INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

Karlsruhe Institute of Technology

International standardization on graphene and other 2D materials: Status and future prospects

Recent Progress in Graphene & 2D Materials Research, RPGR 2017, Singapore, September 18-22, 2017

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International Standards Consulting
www.isc-team.eu

IEC/TC 113: NANOTECHNOLOGY FOR ELECTROTECHNICAL PRODUCTS AND SYSTEMS

Standardization is one of the key issues for the industrialization of graphene enabled products

- **October 2012: IEC/TC 113 held a workshop about “graphene” as a new field of interest in Milpitas, CA, USA**
- **October 2013: Establishment of the Graphene Flagship - No need for standardization stated**
- **October 2014: Standardization as part of an open call in the Graphene Flagship**
- **October 2017: IEC/TC 113 has a track record on 19 workshops and invited talks regarding graphene standardization at international conferences**

Content

Standardization is one of the key issues for the industrialization of graphene enabled products

- Introductory remarks on standardization
- IEC/TC 113: The one-stop-shop for graphene standardization
- The international graphene standardization landscape
- Blank Detail Specifications and Key Control Characteristics measurement standards
- Future prospects: Standardization along the value adding chain

Standards


This is a preview - click here to buy the full publication
IEC/PAS 62565-2-1
Edition 1.0 2011-03
PUBLICLY AVAILABLE SPECIFICATION
PRE-STANDARD
Nanomanufacturing – Material specifications –
Part 2-1: Single-wall carbon nanotubes – Blank detail specification
INTERNATIONAL ELECTROTECHNICAL COMMISSION
PRICE CODE P
ICS 07.030
ISBN 978-2-88912-420-6


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IEC/TS 62607-2-1
Edition 1.0 2012-05
TECHNICAL SPECIFICATION
Nanomanufacturing – Key control characteristics –
Part 2-1: Carbon nanotube materials – Film resistance
INTERNATIONAL ELECTROTECHNICAL COMMISSION
PRICE CODE P
ICS 07.030
Warning! Make sure that you obtained this publication from an authorized distributor.

- Formal consensus based document developed by a Standard Developing Organization (SDO)
- SDOs provides consensus processes and rules to develop standards.
- SDOs provides maintaining procedures to ensure that standards at any time represents the state of the art of technology.
- Technical content of standards is within the responsibility of technical experts active in project teams of the SDO.
- IEC/ISO work according to the WTO Standards Code

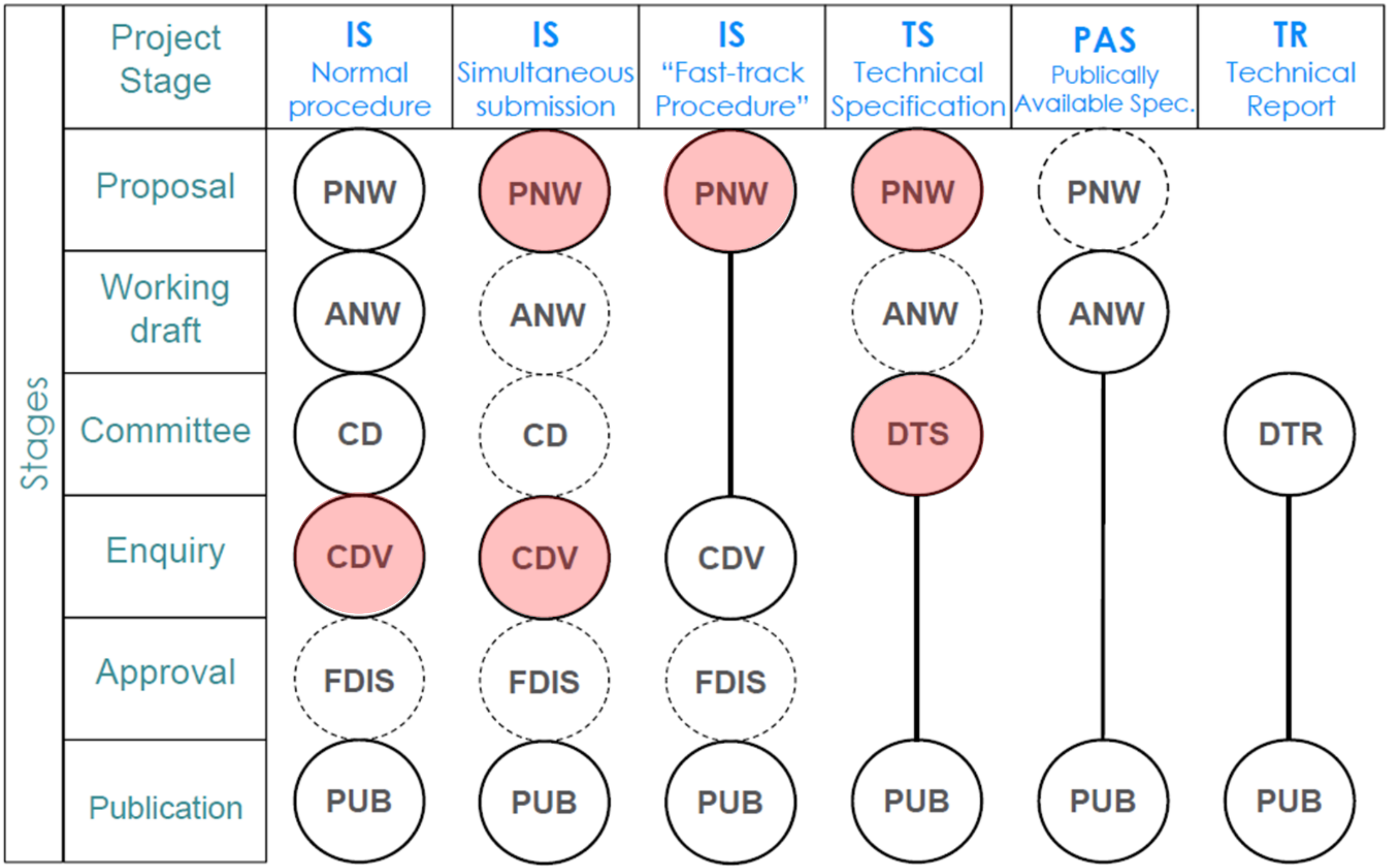
ISO IEC ASTM INTERNATIONAL Standards Worldwide IEEE STANDARDS ASSOCIATION IEEE semi cen CENELEC

The IEC/ISO process

Success Factors:

- Technical maturity of the content
- Engagement of technical experts
- Number of meetings:
  - 2 Face-to-face meetings per year
  - Online in between
- Not reinvent the wheel:
  - Prevent double work
  - Use or adopt-and-modify existing standards

