# Électrodes à cristaux bidimensionnelles pour micro/nano-résonateurs piézoélectriques à haut facteur de qualité


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# Outline

1. Introduction
2. Graphene transfer
3. Application to quartz resonators
    a. Direct transfer and patterning
    b. Conductive ink deposition
    c. Ion etching of graphene
4. Conclusion

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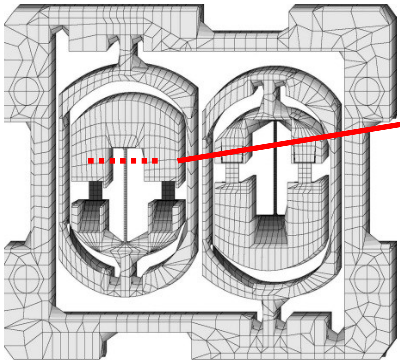
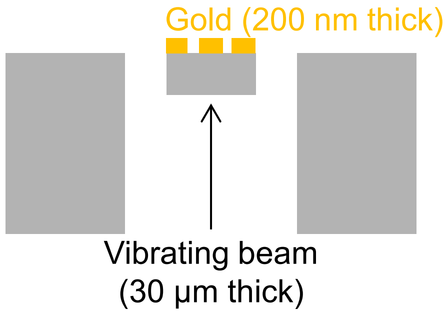
# 1. Introduction
## Graphene electrodes for MEMS resonators



*DIVA accelerometer*

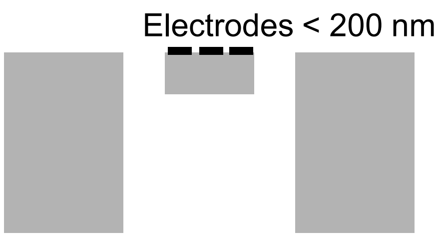



*DIVA accelerometer on its packaging*

- Main damping mechanisms for flexural beams:

$$Q_{TED} \sim 560\ 000\ @\ 22\text{kHz}$$
$$Q_{visc} \sim 140\ 000$$

- **Viscoelastic losses proportional to the electrode thickness:**



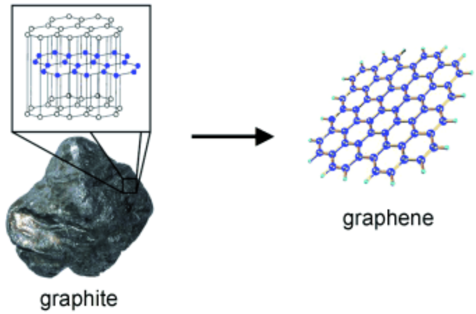

$$\frac{1}{Q_{visc}} = \omega \frac{\eta_{electrode}}{E_{cristal}} \frac{t_{electrode}}{t_{cristal}}$$

➔ Aim: replace gold by a thinner material onto the vibrating region

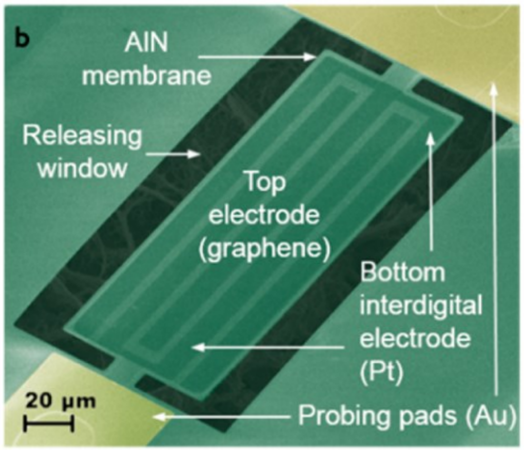
O. Le Traon et al., *Solid State Sciences* (2010), pp. 318-324.

