## Lecture 13: Glass Finishing (Grinding & Polishing)

### Glass Processing Course (Lehigh University; Spring 2015) International Materials Institute for New Functionality in Glass (IMI-NFG) March 5, 2015 1:00 -2:15 pm (EST)



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LLNL-PRES-668080

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. Lawrence Livermore National Security, LLC

## **Useful Reading Material**

H. Karow, "Fabrication Methods of Precision Optics" John Wiley & Sons (1993)

N. Brown, "Optical Fabrication" LLNL Report MISC4476 (August 1989)

L. Cook, "Chemical Processes in Glass Polishing" Journal of Non-Crystalline Solids 120 (1990) 152-171

T. Izumatani, "Optical Glass" (Kyoritsu Shuppan Company, Tokyo 1984; Lawrence Livermore National Laboratory (USA); American Institute of Physics (New York 1986)

D. Anderson, J. Burge, "The Handbook of Optical Engineering: Chapter 28: Optical Fabrication"

D. Malacara, "Optical Shot Testing" Wiley-Interscience (2007)

Other references quoted through the presentation



## What is optical fabrication?

The objective of optical fabrication is to manufacture an optical element (e.g., lense, flat, mirror, active optic) which is often made of glass

#### **Key Requirements**

- 1) Surface Figure (affects wavefront)
- 2) Surface Quality (affects scatter and laser damage resistance) a) Roughness
  - b) Sub-surface damage (scratch/dig)



## An example of specifying the requirements of an optic



<sup>1</sup>For typical 3ω NIF optics; <sup>2</sup>Post-etch with number of scratches (width>8μm) <12-50

## **Typical steps of an optical fabrication process**





## **Examples of grinding techniques**













## **Examples of polishing techniques**







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## The materials science behind grinding & polishing

1. Sub-surface Damage (scratch/dig)

- 2. Roughness
- **3. Surface Figure**



## There are numerous mechanical, structural and chemical effects on the glass surface during grinding and polishing







### There are numerous mechanical, structural and chemical effects on the glass surface during grinding and polishing





### There are numerous mechanical, structural and chemical effects on the glass surface during grinding and polishing





## The load/particle determines the removal mechanism

Removed

material



### **Chemical removal Chemical Polishing**



### P<sub>crit</sub>< 5x10<sup>-5</sup> N

- Removal at the molecular level  $(Si(OH)_4)$ by condensation & **hydrolysis**
- Creates smooth surface
- Determined removal amount ~0.04 nm



# Approach for the management of sub-surface fractures (i.e. scratches/digs)

Schematic of material removal during various steps of the grinding/polishing process illustrating surface fracture removal



- Removal at each step is aimed at removal of deepest damage decreasing it to the level of deepest damage expected at current step (most economical design)
- Note each subsequent step has much lower removal rate
- This approach has been generally followed for hundreds of years

\*Preston (1921), Aleinikov (1957), Edwards & Hed (1987), Brown (1980), Lambropoulos (1996)



## There are five major areas of effort that have aided in managing sub-surface fractures

#### GRINDING



1. Developed fracture mechanics understanding of sub-surface fracture distributions

### POLISHING



2. Identified/characterized behavior of rogue particles causing sub-surface fractures

### **CHEMICAL ETCHING**



3. Established techniques using etching to reveal and remove subsurface fractures



4. Developed quantitative rules for post-diagnosis of cause of surface fractures



5. Showed link between subsurface fracture removal & improved laser resistance

#### surface fractures



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## There are three basic types of cracks created by static brittle indentation





# The fracture initiation and growth constants need to be known to quantitatively use these relationships





# Friction strongly influences fracture initiation for a sliding particle indentation (i.e. scratching)





<sup>1</sup>Lawn, Fracture of Brittle Solids (1993) <sup>2</sup>Lawn, Indentation Fracture: Principles and Applications (1975)



## The effect of load on the fracture behavior of scratches has been measured

## Schematic description of fractures associated with a scratch



- At low loads (P<0.1 N), no cracking is observed just a ductile track
- At intermediate loads (0.1 N< P < 5 N), well defined median and lateral cracks form
- At high loads (P> 5N),

the plastically observed track appears to shatter and the median and lateral crack are not as extending as in the higher end of the intermediate loads

Refs: Review: K. Li, Journal of Materials Processing Technology 57 (1996) 206 Review: M. Swain, Proc. R. Soc. London A, 366 (1979) 575



