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Structural, Nanomechanical, and Nanotribological Characterization of Human Hair and Conditioner using AFM and Nanoindentation

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Nanoprobe Laboratory for Bio- & Nanotechnology and Biomimetics



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Outline

- Introduction
 - Hair structure
 - Why is tribology important to desired hair features?
 - Role of shampoos and conditioners
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 - Nanoindentation technique
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- Hair Cellular Structure Characterization
- Nanomechanical Property Characterization
- Nanotribological Characterization
- Conditioner Distribution Studies
- Surface Potential Measurements Using Kelvin Probe Microscopy (KPM)

Bharat Bhushan

BIOLOGICAL AND MEDICAL PHYSICS, BIOMEDICAL ENGINEERING

Biophysics of Human Hair

Structural, Nanomechanical,
and Nanotribological Studies

 Springer

B. Bhushan, The Ohio State University, Columbus, OH, USA

Biophysics of Human Hair

Structural, Nanomechanical, and Nanotribological Studies

This book presents the biophysics of hair. It deals with the structure of hair, its mechanical properties, the nanomechanical characterization, tensile deformation, tribological characterization, the thickness distribution and binding interactions on hair surface. It is the first book on the biophysical properties of hair... *more on <http://springer.com/978-3-642-15900-8>*

- Presents the biophysics of human hair Contains nanostudies of the properties of hair
- Gives a macroscale tribological characterization of hair Discusses hair damage

2010. 295 p. 100 illus. (Biological and Medical Physics, Biomedical Engineering) Hardcover

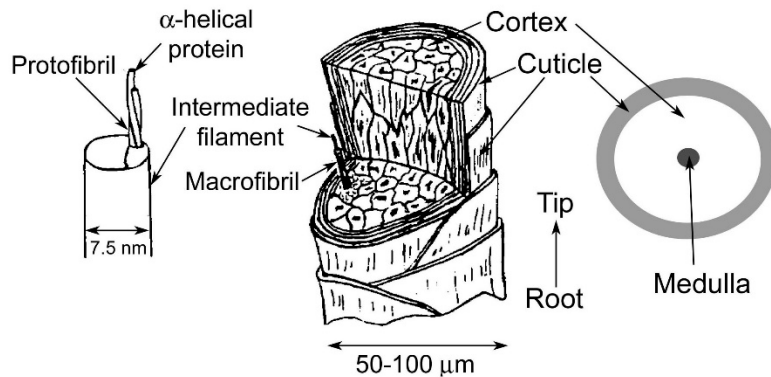
ISBN 978-3-642-15900-8

Introduction

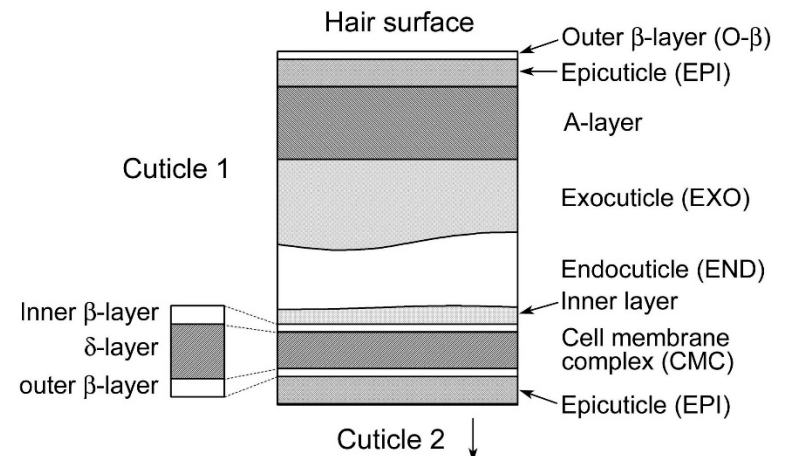
- Hair is nanocomposite biological fiber
- It is composed of dead cells, mostly keratin proteins (cystine) (65-95%), water, lipids, pigments.

Hair structure

Schematic of a human hair fiber structure with cuticle sublamellar structure

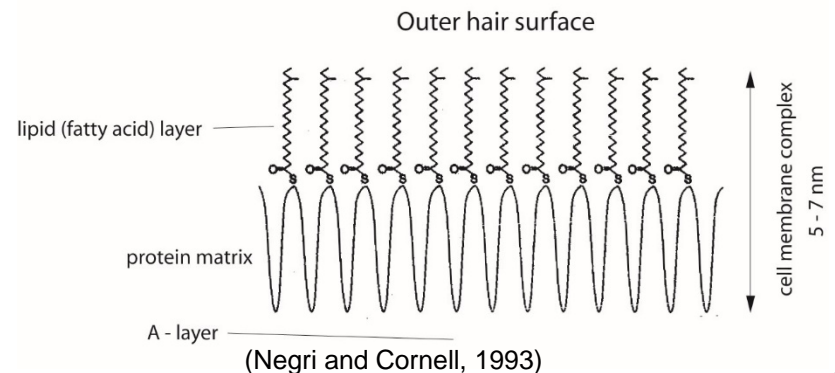


(Robbins, 1994; Smith and Swift, 2002)



Hair fiber consists of three main components:
Cuticle, cortex, and medulla

- Cuticle is important in protection of hair.
- Cuticle consists of flat overlapping scales, and appears like tiles on a roof.
- Cuticle is multi-layered. The outermost epicuticle layer is covered with lipid layer (18-MEA) which provides lubrication.

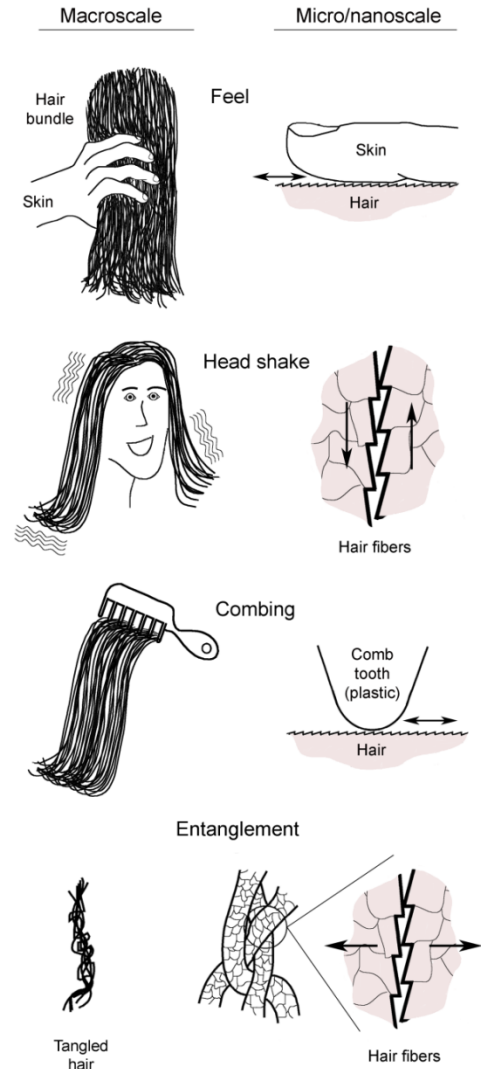


B. Bhushan, *Biophysics of Human Hair: Structural, Nanomechanical and Nanotribological Studies*, Springer, 2010.

Why is tribology important to desired hair features?

Desired hair feature	Tribological attributes
Smooth feel in wet and dry environments	Low friction between hair and skin in respective environment
Shaking and bouncing during daily activities	Low friction between hair fibers and groups of hair
Easy combing and styling	Low friction between hair and comb (plastic) and low adhesion. Combing results in physical damage such as scratching and hair stretching. Note: More complex styles may require higher adhesion between fibers.

Various functions requiring low friction and adhesion



- Maintaining health, feel, shine, color, softness, and overall esthetics is highly desired.
- Natural weathering and normal grooming actions such as combing, cutting, blow drying with hot air, chemical dyeing, and permanent wave treatments result into
chemical, mechanical and chemomechanical damage
which degrades mechanical and tribological properties.

Role of Shampoos and Conditioners

- Hair care products including shampoos and conditioners are used to clean and repair.

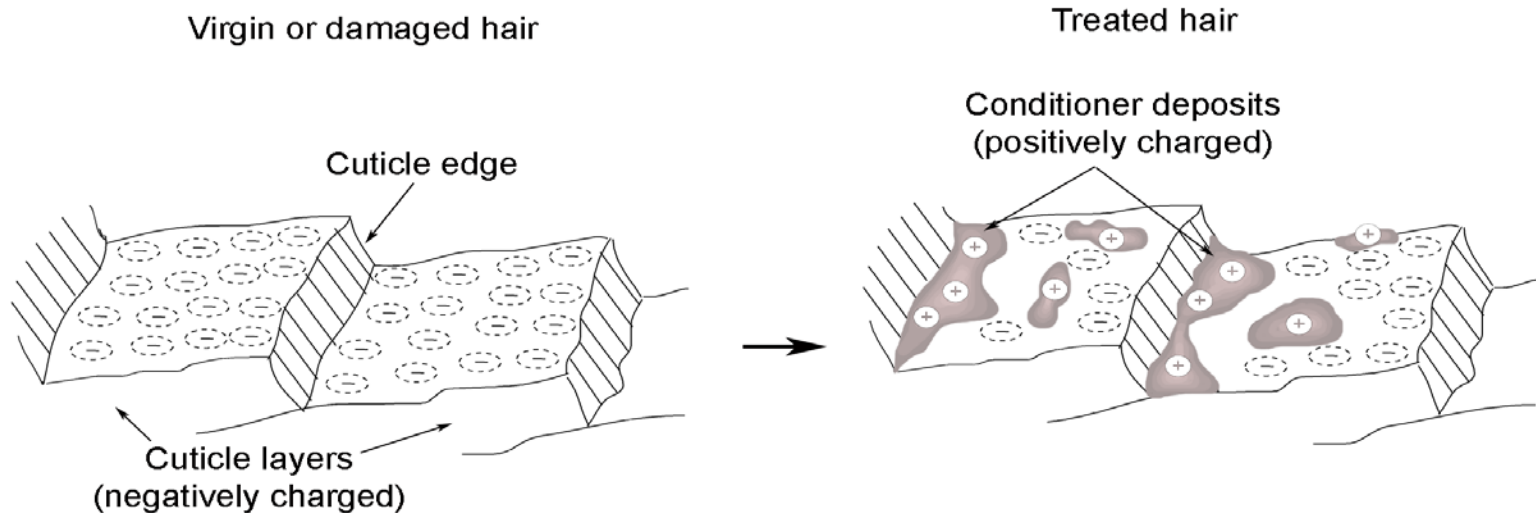
Shampoo – Cleans hair and scalp of oils and dirt.

Conditioner (gel network of cationic surfactants, fatty alcohols, silicones, and water)

– Repairs hair damage and make the hair easier to comb, prevent flyaway, add feel, shine and softness.

- This industry is about \$20 billion dollars in annual sales. Top three hair companies (P & G, L'Oréal and Unilever) all have annual sales on the order of \$5 billion each.

Interaction of conditioner with hair surface



Hair is negatively charged.

Therefore, positively charged conditioner molecules (cationic surfactants) are attracted to the hair surface which results in conditioner deposition on the hair.

It is even more true for damaged hair, since hair gets even more negatively charged as a result of damaging processes.

Objective and Approach

Objective

- In order to develop better cosmetic products,
Objective of our study is to understand the effect of damage and treatments of hair on morphology, cellular structure, mechanical and tribological properties.
- Study various hair and skin as a function of ethnicity, damage, conditioning treatment, and various environments.

