Insights into Characterization of Large Area Graphene along the Process Chain

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Stephan Adam, SURAGUS GmbH

Agenda

- SURAGUS as company and as EU Gladiator
- Challenges for industrial Graphene applications
- Example application graphene as TCM
  - Quality characteristics and common defects
  - Electrical characterization of Graphene
  - Defectoscopy - information added by sheet resistance imaging
- Summary and conclusion

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

LayTec and SURAGUS Group (> 2200 measurement systems worldwide)

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)

SURAGUS as EU Gladiator & Associated Flagship Member

http://graphene-gladiator.eu/

- Fraunhofer COMEDD (Germany)
- Graphenea S.A. (Spain)
- Danmarks Tekniske Universiteit (Denmark)
- Horiba Jobin Yvon S.A.S. (France)
- AIXTRON SE (Germany)
- AIXTRON Ltd. (United Kingdom)
- Suragus GmbH (Germany)
- Commissariat à l'energie atomique et aux energies alternatives (France)
- Amcor Flexibles Kreuzlingen AG (Switzerland)
- Amcor Flexibles Singen GmbH (Germany)
- Leibniz–Institut für Oberflächenmodifikation (Germany)
- Det National Forskningscenter Forarbejdsmiljo (Denmark)
- Aristotelio Panepistimio Thessalonikis (Greece)
- Organic Electronic Technologies (Greece)
- Amanuensis GmbH (Switzerland)

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

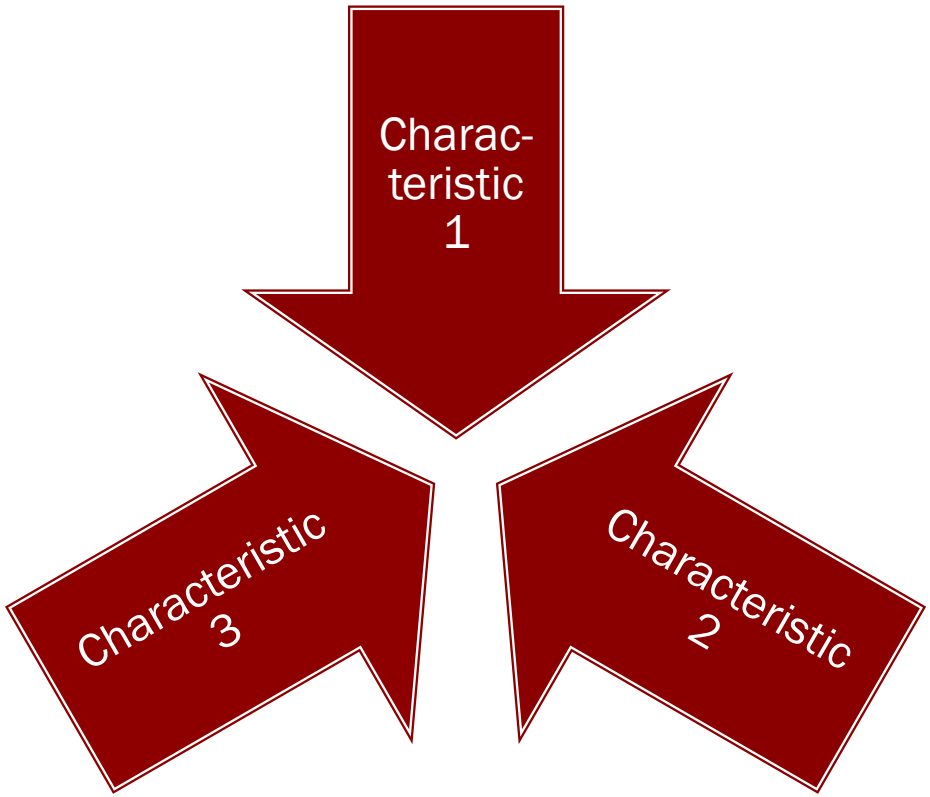
- Electrical properties
- Mechanical properties
- Chemical properties
- Optical properties
- Etc.

Soft characteristics

- Ability for flexible substrates
- Stable over time

Financial Characteristics

- Cost per performance



Challenges the Industry Is Facing from an Commercial/Industrial Application Point of View

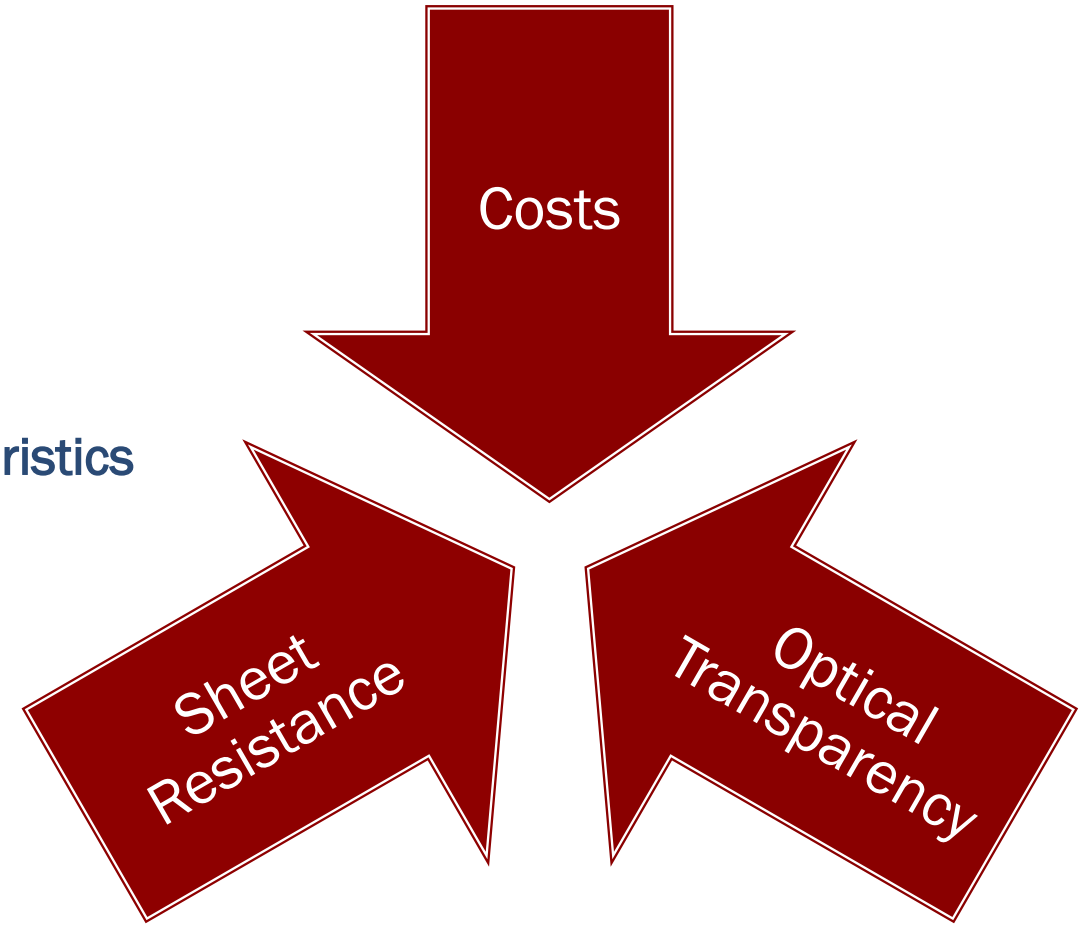
Example: Graphene as transparent electrode

Requirements/ trends

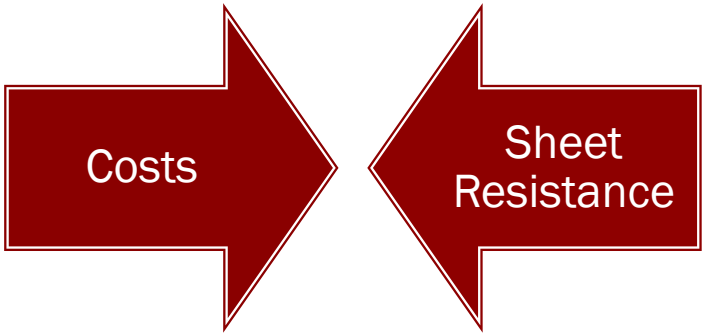
- Low cost
- Low sheet resistance
- High transparency

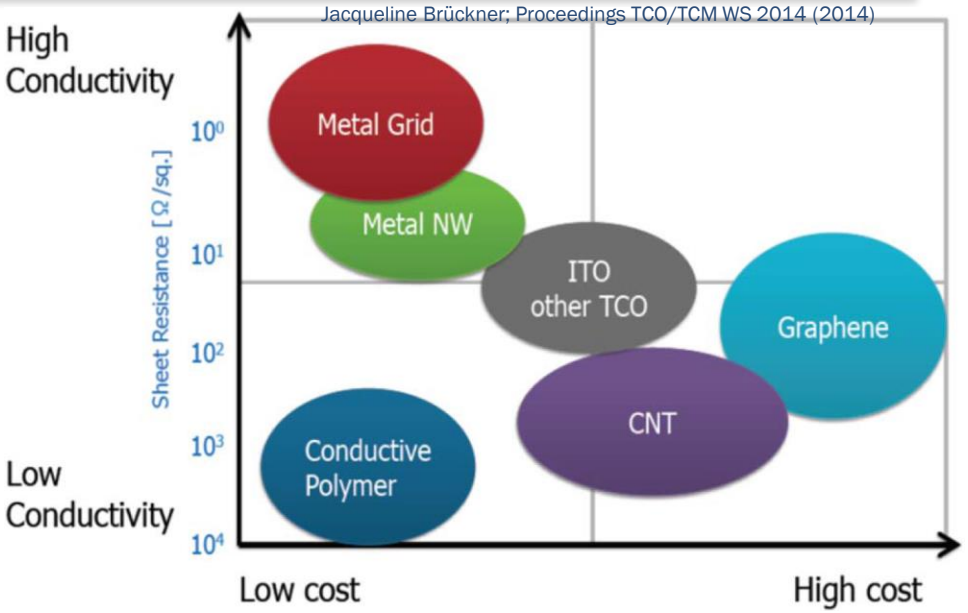
Beneficial/ further characteristics

- Ability for flexible substrates
- Low aging effects



Drivers & Challenges in Manufacturing





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 e.g. reuse of growth substrates

