Stress via Twyman Effect and Subsurface Damage in Polycrystalline Silicon Carbide

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Mirror Tech Days
August 26, 2008

Outline

- Motivation: Impact of Stress and Subsurface Damage in SiC Optics
- Description of Experiment
- Twyman Effect Overview
- Results
- Subsurface Damage (SSD) Background
- Techniques for Measuring SSD or Stress
- Results

Impact of SSD and Stress in SiC

- SiC of interest for space based optics
  - Stiff, light weight with high thermal conductivity and low CTE
- SiC is very hard; requires significant tool pressure during grinding
- Stress can adversely impact the figure at cryogenic temperatures
- SSD microcracking introduces scatter, reduces the strength which could lead to failure

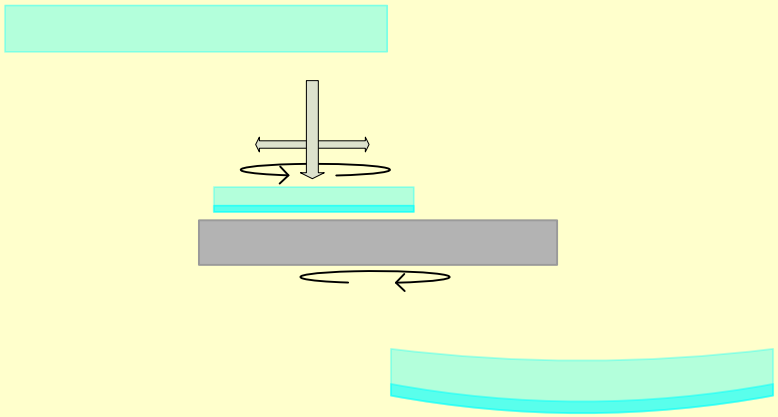
Description of Experiment

- Stress in CVD SiC was compared for various processing conditions by measuring the deflection due to the Twyman Effect
- The depth of the subsurface damage (microcracks) was measured for the various processing steps using MRF
- Comparisons were made for surfaces lapped with 3 µm diamond on a cast iron plate, 3 µm diamond polished on polyurethane pad, 1 µm diamond polished on a pad, chemically etched, and magnetorheologically finished (MRF) and laser ablation.
- SSD is measured using a MRF Spot Technique

Twyman Effect

Twyman observed that a flat, high aspect ratio part double side polished will bow when one of the two surfaces is lapped as a result of the difference in stress between the two surfaces

- Lapping induces compressive stress causing the ground surface to be convex
- Stoney's Equation for thin films can be applied to calculate the stress if the damage layer thickness is known



$$\sigma = \frac{\frac{E}{1-\nu} h^2}{6Rt}$$

where h is the thickness, R is the resulting radius of curvature, and t is the thickness of the damaged layer

SiC Sample Preparation

- TREX CVC SiC 50.8mm dia. Thickness ~1mm
- Coupons were wire sawn using 20-35 µm fixed abrasive diamond (resulting in 7 µm PV) followed by double side lapping and polishing (DSL/DSP) using sequentially finer abrasives

Diamond Abrasive Size	Material removed	Surface Roughness* (PV)
Lapping using steel plate		
6 µm	>15 µm	~300 nm
3 µm	~4 µm	~65 nm
Lapping using polyurethane pads		
3 µm	~4 µm	~50 nm

*Zygo NewView 5000, 20X Mirau, Min/Mod: 5.0, 660X880 µm Field of View

SiC Wafer Figure generated at EOC

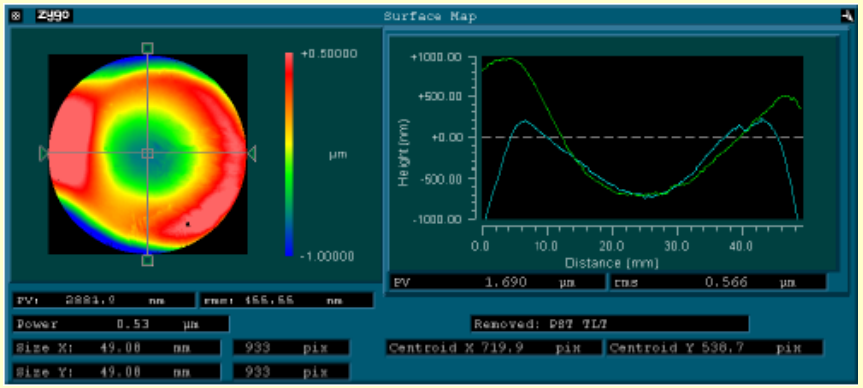
Wafer Group	Double Side Polish Parameters	DSP Figure (Power)	Single Side Polish/Lap Parameters	Final Figure (Power)
1	3 µm dia./pad 23kPa down pressure	-0.195- -0.505 µm	3 µm dia./steel 9.8 kPa down pressure 50 rpm tool spindle >10 µm removed	-2.75- -2.357 µm (CX) 2.346-2.783 µm (CC)
2	1 µm dia./pad 23kPa down pressure	-0.694- -1.284 µm	3 µm dia./pad 9.8 kPa down pressure 50 rpm tool spindle	-6.18- -7.195 µm (CX) 0.143-0.627 µm (CC)
3	1 µm dia./pad 23kPa down pressure	-0.717- -1.225 µm	N/A	N/A

