

# paintistanbul TURKCOAT CONGRESS

## Novel Viscoelastic Measurement Techniques for Multilayer Coatings Utilizing Nano-Indenter

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Sustainability strategy dept.

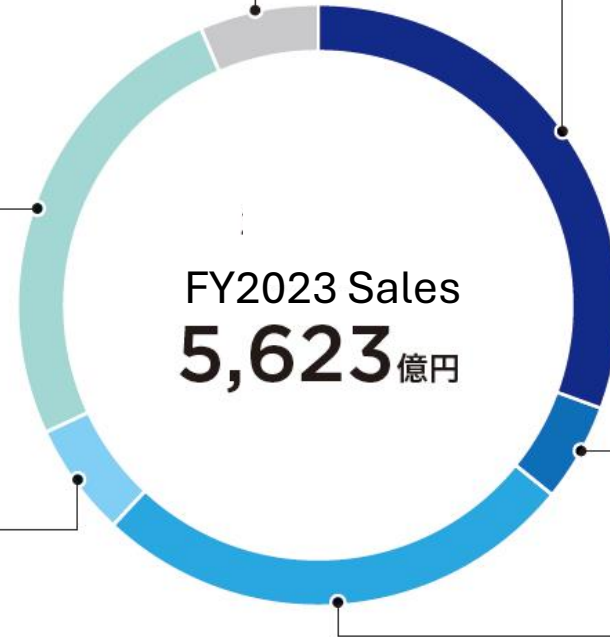
# Kansai Paint Market Share by Sector

The others  
**6.2%** (350億円)

Decorative  
**25.6%** (1,437億円)



Marine & Protective  
**6.0%** (335億円)



Automotive

**30.7%** (1,729億円)



Auto Refinish

**5.2%** (294億円)



Industrial

**26.3%** (1,478億円)

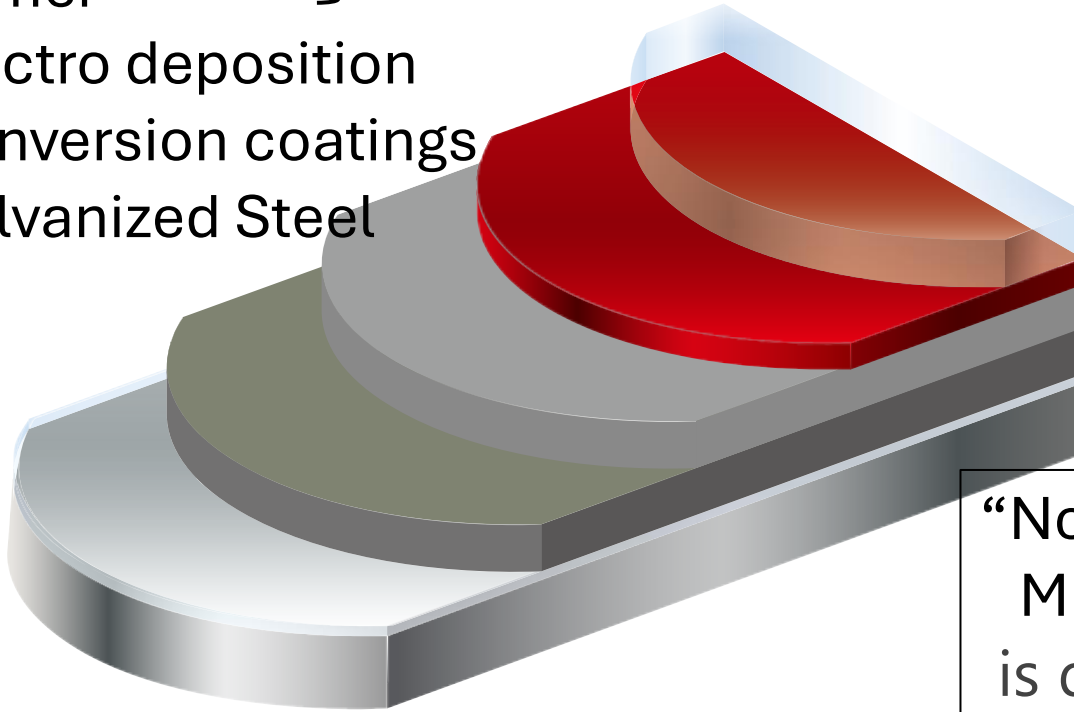


We offer various categories of coatings worldwide.  
Automotive coatings are one of our most important sector.

# Sustainable technology for automotive coatings

## Automotive Coatings

- Clear coat
  - Base coat
  - Primer
  - Electro deposition
  - Conversion coatings
  - Galvanized Steel
- Streamlined process  
3Coat 1Bake



## Eco-friendly Innovations

### -Low VOC

Water borne, High solid

### -High Efficiency Coatings

Low Temperature Cured, Thin layer

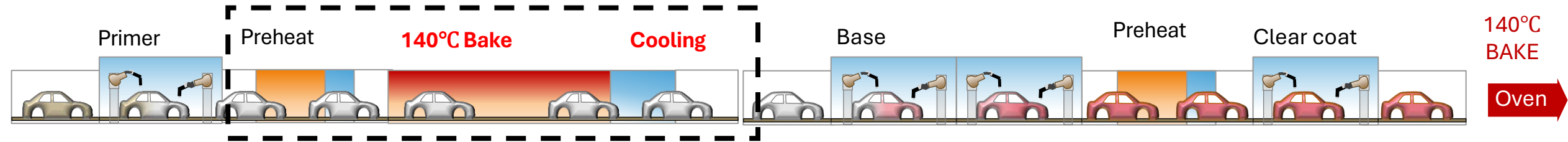
### -Streamlined Process

“Novel Viscoelastic Measurement Techniques for Multilayer Coatings Utilizing Nano-Indenter” is developed to evaluate these coating films.

# Streamlined Process for Automotive Coatings

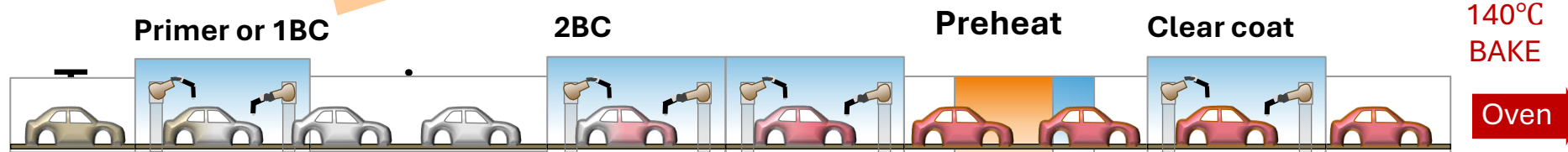
## Conventional Process

3Coat2Bake (150-200m)



## Streamlined Process

3Coat1Bake (120-150m)

