



RPGR 2017
SINGAPORE
SEPTEMBER 19-22, 2017 - SINGAPORE

RECENT PROGRESS IN GRAPHENE
& 2D MATERIALS RESEARCH



SURAGUS
Sensors & Instruments

Challenges & Insights By Characterization Of Large Area Graphene

RPGR2017

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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'énergie atomique et aux énergies alternatives (France)
- ▶ Amcor Flexibles Kreuzlingen AG (Switzerland)
- ▶ Amcor Flexibles Singen GmbH (Germany)
- ▶ Leibniz-Institut für Oberflächenmodifikation (Germany)
- ▶ Det National Forskningscenter Forarbejdsmiljø (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

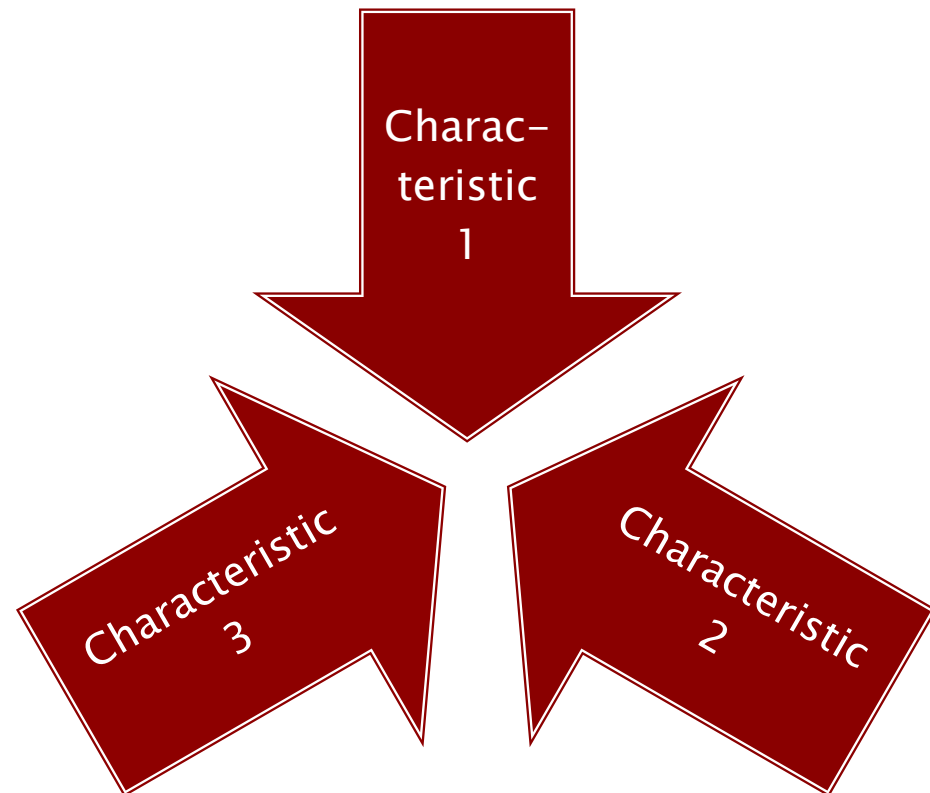
- Electrical properties
- Mechanical properties
- Chemical properties
- Optical properties
- Etc.

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

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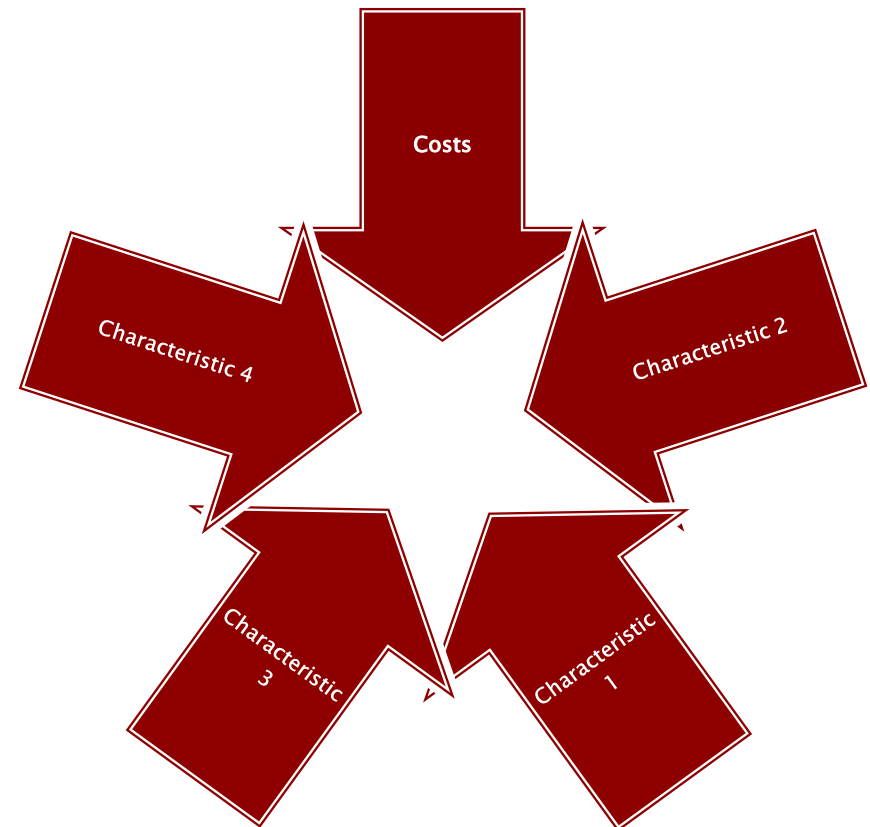
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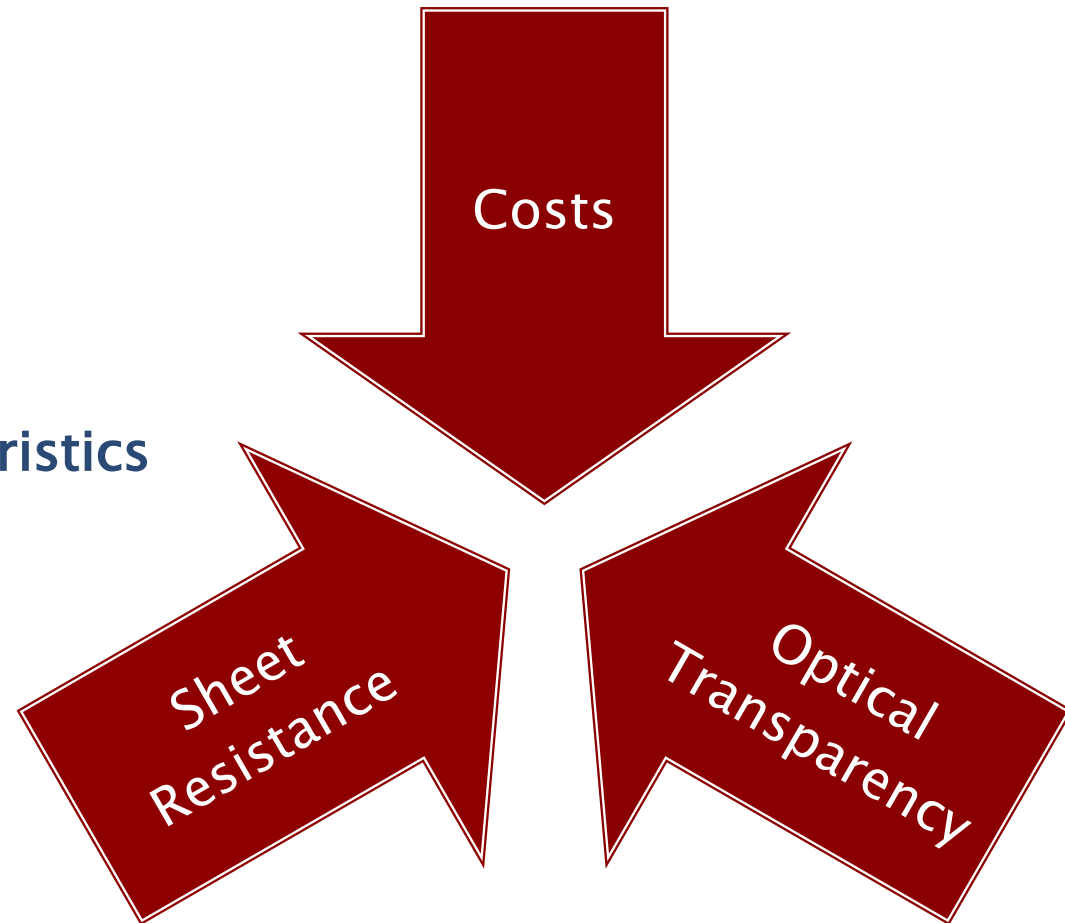
Example: Graphene as transparent electrode

► Requirements/ trends

- Low cost
- Low sheet resistance
- High transparency

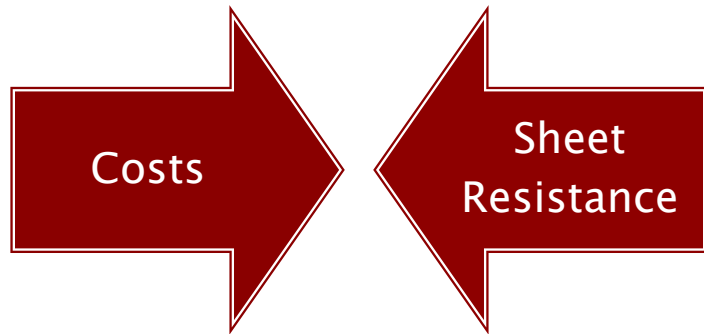
► Beneficial/ further characteristics

- Ability for flexible substrates
- Low aging effects



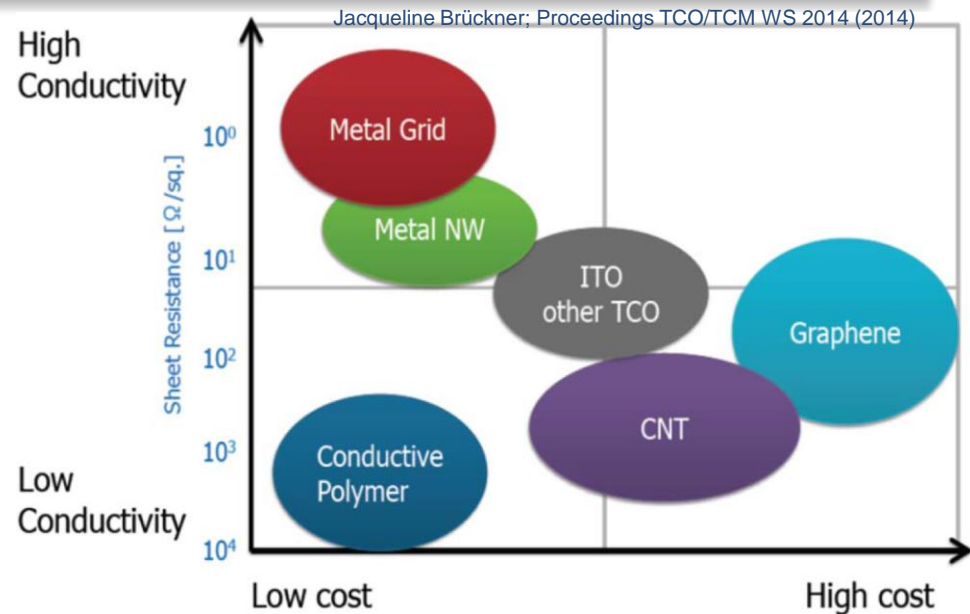


Drivers & Challenges in Manufacturing



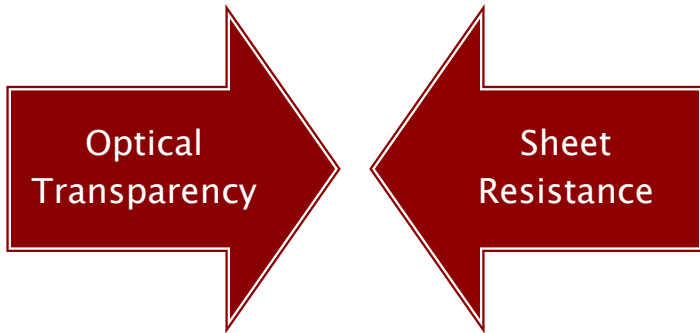
Cost improvement

- 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 eg. reuse of growth substrates



