



EFFECTS OF RADIOFREQUENCY PLASMA TREATMENT ON MECHANICALLY EXFOLIATED GRAPHENE OXIDE PLATELETS

M. Cardinali¹, L. Valentini¹, J. M. Kenny^{1,2}

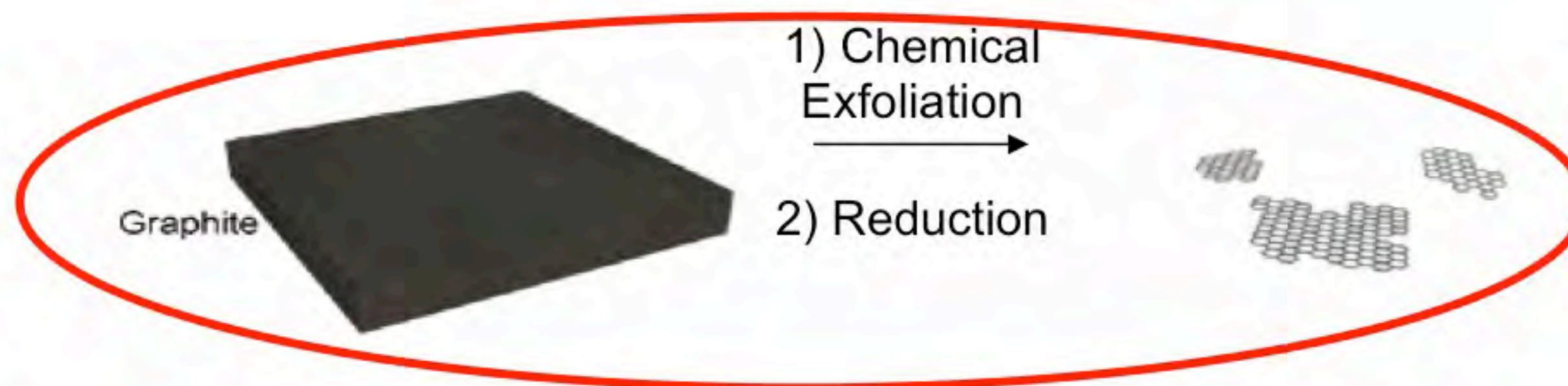