The IEC/ISO process

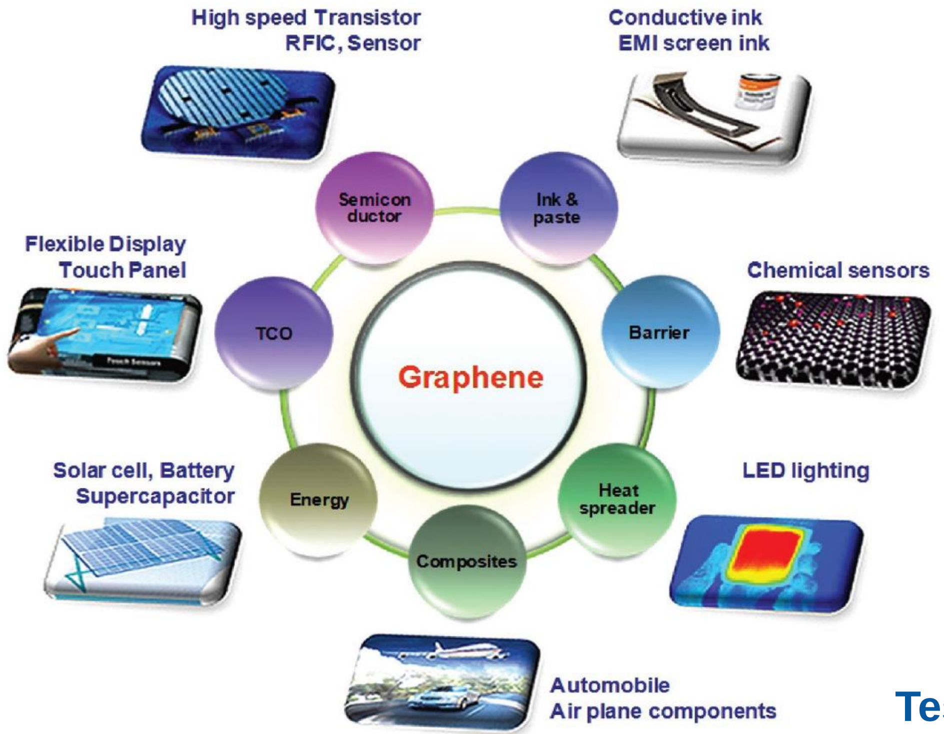


Stakeholder regarding graphene standardization

Raw Material supplier

GRM Manufacturer

End User Products Manufacturer



Fabrication Equipment Manufacturer

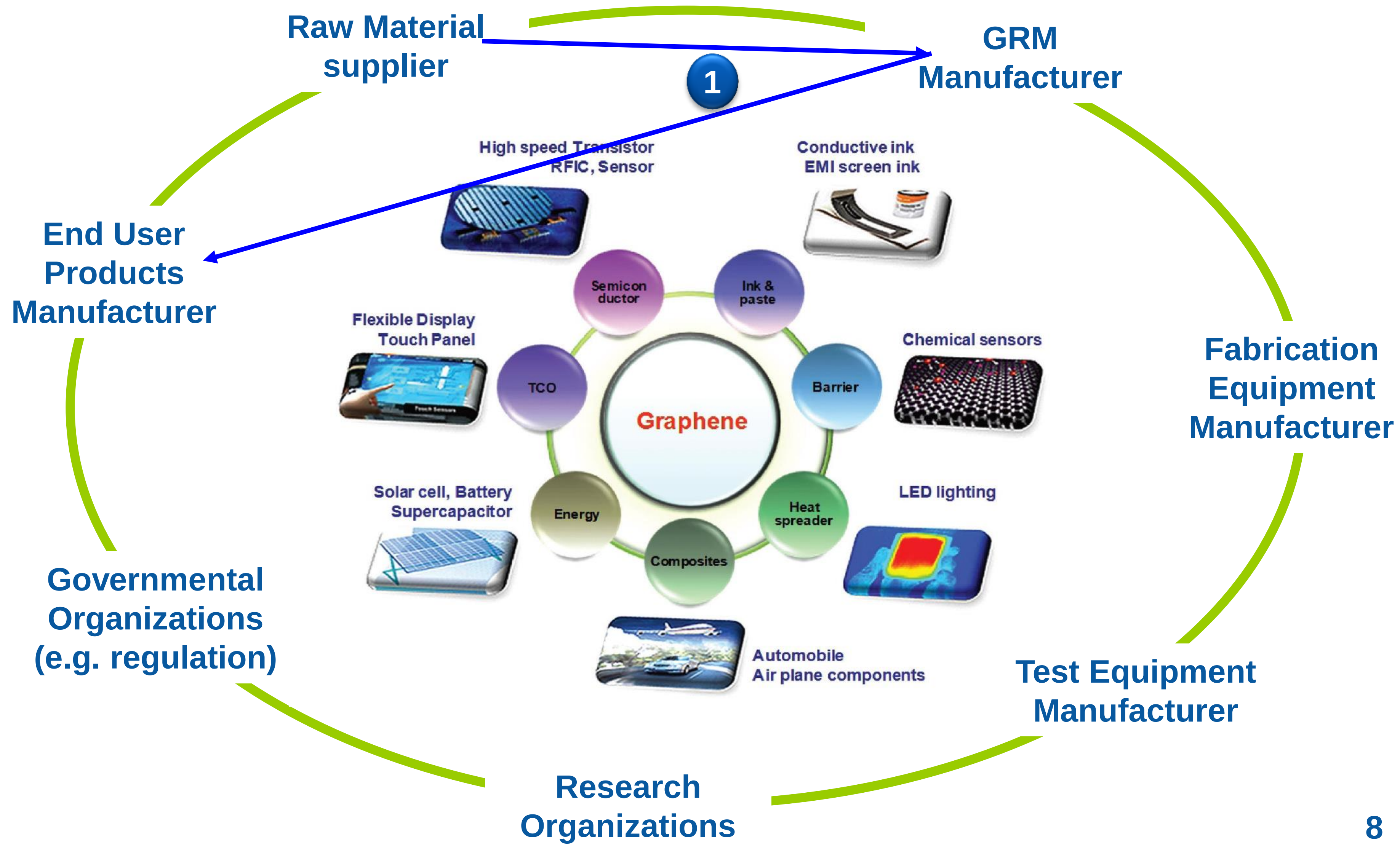
Governmental Organizations (e.g. regulation)

Test Equipment Manufacturer

Research Organizations

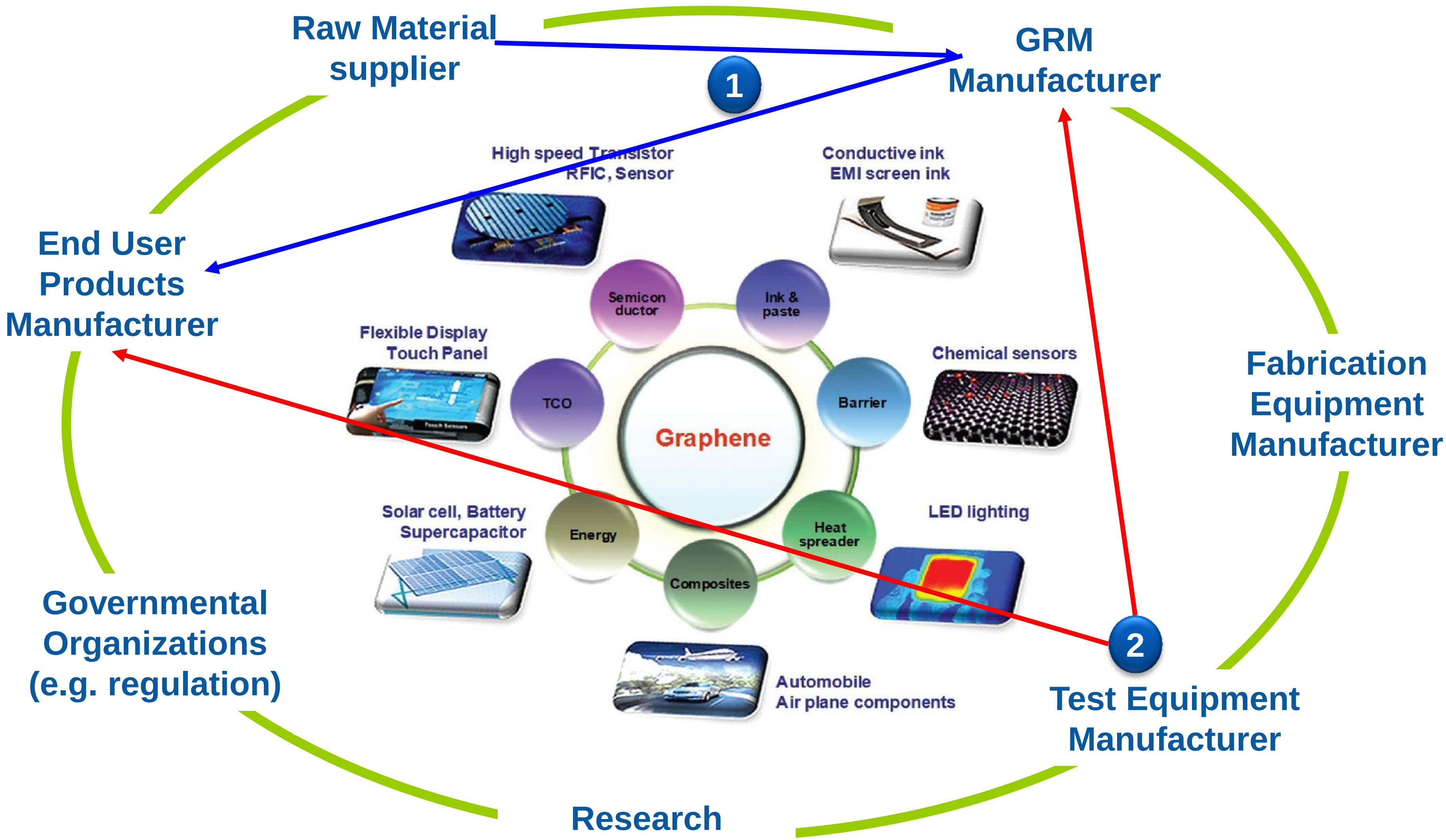
Taken from "Nanoscale: Science and technology roadmap for graphene, related two-dimensional crystals, and hybrid systems", www.rsc.org/nanoscale,

