

Nano-indentation as a characterization technique for implant retrievals

Arevalo, SE¹; Davis, G¹; Van Citters, D²; Pruitt, L¹

¹University of California, Berkeley

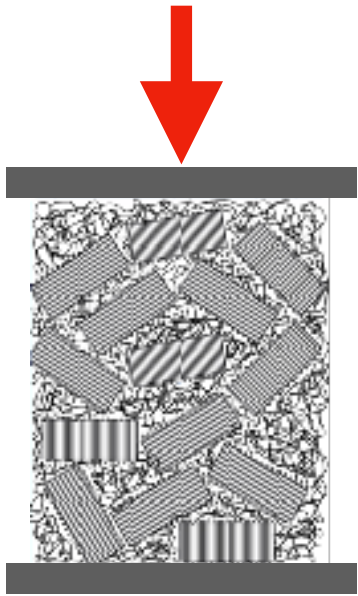
²Dartmouth University, New Hampshire, NH



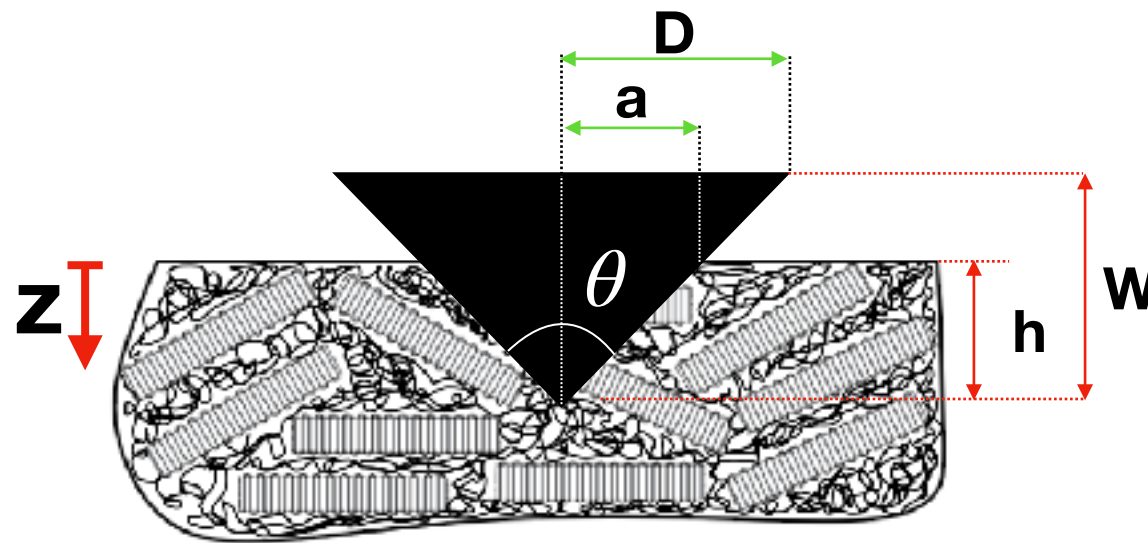
Berkeley

Significance for probing at different length scales and motivation for measuring localized mechanical properties.

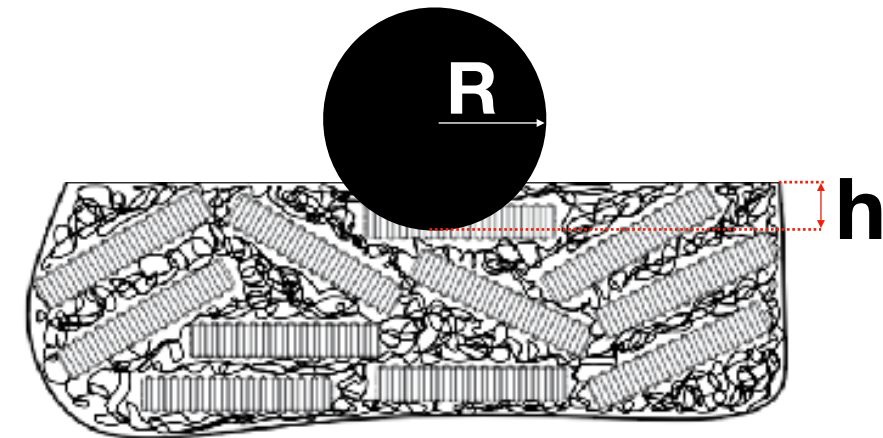
Compression



Microindentation



Nanoindentation



Edidin et al. 1999.

Plasticity-induced
Damage Layer
(Oriented Lamellae)

Wear
Surface

4-9
um

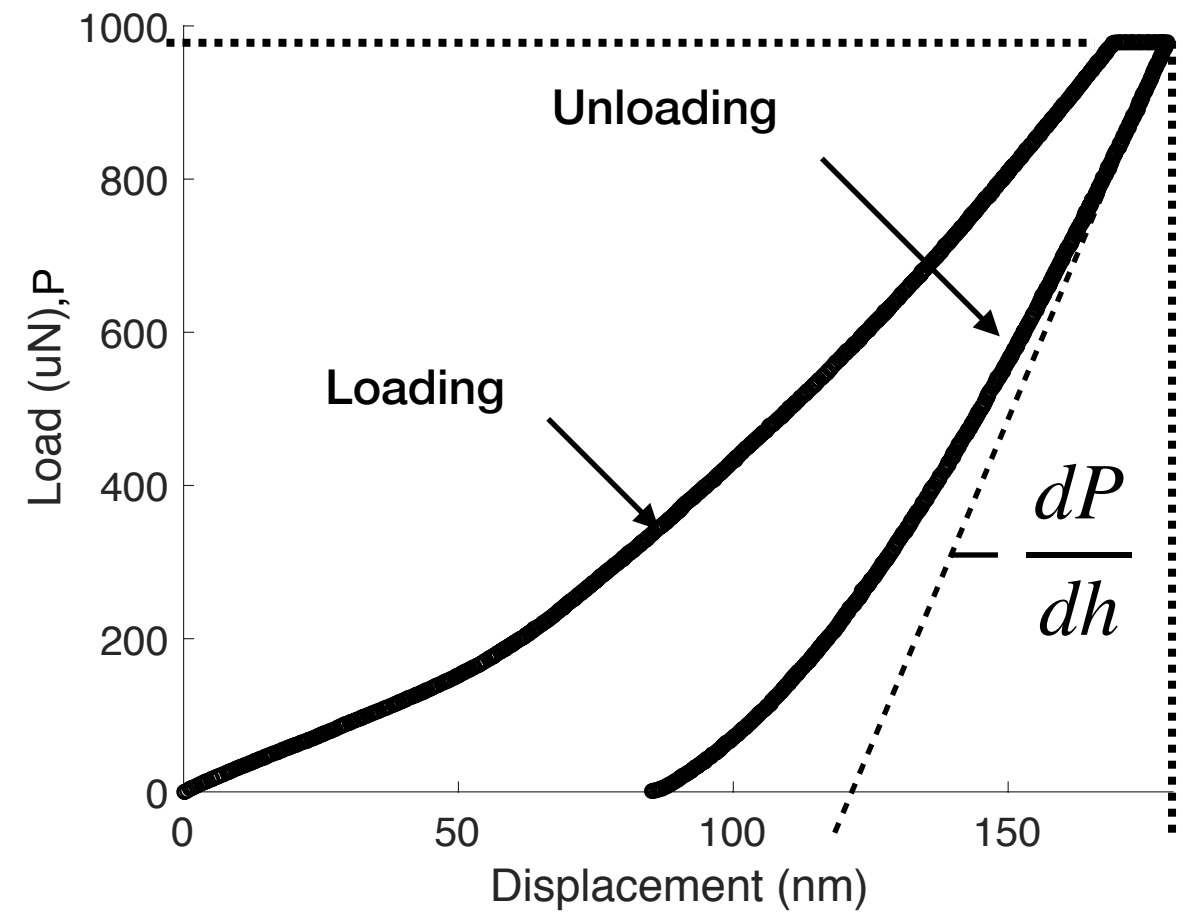
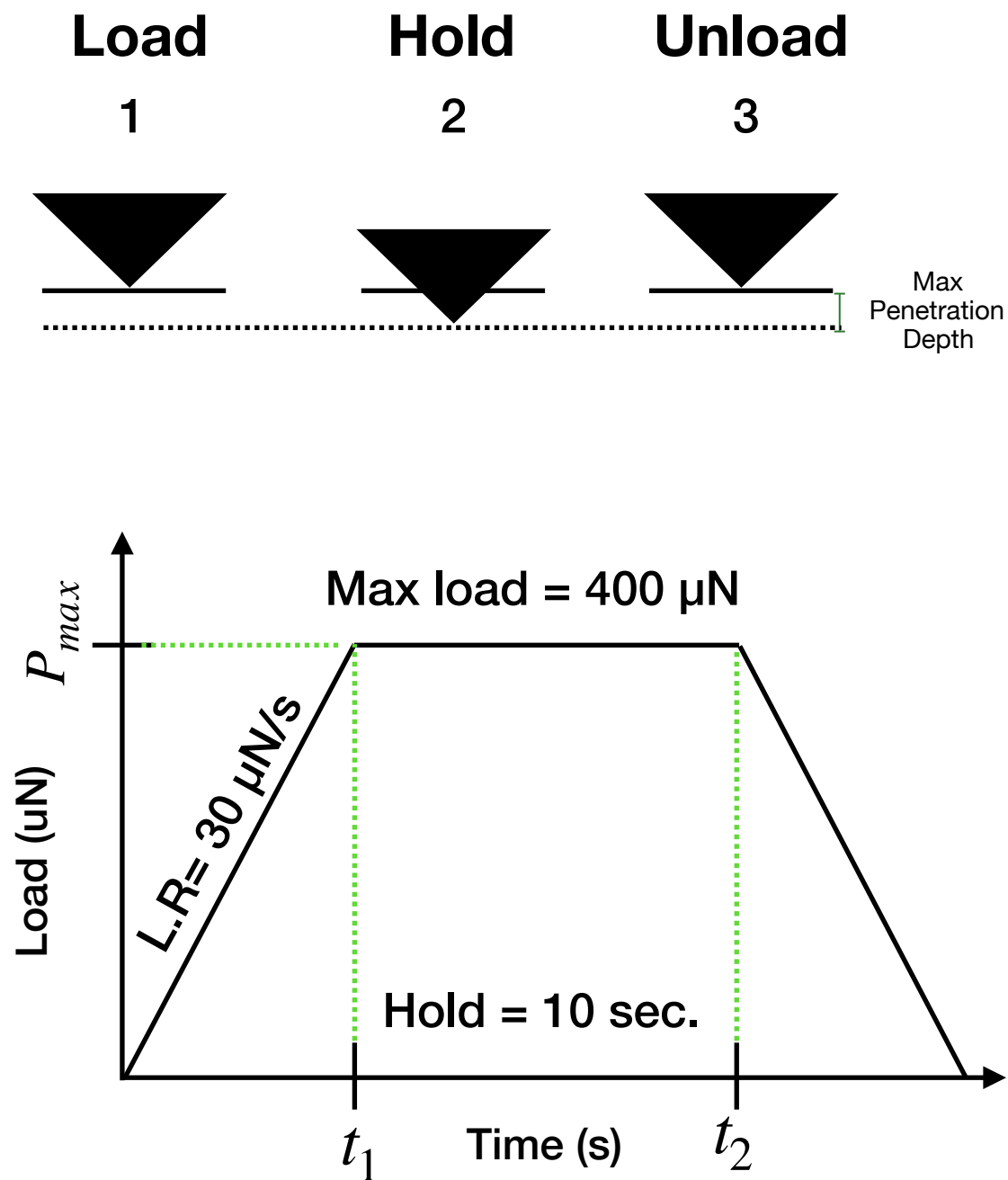
Isotropic Polymer
(Random
Lamellae)