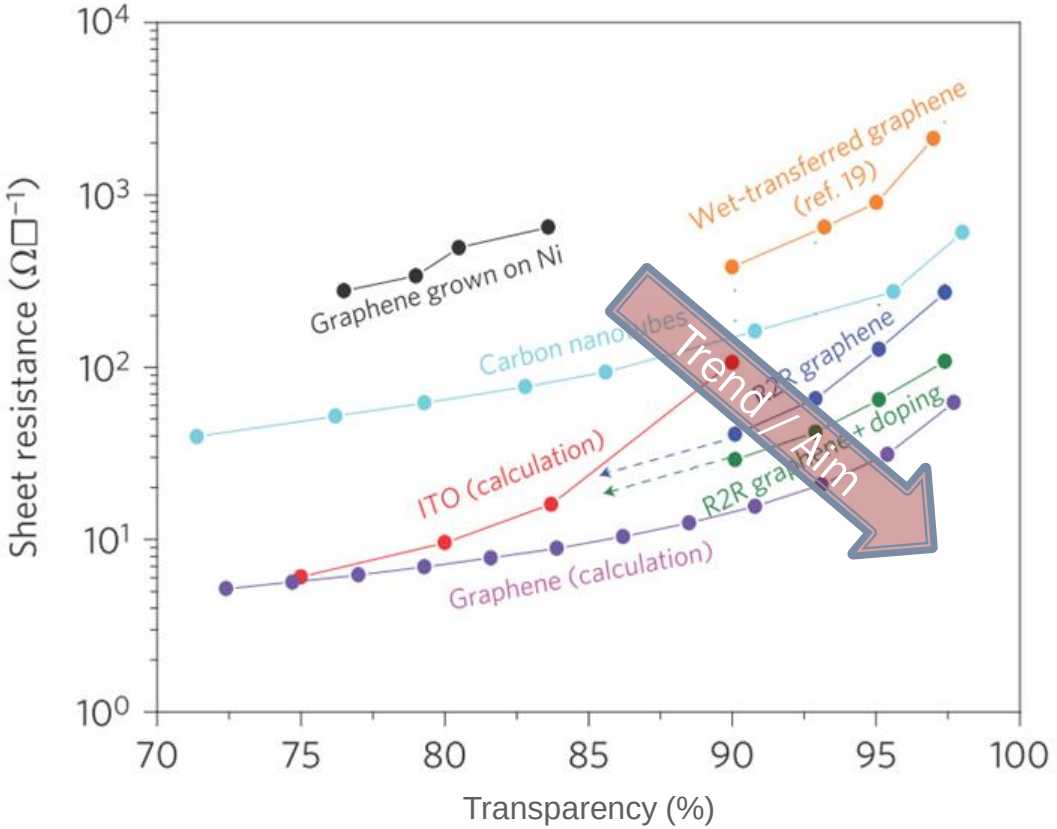
Graphene as Transparent Electrode

Optical Transparency

Sheet Resistance

Main Parameter

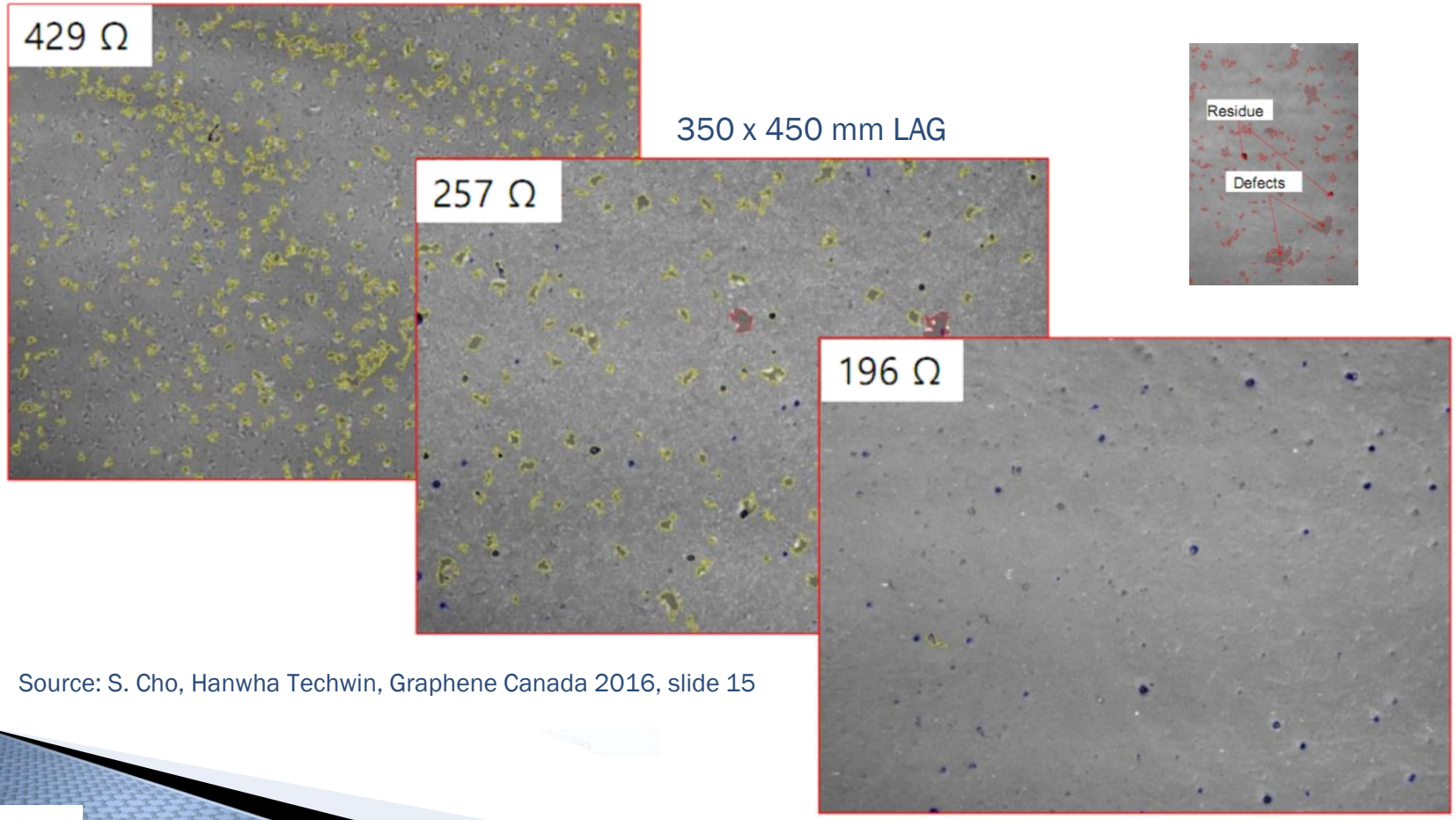
- Low boundary rate / monocrystalline
- Physical integrity
- Doping
- Annealing
- Stacking, patching
- Defect-freeness



S. Bae et al. Nature Nano. 5, 571 (2010)

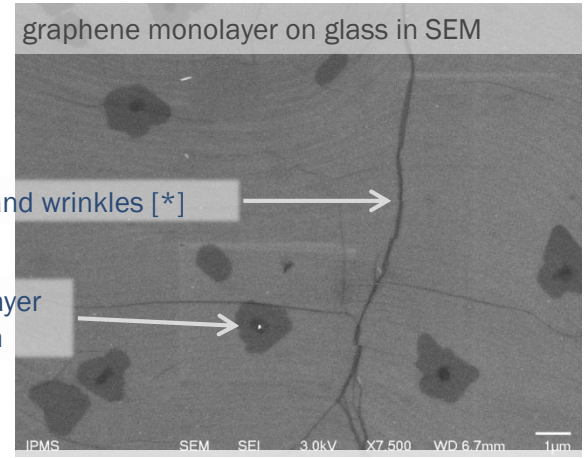
Effects of Defects to the Sheet Resistance

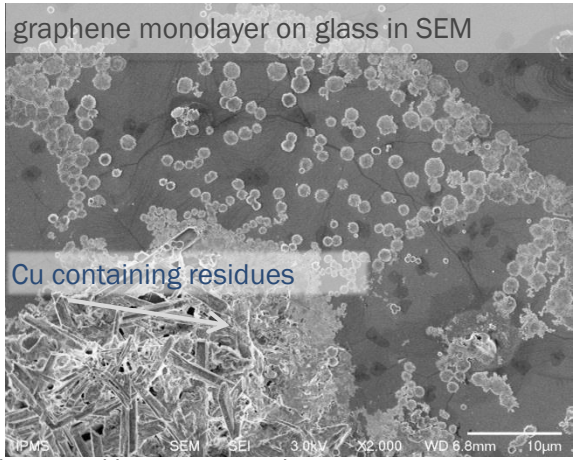
- The defect density significantly affects the sheet resistance

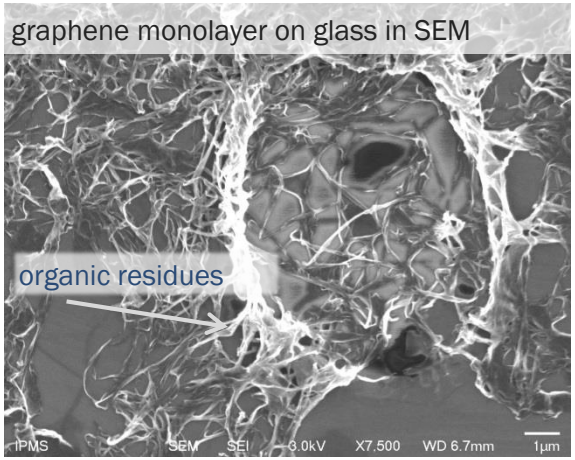


Source: S. Cho, Hanwha Techwin, Graphene Canada 2016, slide 15

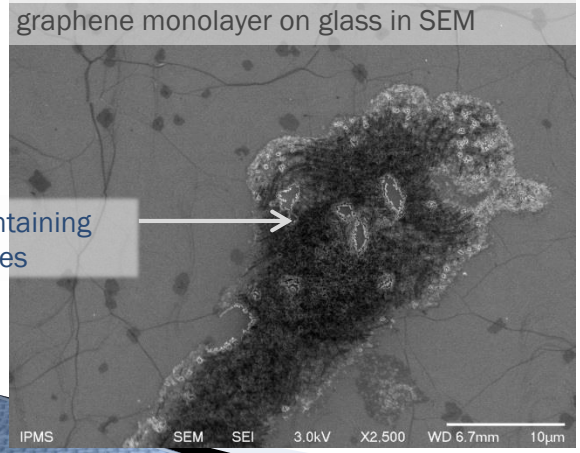
Typical Defects in Graphene

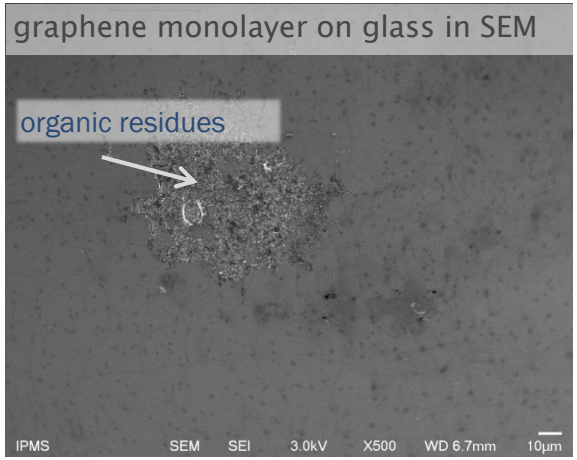


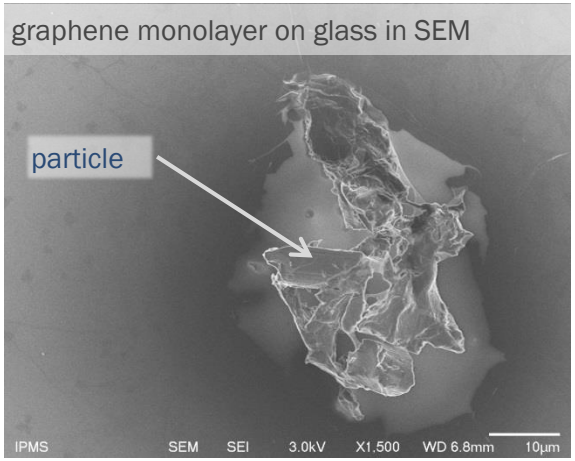




[*] Zhu et al., *Structure and Electronic Transport in Graphene Wrinkles*, Nature Nanoletters, 2012



