Methodologies for Relating Scratch Damage to Its Appearance Properties of Polymeric Materials

- PIC Phase II Proposal -

Principal Investigators

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Performed Extensive Scratch/Mar Literature Review

Variation in Testing Parameters

Test/System	Tip material	Tip geometry	Force Range	Depth Range	Speed/length
AFM	Diamond	Non-ideal < 0.1 μm radius	(1 - 400) μN	(10 –250) nm	(1-70) μm/s (1 –70) μm
AFM	Diamond	90° cone 1 μm radius	50 μN - 4 mN	50 nm - 1 μm	(35-70) μm/s 70 μm
Ford	Steel	sphere 500 μm radius	(0.6 - 7) N 30 N	(0.5 –10) μm	100 mm/s ?
LTDS	Diamond	Berkovich pyramid < 0.1 μm radius	(1 – 7) N	50 μm	500 μm/s (1 – 10) mm
CSM	Diamond	Sphere 2 μm radius	(0 - 5) mN	(0.5 – 1) μm	5 μm/s ?
CSM	Diamond	Sphere 200 μm radius	(0.5 – 10) N	?	200 μm/s ?
CSM	Diamond	? 10 μm radius	(0 – 190) mN	(0 – 20) μm	50 μm/s 3 μm
DuPont	Diamond	60° cone, 3 μm radius ?, (1-2) μm radius	(0 – 8) mN	(1 – 4) μm	25 μm/s (1 – 10) mm
MTS	Diamond	Berkovich pyramid < 0.1 μm radius	(0.02 – 16) mN	(0 – 1.5) μm	(10-25) μm/s 500 μm
MTS	Diamond	Cube corner pyramid (0.5-2) μm radius	(0.02 – 16) mN	(0 – 2.5) μm	25 μm/s 500 μm

Motivations

- **Scratch and mar resistance is a critical performance property for polymeric materials and coatings in various applications.**

- **No universal standardized test method for scratch and mar resistance study**
  - Current scratch and mar test methods are highly dependent on test conditions (**Test/System, tip material/geometry, force/depth range, speed/length**)
  - Scratch and mar resistance assessment is often based on "relative but not objective" types of measurements, such as visual inspection, gloss changes, changes in gray scale level or lightness.
  - Most results are used for qualitative assessment purposes.
  - Lack of a standardized test method, which is repeatable and reliable for polymer materials communities.

Motivations- cont.

Needs for Scratch and Mars Study

- Basic understanding of the relationship between material/ mechanical properties, morphological deformation, and appearance (optical properties) of scratch and mars

- Standardized test methods for scratch test

- Well-defined measurement protocols for assessing scratch and mar resistance

  - when a scratch “is” a scratch?

Objectives of the Proposed Project

- To develop a methodology for relating scratch damage to material/mechanical properties of polymeric materials
- To develop a methodology for correlating scratch parameters (size, depth, density...) with optical scattering properties (appearance) for polymeric materials.
- To develop well-defined measurement protocols for assessing scratch and mar of polymeric materials.
- To develop a science-based standardized scratch test (ISO) based on nanoindentor for assessing scratch and mar resistance of polymeric materials.

Recent developments at NIST

- Recent efforts on correlating surface mechanical response to performance characteristics using depth sensing indentation provide a good measurement guide (current PIC project 1).
  - Development of a general procedure for nanointentation test
  - Development of a preliminary measurement protocol for scratch test

