Insights by Combined Electrical and Optical Characterization of Large Area Graphene

GRAPHENE 2018

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Company – SURAGUS GmbH

SURface ArGUS = Surface guard

Technology

HF Eddy current-based testing solutions for innovative materials

Location and Presence

R&D and production in Germany, Dresden, near Airport and A14

EddyCus systems are present on six continents

Ownership

German privately owned company

Applications

Quality assurance of functional thin-films

Values

Accurate and reliable solutions

Smart solutions (inline reverse calibration, automated self-reference, temperature stabilized)

High technical flexibility (various gap sizes, different sensor setups, traverse and fixed)

Excellent service (Close contact / short response times)

Challenges the industry is facing from an commercial/industrial application point of view

The key challenge is finding an application where Graphene can achieve a superior and competitive set of characteristics
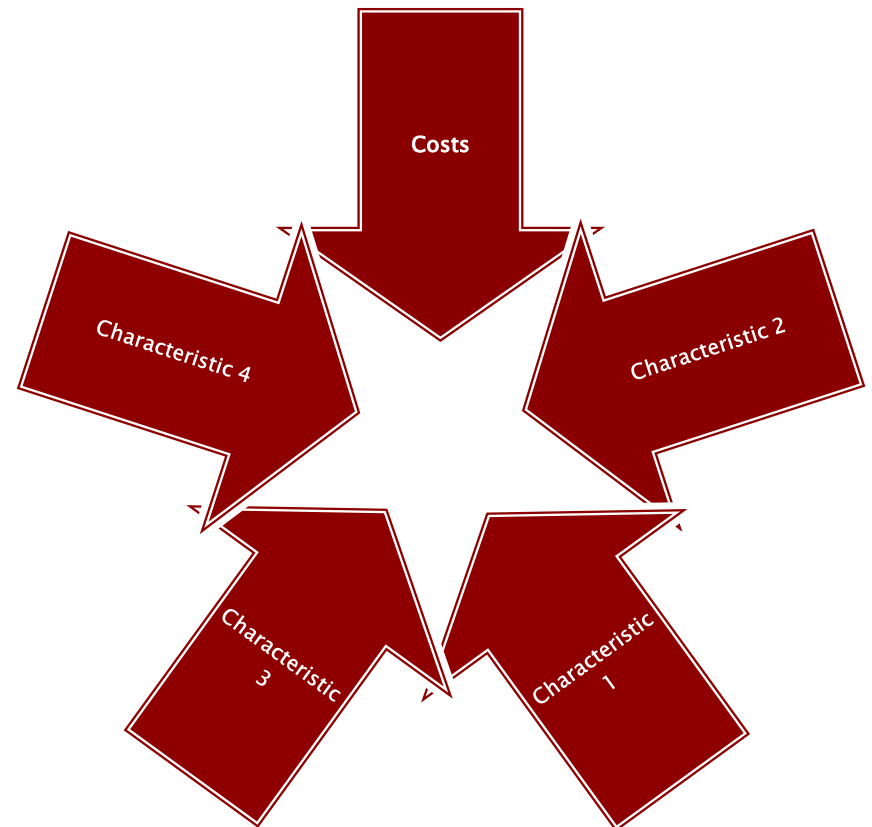
Physical Characteristics

- Electrical properties
- Mechanical properties
- Chemical properties
- Optical properties
- Medical / biological etc.

Soft characteristics

- Ability for flexible substrates
- Stable over time

Financial Characteristics

- Cost per performance

Costs

Characteristic 4

Characteristic 2

Characteristic 3

Characteristic 1

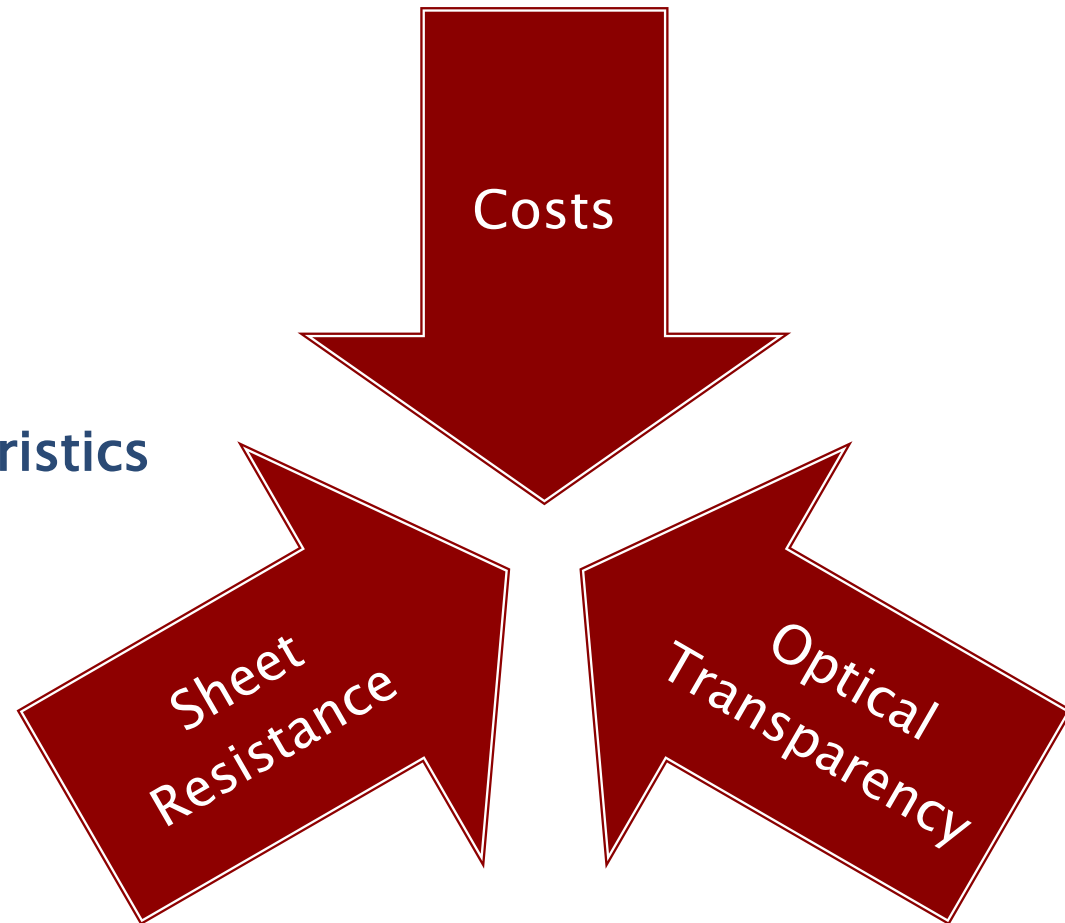# Challenges the industry is facing from an commercial/industrial application point of view

Example: Graphene as transparent electrode

▸ **Requirements/ trends**
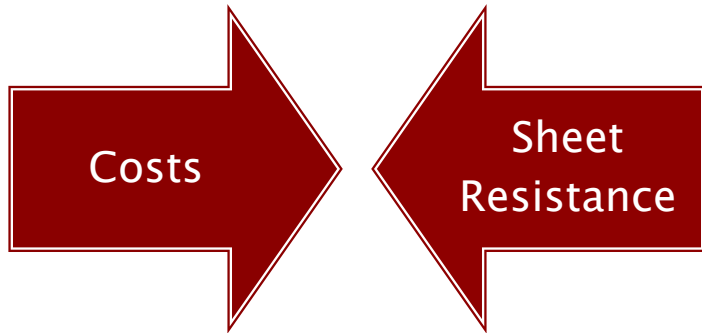- Low cost
- Low sheet resistance
- High transparency

▸ **Beneficial/ further characteristics**
- Ability for flexible substrates
- Low aging effects

Costs

Sheet Resistance

Optical Transparency

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Sensors & Instruments

Drivers & Challenges in Manufacturing

Costs → ← Sheet Resistance

High conductivity

Metal mesh

Silver NW

Graphene

ITO

CNT

Conductive polymer

Low conductivity

Source: Touch Display Research ITO-replacement 2016 report

Low cost   High cost

Cost improvement

- Larger substrate sizes / gapless production/R2R
- Higher throughput due to higher deposition/growth rate
- Cheaper processes: atmospheric pressure, low temperature
- More automation / continuous processes?
- Optimized material input eg. reuse of growth substrates