#### GRINDING

### A wedge or taper polishing\* technique was developed to directly measure the SSD distribution



\*J. Menapace, SPIE 2005, Boulder Damage Symposium; Based on tapering technique used by Hed & Edwards (1987)







#### GRINDING

## The SSD depth distribution has been measured for a series of standard grinding processes





## **Coarse Generator Grind (120 grit) (Sample B)**



T. Suratwala, JNCS 352 (2006) 5601 Lawrence Livermore National Laboratory

NIF

#### GRINDING

### Microscope images of the fractures show a unique size character for each grinding step





#### GRINDING

### Microscope images of the fractures show a unique size character for each grinding step





### A brittle fracture model has been successfully used to explain the observed distribution of crack depth and lengths



Key assumption: The load on particle is proportional to its vertical dimension

\*T. Suratwala, JNCS 352 (2006) 5601. \*P. Miller, SPIE 5991 (2006).



#### GRINDING

## We recommend using the '90' rule for material removal (c<sub>90</sub>=0.9<L>) for isolated SSD observed on polished parts





#### GRINDING

## The addition of a small amount of 15 $\mu$ m particles in a 9 $\mu$ m slurry results in a significant increase in SSD





## The loaded particles are the largest particles in the abrasive particle distribution





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### CHEMICAL ETCHING



 Established techniques using etching to reveal and remove subsurface fractures





## Rogue particles of diamond were added to a ceria slurry during polishing at various sizes & concentrations





## Rogue particles can cause multiple types of scratches





## The scratch length increases with rogue particle size







# The observed scratch lengths can be explained by the viscoelastic penetration of a rogue particle



This behavior has been modeled using hard sphere penetration into a linear viscoelastic lap at large penetration



## The observed scratch lengths can be explained by the viscoelastic penetration of a rogue particle



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## The observed scratch lengths can be explained by the viscoelastic penetration of a rogue particle

$$L_{scratch} = 8.9 \frac{v_{ave} \eta R^2}{P}$$

 $t = t_2$  $P_1 = Load on rogue particle$ 



**Viscoelastic Lap** 

## This behavior has been modeled using hard sphere penetration into a linear viscoelastic lap at large penetration


## The observed scratch lengths can be explained by the viscoelastic penetration of a rogue particle

$$L_{scratch} = 8.9 \frac{\nu_{ave} \eta R^2}{P}$$

 $t = t_3$  $P_1 = Load on rogue particle$ 

Optic movement



**Viscoelastic Lap** 

### This behavior has been modeled using hard sphere penetration into a linear viscoelastic lap at large penetration

T. Suratwala, JNCS 354 (2006) 2023 Lawrence Livermore National Laboratory



## The observed scratch lengths can be explained by the viscoelastic penetration of a rogue particle

$$L_{scratch} = 8.9 \frac{\nu_{ave} \eta R^2}{P}$$

 $t = t_4$  $P_1 = Load on rogue particle$ 





Viscoelastic Lap

### This behavior has been modeled using hard sphere penetration into a linear viscoelastic lap at large penetration

T. Suratwala, JNCS 354 (2006) 2023 Lawrence Livermore National Laboratory



## The observed scratch lengths can be explained by the viscoelastic penetration of a rogue particle

$$L_{scratch} = 8.9 \frac{\nu_{ave} \eta R^2}{P}$$

 $t = t_5$ P = Load on all particles

**Optic movement** 



**Viscoelastic Lap** 

### This behavior has been modeled using hard sphere penetration into a linear viscoelastic lap at large penetration

T. Suratwala, JNCS 354 (2006) 2023 Lawrence Livermore National Laboratory



#### The scratch length correlates with viscoelastic model wrt rogue particle size, pressure, lap viscosity, and lap temperature





T. Suratwala, JNCS 354 (2006) 2023; T. Suratwala OPN (Sep 2008) 12 Lawrence Livermore National Laboratory



## There are five major areas of effort that have aided in managing sub-surface fractures



1. Developed fracture mechanics understanding of sub-surface fracture distributions

#### POLISHING



2. Identified/characterized behavior of rogue particles causing sub-surface fractures

#### **CHEMICAL ETCHING**



3. Established techniques using etching to reveal and remove subsurface fractures



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ETCHING

## HF:NH<sub>4</sub>F etching of fused silica glass allows for removing the Bielby layer and visually observing surface cracks