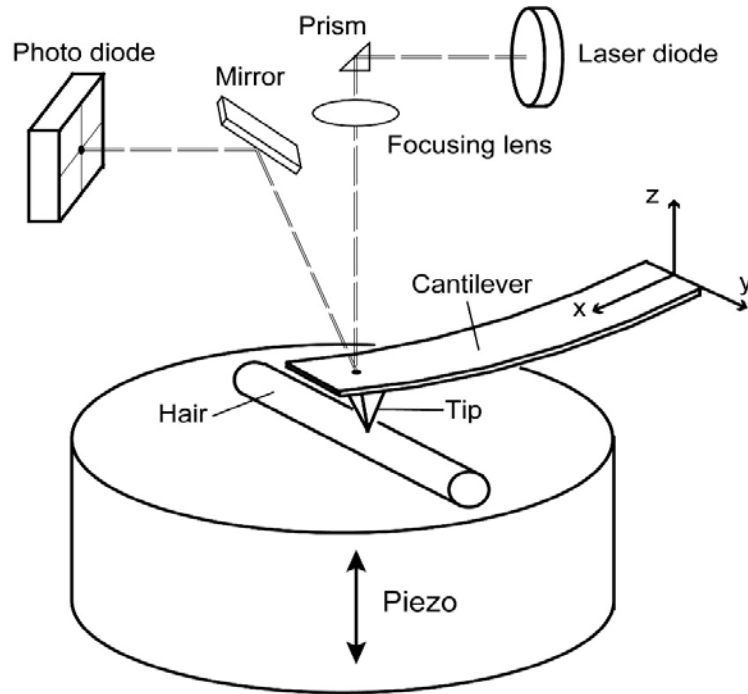
Approach

- Hair cellular structure characterization using SEM and AFM
 - Various ethnic hair including effect of location
 - Cross-section and longitudinal sections of human hair
- Nanotribological characterization using AFM
 - Surface roughness, friction, and adhesion
 - Directionality effects and scale effects on friction and adhesion
- Nanomechanical property characterization using nanoindenter and AFM
 - Hardness, elastic modulus, creep, scratch resistance
 - In-situ surface characterization of localized deformation of hair
- Macrotribological Characterization using tribometer
- Conditioner distribution studies using AFM
 - TR mode, force calibration mode, and adhesive force mapping
- Surface potential studies using KPM
 - Electrostatic charge build-up on hair surface

Experimental Techniques

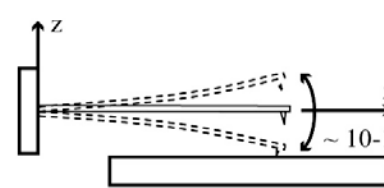
AFM and various operating modes

Schematic diagram of AFM

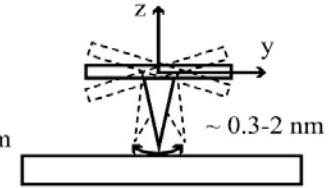


Three different AFM operating modes

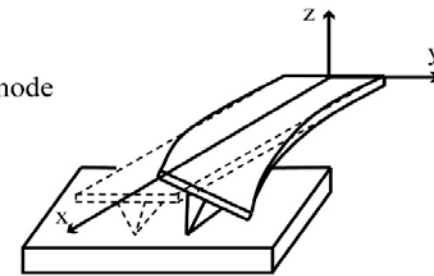
Tapping mode



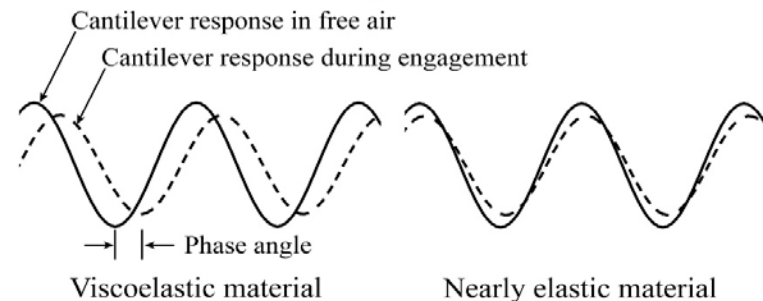
Torsional resonance mode



Contact mode



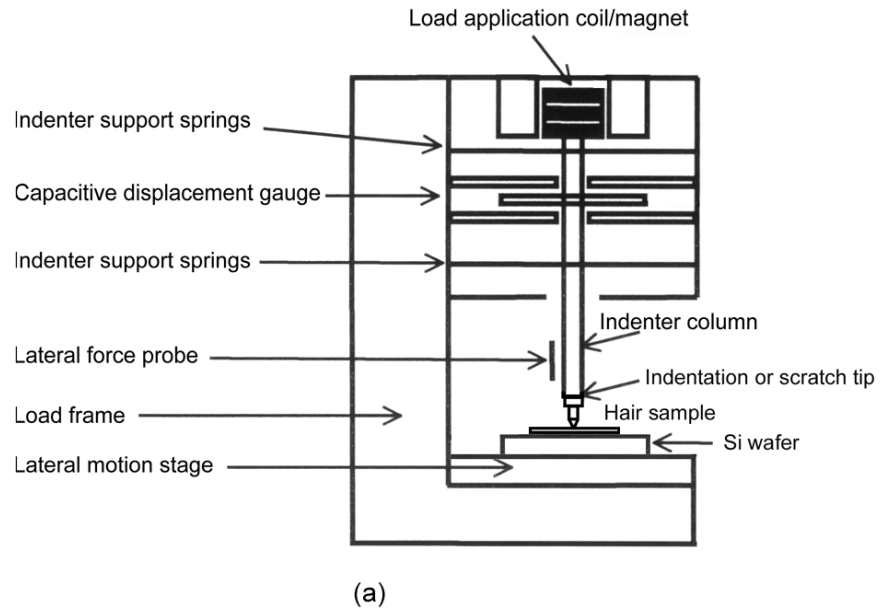
Phase angle definition



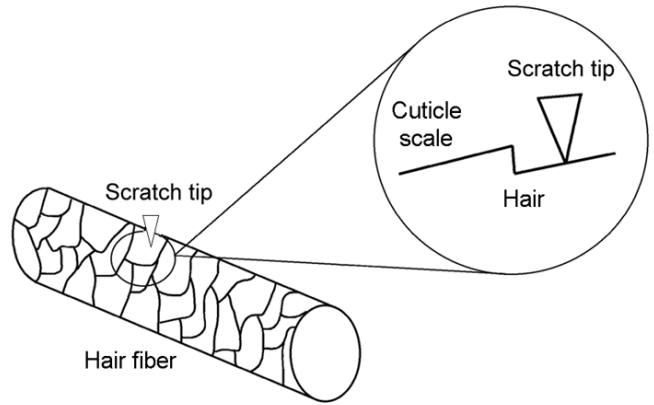
- *Roughness* measurements were made using the Tapping mode (constant amplitude).
- *Adhesion, friction and durability* studies were carried out in the Contact mode.
- *Elastic and viscoelastic properties* were measured using the Torsional resonance (TR) mode (constant load)

Nanoindenter

Schematic of nanoindentation and nanoscratch test on hair



(a)



(b)

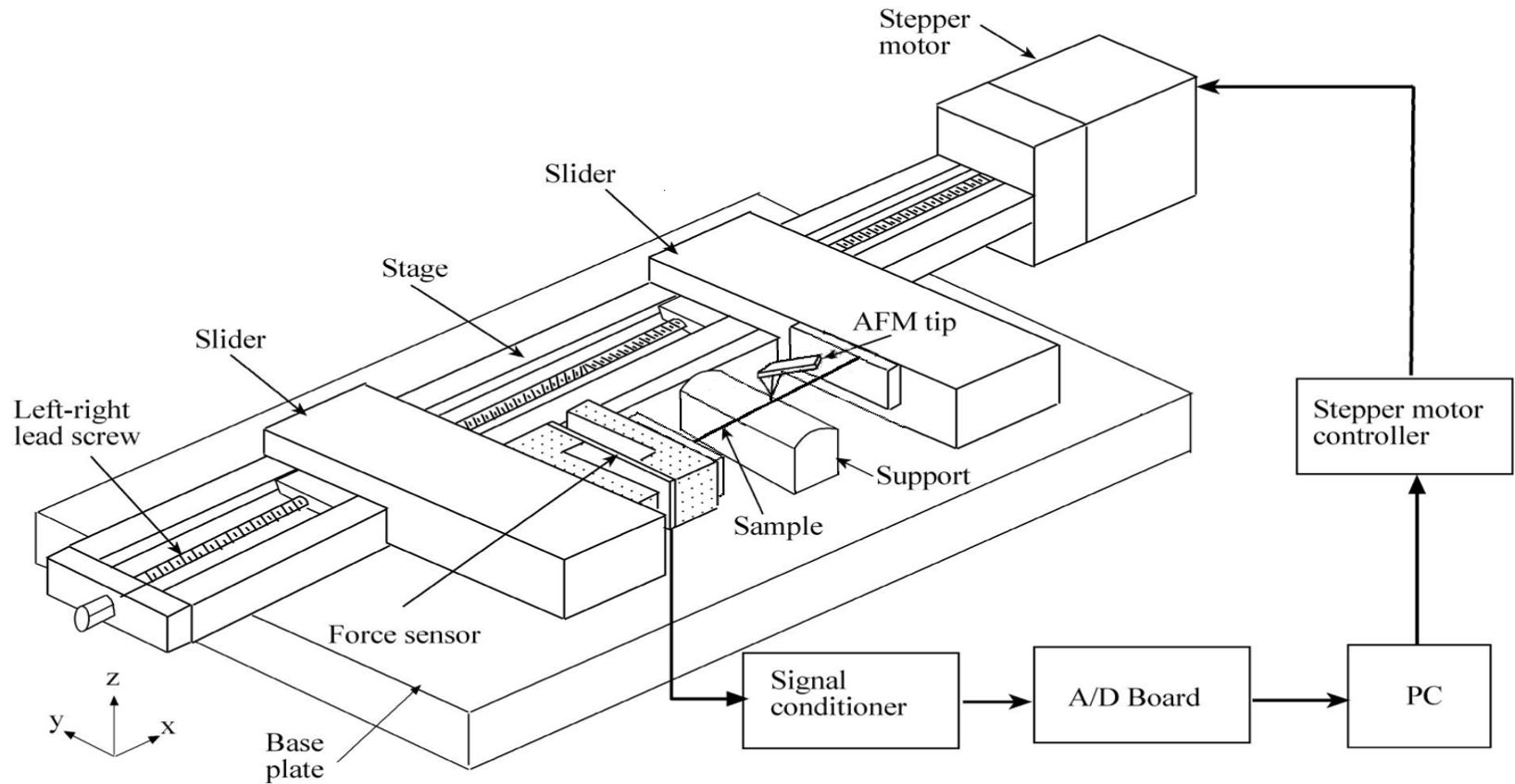
- Nanohardness, elastic modulus, creep, and scratch resistance measurements were made using a Nanoindenter II.