Graphene as transparent electrode
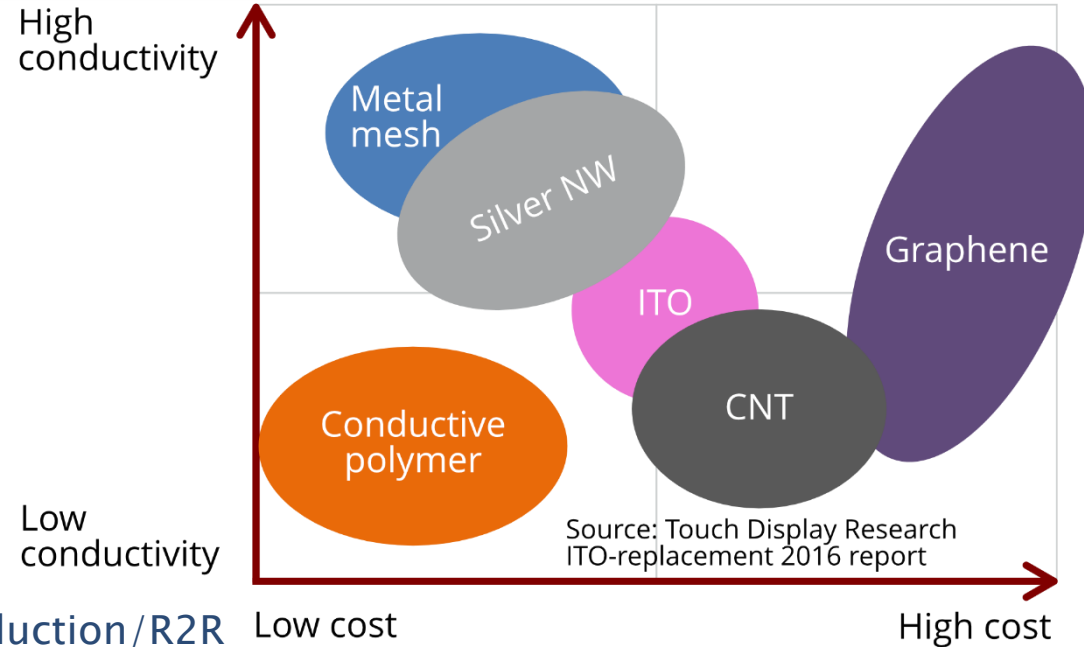
Optical Transparency ⟶ ⟵ **Sheet Resistance**
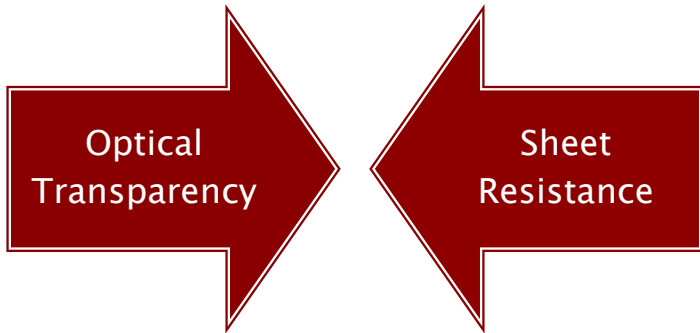
Main Parameter

- Low boundary rate / monocrystallin
- Physical integrity
- Doping
- Annealing
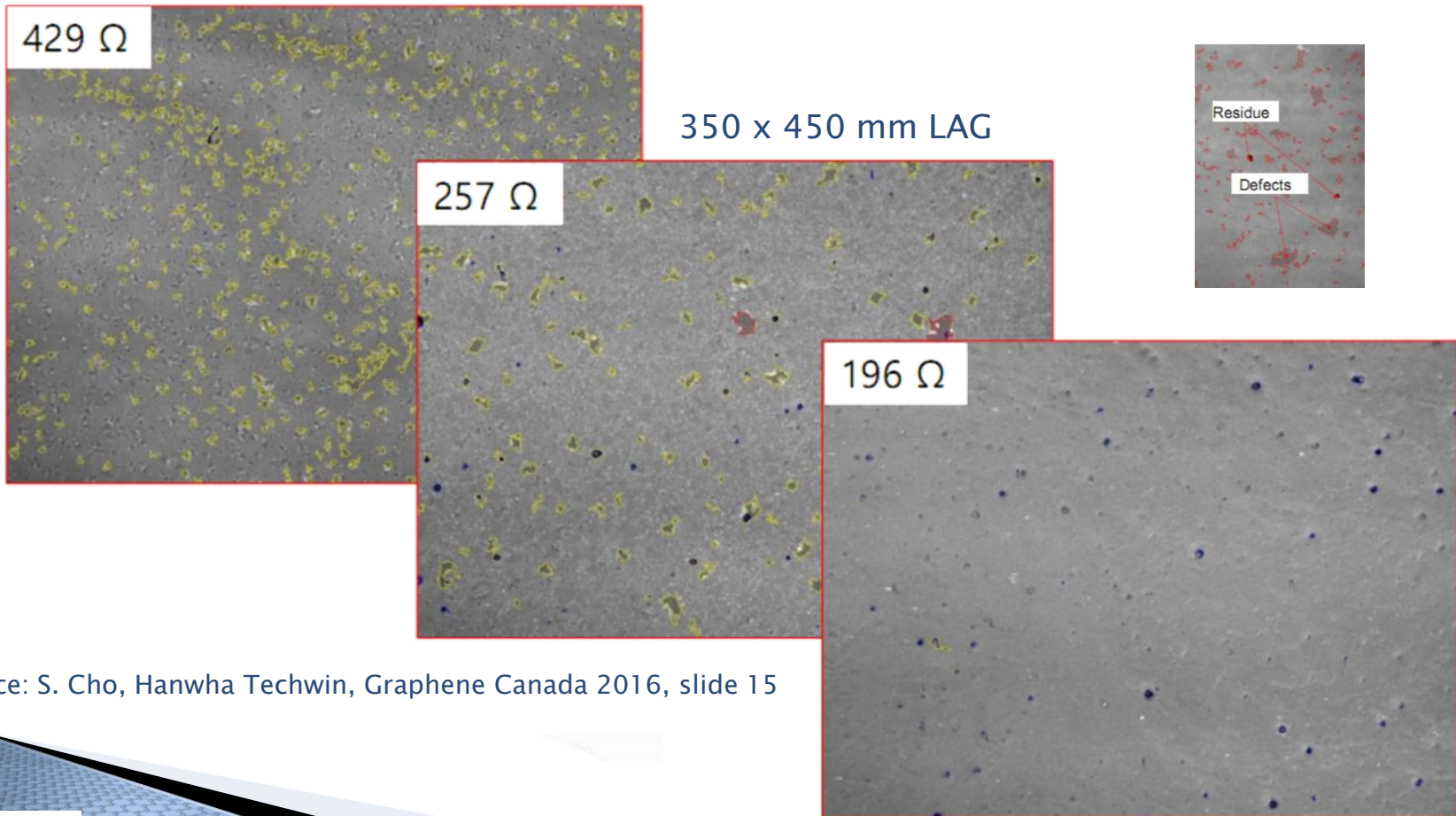- Stacking, patching
- **Defect-freeness**
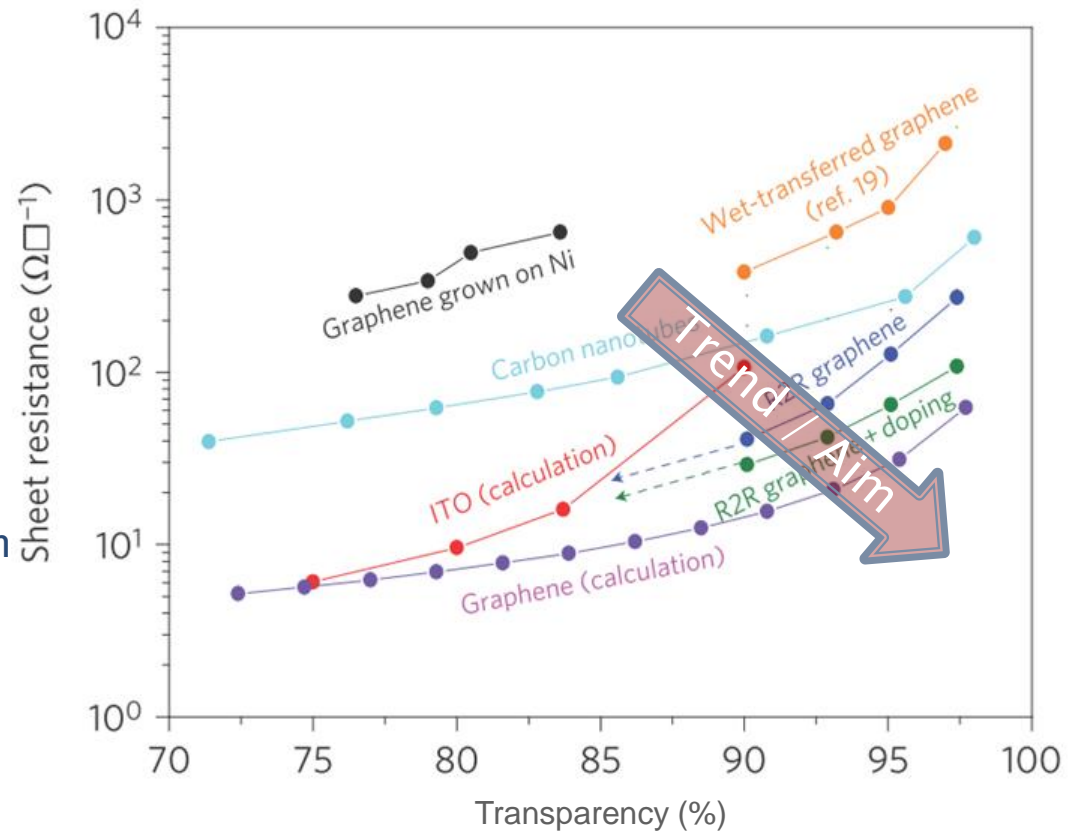
S. Bae et al. Nature Nano. 5, 571 (2010);
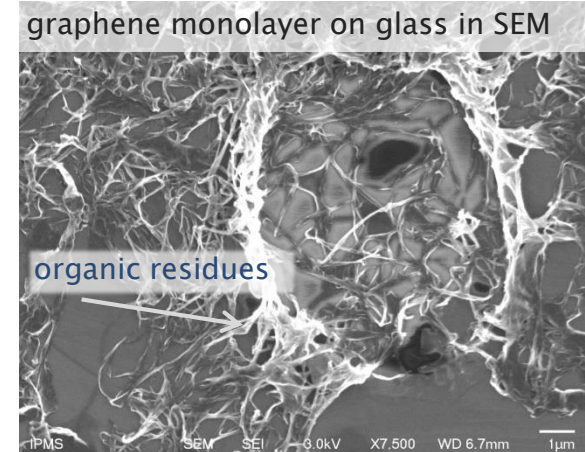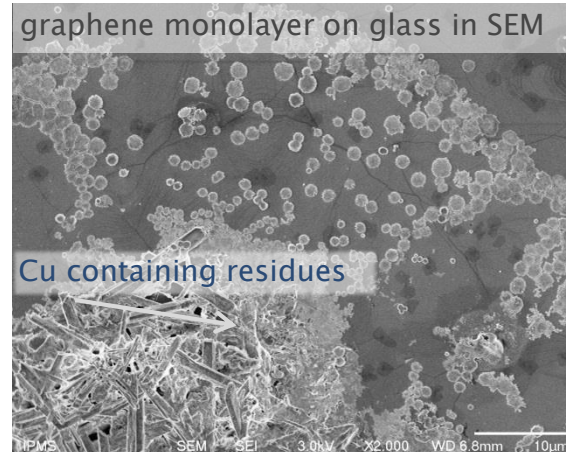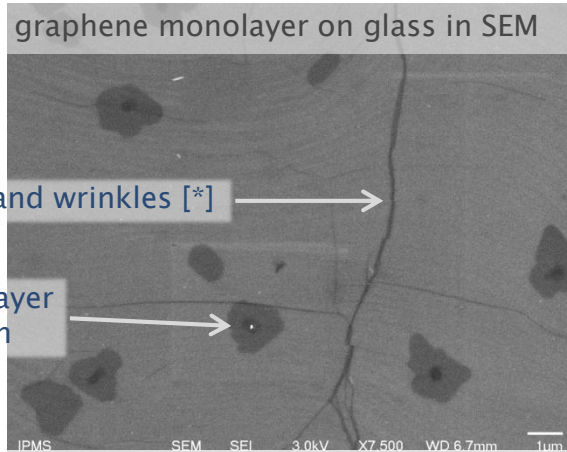
Effects of Defects to the Sheet Resistance