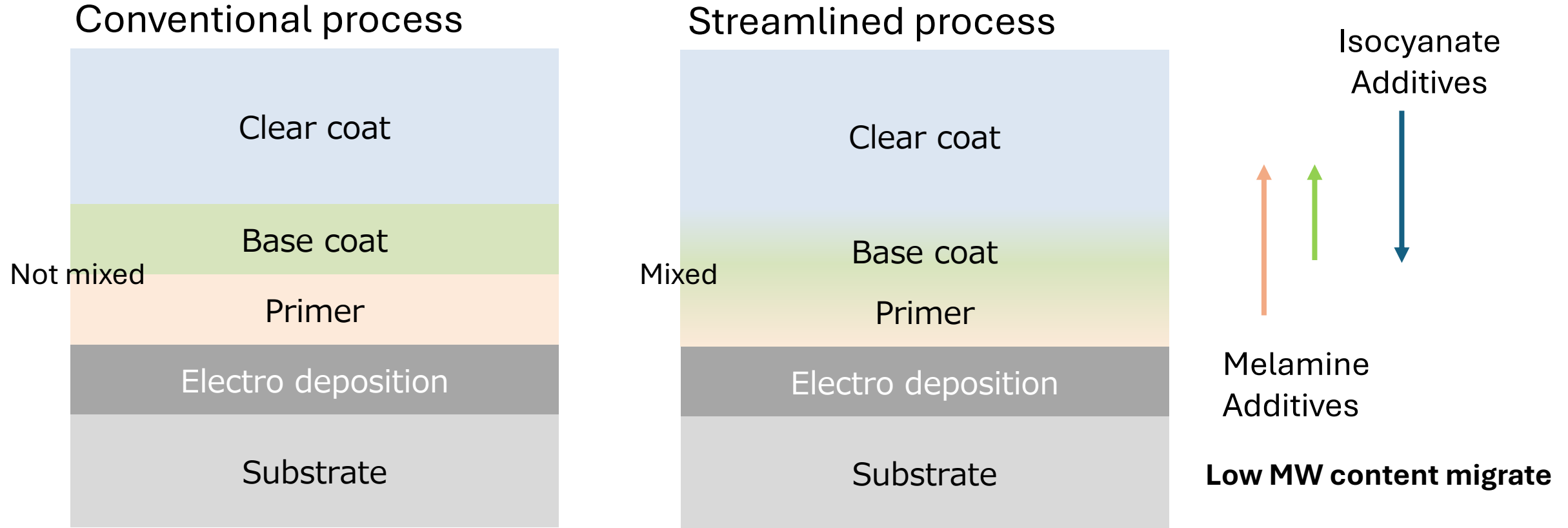


Shorten line length  
Low energy consumption



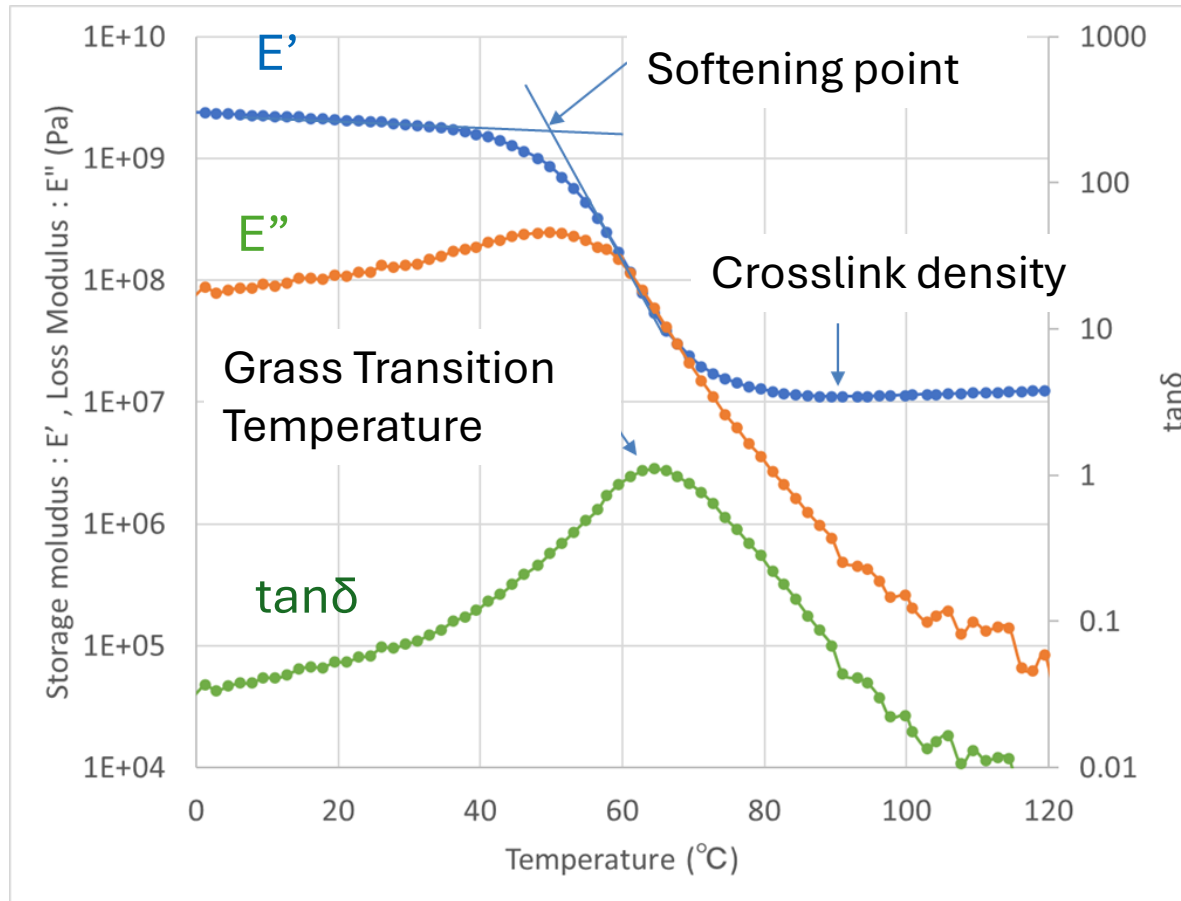
We faced challenges in evaluating physical properties during development.

# Layered Films by Streamlined Process

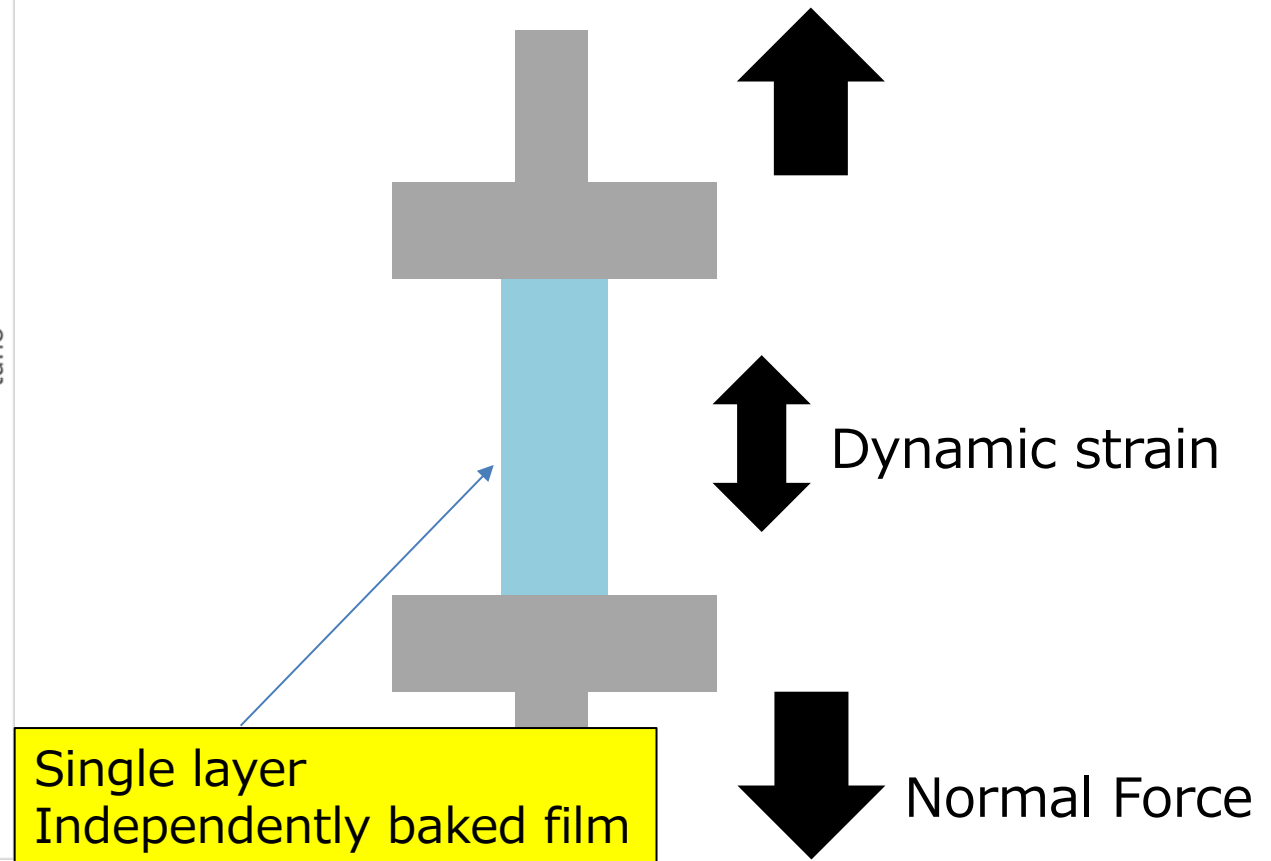


The layered films obtained by the streamlined process are quite different from independently baked films. Our new product are designed to utilize this migration.

