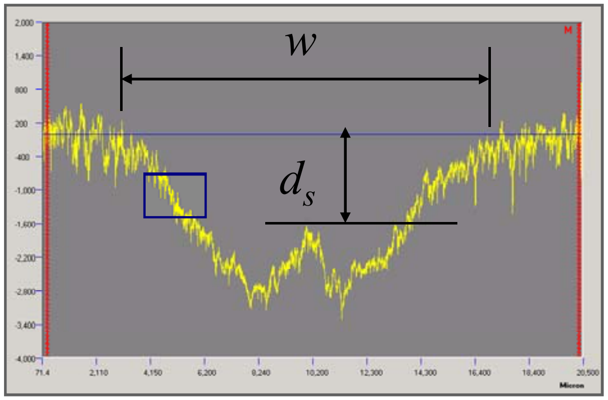
- Wear depth d = O(μm)
- Wear width w = O(mm)
- Estimated ~ processing time, t_p per cell:

$$t_p \approx \frac{A_{cell} d_d}{2\pi R_{ave} \dot{d}_s w}$$

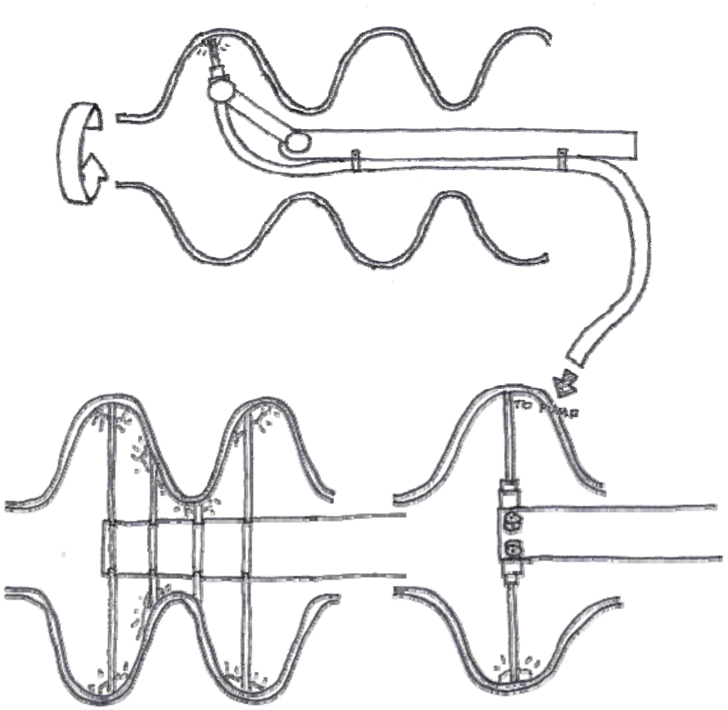
$$\dot{d}_s, w \propto f(\vec{V}_{jet}, \alpha, \frac{1}{H}, v, pH)$$

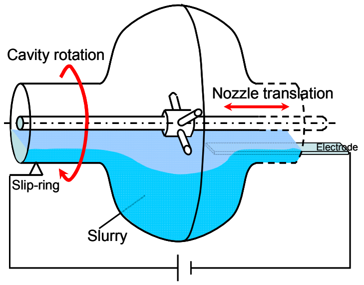
- A_{cell} : Internal area
- d_d : Required depth of material to be removed
- R_{ave} : Average radius of the cell
- $\mathrm{d}(d_s)/\mathrm{d}t$: Measured wear rate of stationary jet wear depth
- H : Hardness
- α : Jet orientation
- v : Applied potential

Roughness improved:
150 nm



Material removed:
3 μm

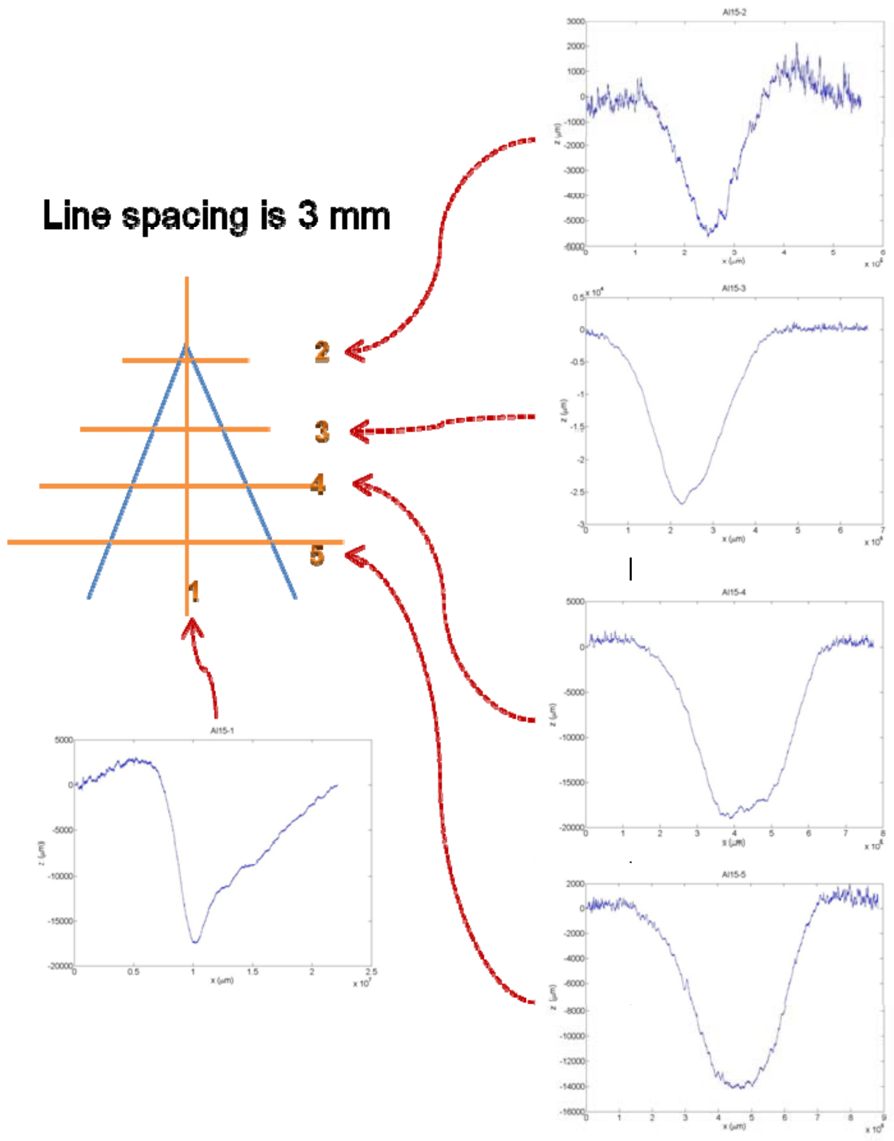




Thank you

Aluminum, stationary plate,15 deg, 8 min

Line spacing is 3 mm



w = 3 mm, d = 5um

w = 4 mm, d = 25 um

w = 6 mm, d = 18 um

w = 7 mm, d = 14 um