Example 1: Standardized specifications supports the supply chain



Taken from “Nanoscale: Science and technology roadmap for graphene, related two-dimensional crystals, and hybrid systems”, www.rsc.org/nanoscale,

Example 2: Measurement standards supports equipment availability



Taken from "Nanoscale: Science and technology roadmap for graphene, related two-dimensional crystals, and hybrid systems", www.rsc.org/nanoscale,

Company standardization strategy

If a company is part of a well established network based on close and long term bilateral co-operations:

- **There is no need for standardization**
- **Those companies typically try to impede standardization**

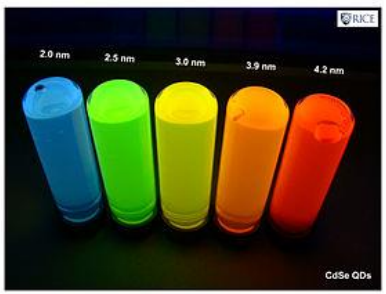
If a company wants to work with alternating suppliers to take profit from the competition among their suppliers

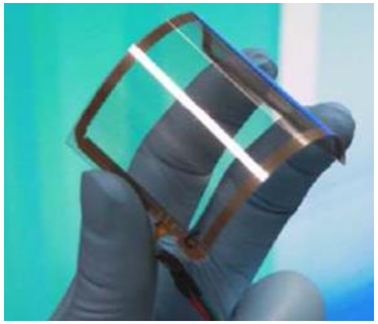
- **They strongly depend from reliable standardized specifications**
- **Those companies support standardization**

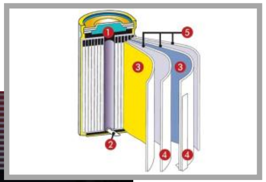
The experience is that companies who are active participating on standardization are more successful on the long term

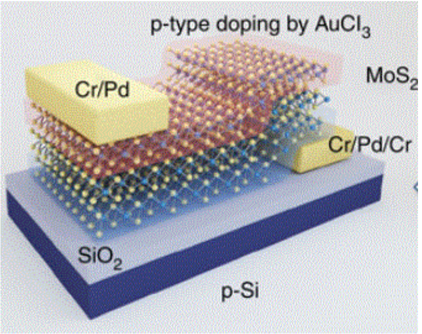
Title and scope of IEC/TC 113

- **TC 113 – Nanotechnology for electrotechnical products and systems**
- **Standardization of the technologies relevant to electrotechnical products and systems in the field of nanotechnology in close cooperation with other committees of IEC and ISO.**









Nano-enabled electrotechnical product: "electrotechnical product exhibiting function or performance only possible with nanotechnology"

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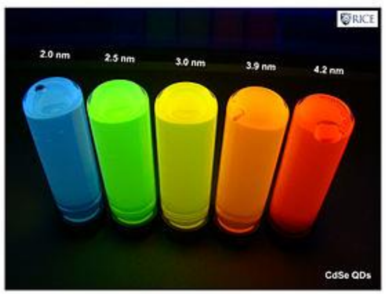

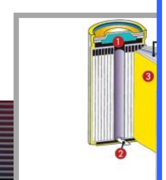

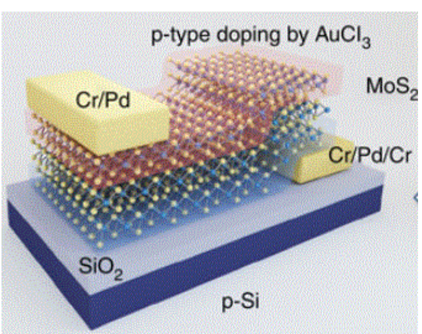

IEC/TC 113 projects:
- **Total under work: 37**
- **Total published: 29**
- **Topics**
  - **Carbon nanomaterials**
  - **Luminescent nanomaterials**
  - **Nanoscale contacts**
  - **Nano-enabled PV**
  - **Nano-enabled energy storage**
  - **Nanoelectronics**

Nano-enabled electrotechnical product: “electrotechnical product exhibiting function or performance only possible with nanotechnology”

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Graphene related materials: WG 8

22 (+4 in the definition phase)

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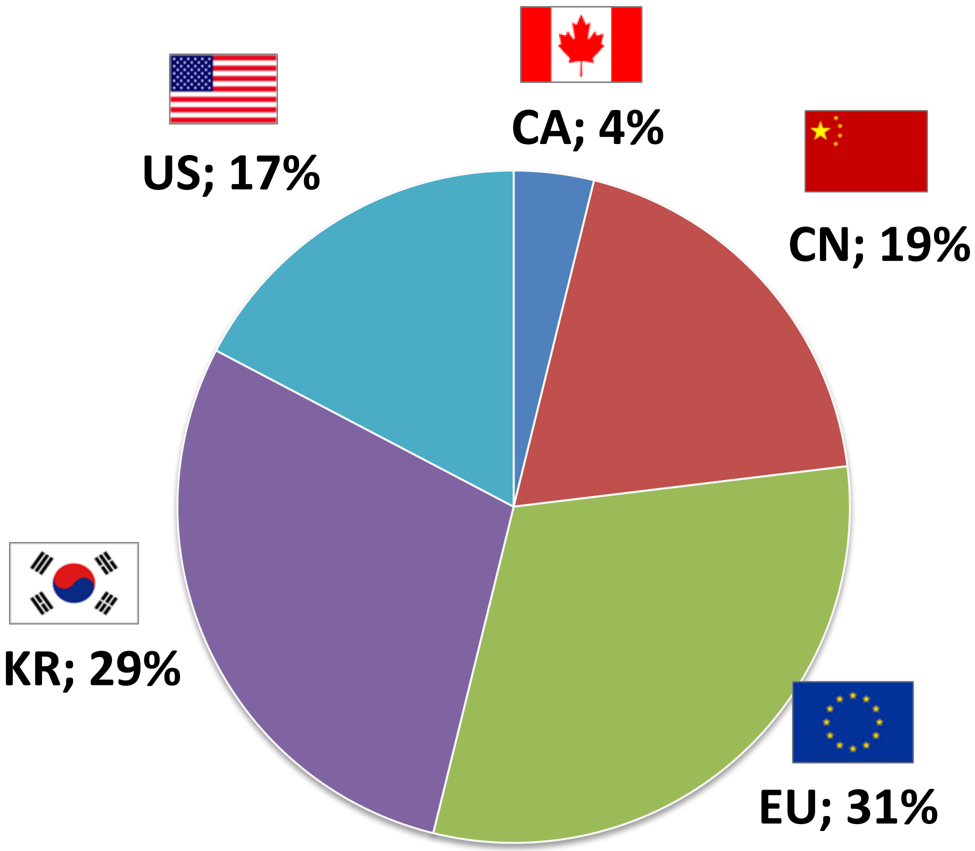
Nano-enabled electrotechnical product: “electrotechnical product exhibiting function or performance only possible with nanotechnology”

Standardization landscape „Graphene and other 2D-Materials"