# Viscoelastic Property of Coating Film



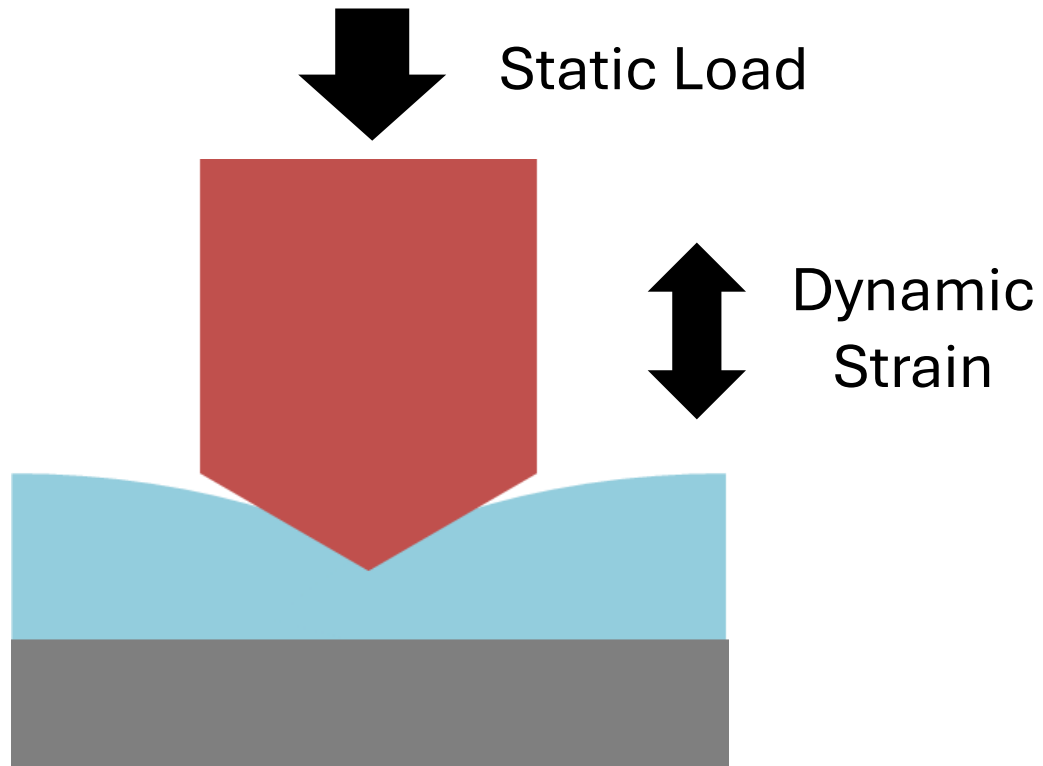
## DMA measurement of coating films



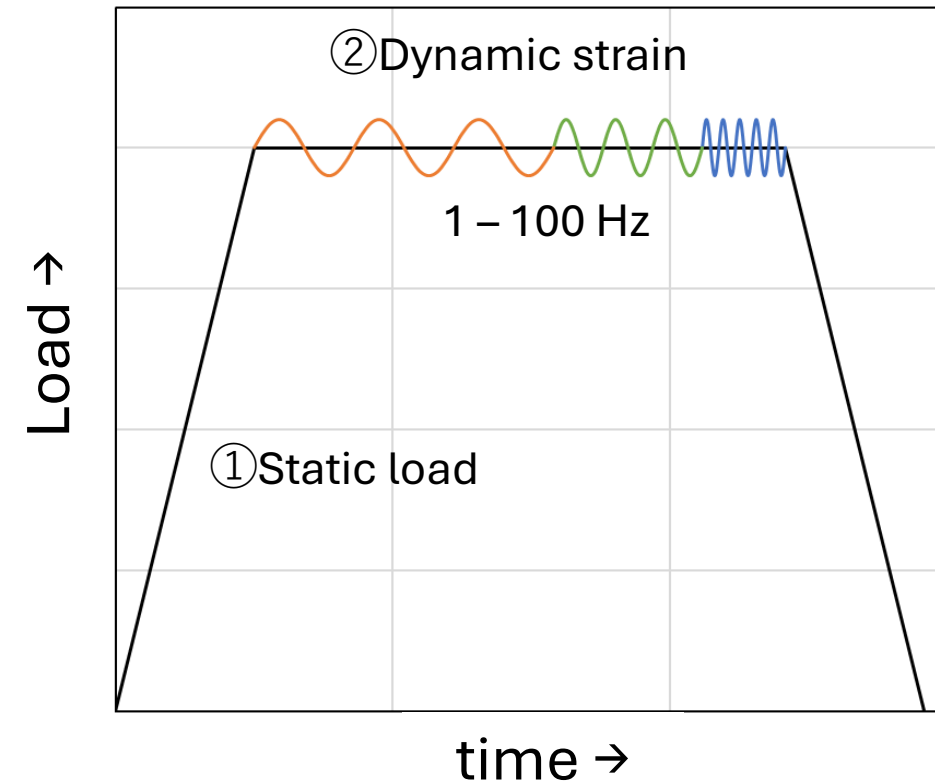
For our streamlined process, this method is becoming less meaningful.

