Acoustic Spectroscopy of Silicon Carbide Mirror Materials

Richard Haber

Andrew Portune

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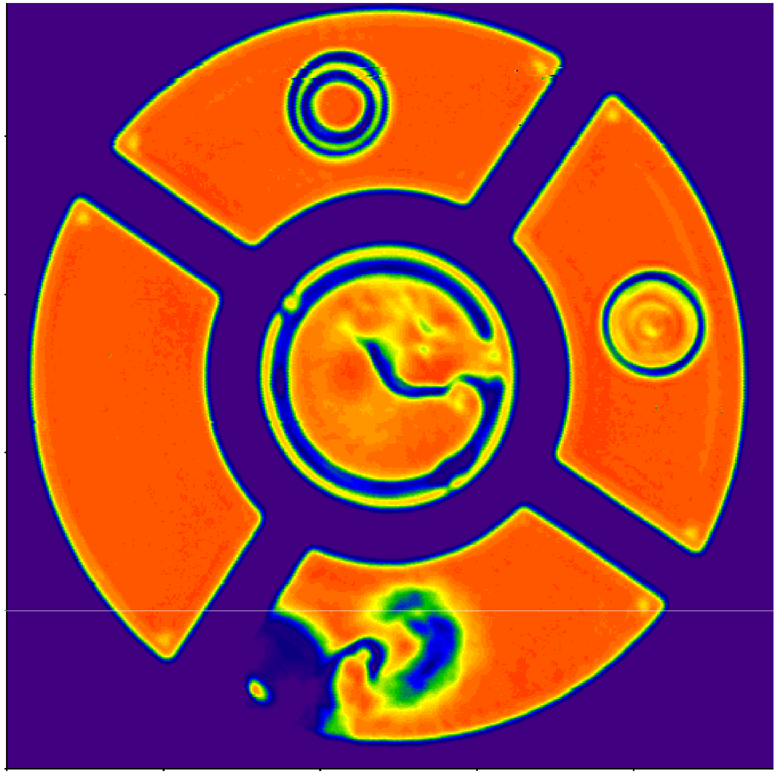
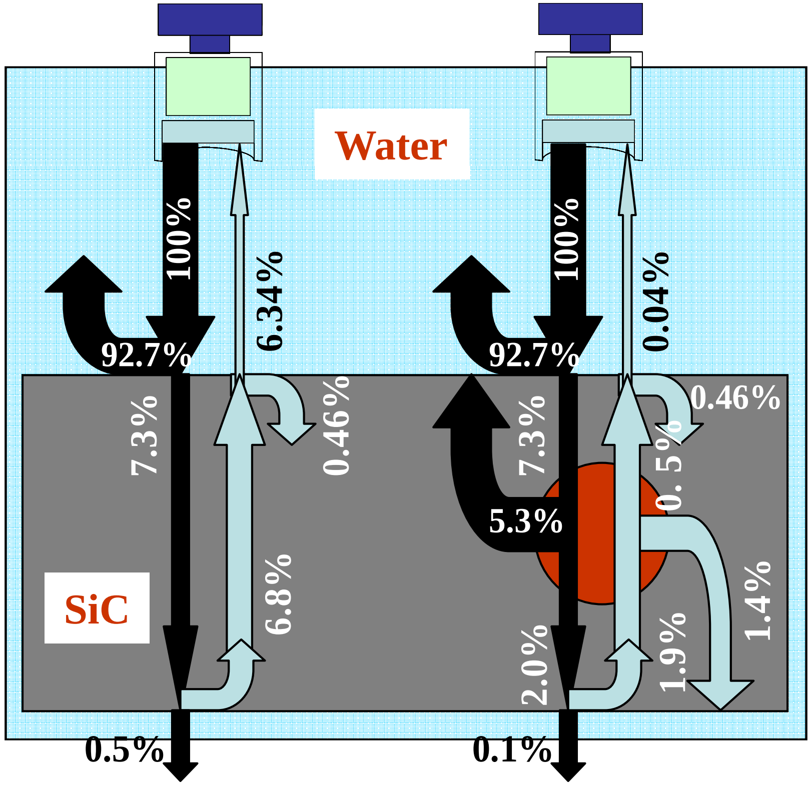
Outline of Presentation

- Ultrasound Fundamentals
- C-Scans of SiC Mirrors
  - Single Transducer
  - Phased Array
- Acoustic Spectroscopy
  - Fundamentals
  - Defect Engineered SiC
  - Area Spectroscopy Maps
- Summary and Conclusions



Ultrasound Fundamentals

- **Time-of-Flight (TOF)** – travel time of an acoustic wave through a material from top to bottom surface
- **Material Velocity (c)** – speed at which ultrasonic vibrations pass through a material which is dependent on elastic properties of material and mode of vibration
- **Acoustic Impedance (Z)** – the product of sound pressure to particle velocity in a medium
- **Attenuation** – the loss in acoustic energy that occurs between two points of travel which is the combined effect of scattering and absorption



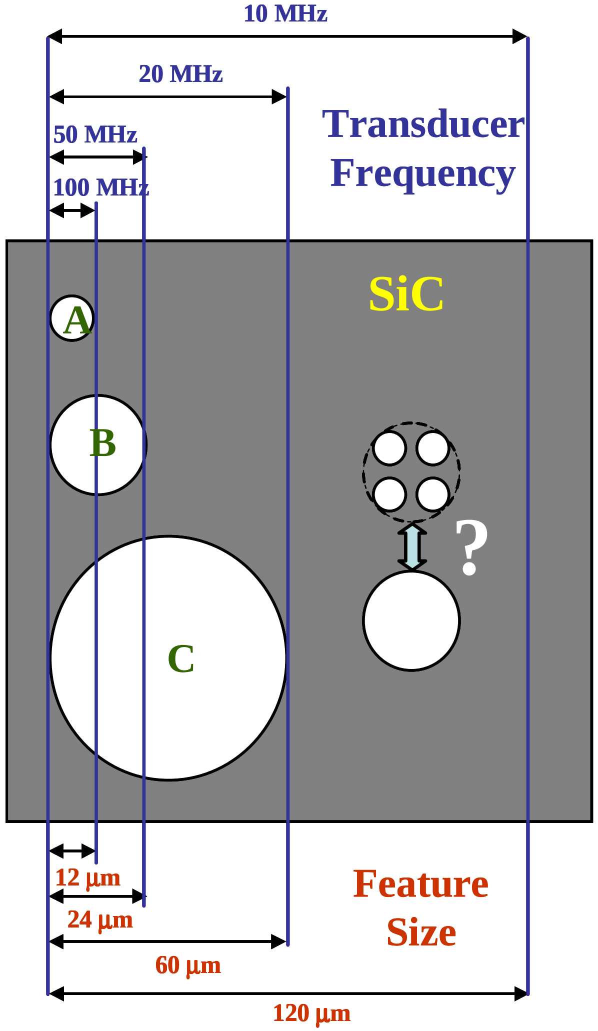
Reflection/Transmission of SiC in Water

$$Z = \rho C$$

$$C = f \cdot \lambda$$

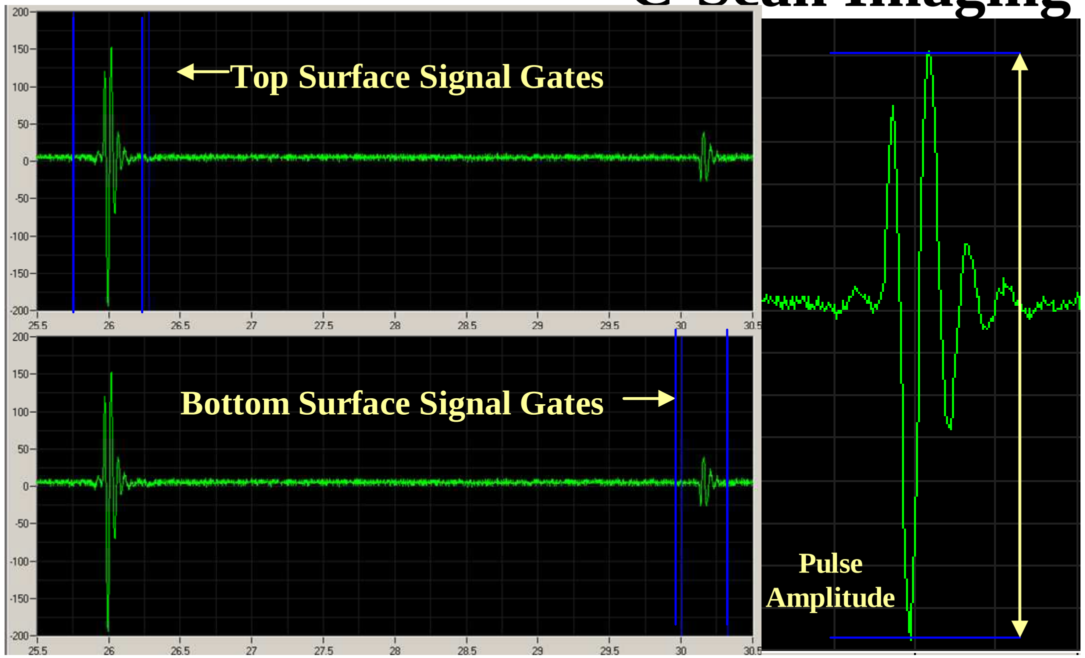
$$R = (Z_2 - Z_1)^2 / (Z_2 + Z_1)^2$$

$$T = 4Z_1Z_2 / (Z_2 + Z_1)^2$$



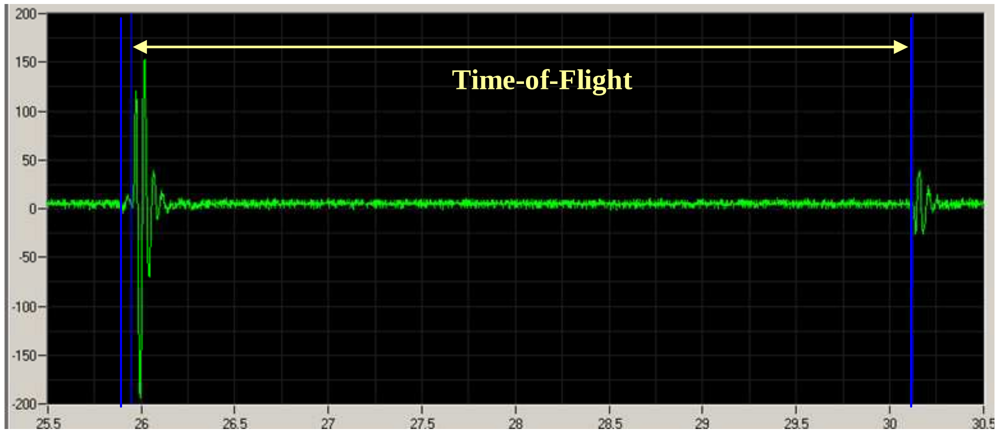
- **Higher frequency equates with smaller wavelength**
- ***Top surface peak representative of surface and sub-surface homogeneity***