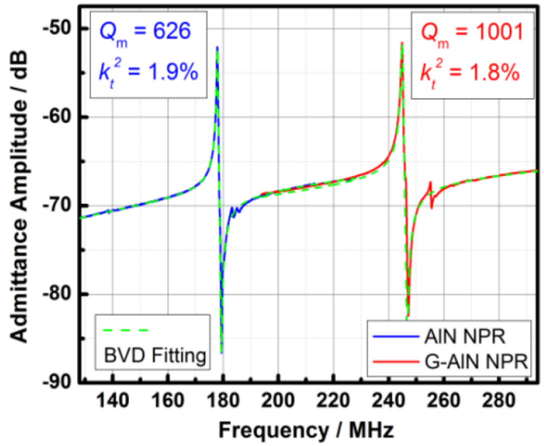
# 1. Introduction
## State of the art

- Graphene:
  - 2D crystal, derivative of graphite
  - One atom thick layer of C atoms
  - Low intrinsic viscosity (?)




- Aluminum nitride (AlN) resonator with a graphene top electrode

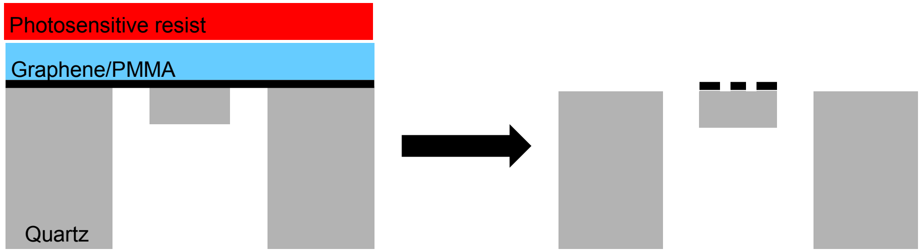


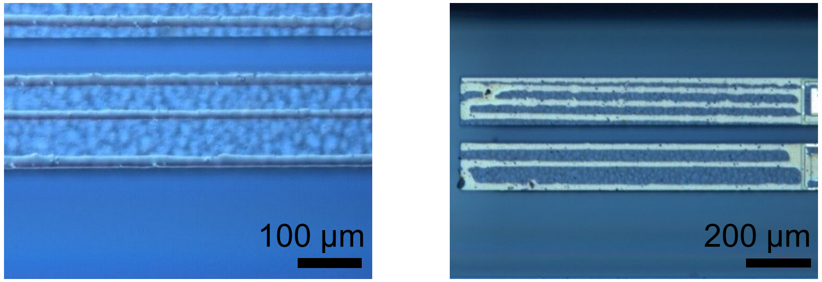

Z. Qian et al., *Proceedings of the 2013 IEEE International Frequency Control Symposium*, pp. 559-561.

- ✓ Higher resonance frequency and improved quality factor
- × Bottom platinum electrode

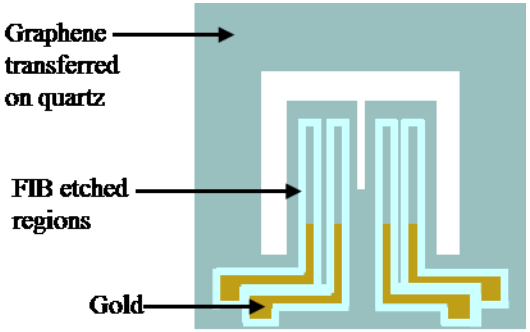
- Different strategies for **less intrusive electrodes onto the vibrating parts of quartz resonators:**
  - **Direct transfer and patterning on resonators**




  - **Conductive inks**




  - **Focused Ion Beam (FIB) etching of graphene**



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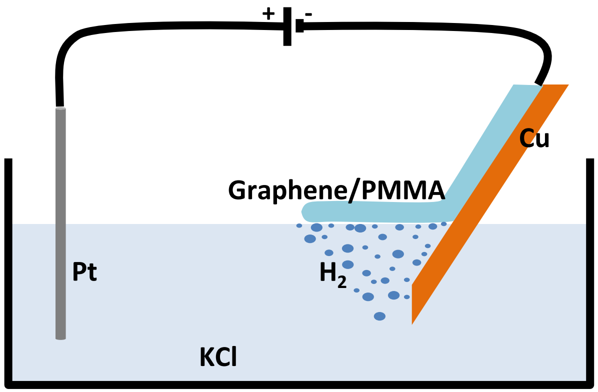
*Graphenea*