Importance of studying the surface mechanical behavior

- Poor surface mechanical behavior may result in wear, fracture, and surface fatigue mechanisms.
- Nanomechanical testing may provide a link between surface behavior and wear mechanisms.
- The role of cross linking on the local mechanical properties at the articulating surface remains to be studied.

Klapperich et. al. 2001; Chen et. al. 1997.

Nanoindentation: Extracting the stiffness from load displacement curves



Sneddon's equation:

$$E = \frac{1 - \nu^2}{2} \frac{\sqrt{\pi}}{\sqrt{A}} \frac{dP}{dh}$$

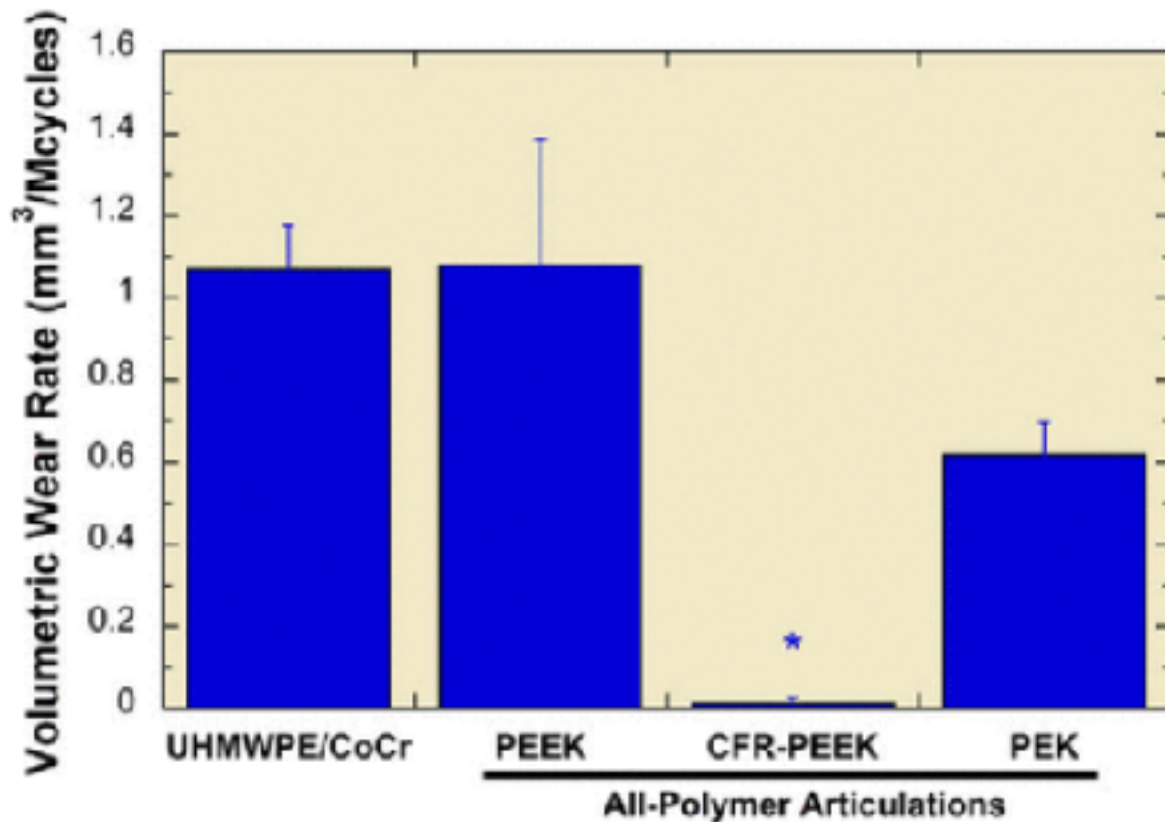
ν : Poisson's ratio
 A : Contact area

Nanoindentation as a tool for characterizing PEEK and PEEK composites

- Tip dependence on nano mechanical measurements
- Mechanical behavior of the constituents present in PEEK composites
- Structure-property relations
- Thermal treatment effects

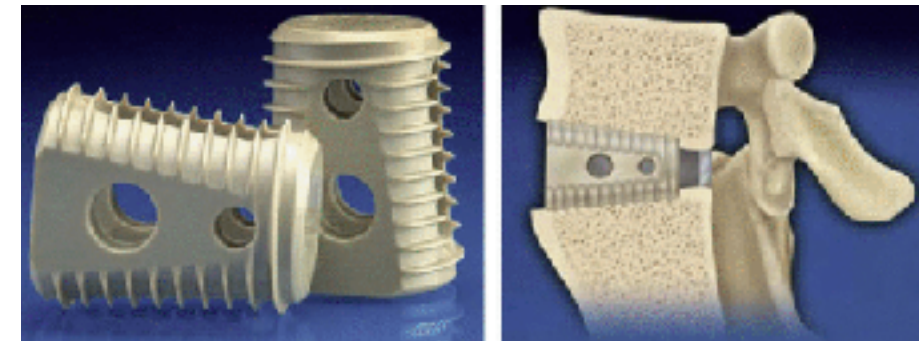
Mechanical properties and biomedical applications of PEEK

Mechanical Properties



[3] Image obtained from Kurtz et. al. 2013

Applications



Tapered PEEK cage for the lumbar spine (LT-CAGE system; medtronic Spinal and Biologics, Memphis, TN)

Benefits of PEEK in biomedical applications:

- Radiolucency and radiative stability
- Sterilization with minimal degradation to mechanical properties.