Wafer Polishing at QED using MRF

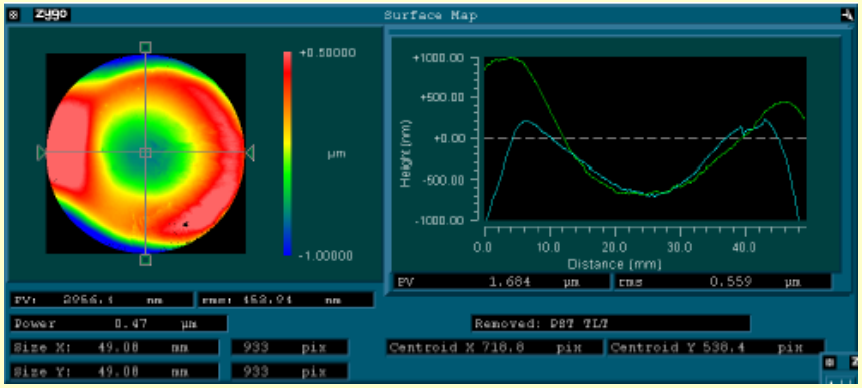
- MRF can remove uniform layers of material at specific removal depths without attention to pre-existing wafer bow
  - Diamond based MR Fluid was used
  - Parts held by vacuum using an acrylic backing plate
  - Each polishing step removed 100 nm
  - For each polishing step figure was measured with an interferometer to observe relaxation of the Twyman Effect
- 3 SiC wafers with different surfaces were polished and measured at QED

Chemically Etched SiC

- 3 µm diamond double side polished→3 µm single side lapped on steel (>10 µm removed)
- Chemically etched on single side lapped until wafer relaxed
  - Two orthogonal line scan indicate surface nearly identical
  - Change in power is most likely due to Zernike calculation
  - 100 nm additional material removed to verify no change in figure
  - Final roughness: 1300 nm PV, 14 nm rms after 200 nm removal



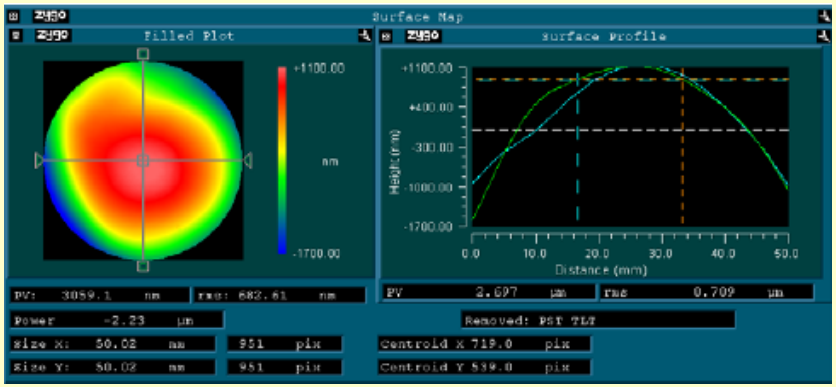
Power = 1370 nm



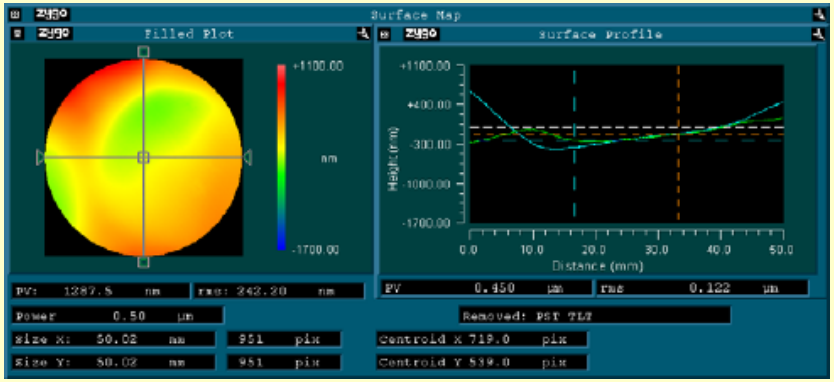
Power = 530 nm

3 µm DSP with 3 µm SSL on Steel

- Power changes 2730 after 100 nm of material removal using MRF
- Removal of 100 nm from the compressive surface results in the stress in each surface inverting (i.e. the compressive side becomes tensile and vice versa