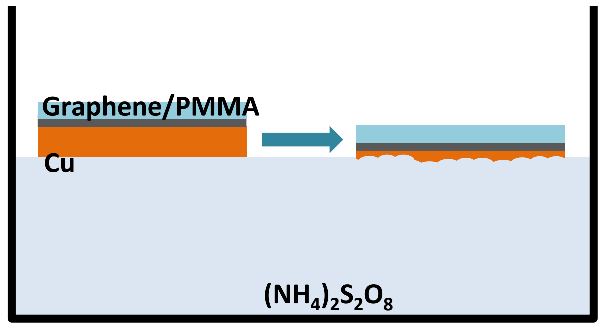
- Graphene monolayer grown by Chemical Vapor Deposition (CVD) on a copper substrate
- Transfer = graphene separation from copper + transfer on target substrate


Spin coating of PMMA resist on top of graphene for mechanical support during the transfer process

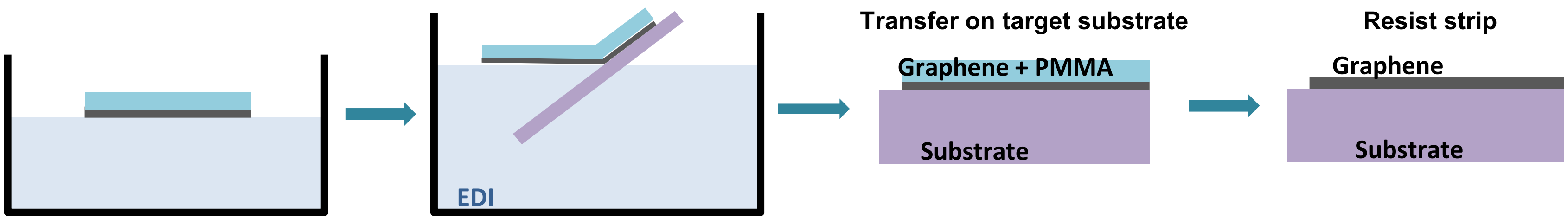
### Electrochemical delamination




### Copper etching

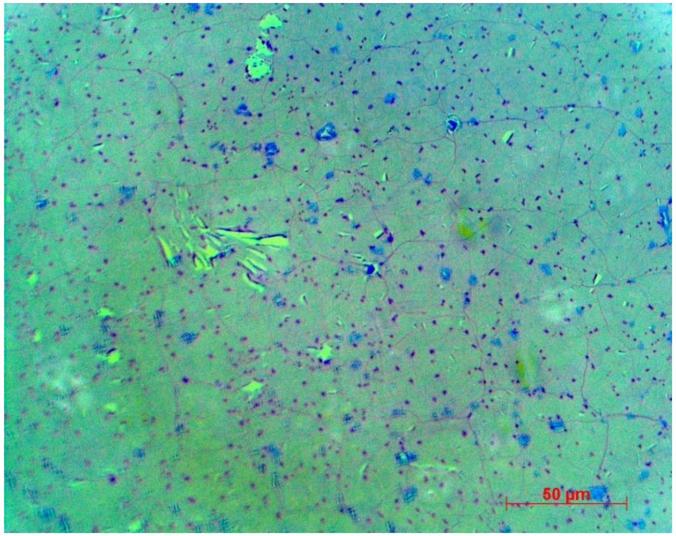


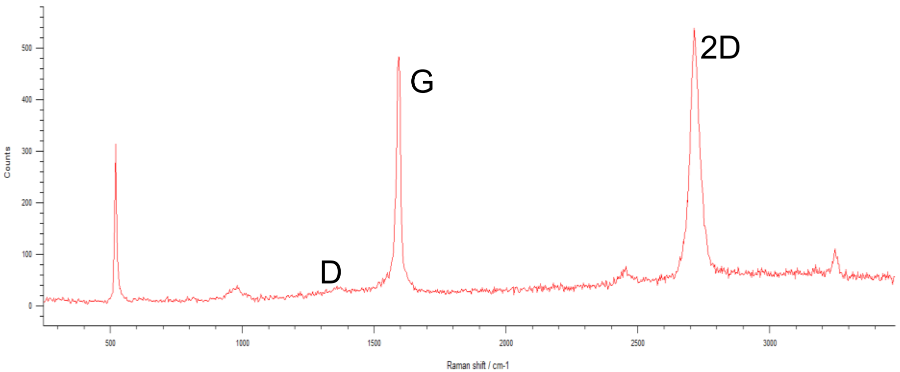

### Transfer



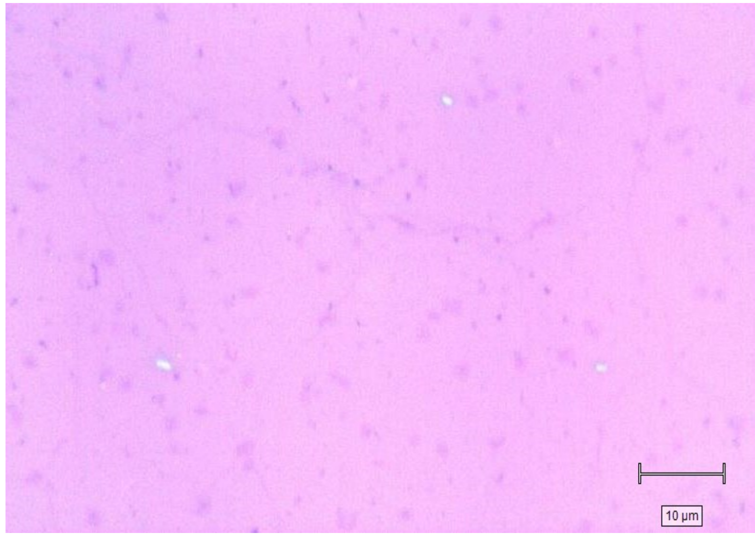