SEM imaging

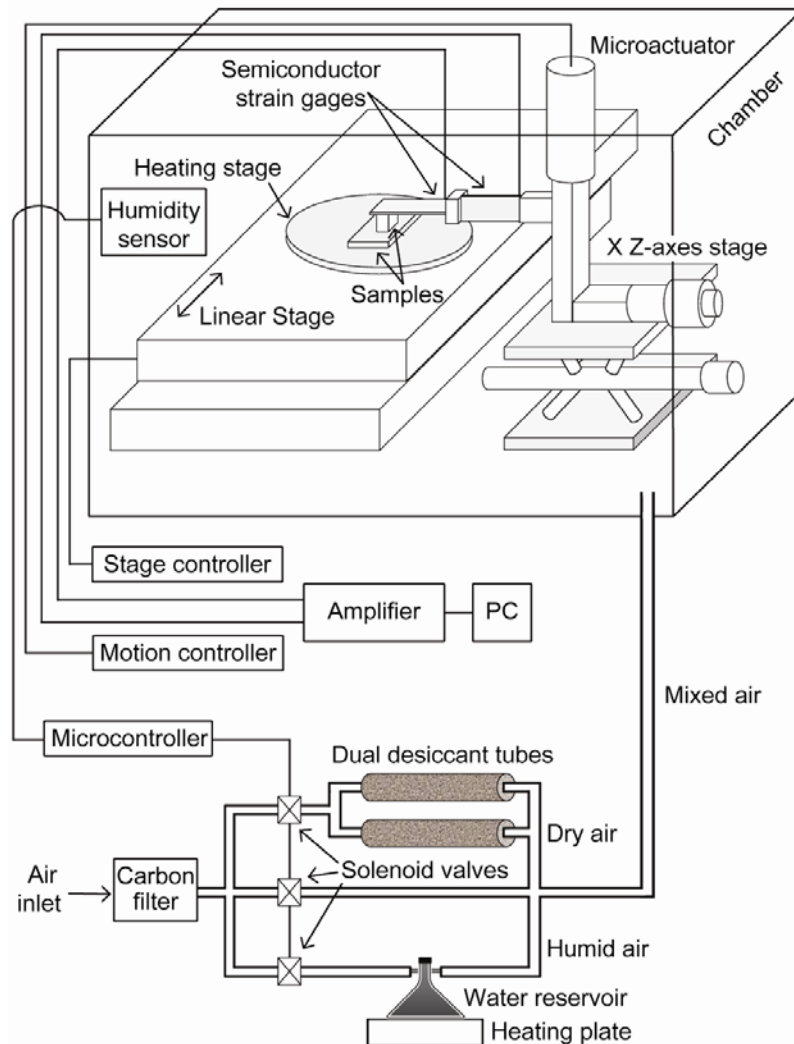
- Philips XL-30 ESEM
- Hair sample sputtered with thin gold coating prior to SEM measurements



In situ surface characterization of localized deformation conducted using a tensile stage inside an AFM



Macrotribological Characterization Using Tribometer with an environmental chamber



- Macroscale friction and durability studies were carried at various temperature and humidity conditions were carried out using a tribometer.

Hair Samples

- Virgin and damaged
 - Virgin
 - Chemically damaged (bleached and colored)
 - Mechanically damaged (exposed to combing which shows high degree of cuticle damage)
- Ethnic Hair
 - Caucasian, Asian, and African
- Treatment
 - Polydimethylsiloxane (PDMS) silicon based conditioner
 - Aminosilicone based conditioner that attaches chemically to the surface
 - Soaked in DI water for 5 min
(Time for water to penetrate)

Contact angle of hair, human skin, and AFM tips

Contact angle affects wettability of hair; one also needs to know how care products affect contact angle.

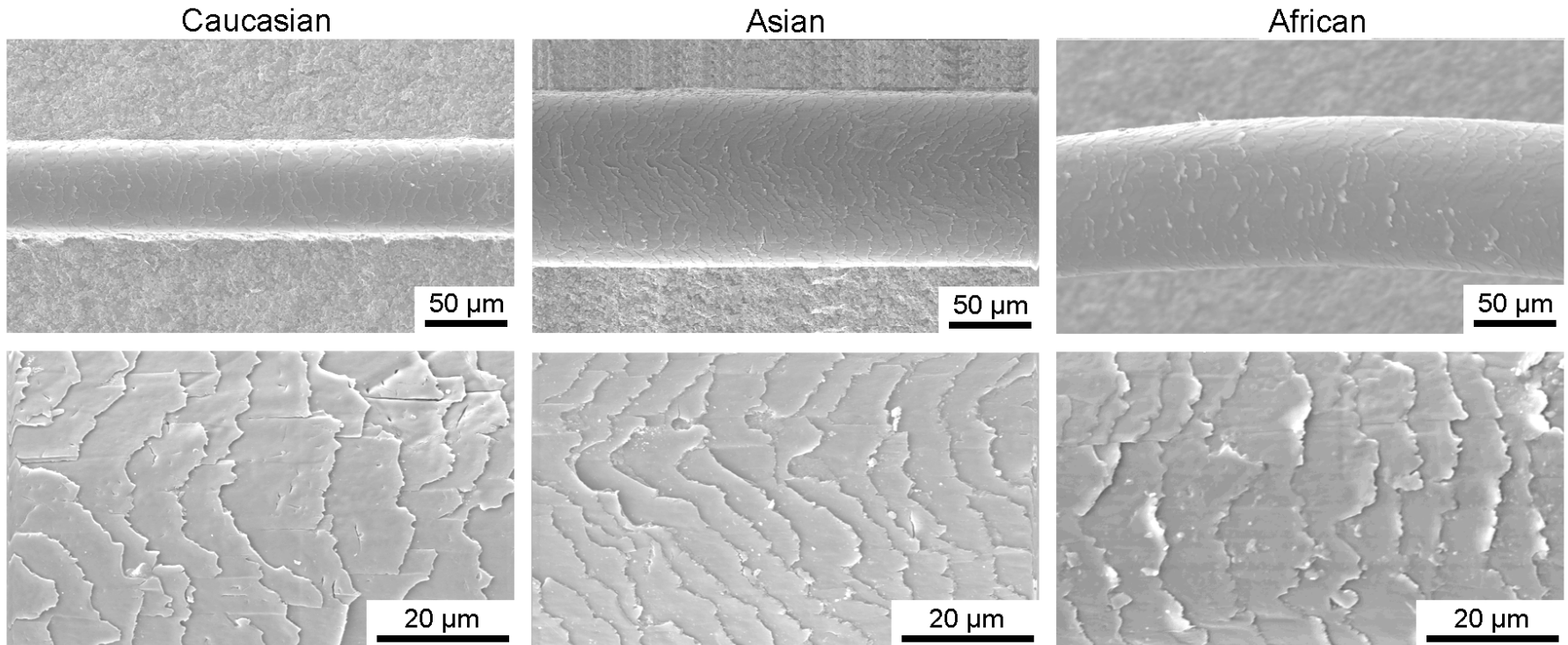
Contact angles need to be simulated in our measurements.

Sample	Contact angle (°)		Surface energy (N/m)
	Dry	Soaked	
Virgin Caucasian hair	103 ^a	98 ^a	0.028 ^b
Virgin treated	88 ^a	92 ^a	
Chemically damaged	70 ^a	70 ^a	
Chemically damaged, treated	79 ^a	84 ^a	
Mechanically Damaged	95 ^a		
Asian	92 ^a		
African	80 ^a		
Human skin - forehead	55 ^c		0.043 ^c
- forearm	88 ^c		0.038 ^c
	84 ^d		0.029 ^d
- finger	74 ^d		0.024 ^d
	104 ^e (after soap washing)		0.027 ^e
	58 ^e (before soap washing)		
Si ₃ N ₄ film	48 ^f		
Si tip	51 ^g		



Hair Cellular Structure Characterization

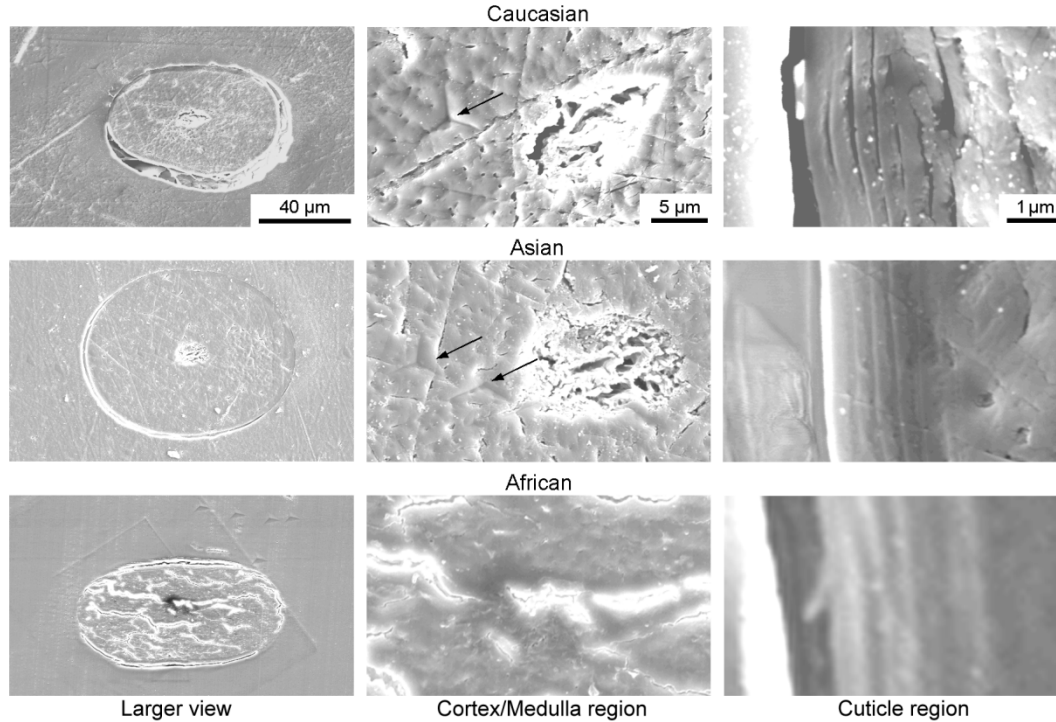
Various ethnic hair using SEM



- Cuticles consists of of flat overlapping scales, observed in all ethnic hair. These appear like tiles on a roof, attached at the root end and point toward the tip end of the hair fiber.
- Hair diameters are different in different ethnic hair.