# DMA Measurement by Nano-indenter

Schematic of Nanoindentation



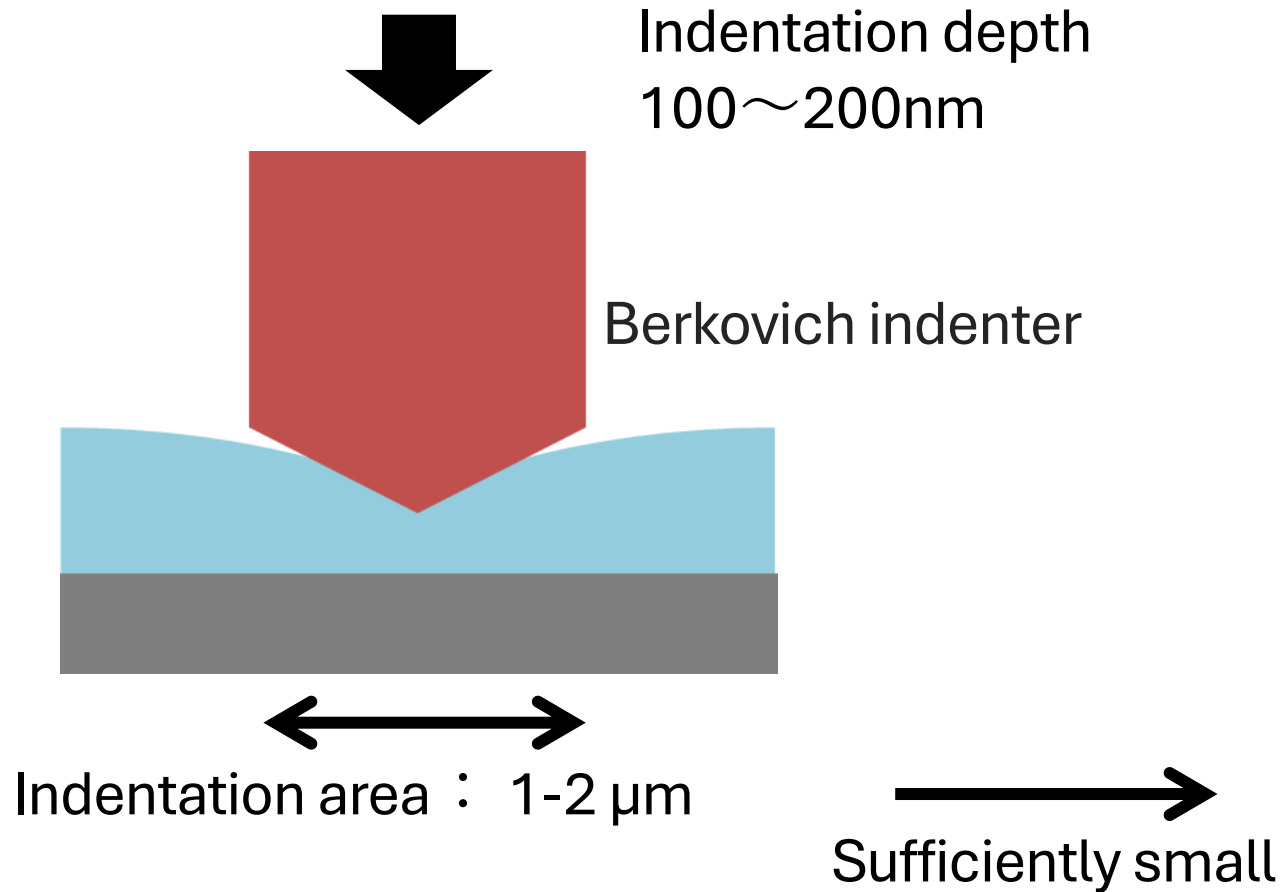
Load function



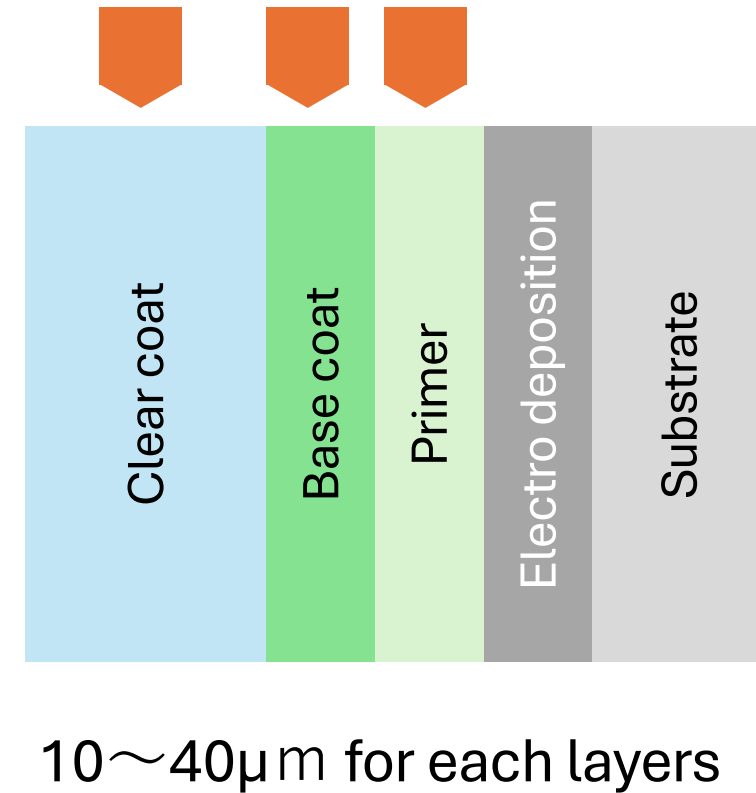
We can measure the viscoelastic properties of the coating panel without peeling off the substrate.

# Measurement of Underlayer from Cross-section

## Schematic of Nanoindentation Method



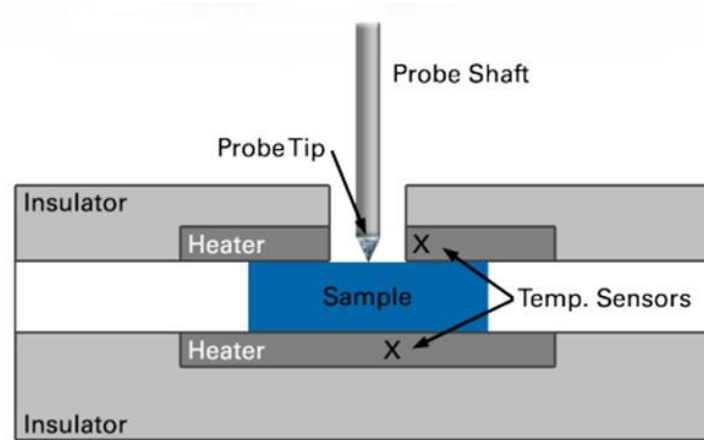
## Measurement from cross-section





# Experiment

## Equipment



Schematic of the xSol High Temperature Stage.

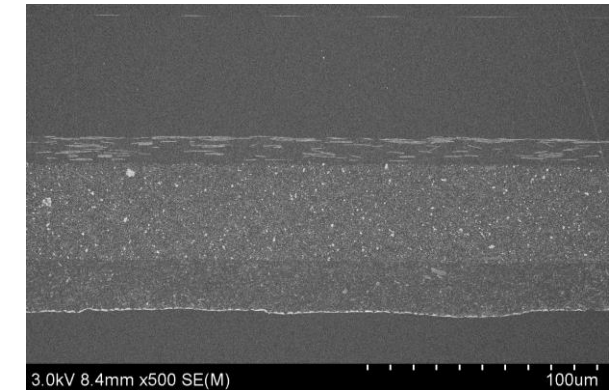
Bruker TI-premier  
with X-sol stage

## Sample

Multi-layer coating films  
for automotive

Preparing of cross-section

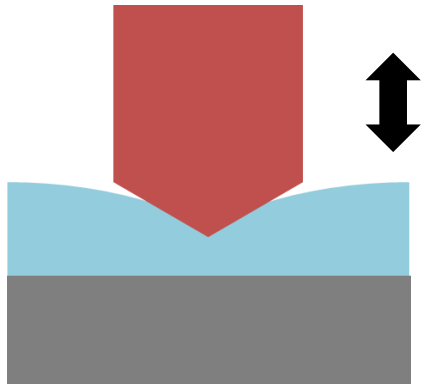
- Machine polishing
- Cross-section polisher



(SEM-image)