#### ETCHING

## HF Etching exposes sub-surface fractures allowing detection

Polished Optic (14 cm x 14 cm) viewed off axis by side lighting



#### **Preston reported this behavior in 1921**

L. Wong, JNCS 355 (2009) 797 Lawrence Livermore National Laboratory



#### ETCHING

# HF etching can be used after grinding to remove subsurface fracture because it annihilates neighboring cracks



L. Wong, JNCS 355 (2009) 797 Lawrence Livermore National Laboratory



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5. Showed link between sub surface fracture removal & improved laser resistance

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#### SCRATCH FORENSICS

#### Our studies have provided <u>new</u> rules that Opticians use to diagnose the cause of or to mitigate scratches

Property of scratch	What can it tell you?		Rule / E	xampl	e
1. Scratch width or	- Size of rogue particle (d)	For grinding			
trailing indent length (L)	- Size distribution of Rogue Particles	$0.15 \ d \le L \le 0.3 \ d$			
	- Process step		For polish	ing	7
	- Depth of fracture (c <sub>90</sub> or c <sub>max</sub> )	0.	$3 d \leq L \leq$	0.5	d
2 Number density	- Roque particle concentration		Sample	<l></l>	
3 Scratch length (I )	- Lan properties and roque particle si	70	B: 120 grit	28.3 μm	
1. Ocratch length (Escratch)	- Lap properties and rogue particle size	26	C: 320 grit	14.9 μm	
4. Scratch type (plastic,	- Load during fracture		D: 15 µm loose	4.6 μm	
brittle, mixed)	- Sharpness of particle		F: 9 μm loose	4.5 μm 1.9 μm	
5. Orientation and	- Particle movement direction		G: 7 μm fixed	8.4 μm	
pattern of trailing indent	- Particle rotation	$c_{90} = 0.9 < L > c_{max} = 2.8 < L >$			
	- Stick slip behavior	$P \approx 0.0$	001 - 0.1 N P	Plastic o	nly
6. Curvature	- Pathway of indenting particle	$P \approx 0.1$	-5 N Plast	ic & Br	rittle
or scratch pattern	- Shape of tool	P > 5N Plastic & rubble			
	- Handling vs polishing	$L_{s}$	$_{cratch} = 8.9 \frac{v_{d}}{2}$	$\frac{\eta R^2}{R}$	

- Material removal and surface figure

7. Location on optic

#### T. Suratwala, JNCS 354 (2006) 2023; T. Suratwala OPN (Sep 2008) 12 Lawrence Livermore National Laboratory



Ρ

#### **Example of scratch forensics**







#### **Example of scratch forensics**





#### 1. Measure the SSD at each step

- 2. Define proper removal rate at each step such that all the SSD from previous step is removed
- 3. Can use etching as a means to remove SSD just after grinding
- 4. Ensure handling and cleaning at each step does not let rogue particles make contact with surface
- 5. Remove all rogue particles in polishers; Use scratch forensics to determine source
- 6. Use etched scratch dig inspections between steps and at end of process





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237 µm



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Rogue particle sources

3

6

9

**10<sup>-6</sup>** 

I) In slurry from foreign particle or agglomerates

12

Crack Depth (µm)

15

- 2) Dried slurry on components falling in
- 3) Contamination from polisher exterior

18

21

24



- 1. Measure the SSD at each step
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Etching provides a means of revealing



## There are five major areas of effort that have aided in managing sub-surface fractures

#### CERINDING • A: Send blast • B: 520 ptf Generator • C: 52 ptf Generator • D: 53 ptf Generator • D: 54 ptf Gene

1. Developed fracture mechanics understanding of sub-surface fracture distributions

#### POLISHING



2. Identified/characterized behavior of rogue particles causing sub-surface fractures

#### **CHEMICAL ETCHING**



3. Established techniques using etching to reveal and remove subsurface fractures



#### LASER DAMAGE



5. Showed link between subsurface fracture removal & improved laser resistance

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#### LASER DAMAGE

#### SSD-free test optics have been fabricated such it does not laser damage, supporting the "absorber-in-a-crack" theory



Laser testing on a 14 cm x 14 cm test optic to 14 J/cm<sup>2</sup> (351 nm, 3 ns equiv) resulted in the elimination of growing laser initiation site upon SSD removal