C-Scan Imaging Modes



Amplitude C-Scans

- Attenuation due to scattering caused by features in sample one-tenth or less than size of λ reduces peak amplitude
- Higher f causes increased scattering of smaller features to image local changes



Time of Flight C-Scans

- Time of flight (TOF) related to c, ρ, ν, E, G, and K and Z
- Higher TOF equates with lower materials velocity and therefore lower density and elastic properties

ρ (g/cm^3)	c_l (m/s)	c_s (m/s)	ν	E (GPa)	G (GPa)	K (GPa)
3.01	11,500	7,320	0.161	374	161	184
3.09	11,800	7,530	0.159	406	175	198
3.15	12,050	7,700	0.157	431	186	210

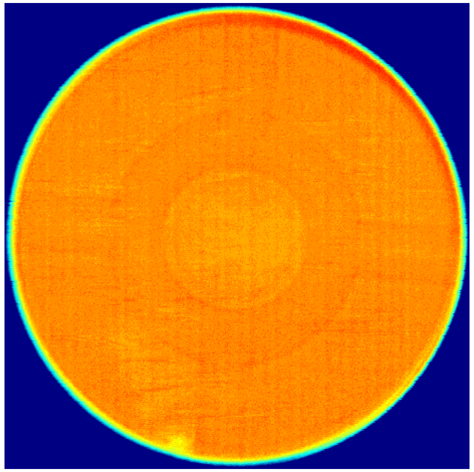
Material Velocity vs. density and Elastic Properties for SiC

TOF

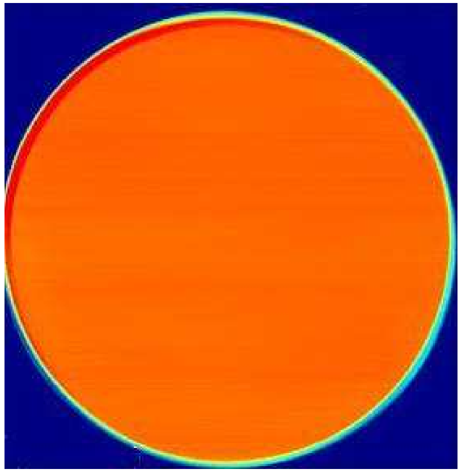
TOF = C / t

C = Z / ρ

Top Surface Reflected Signal Amplitude – M-Cubed

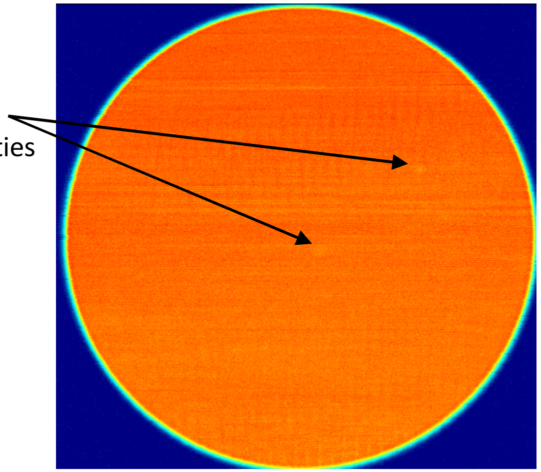


5MHz Top Surface Reflected Signal Amplitude



15MHz Top Surface Reflected Signal Amplitude

Subsurface Inhomogeneities



125MHz Top Surface Reflected Signal Amplitude

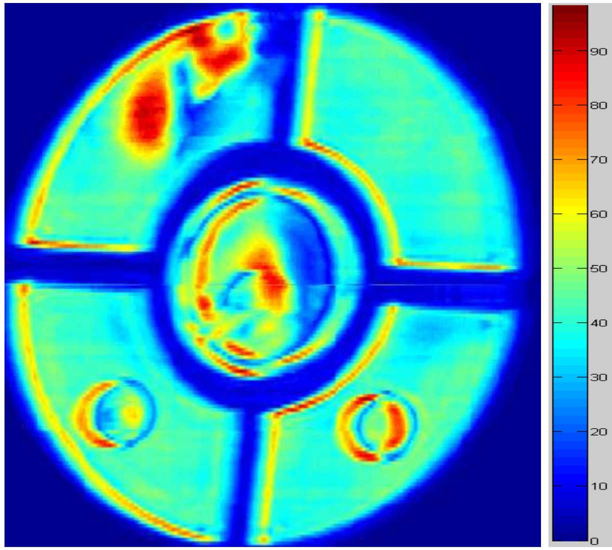
- 5MHz image reveals support structure
- Lower amplitude region caused by imprinted pattern on sample's bottom surface
- 125MHz image shows two small lower amplitude circles where the reflected signal was weaker
- This is likely due to weakly bonded or lower density areas

Why are we using NDE on mirrors?

- For SiC, the most apparent reason is to remove Griffith sized flaws – Why? *Obvious* defects reduce mechanical integrity
- What is obvious? 1mm? 100um, 10um, 100nm?
- Is NDE to strictly cull “bad” blanks from “good” blanks?
- Are blanks that show no *obvious* defect good?

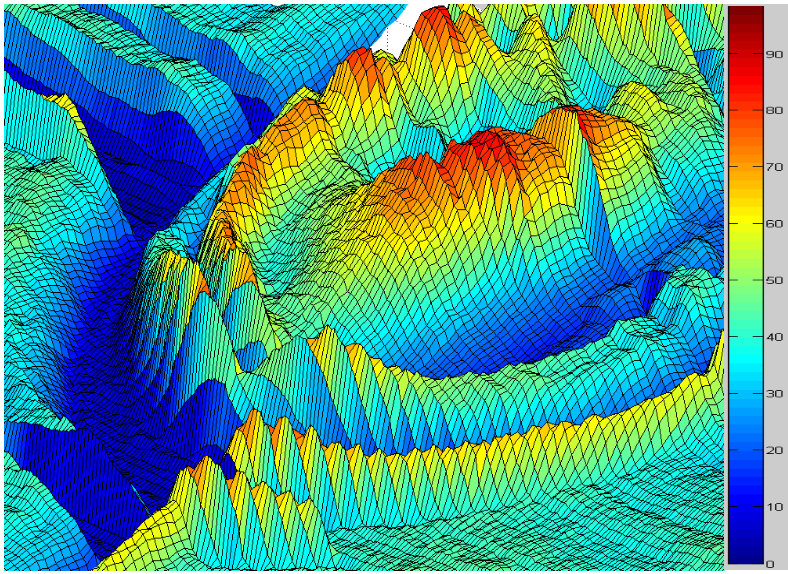
Now we start crossing into where NDE and Non Destructive Spectroscopy become relevant