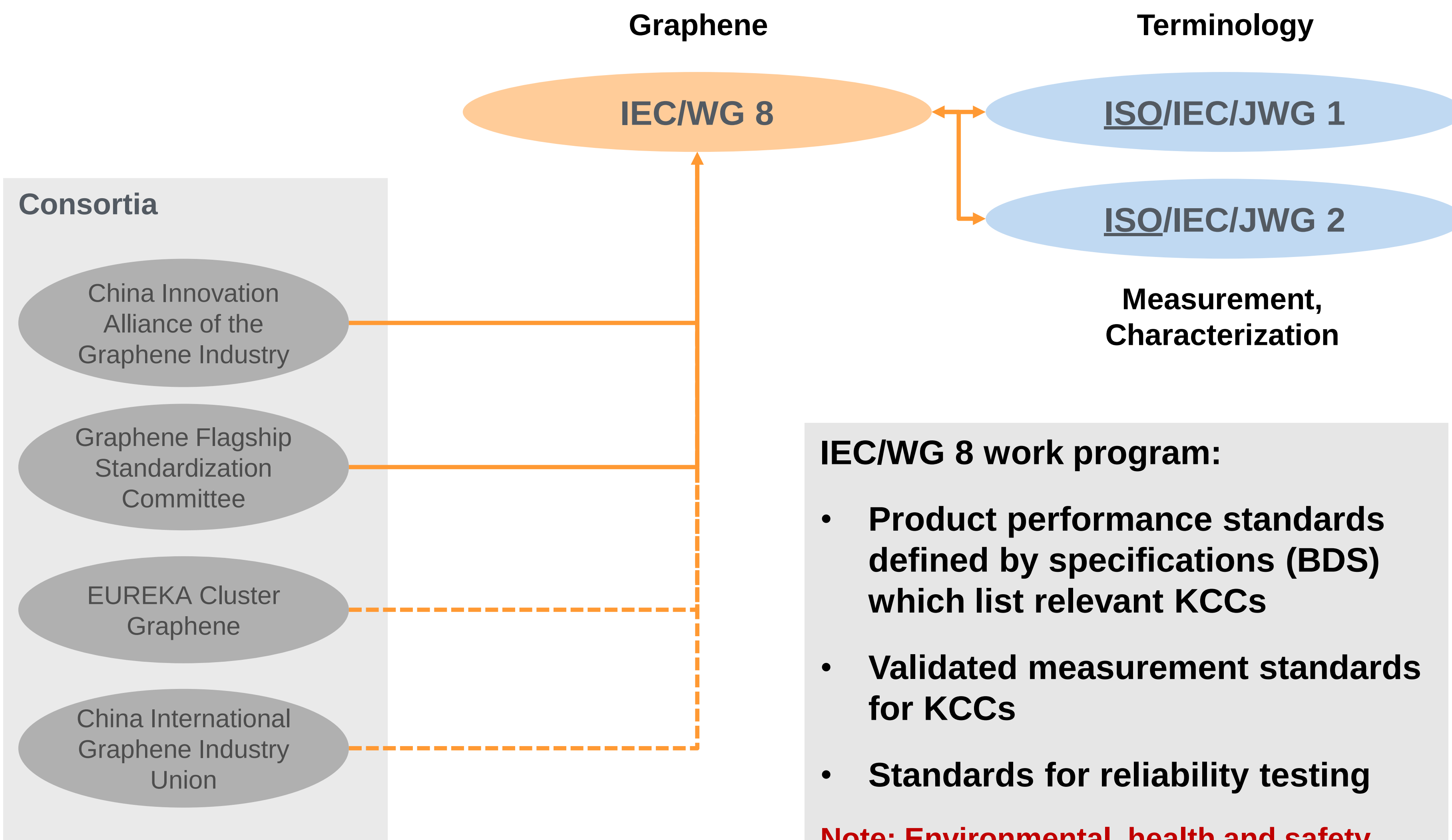
Project leadership by countries (world wide)

Graphene projects IEC/ISO:

- 10/2014: Europe 17%
  - Start of standardization activities in the graphene flagship 10/2014
- 03/2016: Europe 50%
  - China joined 05/2016
- 10/2016: Europe 36%
- 03/2017: Europe 30%
- 10/2017: Europe 31%
  China 19%

- Published: 2 (IEC/ISO)
- Majority led by IEC

Standardization landscape „Graphene and other 2D-Materials“

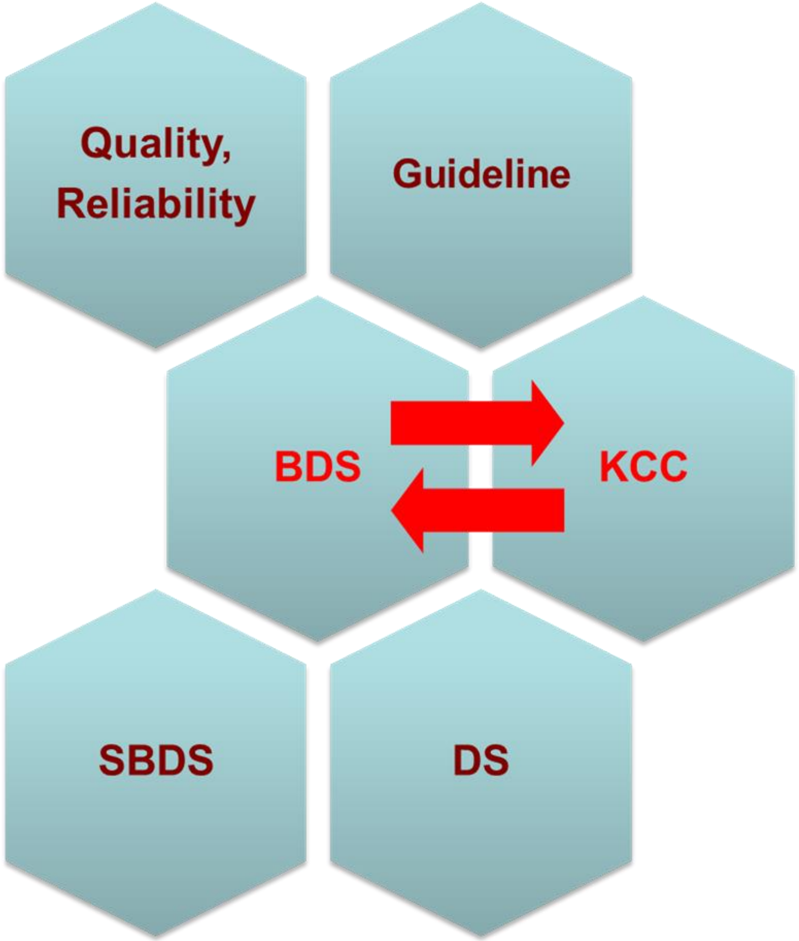


IEC/WG 8 work program:

- **Product performance standards defined by specifications (BDS) which list relevant KCCs**
- **Validated measurement standards for KCCs**
- **Standards for reliability testing**

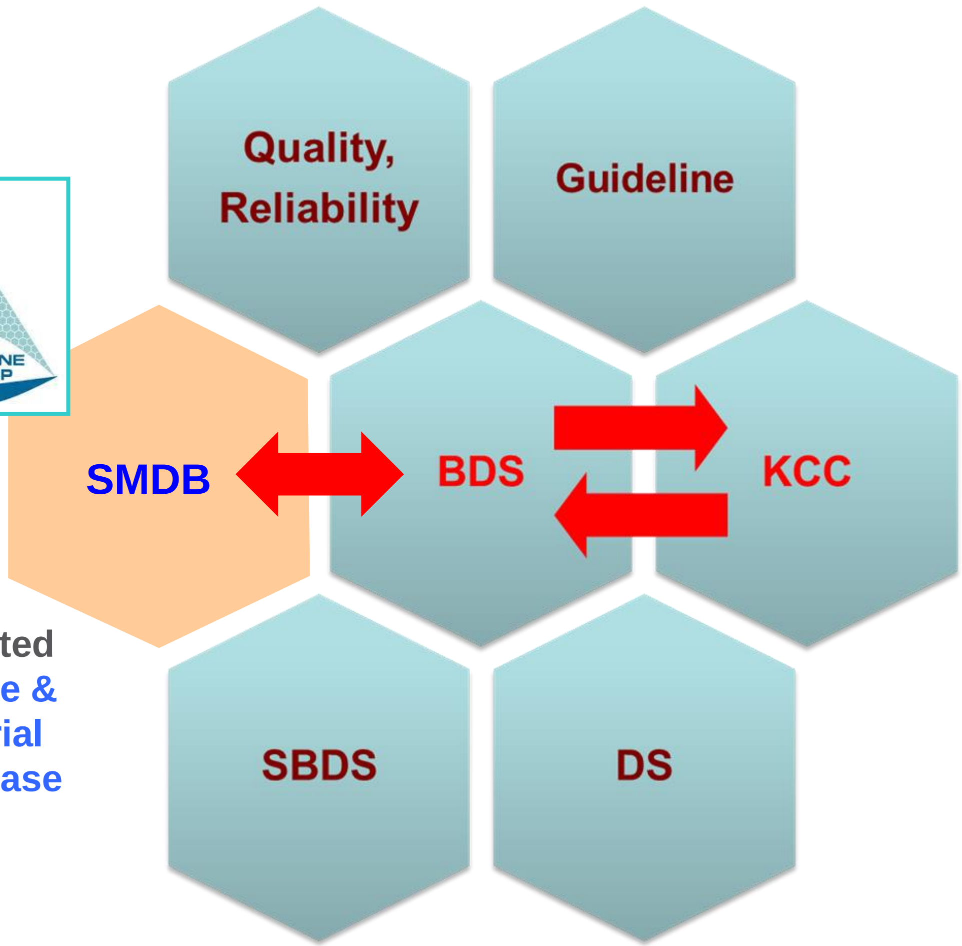
Note: Environmental, health and safety standards via liaison with ISO