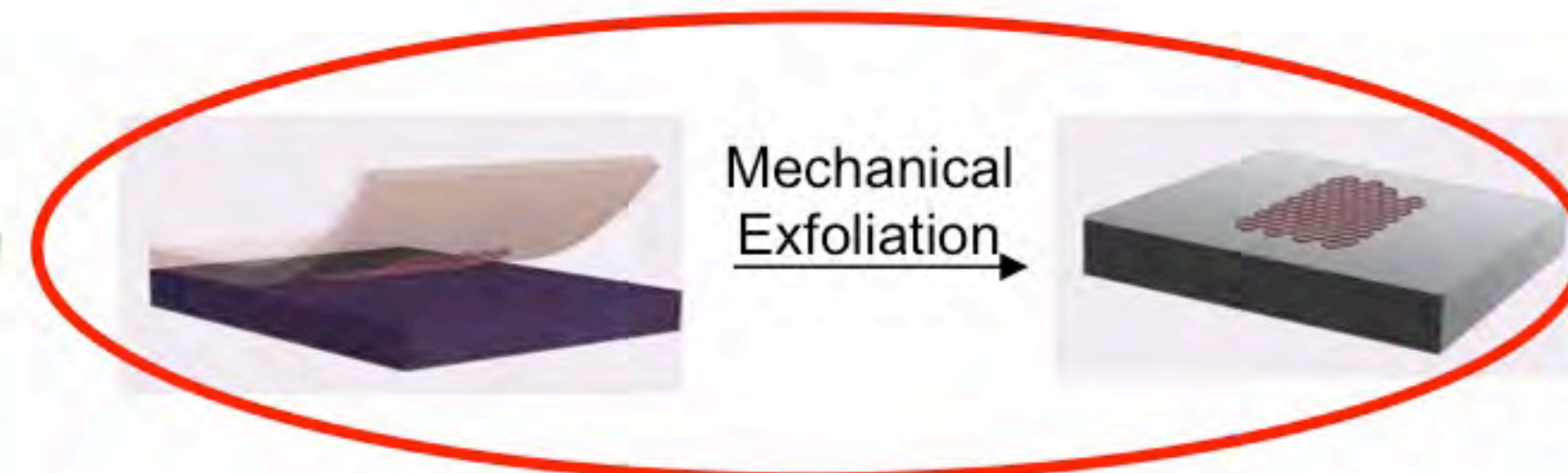
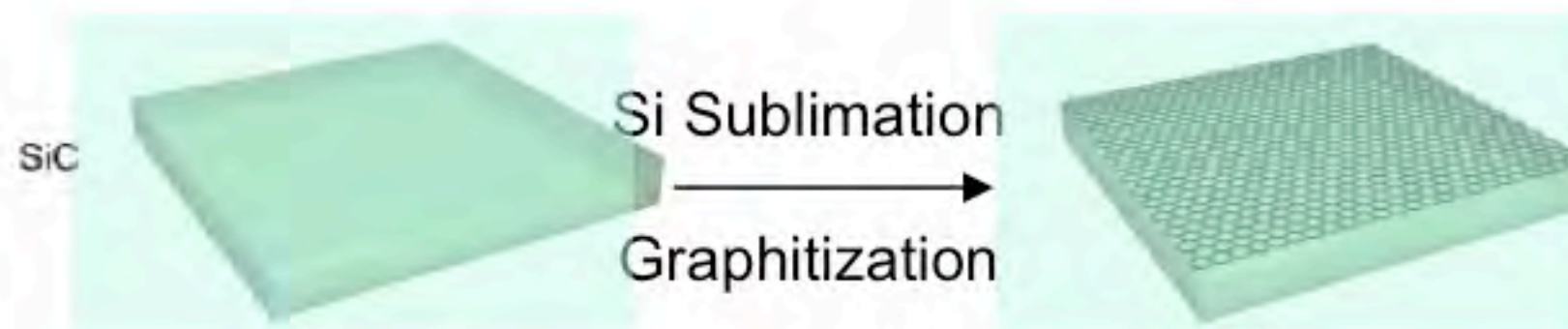
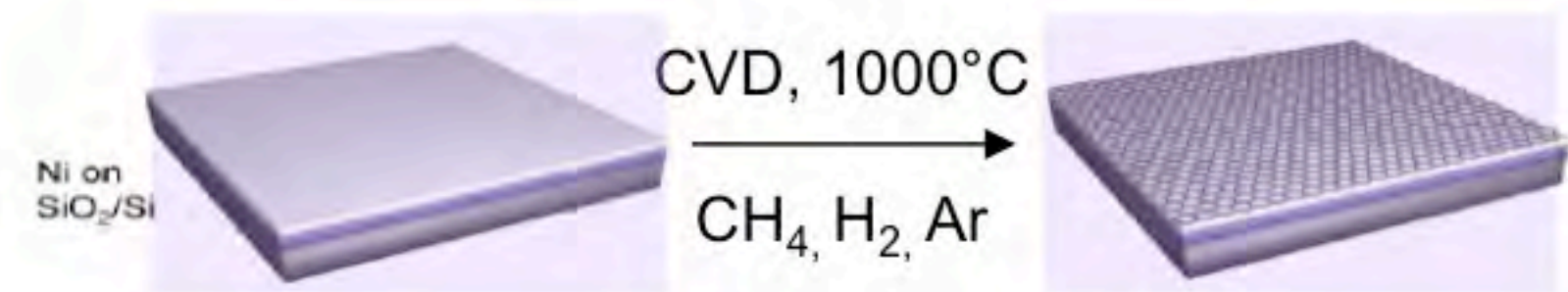
¹Dipartimento di Ingegneria Civile e Ambientale, Università di Perugia, Italy