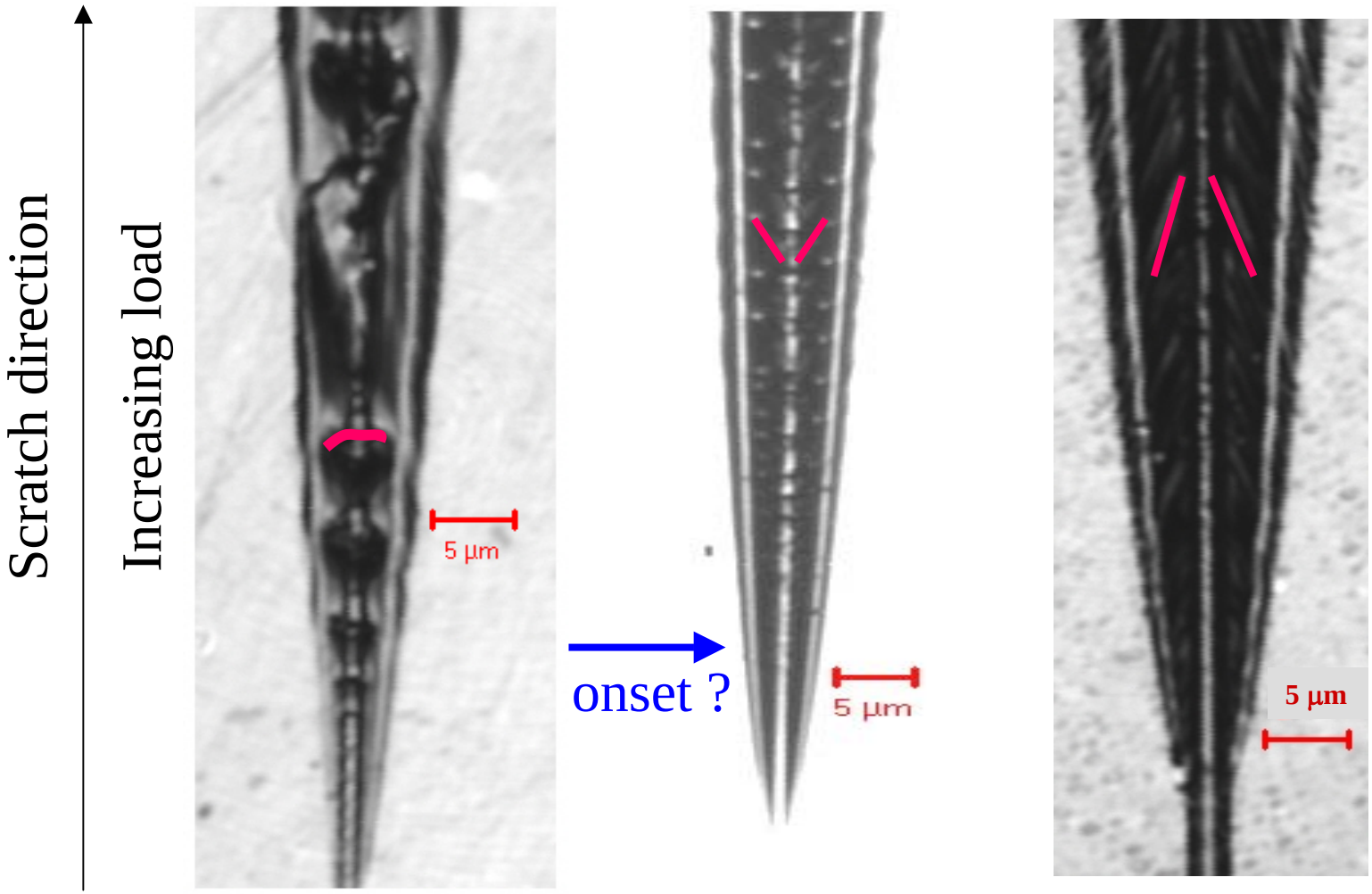
- A methodology has been developed at NIST for linking surface morphology/microstructure to optical reflectance that enables researchers to calculate and predict appearance attributes of polymer coatings.

- Establishing a state-of-the-art light scattering laboratory for characterizing nanoscale structural and optical properties (appearance) of polymers and coatings.