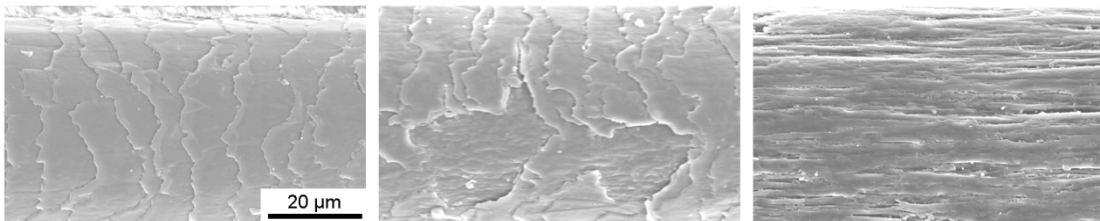
Hair cross section

SEM images of virgin hair cross section



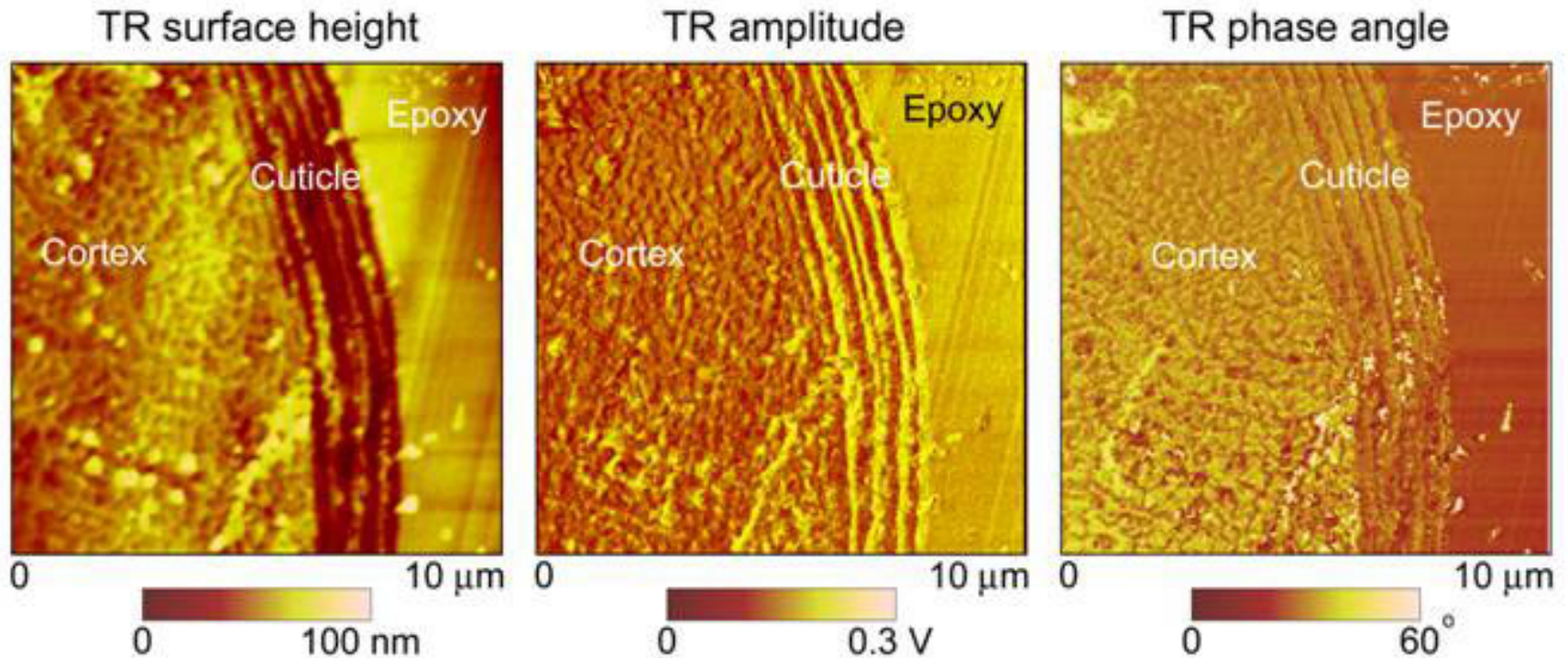
- Asian hair seems to be the thickest (~80-100 μm thick, nearly round), followed by African hair (~50-100 μm thick, significantly elliptical) and Caucasian hair (~50-80 μm thick, elliptical).
- Hair near the scalp (root) has complete cuticle scales while no scales are found near the tip.
- Near the tip, the hair experiences more mechanical damage during its lifetime than near the root.

Effect of location



G. Wei, B. Bhushan, and P. M. Torgerson, *Ultramicroscopy* **105**, 248 (2005).

Cross section of virgin Caucasian hair Using AFM



The cortex region, the cuticle region (5 layers of cuticle cells, total $\sim 2 \mu\text{m}$) and epoxy resin region are easily identified.

Sublamellar structure of the cuticle is observed at high mag. (not shown).

Cortex region shows very fine circular structure of size $\sim 50 \text{ nm}$, which represents the transverse face of the macrofibril and matrix.

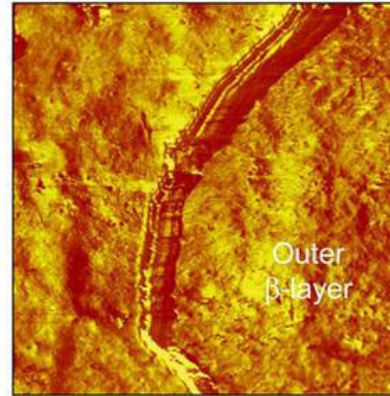
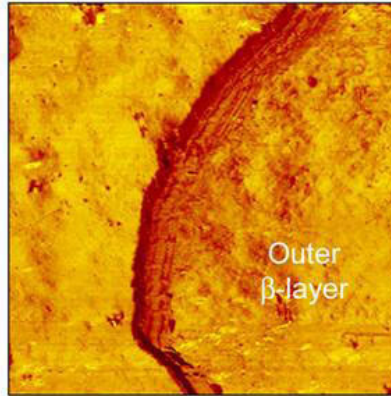
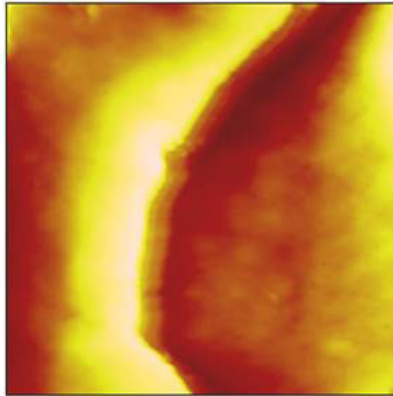
Effect of location (near root and near tip)

TR surface height

TR amplitude

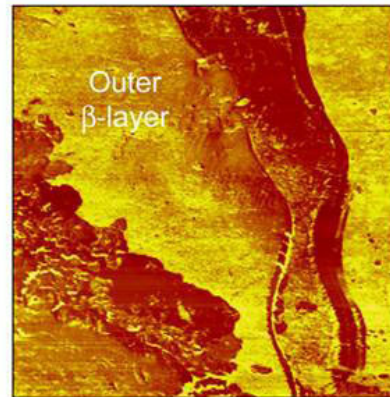
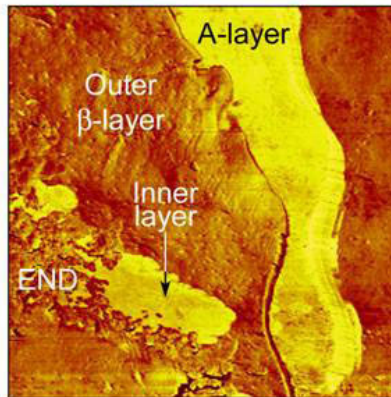
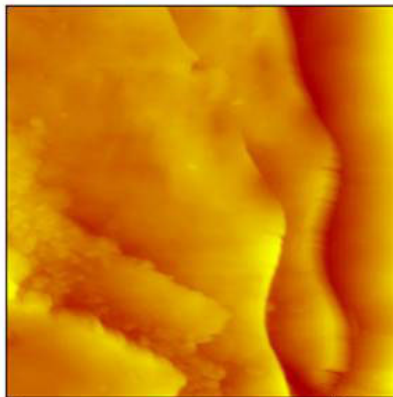
TR phase angle

Position 1
(near root)



Intact cuticle edge

Position 2
(near tip)



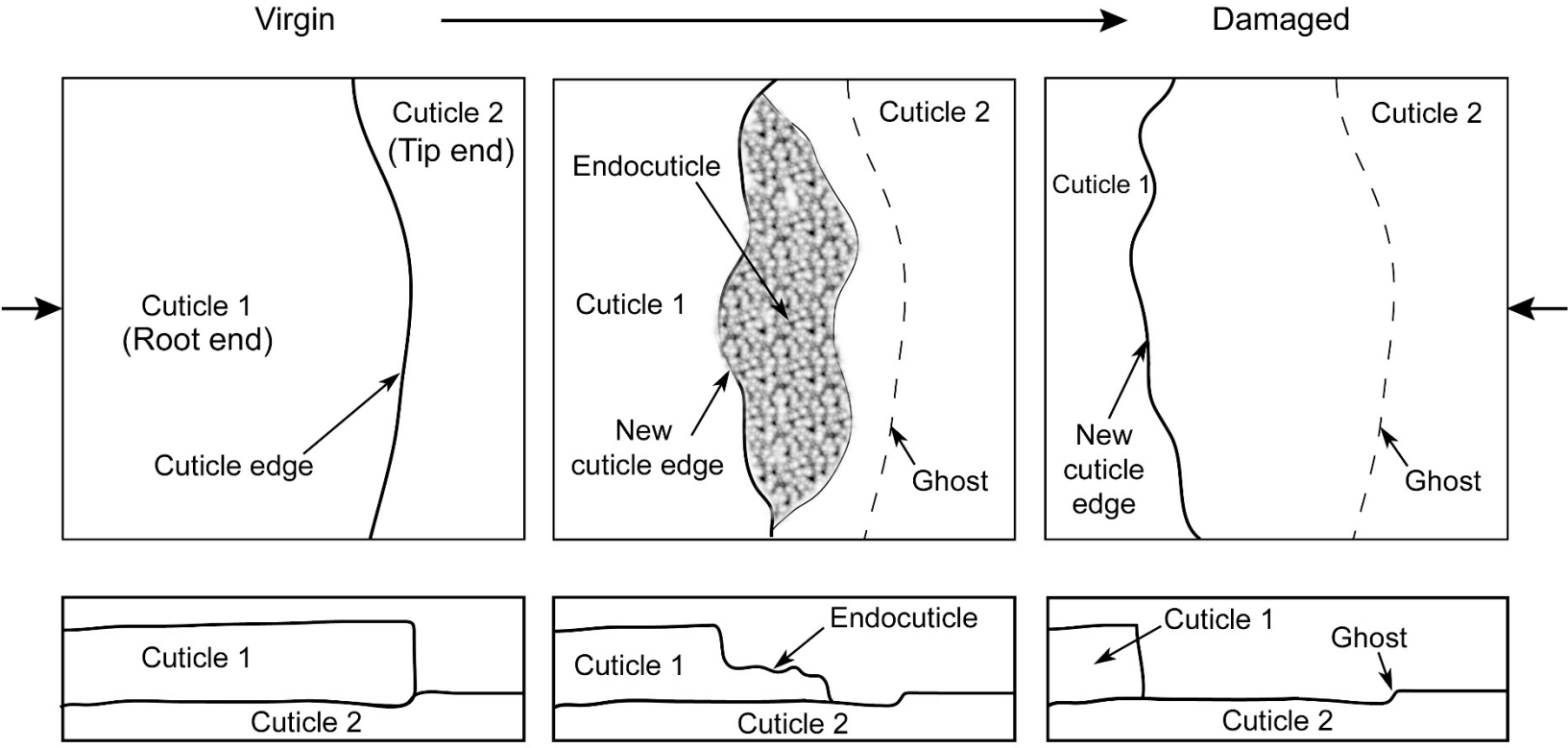
Lots of damage along the cuticle edges



Near the tip, parts of the cuticle outer sublamellar layers were removed and underneath layers (the A-layer, the endocuticle, the inner layer) are exposed

N. Chen and B. Bhushan, *J. Micros.* **220**, 96 (2005).

Damage process of hair surface



Due to natural weathering or mechanical/chemical damage, parts of the cuticle outer sublamellar layers wear off and underneath layers are exposed.

Further damage will cause entire piece of cuticle to be broken off. The ghost which delineate the original boundary of the cuticle edge is seen.

N. Chen and B. Bhushan, *J. Micros.* **220**, 96 (2005).

Summary

- Various sublamellar cellular structures of cuticle are easily observed using AFM with TR mode technique because of their distinct stiffness and viscoelastic properties.
- Surface of virgin hair near its root end shows little damage and intact smooth cuticle edges.
- Damage occurs near tip end of hair surface because of natural weathering and mechanical/chemical damage, exposing various sublamellar cuticle layers.