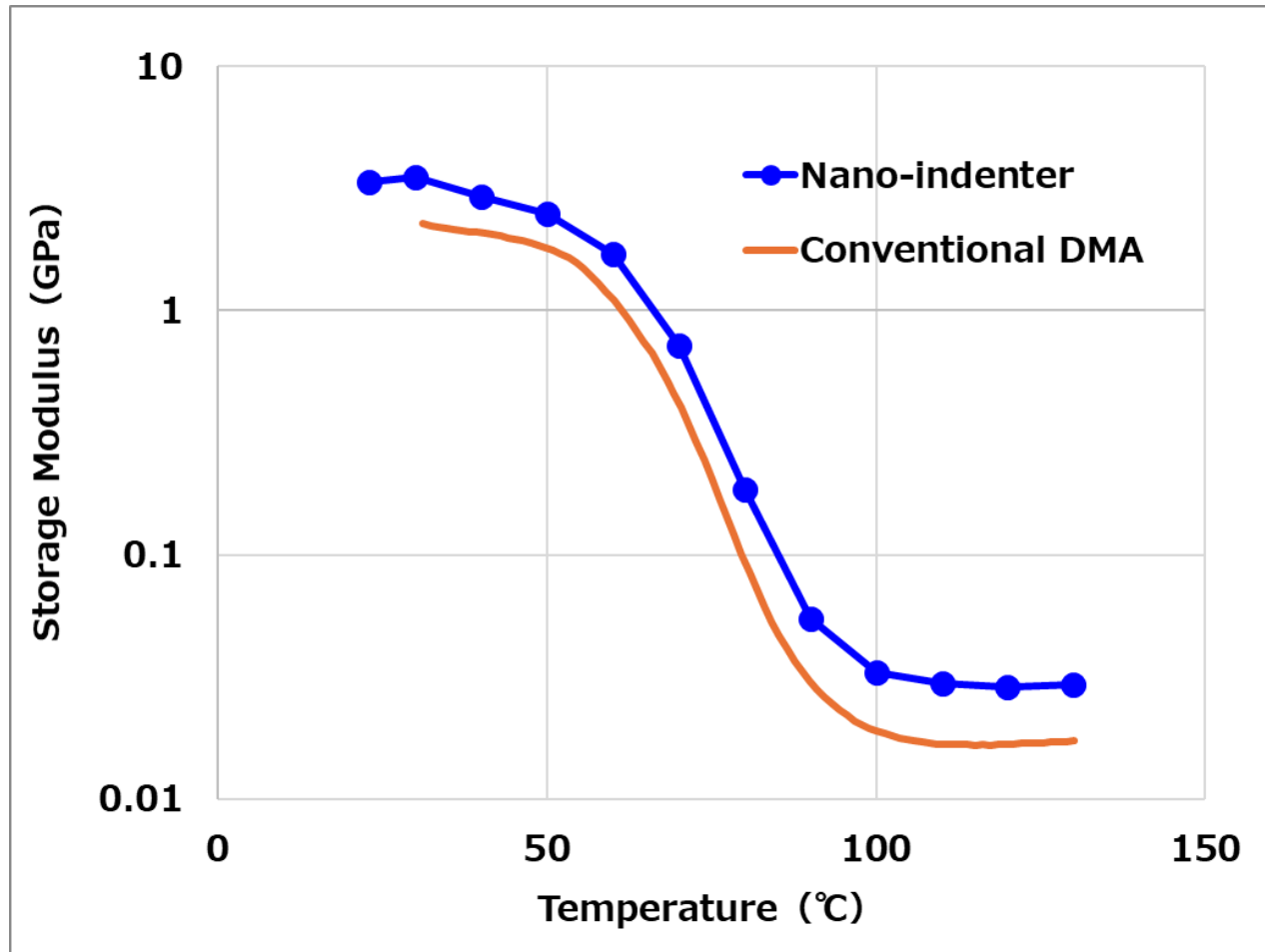
# The Comparison Between Nano-Indenter and Conventional DMA, E'

## Nano-indenter

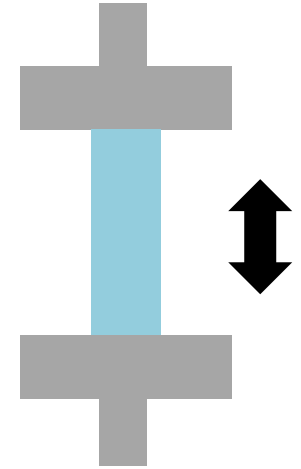


2-3hours

Frequency : 10Hz  
Temperature:  
23°C-130°C



## Conventional DMA

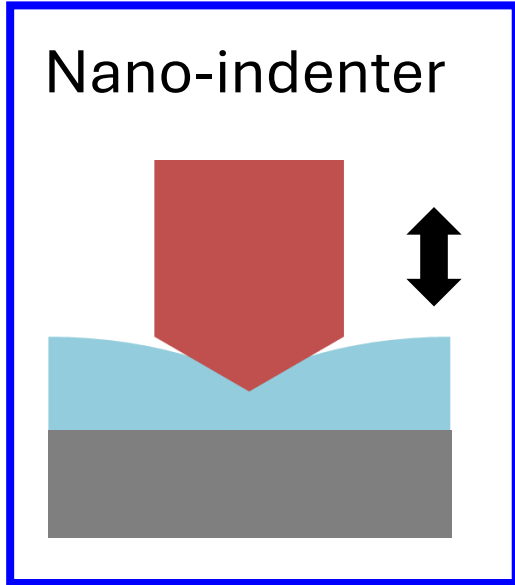


30-40minutes

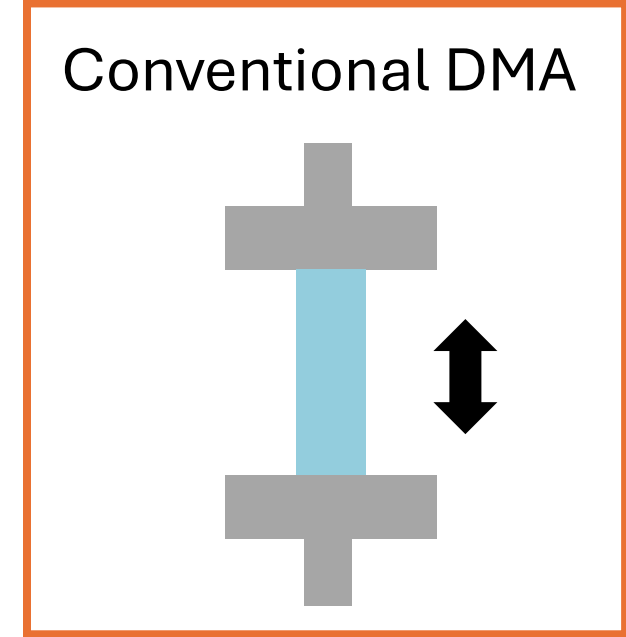
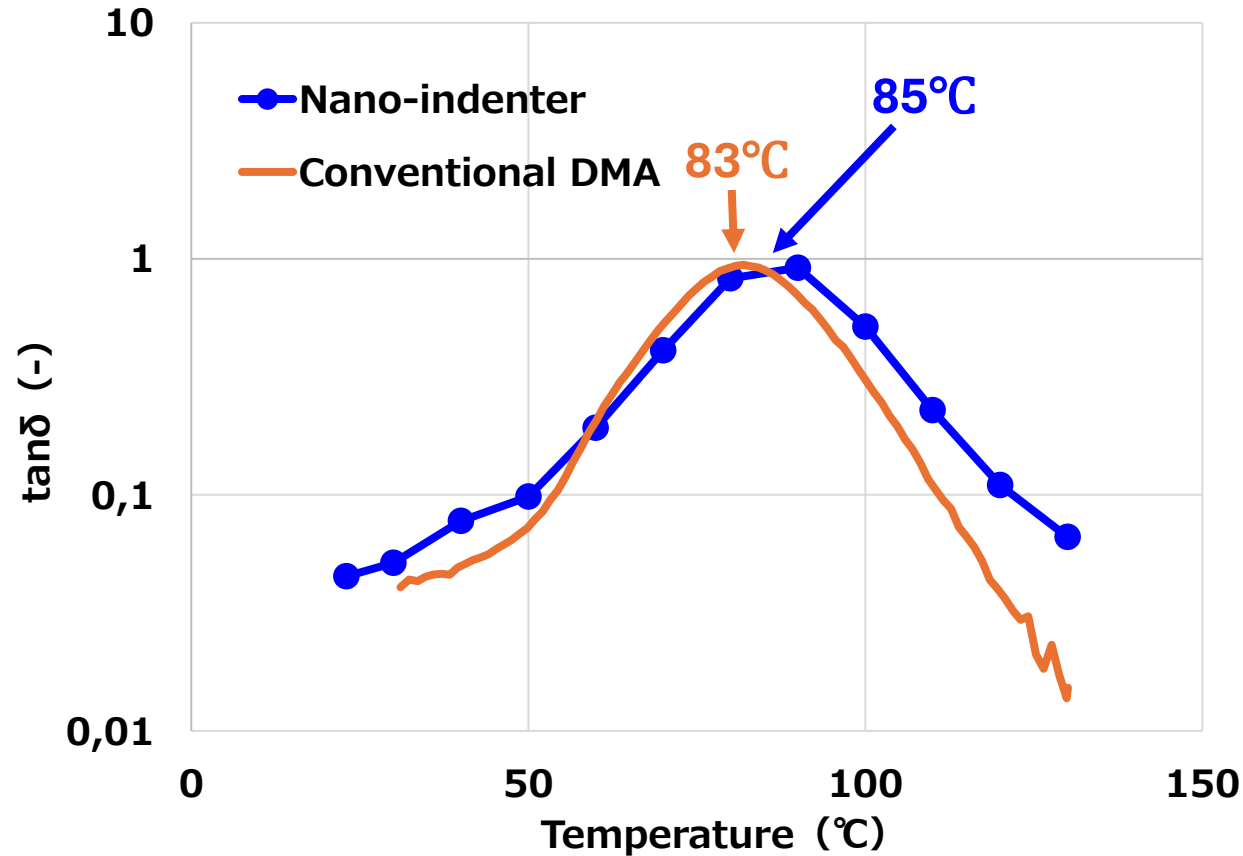
Frequency : 10Hz  
Temperature:  
40°C-130°C