[3] Kurtz et. al. 2013

Tensile modulus of PEEK composites

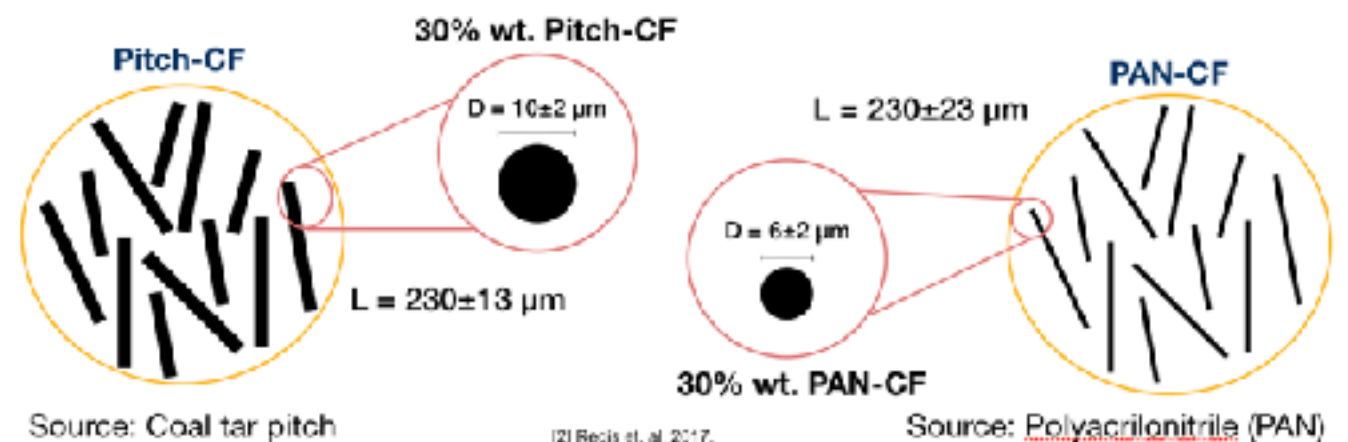
	Unfilled	Pitch	PAN
E	3.9±0.2	12.5±1.3	18.5±2.3

Carbon Fiber Modulus:

PAN: 540 GPa
Pitch: 280 GPa

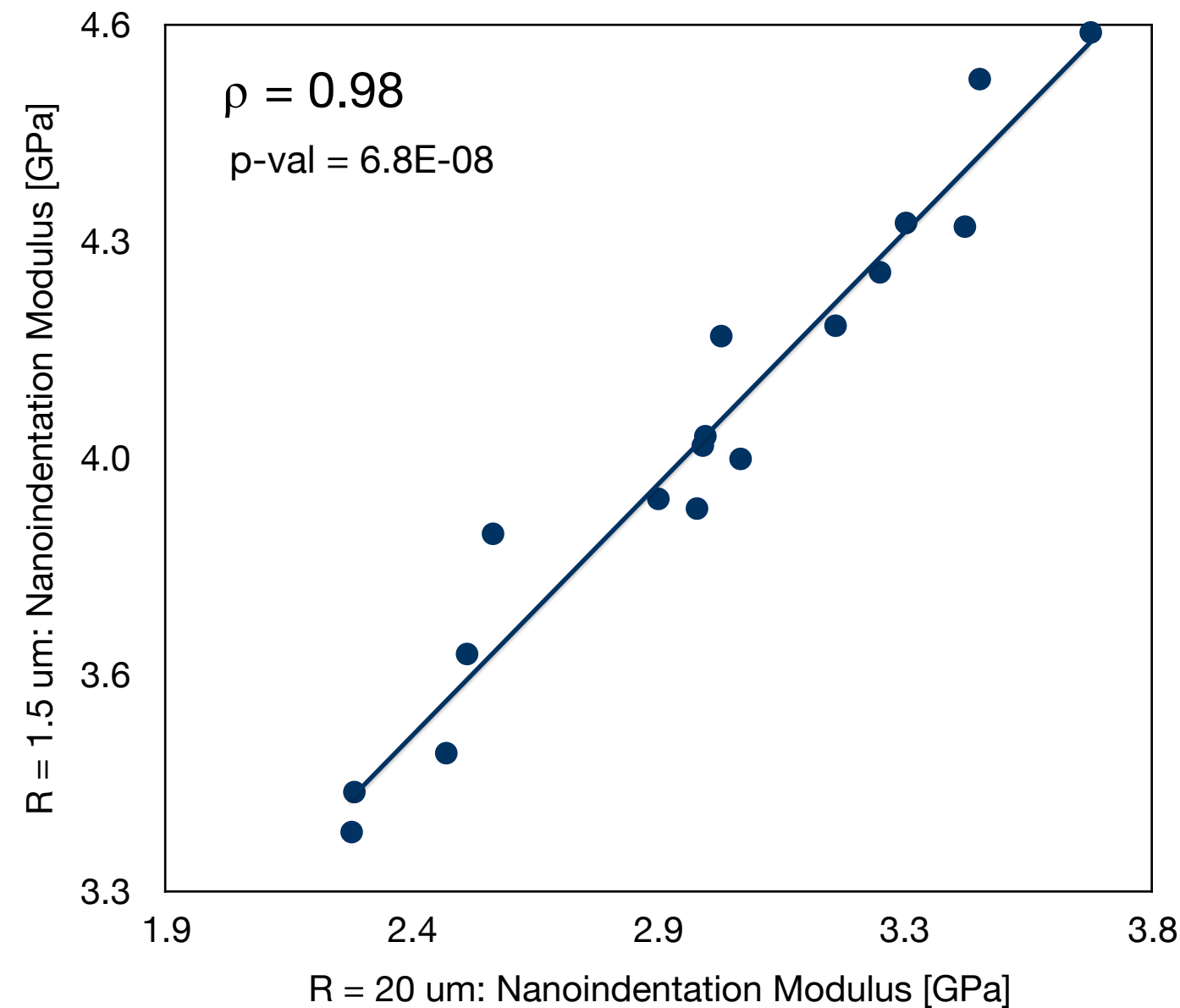
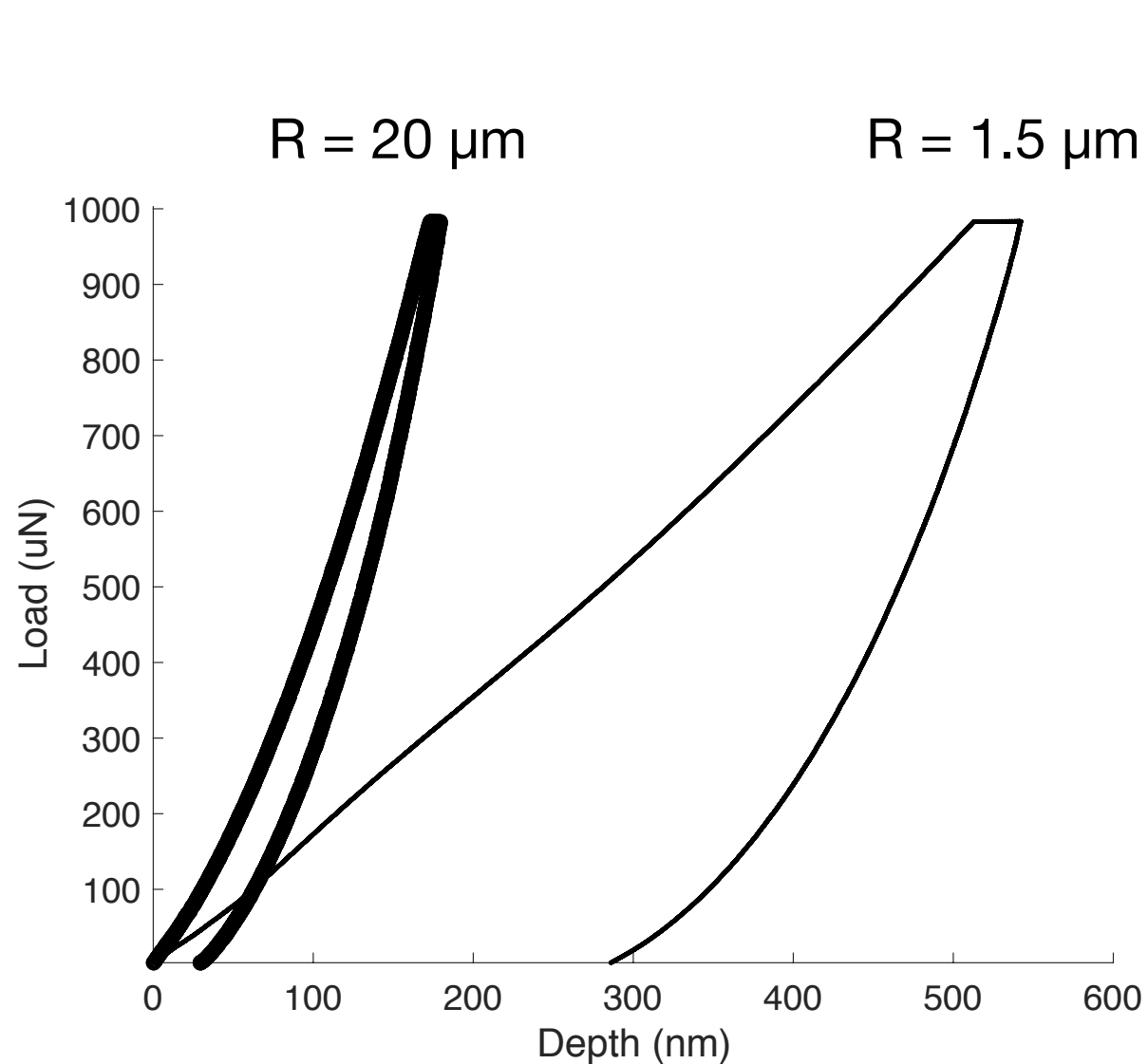
[4] Bonnheim et. al. 2018