Nanomechanical Property Characterization

Hardness and elastic modulus plots across various virgin hair

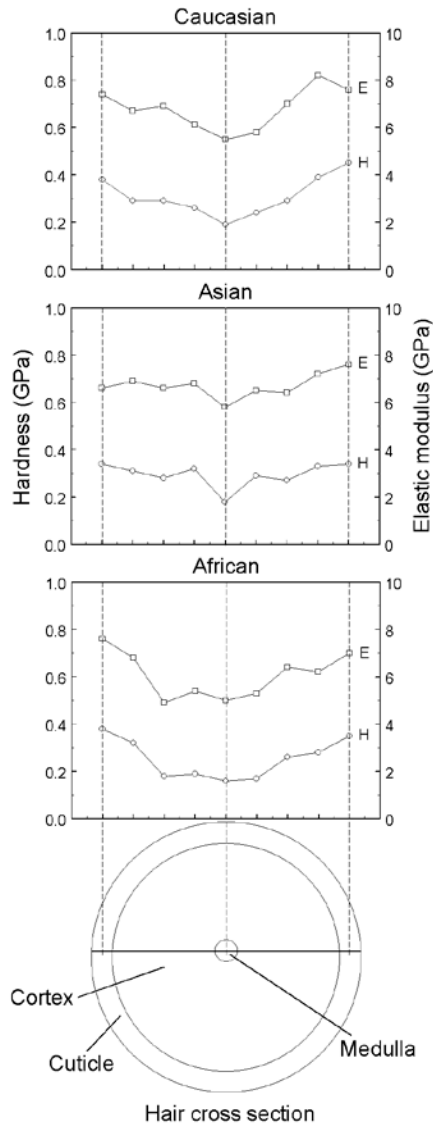


Table Summary of hardness and elastic modulus of human hair.

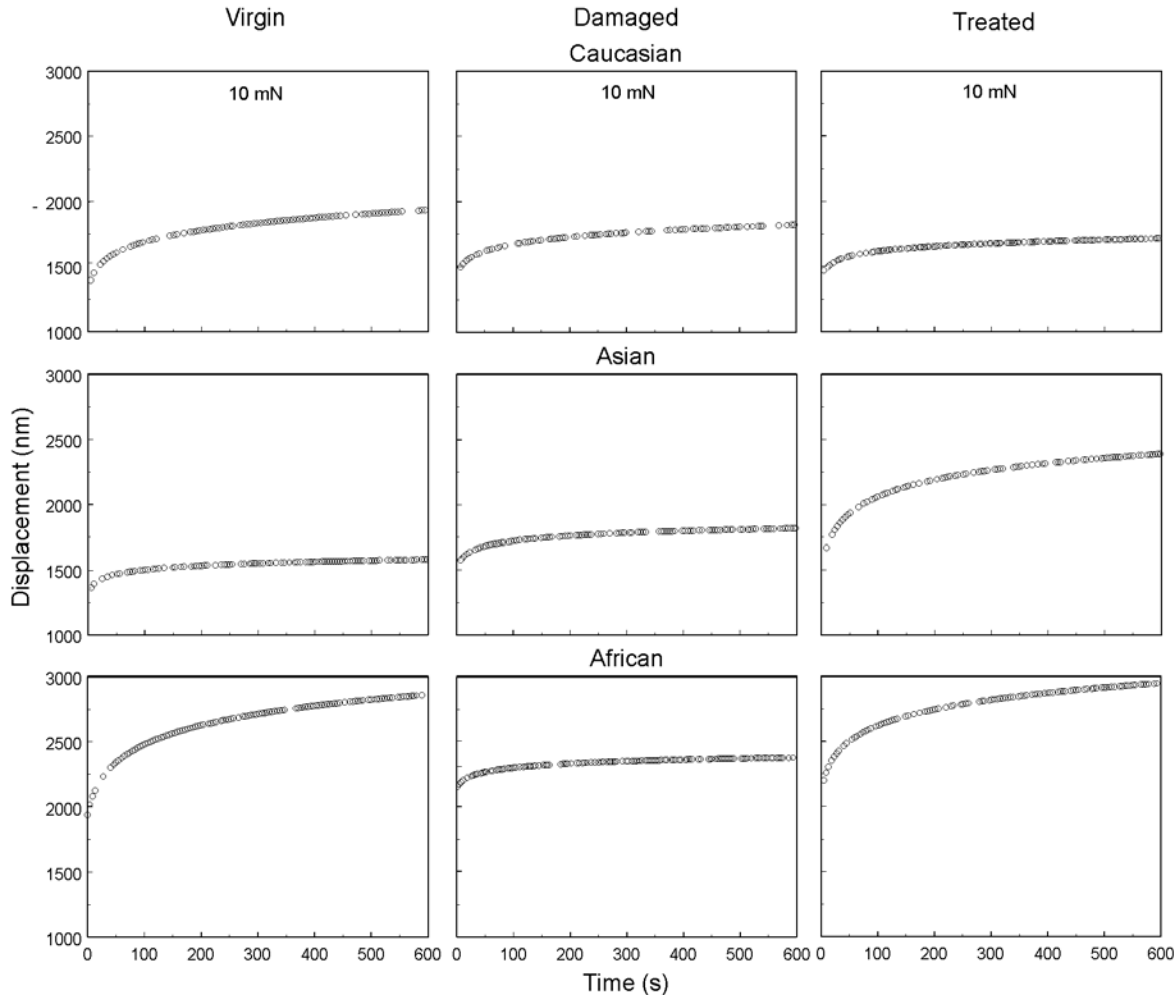
	Hardness (GPa)			Elastic modulus (GPa)		
	Cuticle ^a	Cortex ^b	Medulla ^b	Cuticle ^a	Cortex ^b	Medulla ^b
Caucasian	0.32 ± 0.04	0.27 ± 0.02	~ 0.19	6.0 ± 0.4	6.5 ± 0.5	~ 5.5
Asian	0.39 ± 0.06	0.30 ± 0.02	~ 0.18	7.5 ± 0.8	6.7 ± 0.3	~ 5.8
African	0.24 ± 0.05	0.23 ± 0.06	~ 0.16	4.8 ± 0.6	5.8 ± 0.7	~ 5.0

^aObtained from the hair surface at normal load of 1.0 mN

^bObtained from the hair cross section at normal load of 1.0 mN

- Hardness and elastic modulus of hair decreased going from the surface to the core.
- The Caucasian, Asian and African hair have different nanomechanical properties.
- It appears that Asian hair has the highest hardness and elastic modulus, followed by Caucasian and African hair.

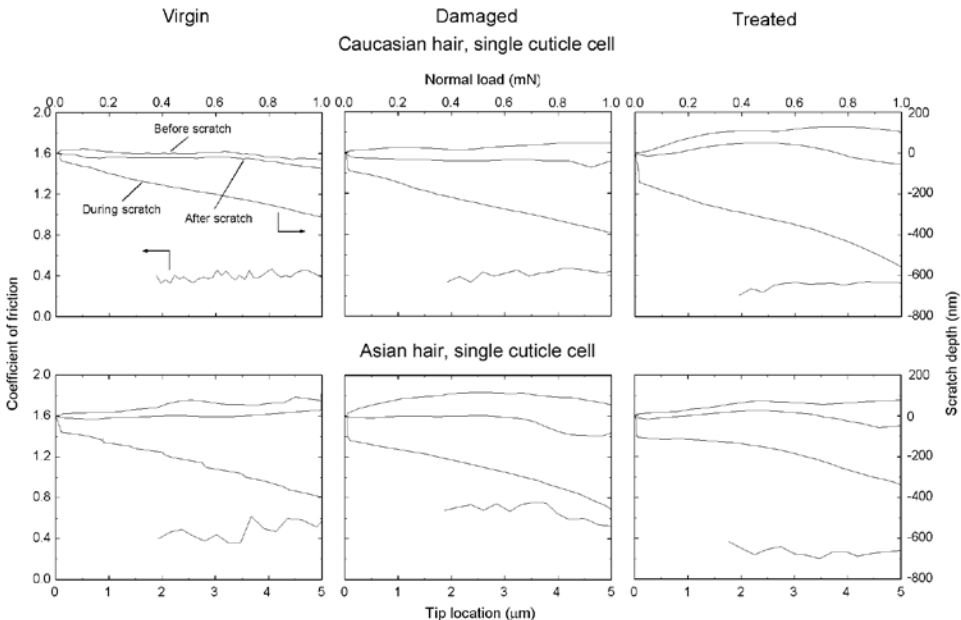
Creep displacement vs. time curves for various hair



- In all cases, the displacement increases with time at a constant load, i.e., hair creeps.
- It is difficult to correlate the creep behavior of each hair to its ethnicity and condition (virgin, damaged, or virgin treated).

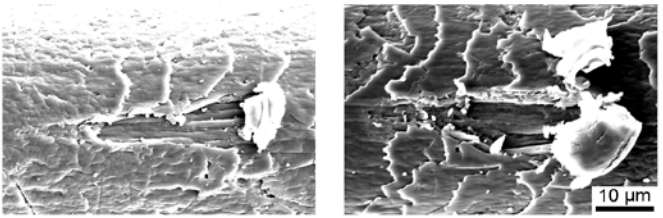
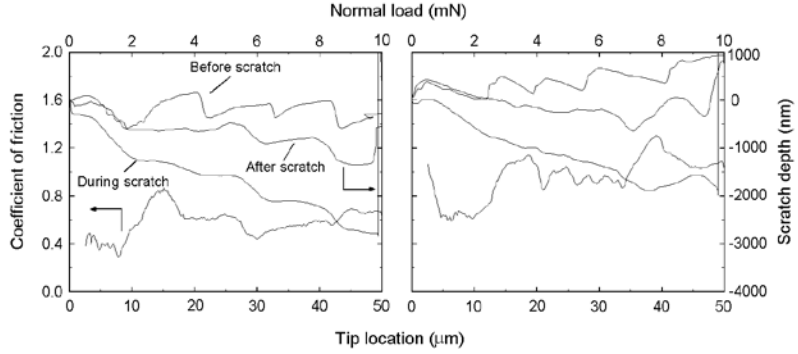
Scratch resistance

Comparison of coefficient of friction and scratching



Directionality effects on coefficient of friction and scratching

Damaged Caucasian hair, multiple cuticle cells
 Along cuticle Against cuticle



- Scratch depth during scratch is higher for the chemically damaged hair than virgin hair. Chemical damage softens the hair surface, which allows tip to plow into the hair easily.
- Scratch depth in treated hair is high because the tip can easily penetrate into a thin layer of conditioner.

- Coefficient of friction and damage to hair surface is higher when the tip is scratches the hair against the cuticle.