10MHz Phased Array Study of M-Cubed SiC Mirror

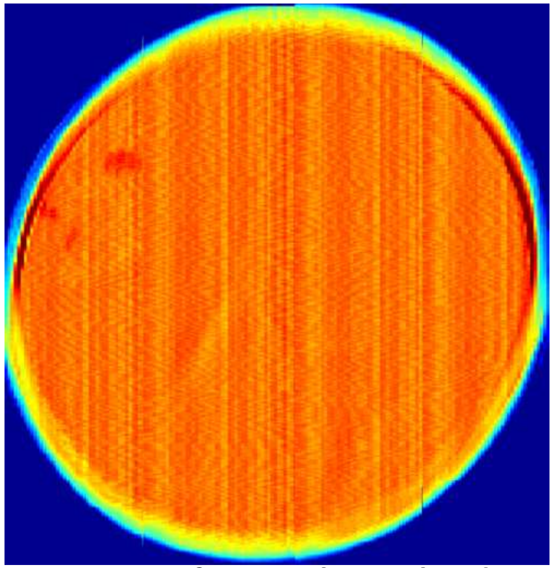


Amplitude of back surface reflection

Photograph of sample bottom



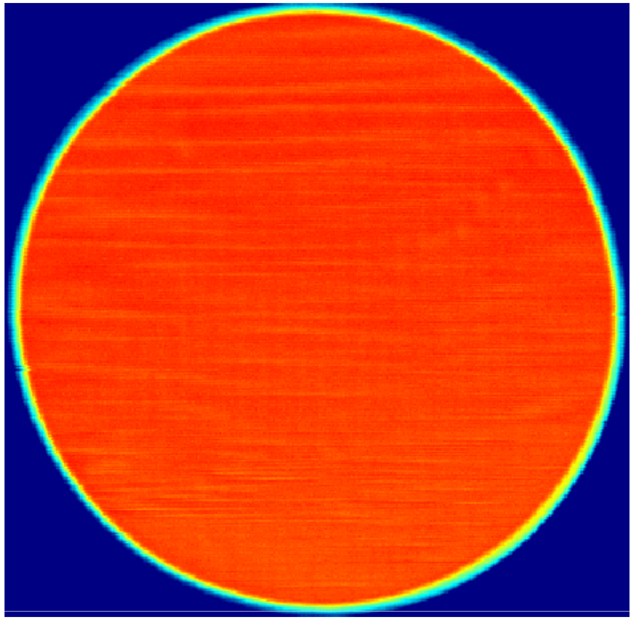
Mesh plot of central region



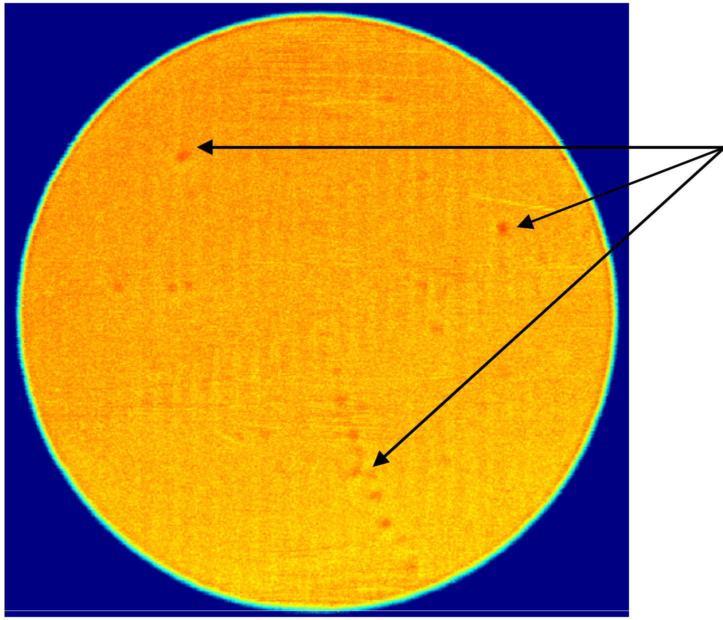
Top Surface Peak Amplitude

- Scan of back surface reflected signal amplitude show abnormalities which correspond to surface aberrations
- Full scan took 2 minutes to run with phased array – scans take 20 minutes with single transducer
- Top surface scan reveals small abnormality in upper left corner

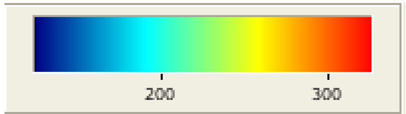
Top Surface Reflected Signal Amplitude – Schafer



15MHz Top Surface Reflected Signal Amplitude

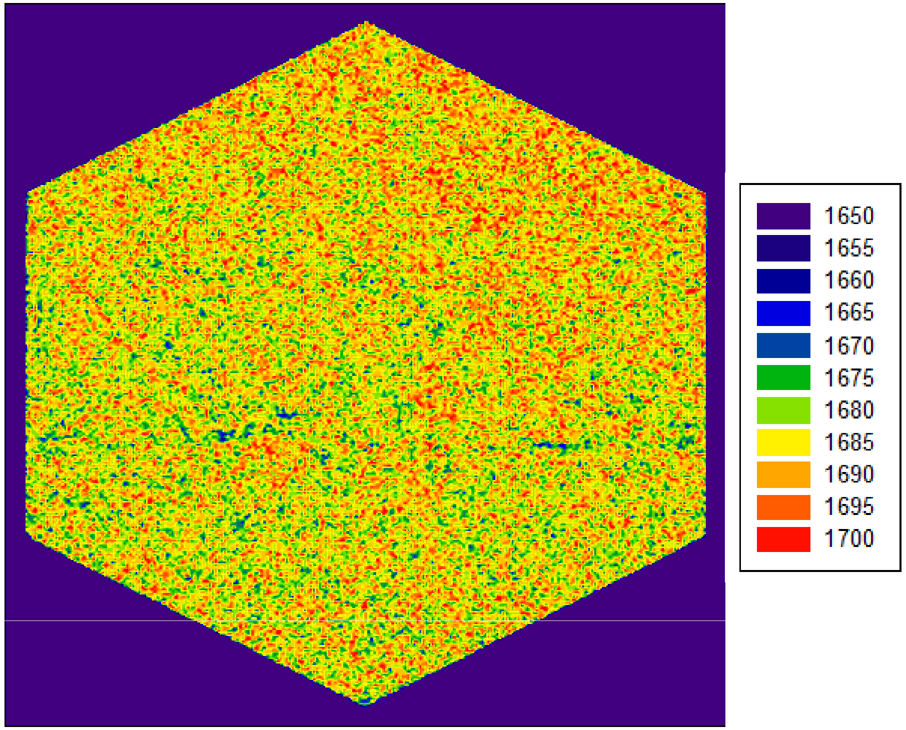


125MHz Top Surface Reflected Signal Amplitude

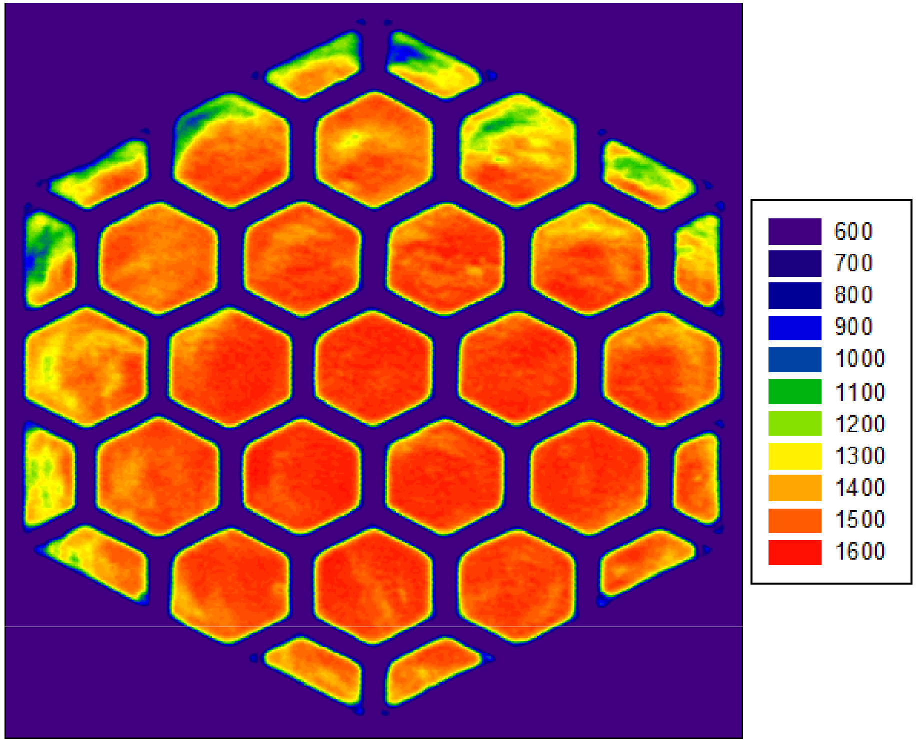


- 15MHz image shows homogeneous surface
- 125MHz scan reveals inhomogeneous areas in the substructure
- These areas caused a greater reflected signal amplitude and thus exhibit a higher acoustic impedance
- Likely caused by local high density regions

Reflected Signal Amplitude – SSG SiC Mirror



20MHz Top Surface Reflected Signal Amplitude

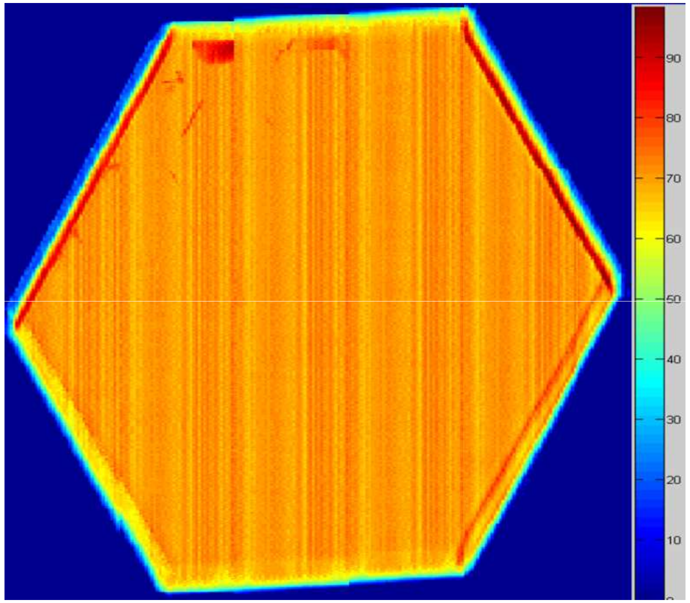


20MHz Bottom Surface Reflected Signal Amplitude

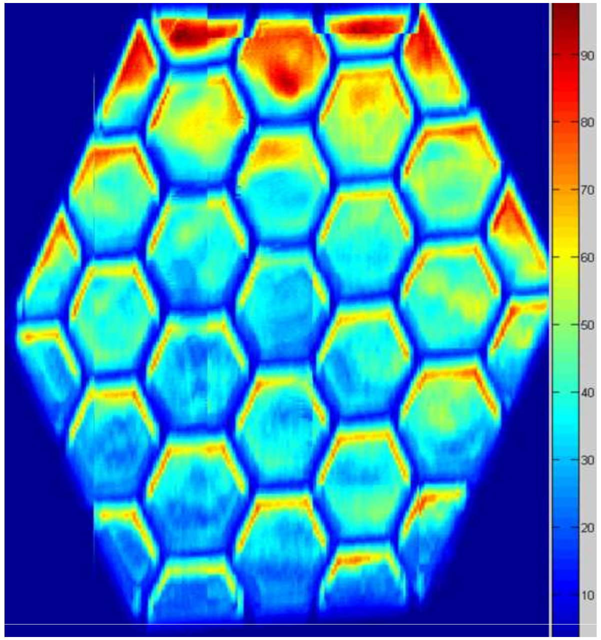
- Top surface amplitude map reveals only slight variations in signal amplitude – no significant subsurface flaws
- Bottom surface map clearly reveals hexagonal support structure
- Local variations in bottom surface amplitude could indicate a lack of microstructural homogeneity within the bulk of the mirror