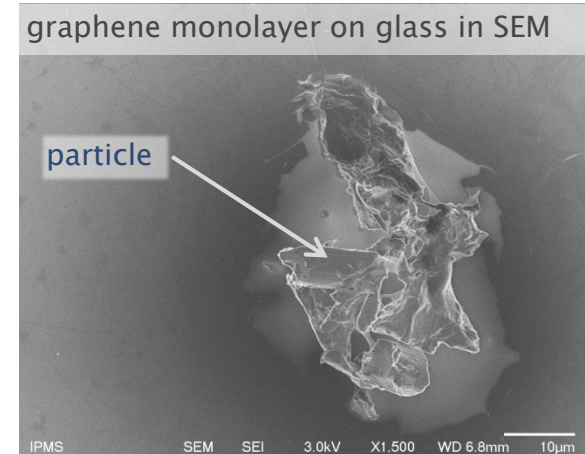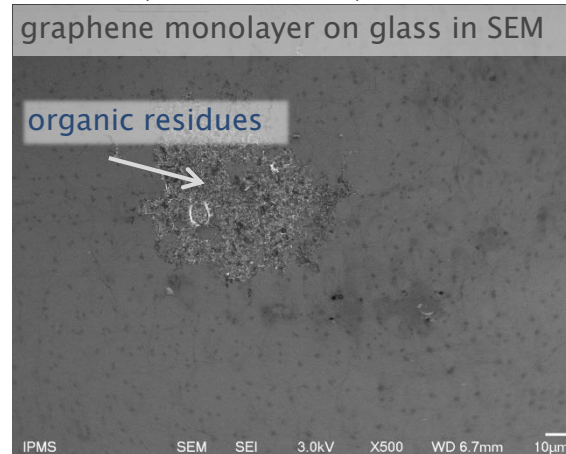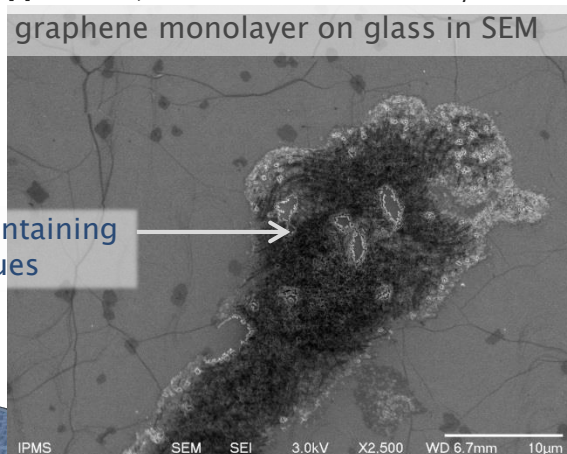- The defect density significantly affects the sheet resistance

429 Ω

350 x 450 mm LAG

257 Ω

196 Ω

Residue

Defects

Source: S. Cho, Hanwha Techwin, Graphene Canada 2016, slide 15

Typical Defects in Graphene

graphene monolayer on glass in SEM

folds and wrinkles [*]

multilayer growth

[*] Zhu et al., *Structure and Electronic Transport in Graphene Wrinkles*, Nature Nanoletters, 2012

graphene monolayer on glass in SEM

Cu containing residues

graphene monolayer on glass in SEM

organic residues

graphene monolayer on glass in SEM

Cu containing residues

graphene monolayer on glass in SEM

organic residues

graphene monolayer on glass in SEM

particle

Source: D. Waynolds et al., Fraunhofer FEP

Quality Characteristics of Graphene as TCM

Thickness/ # of layers	Defect freeness	Sheet Resistance	Optical transparency	Robustness
▪ Homogeneity	▪ Cracks/ gaps, tearing ▪ Holes Folds/wrinkles ▪ Impurities (before and after doping) ▪ Point defects, vacancies, rotated bonds ▪ Dopant atoms ▪ Contaminant particles from catalysts and CVD process ▪ Missing Interlayer conductivity ▪ Not connected flakes ▪ Too low overlap after patching ▪ Multilayer regions	▪ Quantification in a range of 10 Ohm/sq to 3000 Ohm/sq ▪ Homogeneity ▪ How to define it	▪ Optical transparency from UV up to IR wave lengths ▪ Quantification ▪ Range: Transmittance from 80 – 97.7% or absorbance ▪ Homogeneity ▪ How to define it ▪ Other optical parameters such as haze	▪ Stability/Aging ▪ Ability of flexible substrates ▪ Heat resistance

- ▪ How to define homogeneity and defect density?

- ▪ By result / resistance for TCM?

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Sensors & Instruments

Metrology for Graphene

Commonly applied testing technology

- Raman

- TEM and SEM

- High magnification optical microscopes

- Optical spectrometer (reflection and transmission)

- 4PP

- Eddy Current

- Van der Pauw

- Terahertzspectroscopy

- Mechanical testing

- Others

Quality determination of Graphene as transparent electrode

- **Sheet resistance**

  - 44P

  - Van der Pauw

  - Eddy Current

- **Optical transparency**

  - Optical transmission measurement

} Non-contact

Electrical testing of Graphene

4-point-probe testing

- Contact quality artifacts

- Possible damage to sensitive layers

- No measurement of encapsulated films

- Wearing of probe with time

Non-contact eddy current testing

- No influence of contact quality

- No harm or artifacts to sensitive films

- Measurement of encapsulated films

- Very fast measurement

Sample Number	4PP-measurement				Eddy Current			
	Mean [ohm/sq]	Standard deviation [ohm/sq]	Min [ohm/sq]	Max [ohm/sq]	Mean [ohm/sq]	Standard deviation [ohm/sq]	Min [ohm/sq]	Max [ohm/sq]
1	0.496	0.052	0.467	0.64	0.485	0.0002	0.4842	0.4847
2	1.120	0.022	1.079	1.16	1.120	0.0001	1.1203	1.1206
3	1.759	0.032	1.720	1.81	1.772	0.0002	1.7715	1.7721
4	4.430	0.100	4.300	4.61	4.425	0.0006	4.4244	4.4263
5	11.840	0.200	11.350	12.09	11.622	0.0102	11.6055	11.6421
6	30.400	0.500	29.800	31.30	30.498	0.0241	30.4544	30.5360
7	82.500	0.700	81.500	83.40	81.359	0.1145	81.2294	81.4972

Correlation EddyCurrent vs. 4 Point Probe

$y = 0.987x + 0.0649$

Sheet resistance EddyCurrent measureemnt [Ohm/Sq]

Sheet resistance 4PP measurement [Ohm/Sq]

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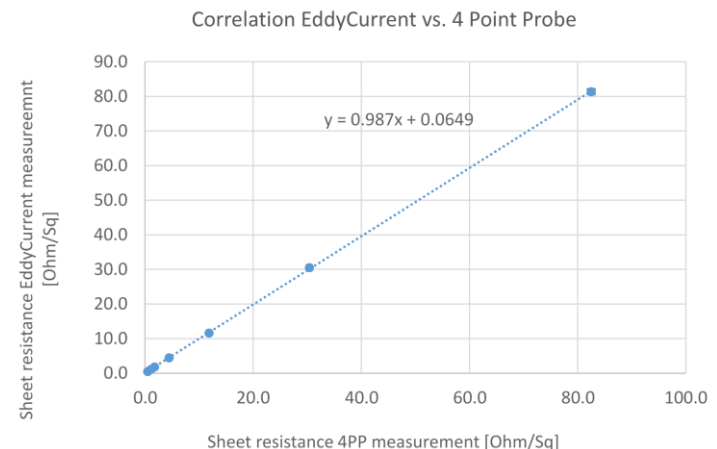
Sheet resistance measurement by Eddy Current