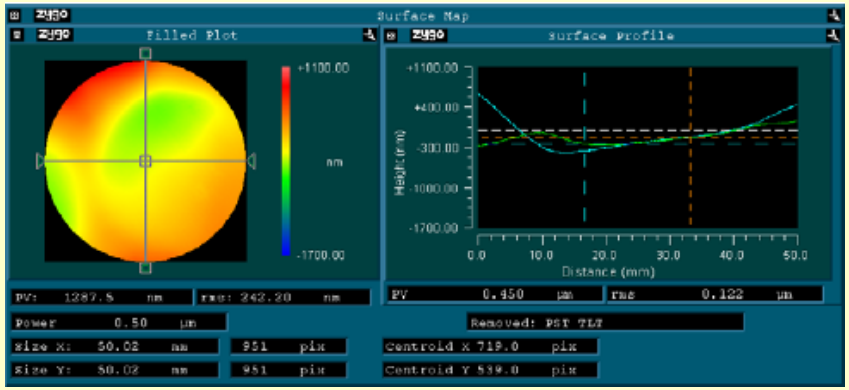
Initial
Power = -2230 nm



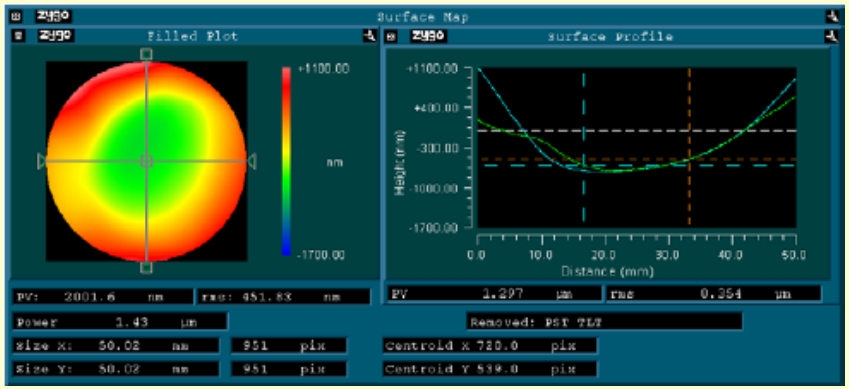
After 100 nm
Power = 500 nm

2nd Polishing Iteration: 200 nm removed

- Total of 200 nm removed using MRF
- MRF continues to remove stress from lapping with 3 µm diamond on steel
- Surface being MRF polished has less stress than 3 µm double side lapped surface



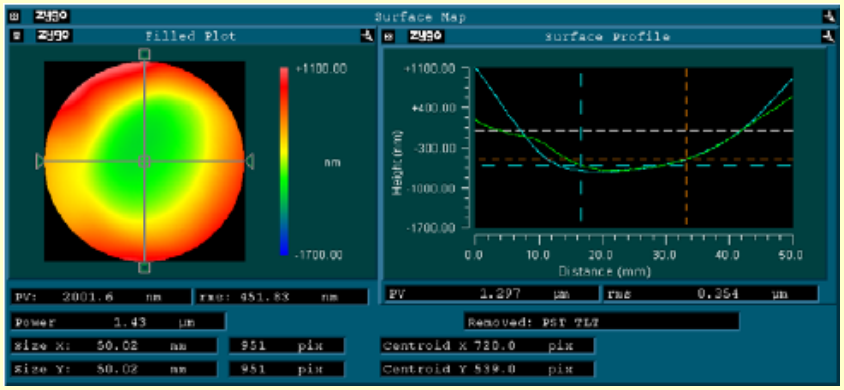
After 100
Power = 500 nm



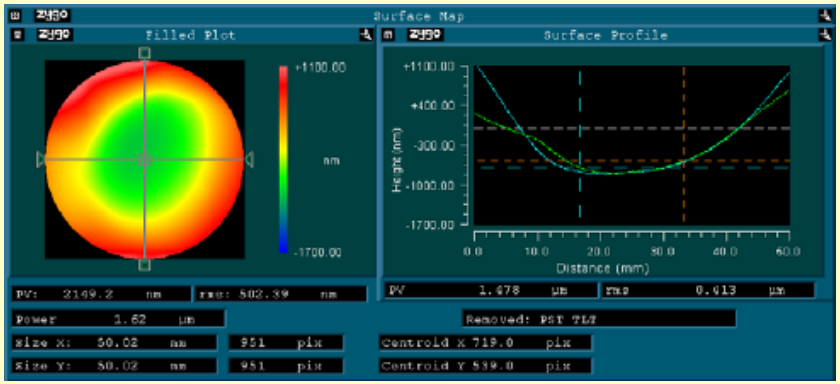
After 200 nm
Power = 1430 nm

2nd Polishing Iteration: 300 nm removed

- Surface has reached its final figure, subsequent polishing will not cause figure deformation
- 3 µm diamond polish renders an SSD layer between 100-200 nm thick