# The Comparison Between Nano-Indenter and Conventional DMA, $\tan\delta$

## $\tan\delta$ of Clear Coat Film



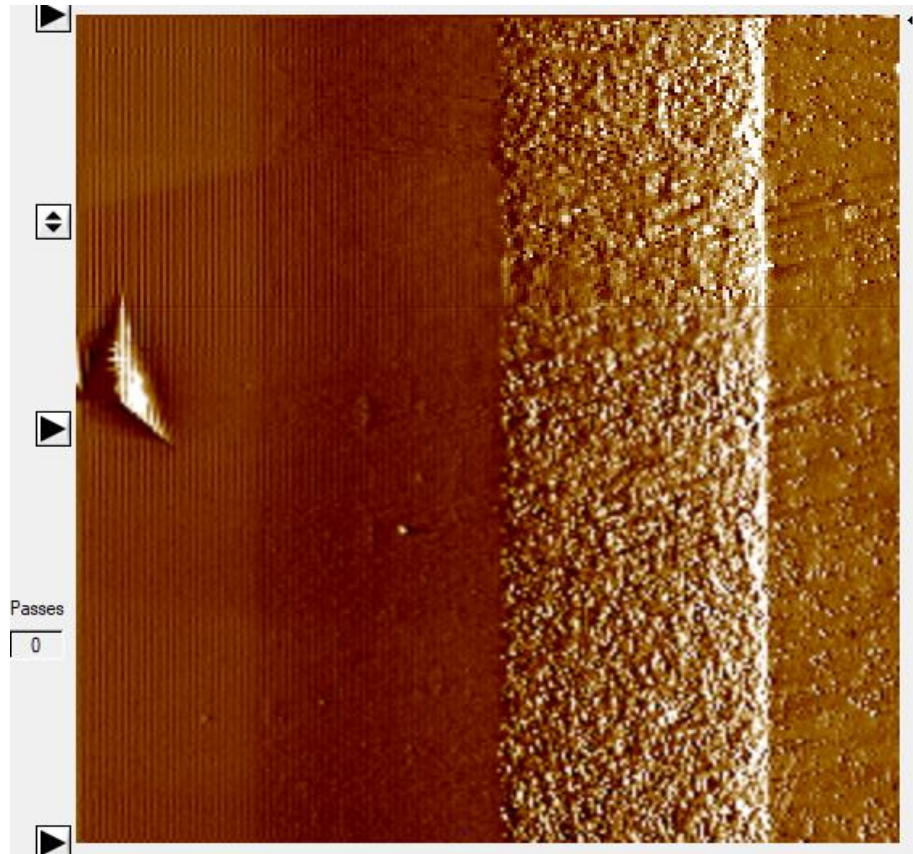
Frequency : 10Hz  
Temperature:  
23°C-130°C



Frequency : 10Hz  
Temperature:  
23°C-130°C

# DMA of Multi-Layer Film : SPM Images of Cross Section

Streamlined Process (3Coat1Bake)



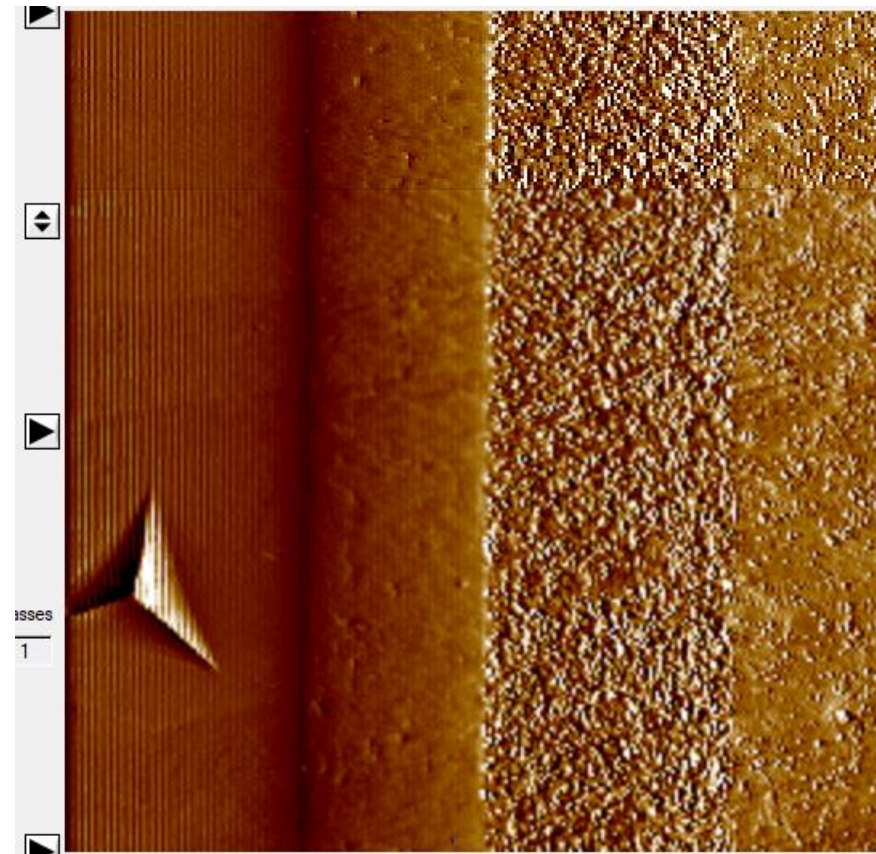
CC

BC

Pr

ED

Conventional Process (3Coat3Bake)