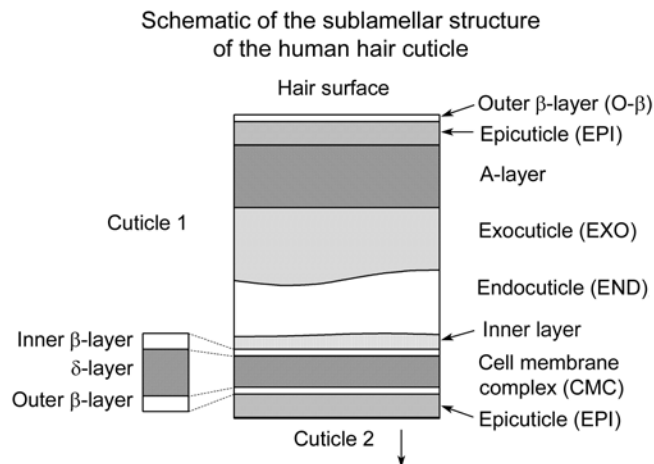
G. Wei and B. Bhushan, *Ultramicroscopy* **106**, 742 (2006)

Cuticle response to tension – by using *in situ* surface characterization of localized deformation

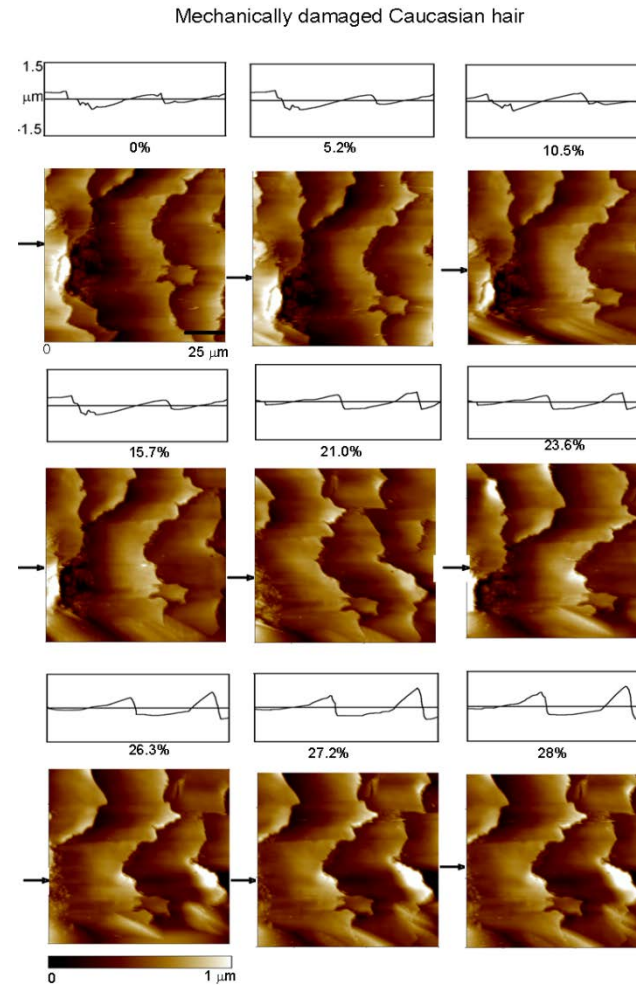
Virgin Hair

Most significant effect of tension – cuticle lift off due to extensibility differences occurs at 20% strain

- Endocuticle and cell membrane – weak, extensible;
- exocuticle, epicuticle – cross linked, rigid
- Difference in extensibility - interlayer shear forces
- 20% strain – failure in endocuticle and delamination - Outer cuticle lifts off



Damaged Hair



- In damaged hair, lift off of cuticle, and fracture and disappearance of cuticle edges occurs.

Summary

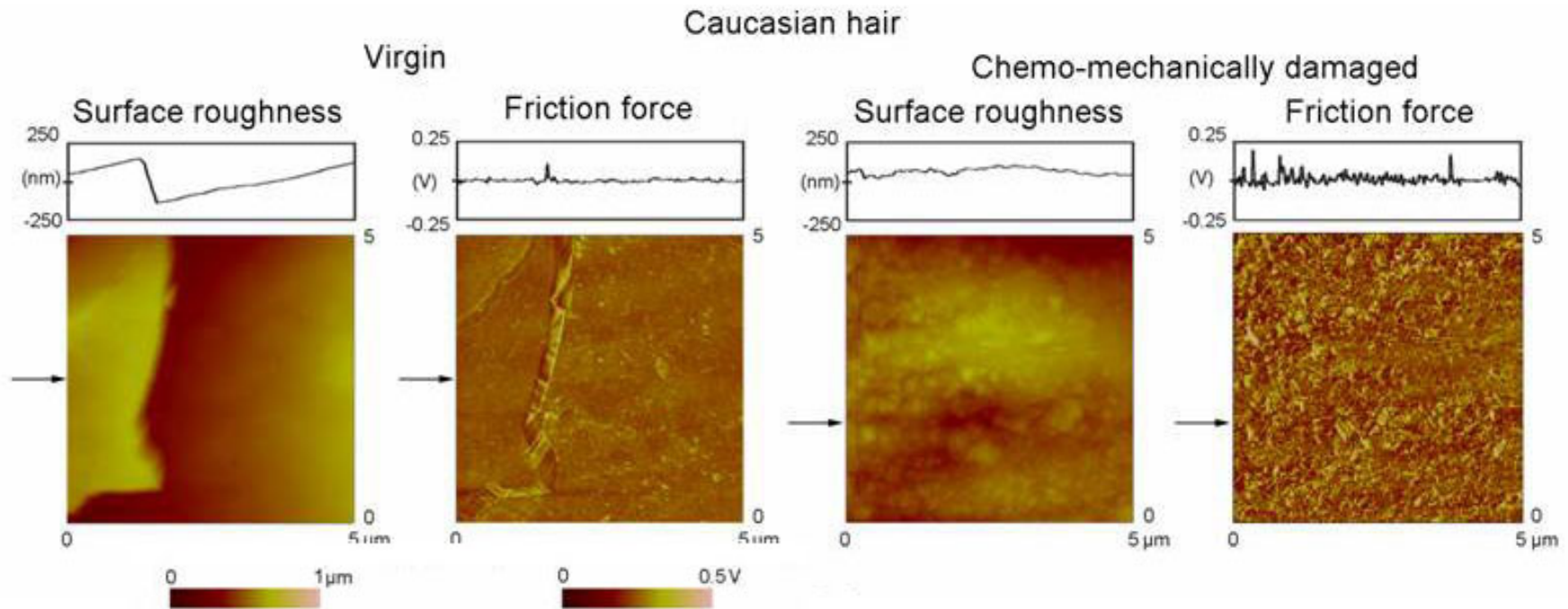
- The hardness and elastic modulus of hair decreased going from the surface to the core. The cystine content variations in cuticle substructures and cortex are believed to be responsible for the variations.
- The Caucasian, Asian and African hair have different nanomechanical properties. It appears that Asian hair has the highest hardness and elastic modulus, followed by Caucasian and African hair.
- Hair exhibits viscoelastic properties.
- Hair scratches and gets damaged at high loads. Scratch depth is higher for chemically damaged hair than virgin hair because it is softer.
- Tensile stresses cause lifting of outer cuticle at ~20% strain in virgin hair, due to interlayer shear forces and separation between inner and outer cuticle layers.
- Chemical and mechanical damage cause lift off and fracture of outer cuticle (at 10% strain in mech. damaged hair, 20% in chem. damaged).



Nanotribological Characterization

Surface Roughness and Friction

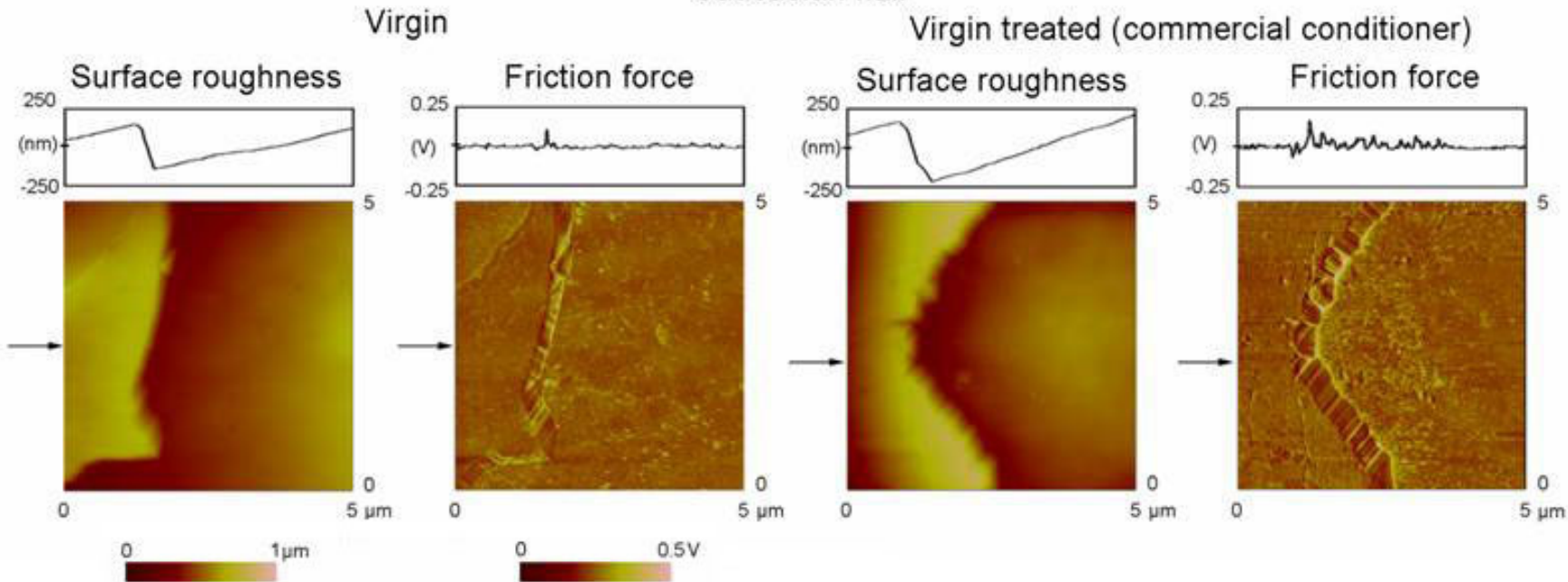
Virgin and chemo-mechanically damaged hair



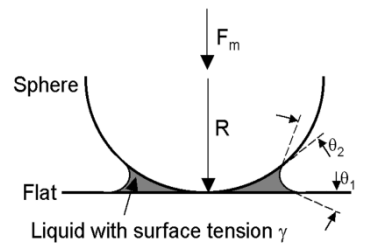
- Virgin hair: Intact cuticle, low friction
- Damaged hair: Severe cuticle wear, high friction due to an increase in roughness and change in surface properties.
- True for all ethnicities

Virgin and virgin treated (commercial conditioner) hair

Caucasian hair



- Friction is similar between virgin and virgin treated hair
- Virgin treated hair: high friction contrast due to meniscus contributions due to accumulation of conditioner near cuticle edge
- True for all ethnicities



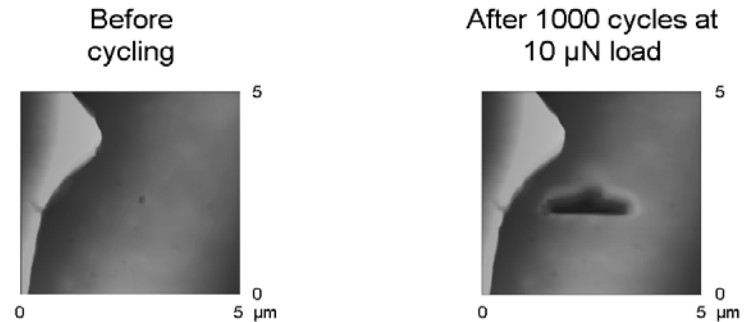
$$F_m = 2\pi R\gamma (\cos\theta_1 + \cos\theta_2)$$

C. LaTorre and B. Bhushan, *Ultramicroscopy* **105**, 115 (2005).

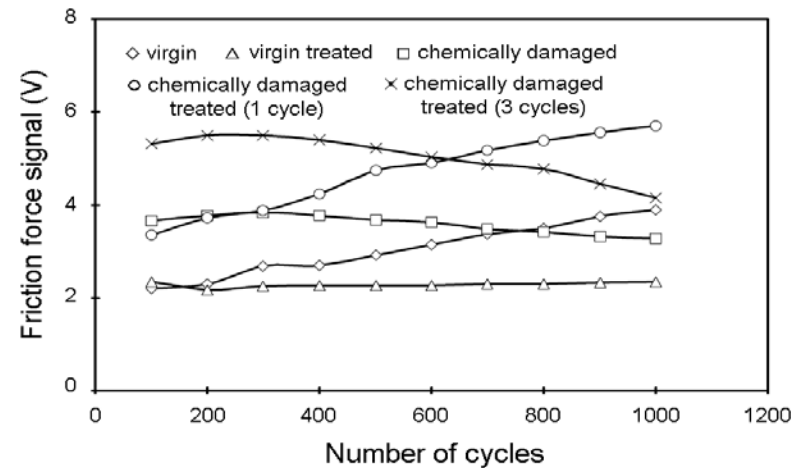
Durability Test - Effect of cycling with hard Si AFM tip