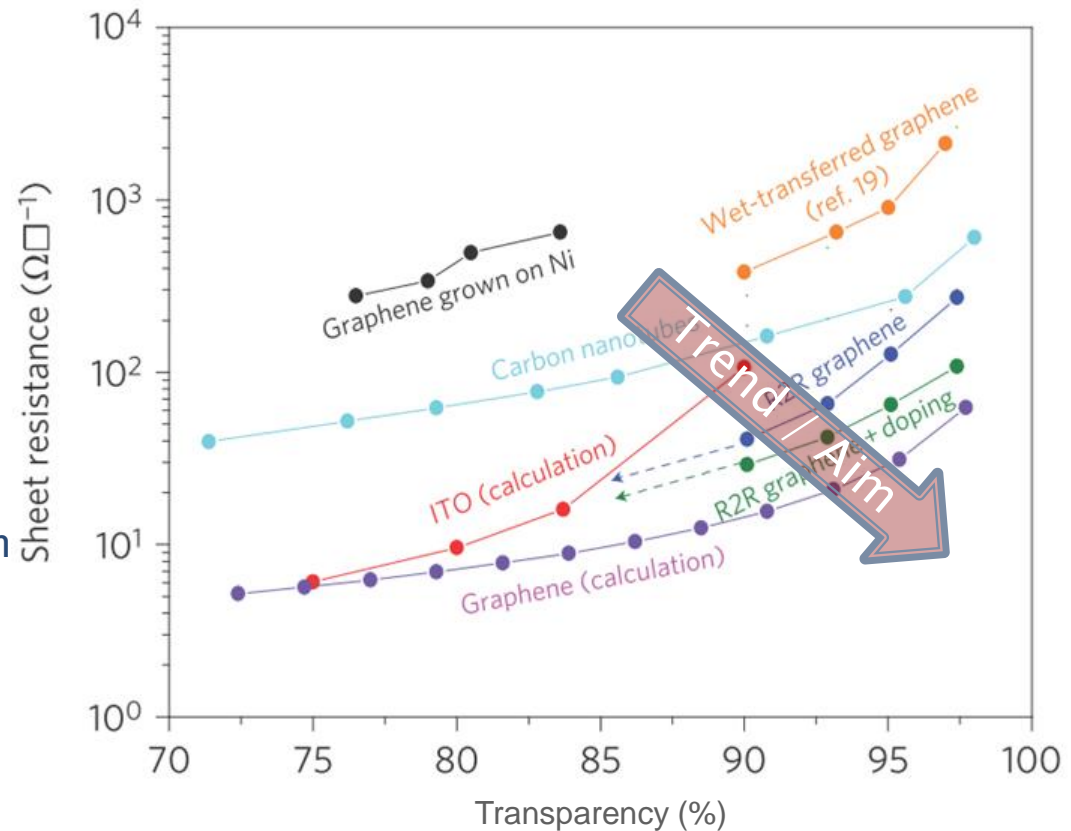


Graphene as transparent electrode



Main Parameter

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

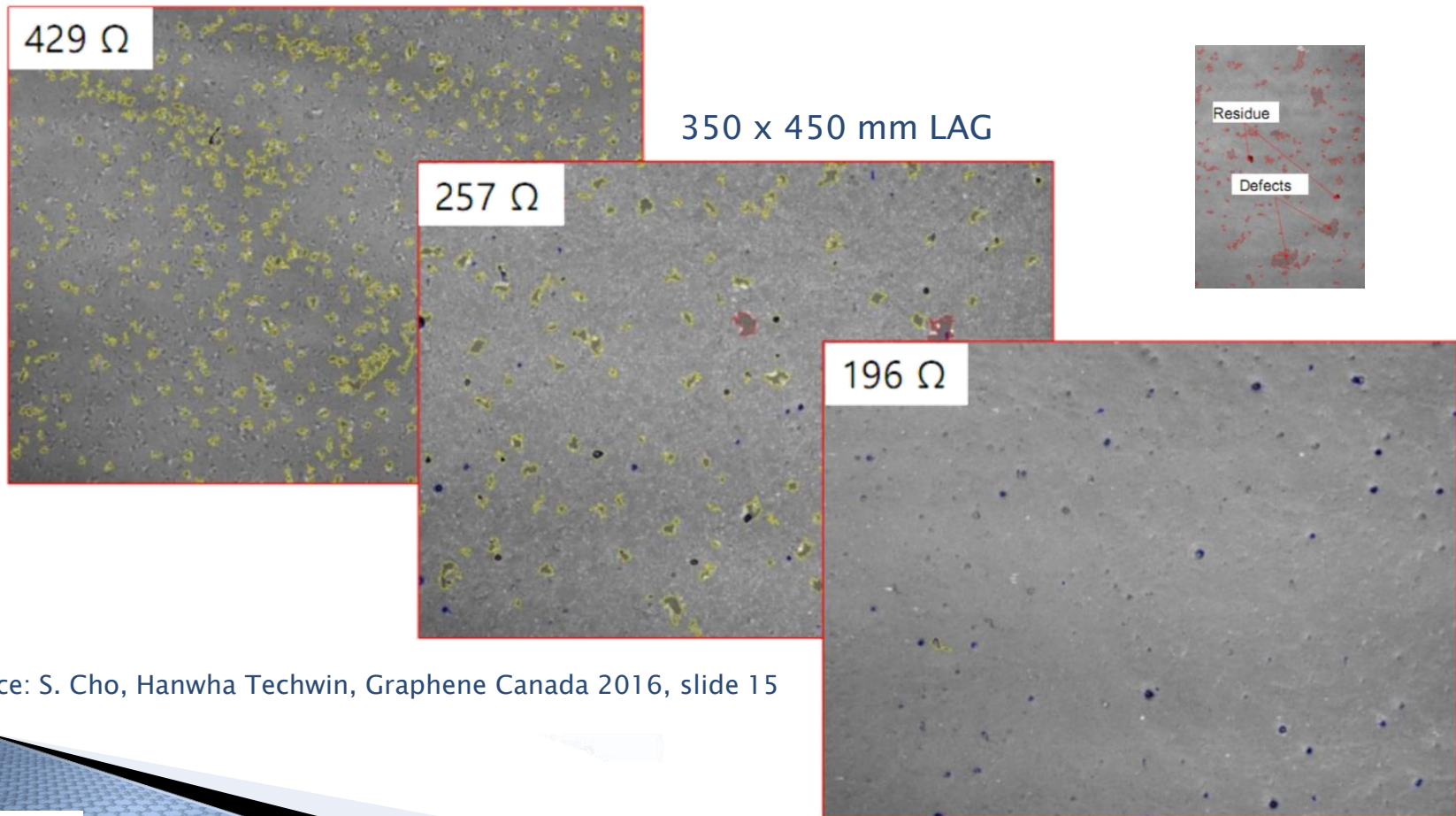


S. Bae et al. Nature Nano. 5, 571 (2010);



Effects of Defects to the Sheet Resistance

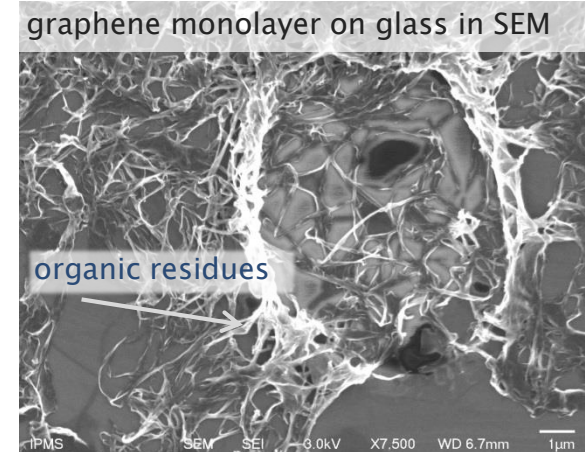
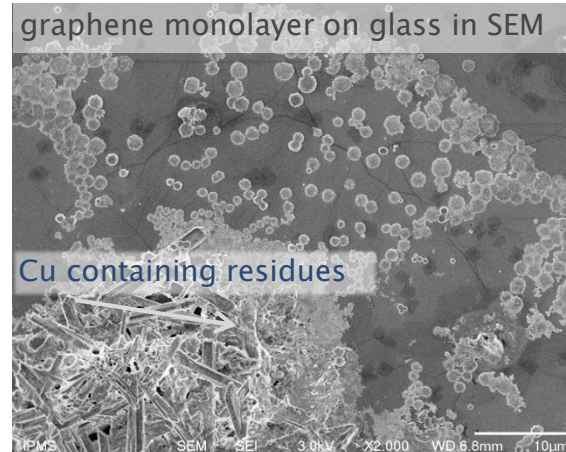
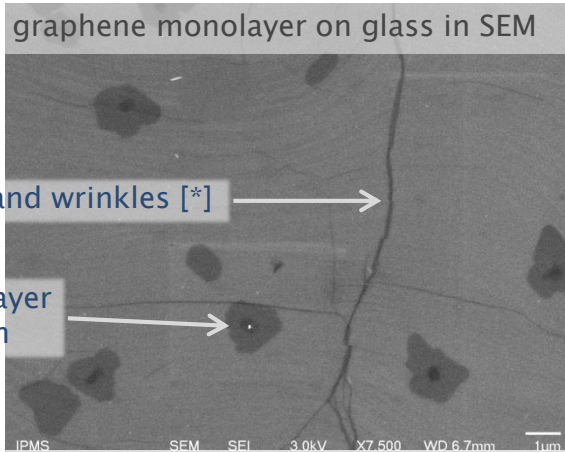
- The defect density significantly affects the sheet resistance