Primary Field
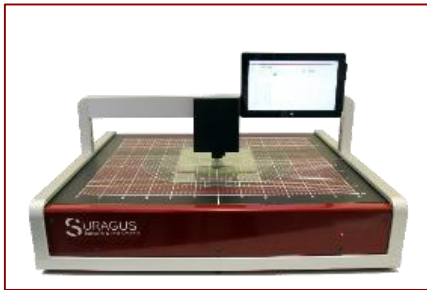
Induction Coil

Pickup Coil

Secondary Field

Eddy Currents
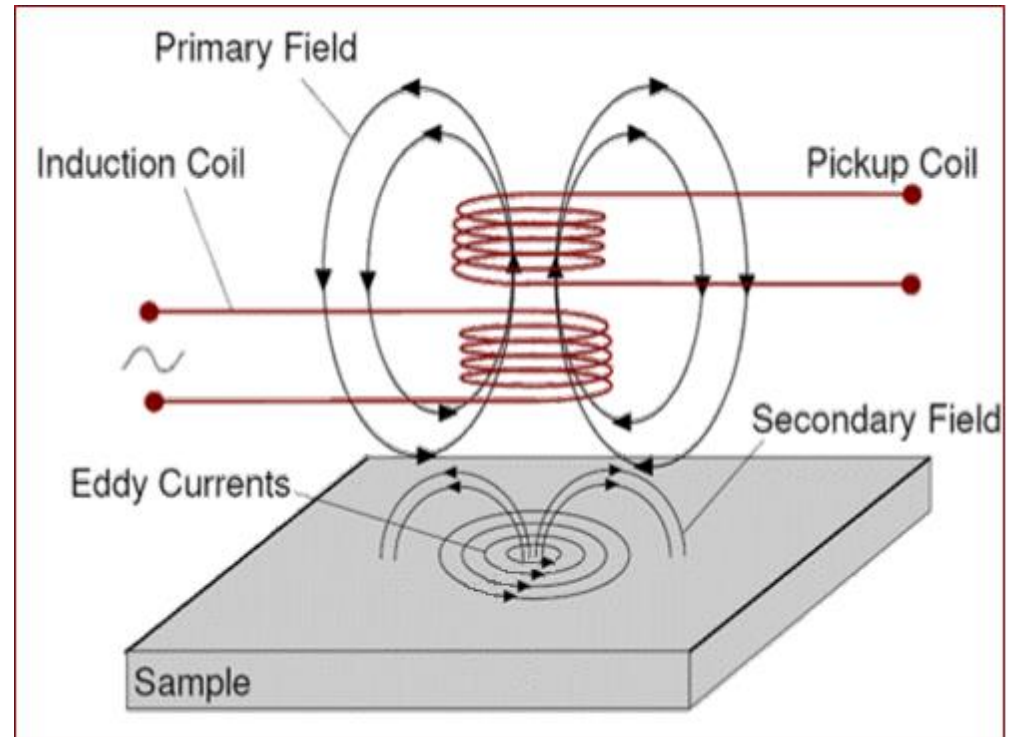
Sample

+ Non-contact
+ High sample rate
+ High sensitivity

– Limited to conductive materials

General testing types
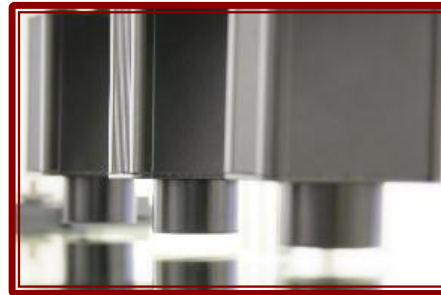
Single Point Testing	**Inline Testing**	**Imaging solutions**
EddyCus® TF lab Series	**EddyCus® TF inline Series**	**EddyCus® TF map Series**

Sheet resistance & OT
- After transfer
- Doping
- Annealing
- Ageing

Sheet resistance & OT
- Concept for inline graphene manufacturing and inline testing is currently created

Sheet resistance & OT imaging

Defect detection
- Impurities,
- deposition effects
- Many more

Information Obtained by Combined Testing

Information obtained by optical testing

- Reflection
- Transmission
- Diffuse Transmittance / Haze
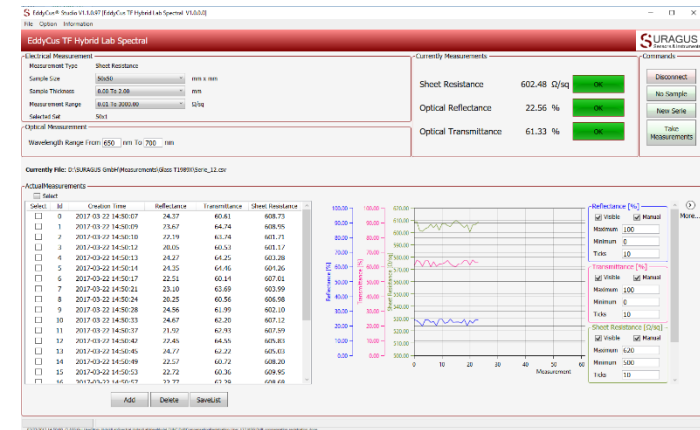
Information obtained by electrical testing

- Sheet resistance
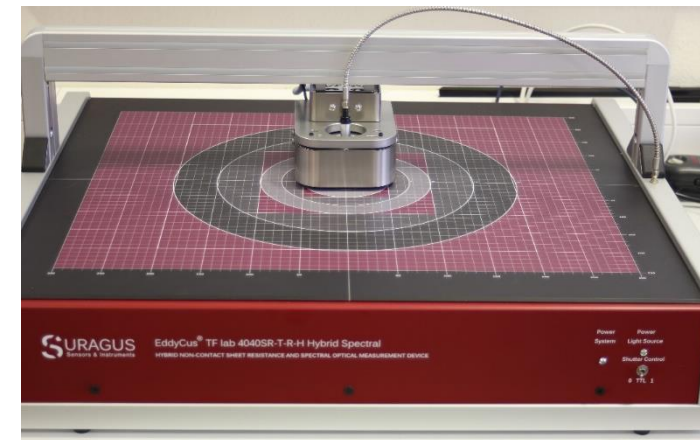
Combined Testing Measurement Head

Capabilities

- Measurement at same position

- Measurement at the same time

- Integration of optical and electrical sensors
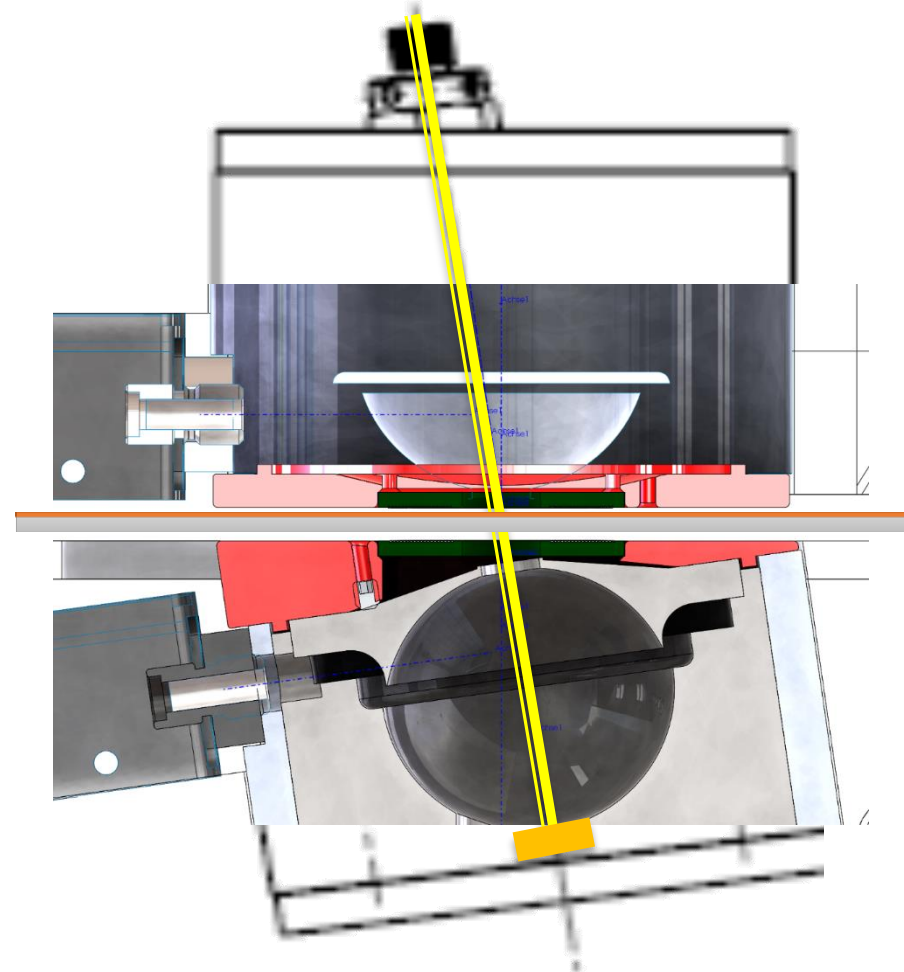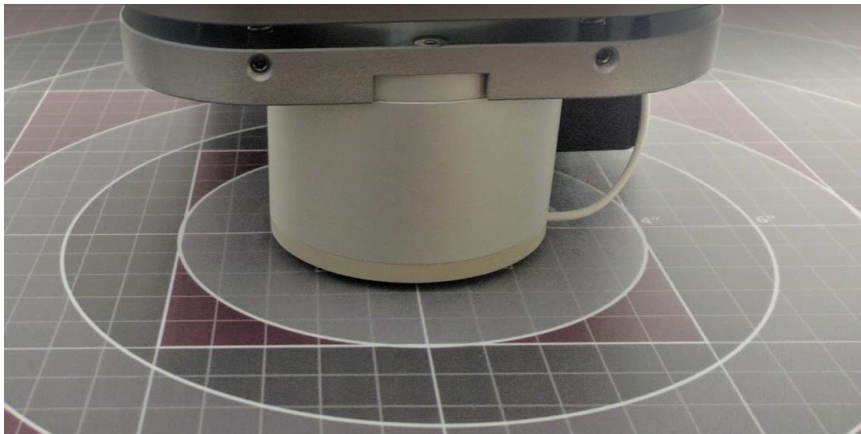
Benefits of Combined Testing

What is the beauty in combined testing?