Lawrence Livermore National Laboratory





#### **AMP process system**



T. Suratwala JACS 94(2) (2010) 416; P. Miller US Patent 0079931 (2011) Lawrence Livermore National Laboratory



## AMP process significantly reduces laser damage initiation per unit scratch length



T. Suratwala JACS 94(2) (2010) 416; P. Miller US Patent 0079931 (2011) Lawrence Livermore National Laboratory



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The materials science behind grinding & polishing

1. Sub-surface Damage (scratch/dig)

2. Roughness

**3. Surface Figure** 



#### To understand surface <u>roughness</u>, one need to understand the complex microscopic & chemical interactions during polishing





## The tail end of the slurry's PSD\* strongly correlates with workpiece roughness



Lawrence Livermore National Laboratory LLNL-PRES-668080 Suratwala et. al., J. Am. Cer. Soc. 97(1) 2014



## The tail end of the slurry's PSD\* strongly correlates with workpiece roughness



The tail end of each slurry follows a single exponential distribution

\*Particle size distribution

### Exponent constant in PSD of slurry vs RMS roughness of polished surface



### The slope of the slurry's PSD quantitatively scales with the rms roughness

Suratwala et. al., J. Am. Cer. Soc. 97(1) 2014



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## Novel chemical slurry stabilization and engineered filtration has resulted in improve slurry PSD





#### Improved Particle Size Distributions



- Surfactant dramatically reduces agglomeration without reducing removal rate
- Appropriate filtration further improves PSD

US Patent Application WO 2012129244 A1 (September 27, 2012) R. Dylla-Spears, Colloids & Surfaces A 447 (2014) 32 T. Suratwala, JACS 97 (2014) 81



#### Single pass ceria particle sliding experiments suggest 'plastic' - type removal can occur during polishing



Sample 4: using Stabilized Hastilite

Suratwala et. al., *J. Am. Cer. Soc.* 97(1) 2014 Lawrence Livermore National Laboratory LLNL-PRES-668080



#### Single pass ceria particle sliding experiments suggest 'plastic' - type removal can occur during polishing



Sample 4: using Stabilized Hastilite

Suratwala et. al., J. Am. Cer. Soc. 97(1) 2014 Lawrence Livermore National Laboratory LLNL-PRES-668080

### Lineout of AFM Perp. to slide particle slide direction



 Single pass of ceria particle removes ~1 nm of material (~7 Si-O units)



# Ensemble Hertzian Gap (EHG) model is used to determine the gap, fraction of particles loaded, & the load per particle



Suratwala et. al., J. Am. Cer. Soc. 97(1) 2014

#### In this formalism, only need to solve for g

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#### **Governing Relationships**

Load Balance

$$\mathbf{P}_{A} = \mathbf{N}_{b} 2\pi r_{o}^{2} \underbrace{\int}_{g}^{\infty} F(r) P(r,g) dr$$

Number density of particles in gap

$$N_{b} = f_{A}N_{p} = f_{A}\frac{\int F(r) dr}{\int F(r) 2\pi r dr}$$

Load on each particle (Hertzian Contact)

$$P(r,g) = \frac{4}{3} \operatorname{E}_{eff} \sqrt{r(2r-g)^3}$$

- g = gap at interface
- f<sub>A</sub>= fraction of pad area making contact with workpiece
- N<sub>p</sub>= # of particles/area



## Using the EHG model, the calculated fraction of "active" particles is very small

#### Full PSD for stabilized and unstabilized Hastilite Polishing Slurry



- Shaded region represents active (i.e., loaded) particles (using f<sub>A</sub>=1.5x10<sup>-4</sup>)
- Stabilized Hastilite uses much smaller particles during polishing compared to Unstabilized Hastilite

Slurry	Gap	Fraction of active particles
Stabilized Hastilite	<b>0.07</b> μm	0.224
Unstabilized Hastilite	<b>0.42</b> μm	0.0005

Suratwala et. al., J. Am. Cer. Soc. 97(1) 2014 Lawrence Livermore National Laboratory LLNL-PRES-668080

# Using the EHG model, polished surfaces using different PSDs have been simulated over multiple spatial scale lengths



Suratwala et. al., *J. Am. Cer. Soc.* 97(1) 2014 Lawrence Livermore National Laboratory LLNL-PRES-668080



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The materials science behind grinding & polishing

- 1. Sub-surface Damage (scratch/dig)
- 2. Roughness

**3. Surface Figure** 



## The surface figure of an optic is typically measured by interferometry





## Material removal on a workpiece is governed by a large number of phenomena



IJAGS 3(1) 14-28 (2012); J. Am. Ceram. Soc., 97 [6] 1720-1727 (2014);