# 2. Graphene transfer and patterning
## Transfer techniques – Graphene characterization

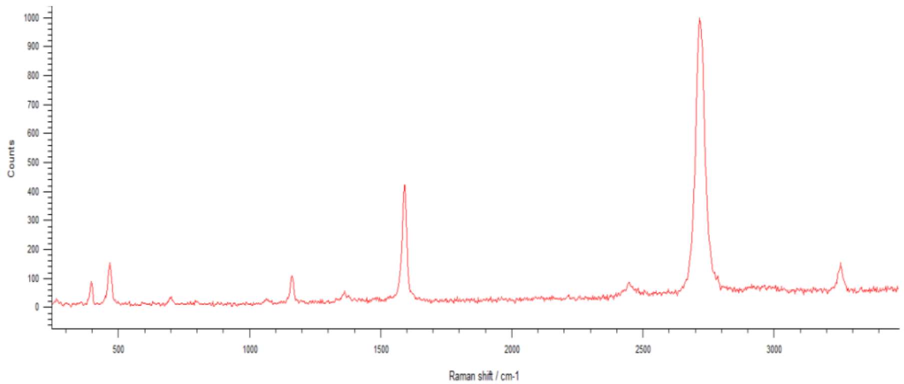
**Electrochemical delamination**




**Copper etching**




- ✓ Low density of defects for both processes
- ✓ No luminescence (no resist residue)
- ✓ Less cracks with copper etching

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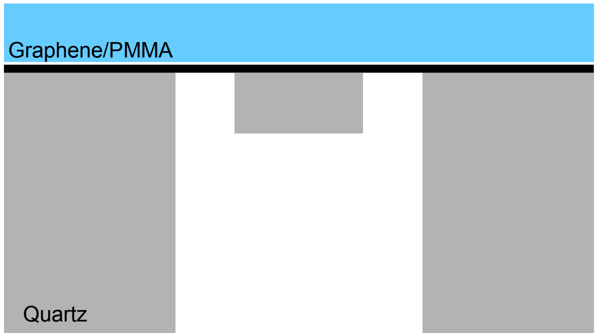
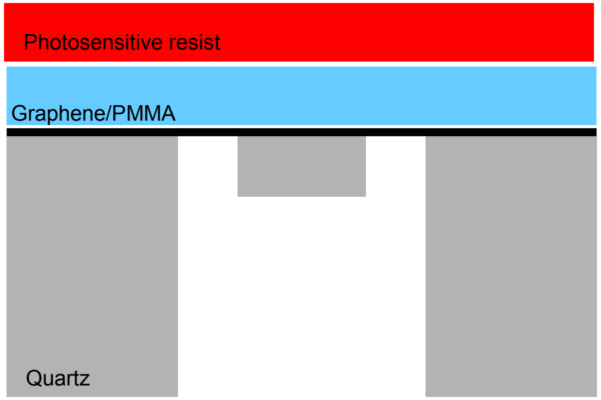
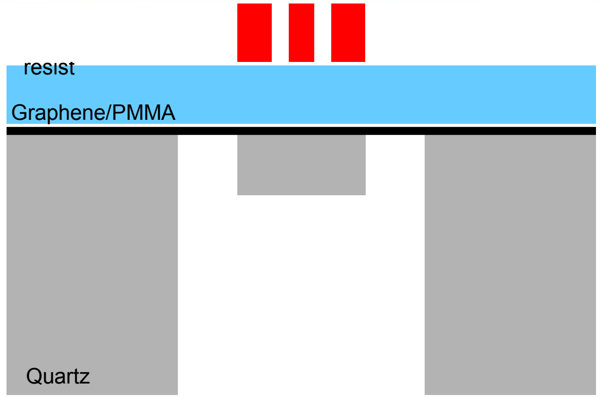
# 3. Application to quartz resonators
## a. Direct transfer and patterning