- Lower investment costs
  - One sample holder
  - One sensor holder
  - One PC and set of drives
- Lower space requirements (one tool instead of 4)
- Measurements are obtained at the same position
- Measurements are obtained at the very same time
- Lower risk of contaminations and handing defects
- Time efficient
  - One time sample handing
  - Data merging
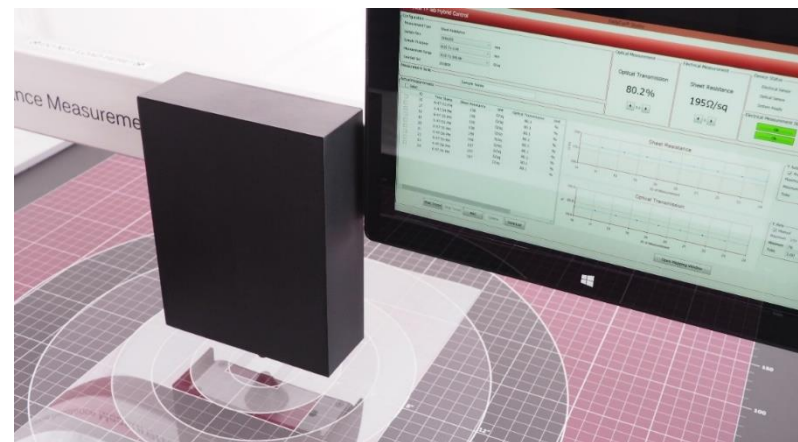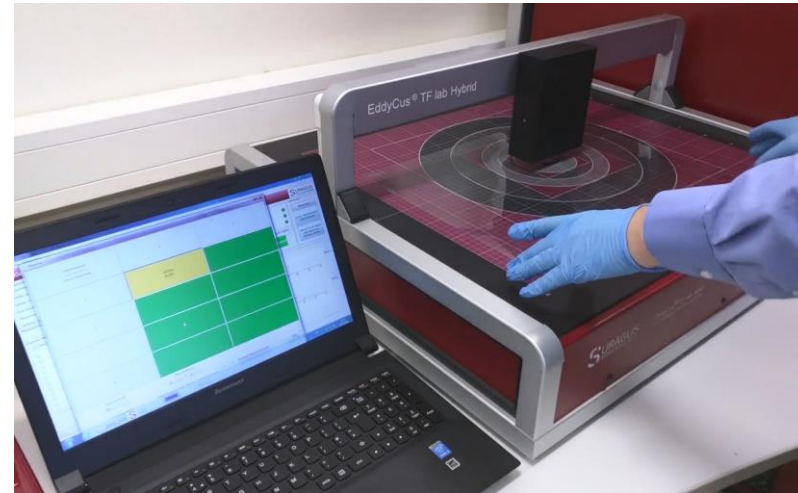
Data integrity @ time & cost savings!

Demonstration of sheet resistance imaging of large area graphene
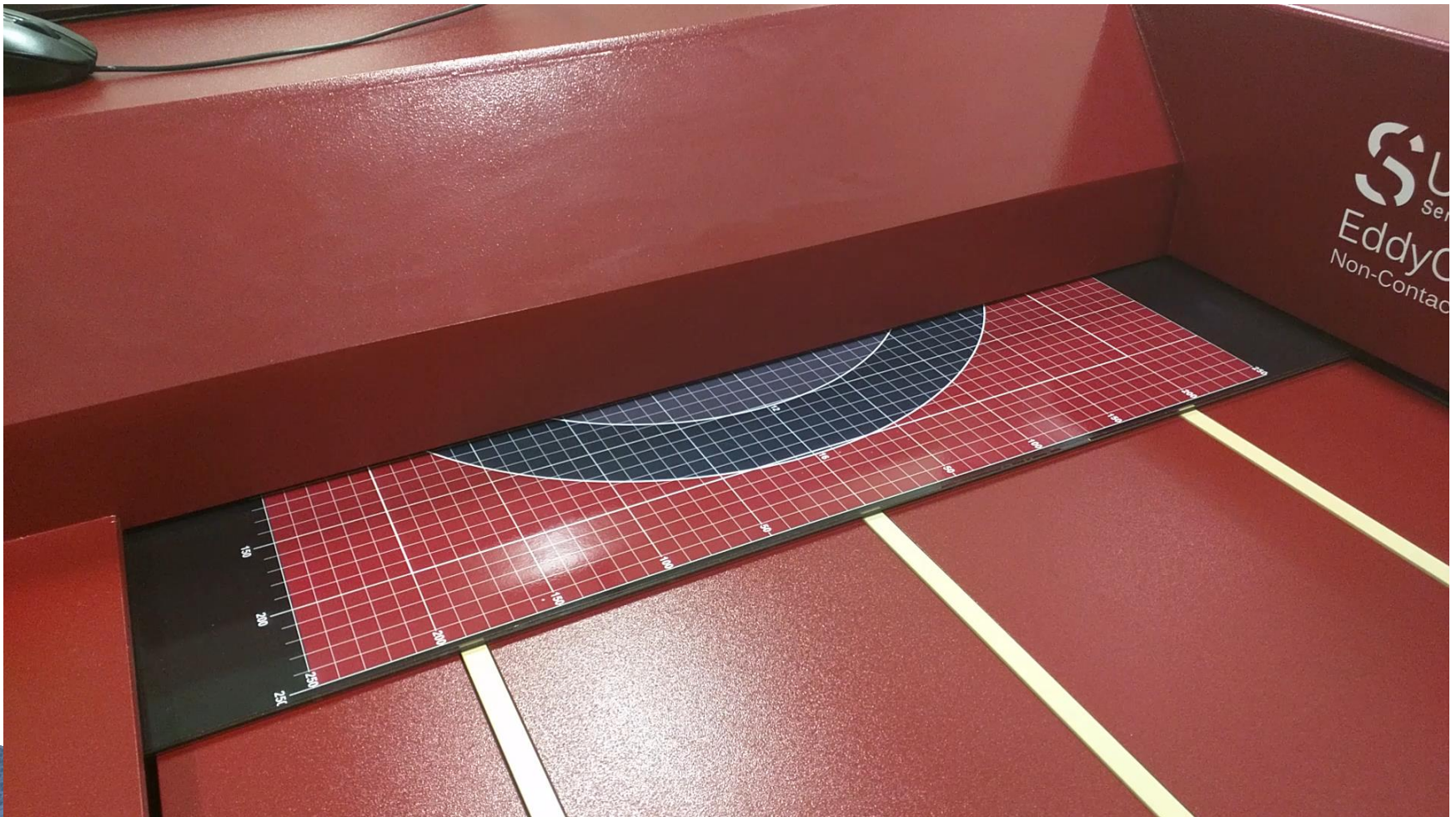
Sheet resistance imaging analysis – defectoscopy

Sheet resistance imaging [ohm/sq] on 200 x 200 mm
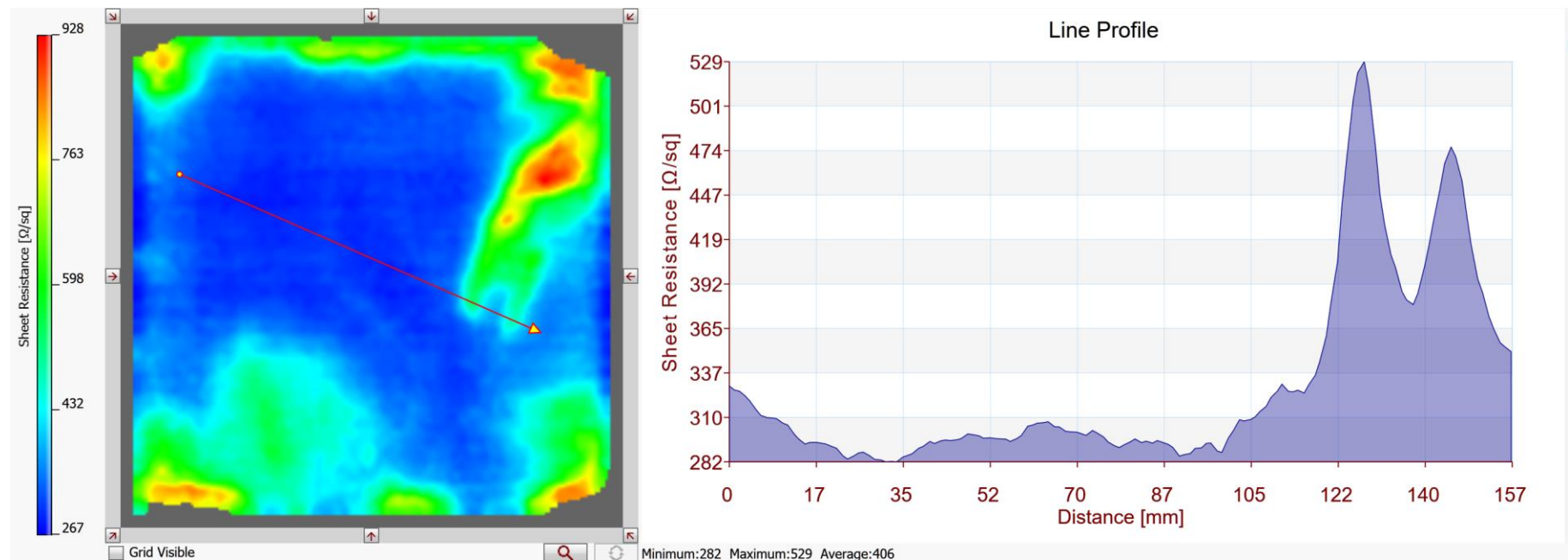
Sheet resistance imaging analysis – defectoscopy