10MHz Phased Array Study of Hexagonal Backed SiC Mirror

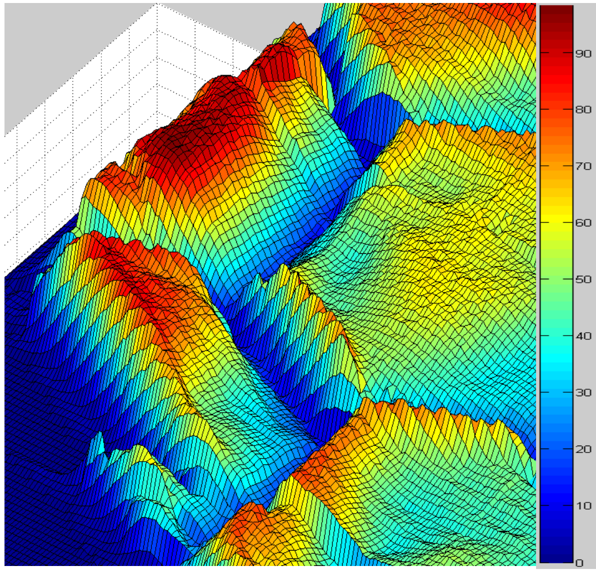
- Scan of back surface reflected signal amplitude reveals the hexagonal backing matrix
- Variations seen within each hexagonal area indicative of differences in the microstructure of the mirror sample
- Mesh plot reveals the extent of variations in the upper-left corner
- Top surface scan shows some high amplitude features in the upper left of the sample



Top Surface Peak Amplitude

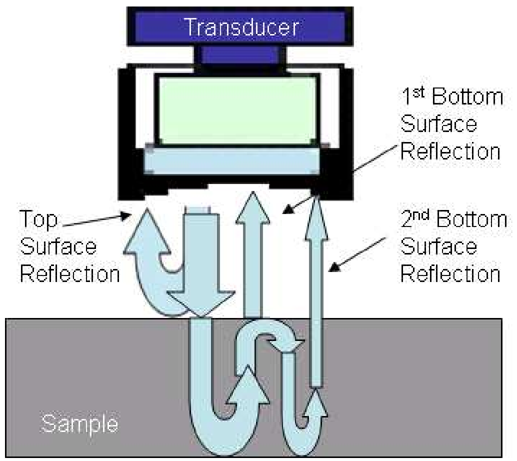


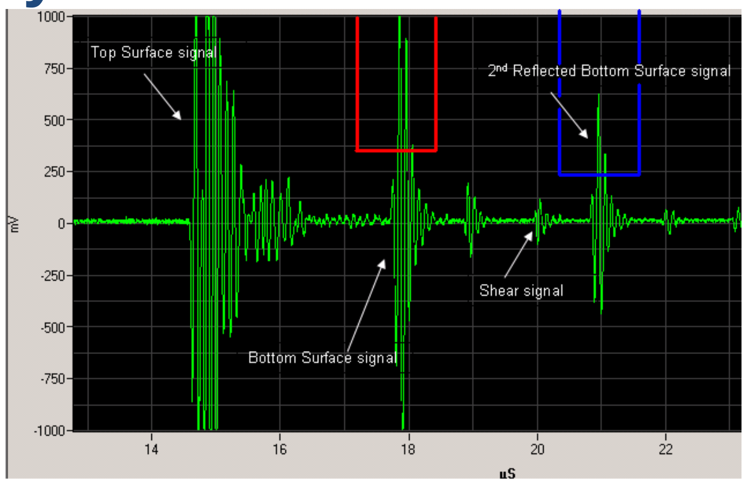
Bottom Surface Peak Amplitude



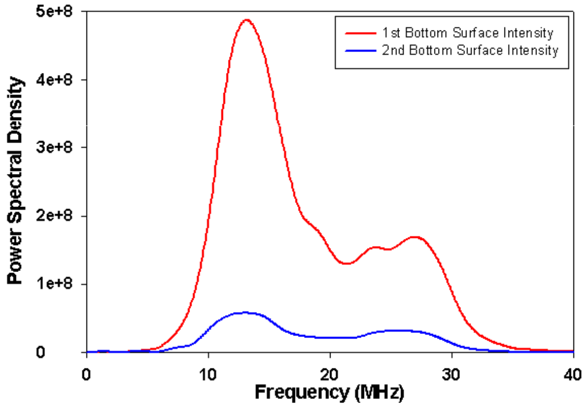
Mesh plot of upper left corner

Acoustic Spectroscopy in Elastic Solids



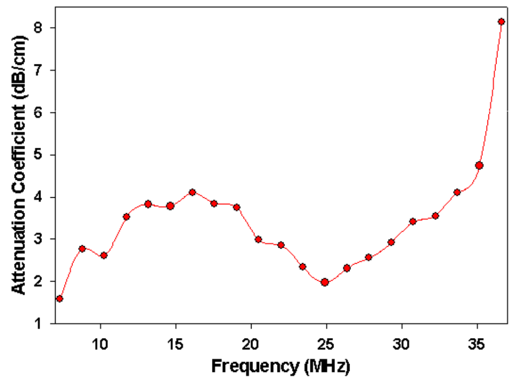


FFT



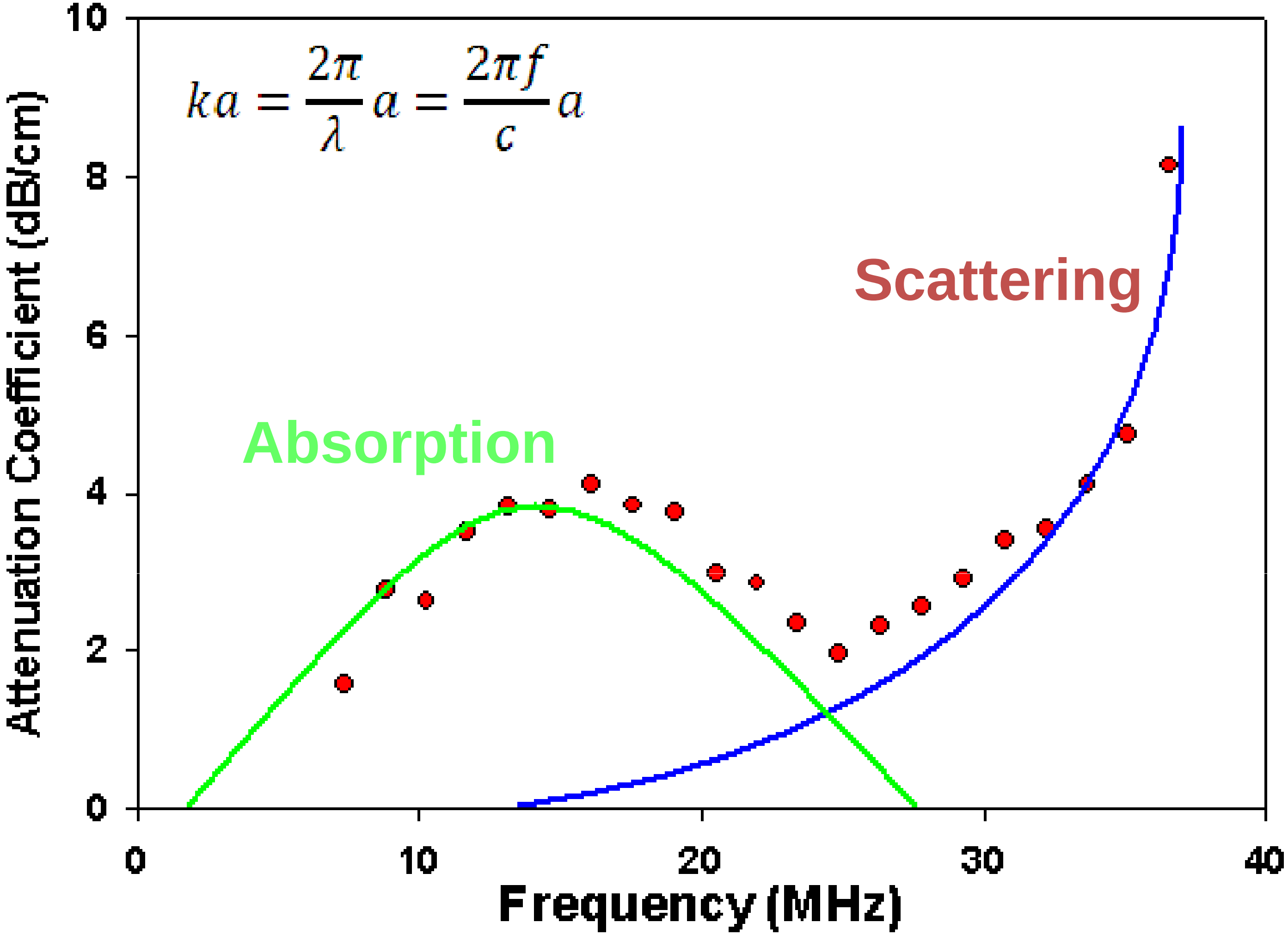
$$\alpha = -\frac{8.686}{2 * \text{thickness}} \ln\left(\frac{I}{I_o}\right)$$