Systematics of standards



- BDS: Blank Detail Specification lists the KCCs and the related measurement methods
- KCC: Measurement standards for Key Control Characteristics
- Guideline: Guiding documents to write specifications
- SBDS: Sectional Blank Detail Specifications
- DS: Detail Specifications for special applications
- Quality, Reliability: Standards regarding quality and reliability assessment

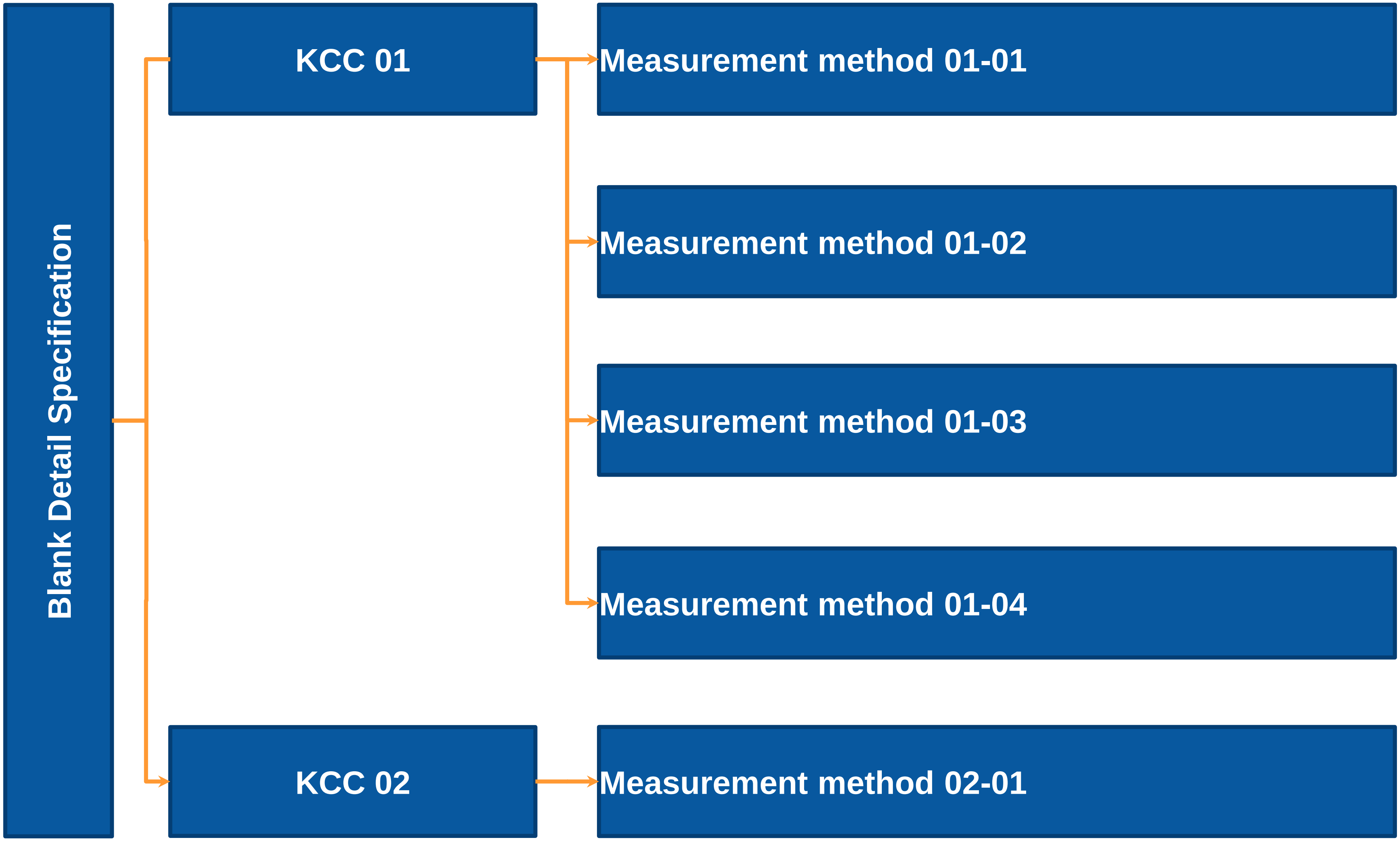
Systematics of standards / Samples & Material Database (SMDB)



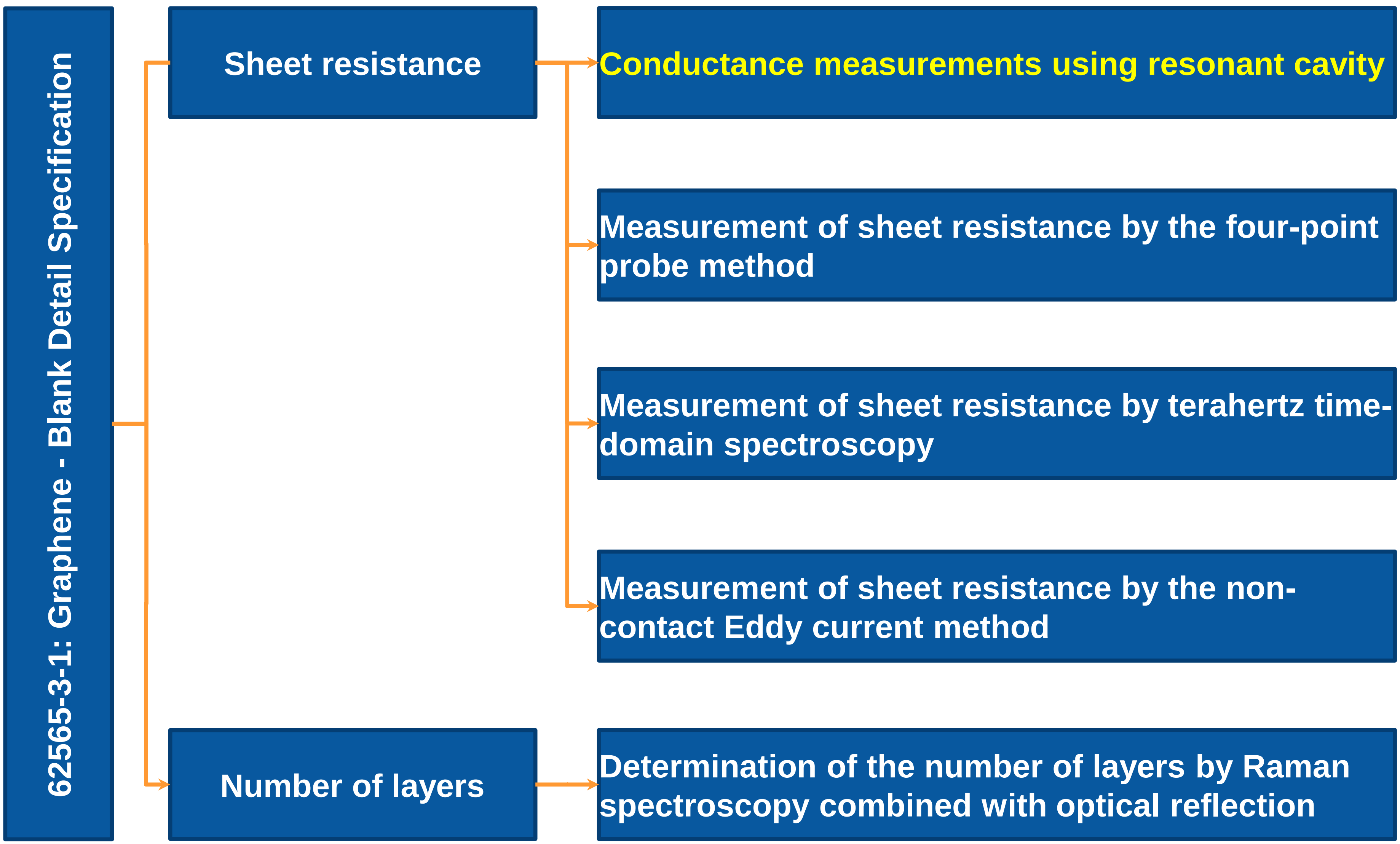
Validated Sample & Material Data Base

- BDS: Blank Detail Specification lists the KCCs and the related measurement methods
- KCC: Measurement standards for Key Control Characteristics
- Guideline: Guiding documents to write specifications
- SBDS: Sectional Blank Detail Specifications
- DS: Detail Specifications for special applications
- Quality, Reliability: Standards regarding quality and reliability assessment