J. Am. Ceram. Soc., 93 [5] 1326–1340 (2010) Lawrence Livermore National Laboratory


# Material removal on a workpiece is governed by a large number of phenomena





#### Friction

### The optic/lap can have different modes of contact which strongly influences the amount of material removal

#### **Contact Mode**







- Friction μ>0.1
- Optic/pad mechanically make contact
- High pressure/low velocity
- Real contact area < nominal contact area
- Plastic deformation of optic/ pad occurs
- Fluid film is discontinuous



- Friction μ~0.01 to 0.1
- Transition mode during pressure or velocity changes
- Contact is made between lap asperities and optic





- Friction μ~0.001 to 0.01 (due to shear of viscous fluid)
- Optic glides on fluid film without directly touching pad
- Low pressure/high velocity
- Pressure build-ups in fluid to support normal load of optic
- Pressure gradient is sensitive to wedge angle

J. Lai, Thesis (2001); J. Am. Ceram. Soc., 93 [5] 1326-1340 (2010)



# A geometric model is used to estimate the figure during conventional grinding/polishing

#### Schematic of geometric model



The velocity vector at each point on the optic is the velocity relative to the optic rotation minus the velocity relative to the lap rotation

$$\vec{V} = \left(\vec{R}_{optic} \times \vec{\rho}\right) - \left(\vec{R}_{Lap} \times \left(\vec{\rho} - \vec{S}\right)\right) + \vec{V}_{s}$$

#### where the vectors are:



NIF

J. Am. Ceram. Soc., 93 [5] 1326–1340 (2010) Lawrence Livermore National Laboratory

# For a translating workpiece on a viscoelastic lap, stress is highest at leading edge and lowest at end



#### J. Am. Ceram. Soc., 93 [5] 1326-1340 (2010)



### Calculated instantaneous stress distribution is qualitatively similar to measured data

Leading edge

### Calculated instantaneous Stress profile



Measured removal on optic when it is not rotated (Exp B)



High removal was observed at leading edge consistent with viscoelastic mechanism for causing pressure distribution

J. Am. Ceram. Soc., 93 [5] 1326–1340 (2010) Lawrence Livermore National Laboratory



#### **Rigid Punch**

# The pressure distribution across the workpiece can be predicted using the rigid punch indentation model for contact mode





# Our code SurF incorporates these phenomena & does a good job at predicting surface



J. Am. Ceram. Soc., 93 [5] 1326–1340 (2010)



# Workpiece polishing can cause non-uniform wear of the lap

#### Shape of lap after polishing workpiece



T. Suratwala et. al., IJAGS 3(1) 14-28 (2012).





# A novel septum has been designed to counteract non-uniform wear on the pad



#### T. Suratwala et. al., IJAGS 3(1) 14-28 (2012).



### **Temperature variations across workpiece can be** minimized using rotated workpiece and septum







80

100

### Pitch (Stiff) Button Blocking (PBB) and Foam (Compliant) Button Blocking (FBB) allows different workpiece response during polishing for High AR workpieces





### Without stiff blocking, thin workpiece deflects during polishing







# Pitch button blocking (PBB) technique prevents workpiece from bending during polishing

### 265 mm (side) x 8 mm (thick) Fused Silica PBB

### Model vs Experiment: $\Delta PV$ as fn of pitch button area fraction



#### M. Feit et. al., Applied Optics 51(35) (2012) 8350-59



# Fine scale radial material non-uniformity is caused by local islands of slurry on the pad

Schematic representation of islands of slurry on pad



Optical micrograph of grooves observed on non-rotated workpiece



J. Am. Ceram. Soc., 97 [6] 1720–1727 (2014) Lawrence Livermore National Laboratory



# Fine scale radial material non-uniformity is caused by local islands of slurry on the pad





#### Optical micrograph of grooves observed on non-rotated workpiece



### <u>Radial</u> stroke motion dramatically reduces this non-uniformity

J. Am. Ceram. Soc., 97 [6] 1720–1727 (2014) Lawrence Livermore National Laboratory



# Residual grinding stress causes a high aspect ratio workpiece to bend



#### Chemical etching can effectively remove the residual stress and any complications to workpiece-lap mismatch

T. Suratwala, IJAGS 3(1) 14-28 (2012) Lawrence Livermore National Laboratory



# Material removal on a workpiece is governed by a large number of phenomena







The materials science behind grinding & polishing

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