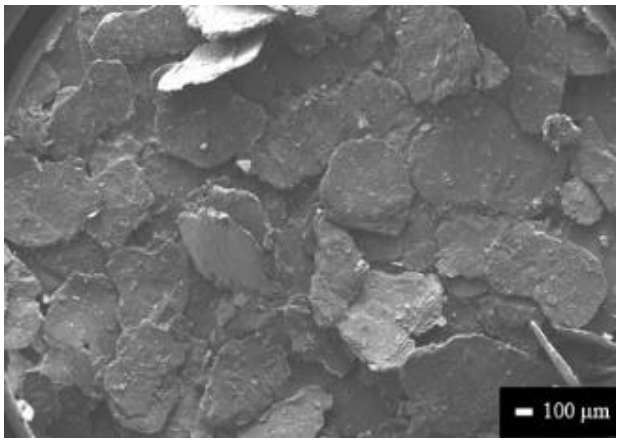


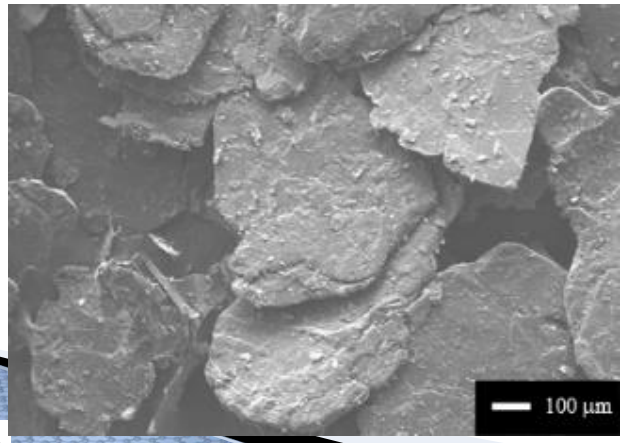


Source: D. Waynolds et al., Fraunhofer FEP

Properties and Common Defects

- Flake size



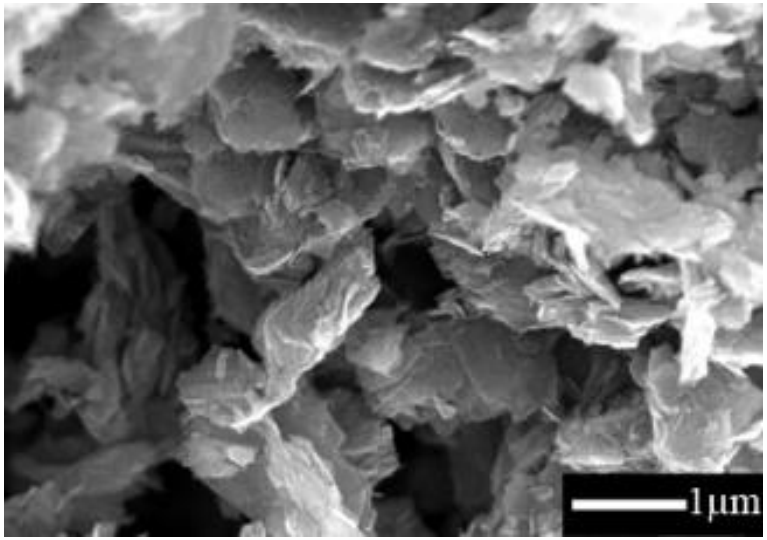


Effect on
sheet resistance

Source: Graphene-supermarket.com

Properties and Common Defects

- Stacking angle and stacking density



Effect on
sheet resistance

Source: Graphene-supermarket.com

Properties and Common Defects

- Monolayer / multilayer Graphene
- Polycrystalline graphene / flake size
- Contaminations, particles and residues
- Doping and doping homogeneity
- Stacking angle and stacking density
- Line defects, wrinkles, holes, missing Graphene

Significant effects on sheet resistance

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?

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

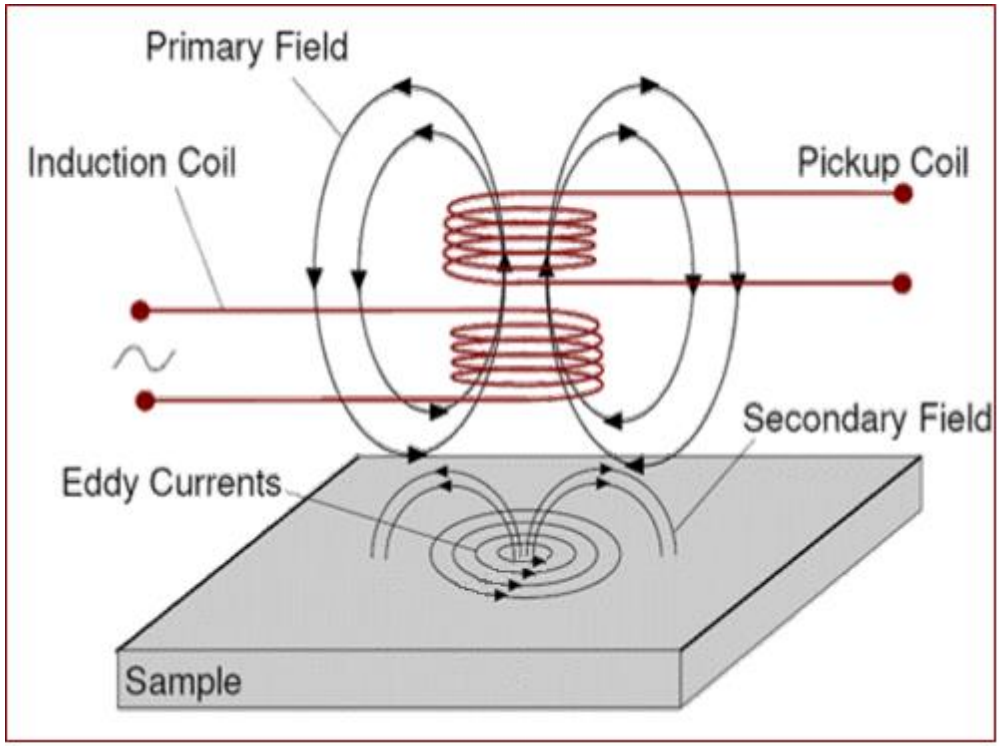
4-point-probe testing

- 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
- High resolution mapping
- Inline measurement possible