²Instituto de Ciencia y Tecnologia de Polimeros, CSIC, Spain

OUTLINE

- **Introduction: what's the best way to produce graphene?**
- **Use of graphene oxide platelets (GOPs): properties and processing parameters**
- **Exfoliation of GOPs by plasma treatment**
- **Characterization of exfoliated GOPs**
- **Conclusions**

INTRODUCTION: PRODUCTION of GRAPHENE SHEETS



GRAPHENE VS. GRAPHENE OXIDE PLATELETS

MECHANICAL EXFOLIATION



- Easy and fast process 😊
- High quality single-layer flakes 😊
- Flakes too small for industrial application 😞

CHEMICAL EXFOLIATION



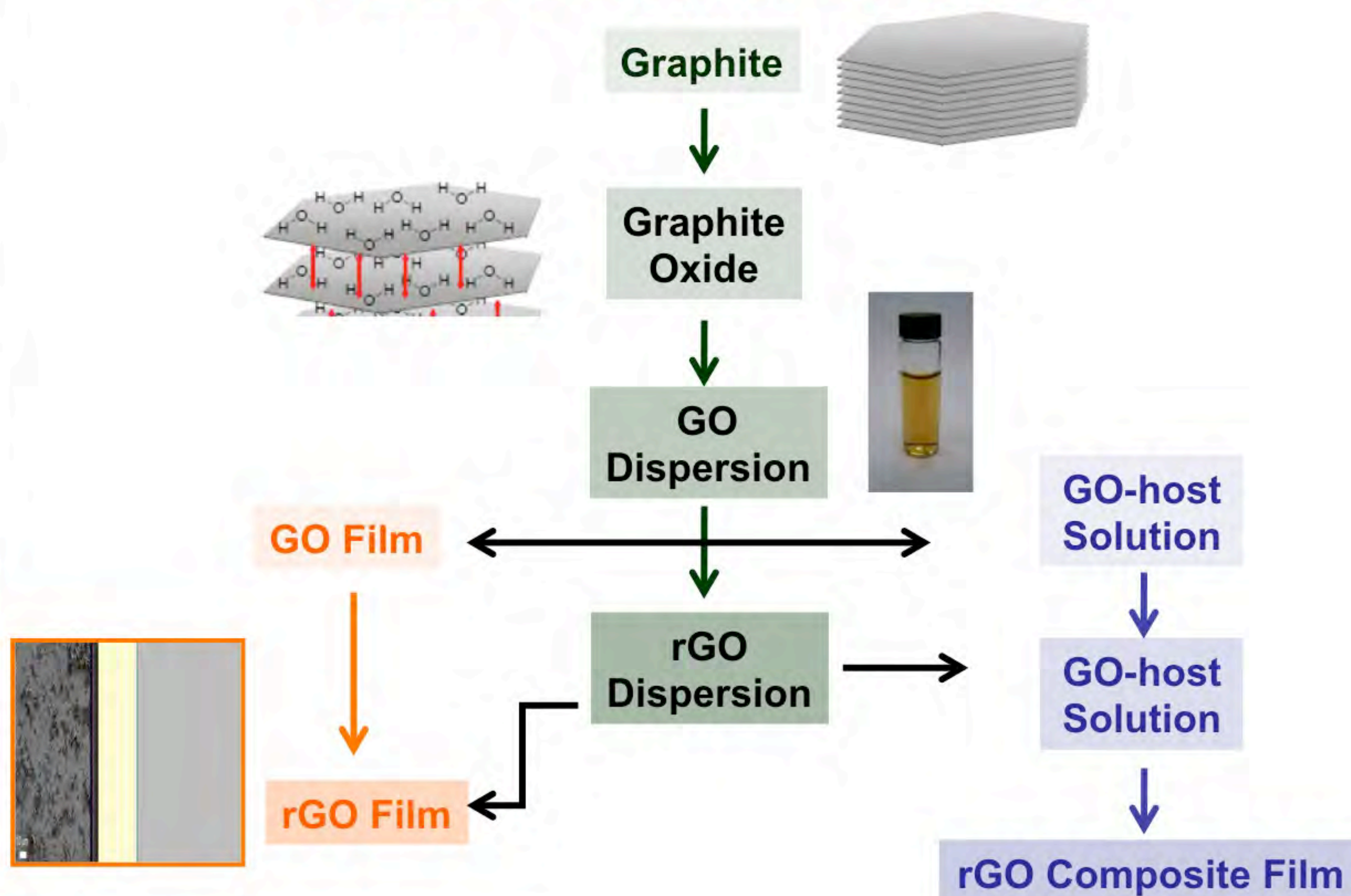
- Low cost of starting materials (i. e. graphite and/or graphite oxide) 😊
- Large scale applications 😊
- Folded final material consisting of stacked flakes 😞