Beer – Lambert Law



- Select back-wall reflections from ultrasound A-Scan of sample
- Use the fast-Fourier transform (FFT) on peaks to obtain the power spectral density (PSD)
- Apply the Beer-Lambert law to determine attenuation as a function of frequency

Acoustic Loss Mechanisms – Absorption and Scattering



Absorption

$\alpha_{Absorption} = A * f^2 / a^2$

- Dominates at low ka values
- Exact equations unknown for elastic solids

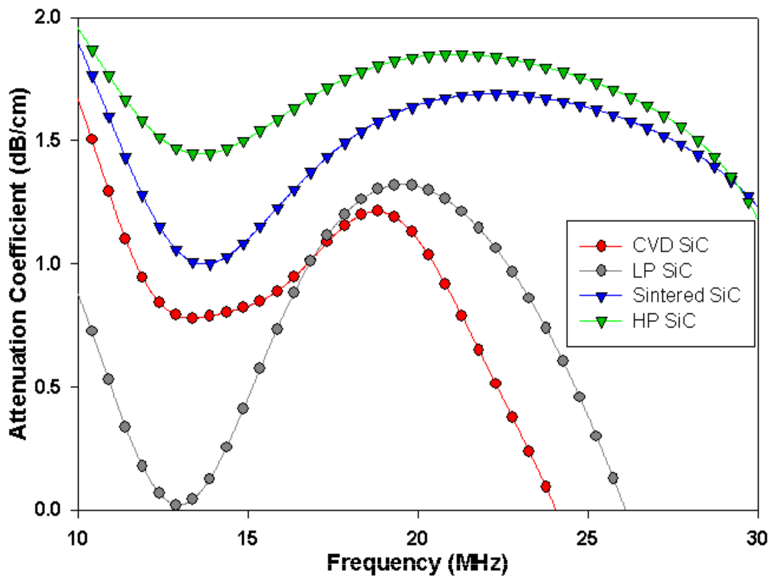
Scattering

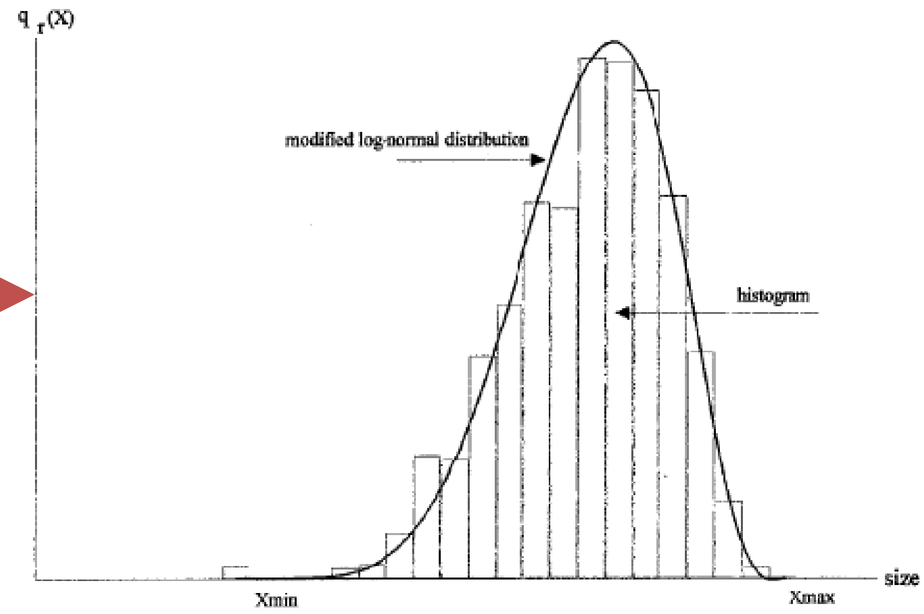
$\alpha_{scattering} = \Sigma\, \sigma_{s,\,Mie}$

- Dominates at high ka values
- Must use Mie solution for scattering cross section

- Mechanism responsible for acoustic attenuation is dependant on the frequency of ultrasound and size of the heterogeneity
- Degree of elastic mismatch describes the severity of signal loss

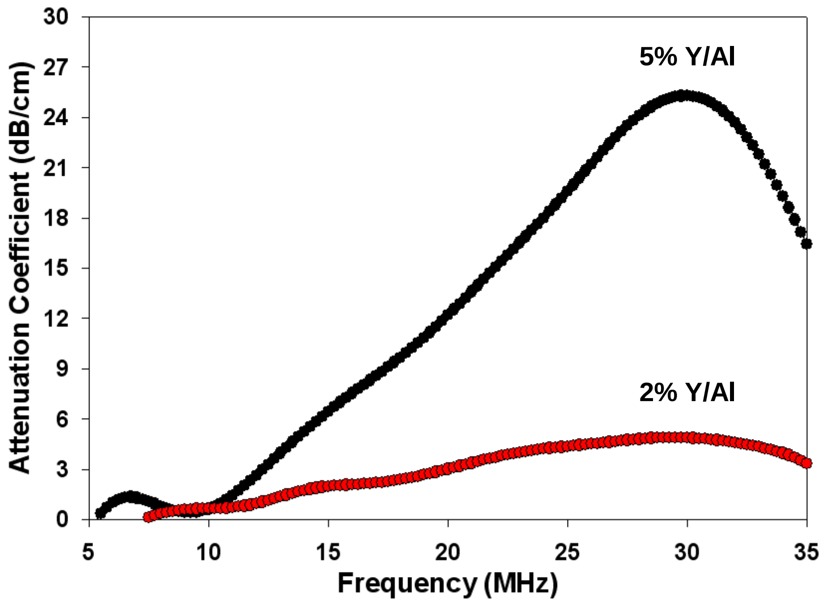
Determining the Defect Size Distribution from Acoustic NDC

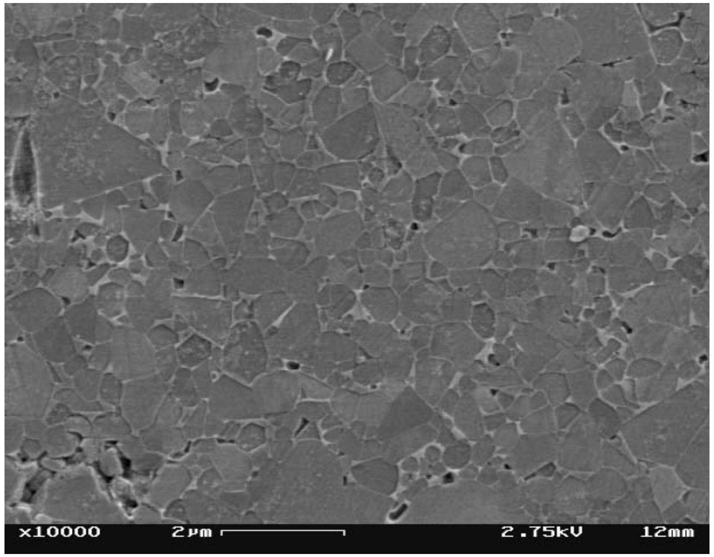




- Apply acoustic physics to attenuation spectra to determine particle size distribution of pores and inclusions
- Current difficulties in implementation:
  - Absorption physics have not been thoroughly explored for elastic solids
  - Looking at low density inclusions in a high density matrix – opposite of literature
- Potential solutions
  - Move to frequency regime where scattering dominates
  - Create and study a series of known samples to develop physics of acoustic attenuation due to absorption by heterogeneities in an elastic solid

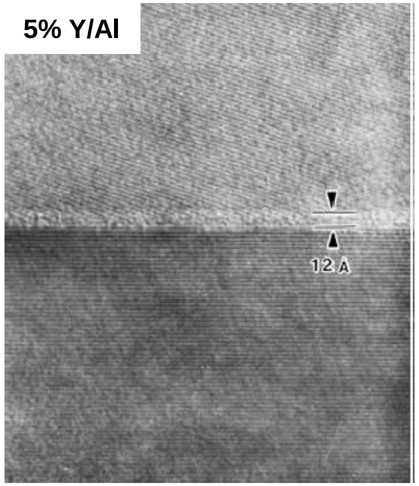
Microstructure and Ultrasound NDE

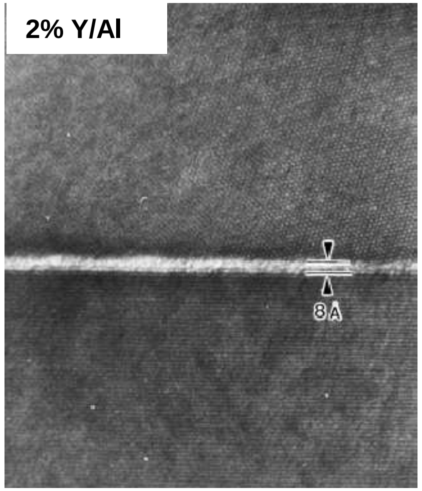




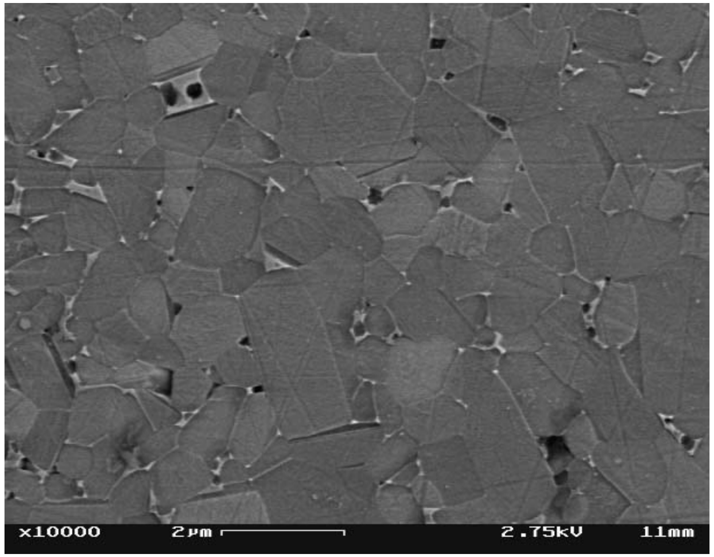
95 wt % SiC, 3.22 wt% Al_2O_3, 1.78 Y_2O_3

How does GB thickness affect fatigue failure?