Sheet Resistance Measurement by Eddy Current



+ Non-contact
+ High sample rate
+ High sensitivity

- Limited to conductive materials

General Testing Setups

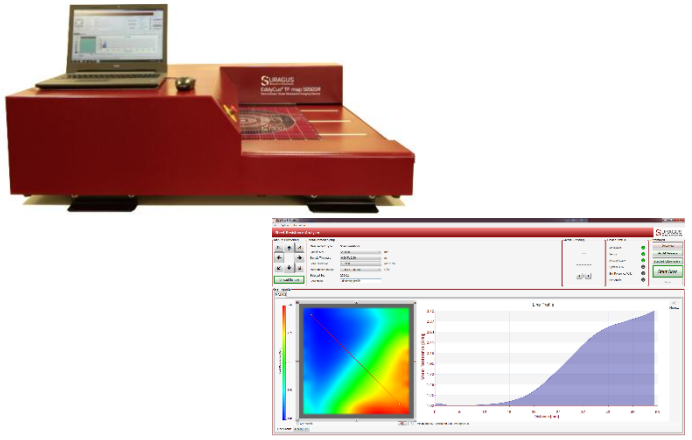
Single Point Testing

EddyCus® TF lab Series

Sheet resistance & OT

- After transfer
- Doping
- Annealing
- Aging

Imaging Solutions



EddyCus® TF map Series

Sheet resistance & OT imaging

Defect detection

- Impurities
- Deposition effects
- Many more

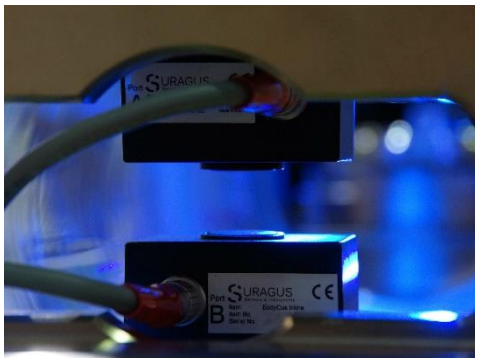
General Testing Setups

Inline Testing

EddyCus® TF inline Series

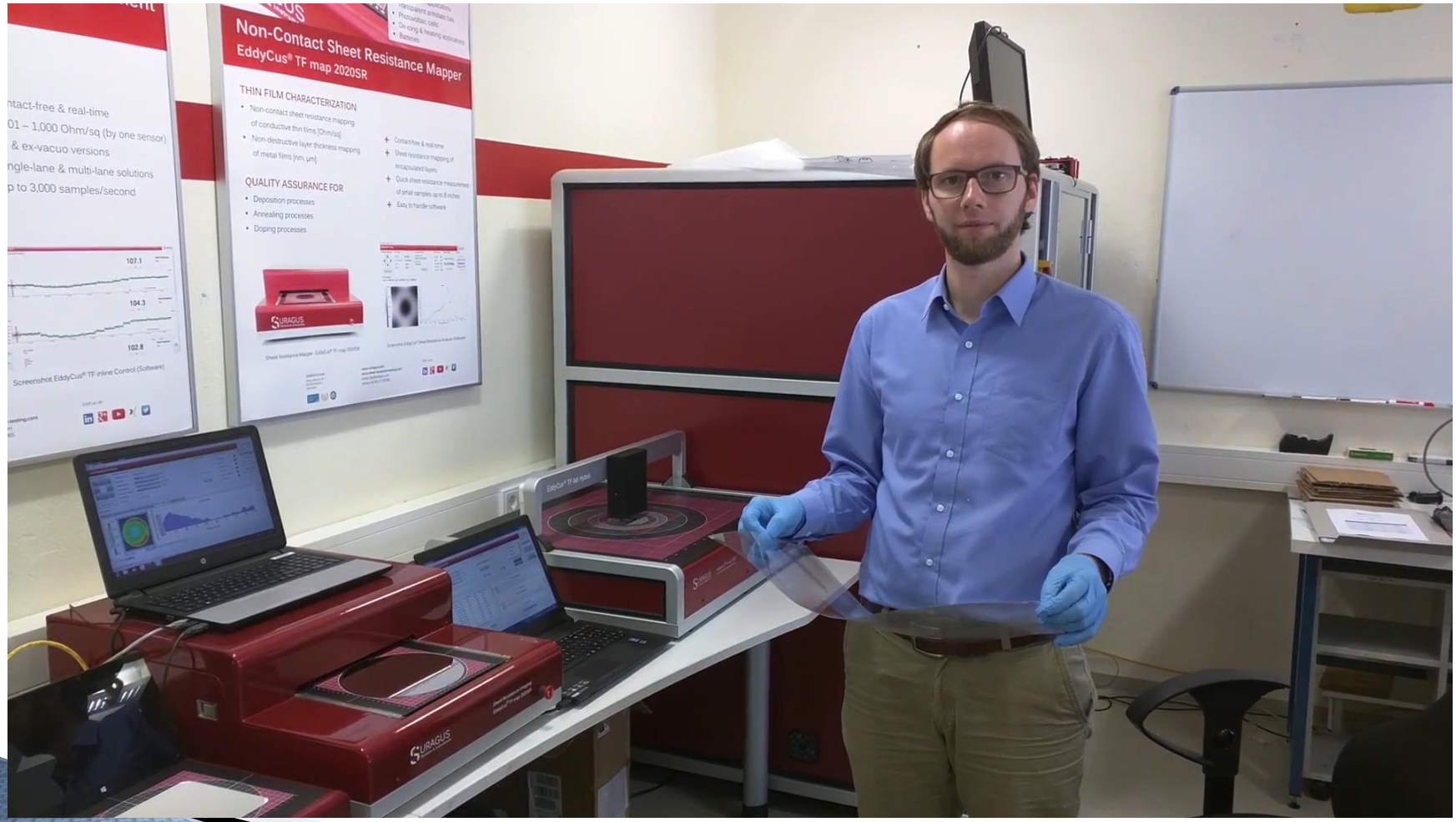
Sheet resistance & OT

- Concept for inline graphene manufacturing and inline testing is currently created



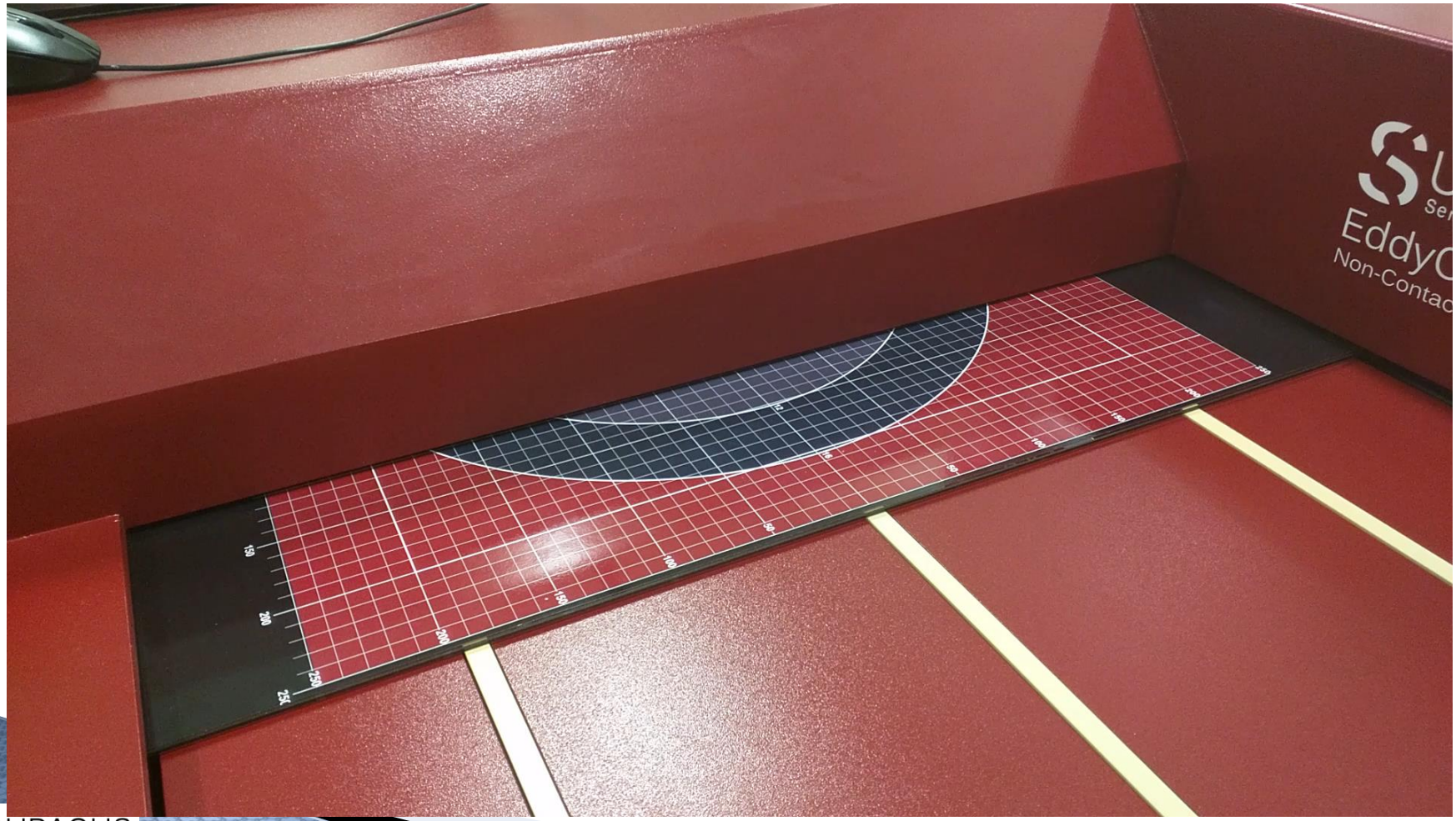
Demonstration – Manual Mapping

Simultaneous Manual Mapping of Sheet Resistance and Optical Transparency of Large Area Graphene (LAG)



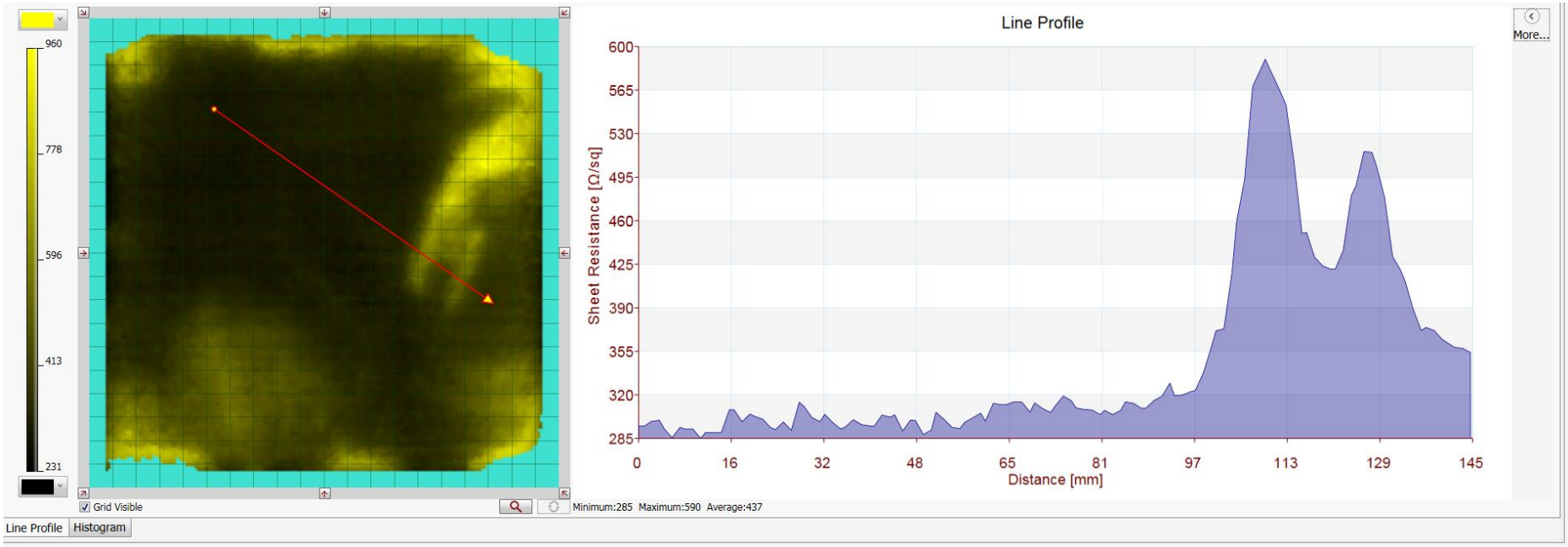
Watch movie on youtube here.