PEEK composites



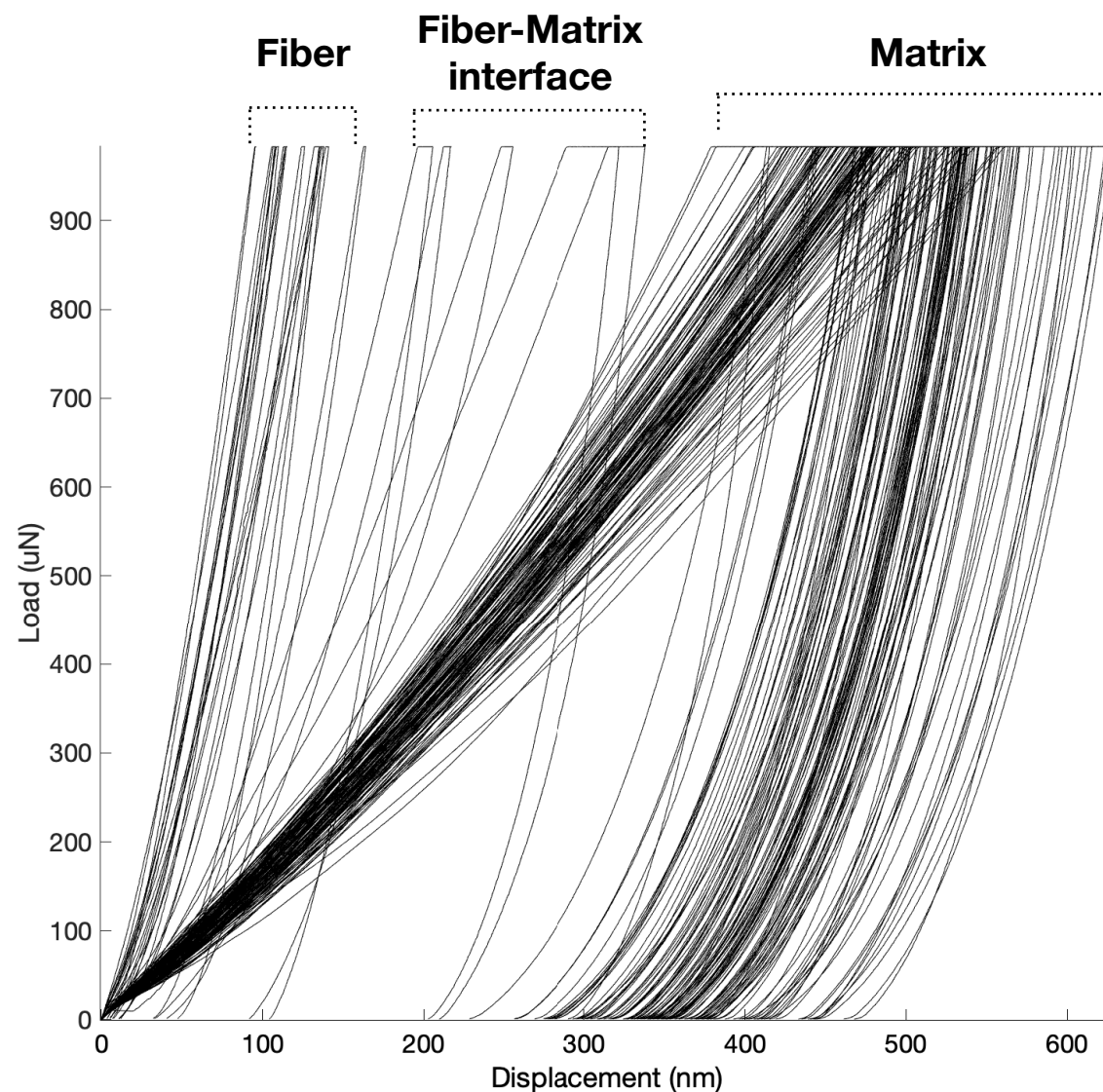
[2] Regis et. al. 2017.

Nanoindentation modulus measurement are sensitive to the tip diameter

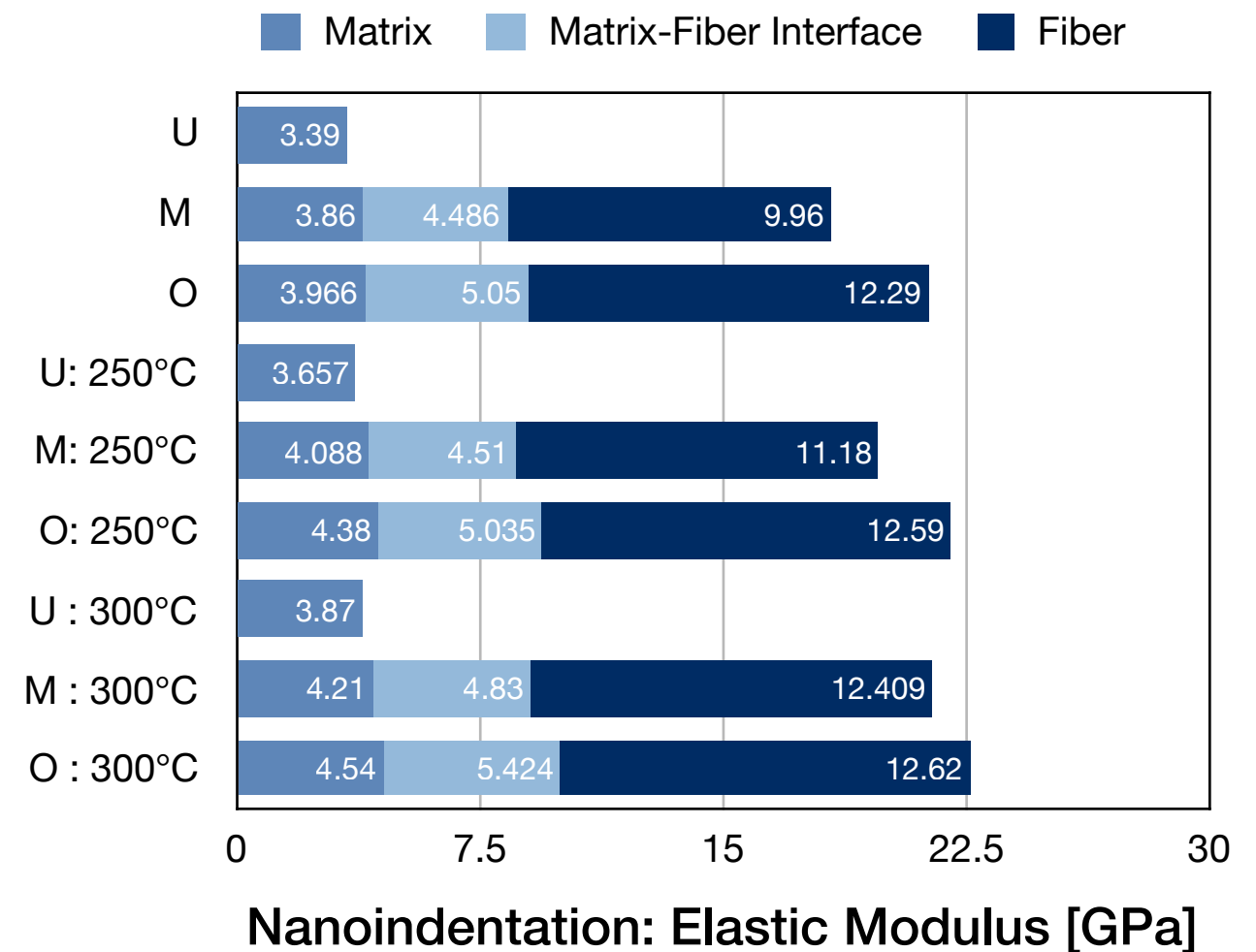


A difference in modulus with increase in tip diameter results from the changes in contact stresses beneath the indenter.