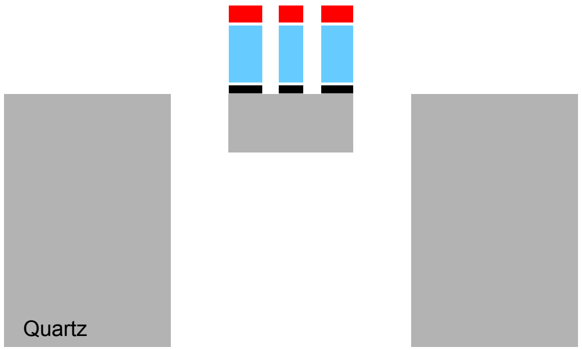

1. Graphene/PMMA transfer



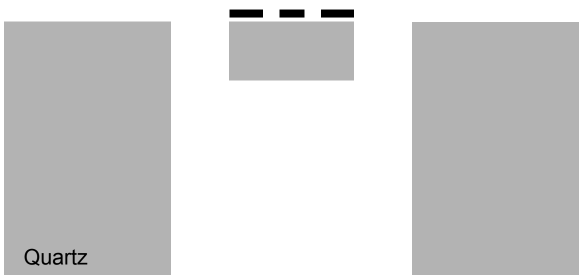

2. Spin coating of photoresist



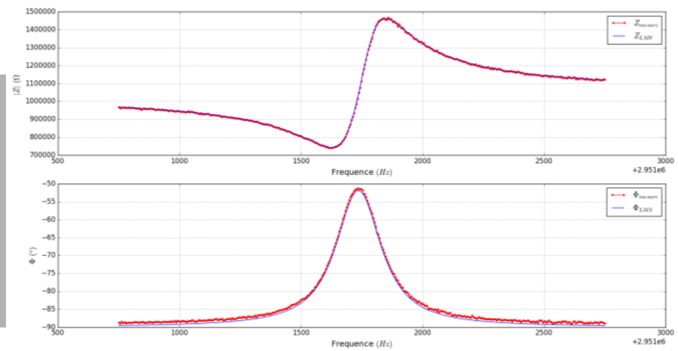

3. Exposure and developement




4. Plasma etching




5. Resists dissolution



ARQOMM ➔ Q = 14 000 @ 2.9 MHz
7 000 with gold electrodes
✓ **Enhancement of Q at atmospheric pressure**