IEC 62565-3-1: Blank Detail Specification Graphene (Principle)



IEC 62565-3-1: Blank Detail Specification Graphene (Example)



Content of IEC 62565-3-1 Blank Detail Specification Graphene

FOREWORD

INTRODUCTION

1 Scope

2 Normative references

3 Terms, definitions

3.1 General graphene terms (Ref. 80004-13)

3.2 Terms related to procurement

3.3 Terms related to material description

3.4 Chemical key control characteristics

3.5 Electrical key control characteristics

3.6 Thermal key control characteristics

4.7 Optical key control characteristics

3.8 Mechanical key control characteristics

3.9 Structural and dimensional key control characteristics

4 Measurement methods

5 Graphene specification format and recommended measurement methods

5.1 Procurement information

5.2 General material description

5.3 Chemical key control characteristics

5.4 Electrical key control characteristics

5.5 Thermal key control characteristics

5.6 Optical key control characteristics

5.7 Mechanical key control characteristics

5.8 Structural and dimensional key control characteristics

Example: Electrical Key Control Characteristics (IEC 62565-3-1)

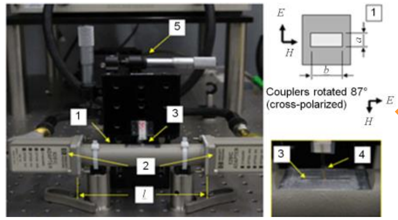
Table 4 – Format for electrical key control characteristics

KCC No	KCC	Specification	Measurement method	F	P	D
4.1	Sheet resistance	Nominal [ ] ± Tolerance [ ] Ω/sq	IEC/TS 62607-06-04 IEC/TS 62607-06-08 IEC/TS 62607-06-09 IEC/TS 62607-06-10	✓		✓ 2
4.2	Sheet conductance	Nominal [ ] ± Tolerance [ ] S/sq	IEC/TS 62607-06-04	✓		✓ 2
4.3	Conductivity	Nominal [ ] ± Tolerance [ ] S/m	IEC/TS 62607-06-01		✓ 1	
4.4	Field effect carrier mobility	Nominal [ ] ± Tolerance [ ] cm²/Vs		✓		
4.5	Hall carrier mobility	Nominal [ ] ± Tolerance [ ] cm²/Vs		✓		
4.6	Work function	Nominal [ ] ± Tolerance [ ] meV		✓		

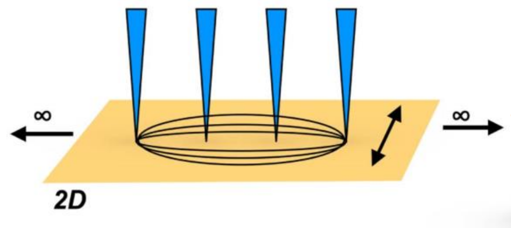
Note
1) Measured on pellets
2) Measured on films solidified according to suppliers specification

Example: Electrical Key Control Characteristics (IEC 62565-3-1)

Microwave cavity



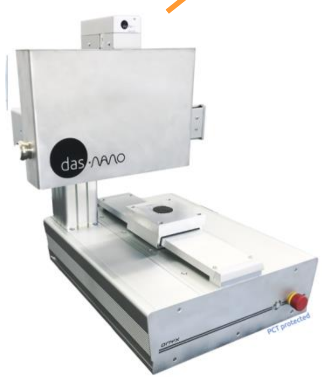
Four point probe



Measurement method	F	P	D
IEC/TS 62607-06-04	✓		✓[2]
IEC/TS 62607-06-08			
IEC/TS 62607-06-09			
IEC/TS 62607-06-10			
IEC/TS 62607-06-04	✓		✓[2]
IEC/TS 62607-06-01		✓[1]	
	✓		
	✓		
	✓		

published

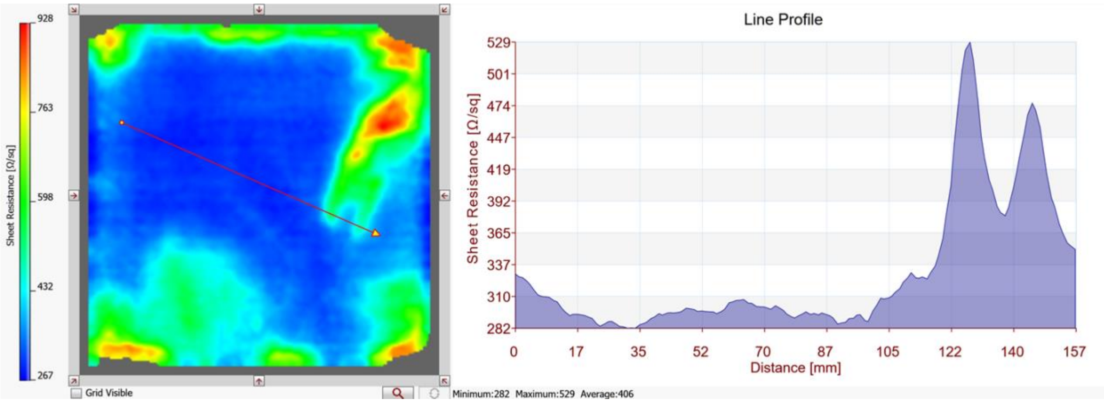
Eddy current



THz-TDS

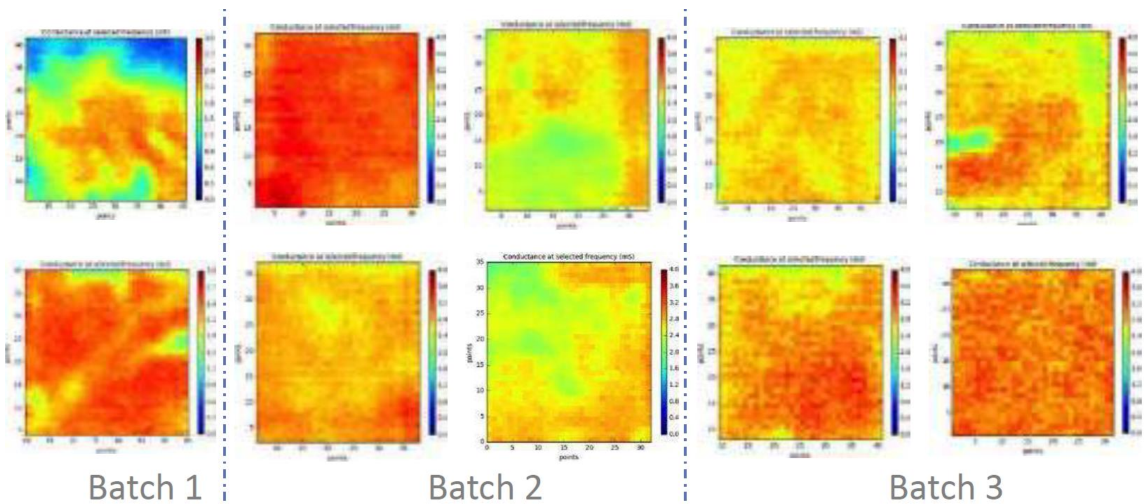
Evaluation of fabrication quality by standardized methods

Example: Analysis of sheet resistance uniformity:



IECTS 62607-6-9
With courtesy of:
Suragus GmbH, Germany

Example: Analysis batch reproducibility:



IEC/TS 62607-6-10
With courtesy of:
das-Nano S.L., Spain

62607-06-10 / THz – TDS: Content

FOREWORD

INTRODUCTION

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4. General

4.1 Measurement principle

4.2 Measurement configuration

4.2.1 Transmission configuration

4.2.2 Reflection configuration

examples

4.3 Measurement mode

4.3.1 Single point mode

4.3.2 Image scanning mode

examples

4.4 Measurement system

4.4.1 Measurement equipment / apparatus

4.4.2 Materials

4.4.3 Calibration standards

4.4.4 Ambient conditions

4.4.5 Sample preparation method

5. Measurement procedure

5.1 Calibration of measurement equipment

5.2 Detailed protocol of the measurement procedure

5.3 Measurement accuracy

6. Data analysis / Interpretation of results

7. Results to be reported

ANNEX

A. Use case description

B. Sampling plan

C. Test report

D. Worked examples

62607-06-10 / THz – TDS: Test report

Test Report

This test report concludes the results of sample characterization according to

IEC/TS 62607-6-10 NANOMANUFACTURING – KEY CONTROL CHARACTERISTICS – Part 10: Graphene – Measurement of sheet resistance by terahertz ti...