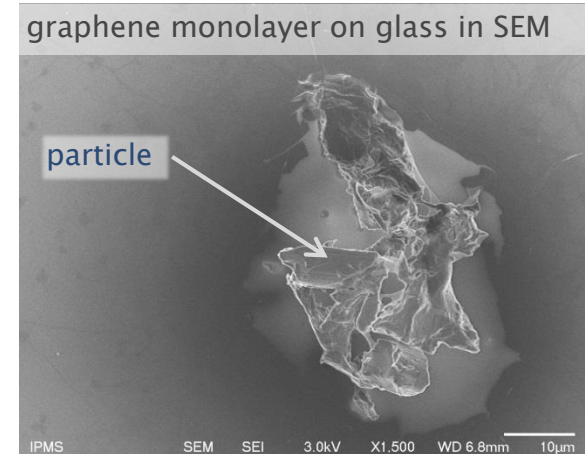
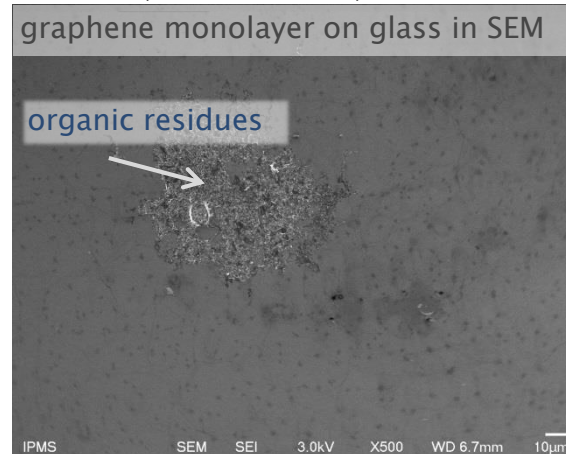
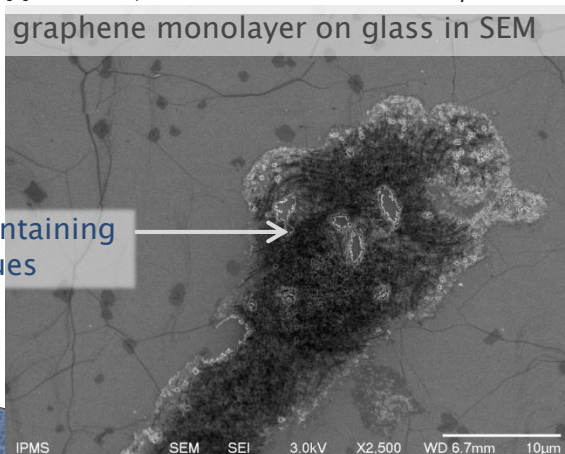
Source: S. Cho, Hanwha Techwin, Graphene Canada 2016, slide 15



Typical Defects in Graphene



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

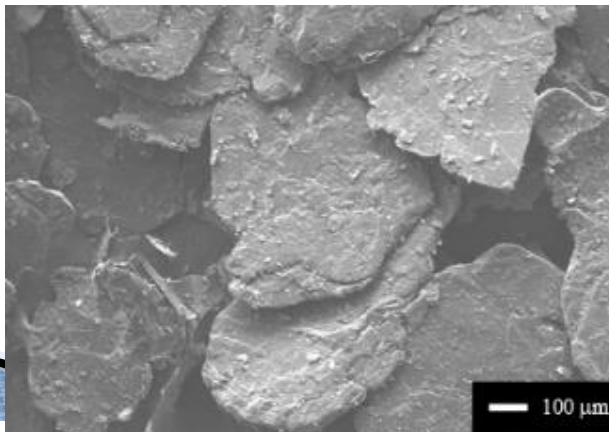
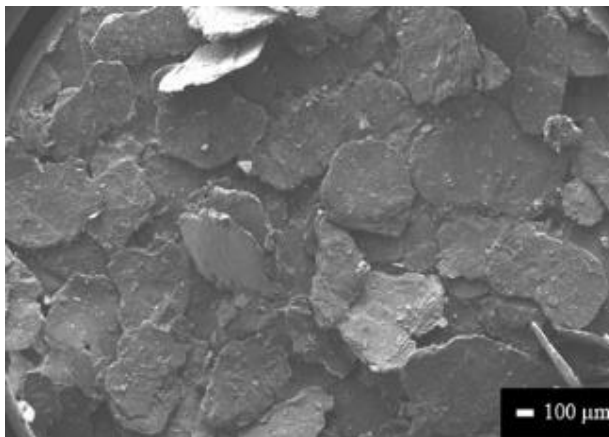


Source: D. Waynolds et al., Fraunhofer FEP



Properties and common defects

- Flake size



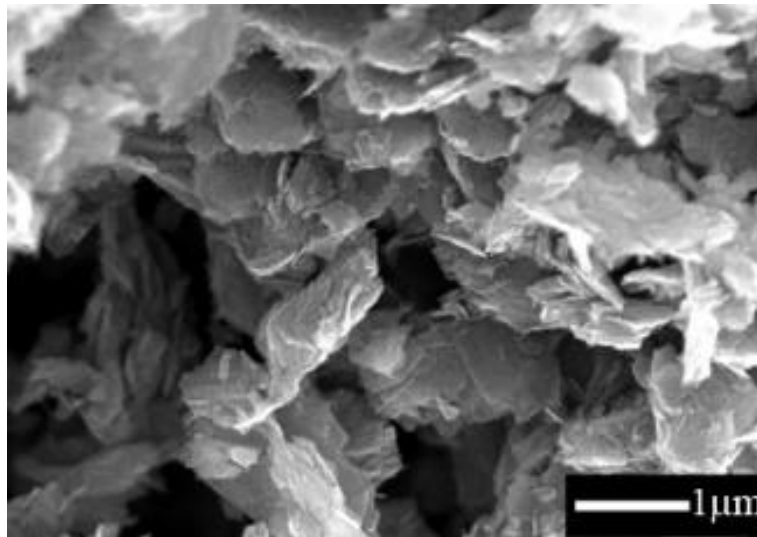
} Effect on sheet resistance

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
<ul style="list-style-type: none"> ▪ Homogeneity 	<ul style="list-style-type: none"> ▪ 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 	<ul style="list-style-type: none"> ▪ Quantification in a range of 10 Ohm/sq to 3000 Ohm/sq ▪ Homogeneity <ul style="list-style-type: none"> ▪ How to define it 	<ul style="list-style-type: none"> ▪ Optical transparency from UV up to IR wavelengths ▪ Quantification ▪ Range: Transmittance from 80 - 97.7% or absorbance ▪ Homogeneity ▪ How to define it ▪ Other optical parameters such as haze 	<ul style="list-style-type: none"> ▪ 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
- Terahertz spectroscopy
- 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

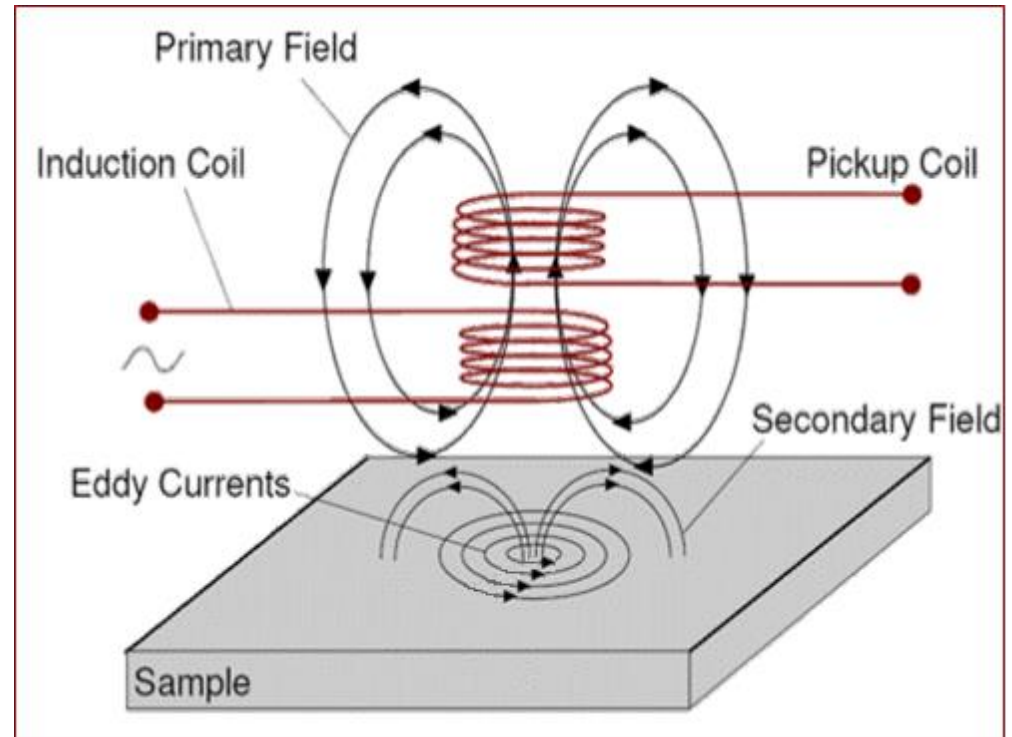
- Contact quality artifacts
- Possible damage to sensitive layers
- No measurement of encapsulated films
- Wearing of probe with time

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



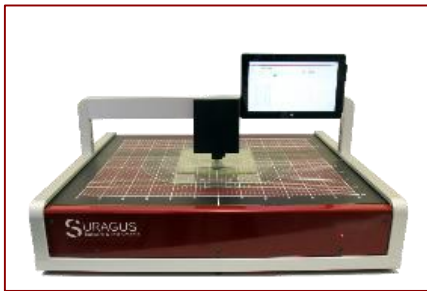
- + Non-contact
- + High sample rate
- + High sensitivity

– Limited to conductive materials



General testing types

Single Point Testing

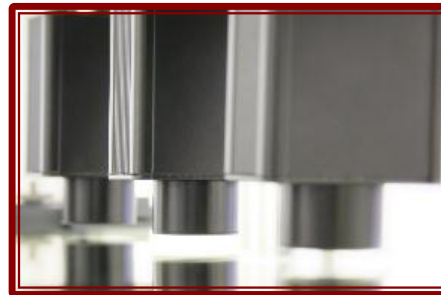


EddyCus® TF lab Series

Sheet resistance & OT

- After transfer
- Doping
- Annealing
- Ageing

Inline Testing



EddyCus® TF inline Series

Sheet resistance & OT

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

Imaging solutions



EddyCus® TF map Series

Sheet resistance & OT imaging

Defect detection

- Impurities,
- deposition effects
- Many more



Demonstration – Single point measurement

<http://www.files.suragus.com/videos/Non-Contact-Sheet-Resistance-Measurement-Device-EddyCus-TF-lab-2020.mp4>



EDDYCUS TF LAB 2020
**NON-CONTACT
SHEET RESISTANCE TESTER**

SURAGUS Sensors & Instruments EddyCus® TF lab 2020
Sheet Resistance Measurement Device