Scratch Damage

(mechanical response for different materials)

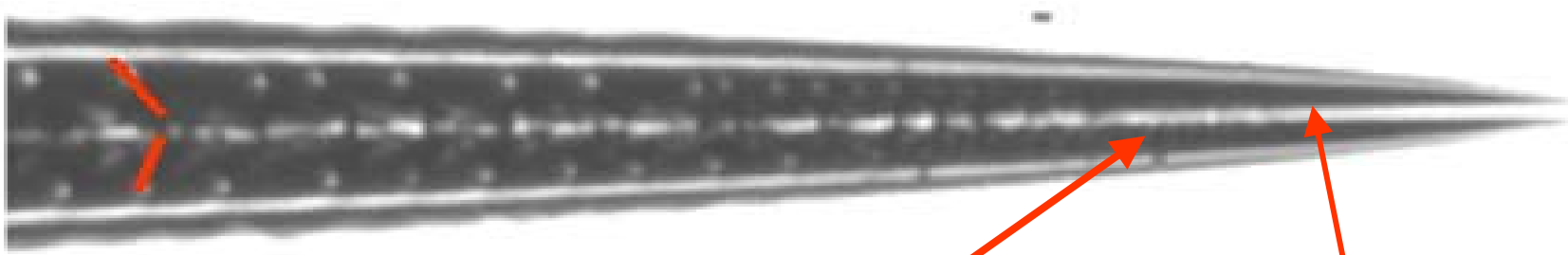
Scratch parameters: size, shape, width, depth, density, crack propagation, …etc.



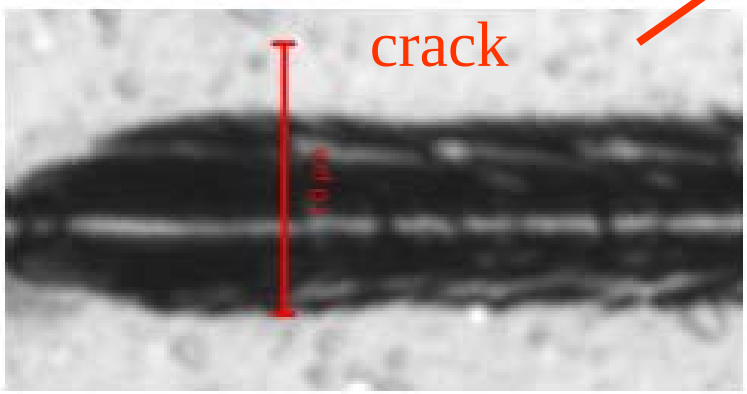
Epoxy PMMA PP

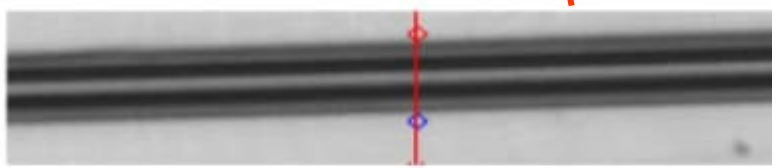
- **Progressive-force test**

Scratch direction



- **Constant-force test**

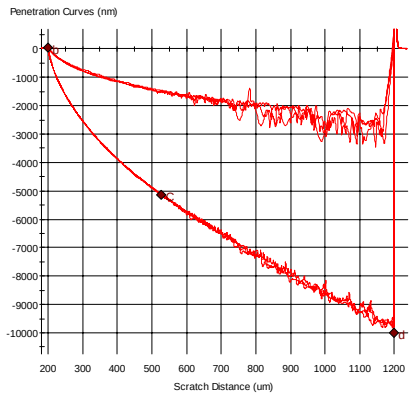




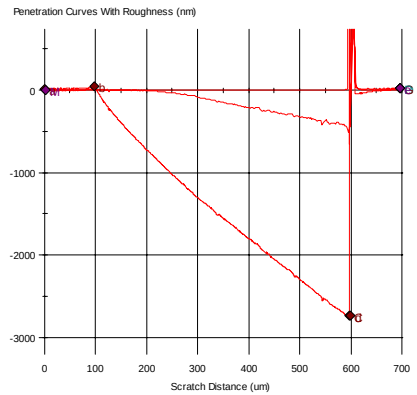
critical strain/force, friction coefficient, scratch depth & width as function of scratch rate ⇔ Viscoelastic Behavior of Materials