<u>Future work:</u>
- Process optimization to obtain continuous electrodes
- Characterization under vacuum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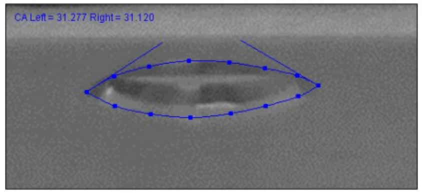
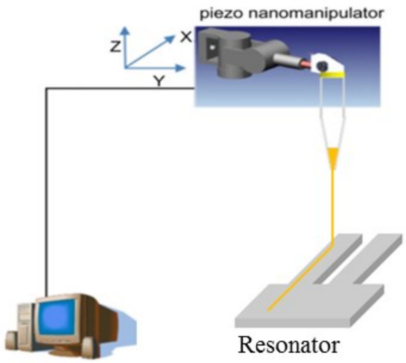
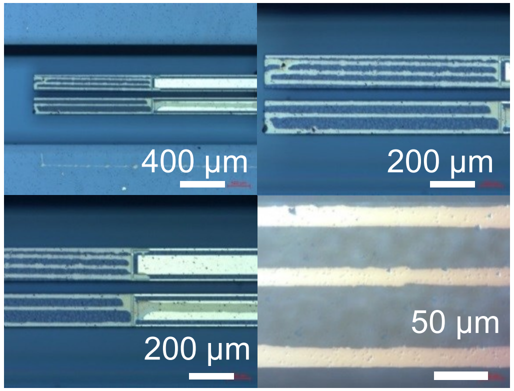
# 3. Application to quartz resonators
## b. Conductive ink deposition – Gold nanoink

- Collaboration with Gilgueng Hwang (C2N)
- Wettability of quartz resonators after the fabrication process




Contact angle left: 31,3°
Contact angle right: 31,1°
➔ hydrophilic




Gold nanoparticles between 2 and 4 nm in diameter

✓ Fine lateral resolution (< 20 µm)

- Gold nanoparticles electrodes on a Vibrating Inertial Gyro (VIG)

| | Gold nanoparticles electrodes (~100 nm) | | Evaporated gold electrodes (200 nm) | |
|---|---|---|---|---|
| | **Frequency (Hz)** | **Quality factor** | **Frequency (Hz)** | **Quality factor** |
| **Drive mode** | 21 825 | 144 640 | 22 387 (+/-0.2%) | 115 000 (+/-5%) |
| **Sense mode** | 22 889 | 146 850 | 23 267 (+/-1.5%) | 137 000 (+/-5%) |

✓ No significant shift in resonance frequency

✓ **Enhancement of Q** compared to standard gyros

$Q_{drive}$: + 26%

$Q_{sense}$: + 6% (limited by the broken decoupling frame?)

➔ Promising first results

Further work:

- Gold nanoparticles electrodes < 100nm thick
- Graphene inks



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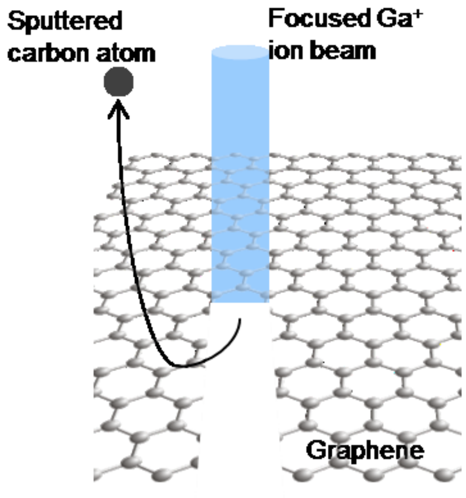
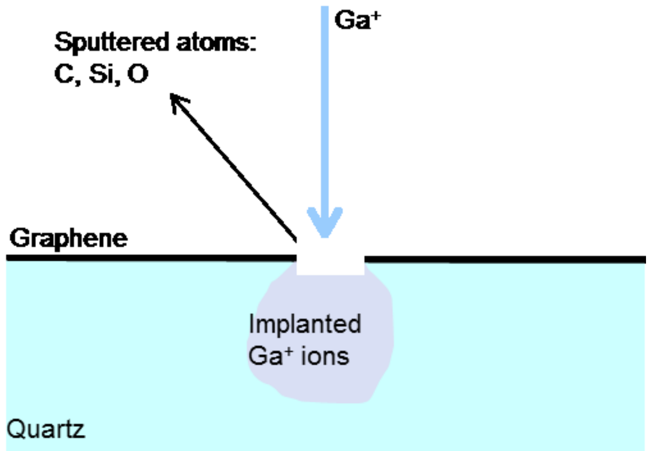
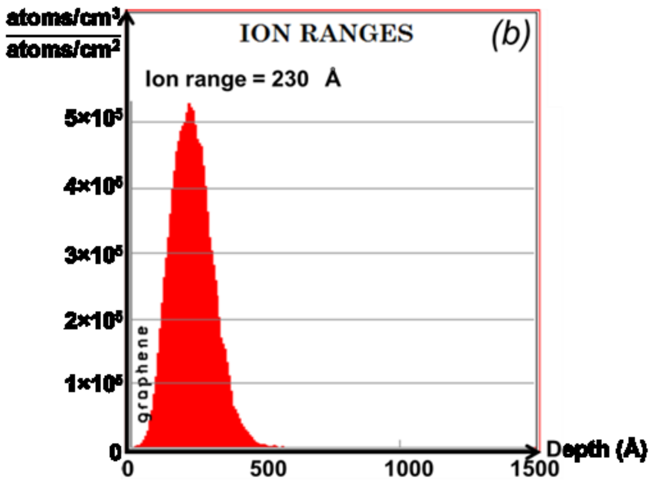
# 3. Application to quartz resonators
## c. Etching of the graphene electrodes on a VIG