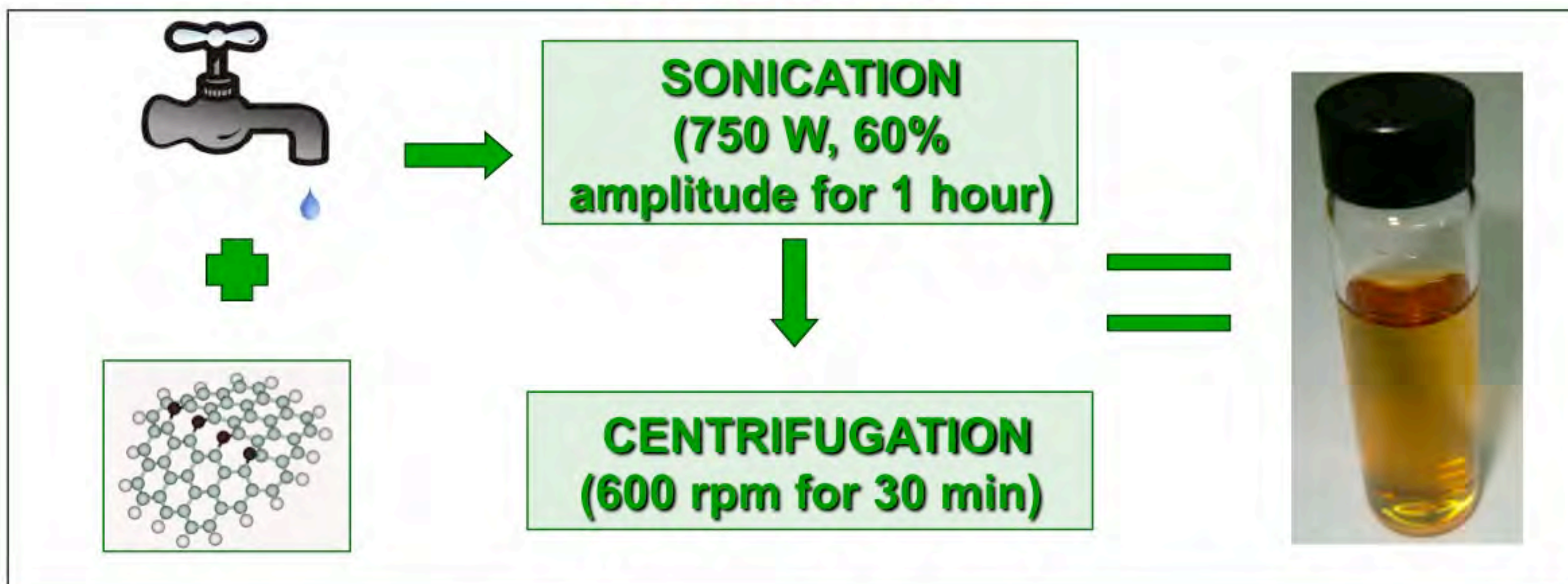
Use of mechanical exfoliation of GO sheets to obtained large flakes to be thinned...