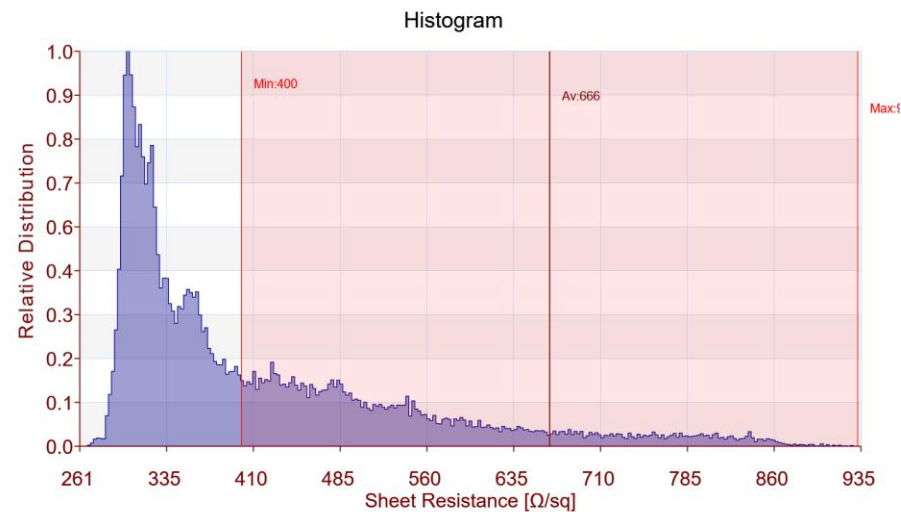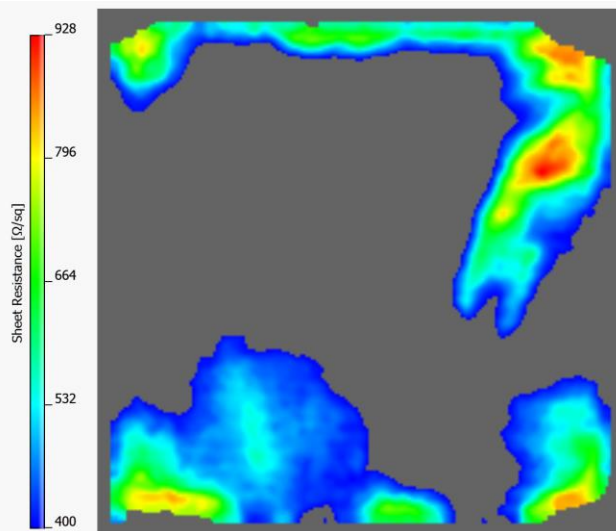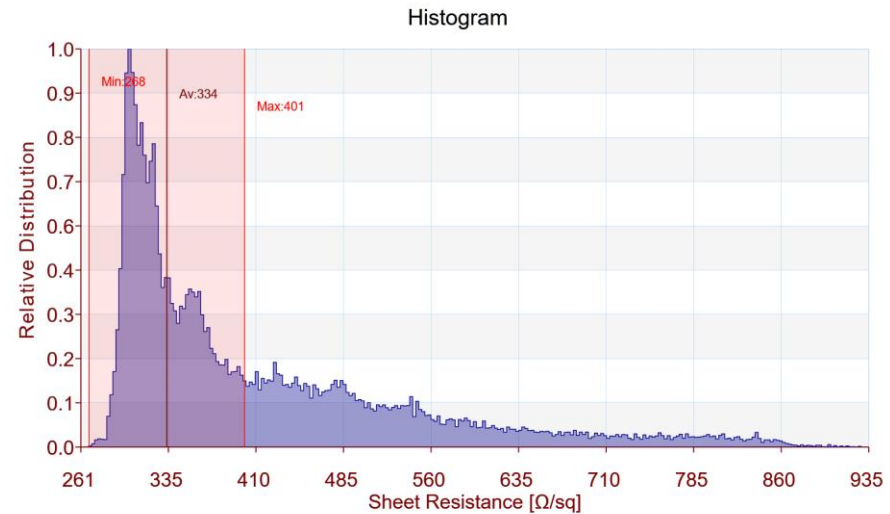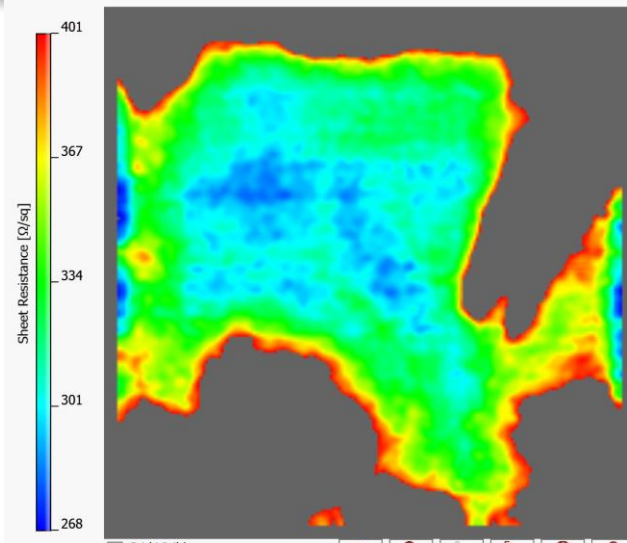## Sheet resistance imaging [ohm/sq] on 200 x 200 mm
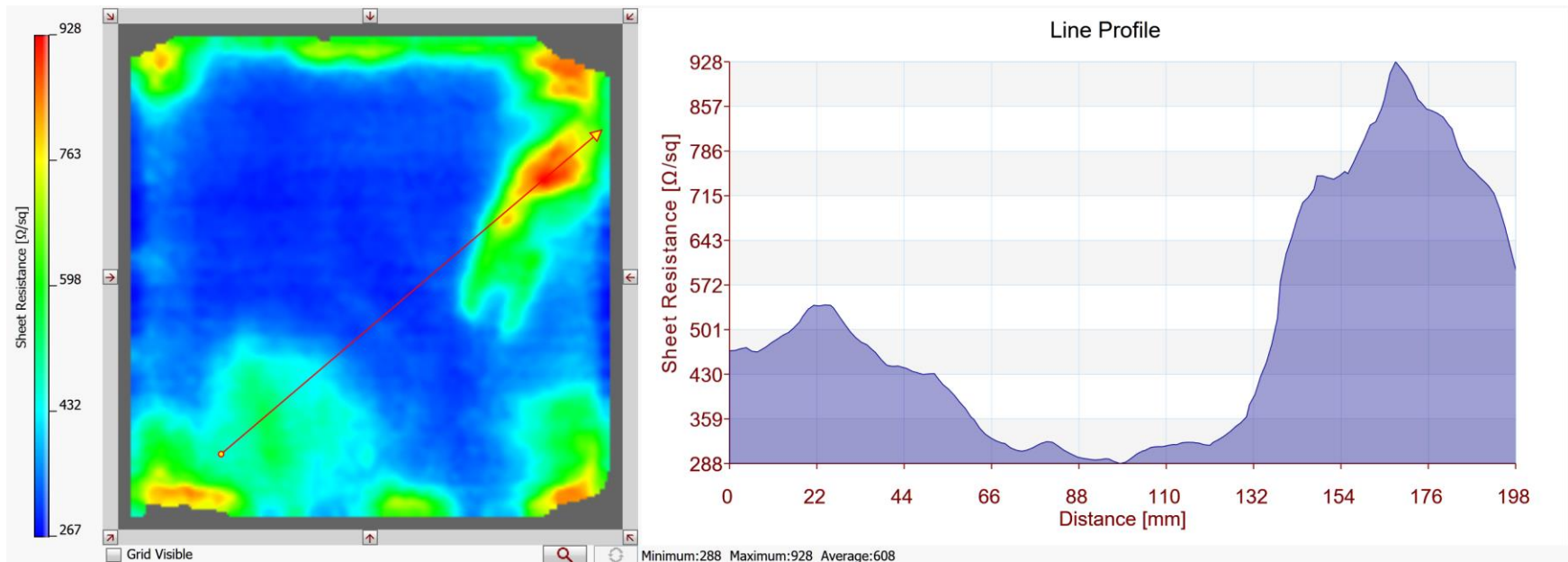
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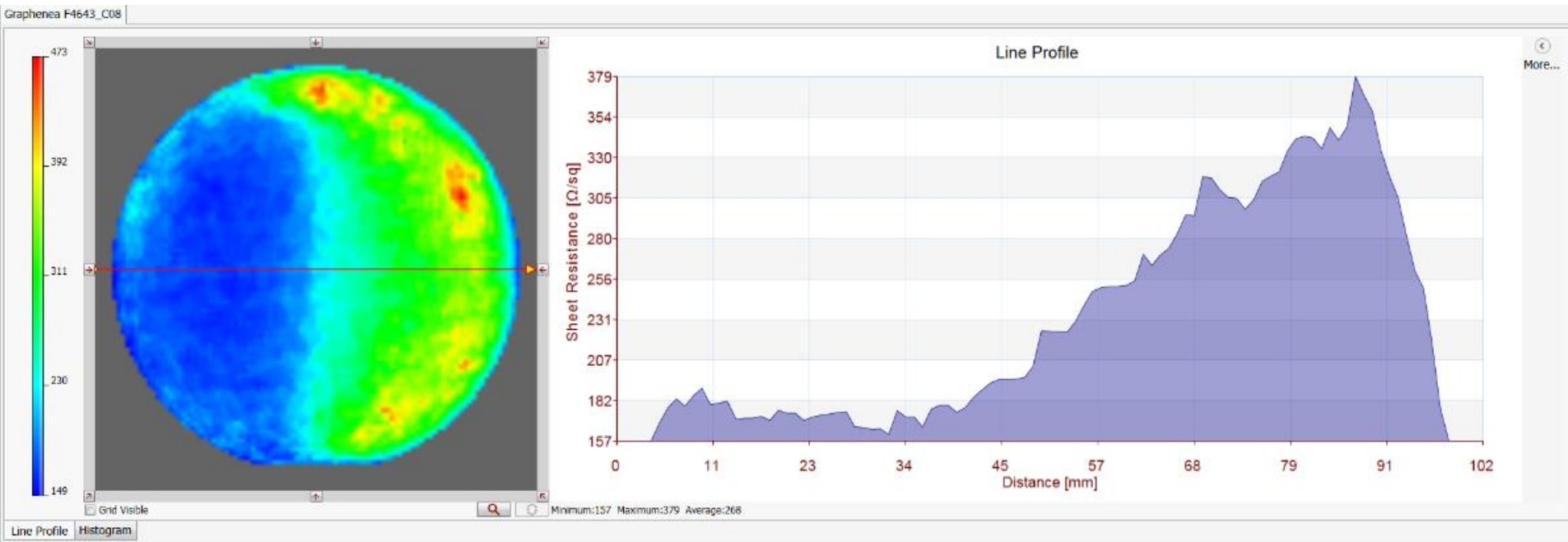
