Graphene 2017
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High sensitive CVD graphene-based gas sensors operating under environmental conditions

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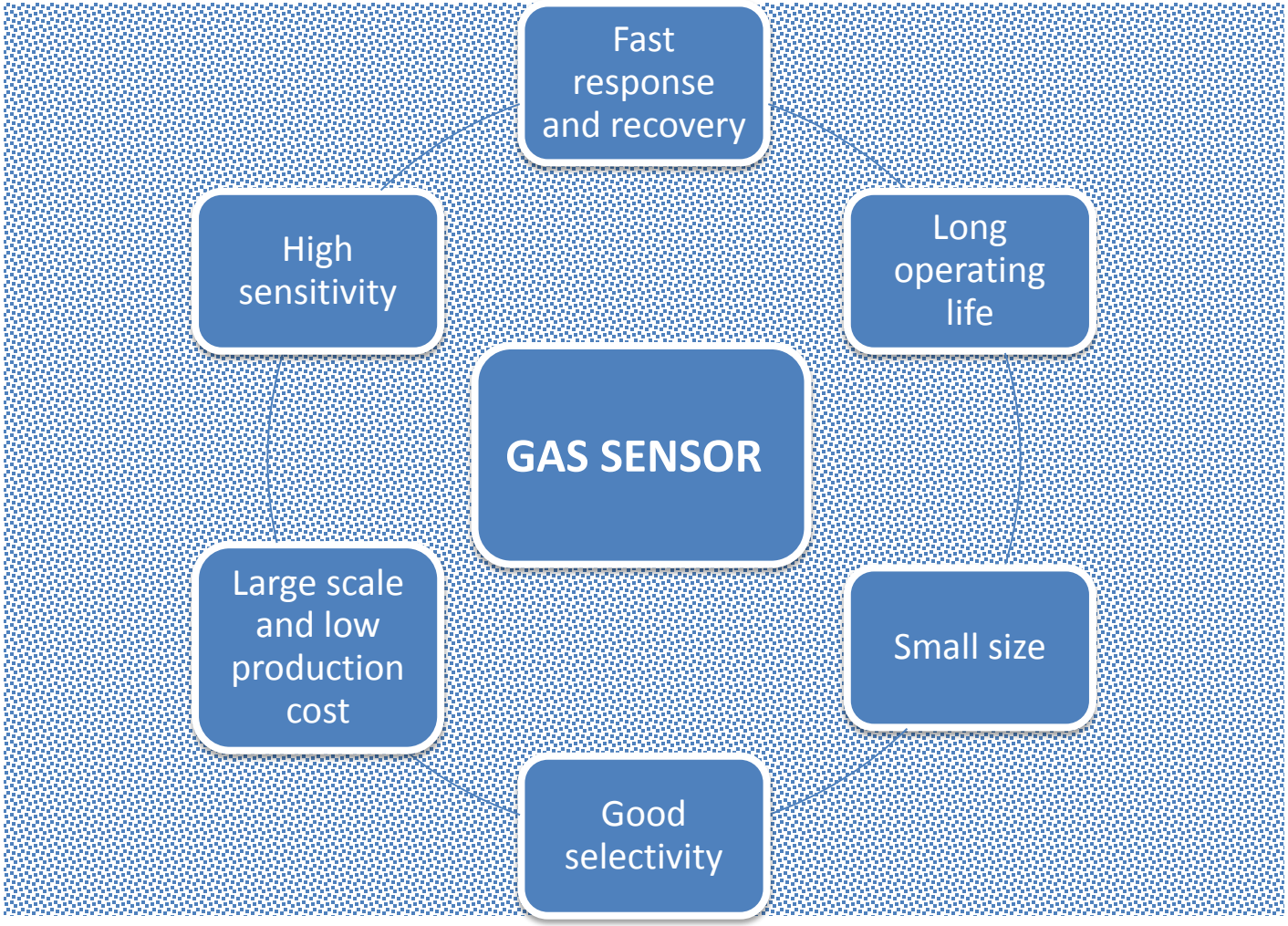
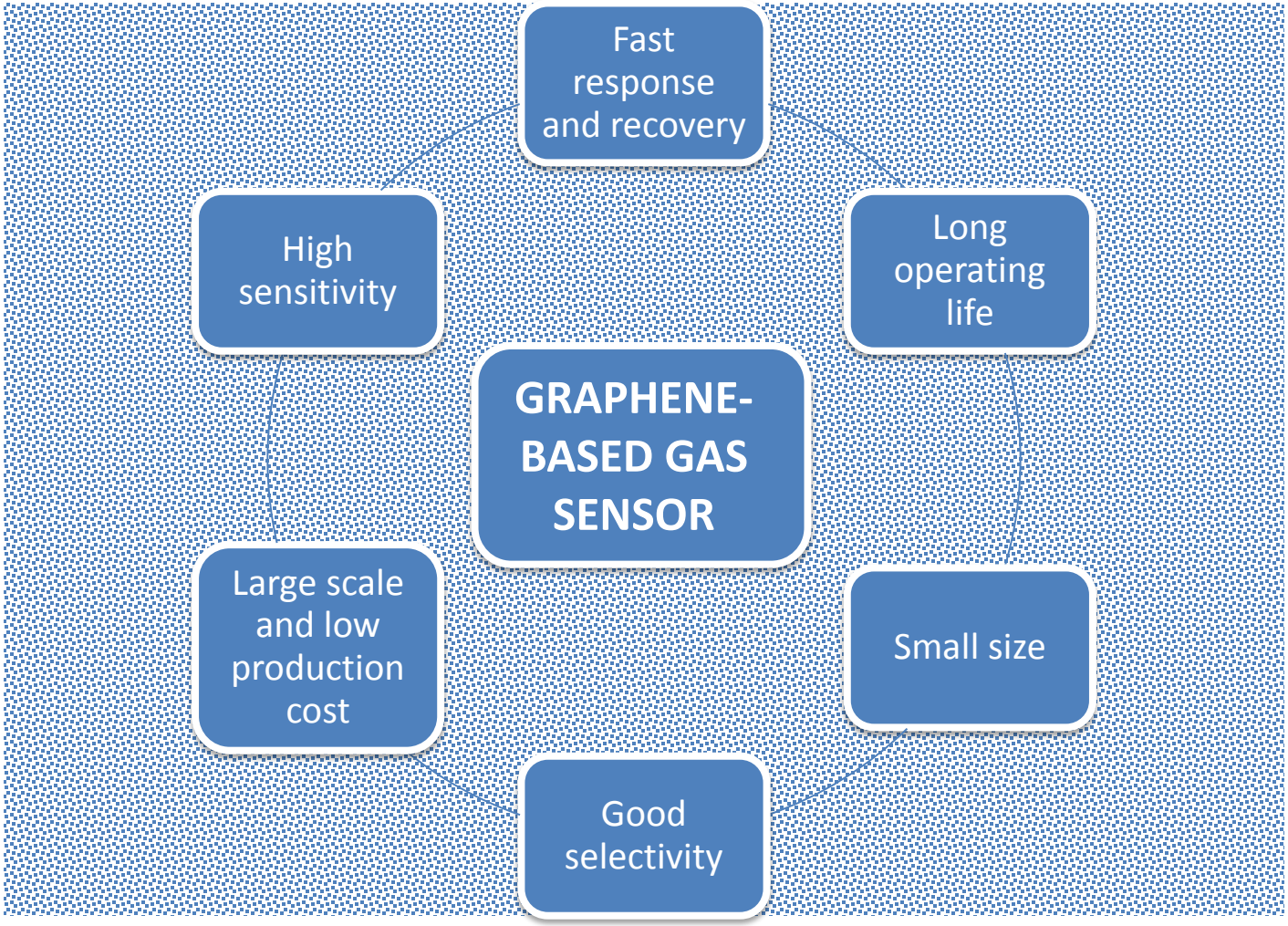
Introduction