1. Identification of test samples according to IEC 62565-3-1

Item No	Item		
1.1	Supplier		
1.2	Trade name		
1.3	ID number		
1.4	Traceability requirements	☐ Batch number ☐ Serial number ☐ Others, specify	
		Manufacturing date	
1.5	Specification	Number	
		Revision level	
		Date of issue	

2. General material description according to 62565-3-1

Item No	Item	Information...
2.1	Material type	
2.2	Physical form	☐ Film on substrate (F) ☐ Powder (P) ☐ Solution (S)
2.3	Composition	
2.4	Manufacturing method	
2.5	Transfer method	
2.6	Substrate	
2.7	Solvent	
2.8	Shelf life	Nominal [ ] s, d
2.9	Curing conditions*	
2.10	Material Safety Data Sheet (MSDS) available	☐ No ☐ Yes, reference ....
2.11	Typical batch quantity	☐ Number of wafers ☐ Weight [kg] ☐ Volume [l]

3. Geometry of the sample

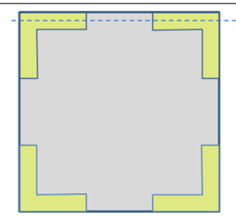

Figure 1: Top view

Figure 2: Cross secti...

4. Test related information

Item No	Item	Inf...
3.1	Sampling plan	☐ Circular, specify C-.... ☒ Square, specify S-.... ☐ others, drawing attache...
3.2	Excitation wavelength	
3.3	Number of spectra/measurement	☐ 10x10 ☐ others, specify
3.4	I(D)/I(G)	☐ not visible
3.5	Mean value and standard deviation of FWHM(2D) distribution	
3.6	Raman maps	☐ attached ☐ available on request
3.7	Environmental humidity	

5. KCC maps

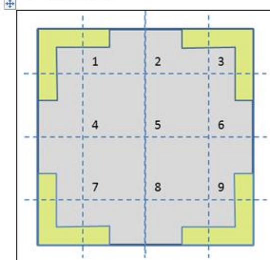

Figure 3: Sampling plan

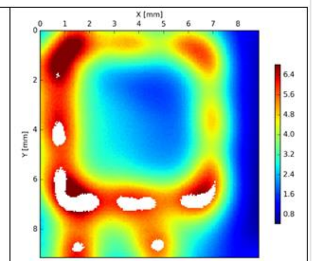

Figure 4: Conductivity map

6. KCC measurement results

Position	Surface conductivity	FET/Hall mobility
1		
2		
3		
4		
5		
6		
7		
8		
9		

Maturity and validation of the measurement standards

Standardization process

Consensus:
„yes“ ≥ 66,7%
„no“ ≤ 25,0%

Stability:
1-3 years

Consensus:
„yes“ ≥ 66,7%
„no“ ≤ 25,0%

Stability:
1-3 years

Voting is performed by the IEC members (countries) after they have achieved a consensus position in their country

Maturity and validation of the measurement standards

Standardization process

Consensus:
„yes“ ≥ 66,7%
„no“ ≤ 25,0%

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1-3 years


IEC/TS 62607-2-1

TECHNICAL SPECIFICATION

Nanomanufacturing – Key control characteristics – Part 2-1: Carbon nanotube materials – Film resistance

Consensus:
„yes“ ≥ 66,7%
„no“ ≤ 25,0%

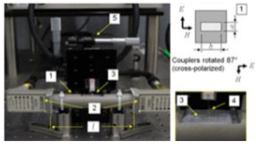
Stability:
1-3 years

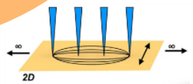

IEC/TS 62607-2-1

TECHNICAL SPECIFICATION

Nanomanufacturing – Key control characteristics – Part 2-1: Carbon nanotube materials – Film resistance

Scientific validation (round robin):