EddyCus® TF lab 2020

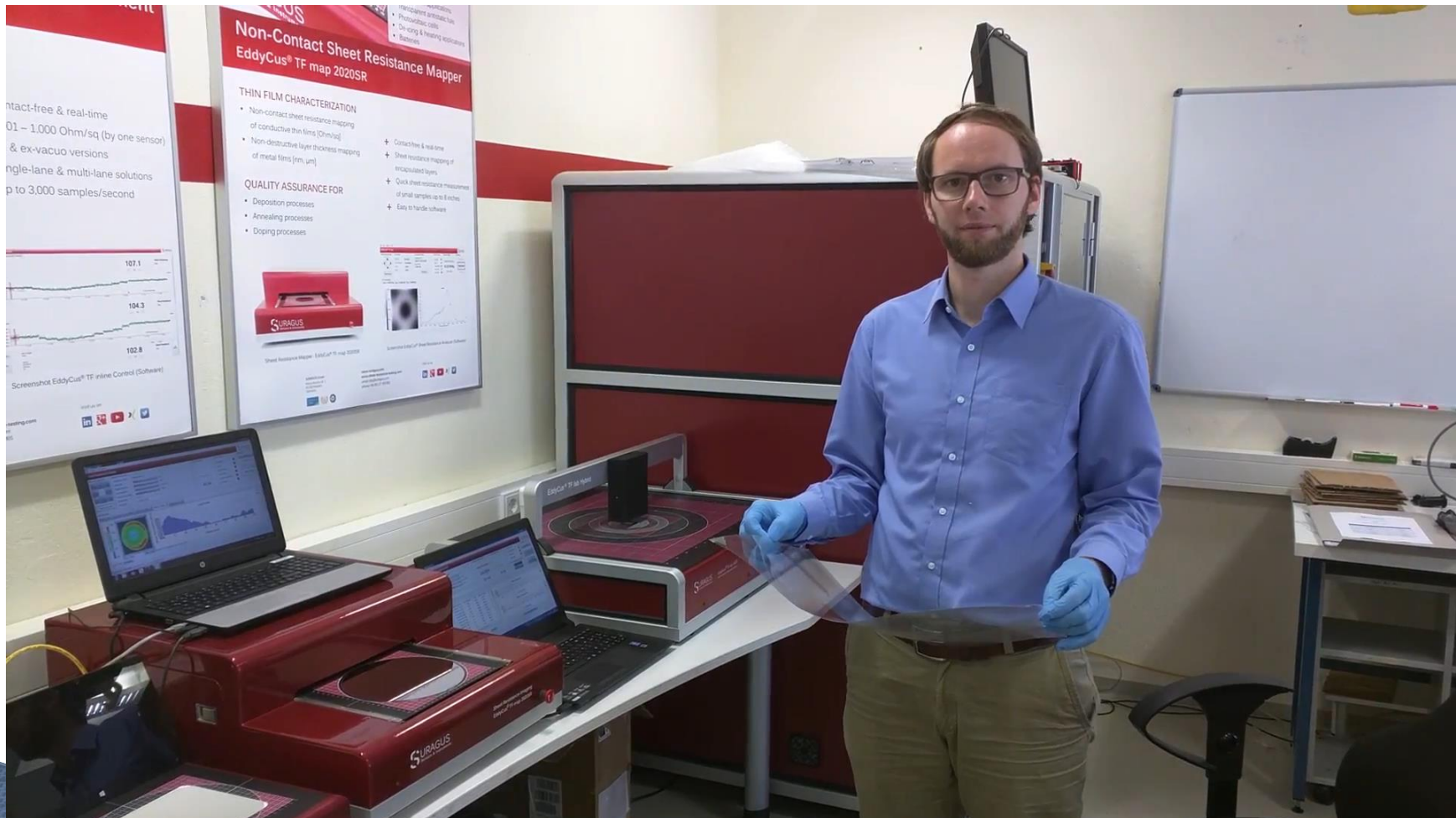


Demonstration – manual mapping





Simultaneous manual mapping of sheet resistance and optical transparency of large area graphene (LAG)



Watch movie on youtube [here](#).

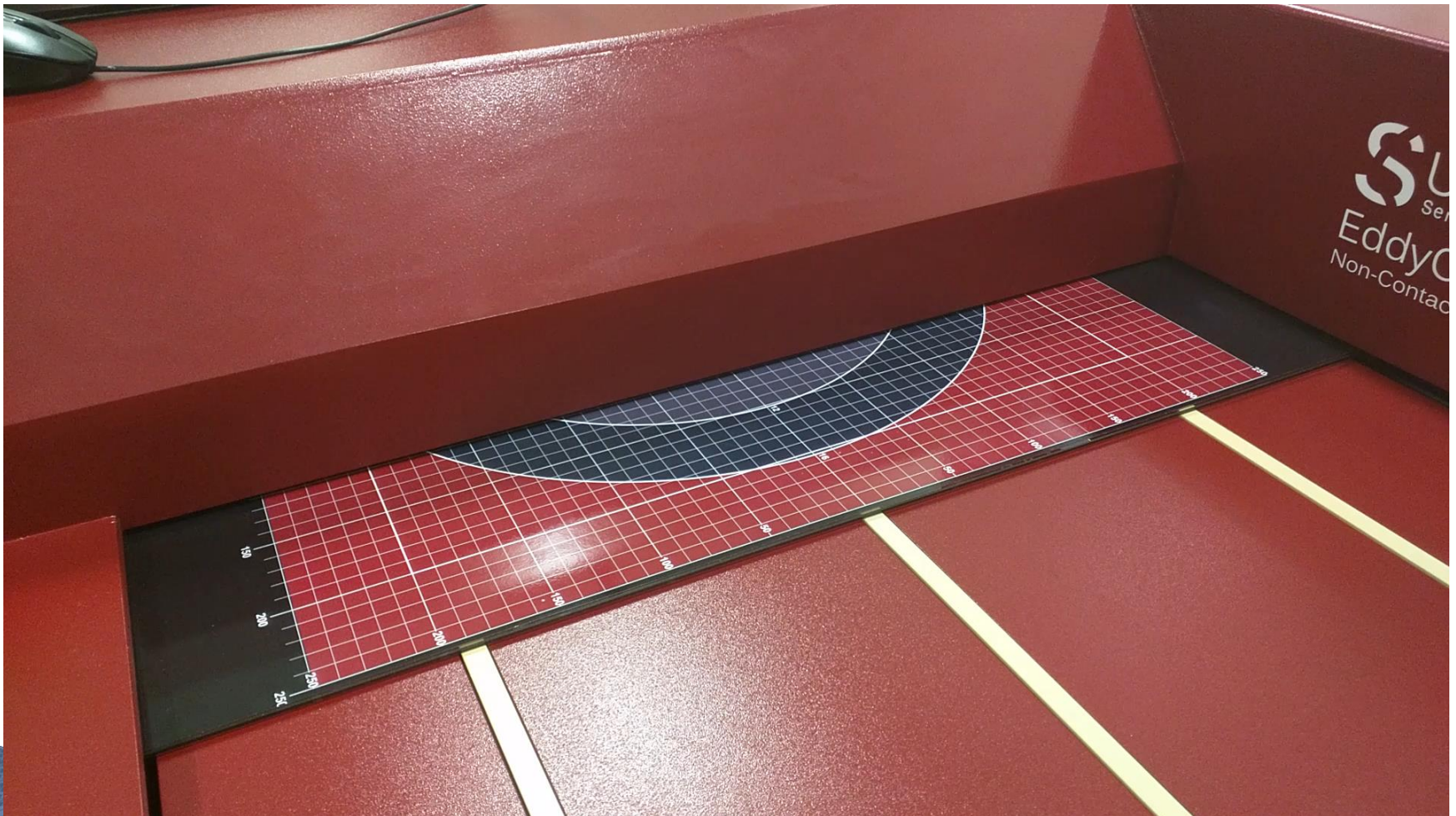


Demonstration of electrical imaging





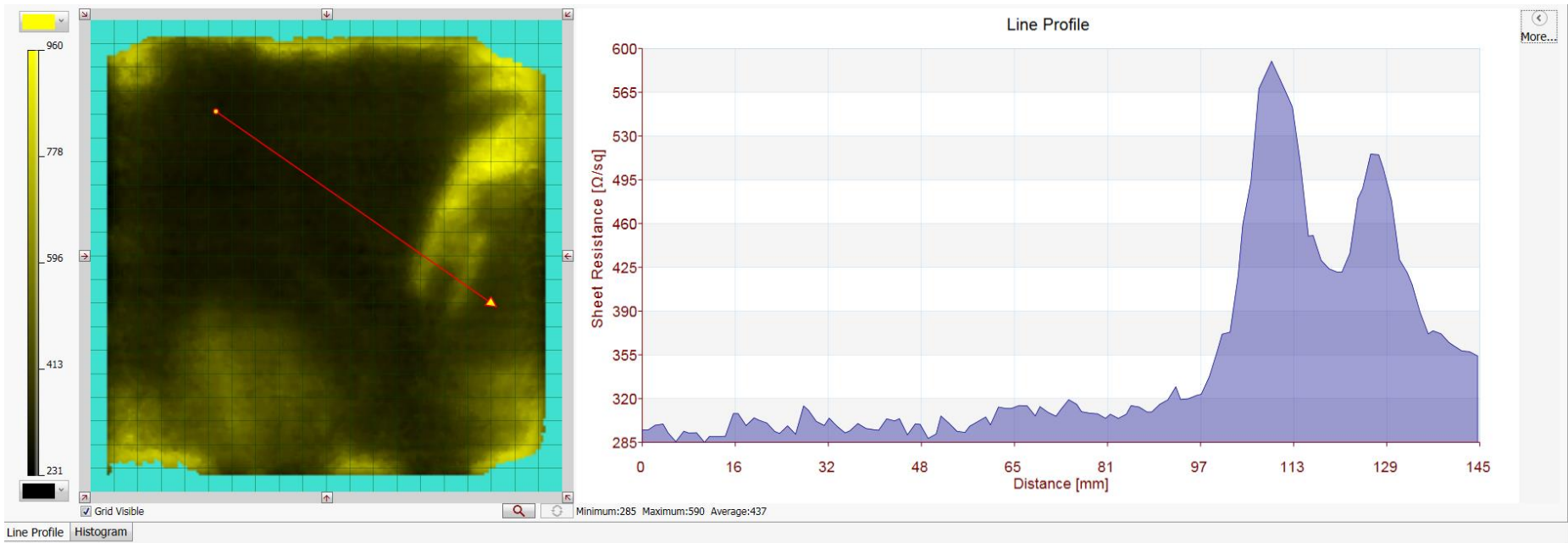
Demonstration of sheet resistance imaging of large area graphene