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

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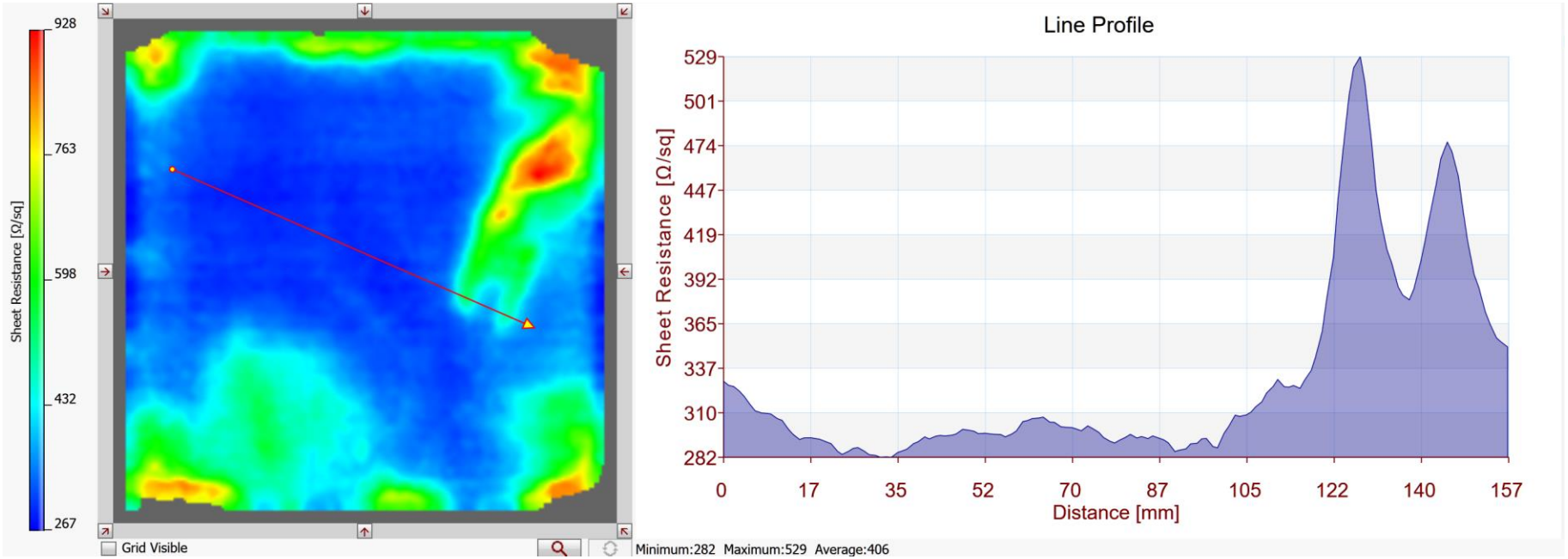
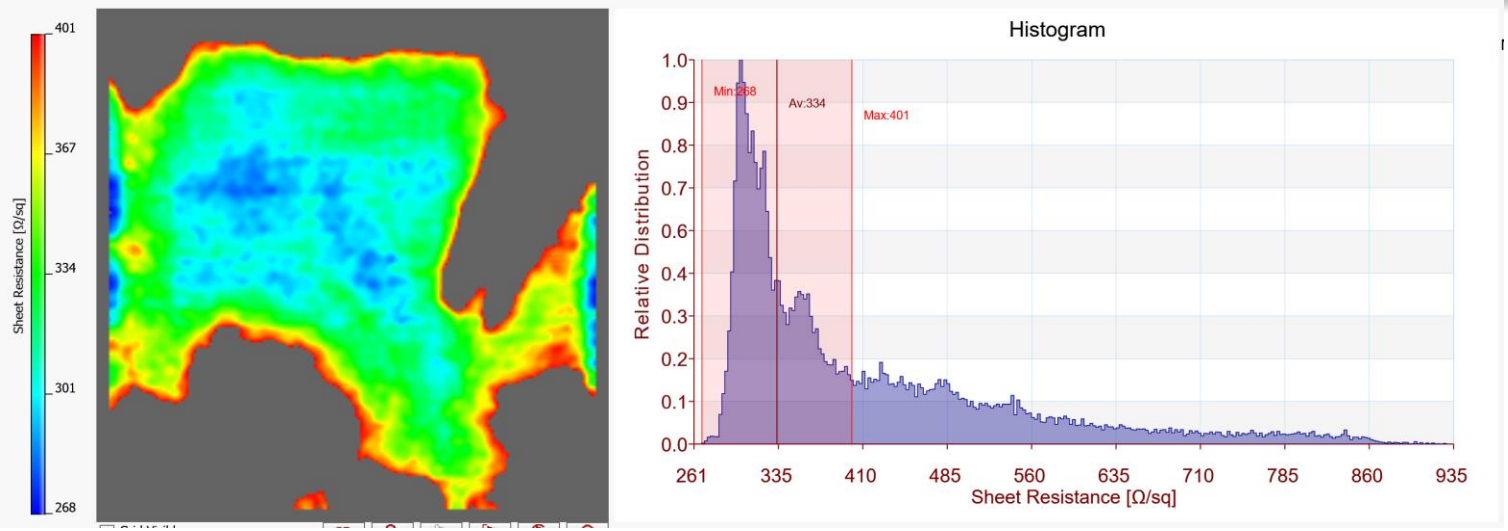
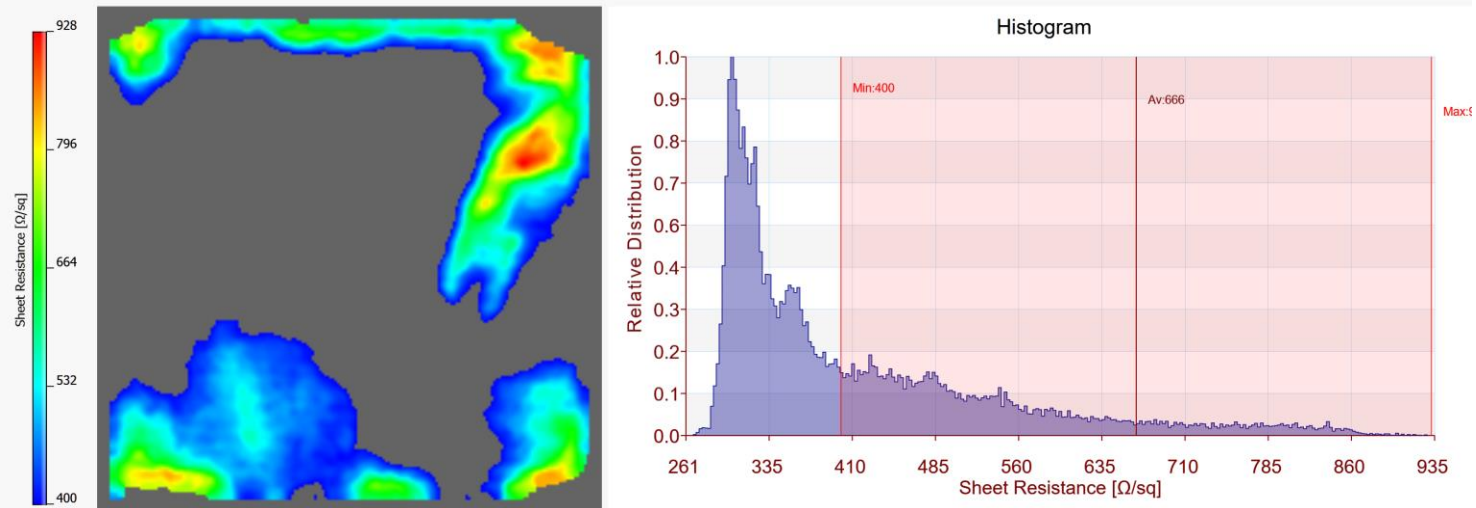
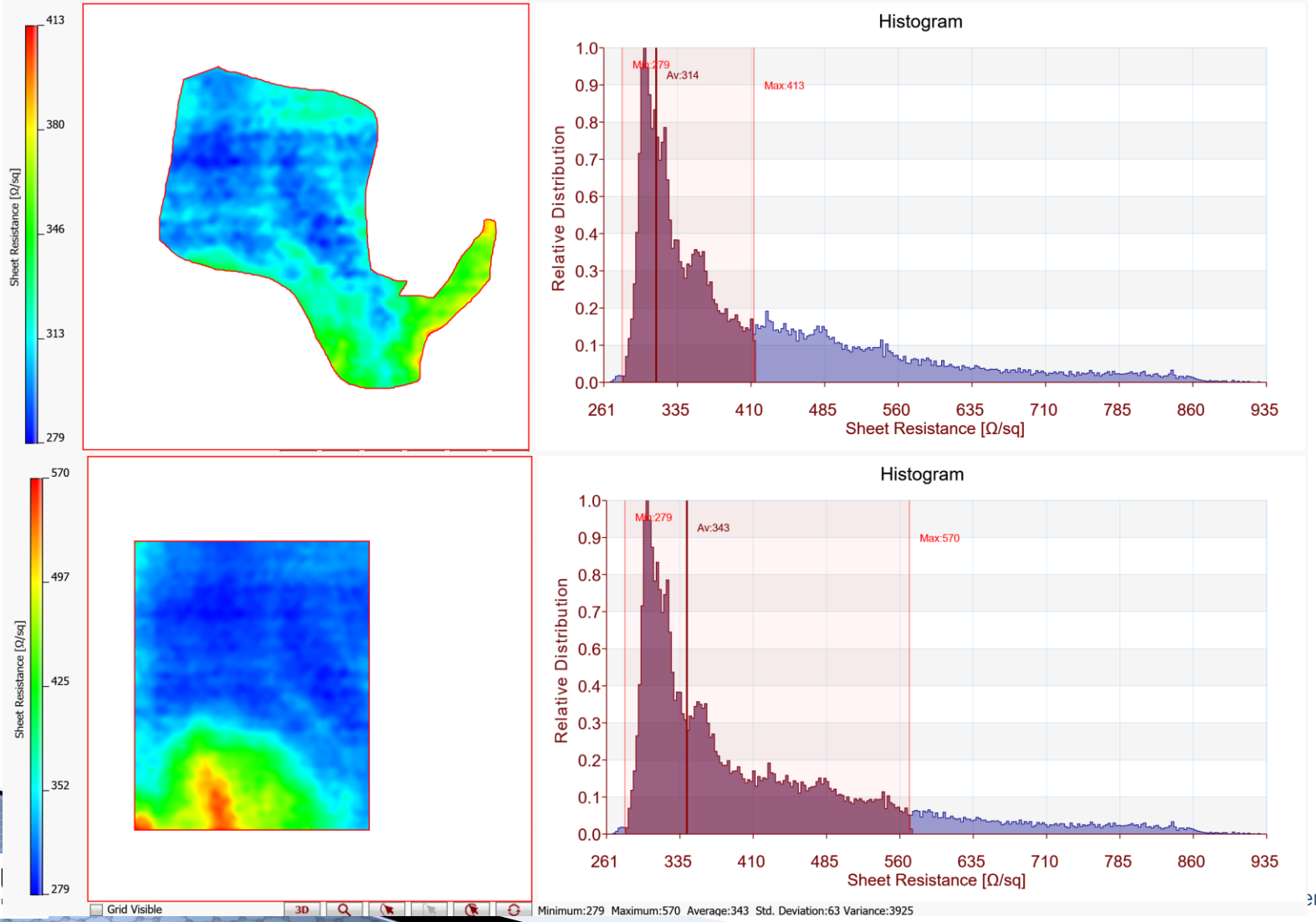