Research activity



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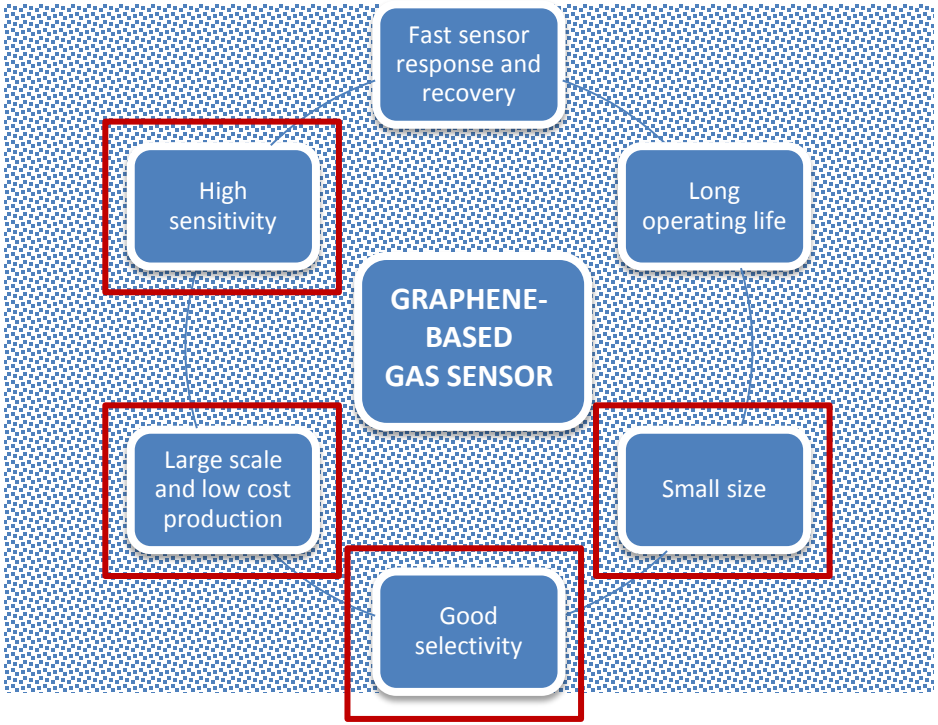
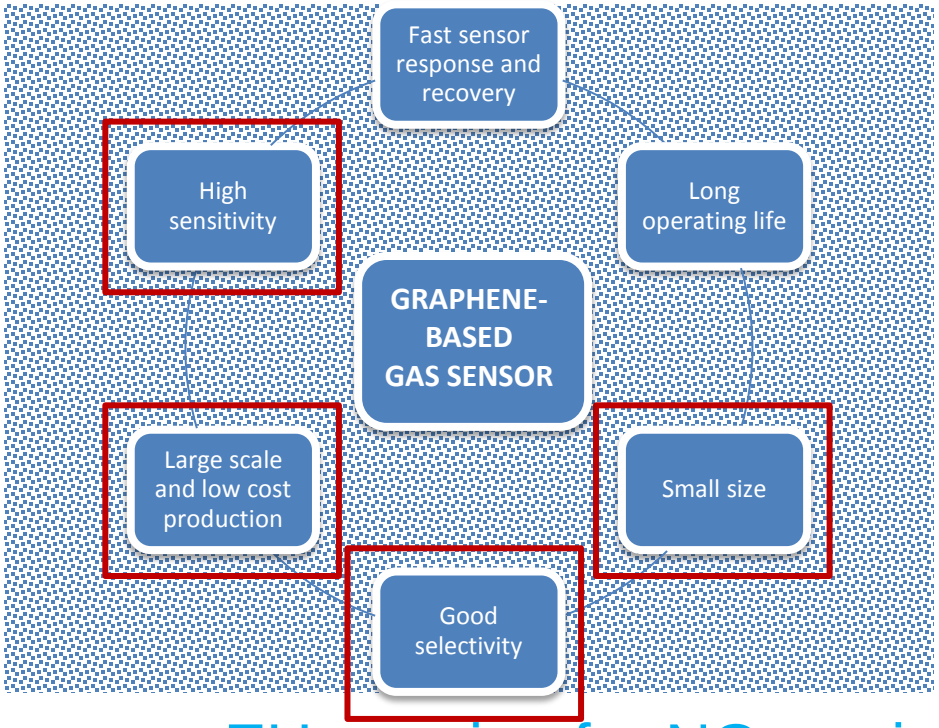


- Wafer scale production
- Small size (device~2, 5, 10 µm)
- Selectivity to NO_2
- High sensitivity (LOD~100ppb @RT)

S. Vollebregt et al.: Proceedings MEMS 2016

F. Ricciardella et al.: Proceedings IEEE Sens. 2016

Research activity

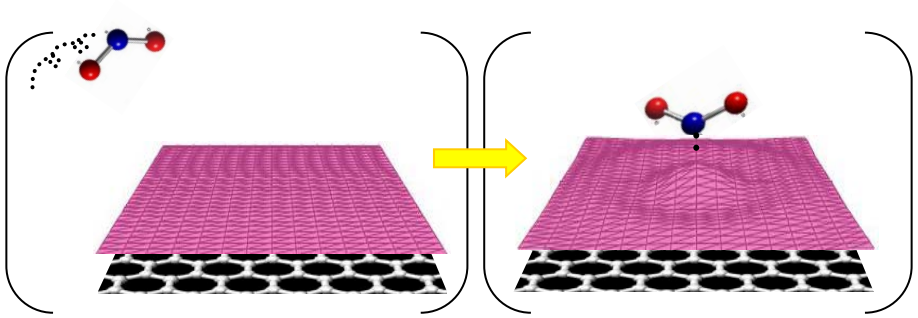


- Wafer scale production
- Small size (device~2, 5, 10 µm)
- Selectivity to NO_2
- High sensitivity (LOD~100ppb @RT)

EU warning for NO_2 emissions (last February)

- France
- Italy
- Germany
- Spain
- Great Britain

Why graphene in gas sensing?