Sheet resistance imaging analysis – defectoscopy

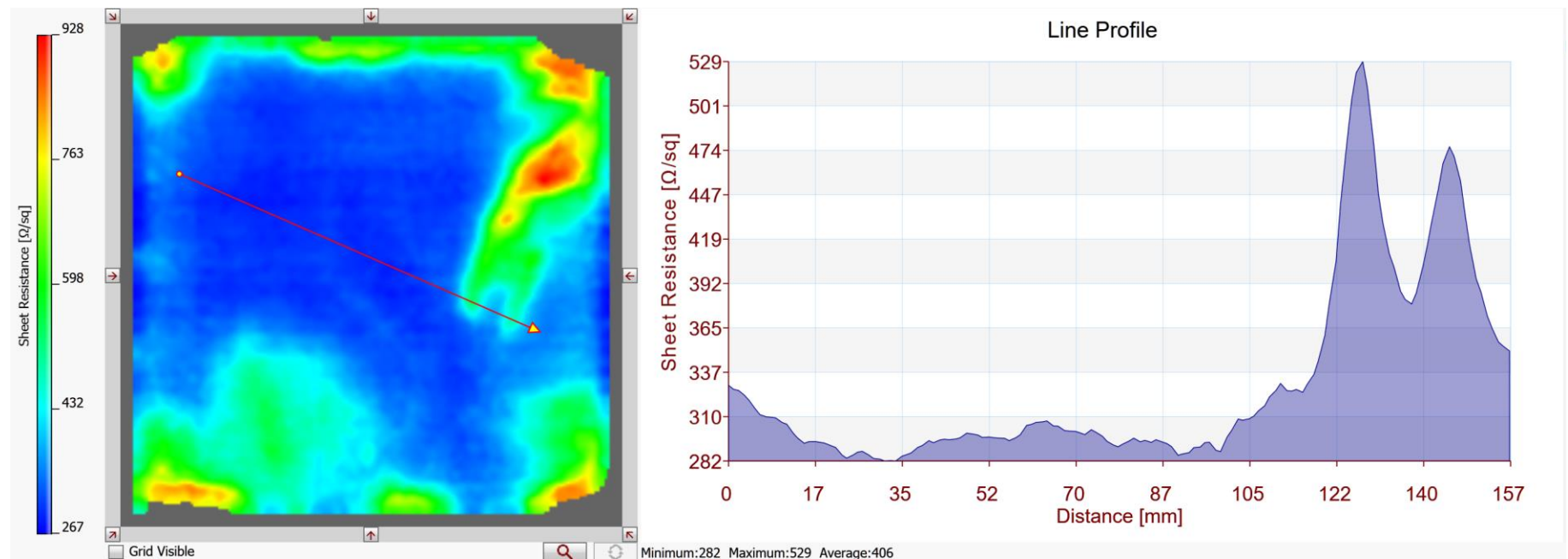
Sheet resistance imaging [Ω/sq] on 200 x 200 mm





Sheet resistance imaging analysis – defectoscopy

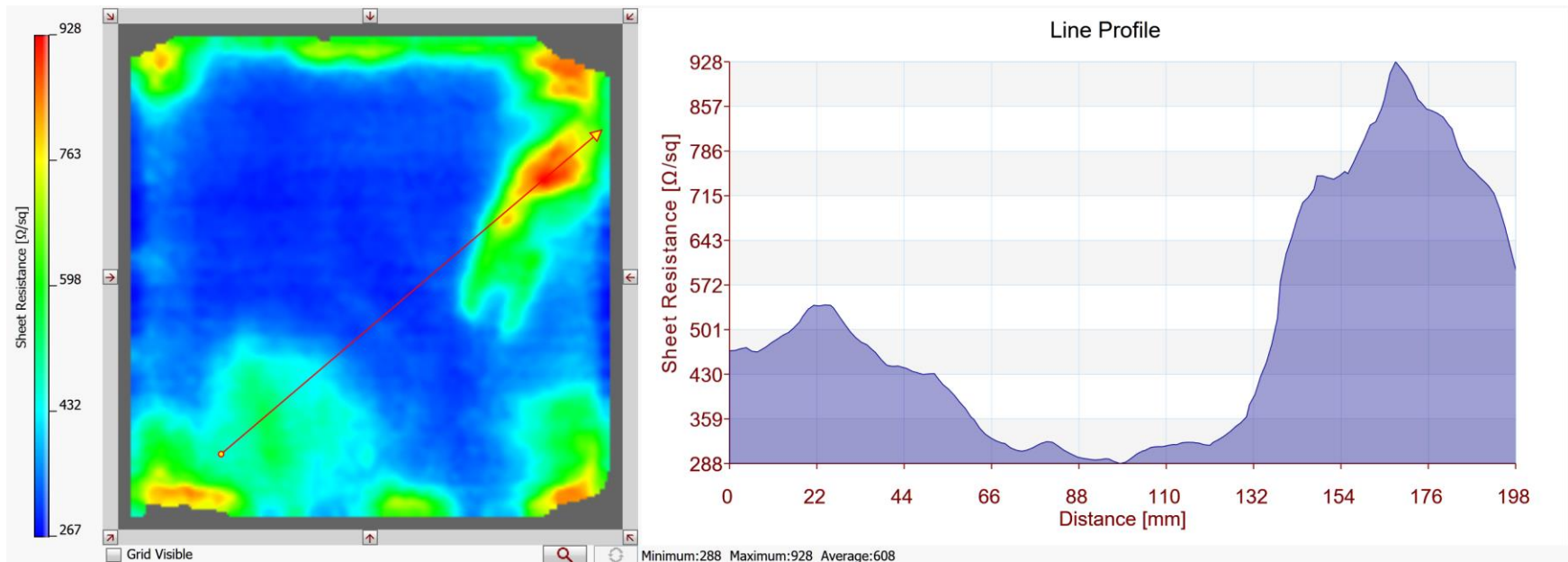
Sheet resistance imaging [ohm/sq] on 200 x 200 mm





Sheet resistance imaging analysis – defectoscopy

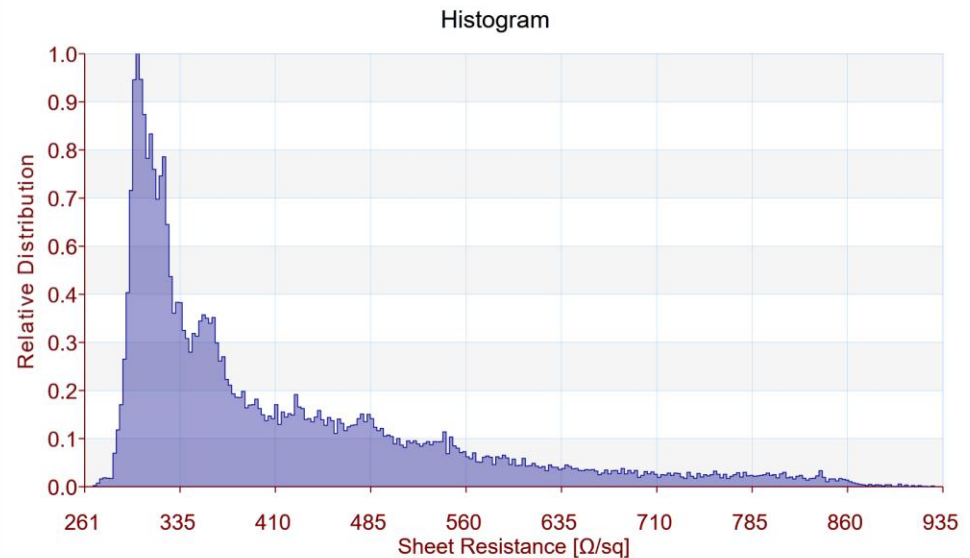
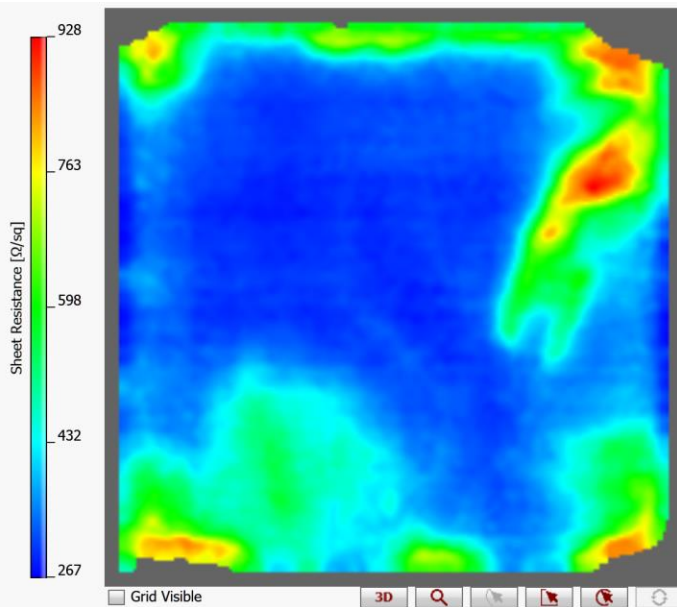
Sheet resistance imaging [ohm/sq] on 200 x 200 mm





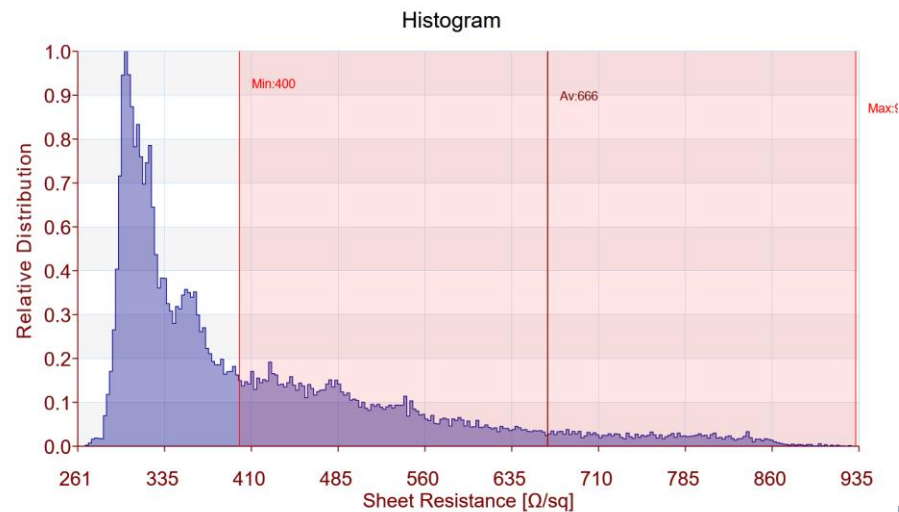
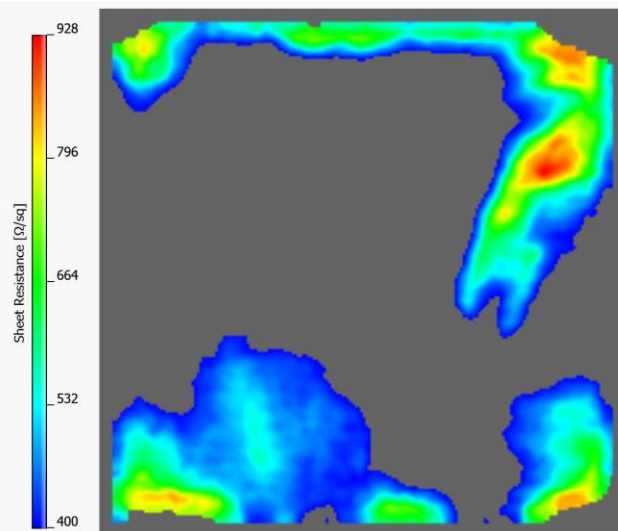
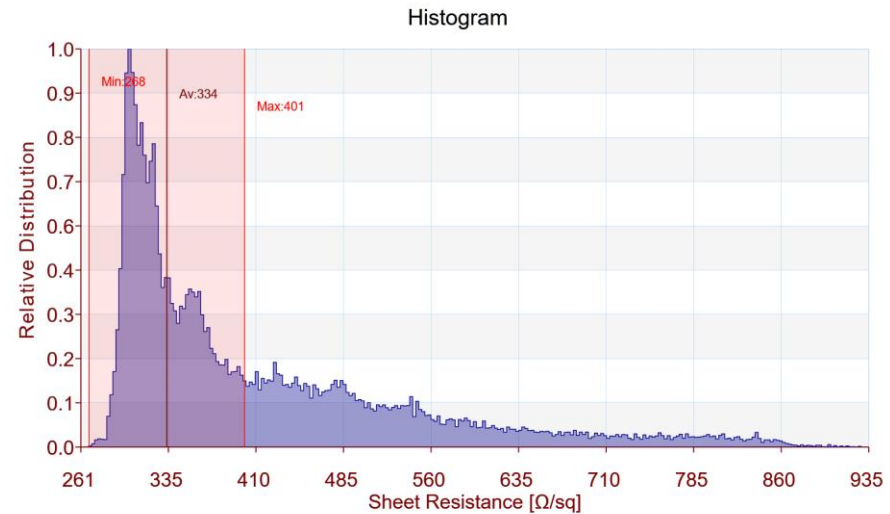
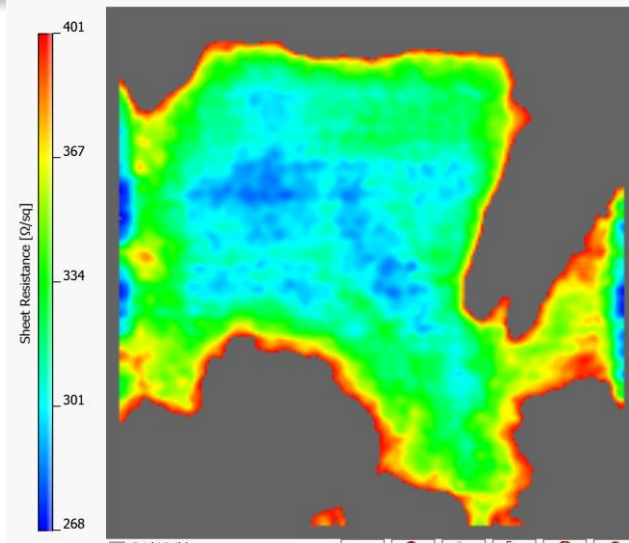
Sheet resistance imaging analysis – defectoscopy