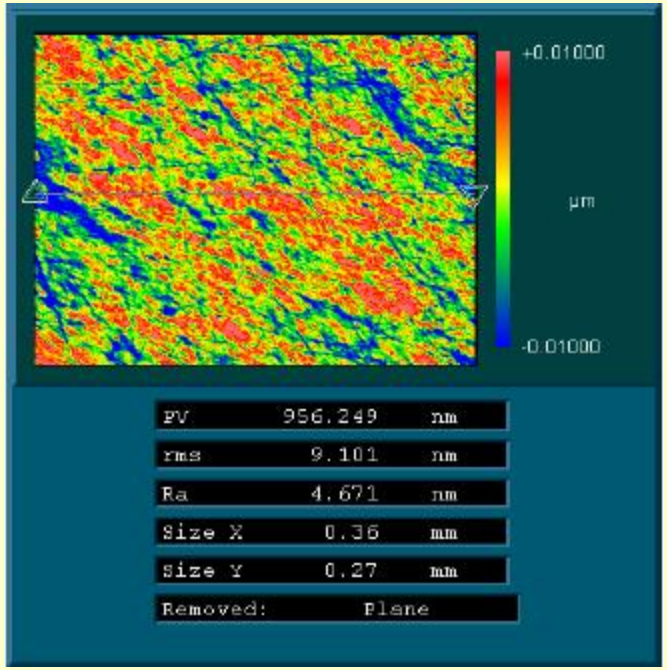


After 200 nm
Power = 1430 nm

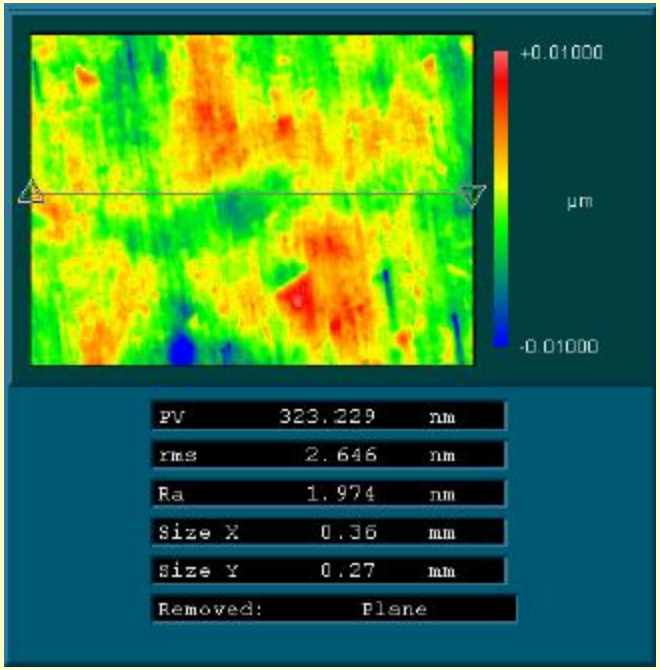


After 300 nm
Power=1420

Improved Roughness from MRF



Initial
PV=956 nm
Rms=9.1 nm



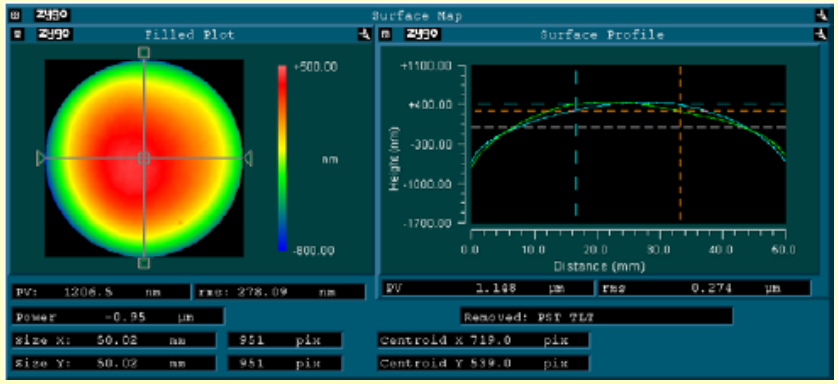
300 nm removed
PV=323 nm
Rms=2.6 nm

- Roughness improved by >3X from 300 nm removed
- Better finish possible with additional material removal

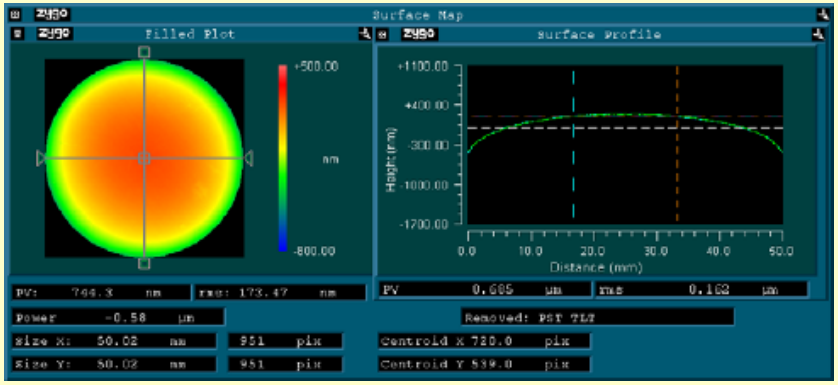
1 µm DSP followed by SS MRF

- Results show that a 1 µm diamond polish has stress compared to MRF, the surface is moving from CX to CC
- Damage occurs within 100 nm of the surface for 1 µm diamond polishing
- Initial roughness:2.4 nm rms→Final roughness: 1.8 nm rms