Sheet resistance imaging [ohm/sq] on 200 x 200 mm
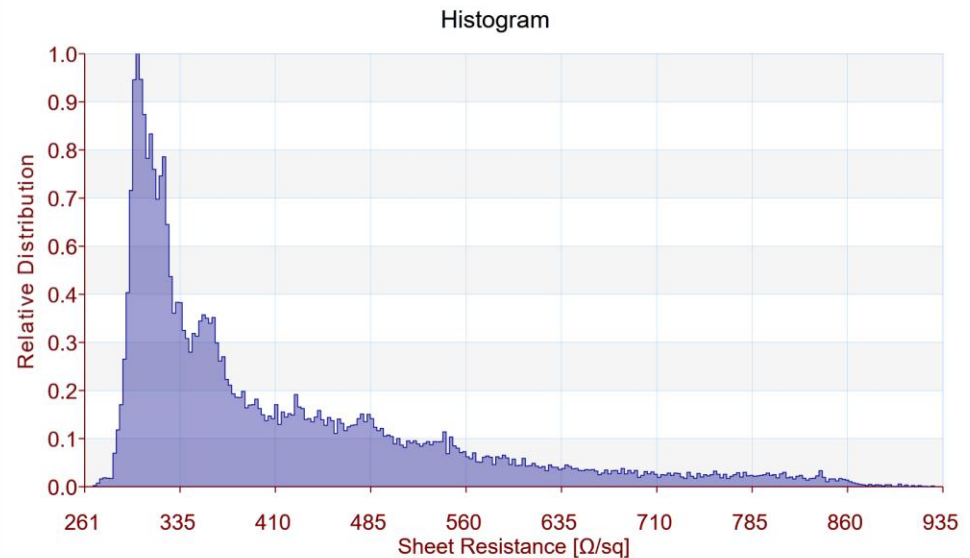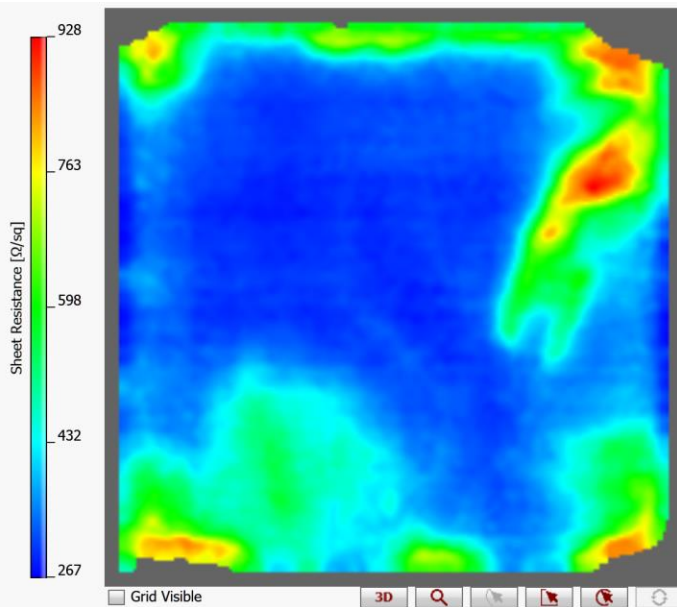
Sheet resistance imaging analysis – defectoscopy
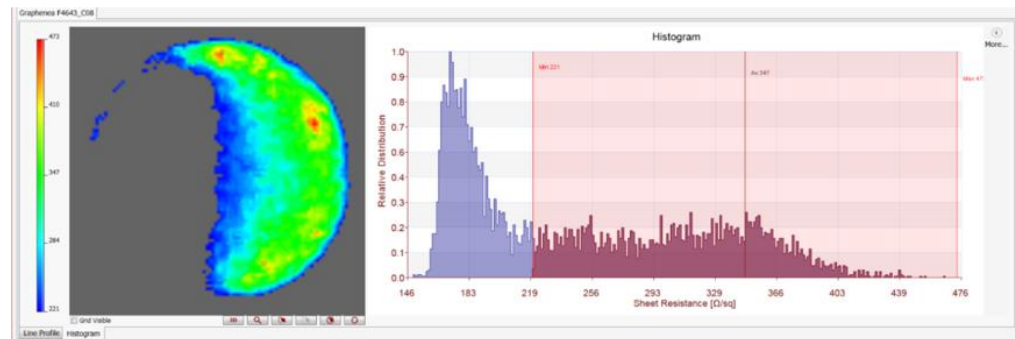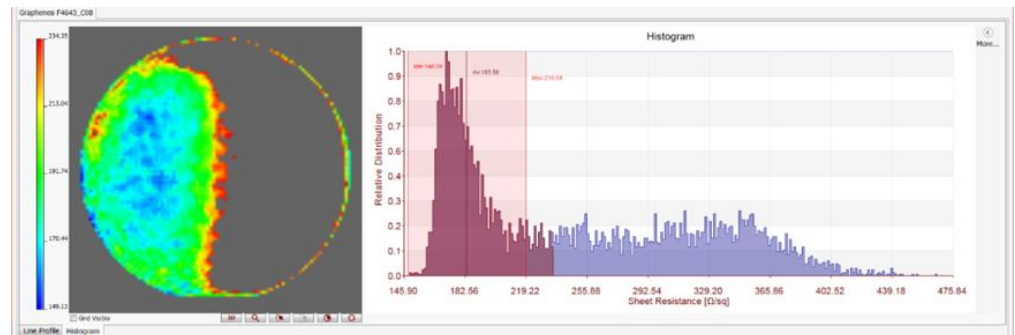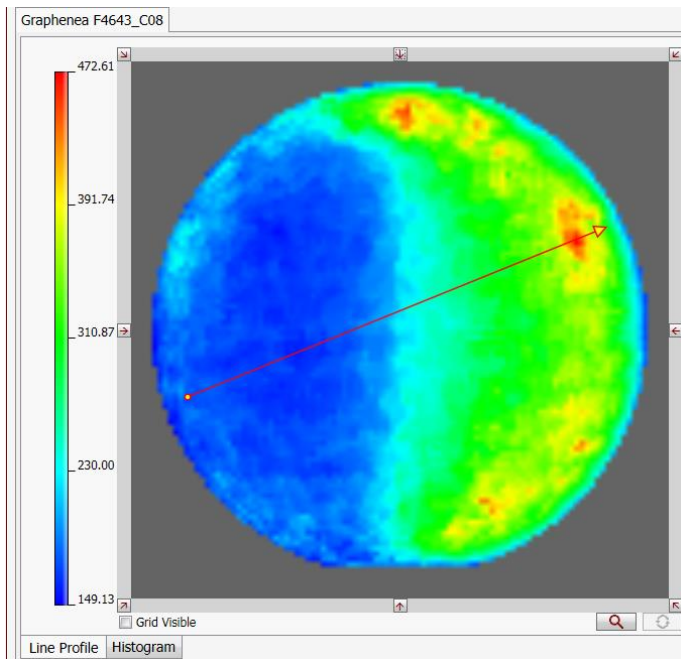
Doping effectivity