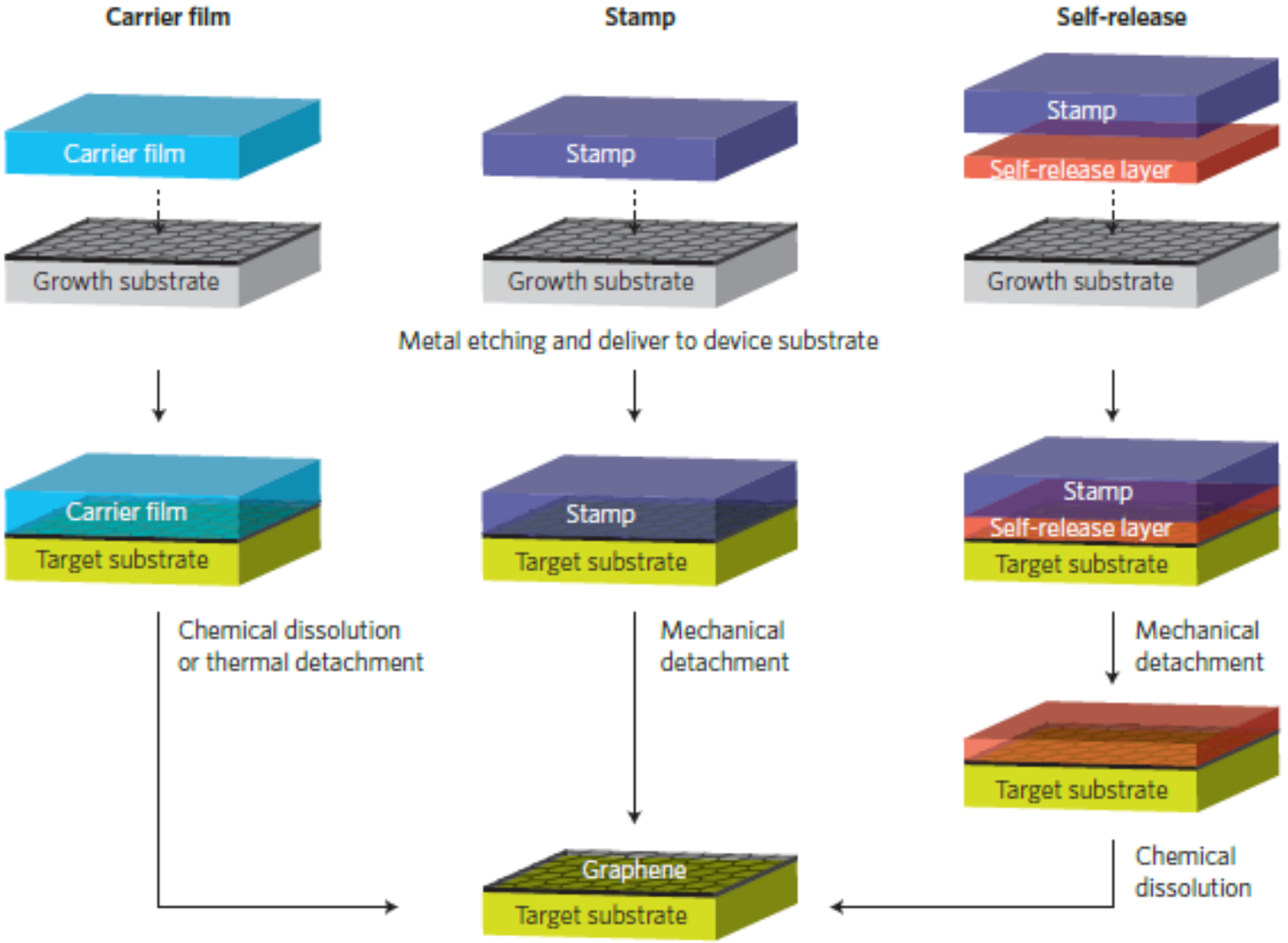
CANDIDATE FOR GAS-SENSORS

2D MATERIAL

- Highest surface volume ratio (2600 m^2g^{-1})
- Surface atoms interaction, no bulk
- **Strong stability @ RT**

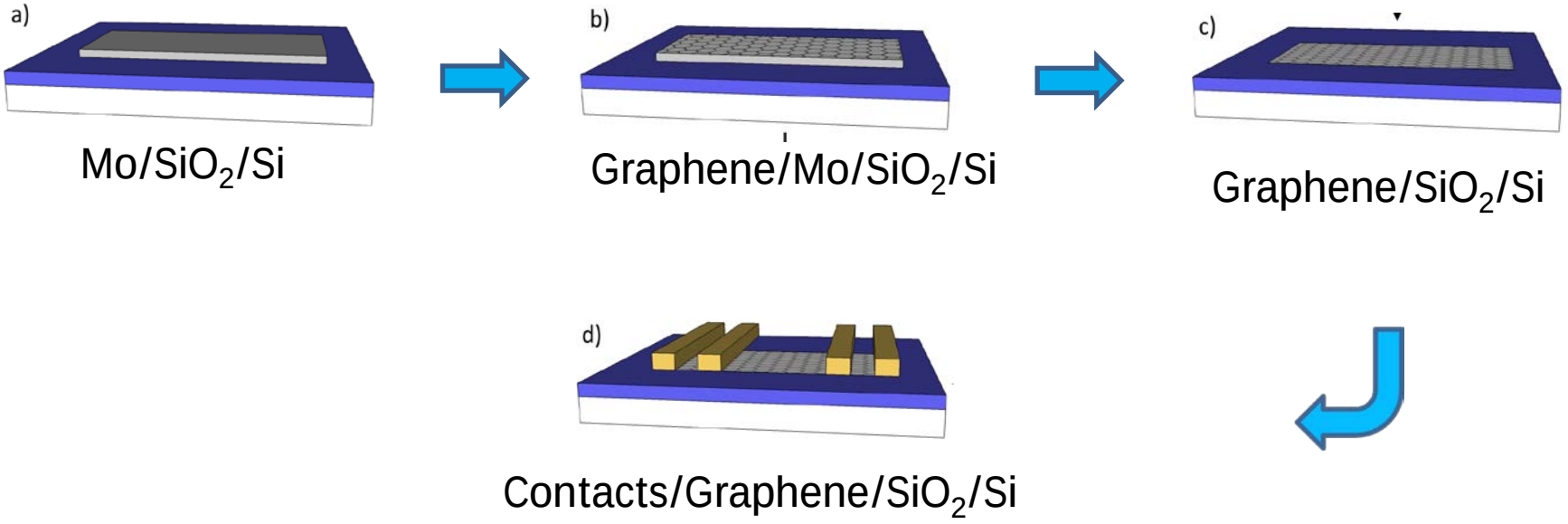
IDEAL CANDIDATE FOR GAS-SENSORS IN ENVIRONMENTAL CONDITIONS

Transfer process after CVD growth



J.-Y. Choi: Nat. Nanotechnol., 8, 311 (2013)