Preliminary Scratch Testing Results

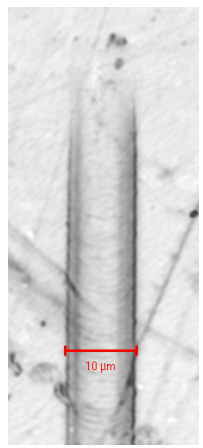
- **Preliminary scratch testing procedure**
  - **Use progressive force tests to identify scratch test parameter ranges of interest**
  - **Use constant force tests to study important ranges of scratch behavior**
- **Tip geometry- important**
- **Scratch velocity- important**
- **Friction coefficient - important**
- **Force level - important, but:**
  - **So-called "critical load" parameter not useful as a measure of scratch resistance**



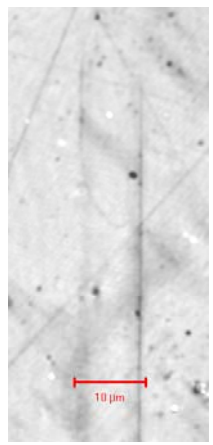
1 μm radius tip



10 μm radius tip



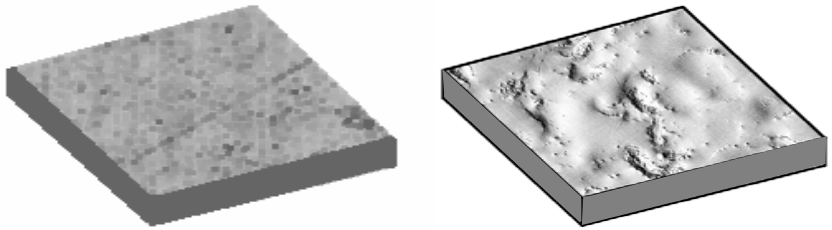
1 μm/s



10 μm/s

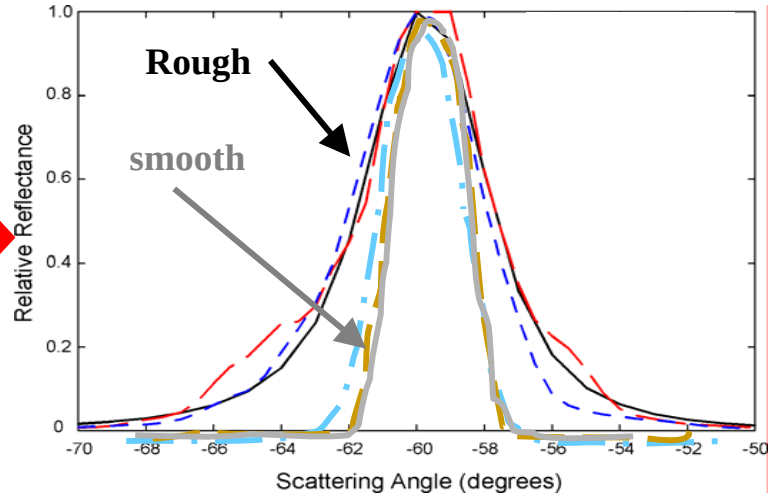
Methodology for Linking Surface Morphology to Optical Reflectance