An appropriate-size tip can assist in measuring the mechanical properties of individual constituents in PEEK composites



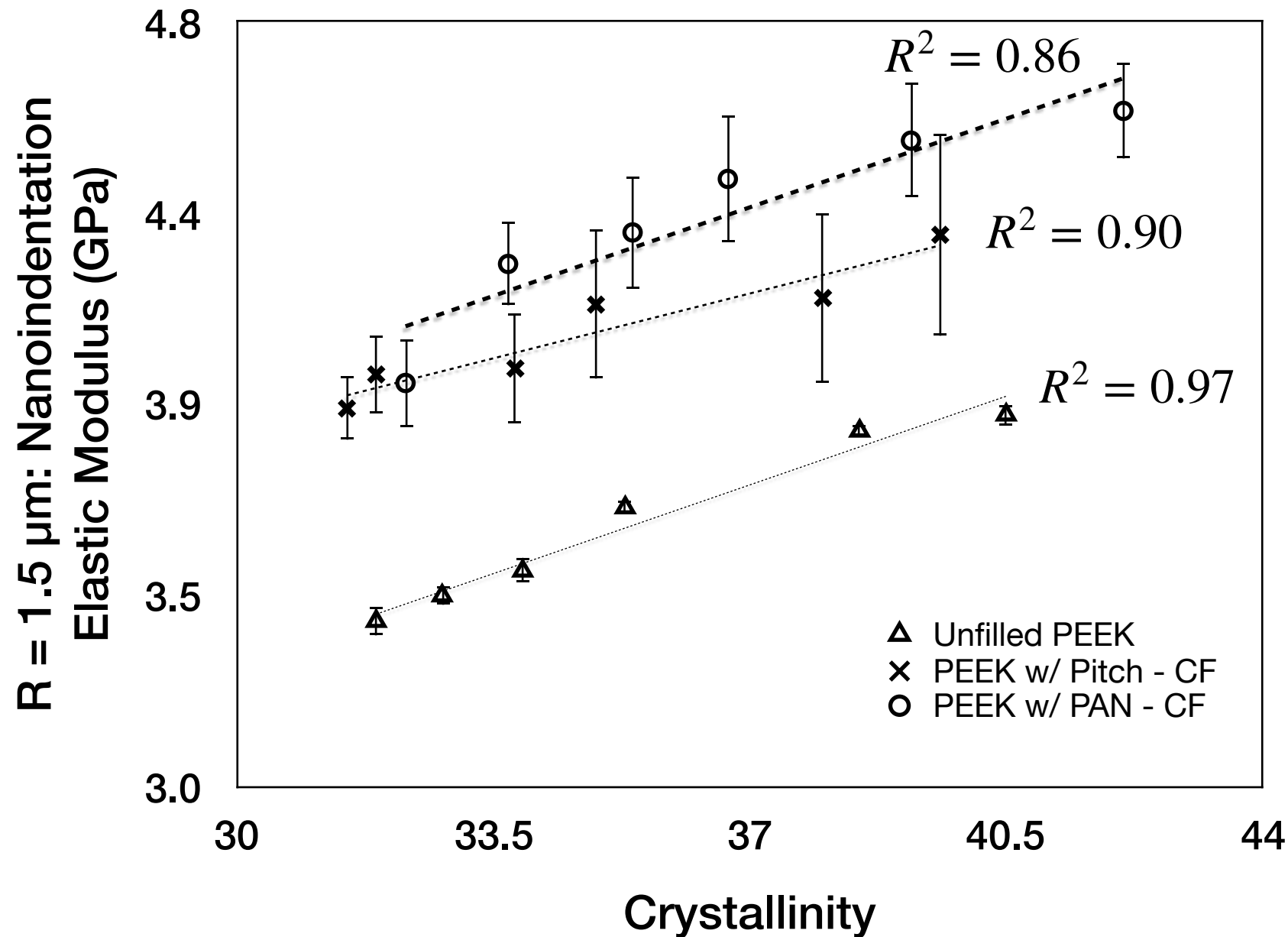
R = 1.5 μm. PEEK w/ Pitch-CF @ 300°C



Average elastic modulus for the matrix, matrix-fiber interface, fiber present in PEEK composites

- The average modulus for the fiber, fiber-matrix interface and matrix is calculated using a statistical method for clustering data.

Assessing the suitability of nano indentation to develop structure-property relation

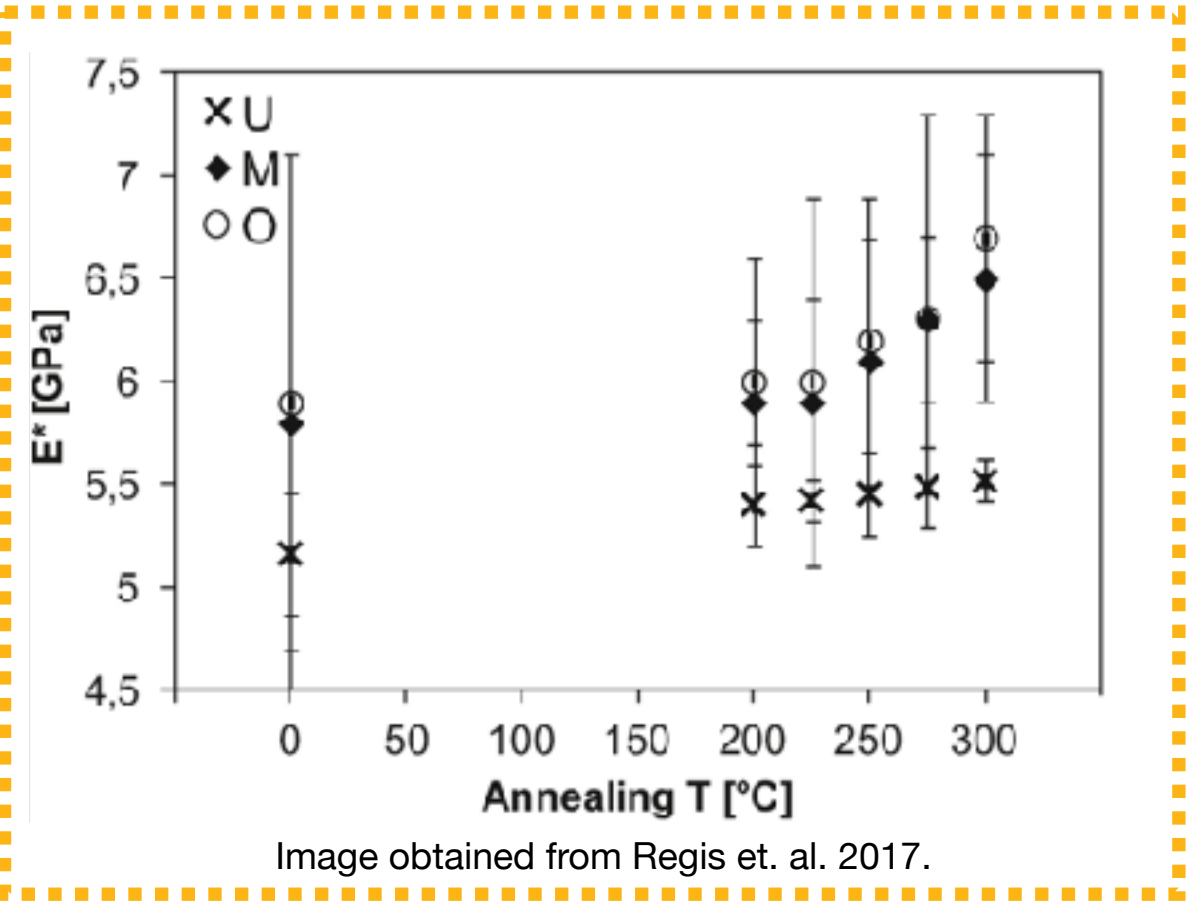
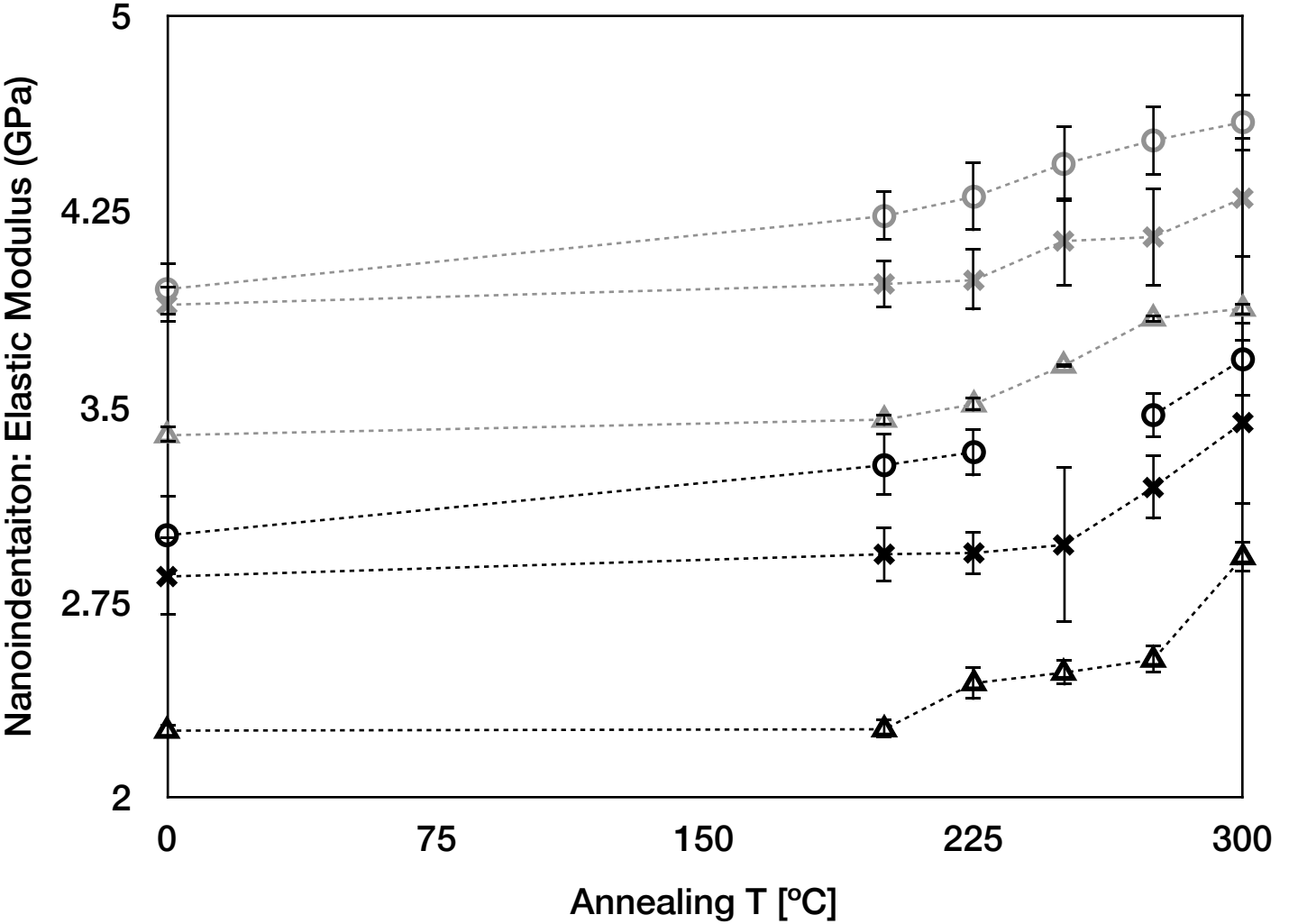