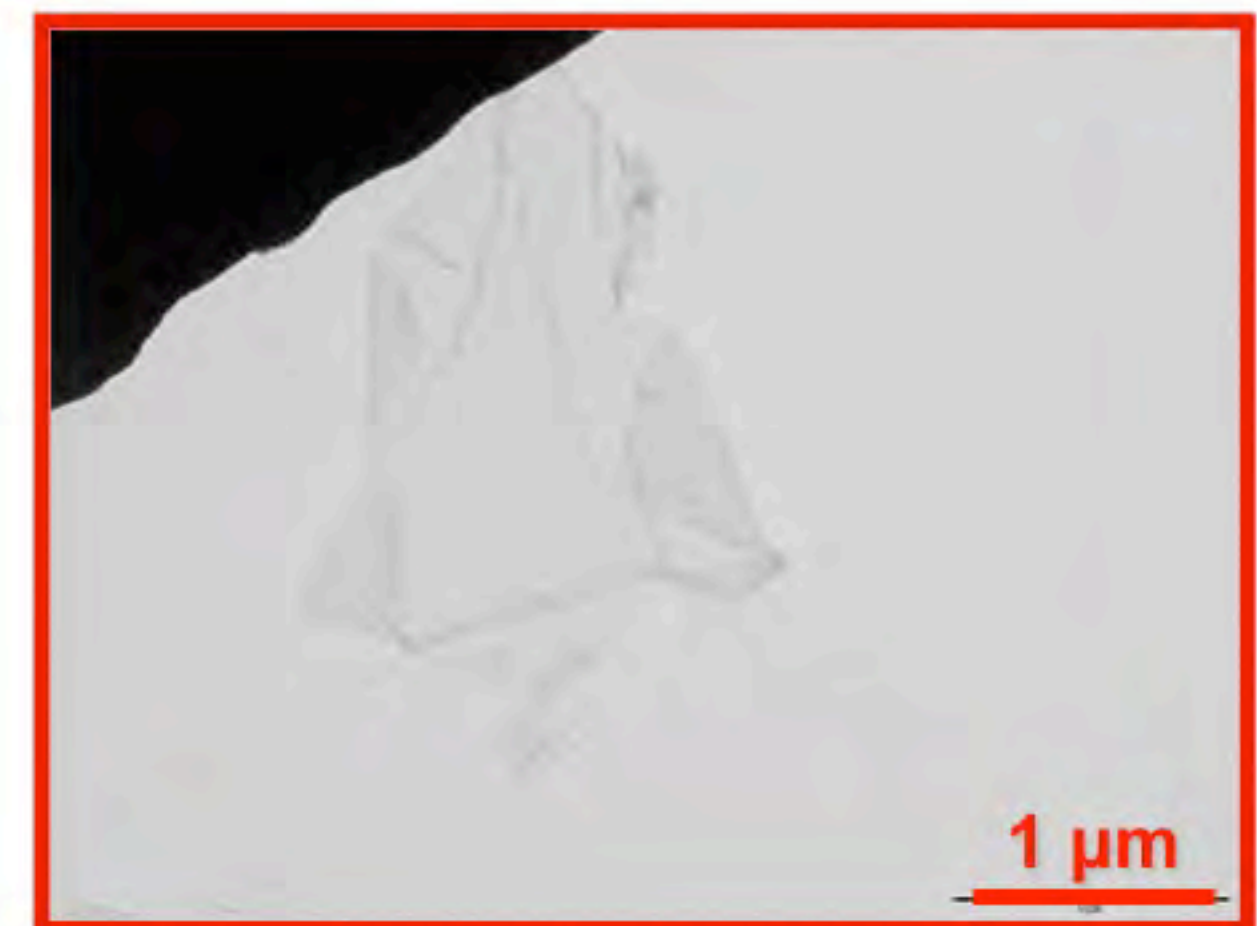