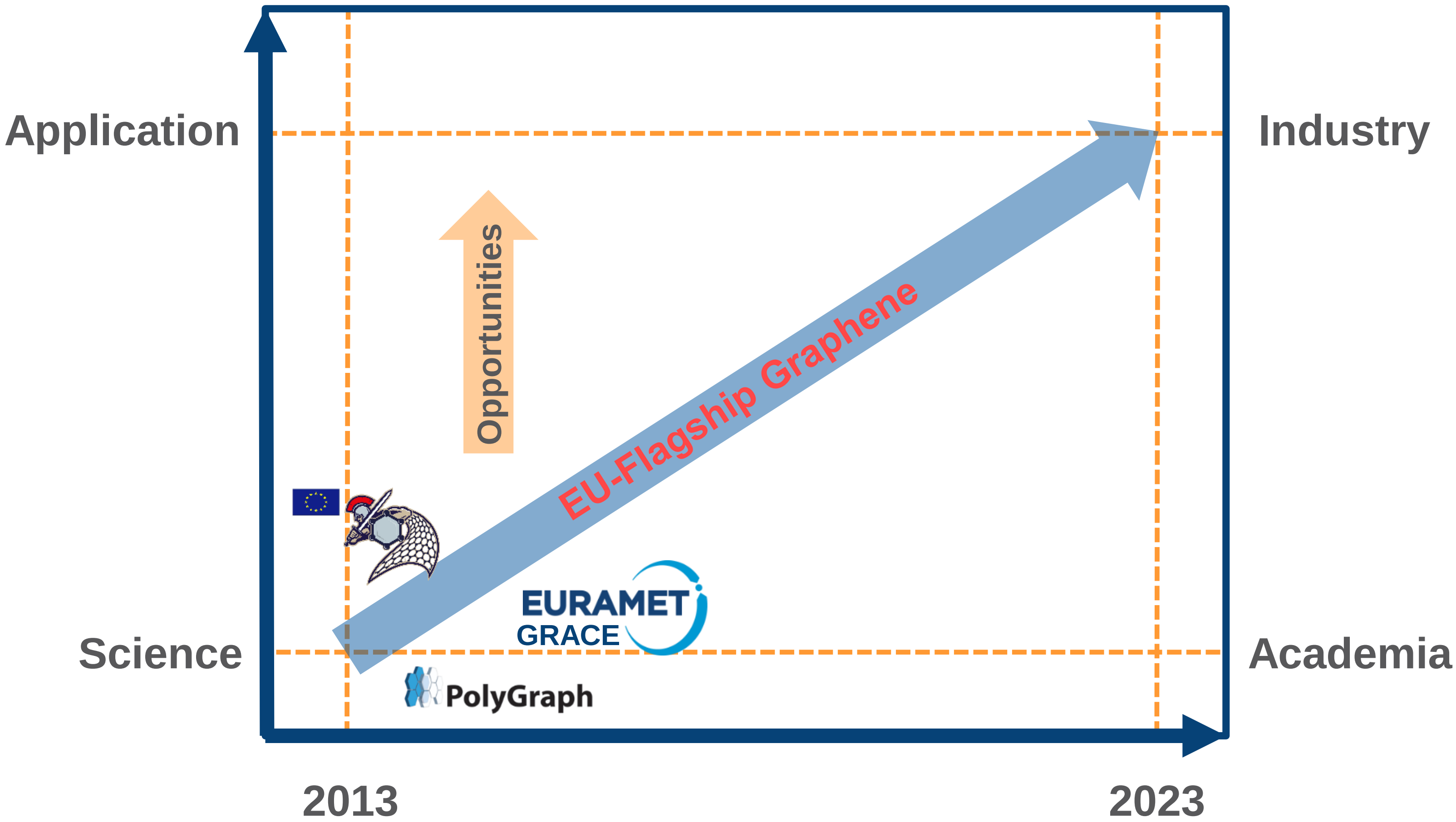


GraCe

EMPIR EURAMET

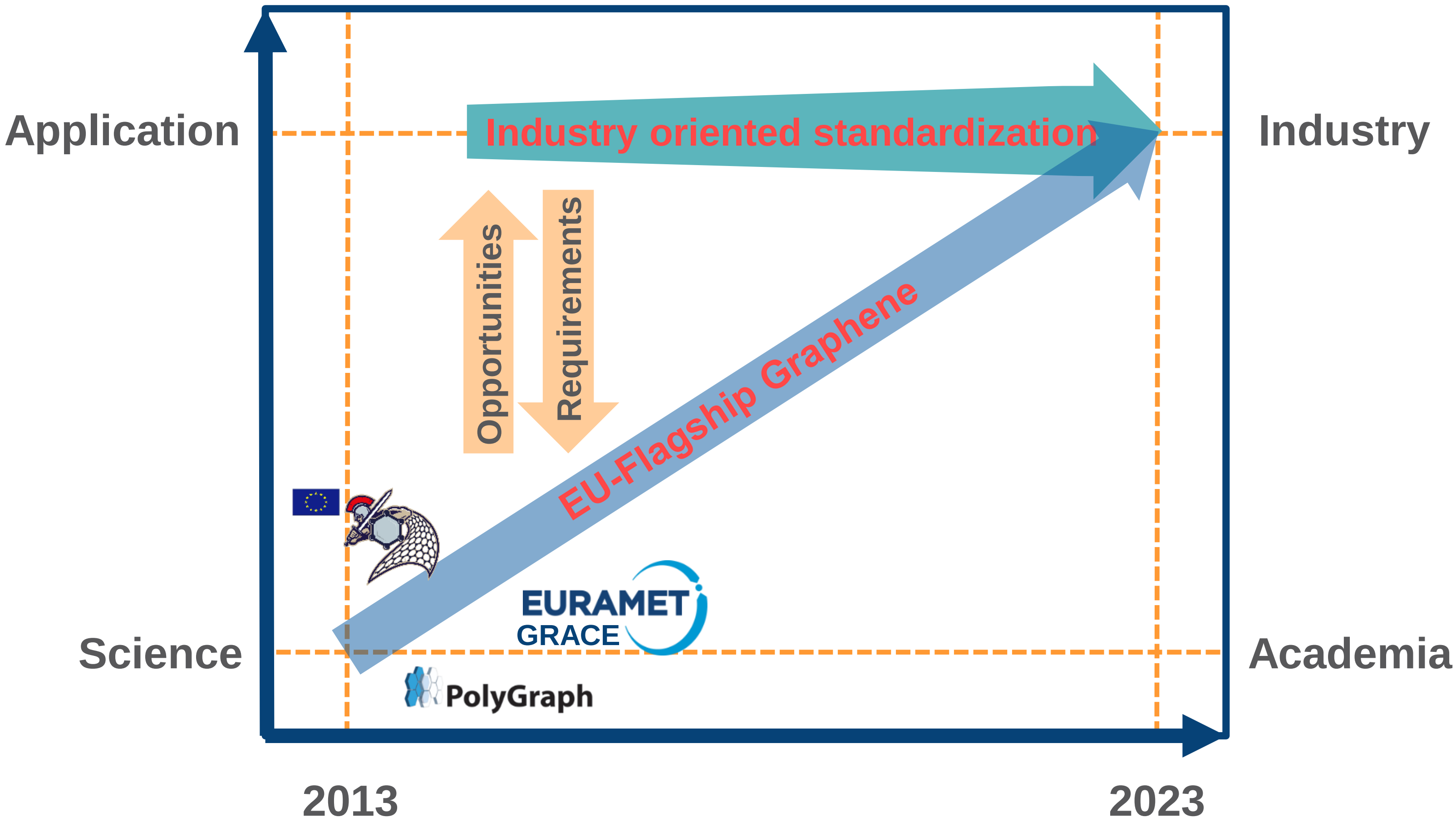
The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Moving towards industry oriented standardization



Standardization supports cooperation between the EU-Flagship and other EU projects like “Polygraph (FP7)”, “Gladiator (FP7)”, “GRACE” (Euramet EMPIR) as well as national funded projects.

Moving towards industry oriented standardization



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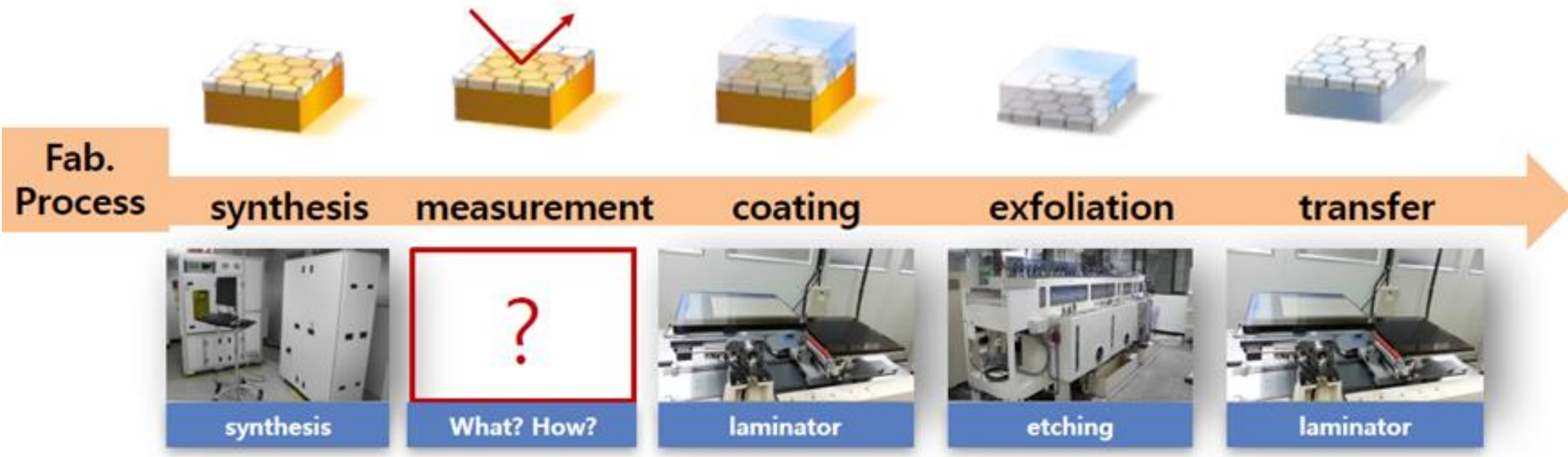
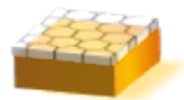
Use case analysis: Value adding chain



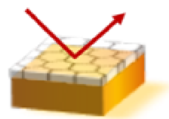
- **Availability of a Standard Operating Procedure (SOP) for each fabrication step is mandatory**
- **Measurement standards for Key Control Characteristics (KCC) are SOPs for controlling and inspection within a fabrication process chain**

Picture: Young Jae Song *Sungkyunkwan University (SKKU), Suwon, Korea*

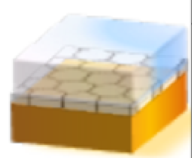
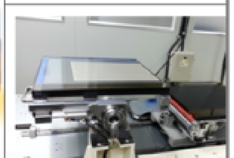
Use case analysis: Value adding chain

No.	Step	Illustration	Equipment
4	Synthesis graphene layer on copper substrate		CVD equipment
	Detailed description of the process		

Process step

No.	Step			Illustration	Equipment
5	Inspection graphene layer on copper substrate				
	KCC	Standard			
		Measure-ment	BDS		
	Averaged domain size	IEC/TS 62607-6-3	IEC 62565-3-1		
	Domain size distribution	IEC/TS 62607-6-3	IEC 62565-3-1		

Inspection step

No.	Step	Illustration	Equipment
6	Support of graphene layer by polymer		Laminator
	Detailed description of the process		

Process step

Karlsruhe Institute of Technology: Contact

Operation of the secretariat IEC/TC 113:
Karlsruhe Institute of Technology (KIT):
norbert.fabricius@kit.edu

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Ass. Secretary Prof. Dr. Werner Bergholz (ISC),
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