Sheet Resistance Imaging Analysis - Defectoscopy





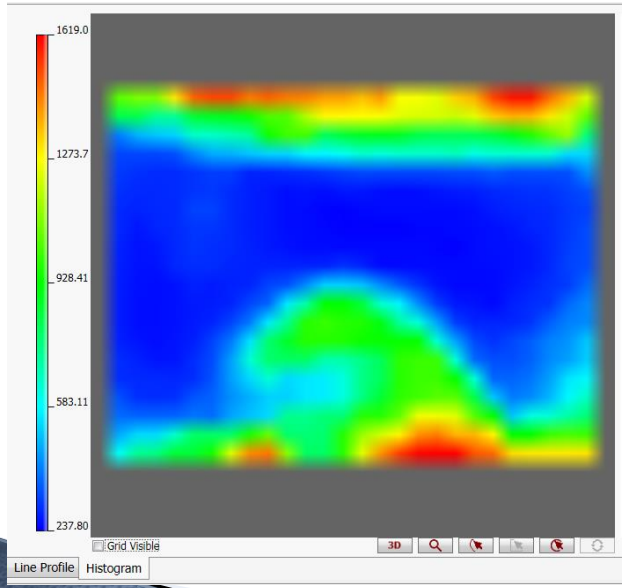
Sheet Resistance Imaging Analysis - Defectoscopy