Tailoring the microstructure to achieve desired nano-mechanical properties.

Nanomechanical properties for PEEK composites increase with increase in annealing temperature

Unfilled	PAN-CF	Pitch-CF
No Heat Treatment	No Heat Treatment	No Heat Treatment
200	200	200
225	225	225
250	250	250
275	275	275
300	300	300
PEEK-OPTIMA™ LT1	PEEK-OPTIMA Reinforced™ 30% wt PAN CF	PEEK-OPTIMA Wear Performance™ 30% wt Pitch CF

○ PAN-CF: Tip R = 20 μm ✕ Pitch-CF: Tip R = 20 μm ▲ Unfilled: Tip R = 20 μm
 ▲ Unfilled: Tip R = 1.5 μm ✕ Pitch-CF: Tip R = 1.5 μm ○ PAN-CF: Tip R = 1.5 μm



Strong correlation exists between micro-indentation and nano-indentation elastic modulus

Unfilled	PAN-CF	Pitch-CF
No Heat Treatment	No Heat Treatment	No Heat Treatment
200	200	200
225	225	225
250	250	250
275	275	275
300	300	300
PEEK-OPTIMA™ LT1	PEEK-OPTIMA Reinforced™ 30% wt PAN CF	PEEK-OPTIMA Wear Performance™ 30% wt Pitch CF

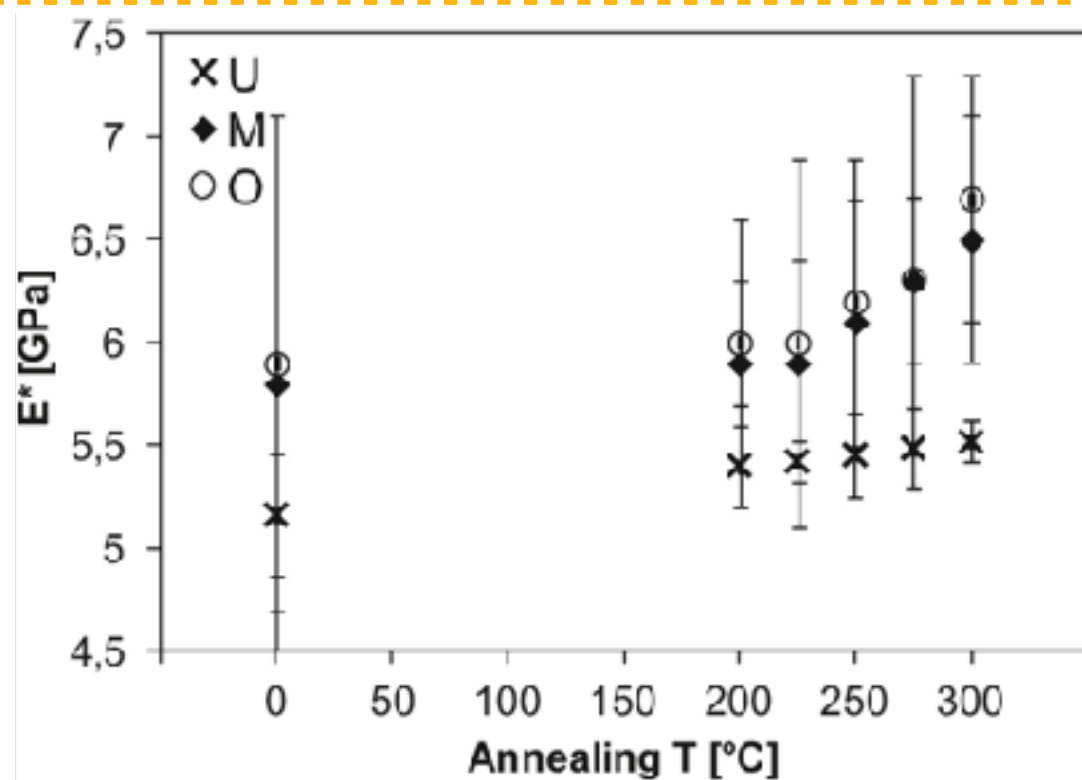
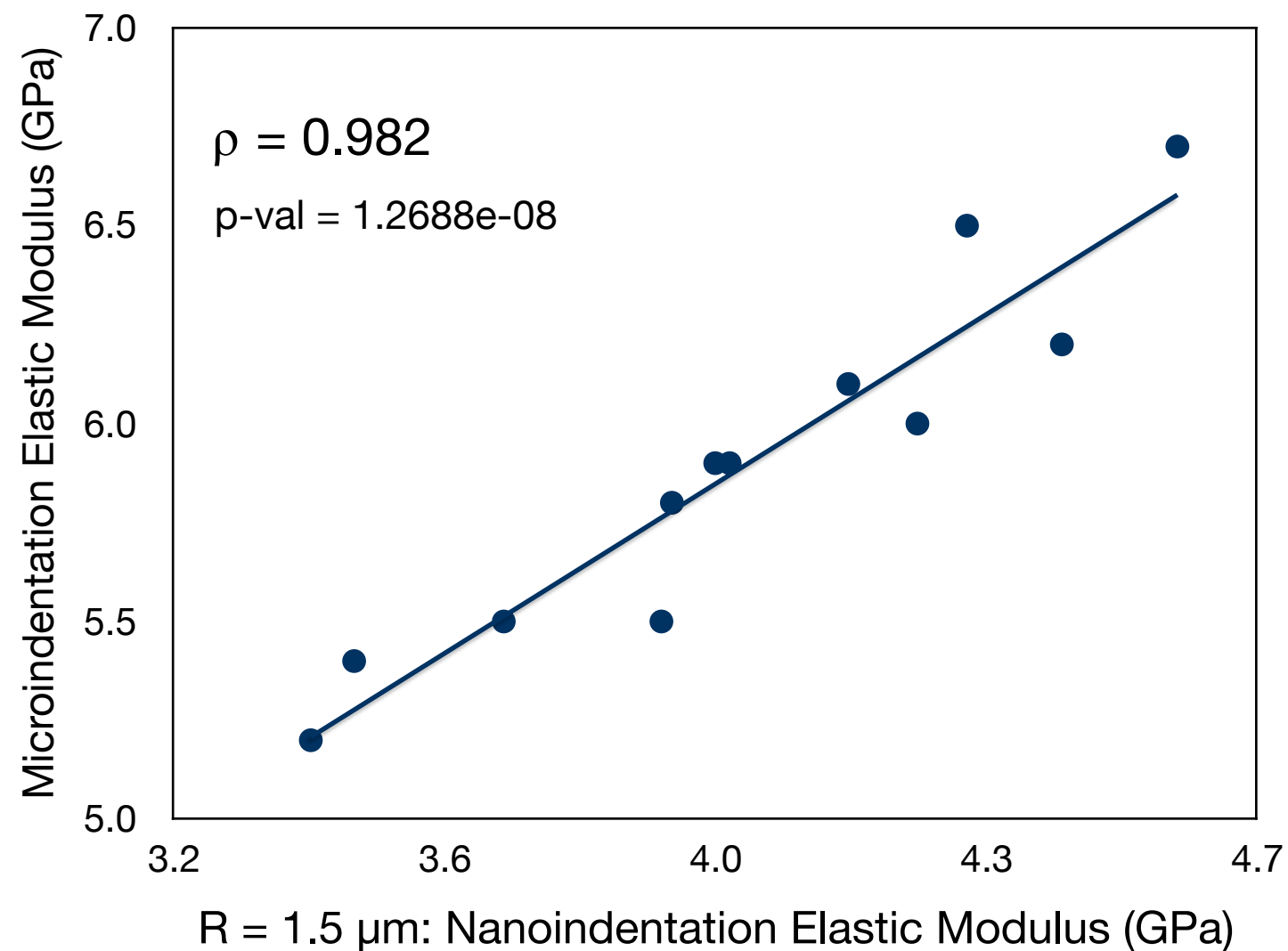
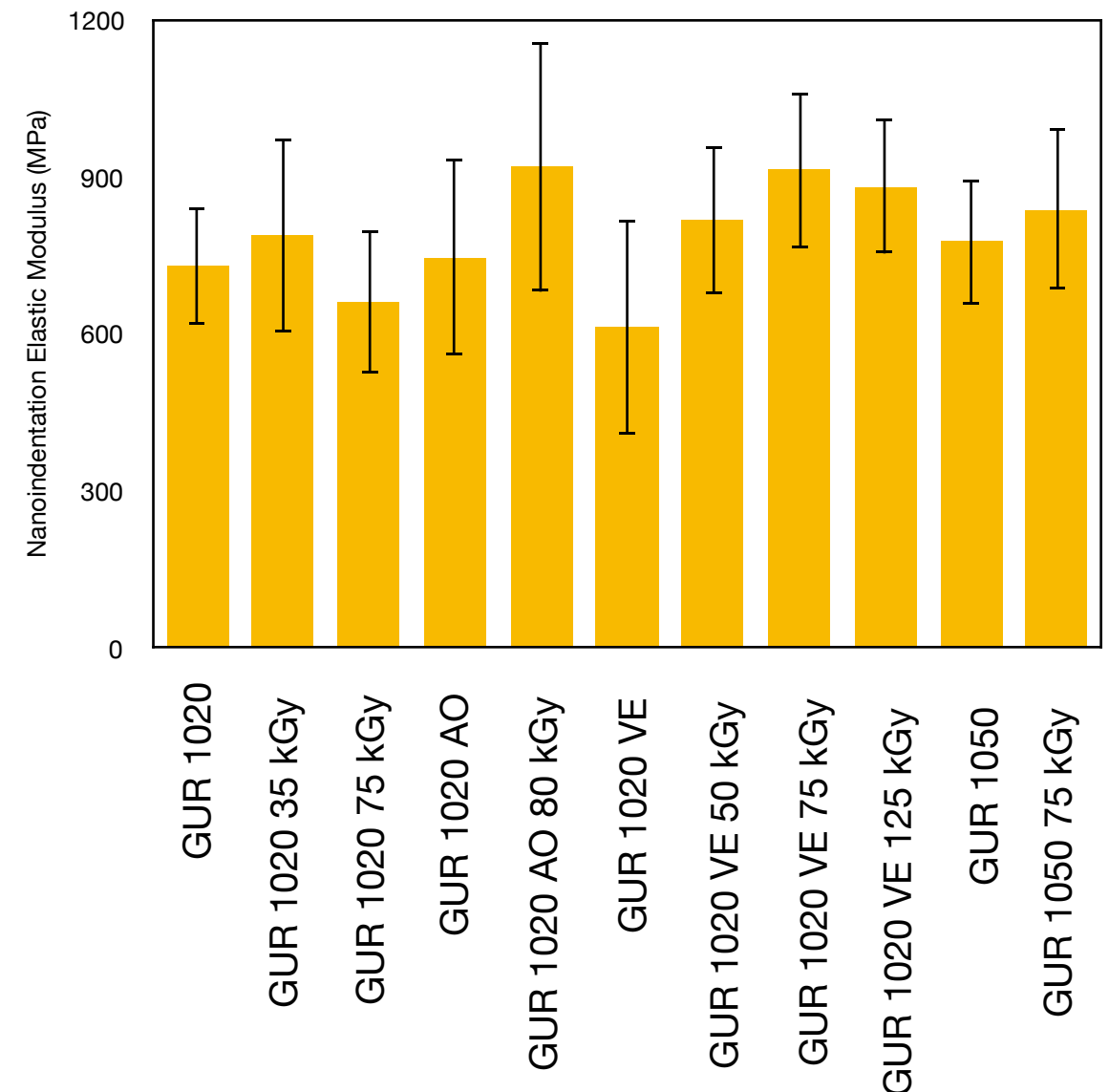