- Simulation of wear such as by sand particles or combing
- Friction for virgin hair increased (lipid layer worn away)
- Virgin treated hair showed no increase of friction (protective covering)
- Friction of chemically damaged hair was high and remained high

Surface roughness of unworn and worn virgin hair



Durability effects on friction force of various hair



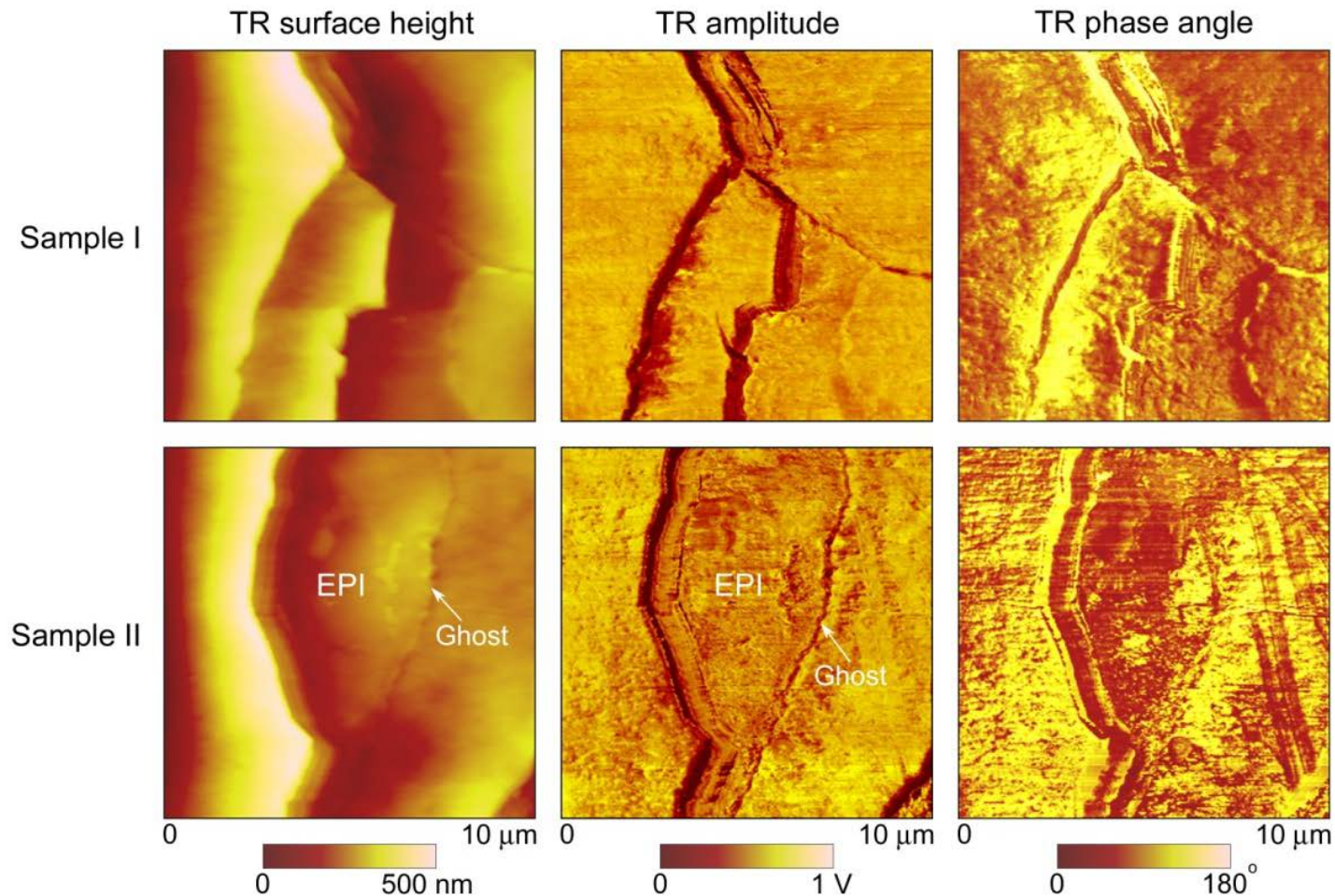
Summary

- Friction is higher on chemo-mechanically damaged hair than on virgin hair, due to an increase in surface roughness and change in surface properties.
- Friction is similar between virgin and virgin treated hair.
- Durability tests were performed to simulate wear such as by sand loose particles.
- Friction for virgin hair increased due to lipid layer being worn away.
- Virgin treated hair showed no increase of friction because of the presence of conditioner which serves as a protective covering.
- Friction of chemically damaged hair was high and remained high.



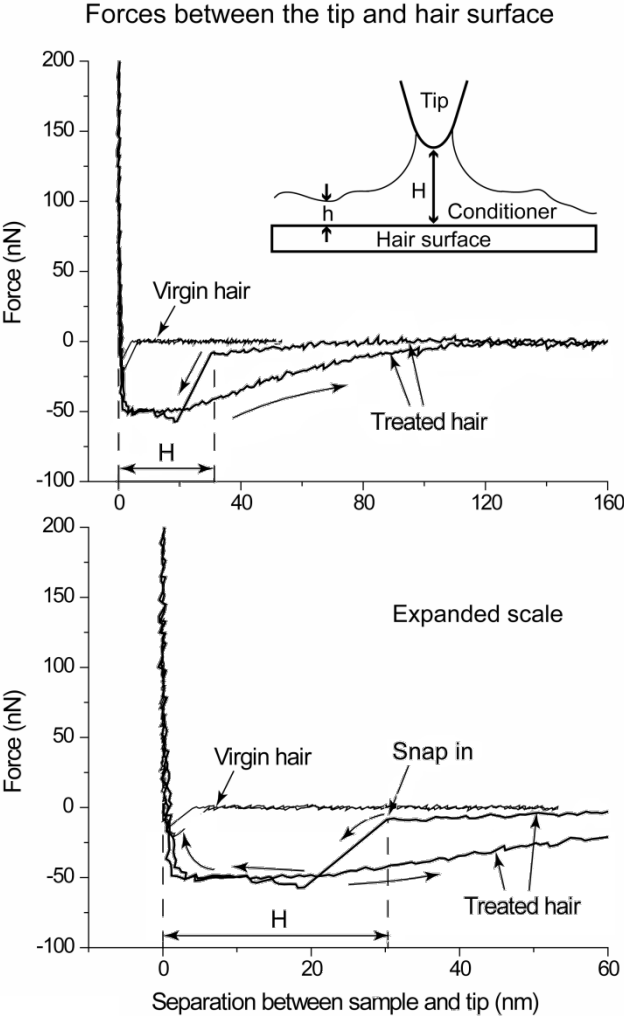
Conditioner Distribution Studies using AFM

Conditioner distribution mapping - treated damaged hair



High TR phase contrast near cuticle edges
- conditioner collected at cuticle edges.
- conditioner layer is unevenly distributed.

Conditioner film thickness estimation

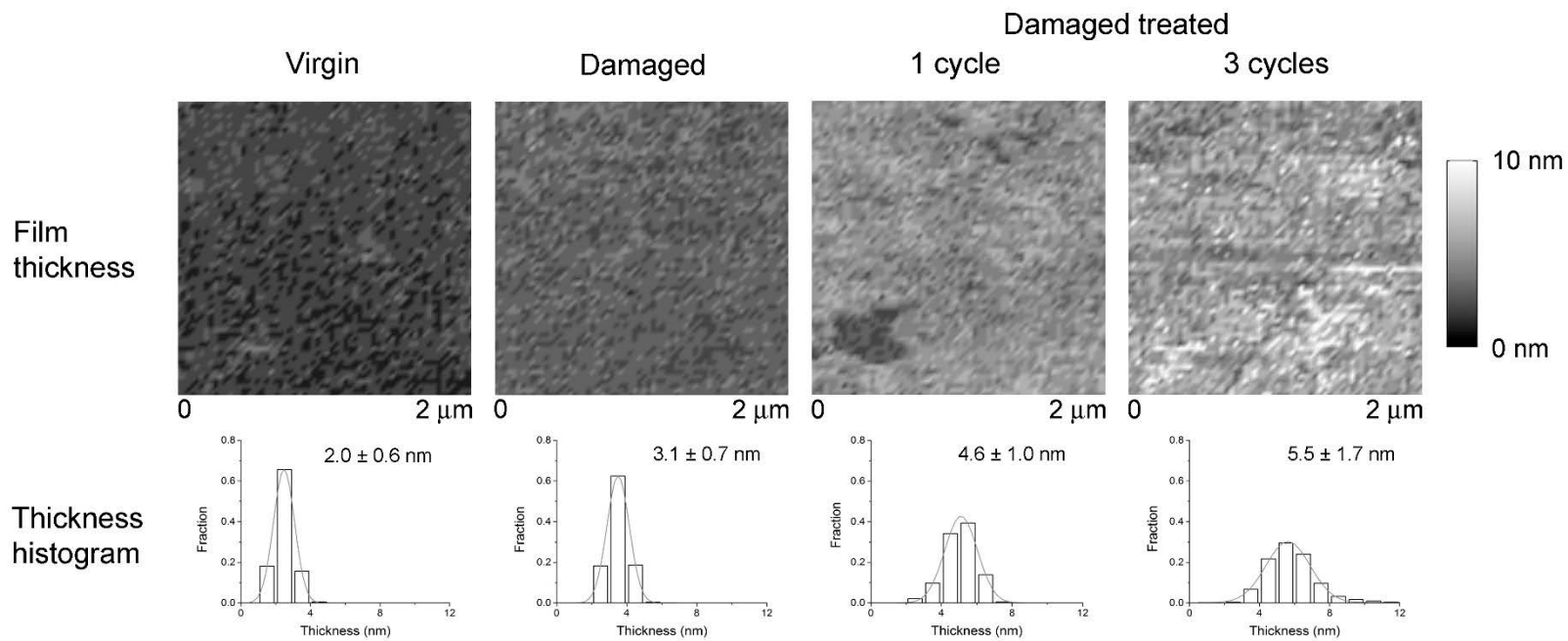


Force calibration plot technique

B. Bhushan and G.S. Blackman, *ASME J. Tribol.* **113**, 452 (1991); N. Chen and B. Bhushan, *J. Micros.* **221**, 203 (2006); R. Lodge and B. Bhushan, *J. Vac. Sci. Technol. A* **24**, 1258 (2006)

Film thickness mapping

Film thickness maps of various hair samples



- Thickness of damaged hair (3.1 nm) is larger than virgin hair (2 nm).
- Damaging will partially remove the fatty-acid lipid layer and make surface hydrophilic and amount of water adsorbed on the hair surface increases.
- After conditioner treatment, thickness increases (4.6 nm and 5.5 nm)

N. Chen and B. Bhushan, *J. Micros.* **221**, 203 (2006)
R. Lodge and B. Bhushan, *J. Vac. Sci. Technol. A* **24**, 1258 (2006)

Summary

- Conditioner unevenly distributes and thicker conditioner film is found near the cuticle edges.
- Amino silicones seem to attach to hair and give low friction (Data not shown).