Sheet resistance imaging [Ω/sq] on 200 x 200 mm



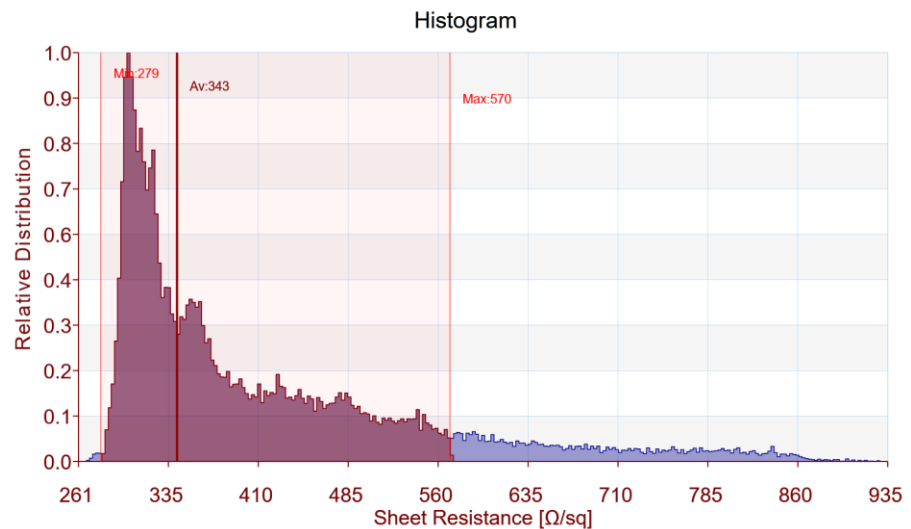
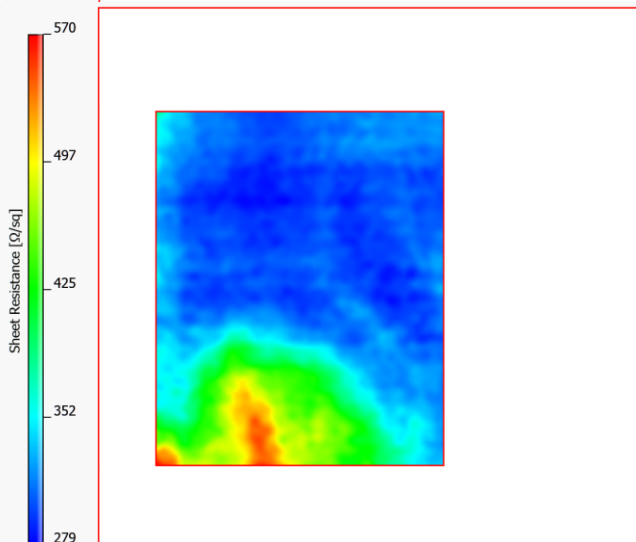
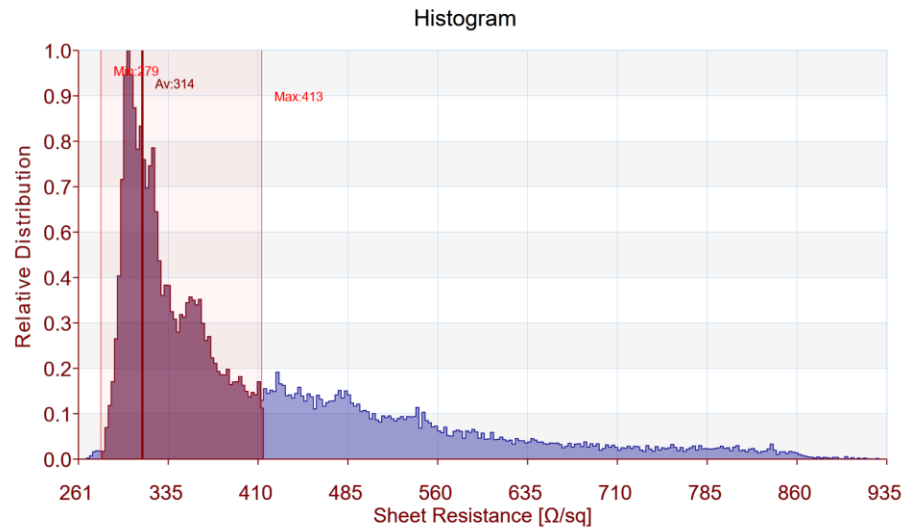
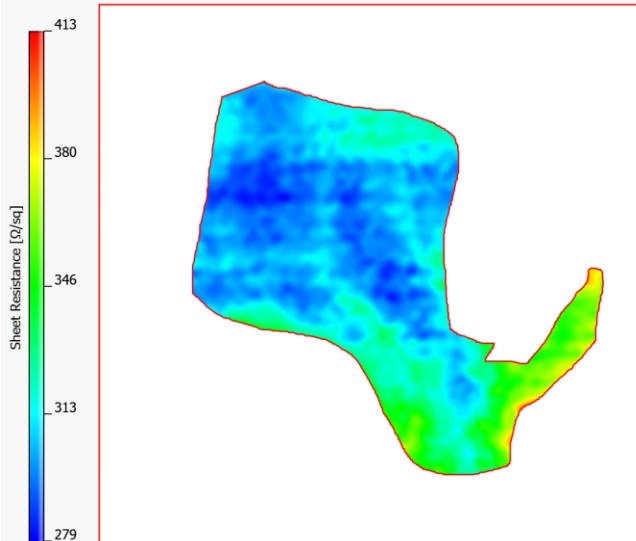