CC

BC

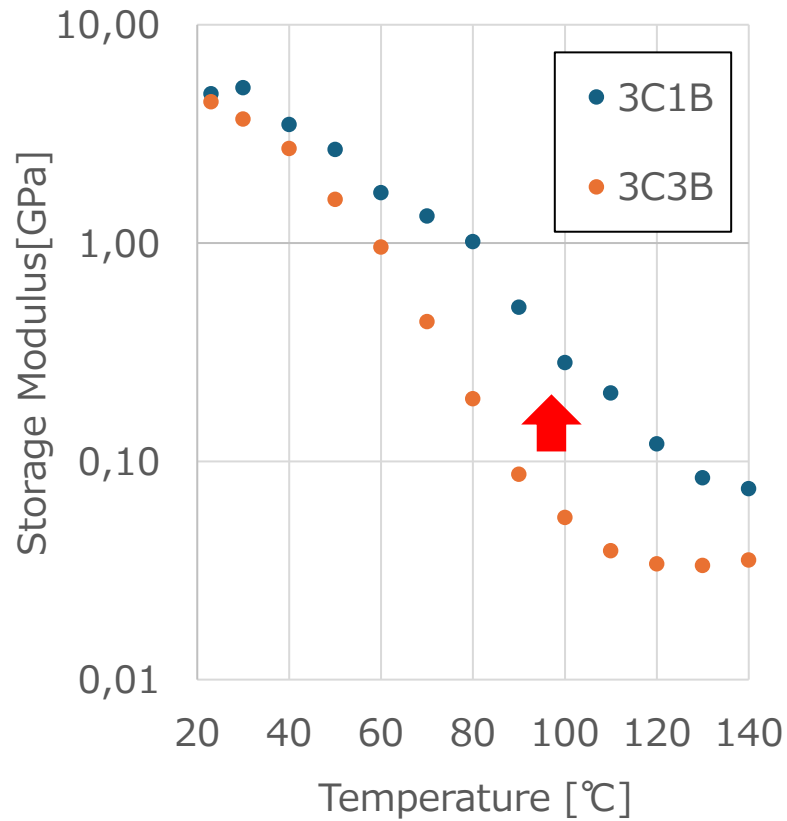
Pr

ED



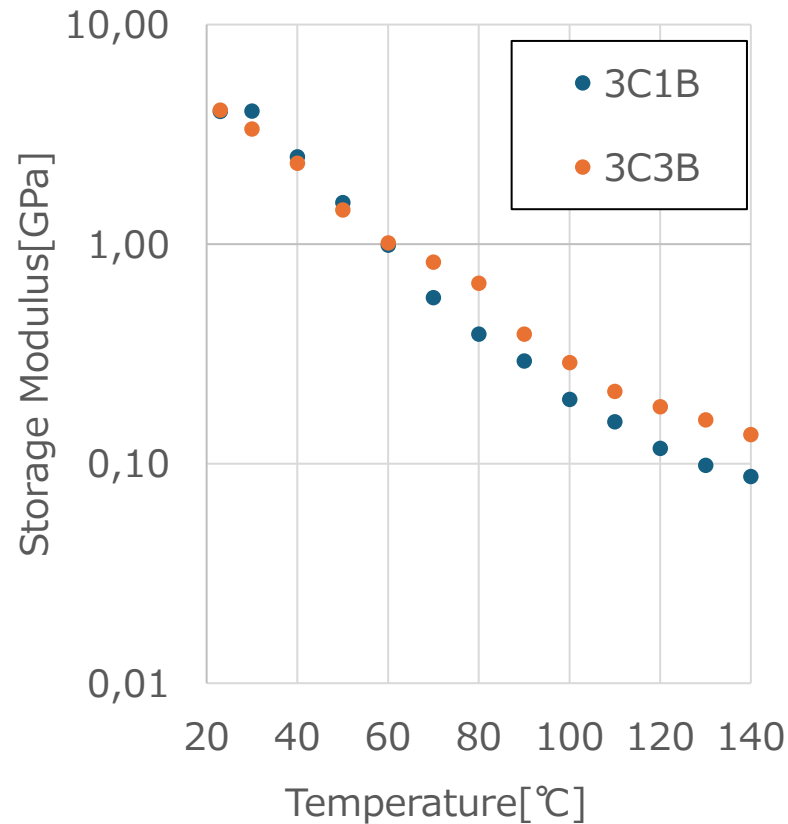
# DMA of Multi-Layer Film : Storage Modulus