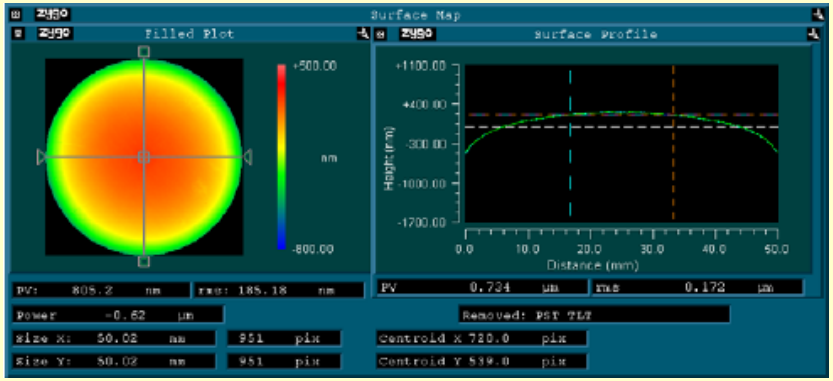
Initial
Power = -950 nm



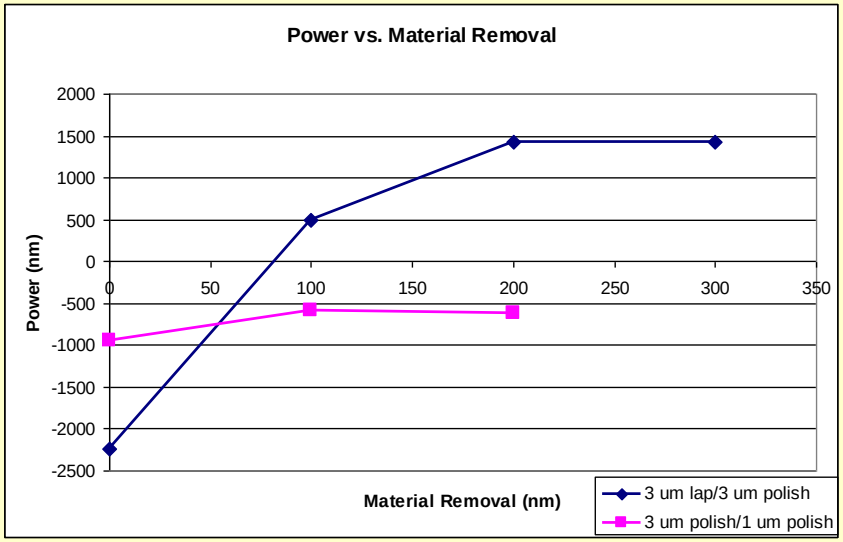
After 100 nm
Power = -580 nm



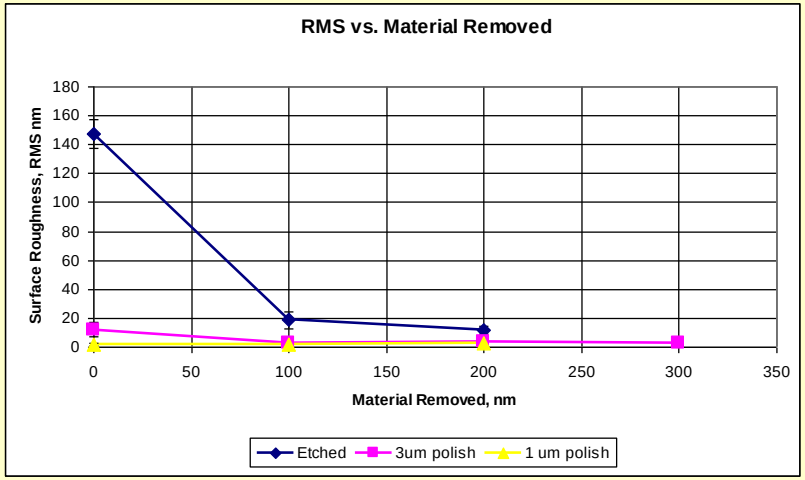
After 200 nm
Power = -620 nm



Twyman Stress vs. Roughness and Material Removed



3 µm lap vs. 3 µm polish has more stress than 3 µm polish vs. 1 µm polish



Roughness decreases to <20 nm after 100 nm removed from the surface

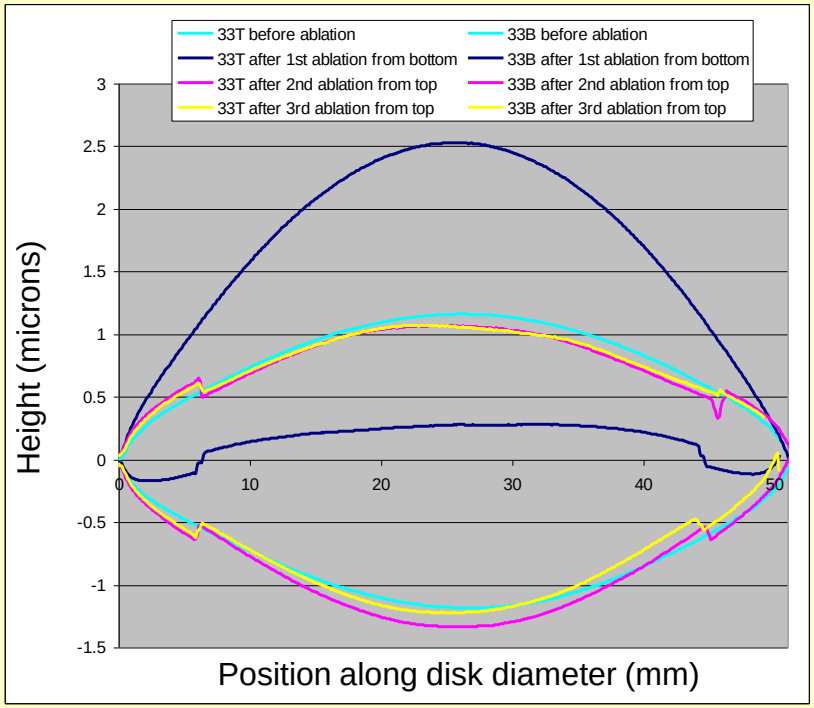
Twyman Effect Conclusions from MRF

- Stress scales with abrasive size for conventional lapping and grinding processes, but does not scale for chemical processes
- MRF reduces stress similarly to chemical etching, i.e. no observable change in wafer bow when MRF removed 200 nm of material from the chemically etched surface
- 3 µm and 1 µm polished surface have 100-200nm and <100 nm thick stress layers

Picosecond Ablation of SiC

Effect of picosecond pulsed laser ablation on Twyman stress of SiC.

- Polished disks of Trex SiC have compressive stress on each face before ablation.
- Ablative removal of ~200 nm appears to completely relieve compressive stress on sample face.
- Figure shows evolution of the shape of each face with successive ablations.
- Laser is ablating away damaged material w/o propagating or creating damage.
- Studies underway removing much thinner layers of material (~20 - 50 nm) to study depth profile suggest damage layer may be < 100 nm.



Light Blue: Original shapes of disc surfaces.

Dark blue: After ablation (stress relief) of bottom face.