HRTEM Showing GB Thickness

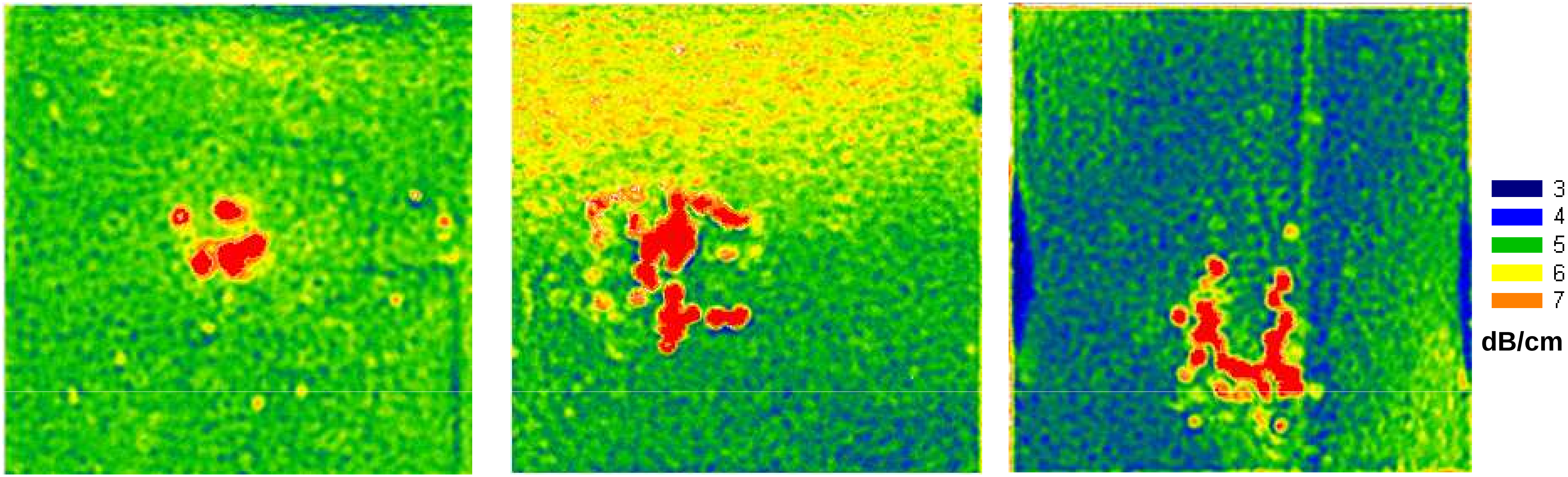


98 wt% SiC, 1.29 wt% Al_2O_3, 0.71 Y_2O_3

Ultrasound Investigation of Defect Engineered SiC

20MHz Overall Signal Attenuation Coefficient C-Scans

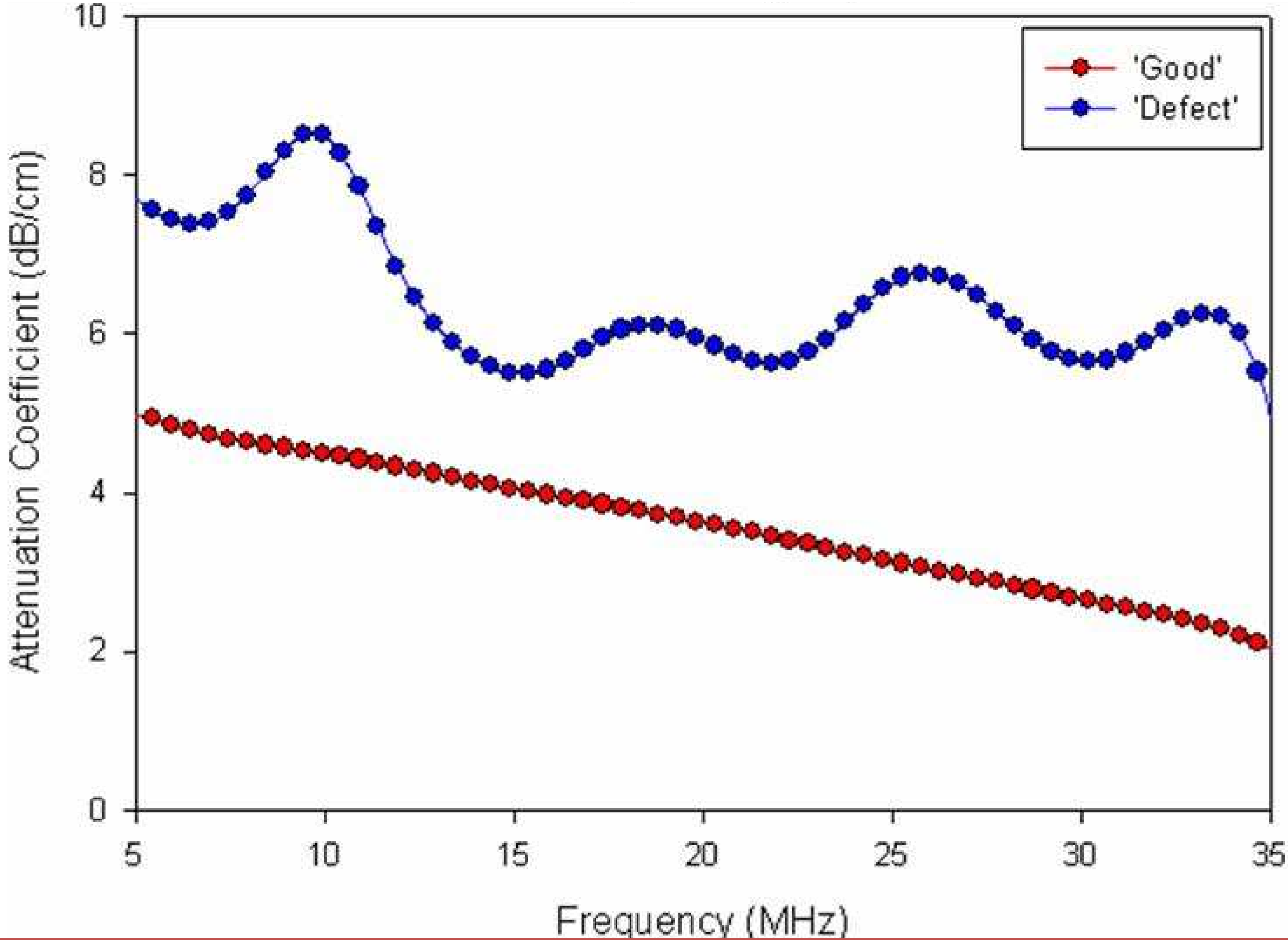
Tile 38 | Tile 77 | Tile 147



Chemical Inhomogeneity | Pitting | Non-spherical Pits

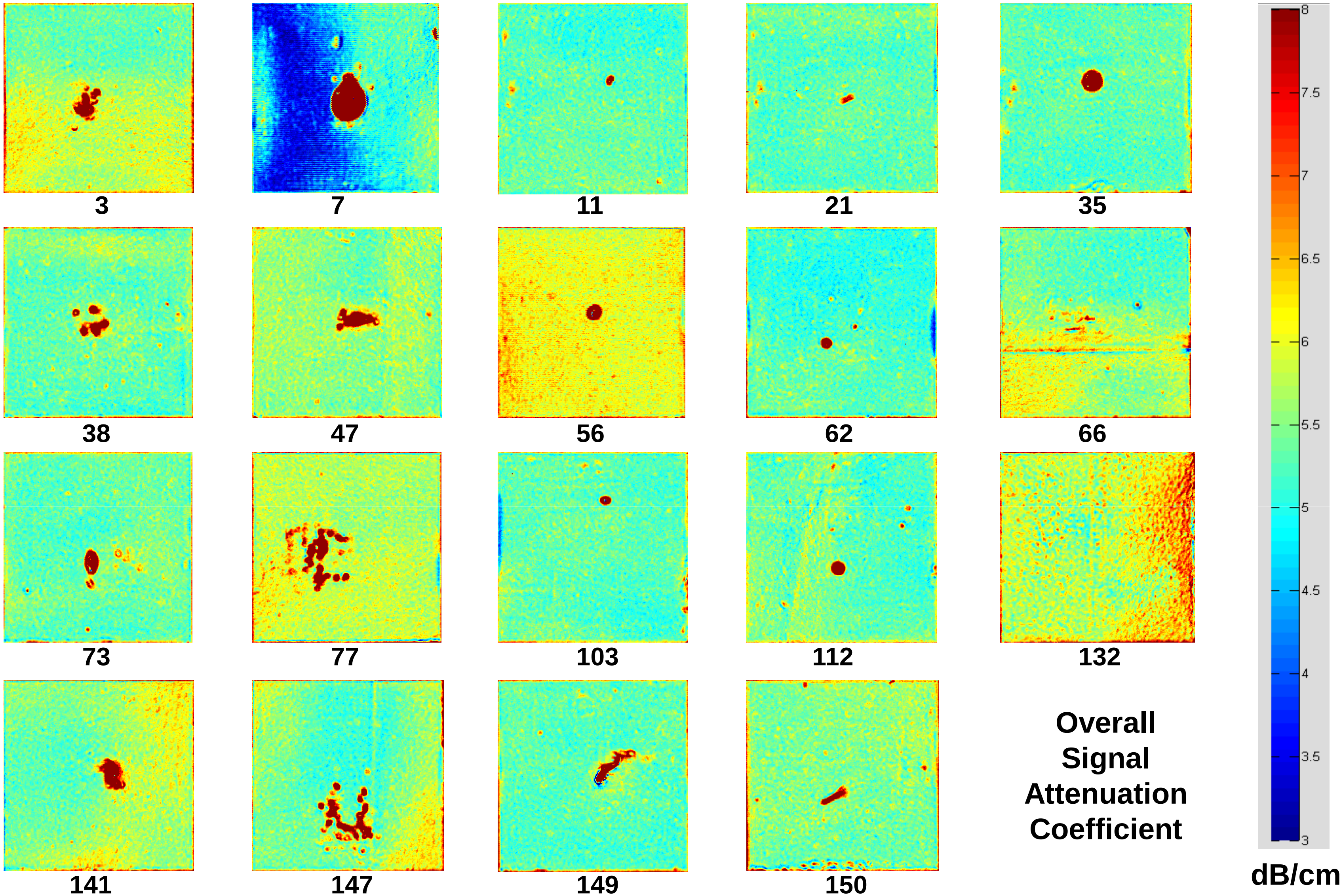
- **20 defect engineered sintered SiC tiles acquired and tested for overall signal attenuation coefficient and elastic properties**
- **Large inclusions show attenuations 2 – 5x higher than mean**
- **Attenuation spectra can be used to get a ‘signature’ of each defect type**
- **Inclusions could damage microstructures in the local vicinity**