Transfer-free process

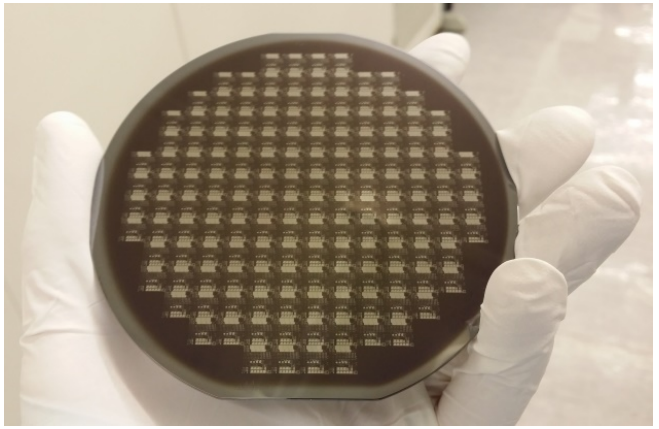


☺ Growth substrate = target substrate

☺ No damages

☺ No polymer residues at interface

☺ Wafer scale process with high yield (>97%)



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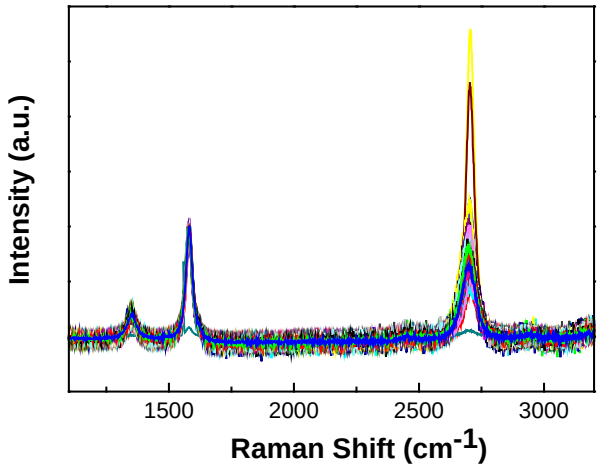
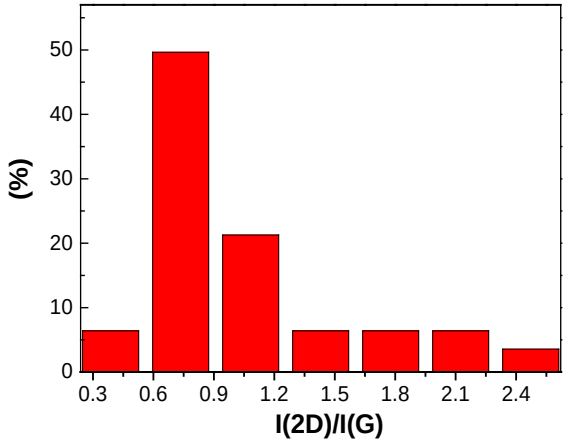
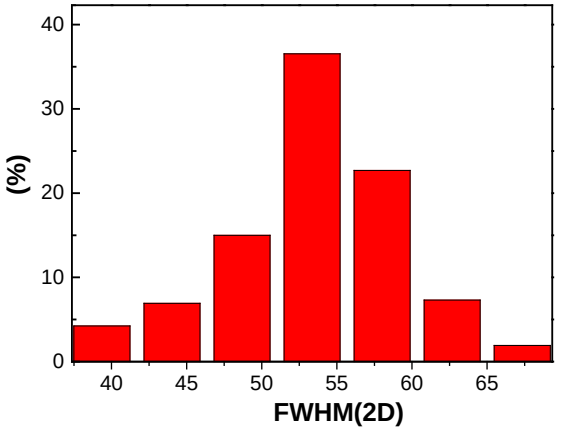
S. Vollebregt et al.: Proc. of 29th Intern. Conf. MEMS 2016

Graphene characterizations

Raman (50X, N.A. 0.50)
100 spectra, area=100 μm x 100 μm







Graphene characterizations

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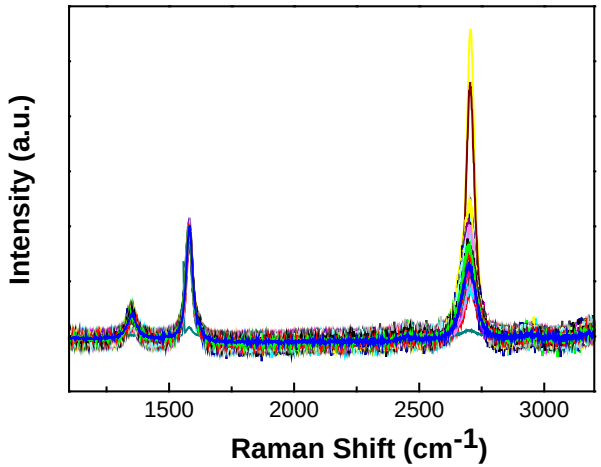
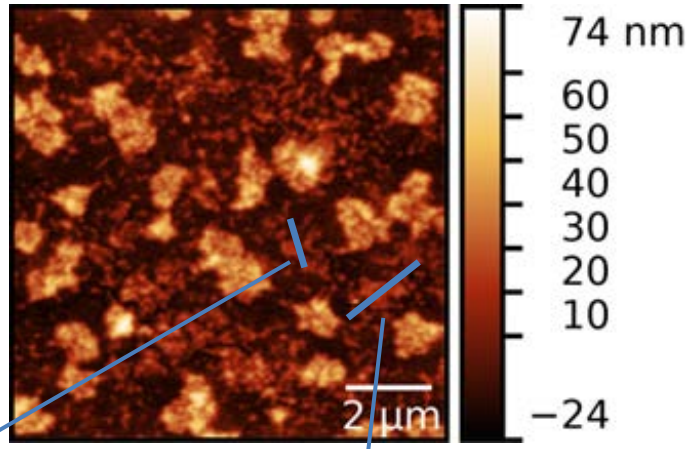
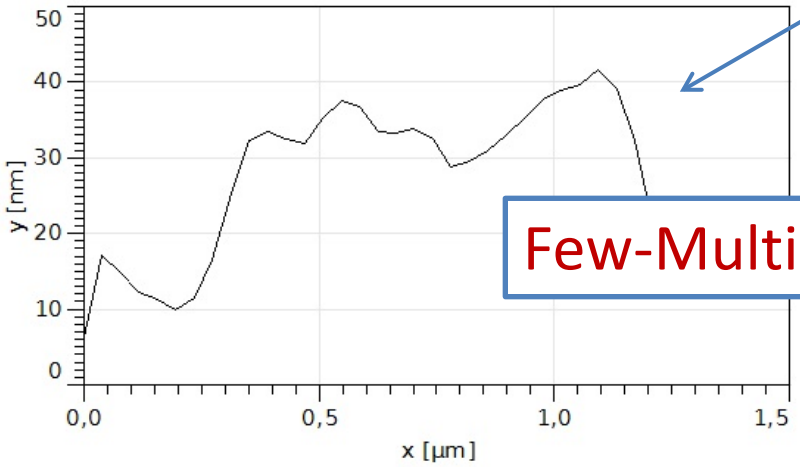
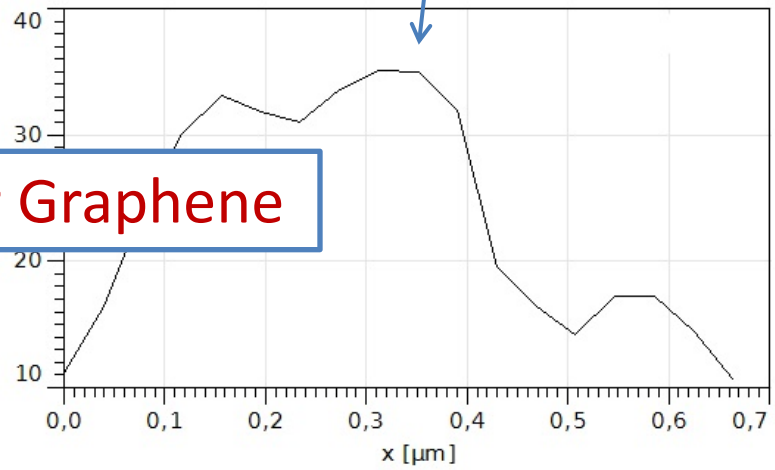