Sheet resistance imaging analysis – defectoscopy





Sheet resistance imaging analysis – defectoscopy

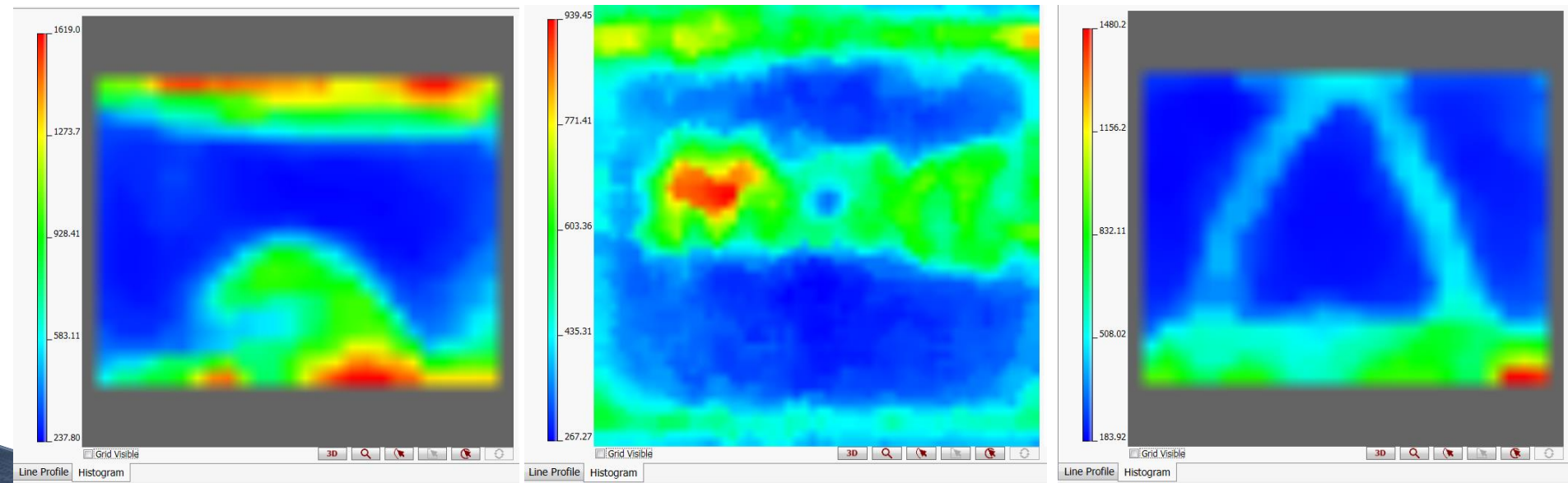




Missing Graphene, holes and cracks

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

Sheet resistance mappings [ohm/sq] on ~ A4

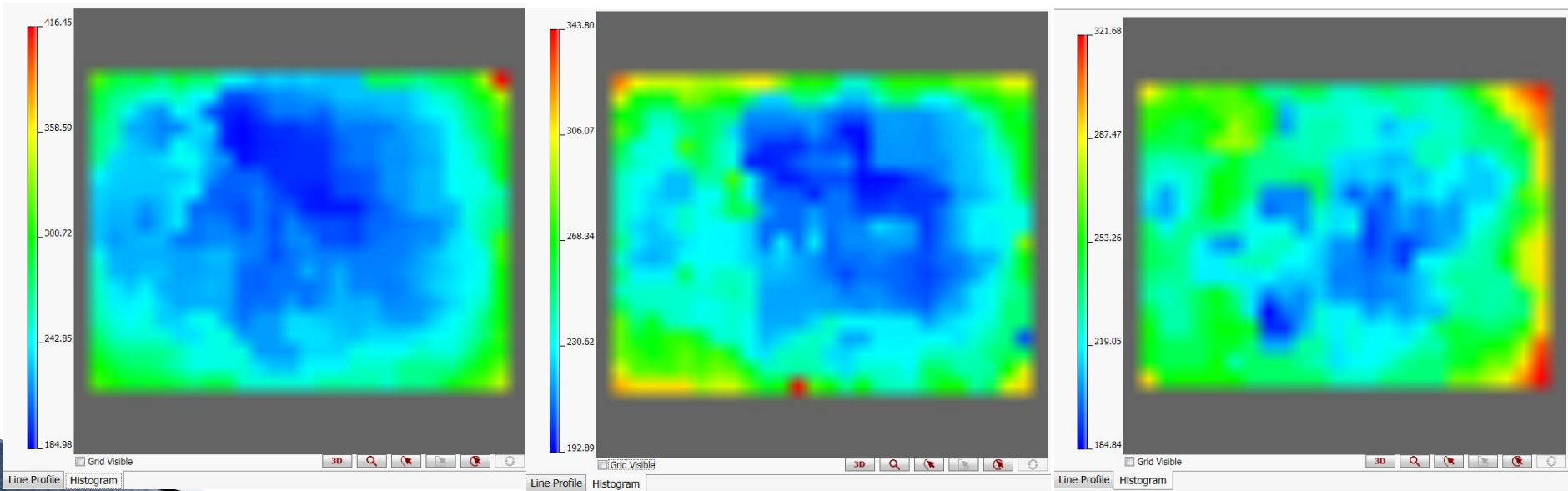




Small inhomogeneity

- Homogeneity assessment

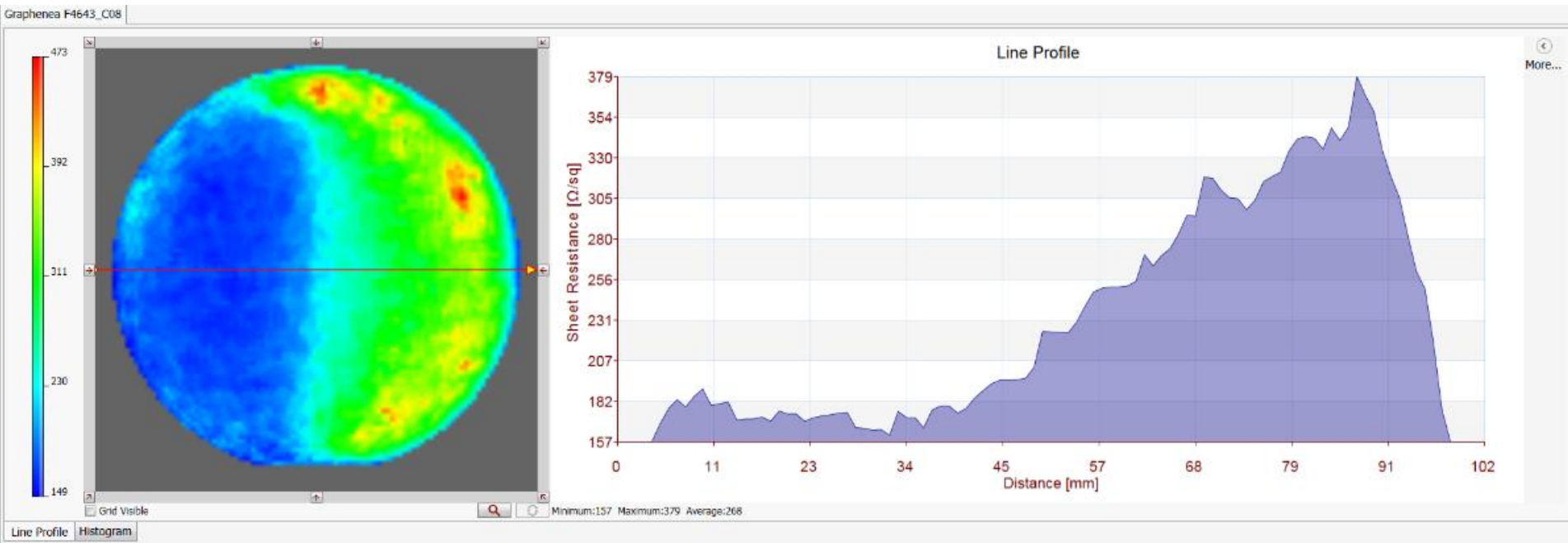
Sheet resistance mappings [ohm/sq] on ~ A4





Doping effectivity

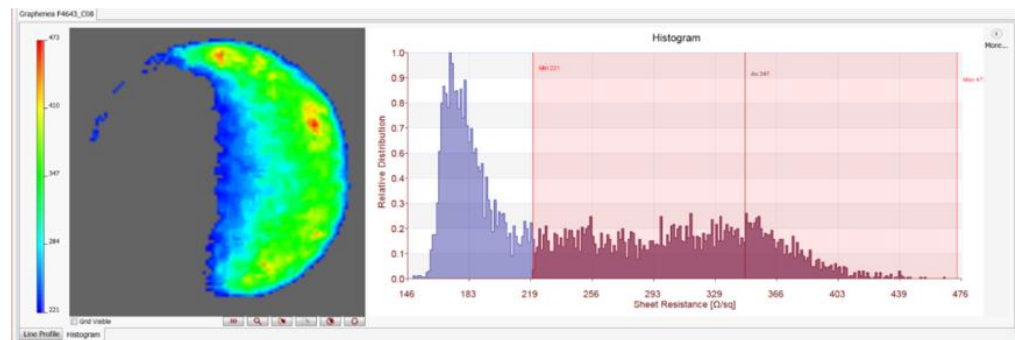
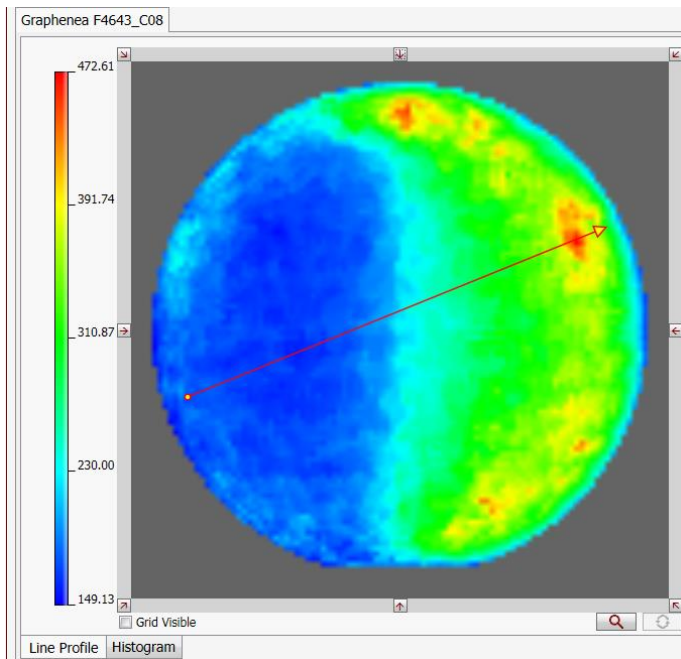
- Doping effectivity
- Doping homogeneity
- Doping stability