Defect Engineered SiC Acoustic Frequency Spectra



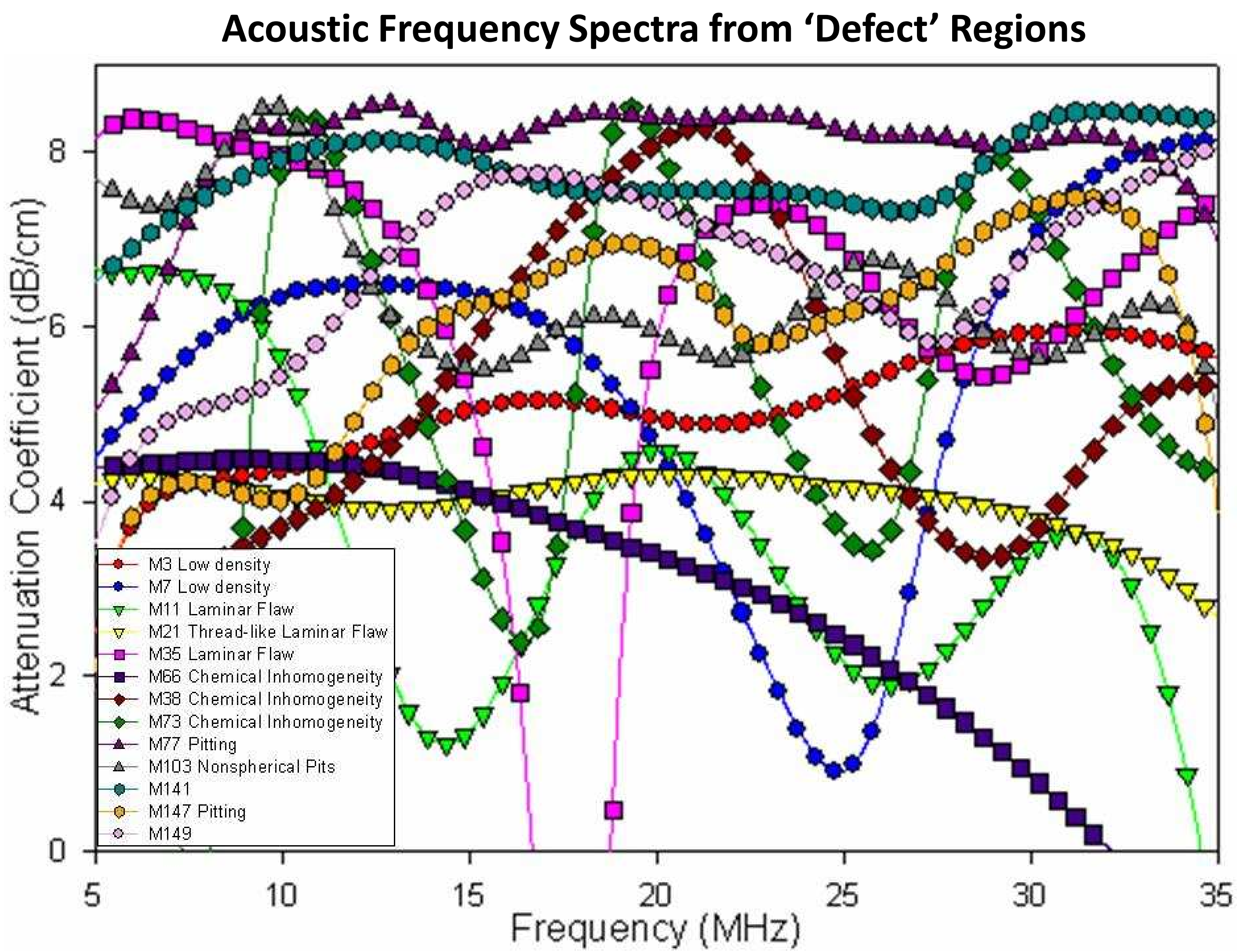
- 'Defect' region shows attenuation behavior characteristic of the inclusion's composition, geometry, and size
- Frequency dependant attenuation studies of defect engineered materials provide insight into how specific defects cause ultrasound energy loss

Defect Engineered SiC C-Scans



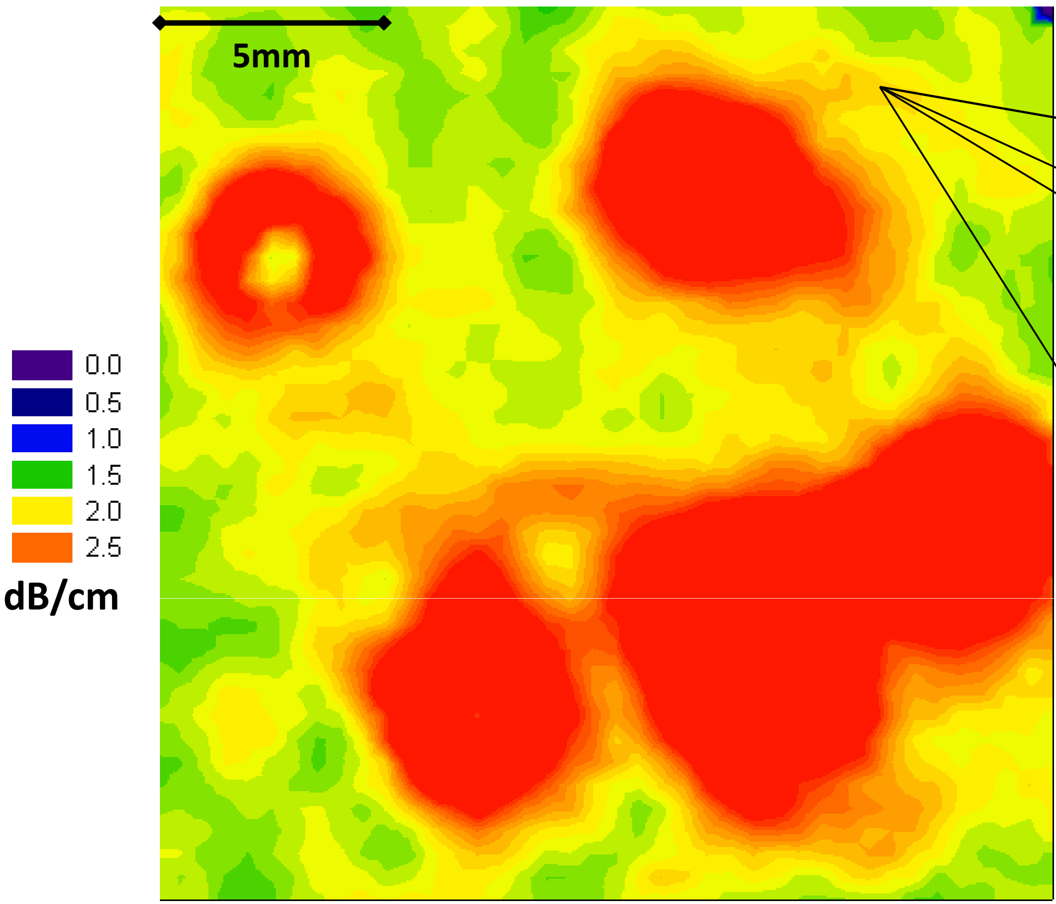
- Specific defects show distinct scanning patterns
- More detailed analysis possible using acoustic spectroscopy

Defect Engineered SiC Acoustic Frequency Spectra

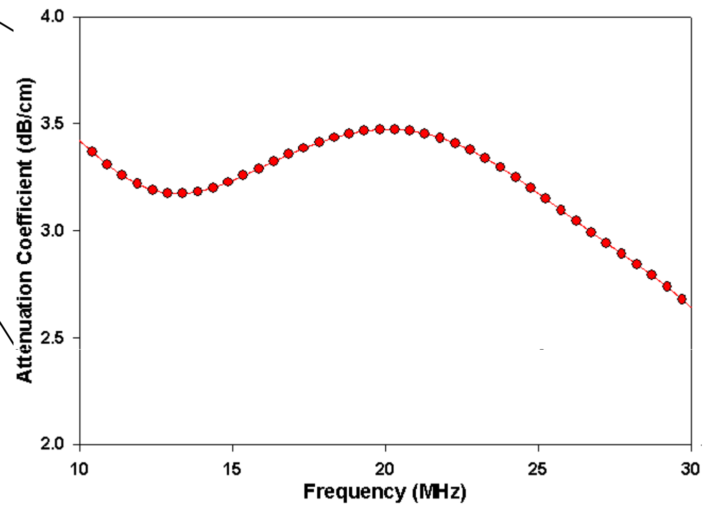


- Each specific inclusion type has its own frequency dependant behavior
- No common pattern exists between defect types
- Extension of study to higher frequencies could provide additional insight into ultrasound loss mechanisms