Image obtained from Regis et. al. 2017.



Nanomechanical characterization of UHMWPE

UHMWPE Material Formulation and Manufacturer			
GUR 1020 (Orthoplastics)	GUR 1020 AO (Depuy)	GUR 1020 VE (Orthoplastics)	GUR 1050 (Orthoplastics)
GUR 1020 35kGy (Orthoplastics)	GUR 1020 AO 80kGy (Depuy)	GUR 1020 VE 50kGy (Orthoplastics)	GUR 1050 75kGy RM (Quadrant)
GUR 1020 75kGy RM (Orthoplastics)		GUR 1020 VE 75kGy (Orthoplastics)	
		GUR 1020 VE 100kGy (Orthoplastics)	
		GUR 1020 VE 125kGy (Orthoplastics)	



- A correlation strength of 0.58 is noted between nano indentation elastic modulus and compressive elastic modulus

Malito et. al. 2018

**Can nano indentation techniques
be used to characterize retrievals?**

**What new information can we learn from
a nanomechanical analysis of retrievals?**

An overview of micro-length scale testing of retrievals

The relationship between the clinical performance and large deformation mechanical behavior of retrieved UHMWPE tibial inserts. [Kurtz et al. 2000]

- clinical performance of UHMWPE tibial inserts is related to the large-deformation mechanical behavior measured near the articulating surface.

Micromechanics of shelf-aged and retrieved UHMWPE tibial inserts: indentation testing, oxidative profiling, and thickness effects [Wernle et al. 2005]

- Observed strong correlation between oxidation index and mechanical properties

On the assessment of oxidative and microstructure changes after in vivo degradation of historical UHMWPE knee components by means of vibrational spectroscopies and nano indentation [Medel et al. 2008]

- Detecting regional differences in physical, chemical and mechanical properties of tibial inserts produced by in vivo oxidation. Methods used: microindentation, FTIR, Raman Spectroscopies

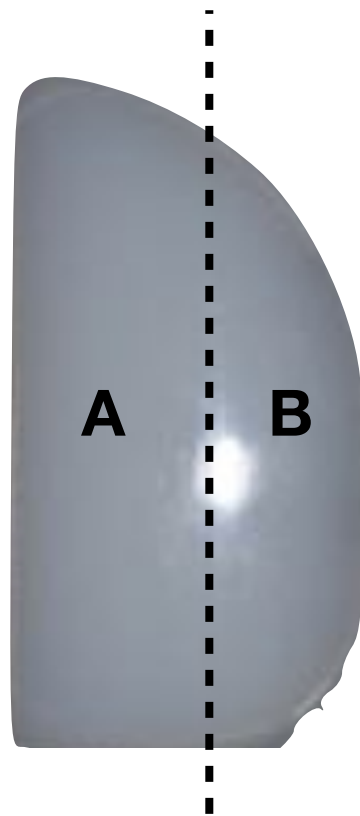
Plasticity induced damage layer is a precursor to wear in radiation-cross-linked UHMWPE acetabular components for total hip replacement [Edidin et al. 1999]

- Effect of cross linking on the tribologic, mechanical and morphologic properties of UHMWPE.
- Relate properties to wear mechanisms in acetabular bearing inserts.
- Using small punch testing for mechanical testing

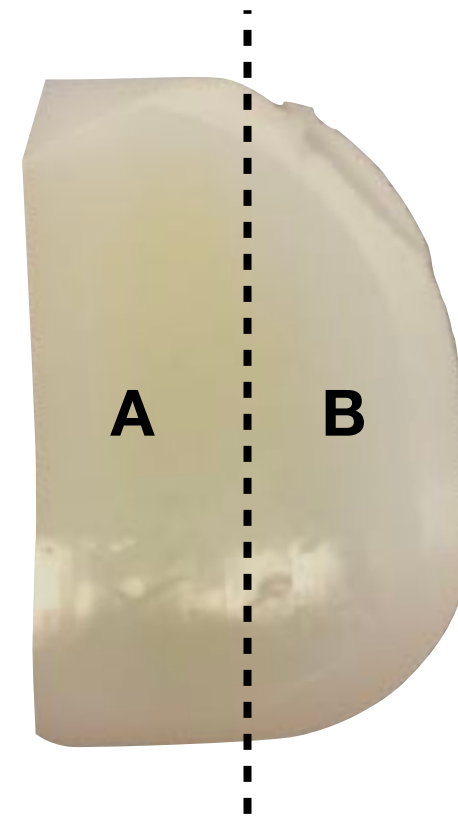
Nano-mechanical characterization of two Prolong® Highly Crosslinked Tibial Inserts

Developing a modulus map of the articulating surface of tibial inserts to:
establish nano-indentation as a suitable surface characterization
technique and to better understand and quantify the changes in
mechanical properties at the articulating surface.