Characterize Surface Roughness

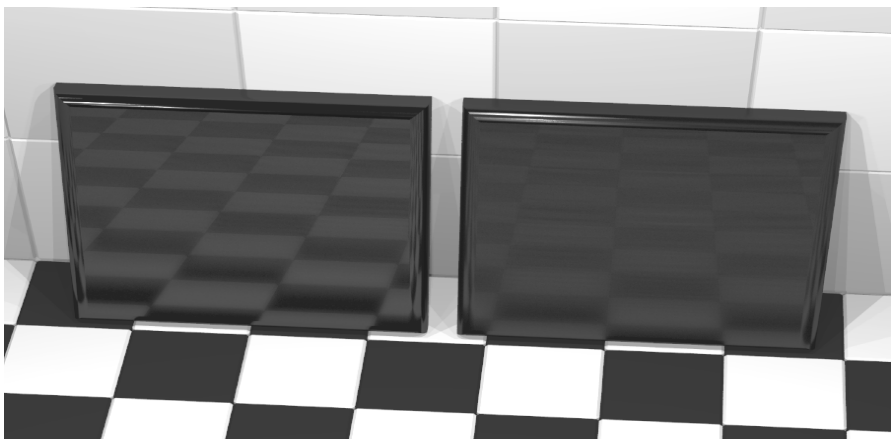


200 nm rms 800 nm rms

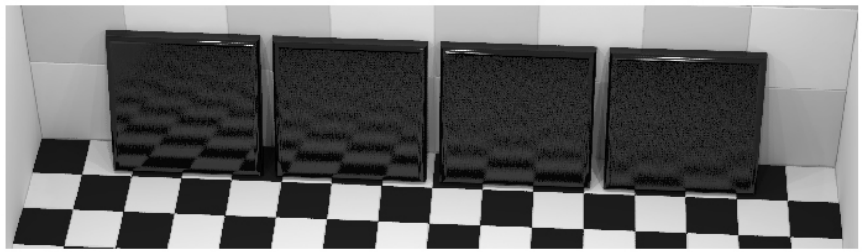
Measure & Model reflectance



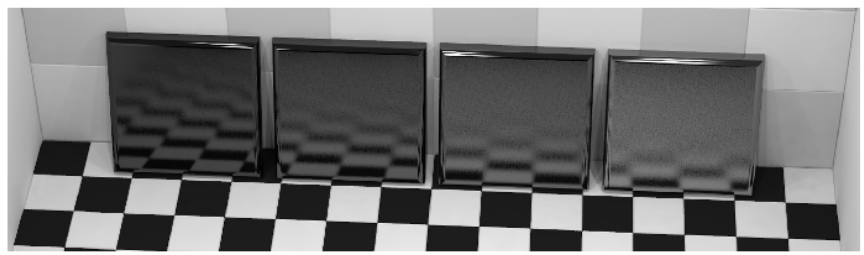
Rendered Image



Computer-based Gloss Standards for Rendering



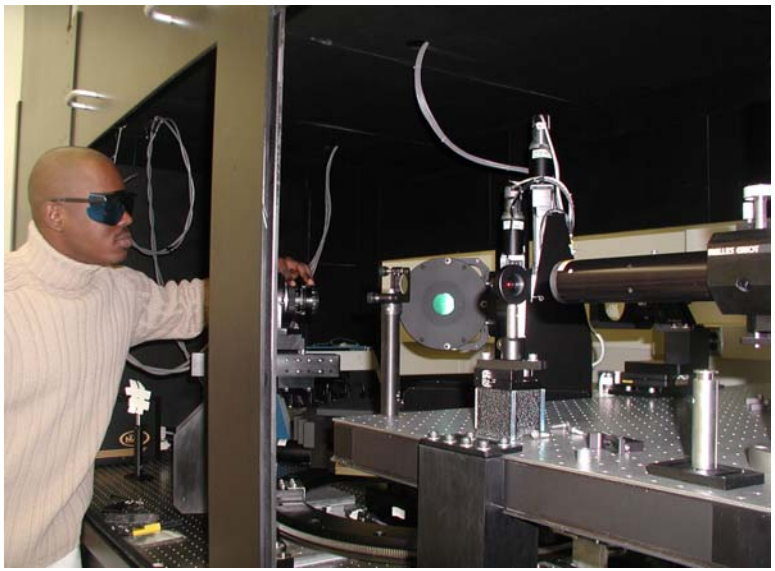