Acoustic Spectroscopy Maps



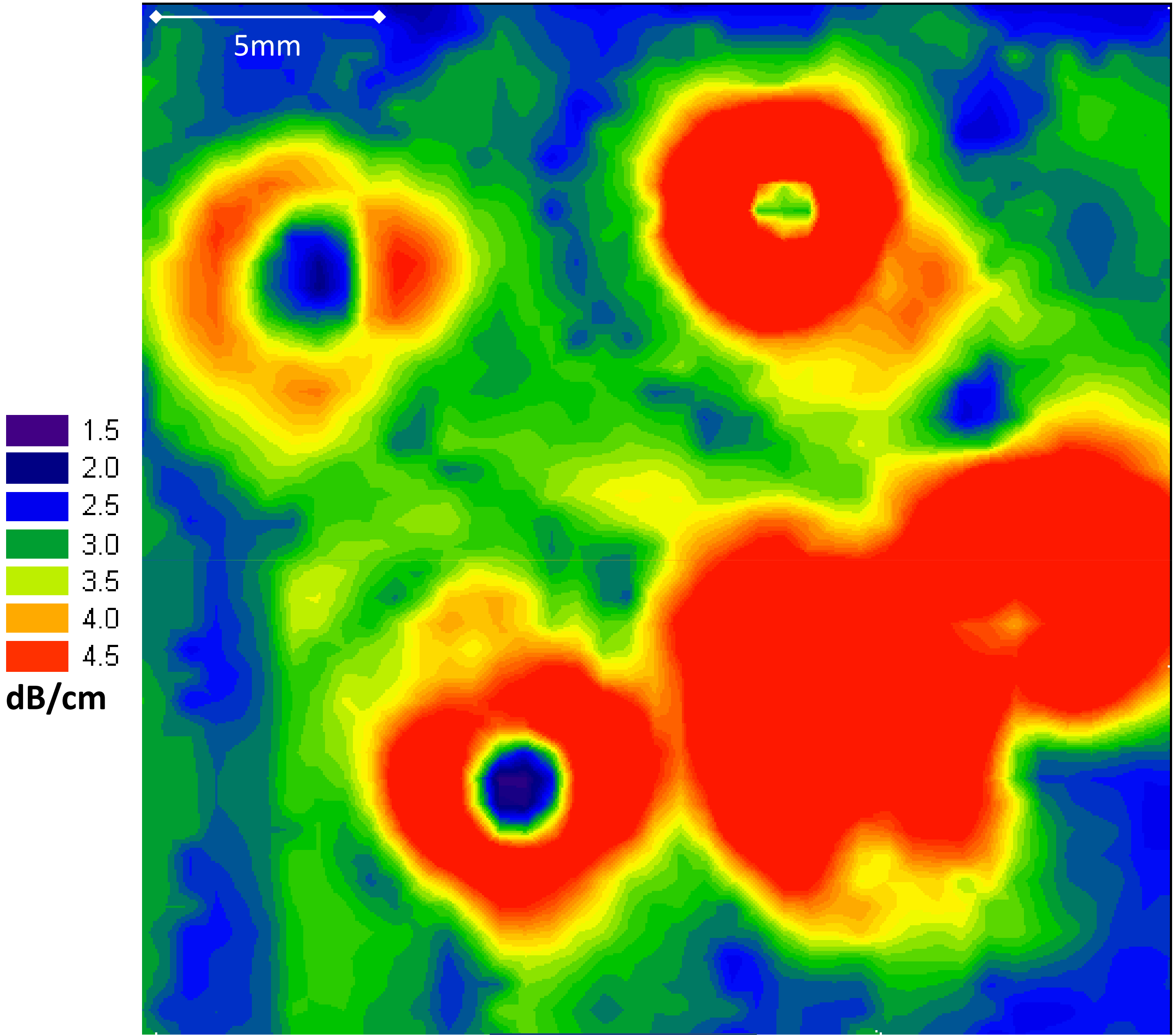
Overall Signal Attenuation Coefficient Map – Tile38



Acoustic Frequency Spectra

- Acoustic spectra have been measured for each point in the above graph
- Maps can be made for the attenuation over the area at a single frequency
- Cycling through the series of graphs enables a visualization of acoustically differentiable microstructure in the measured area

Attenuation Coefficient Map Series 10-30MHz

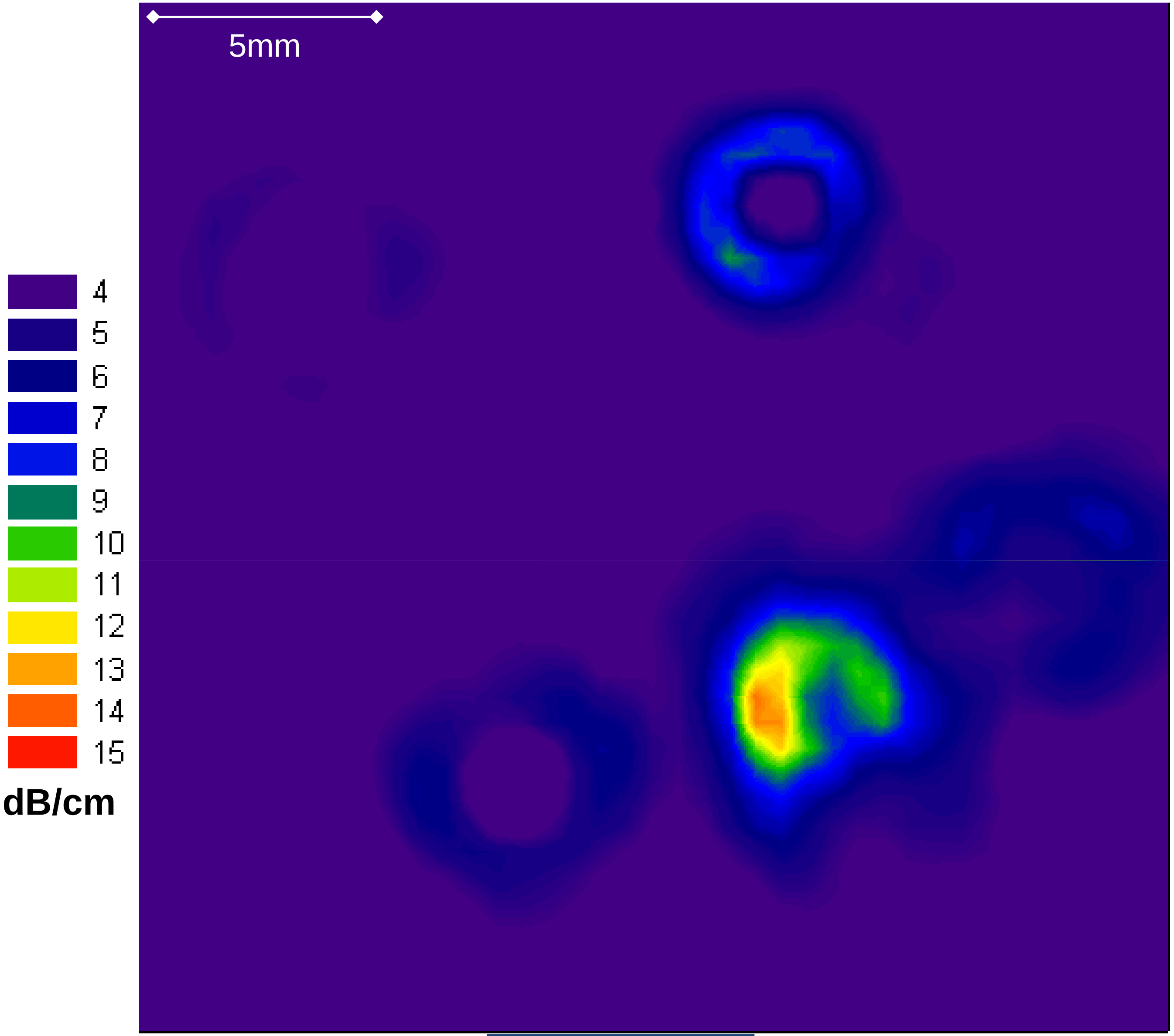


10MHz

Scale zoomed in to see changes in microstructure around silicon inclusions

- Defect areas saturated due to smaller scale used
- Minute differences seen in acoustic response
- Areas in between silicon defects show higher attenuation than areas farther away
- Images demonstrate inherent inaccuracy in taking single point measurements

Attenuation Coefficient Map Series 10-30MHz



10MHz

Scale zoomed out to see changes in silicon defect areas

- Non-defect areas saturated out due to scale used
- Defects display resonant behavior predicted by Mie mathematics
- Resonances are different for each inclusion
- Specific resonant frequencies rely on defect size, morphology, and composition

Conclusions and Summary

- Subsurface flaws in SiC mirrors are found more easily with *lower* frequency scans
  - Lower frequencies penetrate into the subsurface farther
  - Lower lateral resolution is compensated by greater depth interacted with
  - Phased array allows for tighter beam control facilitating ultrasound scans
- Acoustic spectroscopy allows for advanced microstructural characterization
  - Attenuation spectra show contributions of absorption and scattering to energy loss
  - Specific defect compositions and sizes exhibit significantly different acoustic signatures, enabling enhanced characterization of specific flaws
  - Acoustic spectroscopy area maps allow for advanced quantitative and qualitative analysis of large sample areas
  - Large inclusions show resonant behavior which could be used to predict their composition, size, and morphology

Acknowledgements

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