Doping Efficiency Imaging

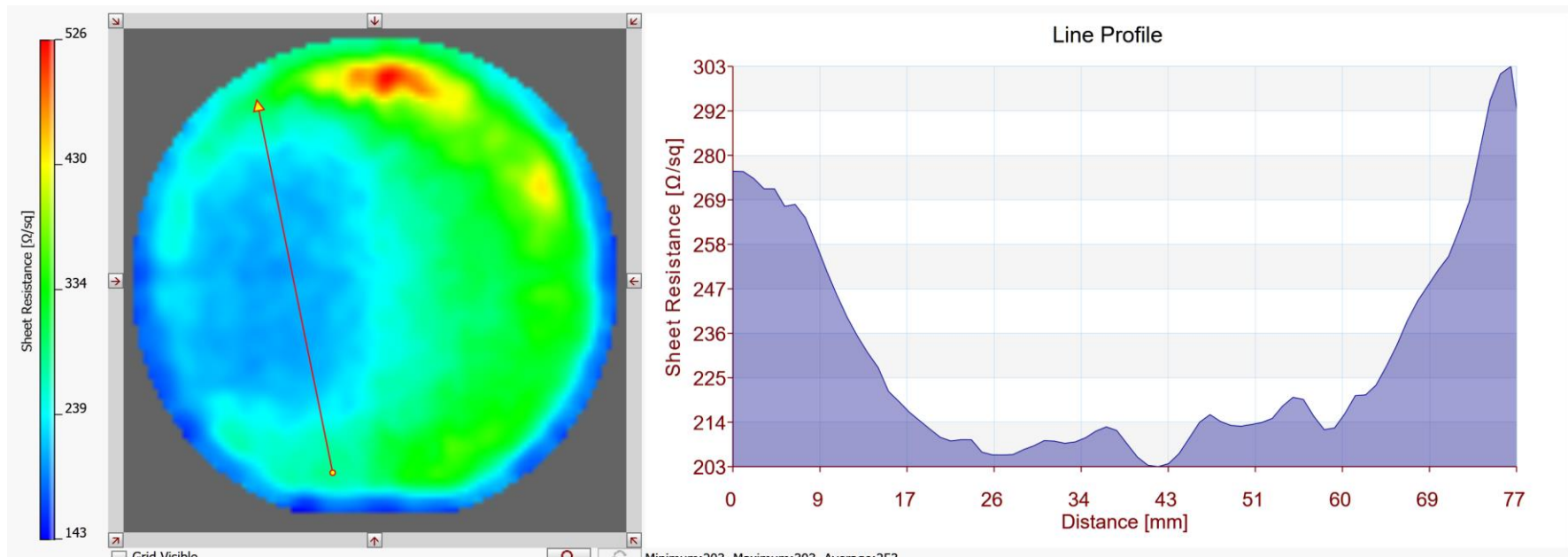
- Inhomogeneous doping





Doping stability imaging

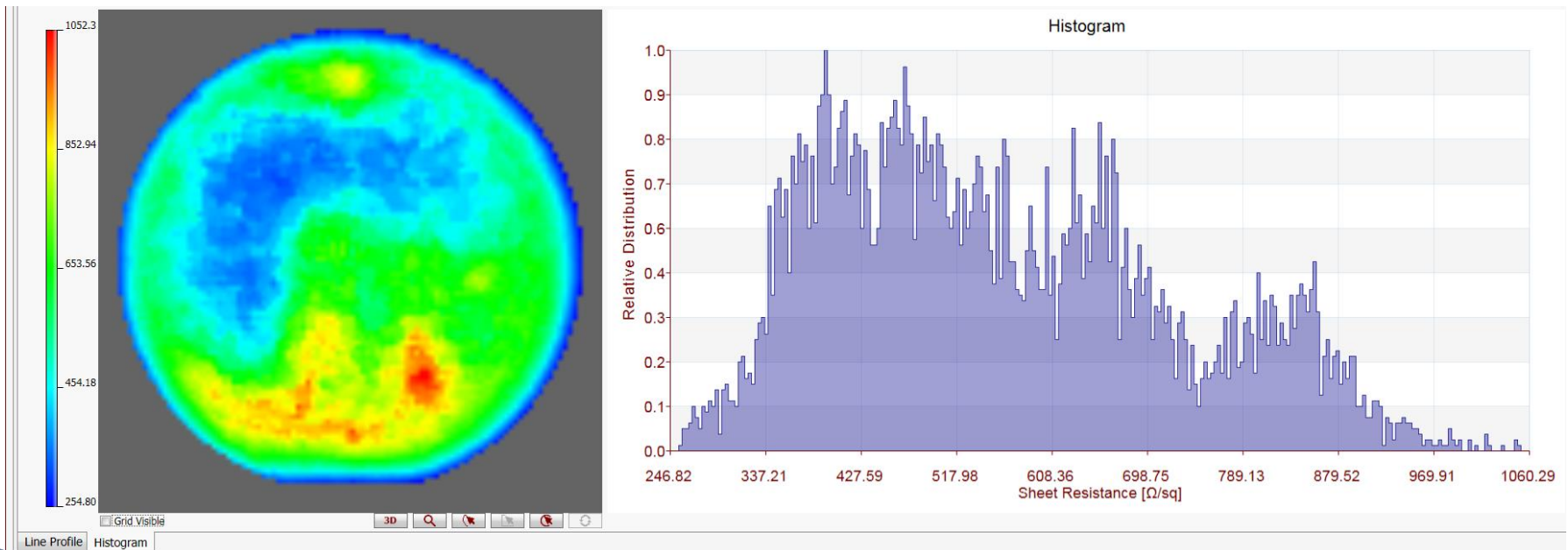
- Doping – measurement after aging





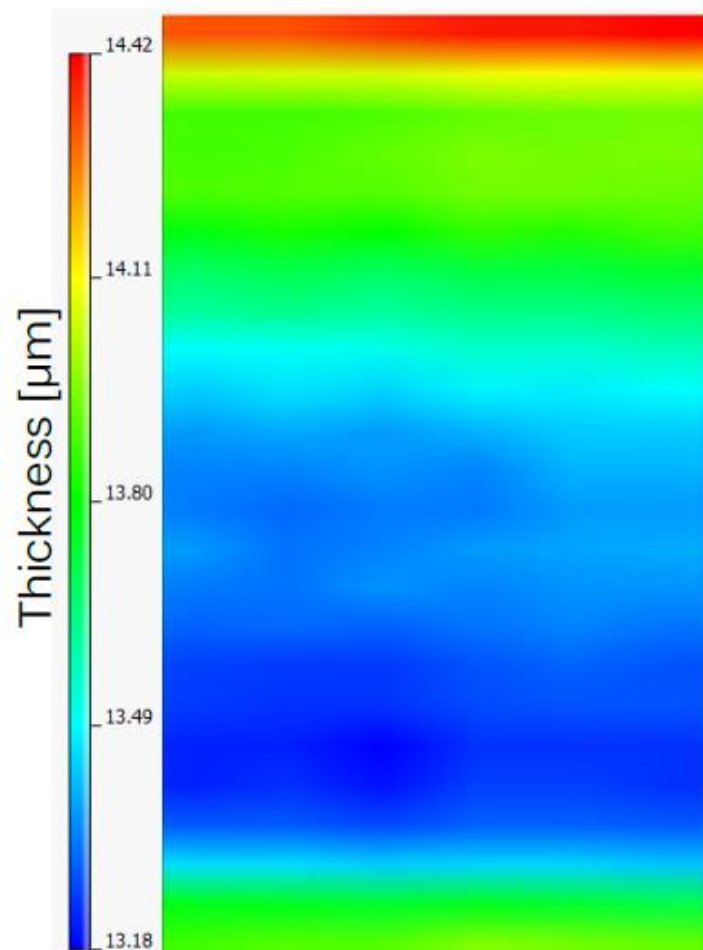
Sheet resistance images - Graphene mappings

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





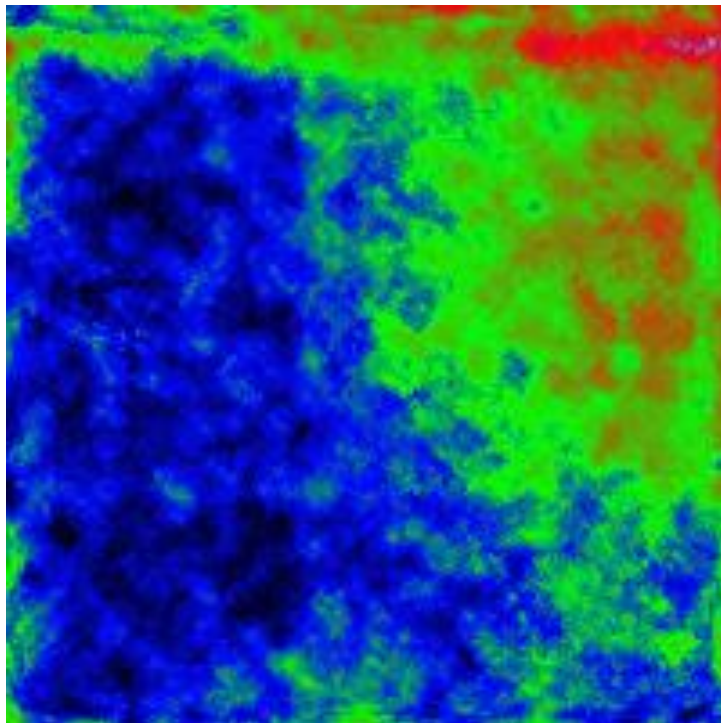
Graphene imaging on Copper?



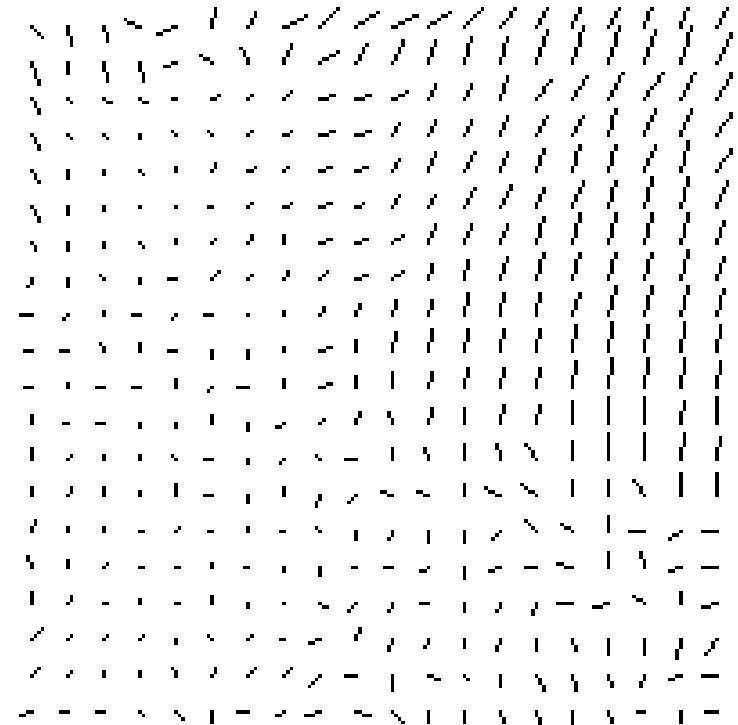


Anisotropy imaging of Graphene? – Example on Ag–NW

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



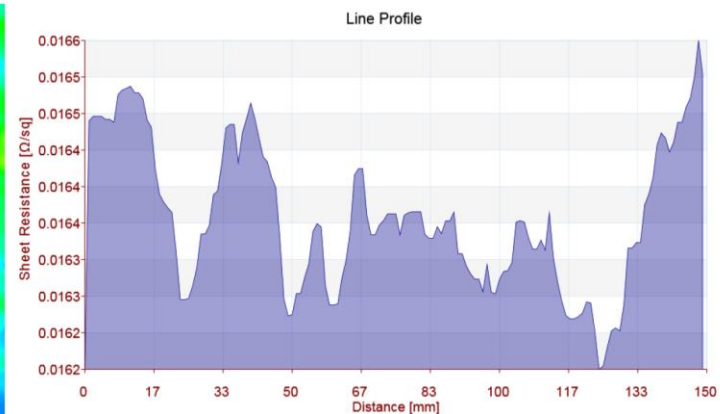
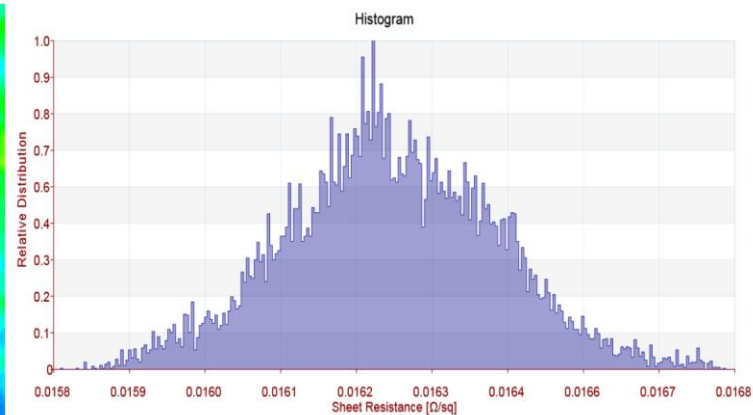
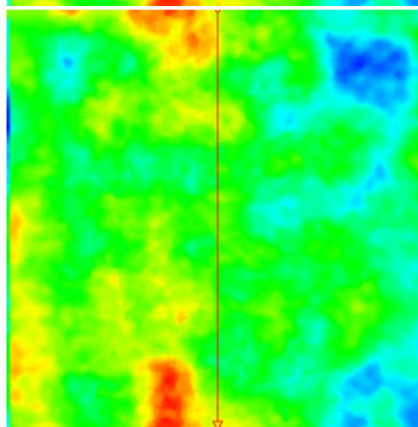
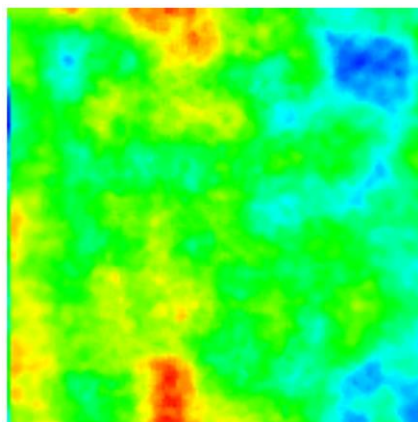
Anisotropy strength(blue=low anisotropy, red=high)



Direction of the least present sheet resistance



Graphite imaging





Inline testing – a look into the Graphene testing future...

- ▶ Inline Sheet Resistance Measurement
- + Contact-free
- + Real-time
- + High sample rate
- + Large measurement gap (1 – 60 mm gap)
- + Compact hardware
- + Optional measurement of optical transparency





Demonstration – future Graphene inline testing



www.sheet-resistance-testing.com

Customized numbers of monitoring lanes
EddyCus TF inline Series



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



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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or visit us in Dresden – e.g. at Graphene 2018