Gloss, D523

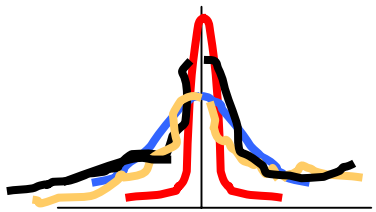


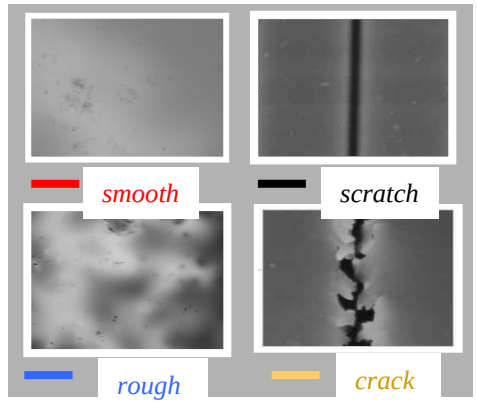
Haze (distinctness of image), D4039

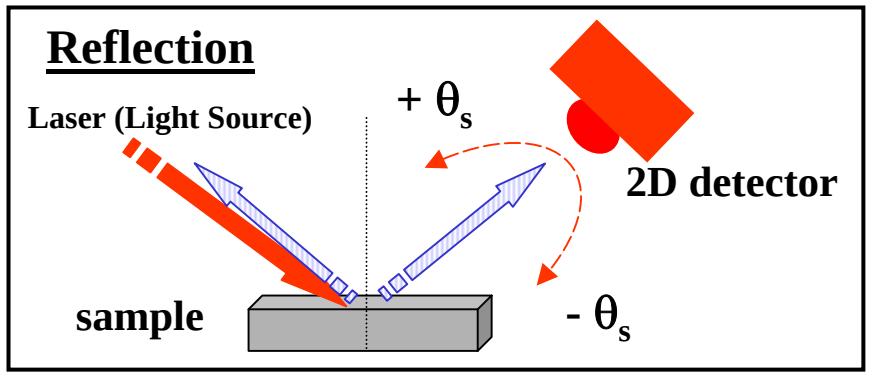
Light scattering lab for appearance measurements of scratch and mar

Light Scattering Lab. (A308/Bldg. 226)