- Etching of graphene by Focused Ion Beam (FIB)



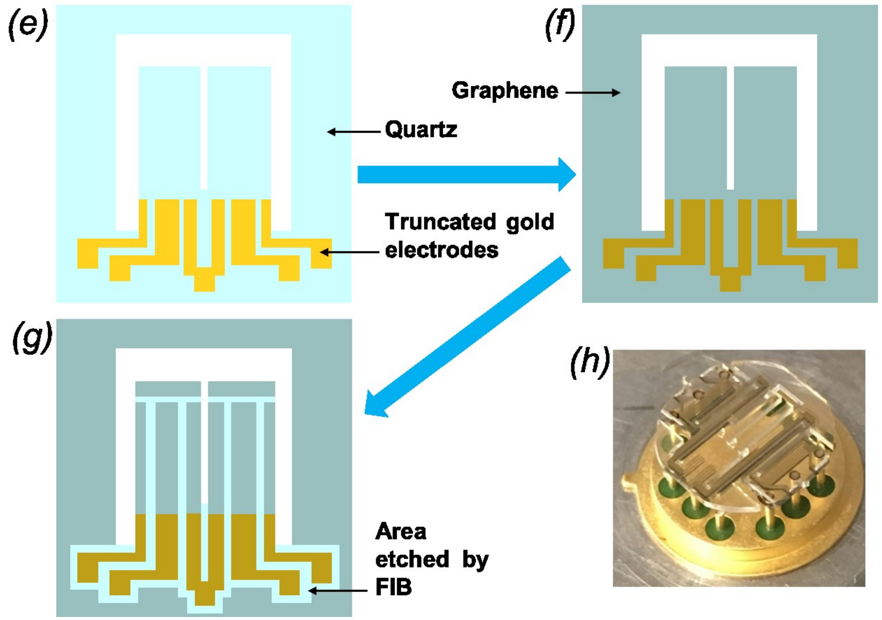




- Stopping and Range Ions in Matter (SRIM) simulation of 30 keV $Ga^+$ ion tracks into a monolayer of graphene on top of a quartz substrate




Normal projected range = **23 nm**
Lateral projected range = **5 nm**
Quartz thickness etched by FIB ~ **3 nm**
**→ Negligible compared to the normal projected range**

# 3. Application to quartz resonators
## c. Etching of the graphene electrodes on a VIG




| | Graphene electrodes | | Gold electrodes | |
|---|---|---|---|---|
| | $f_{resonance}$ (Hz) | Q | $f_{resonance}$ (Hz) | Q |
| **Drive mode** | 22 031 | 46 000 | 22 387 (+/- 0.2%) | 115 000 (+/- 5%) |
| **Sense mode** | 23 157 | 54 000 | 23 267 (+/- 1.5%) | 137 000 (+/- 5%) |

- ✓ No significant shift in resonance frequency
- × **Degradation of Q** compared to standard gyros $Q = 2.5 \times Q_{FIB}$

# 3. Application to quartz resonators
## c. Etching of the graphene electrodes on a VIG

**Degradation of Q by a factor 2.5**

Hypothesis:

- Intrinsic losses at the **graphene/quartz interface**

| | Graphene | Gold electrodes |
|---|---|---|
| Sense mode | 160 000 (+/-6%) | 137 000 (+/- 5%) |