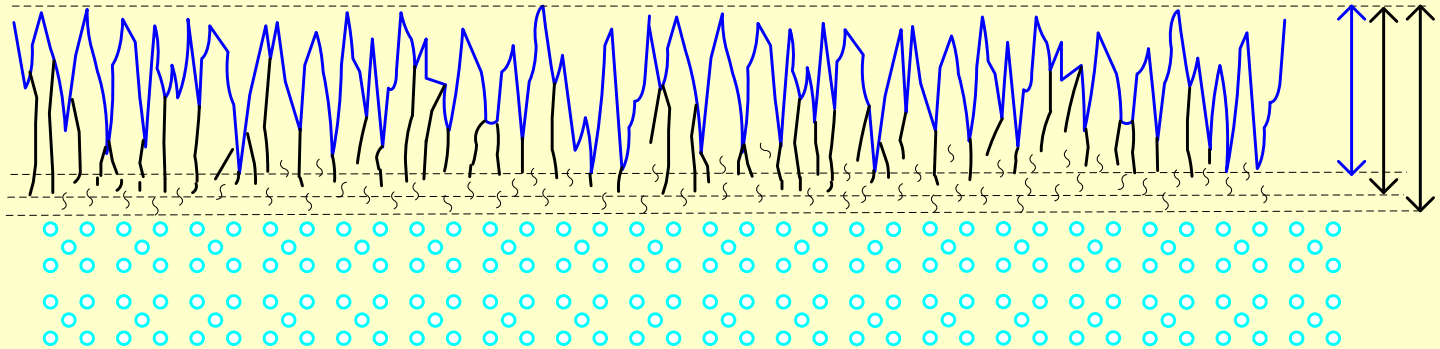
Pink: Ablation of top face restores original shape.

Yellow: Subsequent ablation of top causes no further change. Stress is fully relieved.

Subsurface Damage (SSD)

Subsurface Damage is the top layer of a bulk material that has discernable differences from the bulk as a results of surface processing

- SSD can contain microcracks from brittle material removal from grinding, and residual stress surrounding crack tips or from plastic deformation from ductile grinding or polishing



SSD Measuring Techniques

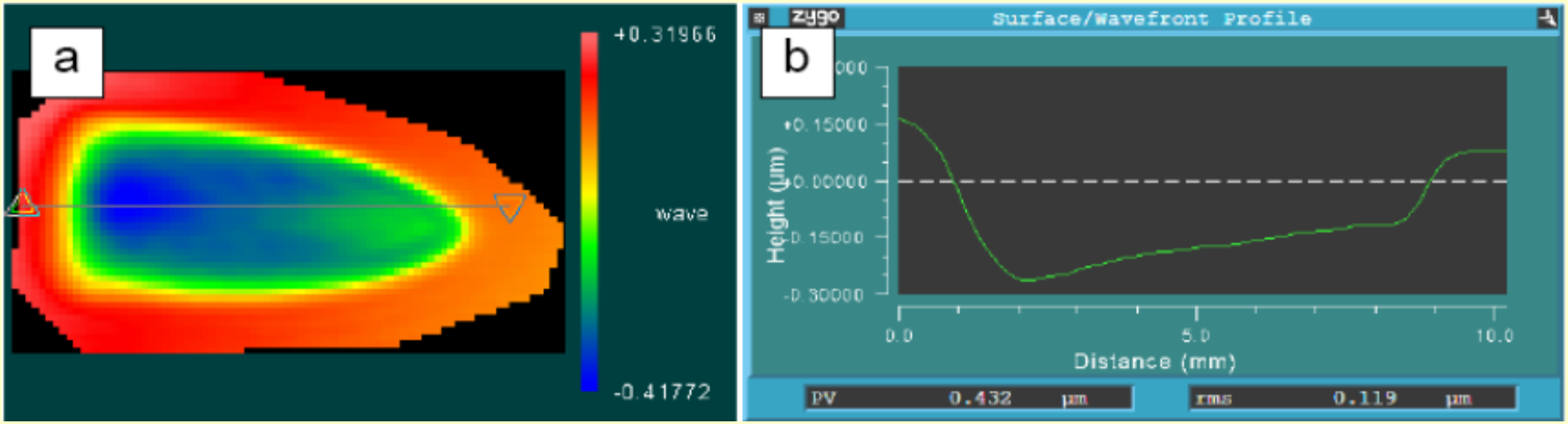
- Destructive: Taper polishing, Etching, Fracture Mechanics
- Non-Destructive Evaluation (NDE):X-ray diffraction, Scanning Acoustic Microscopy, Raman Spectroscopy, Birefringence, Photothermal Microscopy
  - Many of these techniques are qualitative, do not provide an accurate depth of SSD

SSD Measurements using MRF

- SSD measurements are taken using MRF spots to penetrate through SSD and calculate depth base on surface roughness and spot profile
  - MRF spots are taken at sequentially deeper depths until past the depth of SSD
  - Surface roughness measurements using a white light interferometer are made within the deepest region of the spot
  - Roughness decreases as the spot depth increases.
  - The depth of SSD is determined when the roughness levels and the spot is measured with an interferometer or profilometer