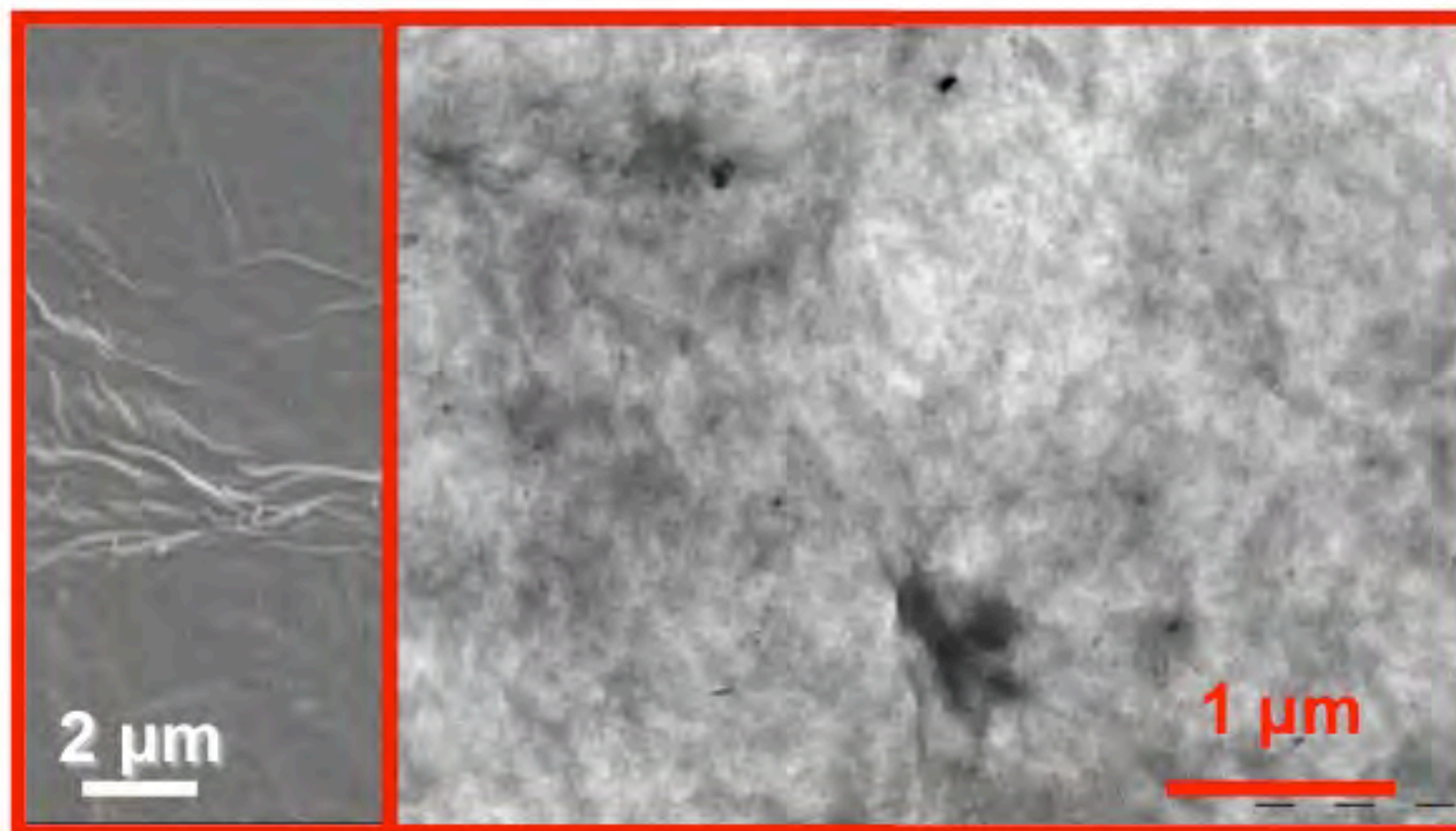
PRODUCTION of GOPs