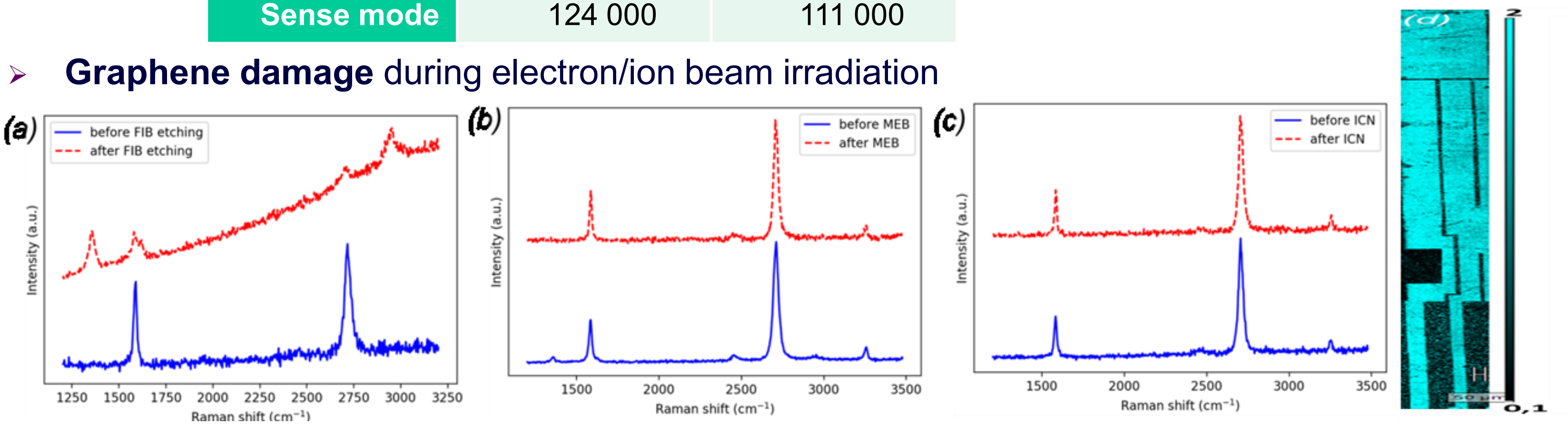
✓ **Improvement of Q with graphene**

- **Quartz amorphization** through ion implantation

| | Q before FIB | Q after FIB |
|---|---|---|
| Drive mode | 112 000 | 105 000 |
| Sense mode | 124 000 | 111 000 |

Slight decreasing of Q attibuted to the dissipation through redepositing of sputtered material

- **Graphene damage** during electron/ion beam irradiation




× **Characteristic peaks of defects**

× **High and homogeneous I(D)/I(G) ratio**

✓ **No graphene degradation with electron beam irradiation**

**Ion beam damage ?**

# Outline

# 4. Conclusion

- Réalisation d'un **gyromètre vibrant avec électrodes en graphène par gravure FIB**
- Caractérisations électriques : **dégradation du facteur de qualité** avec les électrodes en graphène
  - Effet de la dégradation du graphène et/ou dépôt de carbone amorphe par le faisceau d'électron? → observation MEB de résonateurs avec ou sans graphène dans différentes conditions (faisceau d'électrons focalisé ou nuage d'électrons, avec ou sans doigt froid) ➔ **pas de dégradation**
  - Amorphisation du quartz? → simili gravure FIB sur dispositif avec électrodes en or complètes et mesure de l'amortissement avant/après gravure ➔ **légère dégradation due au phénomène de redéposition**
  - Pertes à l'interface graphène/quartz? → mesure de vibrométrie ➔ Q avec graphene autour de 160 000
  - **Dégradation attribuée au faisceau d'ions → réduction du courant d'ions**

- Transfert et mise en forme du graphène sur résonateur usiné : **procédé de transfert + photolithographie**
  - Mesures à pression atmosphériques encourageantes sur des ARQOMM : **amélioration du Q d'un facteur 2**
  - Mesures sous vide en cours

- Étude de l'amortissement de **couches minces par dépôt d'encres conductrices** sur résonateurs : VIG avec électrodes en nanoparticules d'or ➔ **augmentation du facteur de qualité**
  - Dépôts d'encres graphène/CNT sur résonateurs prévus

# Merci de votre attention