AFM

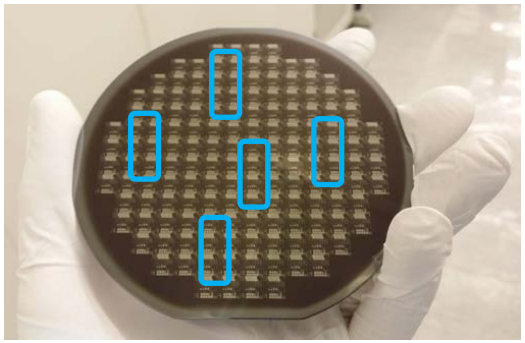




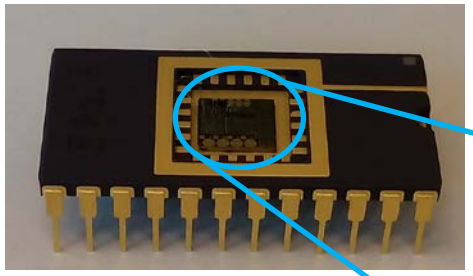


Few-Multi Layer Graphene

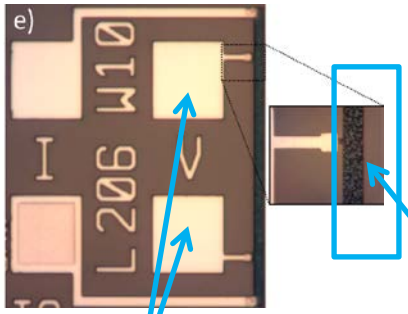
Devices



Packaging



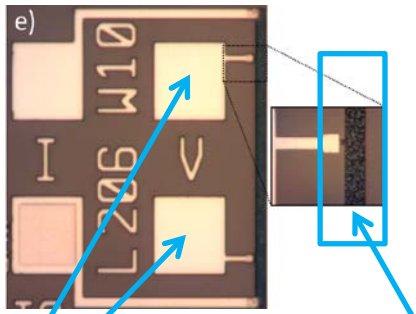
Bonding



Graphene bar

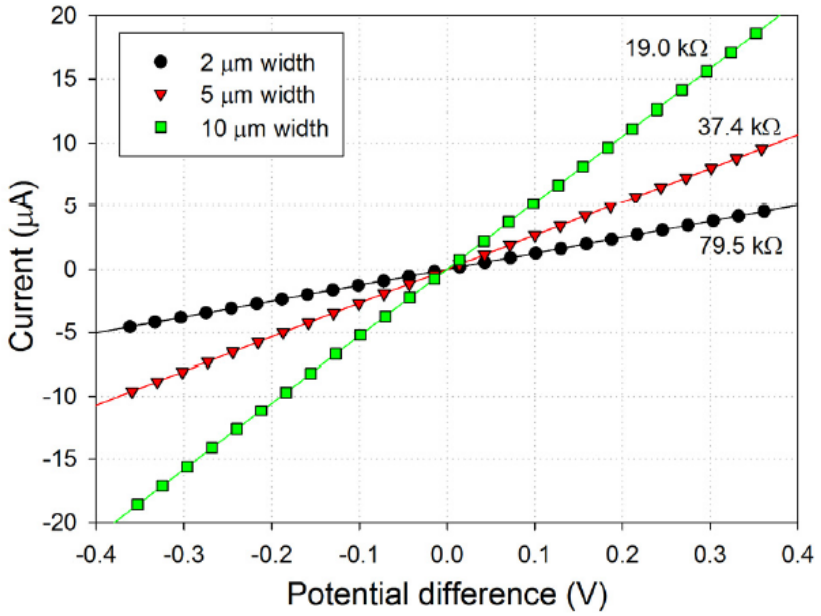
Metal contacts

I-V measurements



Graphene bar

Metal contacts



	Graphene bar dimensions (µm)	
	Length	**Width**
Device 1A	206	10
Device 1B	206	10
Device 2A	206	5
Device 2B	206	5
Device 3A	206	2
Device 3B	206	2

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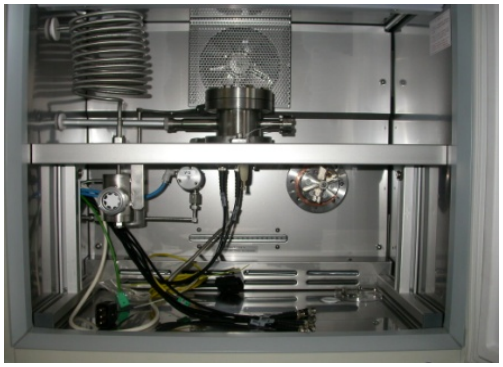
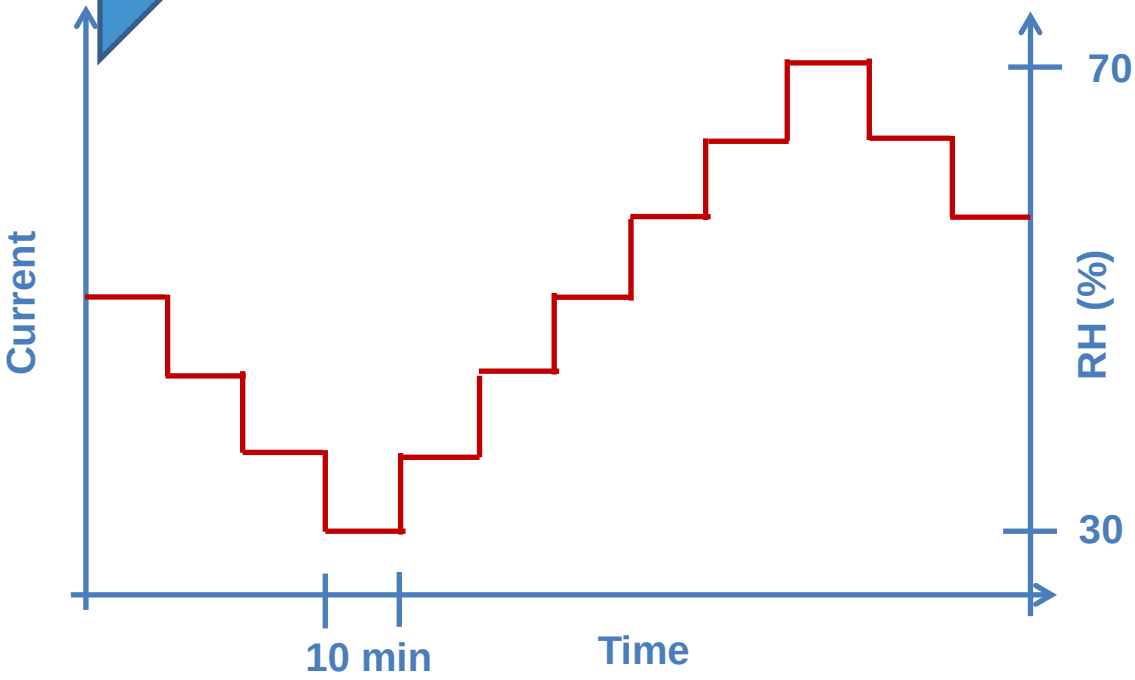
Experimental set-up