Short-term implantation

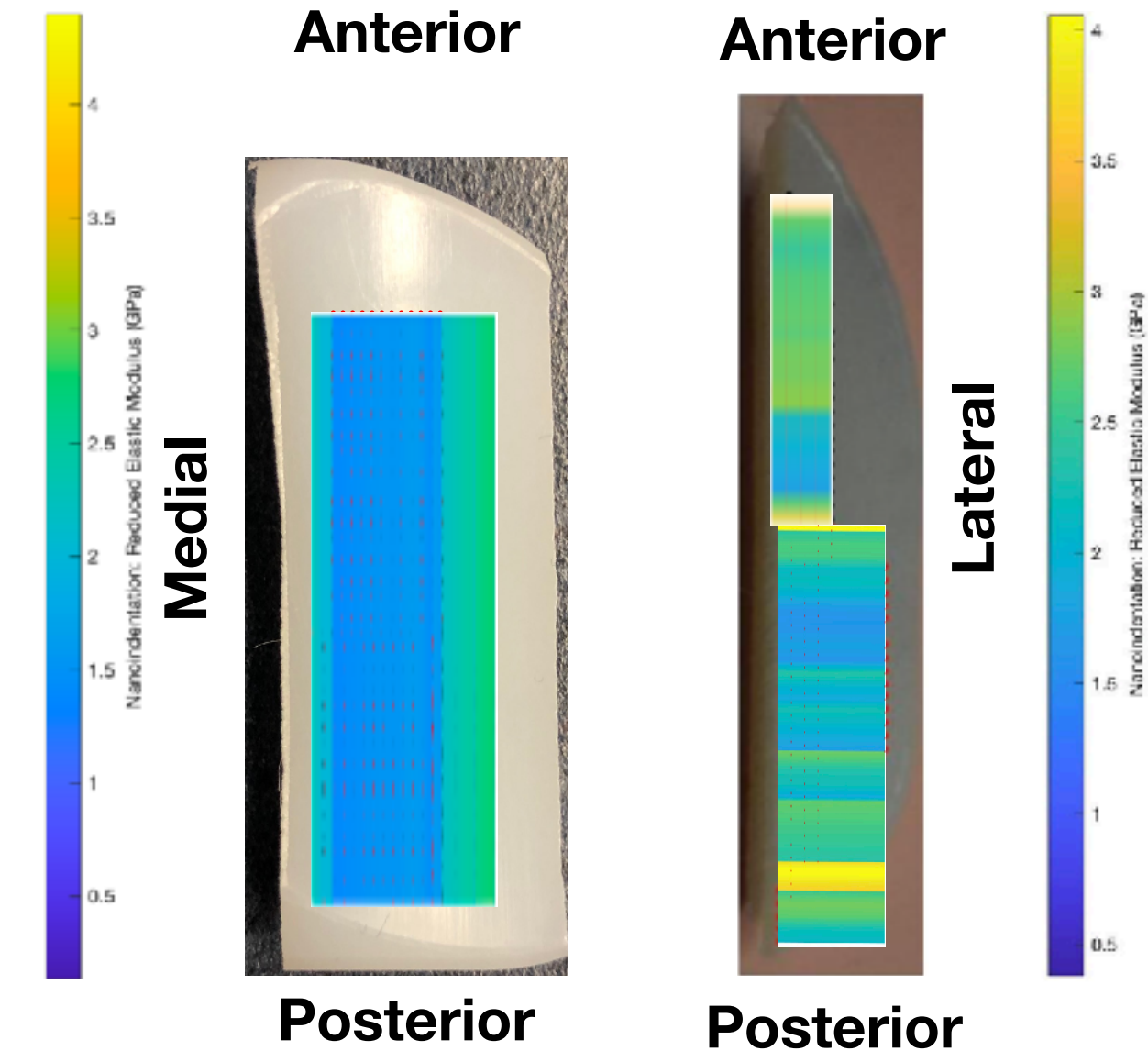


Long-term implantation



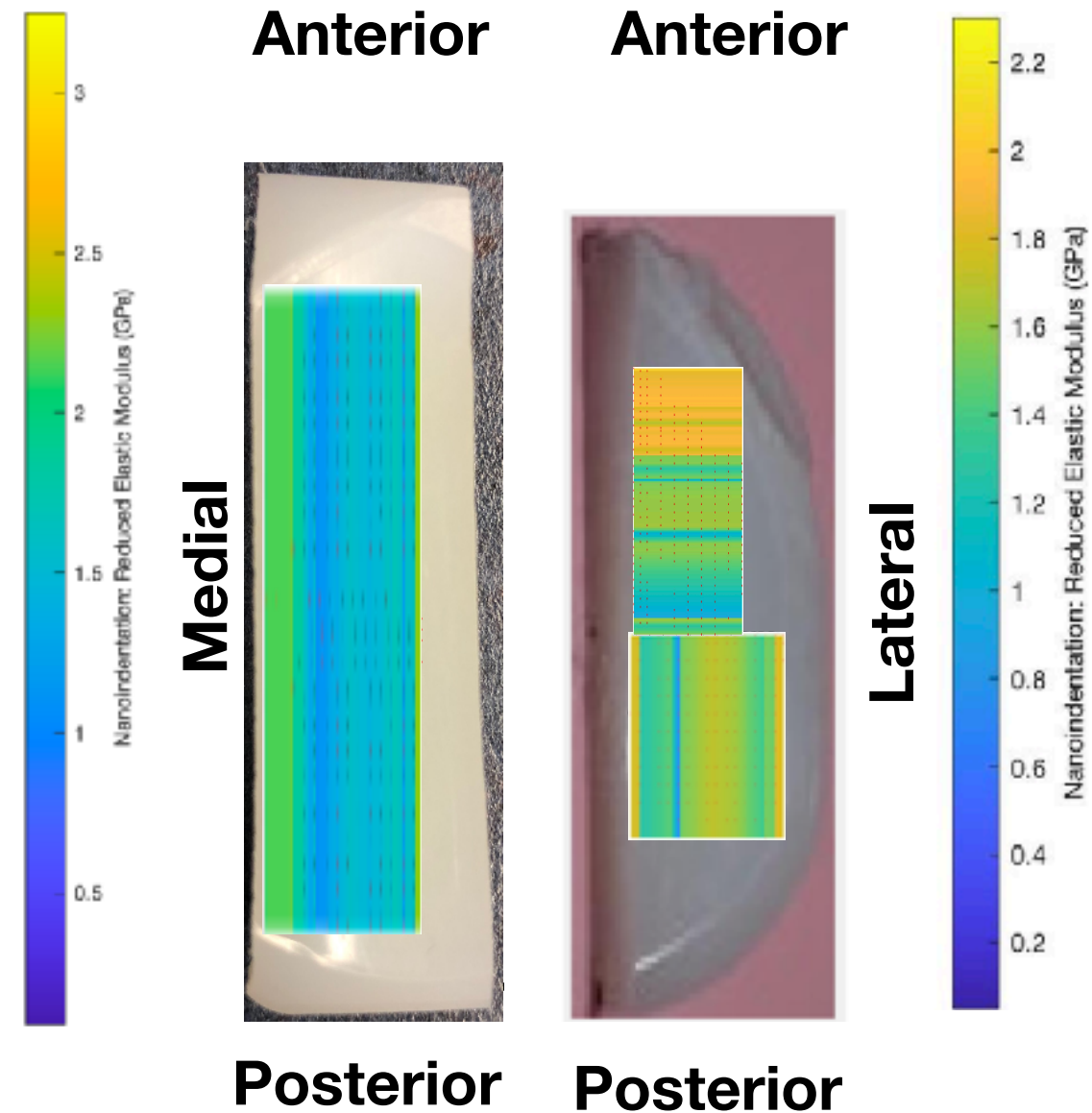
Preliminary results: Assessing the surface properties of tibial inserts

Short-term implantation



Er: 0.84 ± 0.52 GPa Er: 1.15 ± 0.66 GPa

Long-term implantation



Er: 0.77 ± 0.48 GPa Er: 0.59 ± 0.41 GPa

Based on preliminary observations, a strain softening effect is occurring the longer it stays in the body

Summary and Conclusions

- Strong correlations between nano indentation modulus and microstructural properties provide evidence on the utility of nano indentation methods for developing structure-property relations.
- A smaller indentation tip is able to better capture the modulus of the individual component (fiber and the matrix); whereas, larger diameter tips indent over an expanded area containing a mixture of fibers and matrix.
- Finding the effects of indenting at an angle on the mechanical properties.
- Validate and study the effect of tip geometry and size on the measured values.

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