- Doping effectivity
- Doping homogeneity
- Doping stability

Doping Efficiency Imaging

- Inhomogenious doping

- **Doping – measurement after aging**
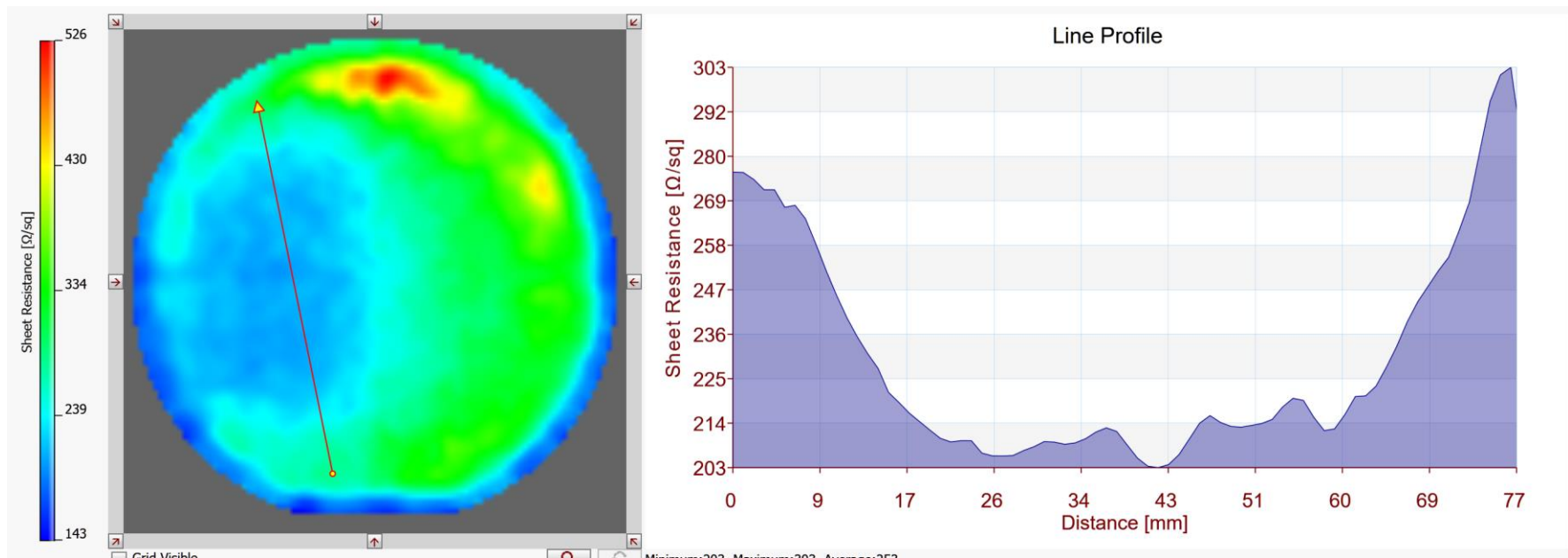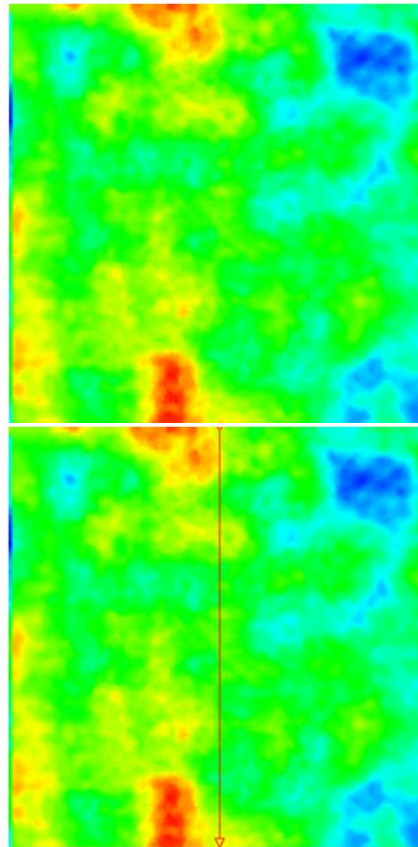
Graphite imaging
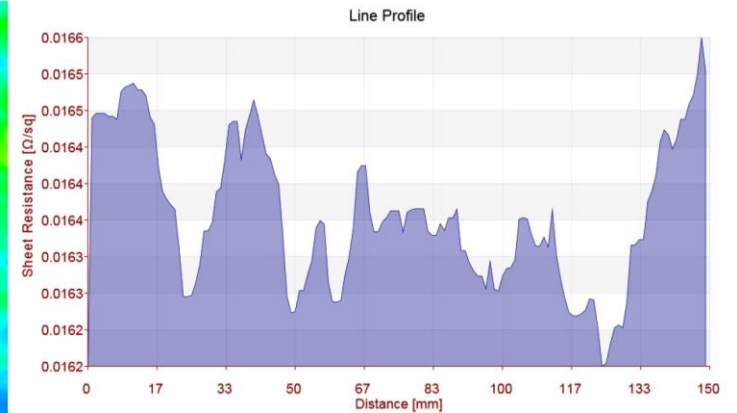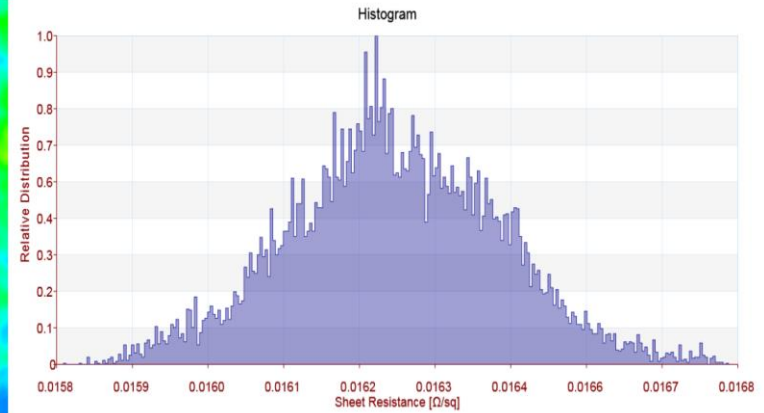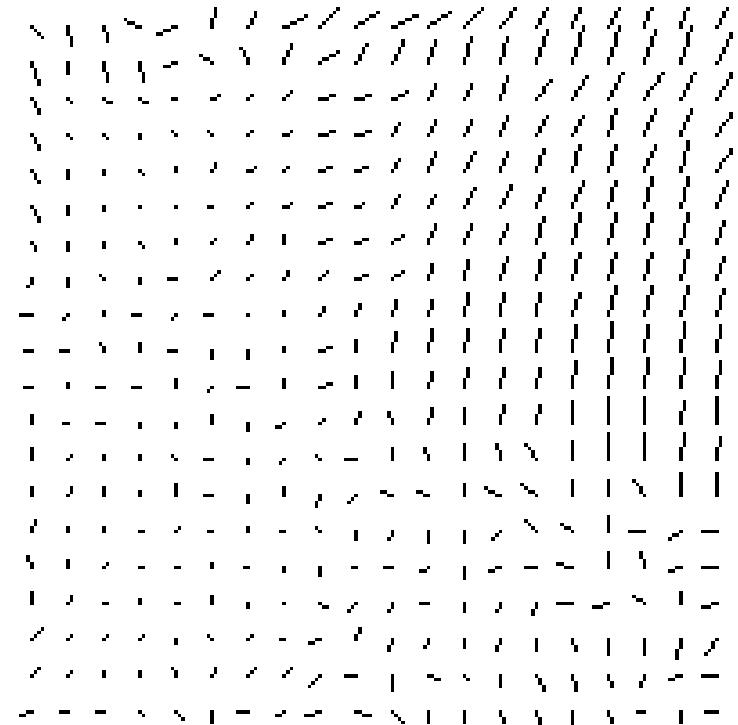
- 200 x 200 mm [8 inch], Measurement pitch 1 mm x 1mm
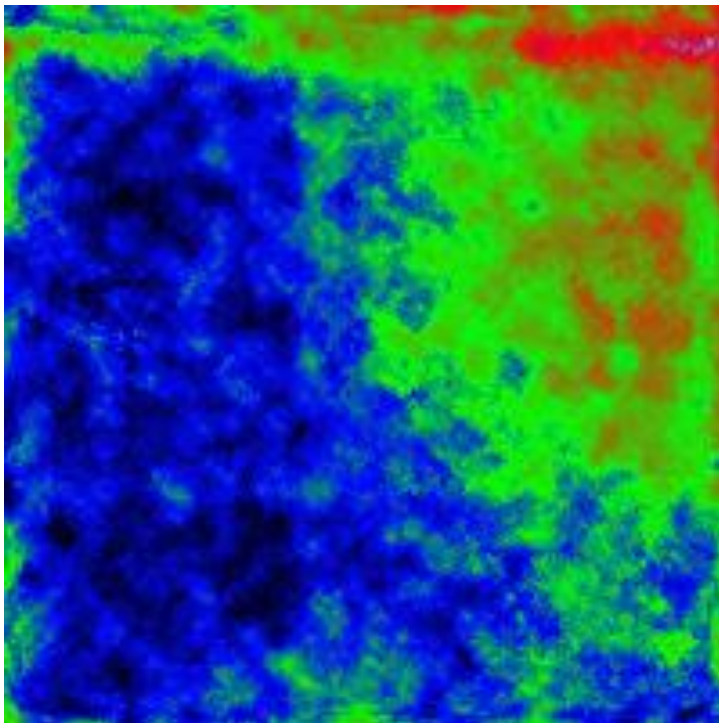
Anisotropy strength(blue=low anisotropy, red=high)

Direction of the least present sheet resistance

Demonstration – future Graphene inline testing

Customized numbers of monitoring lanes
EddyCus TF inline Series

Take home messages

- Graphene opportunities especially if many of its characteristics are required

- TCM was introduced as one example

- There are various characteristics of G. – most of them affect electrical performance

- Combined electrical and optical testing provides additional insights

- HF eddy current testing provides various benefits for Graphene characterization

  - Non- contact, non-destructive, Imaging, defect density assessment

- Imaging provides insights to many quality aspects

- Control of defect density is a key factor for successful application

- Quality assurance and process control needs to be considered along the process chain → Deposition, after transfer, doping, annealing, aging, cleaning, application integration

For questions and requests please feel free to contact us…

- SURAGUS is keen to contribute and participate in graphene application developments

- Please feel free to share your ideas!!

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