TEST CHAMBER

- Stainless steel testing chamber
- Enviromental conditions (RT=25° C, RH=50%, $p=p_{atm}$)
- Carrier: N_2





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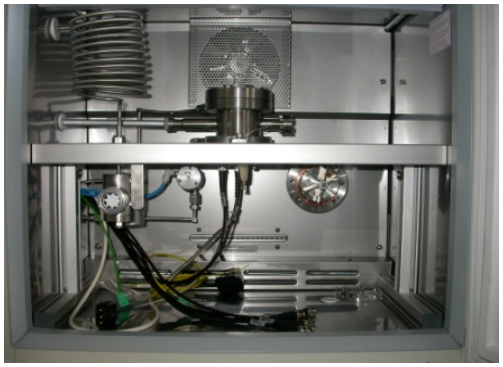
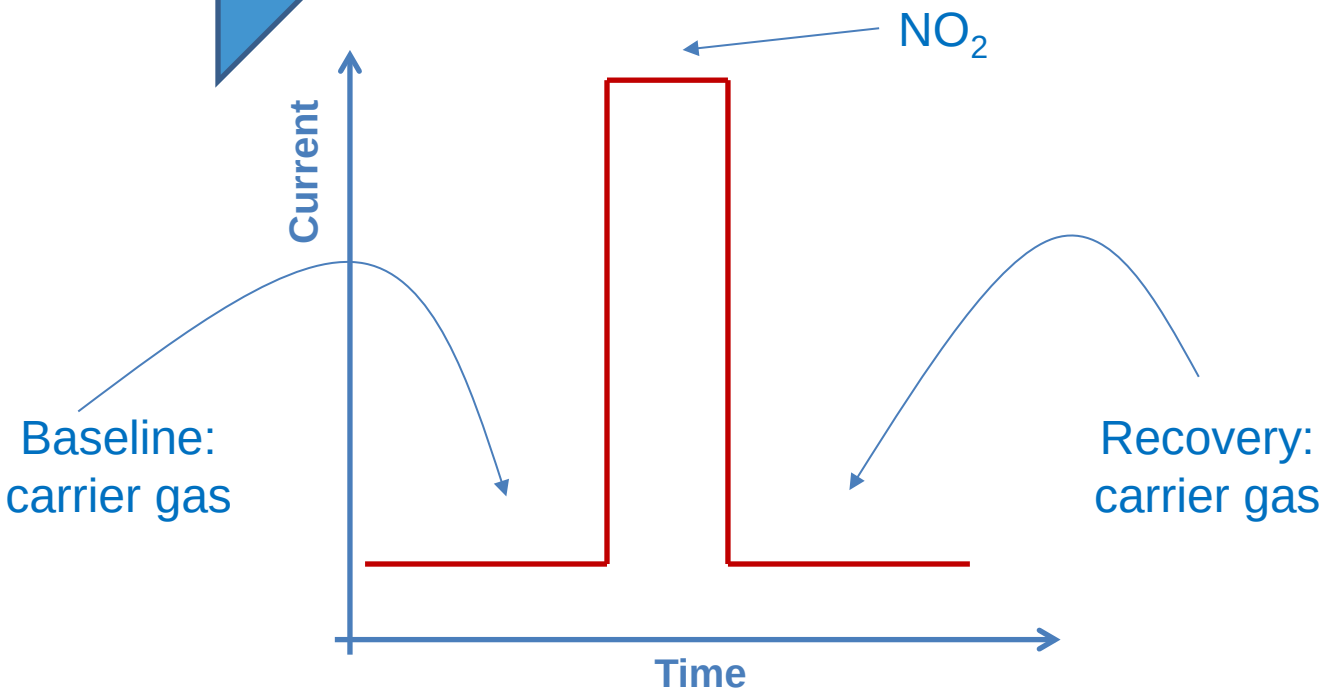
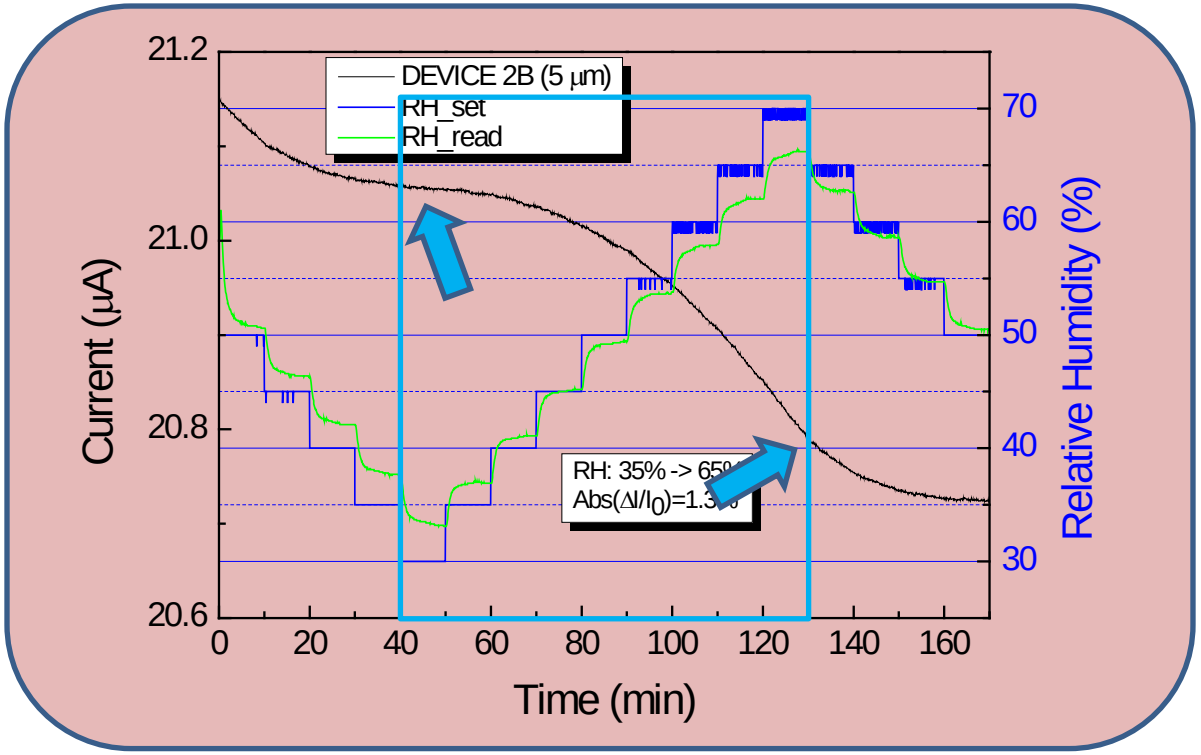