## Clear Coat (1K)

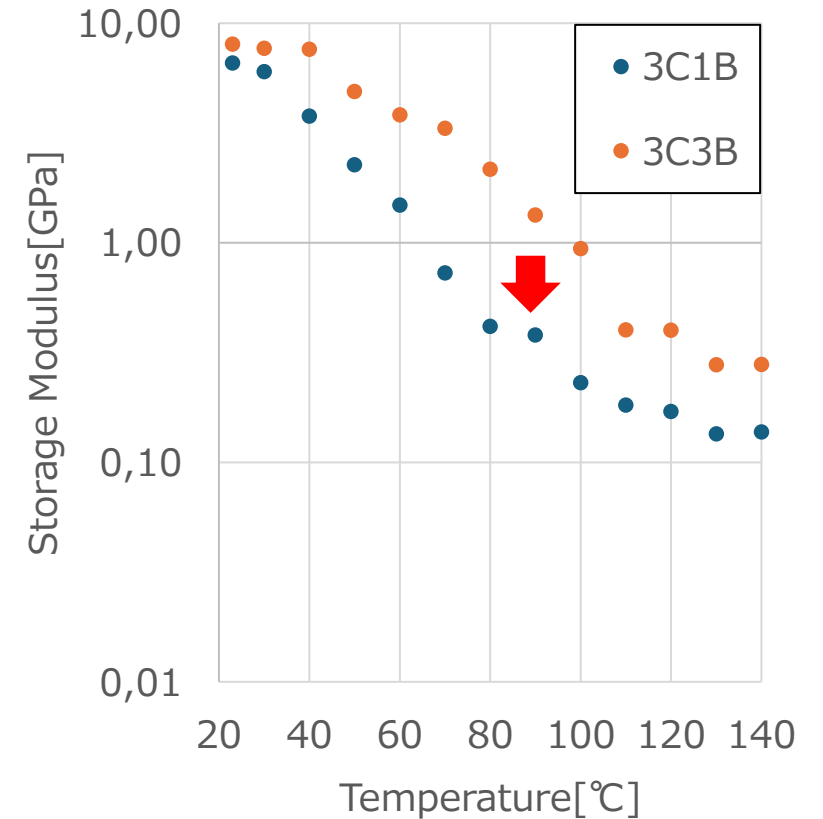


**Harder than 3C3B Process**

## Base Coat



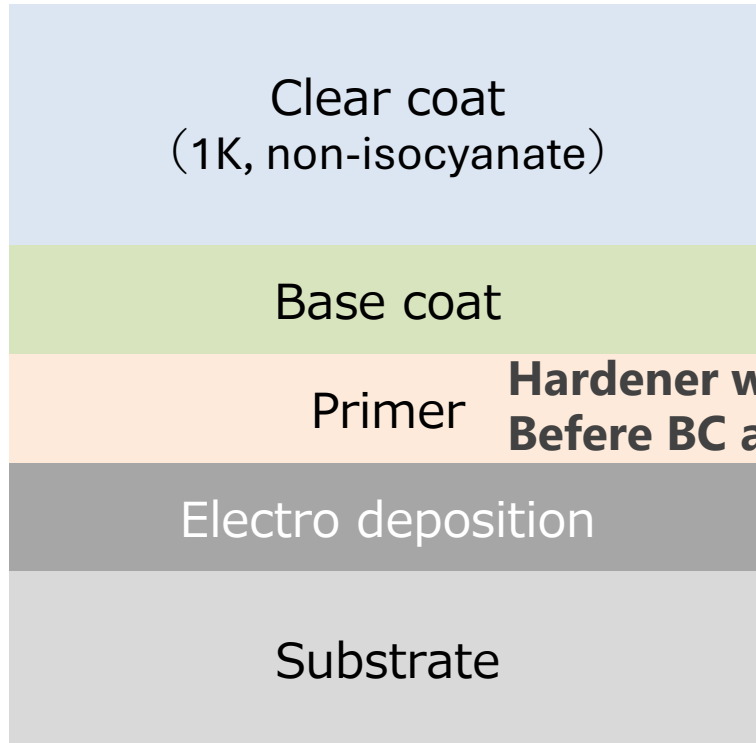
## Primer



**Softer than 3C3B Process**

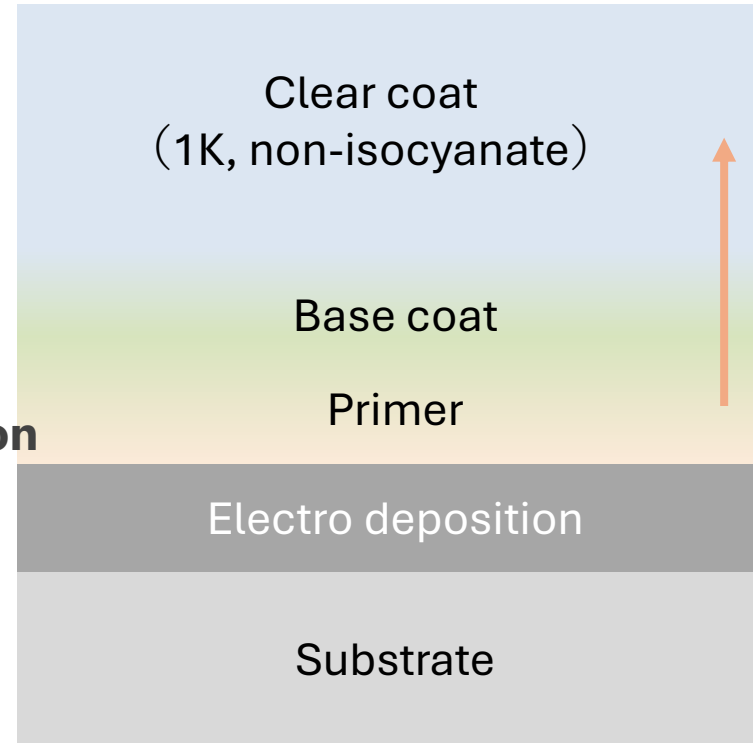
# DMA of Multi-Layer Film : Migration of Melamine

Original process



**Hardener was fixed  
Before BC apprication**

Streamlined process



**Hardener content increase  
→ Film becomes harder**

Melamine

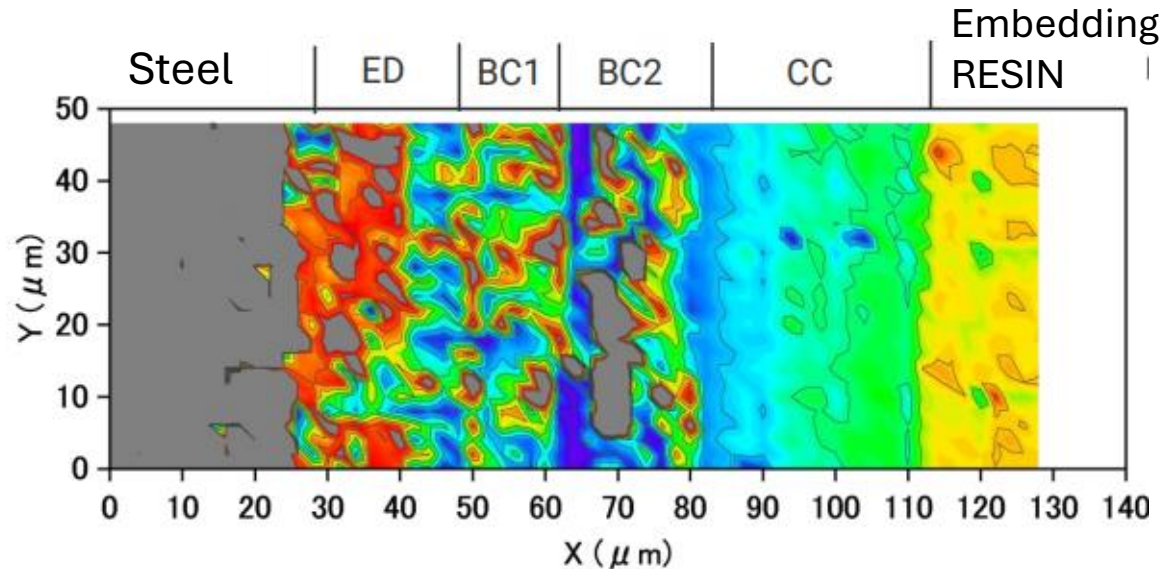
**Hardener content decrease  
→ Film becomes softer**

Streaming-lined process should be designed with consideration of these facts.

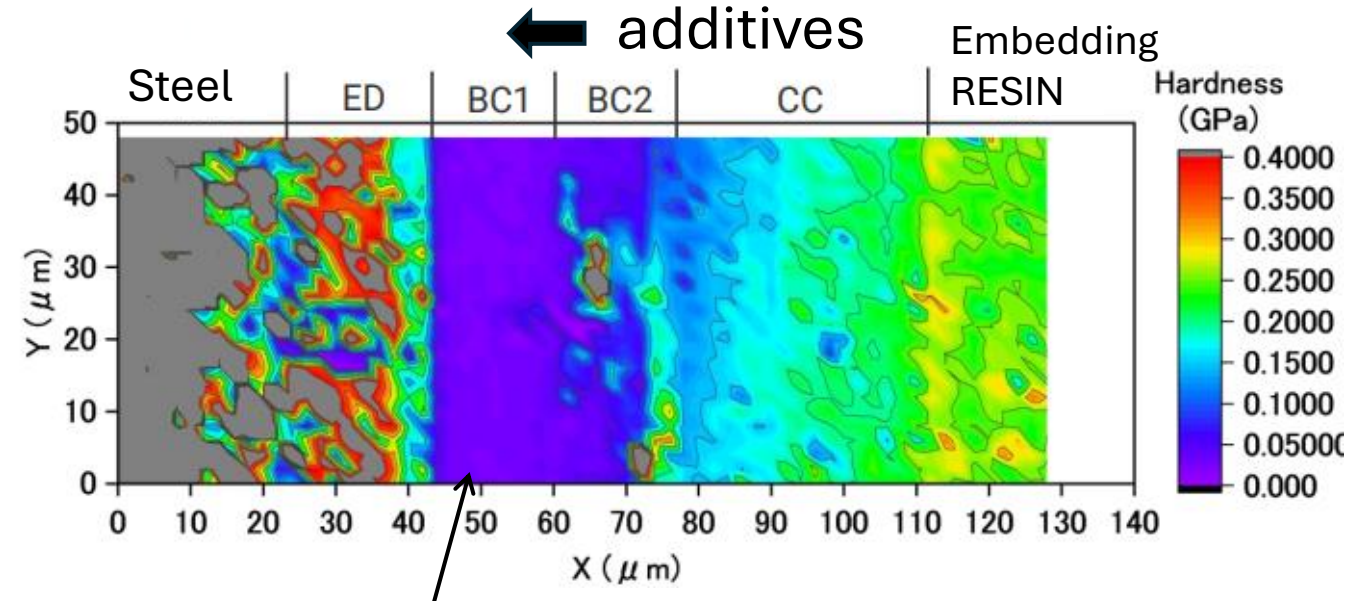
# Analysis for Adhesion Mechanism

## Hardness mapping with XPM

### Good Adhesion Sample



### Poor Adhesion Sample



Peeling off from this layer

Additives present in BC2 inhibited the curing reaction.

This analysis also could not have been achieved without this new method.

# Summary

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Novel viscoelastic measurement techniques using a nano-indenter

Advantage Over Conventional Method:

- DMA Measurement without removing from the substrate
- Analysis of under layers in multilayer coatings from the cross-section.

Applications :

- Streamlined Processes : migration of hardener was suggested
- Adhesion Analysis : Prevention of curing by additives was identified

This method provides a lot of valuable information for development of coatings and will be widely used in the paint industry in the future.