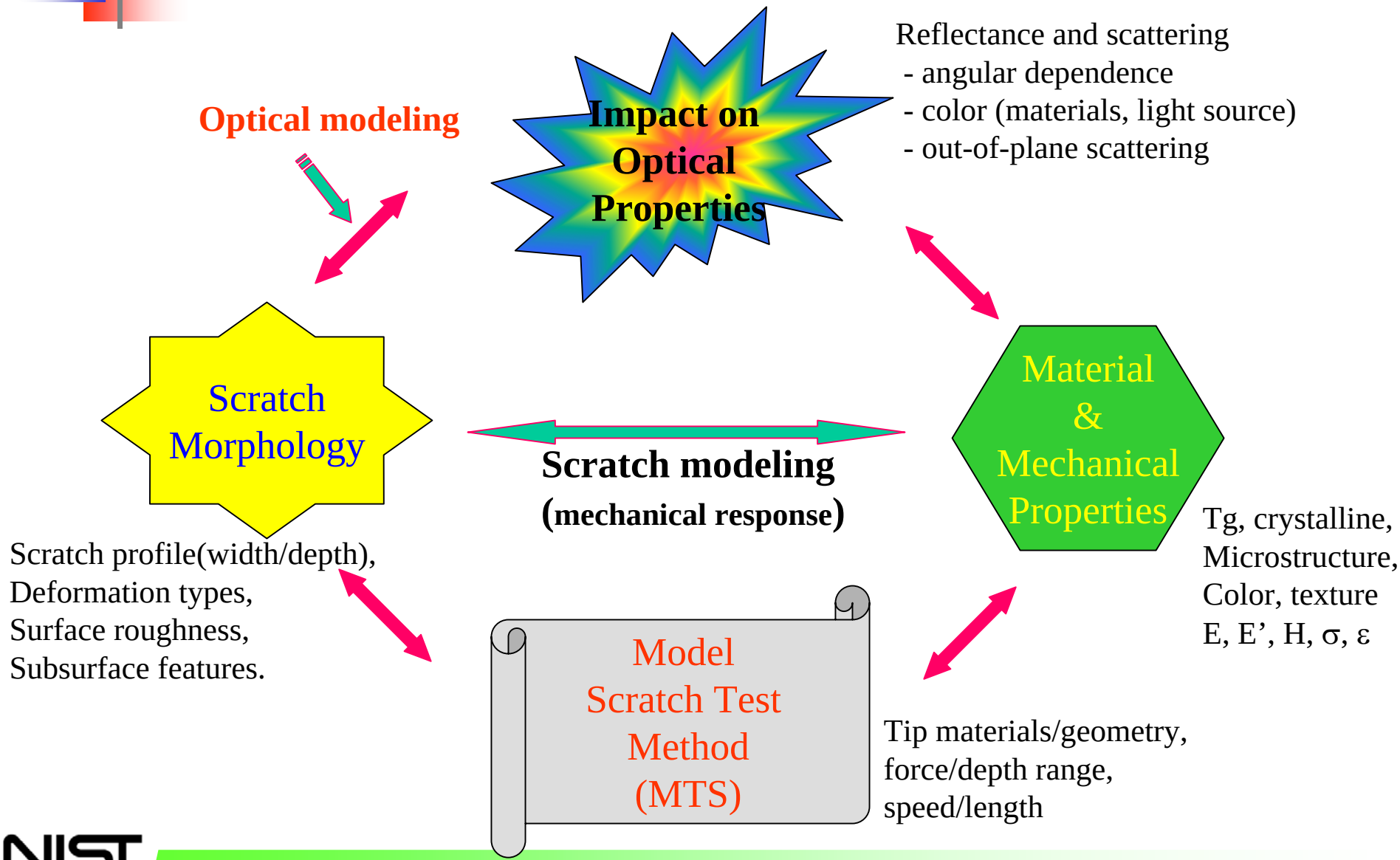




Reflectance measurement

- various scattering angles
- specular, off-specular measurements
- in-plane and out-of-plane measurements
- polarization measurements for detecting craze and other surface deformation

Technical Approach – NIST



Technical Approach

- **Develop a controlled scratch test method using nanointentor – with known strain, calibrated tip geometry, test conditions, etc.**
- **Characterize mechanical responses and relate them to surface deformation (scratch & mar) using confocal microscopy and AFM.**
- **Measure optical reflectance: at various angles, specular & off-specular measurements, out-of-plane measurements.**
- **Develop optical models to link optical scattering measurements to surface deformation (scratch & mar) – provide a foundation for measurement protocols for quantitative characterization of scratch.**
- **Combine scratch models developed in Phase I (by ENISE) and optical modeling to relate "surface mechanical properties" to "scratch & mar resistance" and then to "appearance" of scratch.**
- **Apply the methodology and measurement protocols developed to scratch and surface damage of industrial materials.**

Major Milestones

1. **Complete development of a controlled scratch test method using nano-indenter, and characterize mechanical response and surface deformation.**

2. **Complete development of protocol for measuring optical properties of polymeric materials after scratching.**

3. **Complete assessment of appearance attributes and relate materials mechanical properties to morphological deformation & optical properties of scratches and mars.**

4. **Propose a standardized test method and measurement protocols for assessing scratch and mar resistance of polymeric materials.**

(Milestones 1 to 3 will be conducted in parallel with model materials in Year 1 and progressively toward complex industrial materials in Year 3.)

Impact

Significantly advance in standardization of test methods and measurement protocols for assessing scratch and mar resistance of polymeric materials and coatings.

- A successful development of reliable methods for assessing scratch and mar will greatly improve the commerce and reliability of products that depend on surface appearance performance.