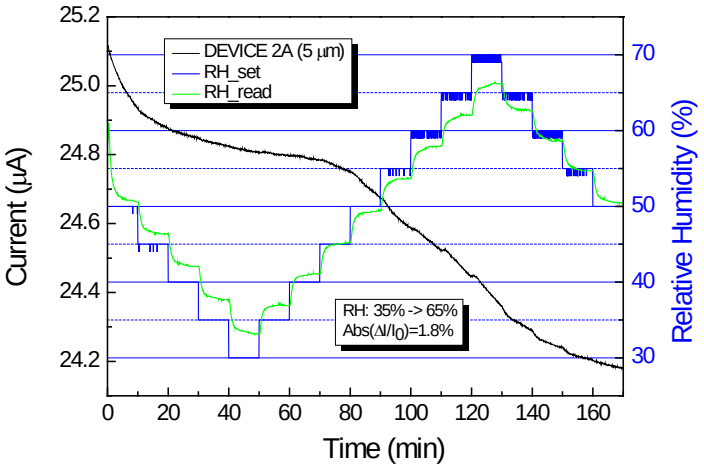


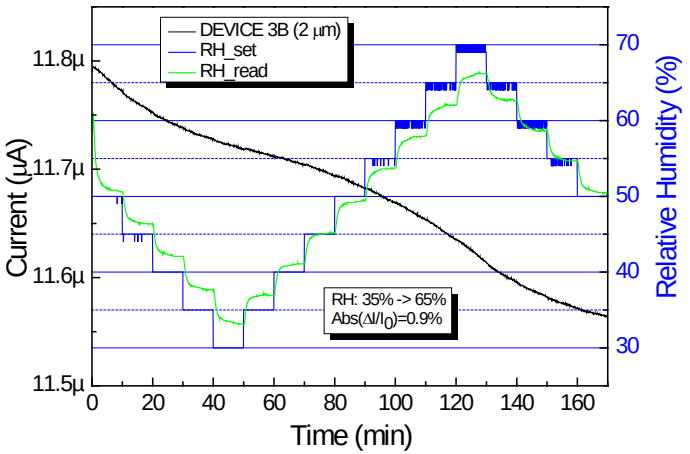
RH test

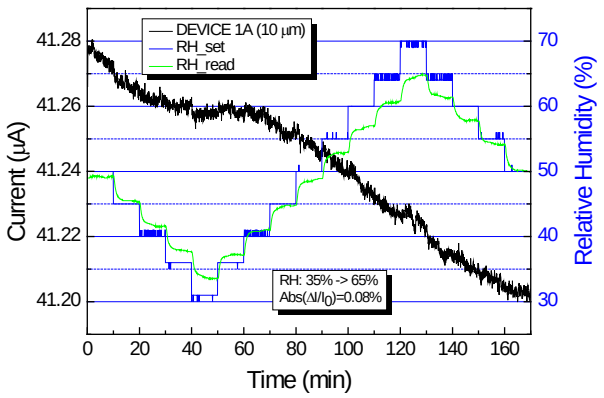


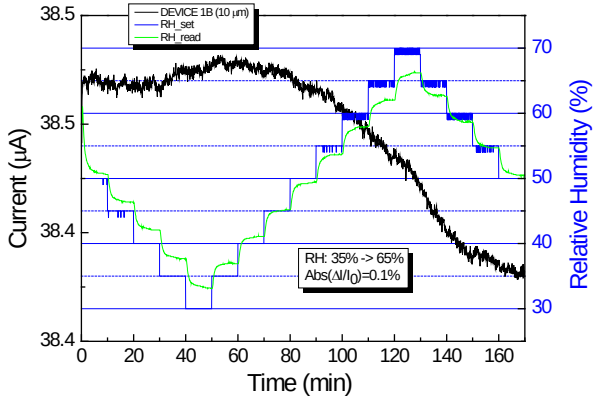
- Drift of base conductance
- Slow kinetics
- RH: 30% -> 70% $|\Delta I/I_0|$~1.3%

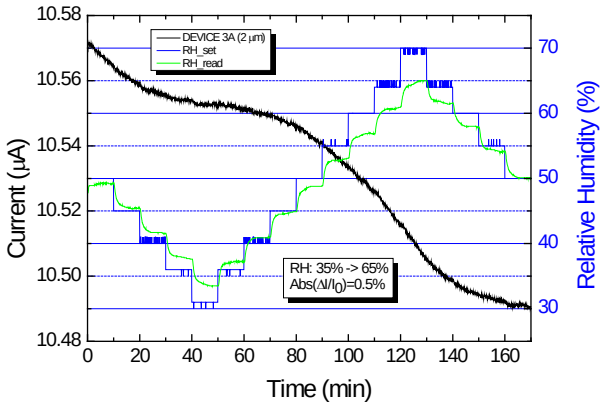
RH tests


DEVICE 2A (5 µm)
RH_set
RH_read
RH: 35% -> 65%
Abs(ΔI/I0)=1.8%
Current (µA)
Relative Humidity (%)
Time (min)

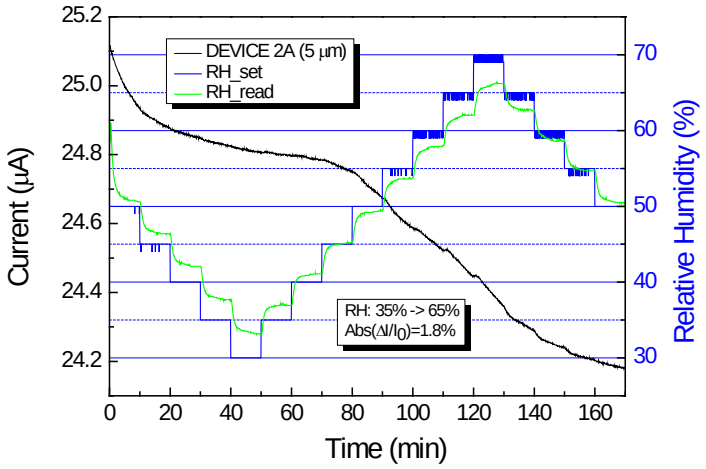

DEVICE 3B (2 µm)
RH_set
RH_read
RH: 35% -> 65%
Abs(ΔI/I0)=0.9%
Current (µA)
Relative Humidity (%)
Time (min)