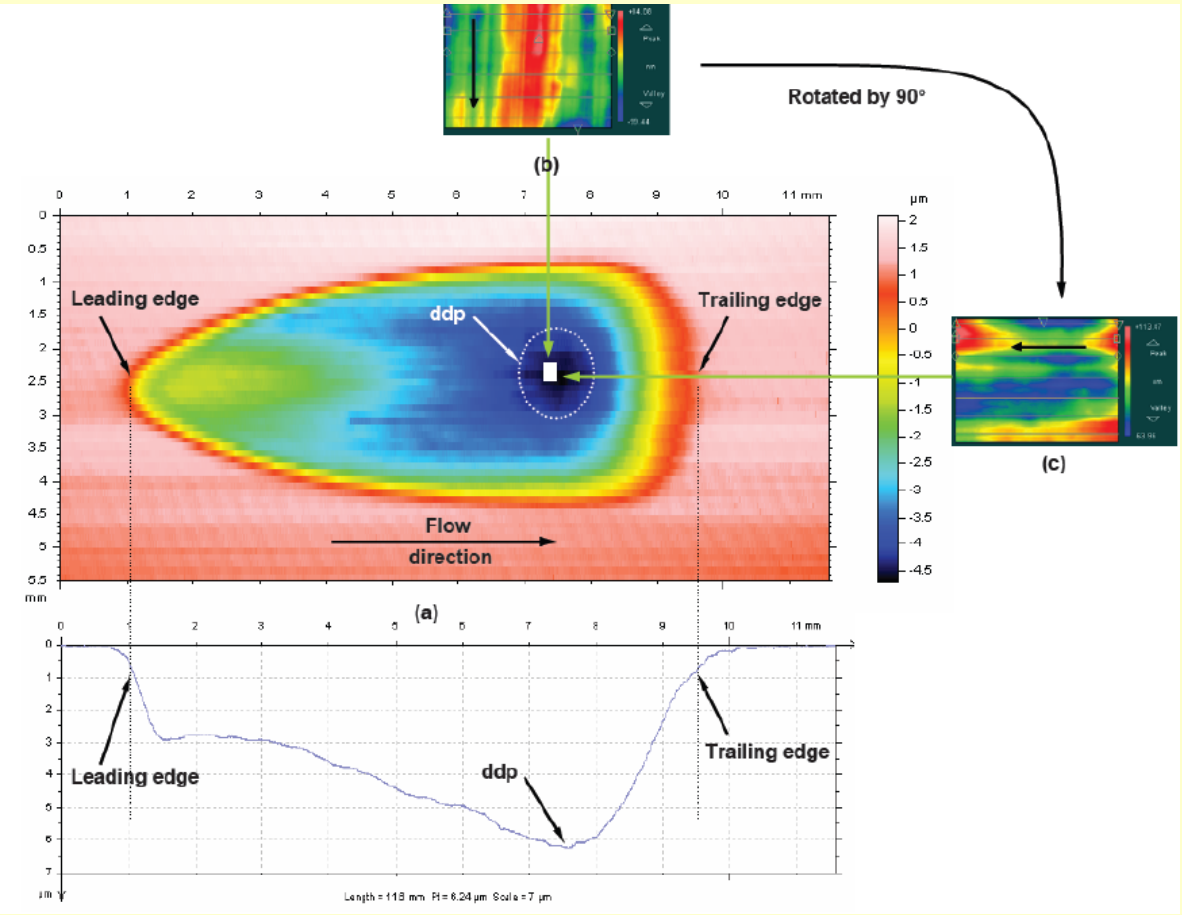
Measuring Spot Profiles

- Previous work shows a strong correlation between surface roughness and SSD-Good estimate of the required spot depth
- Applying this correlation spots with depths <0.5 µm can be profiled using an optical interferometer



MRF Spot Profile measured with an interferometer against a flat reference

Measuring Large Spots with Contact Profilometer



Spot profile from contact profilometer

- Interferometer scans within the deepest area of the spot are taken in a vertical and horizontal orientation to due to interferometer limitations
- Five line scans are collected within each spot, resulting in scan parallel and perpendicular to the fluid flow direction
- Roughness Data collected with NewView 5000, 20X Mirau Objective, 0.35X0.26, MinMod:3%

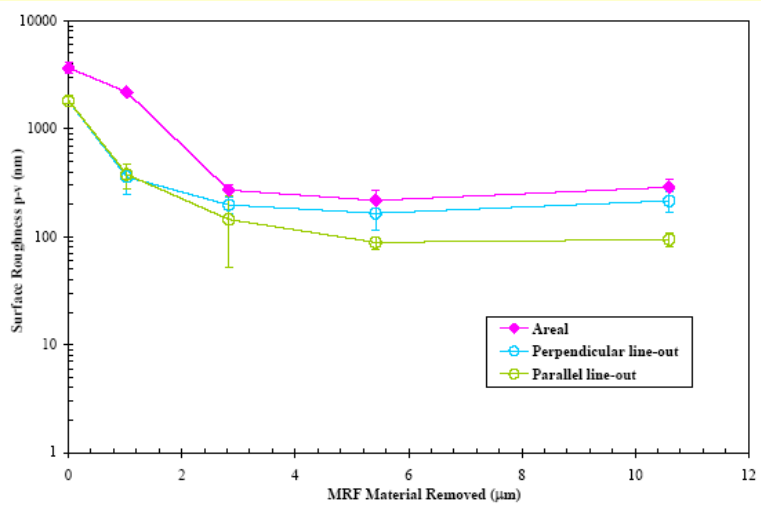
SSD Measurement Procedure

- 3-6 spots are placed on each surface depending on the surface roughness
- 5 random surface roughness measurements were collected within the deepest depth of penetration (ddp) parallel (‖) to direction of flow and perpendicular (⊥) to the direction of flow

Spot #	Time (min)	ddp (µm)	Removal rate (µm/min)	PV (nm)	Rms (nm)
As received	NA	NA	NA	1520 337.7	14 5.0
1	1	0.22	0.22	1261 368.5	114 0.8
2	2	0.34	0.17	258 56.9	16 1.8
3	6	1.32	0.22	139 19.7	23 6.14
4	18	2.86	0.16	129 13.7	21 5.5
5	36	5.95	0.17	181 16.9	31 4.7