Surface Potential Studies using KPM

Introduction

- Hair is a good insulator ($\sim 10^{18}$ ohms/cm), and tends to develop significant static surface charge.
- Surface charge on hair has significant effect on manageability, feel, and appearance – “static flyaway effects”

Objective

- To measure local surface charge buildup on variety of hairs by Kelvin probe microscopy
- Investigate the effect of damage, conditioner treatments, and humidity

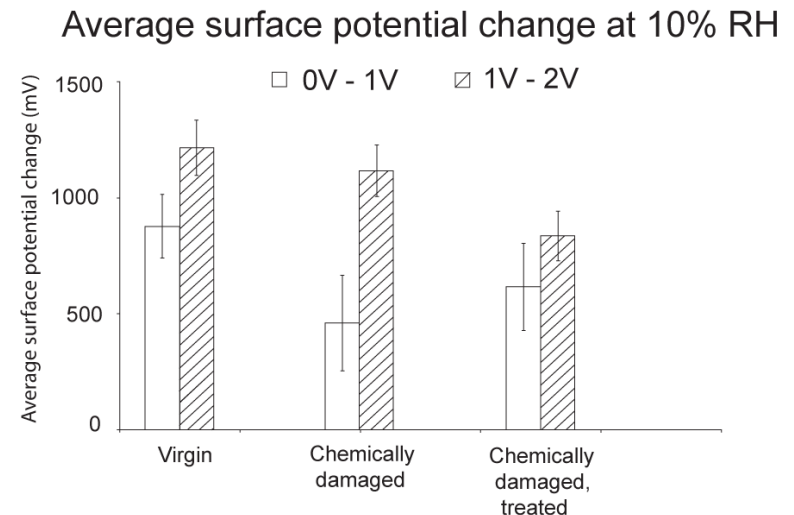
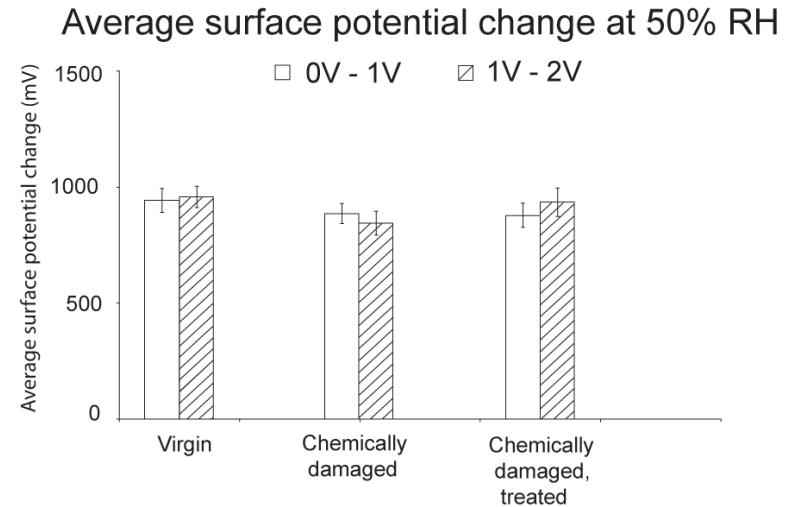
Triboelectric charging

- Voltage applied across hair sample (0 – 2 V)
- Macroscale charging – hair rubbed with latex finger cot, 5 passes
- Nanoscale charging – conducting tip rubbed at 750 nN. 6V bias applied internally to AFM to provide background potential contrast.
- Potential measured before and after rubbing using KPM (in-situ in nanoscale charging)



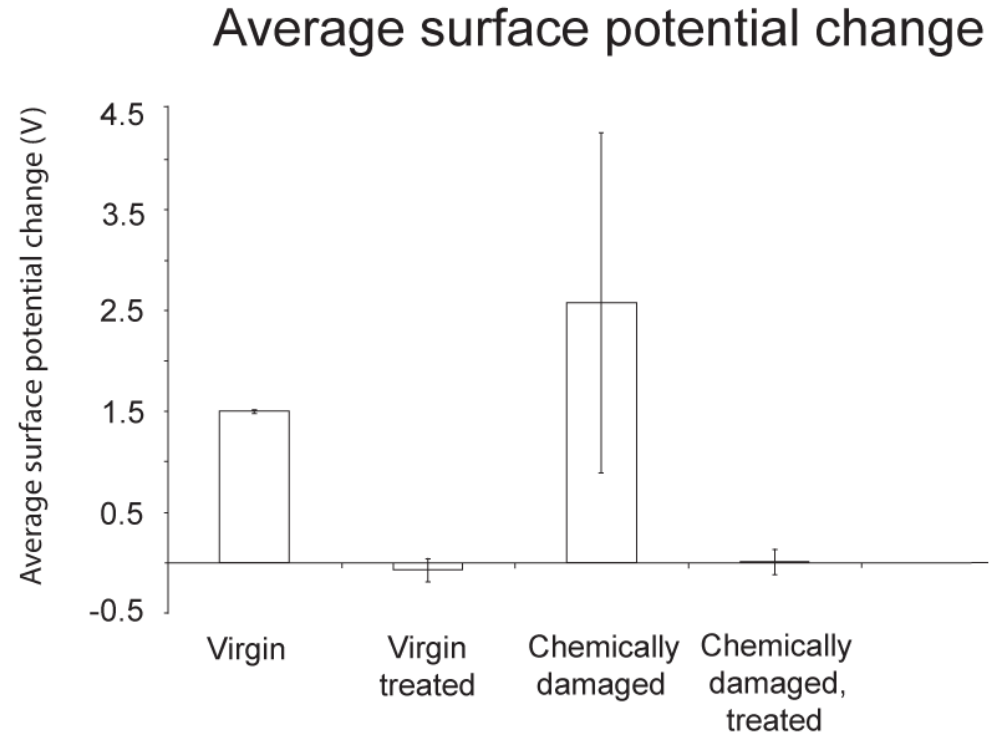
Voltage gradient applied across hair sample

- At 50% RH the potential change is near 1V for 1V change in applied potential. This indicates water vapor in the air improves the charge mobility.
- At 10% RH, the potential change is less than 1V for 1V change in applied potential. The potential change in chemically damaged hair is lower than that for virgin and conditioner treated hair.
- *Both humidity and conditioner treated hair increase the charge mobility which allows dissipation of charge.*

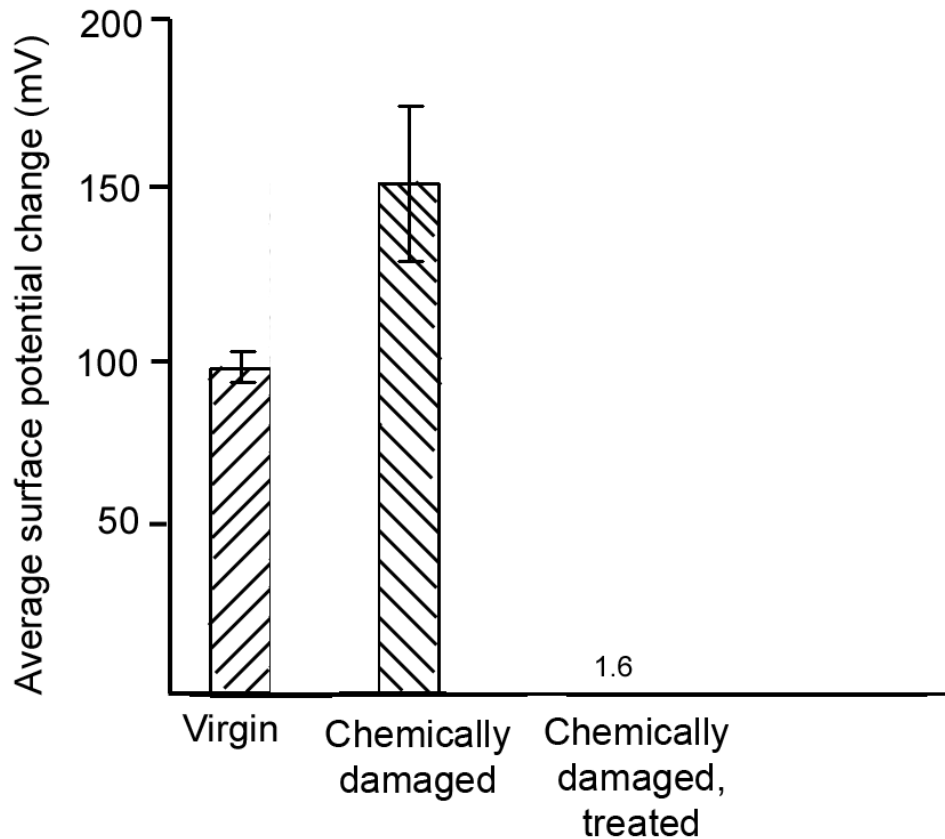


Triboelectric charging - Macroscale

- In chemically damaged hair with no lipid layer accumulated charge is not dissipated.
- *The natural lipid layer on virgin hair surface and conditioner treatment increase mobility of surface charges. This allows dissipation of trapped charges and reduces its amount.*



Triboelectric charging - Nanoscale



- Average potential change magnitudes - lesser on nanoscale
- Surface potential change is highest in chemically damaged hair followed by virgin hair and conditioner treated hair.
- *Lipid layer on virgin hair and conditioner increase the charge mobility responsible for decrease in charge accumulation.*

Summary

- The Kelvin probe technique is a powerful tool to study the surface potential characteristics of human hair.
- Applying voltage gradient across hair shows that conditioner treatment and water vapor significantly increases charge mobility on the hair surface.
- Rubbing with latex finger cot and with an AFM tip increases charge on hair. Chemical damage increases charge accumulation, and lipid layer on virgin hair and conditioner treatment increases charge mobility (reduced accumulation).

Overall Summary

- Human hair is a nano-composite biological fiber. A lipid layer covers the outermost surface of cuticle which gets removed.
- For a good dry and wet feel and combing with ease and without entanglement, the hair surface should exhibit low friction and adhesion against various surfaces.
- Mechanical and chemical damage needs to be minimized. A conditioner with lubricating ingredients can be used to reduce damage.
- Nanoscale characterization of cellular structure and mechanical properties as well as morphological, friction, adhesion and wear properties and role of surface charge are needed.
- These studies have been successfully carried out using an AFM and nanoindenter.

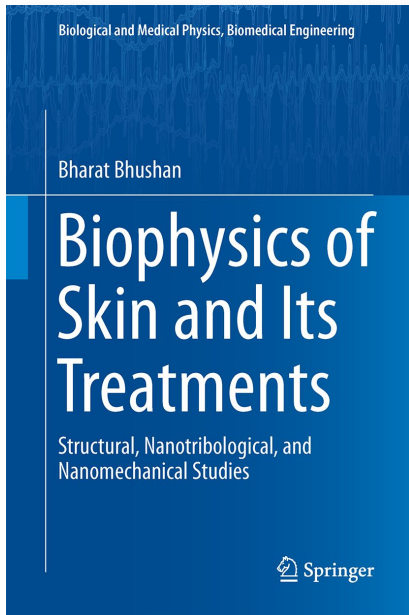


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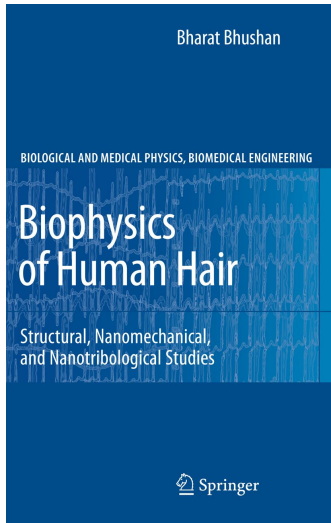
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