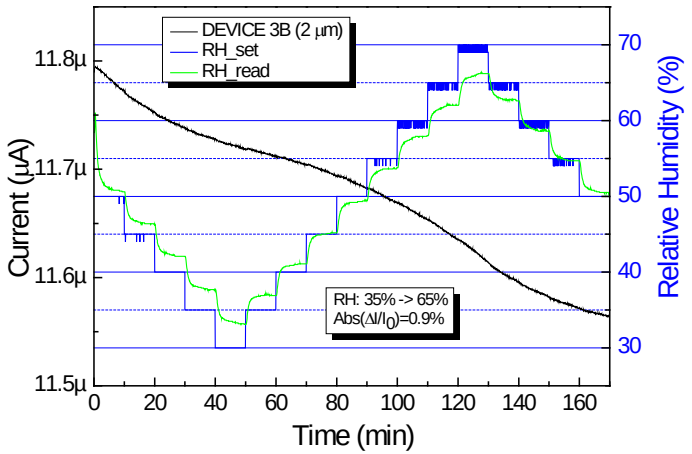

DEVICE 1A (10 µm)
RH_set
RH_read
RH: 35% -> 65%
Abs(ΔI/I0)=0.08%
Current (µA)
Relative Humidity (%)
Time (min)


DEVICE 1B (10 µm)
RH_set
RH_read
RH: 35% -> 65%
Abs(ΔI/I0)=0.1%
Current (µA)
Relative Humidity (%)
Time (min)


DEVICE 3A (2 µm)
RH_set
RH_read
RH: 35% -> 65%
Abs(ΔI/I0)=0.5%
Current (µA)
Relative Humidity (%)
Time (min)

RH tests





- Slow kinetics
- Drift of base conductance
- Repeatability
- Current variation <2%
- Real environment: RH slower variations

	Graphene bar (µm)		RH: 30% -> 70%
	Length	Width	$\|\Delta I/I_0\|$ (%)
Device 1A	206	10	0.08
Device 1B	206	10	0.1
Device 2A	206	5	1.8
Device 2B	206	5	1.3
Device 3A	206	2	0.9
Device 3B	206	2	0.5

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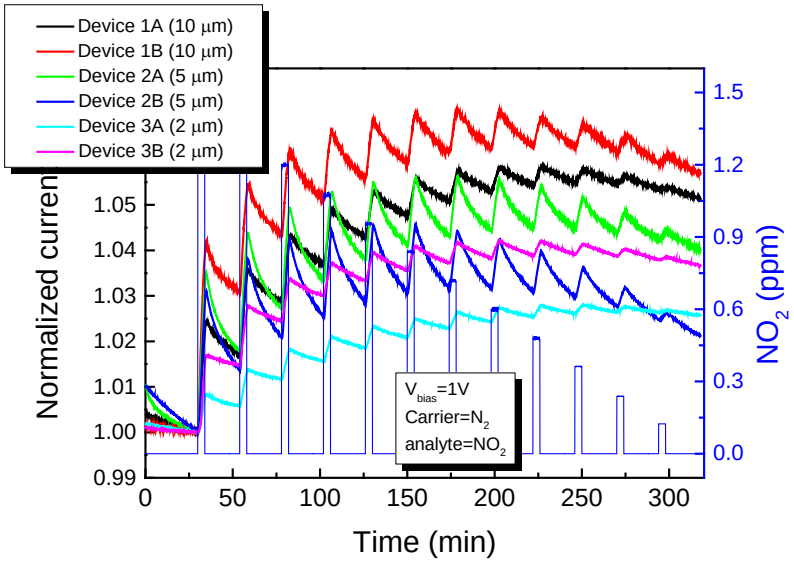
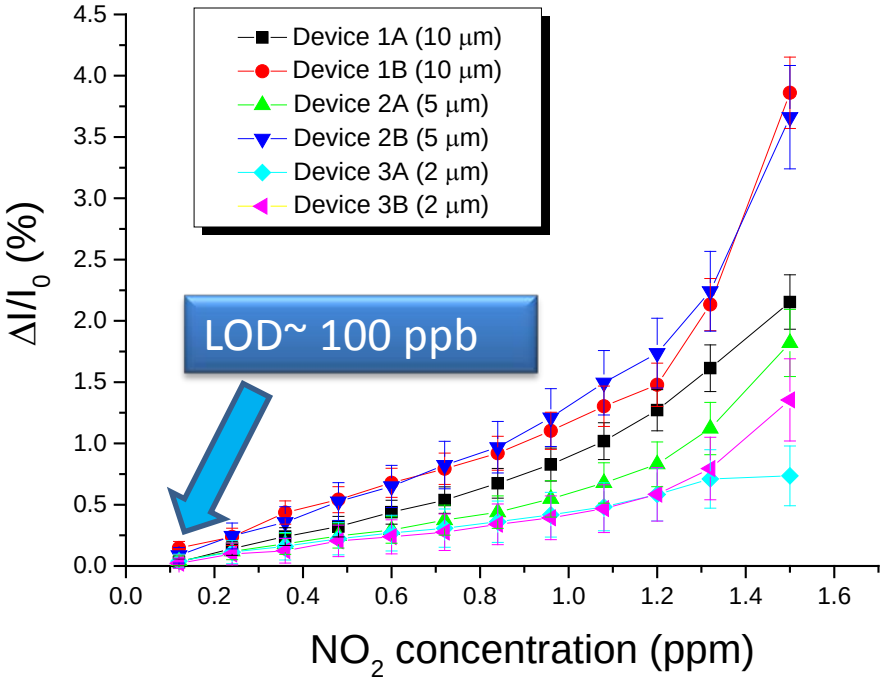
Tests towards NO_2



Test protocol

1. Baseline: 20 min N_2
2. Exposure: 4 min NO_2 @ 0.1-1.5 ppm
3. Recovery phase: 10 min N_2

- Real environment: no drastic RH variation
- @RH=50%: separated contributions
- LOD~ 100 ppb



F. Ricciardella et al., in preparation

Conclusions

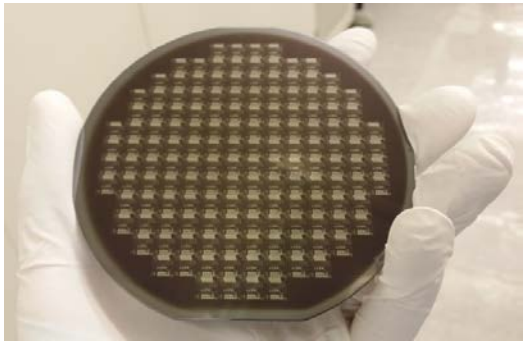
FABRICATION PROCESS

- Transfer-free graphene-based gas sensors
- Wafer scale production (>700 devices/wafer)
- High yield of working devices (>97%)

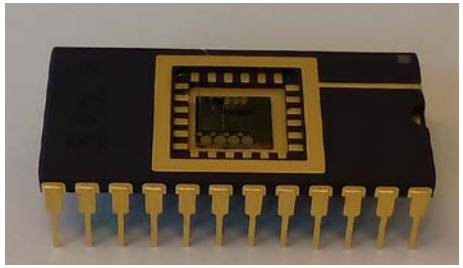
SENSORS

- Distinguishible RH effects on NO_2 sensing
- Repeatability on 2-5-10 µm devices
- High sensitivity in 0.1-1.5 ppm
  - ➢ LOD ~ 100 ppb @ RT

From LAB…



…to FAB



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Acknowledgment

Italian national agency for new technologies, energy and sustainable economic development

…Thanks!

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