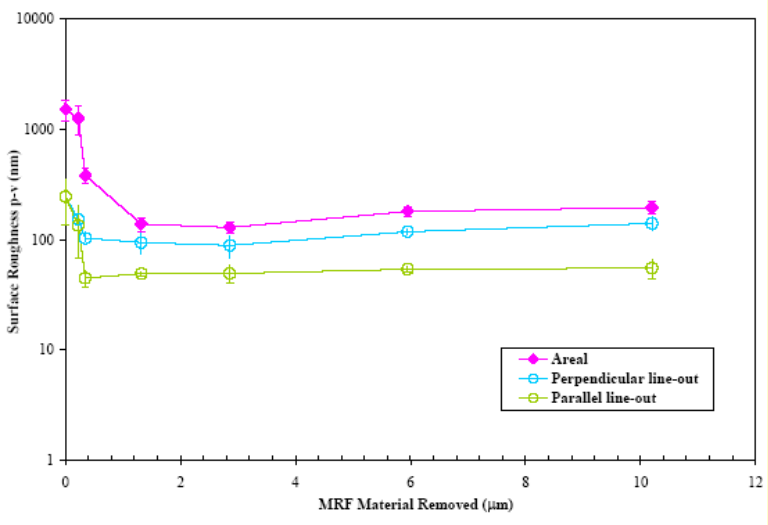
SSD of Etched and Lapped Surfaces

Chemical Etched Surface



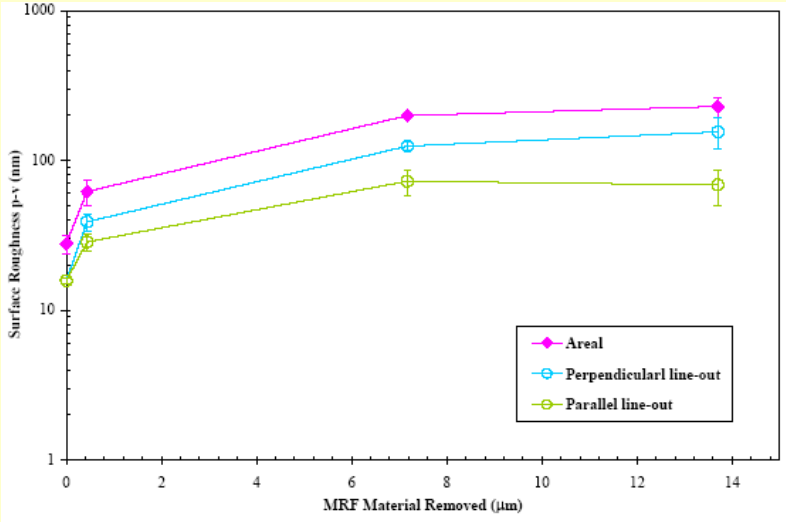
Depth of SSD is ~6µm. Etching has shown to be damage free; therefore, the depth is driven by surface roughness, not SSD.

3 µm lapped on steel

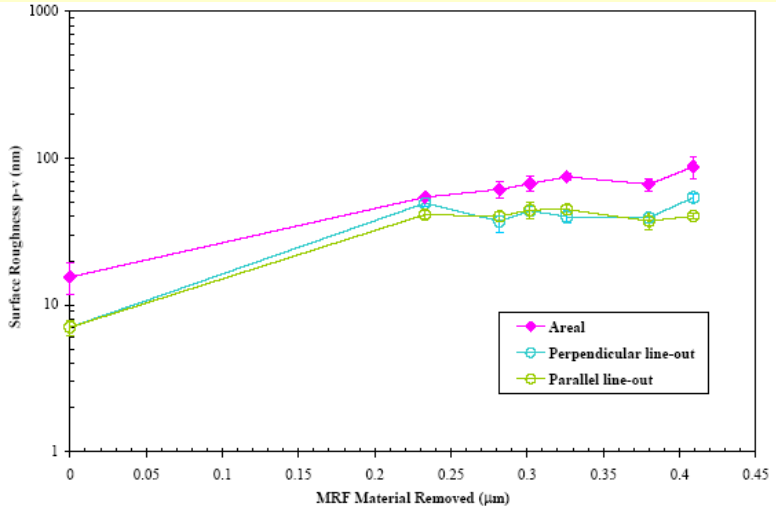


Depth of SSD is ~1.5 µm.

SSD Depth for Polished Surfaces



3 µm lap



1 µm lap

- Roughness increases as MRF removes material
- Spots are placed without part rotation with long dwell times, which causes increased roughness
- Destructive techniques have a resolution of ~ 0.5 µm, therefore SSD depth of ~1 µm is the low threshold

Summary

- Stress can be noticed in surfaces polished with diamond abrasives as small as 1 µm
- SiC lapped against steel with 3 µm diamond results is SSD depth of ~1.5 µm
- Twyman Effect shows the difference in stress between 3 µm diamond and 1 µm diamond polishing and that MRF relieves stress from 1 µm diamond
- SSD can not be measured using MRF for 3 µm and 1µm diamond polishing

Acknowledgements

This material is based upon work supported by the Missile Defense Agency through ATK-MRTS under the contract No. SC-0035-98-2700 Order No. PSU-01

Shai Shafrir and Chunlin Miao are LLE Horton Fellows. Research by Shai Shafrir, Chunlin Miao and Stephen Jacobs was sponsored in part by the U.S. Army Armament, Research, Development and Engineering Center (ARDEC) and was accomplished under Cooperative Agreement Number W15QKN-06-R-0501 and the U. S. Department of Energy Office of Inertial Confinement Fusion under Cooperative Agreement No. DE-FC52-92SF19460, the University of Rochester, and the New York State Energy Research and Development Authority. The views and conclusions contained in this document are those of the authors and should not be interpreted as representing the official policies, either expressed or implied, of U.S. Army ARDEC or the U. S. Government. The support of DOE does not constitute an endorsement by DOE of the views expressed in this article. The U.S. Government is authorized to reproduce and distribute reprints for Government purposed notwithstanding any copyright notation herein.