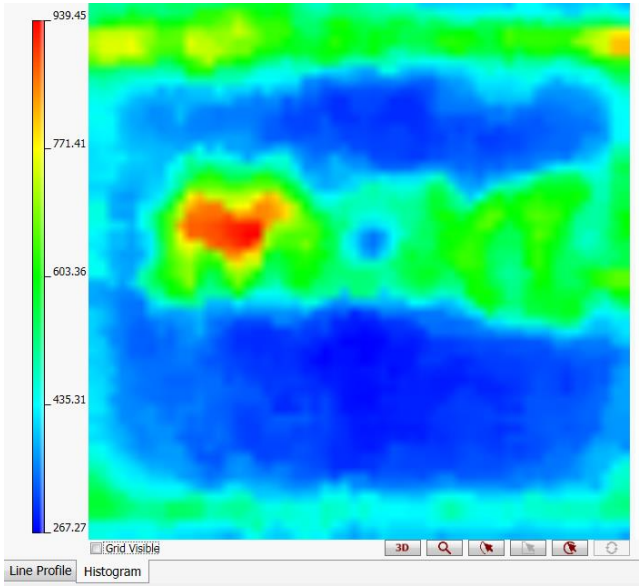


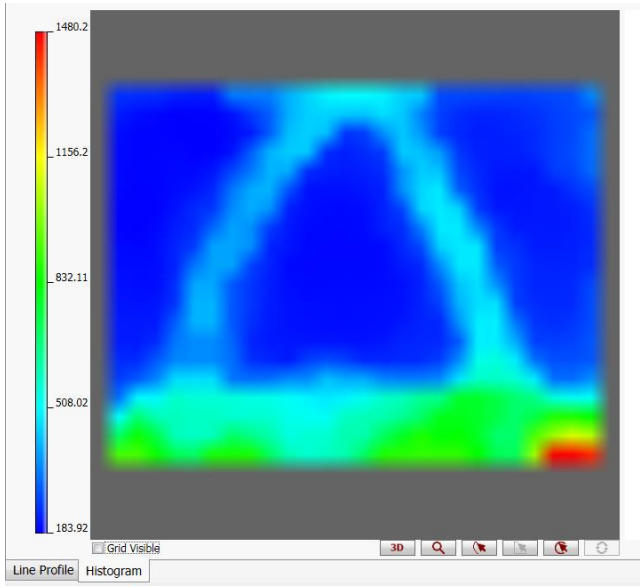
Missing Graphene, Holes and Cracks

- Defect detection
- Selection of “good areas” for further processing

Sheet resistance mappings [ohm/sq] on ~ A4



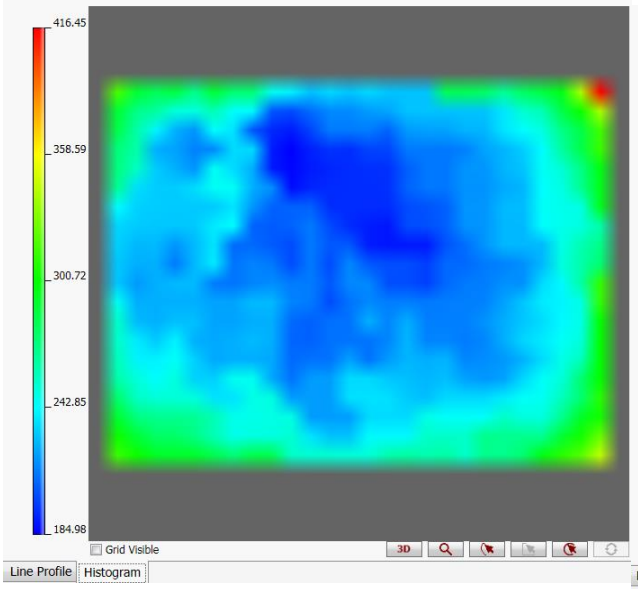


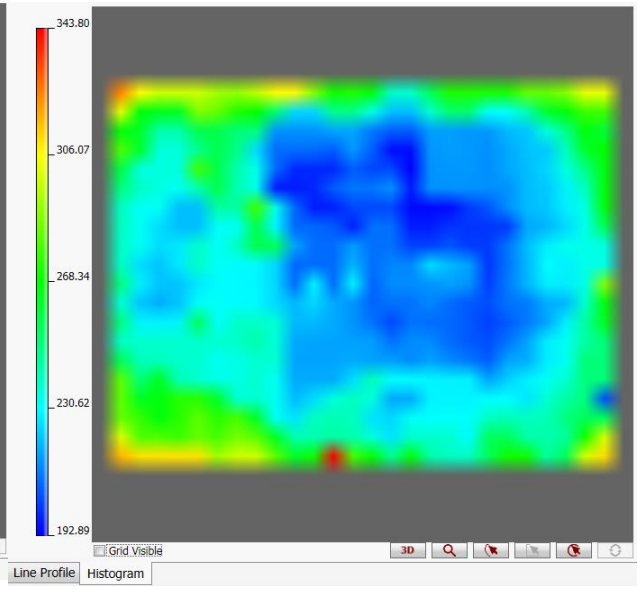


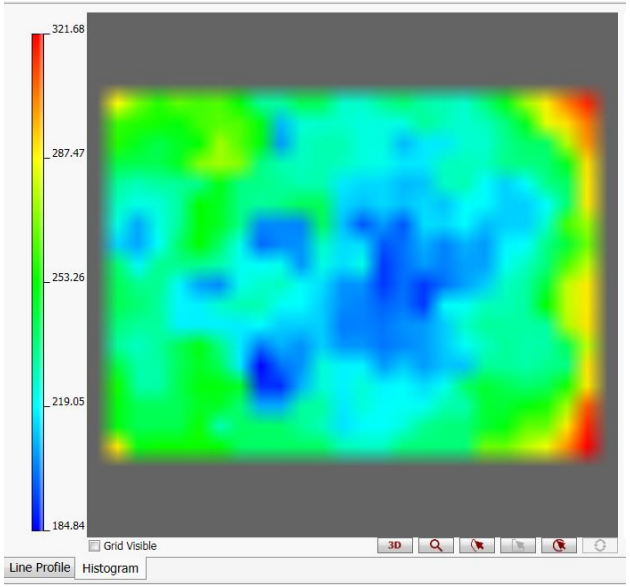
Small Inhomogeneity

- Homogeneity assessment

Sheet resistance mappings [ohm/sq] on ~ A4

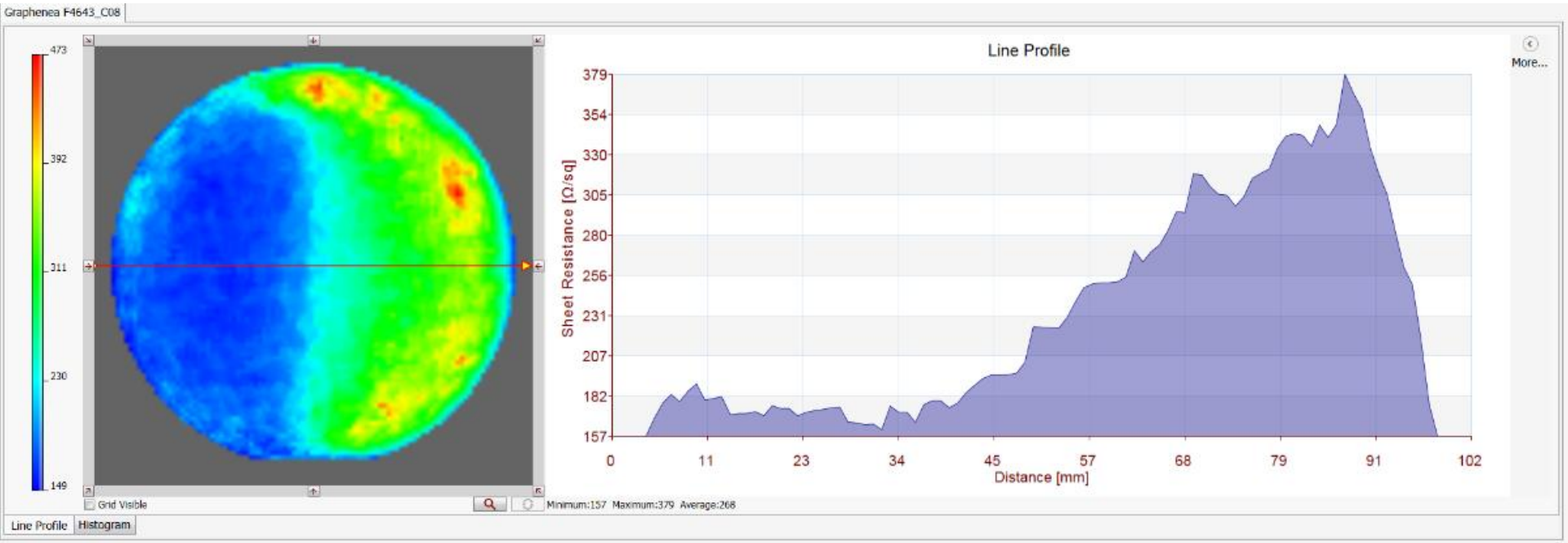






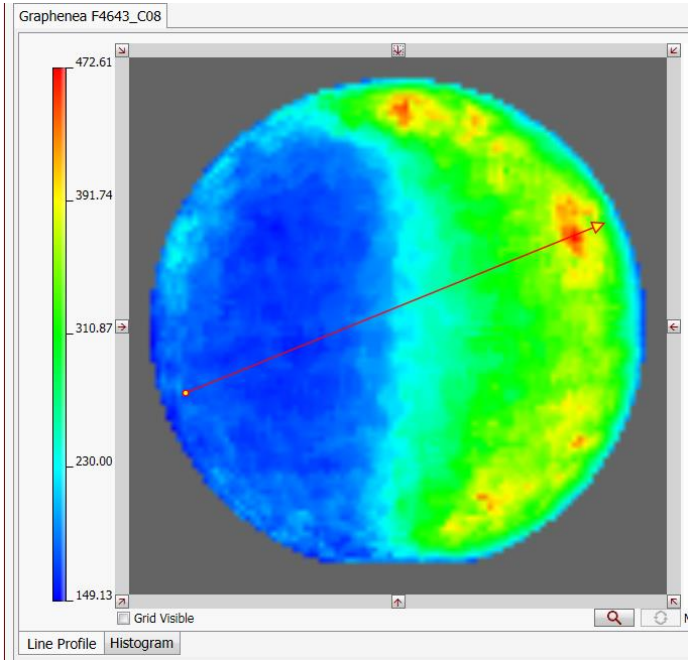
Doping Effectivity

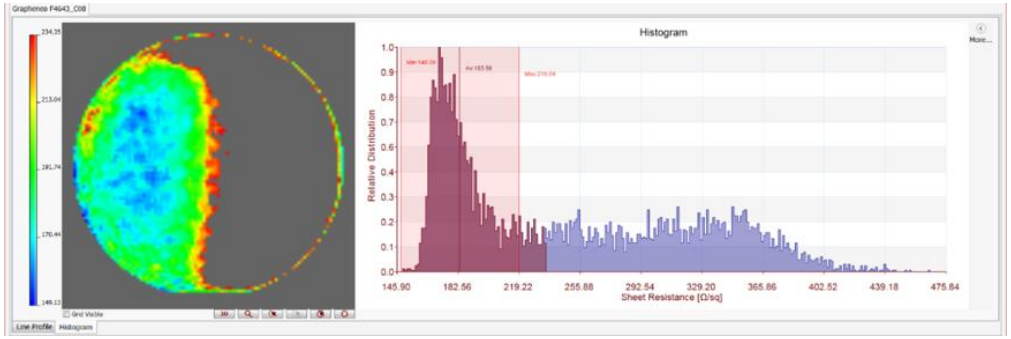
- Doping effectivity
- Doping homogeneity
- Doping stability

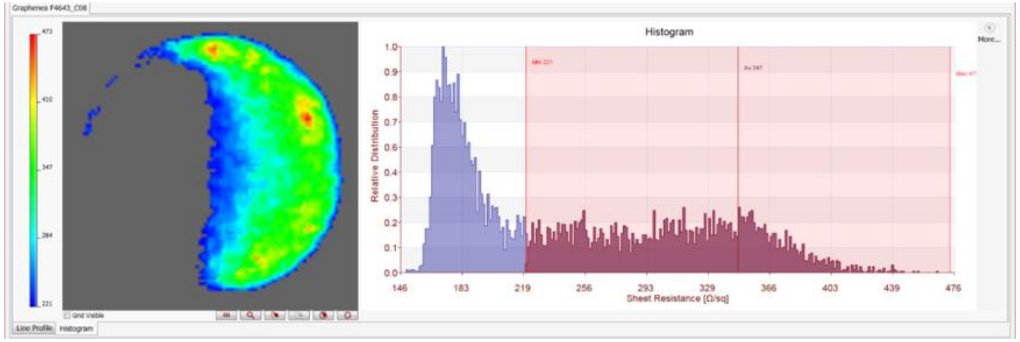


Doping Efficiency Imaging

- Inhomogenious doping

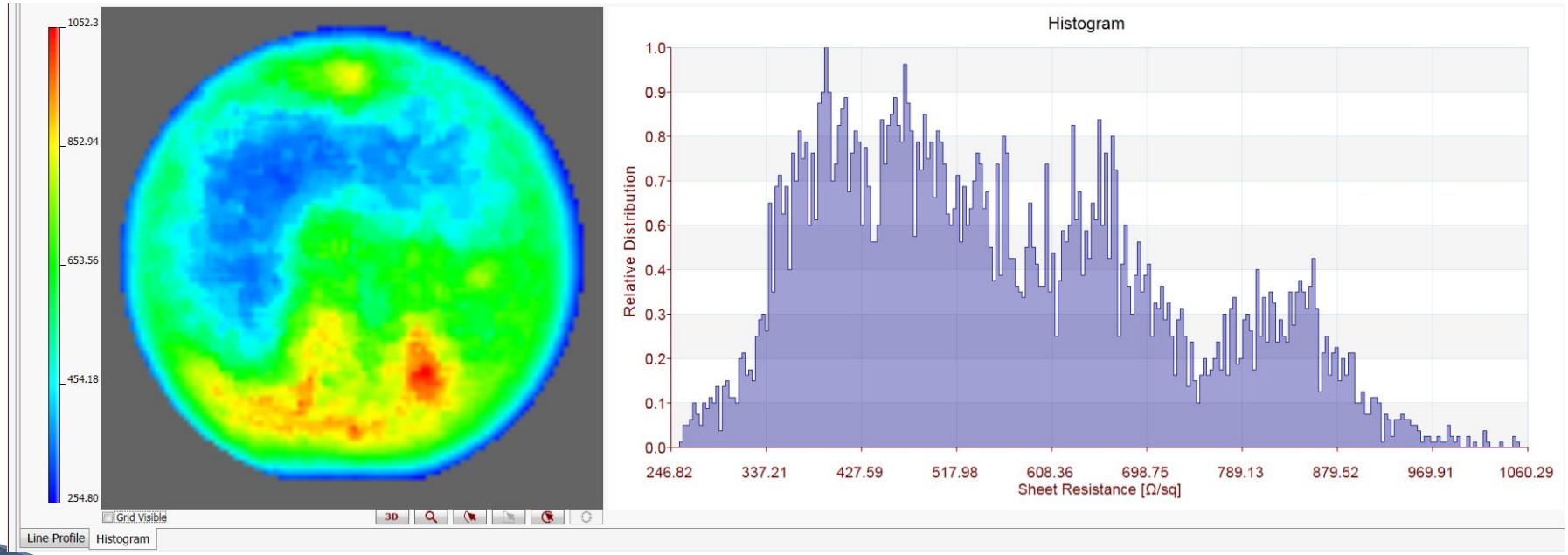




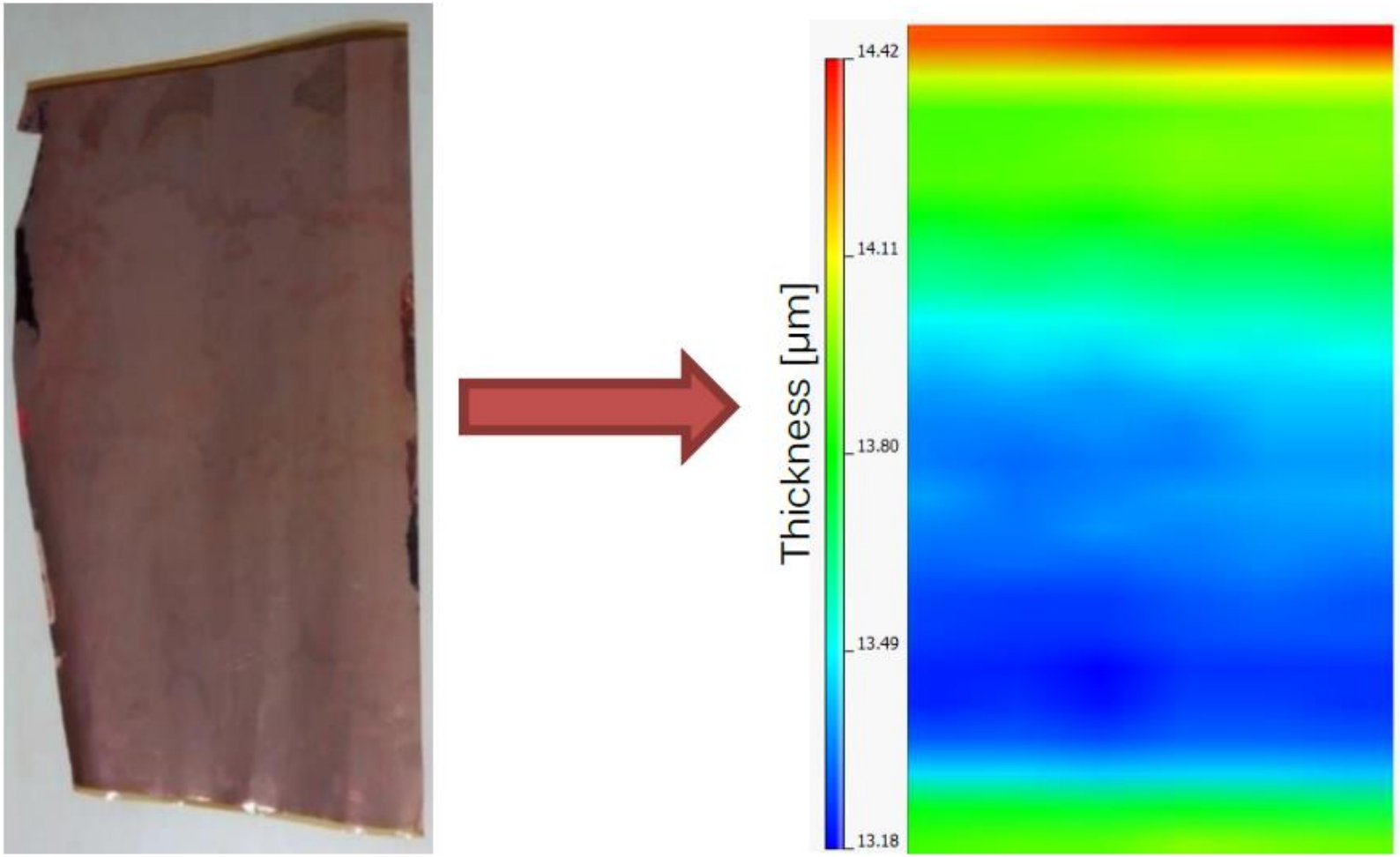


Sheet Resistance Images – Graphene Mappings

- Graphene with local defects on Wafer
- 4 inch
- Measurement pitch 1 mm x 1mm

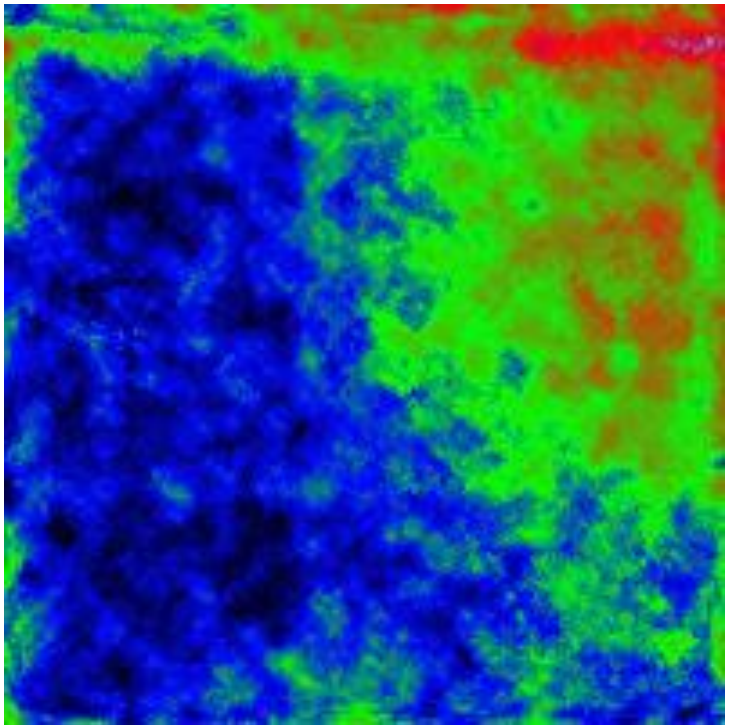


Graphene Imaging on Copper?

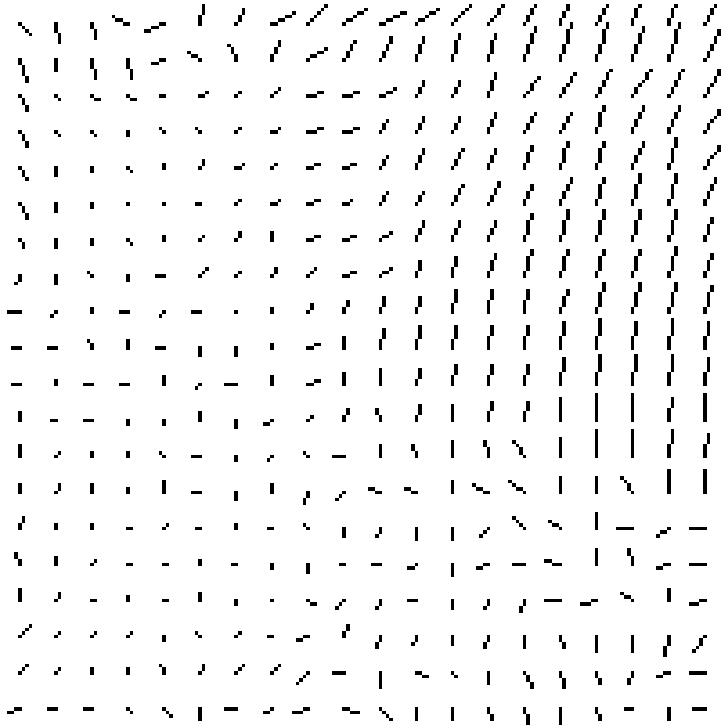


Anisotropy Imaging of Graphene? – Example on Ag-NW

- 200 x 200 mm [8 inch], Measurement pitch 1 mm x 1mm

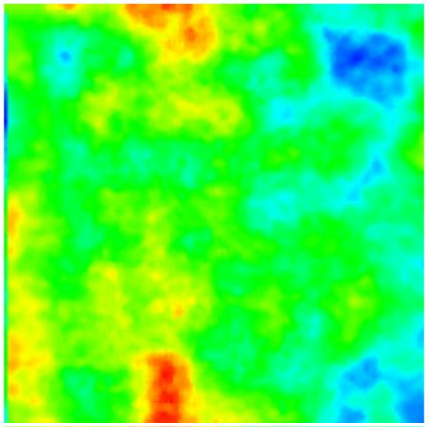


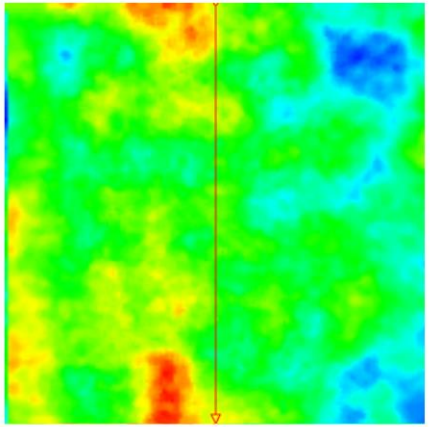
Anisotropy strength(blue=low anisotropy, red=high)

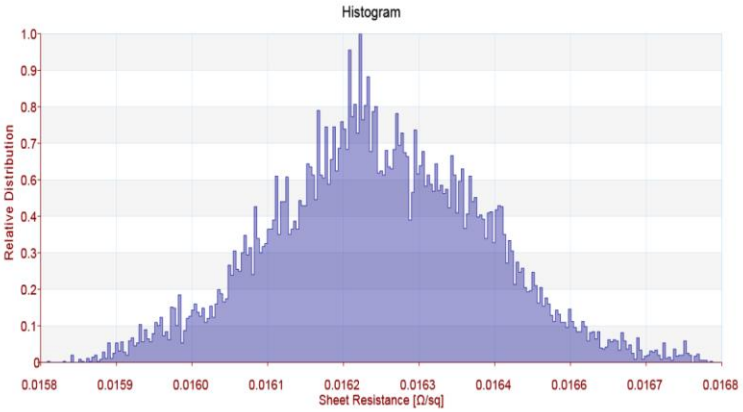


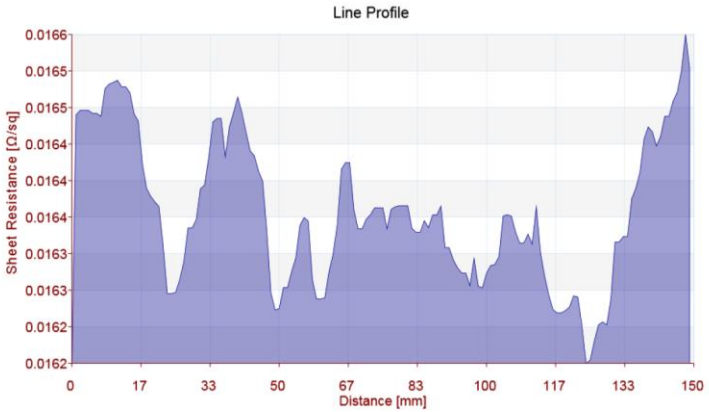
Direction of the least present sheet resistance

Graphite Imaging







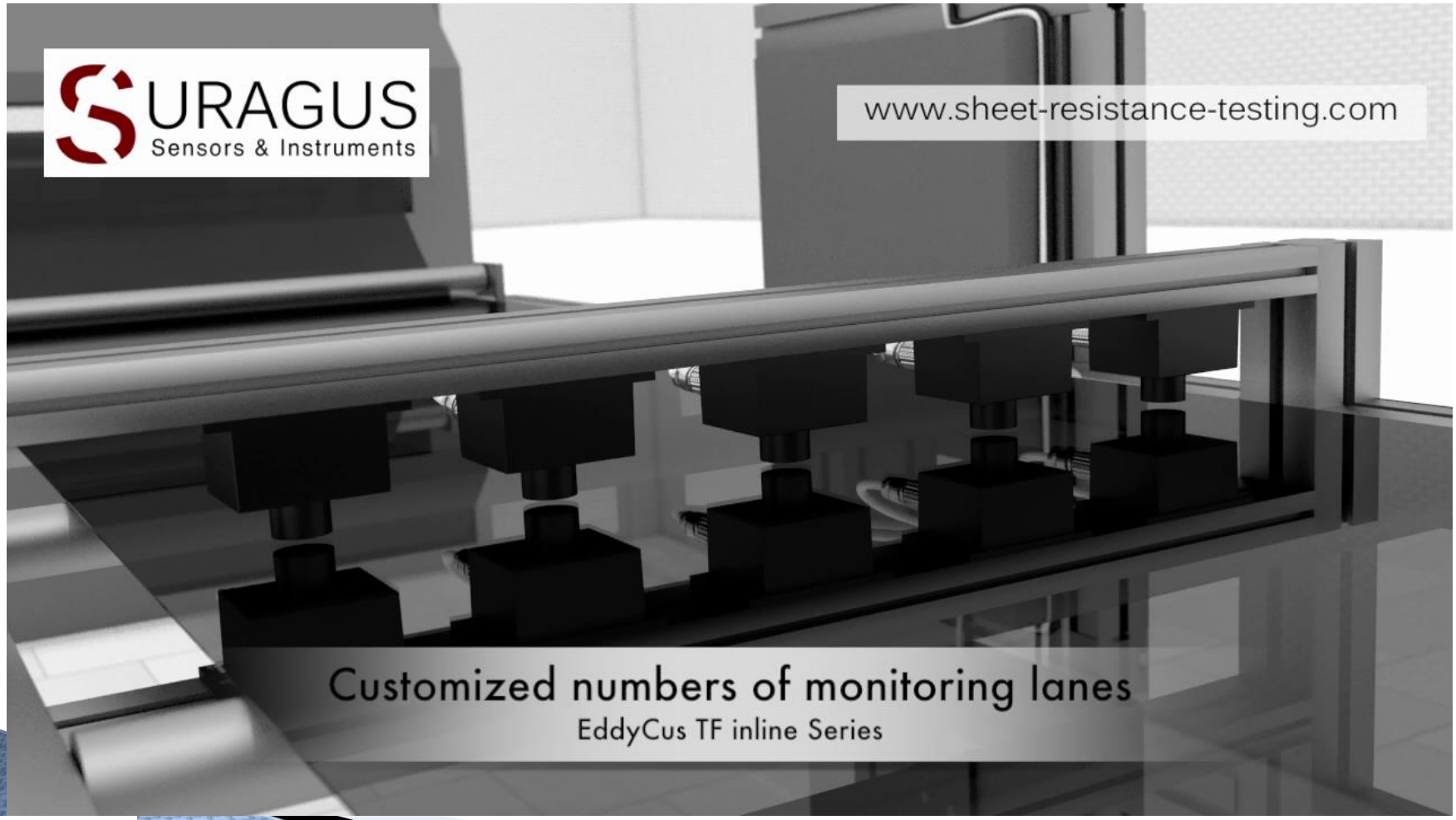


Inline Testing – A Look into the Graphene Testing Future…

Inline Sheet Resistance Measurement

- Non-contact
- Real-time
- QA & Process control after
  - Transfer
  - Cleaning
  - Doping
  - Annealing
  - Potentially stacking
  - Final QA

Demonstration – Future Graphene Inline Testing



Take home messages

- Applications that require complex or sophisticated properties are a focus for Graphene
- TCM was introduced as one example
- There are various characteristics of Graphene – most of them affect electrical performance
- HF eddy current testing provides various benefits for Graphene characterization
  - Non- contact, non-destructive, imaging, defect density assessment
- Defectoscopy by imaging solution 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

Any Questions?

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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!!

SURAGUS GmbH
Maria-Reiche-Straße 1
D-01109 Dresden

E-Mail: info@suragus.com
Web: www.suragus.com

Phone: +49 351 – 32 111 520
Fax: +49 351 – 32 111 509

Or Visit us in Dresden – e.g. at Graphene 2018