PRODUCTION and PREPARATION of GOPs



PRODUCTION and PREPARATION of GOPs



ARGON PLASMA TREATMENT (Why?)



?

- Argon is a noble gas
- Argon has low ionization-energy (1520,6 kJ/mol)



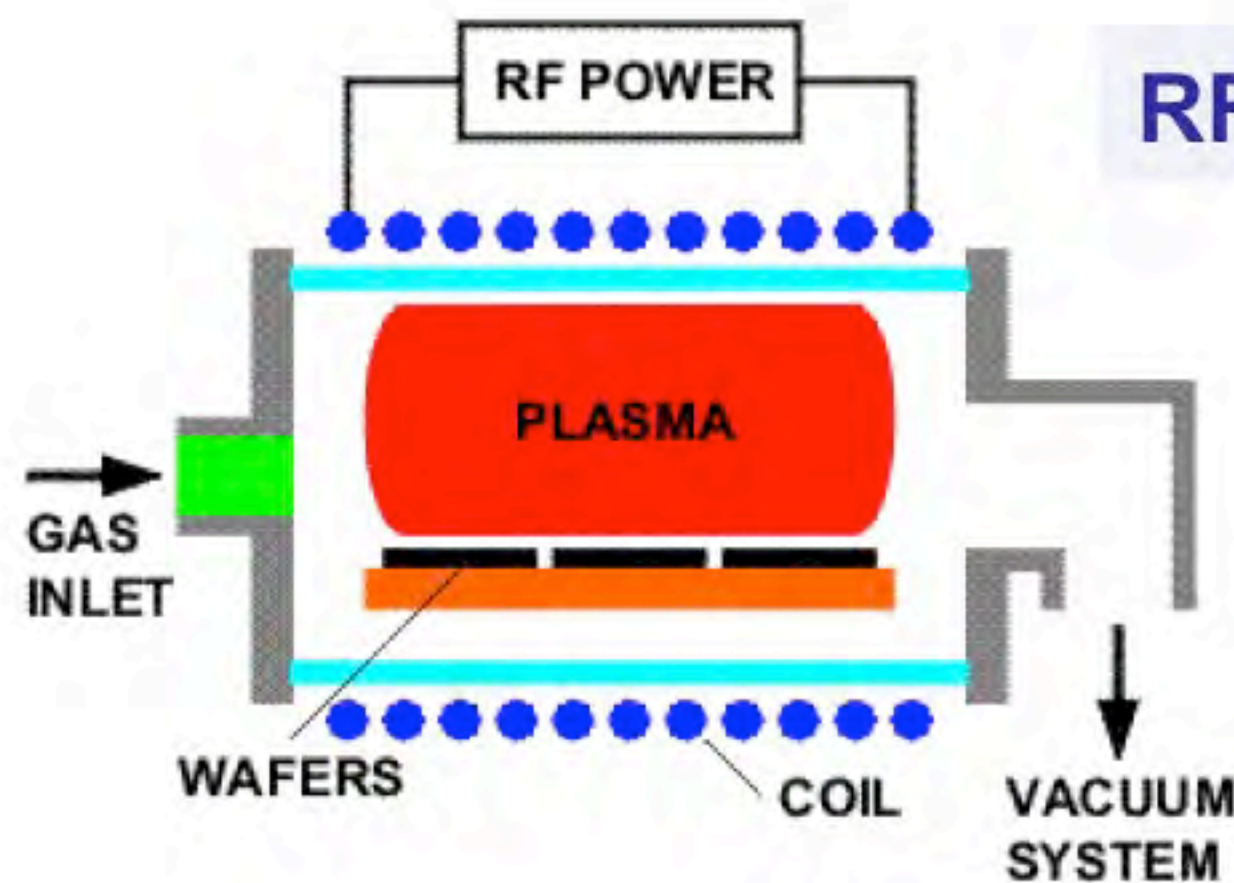
PLASMA

?

- Low cost process
- Possibility to be scaled up industrially
- Possibility to control the exfoliation degree



ARGON PLASMA TREATMENT (Why?)



RF: results in the partial ionisation of the gas

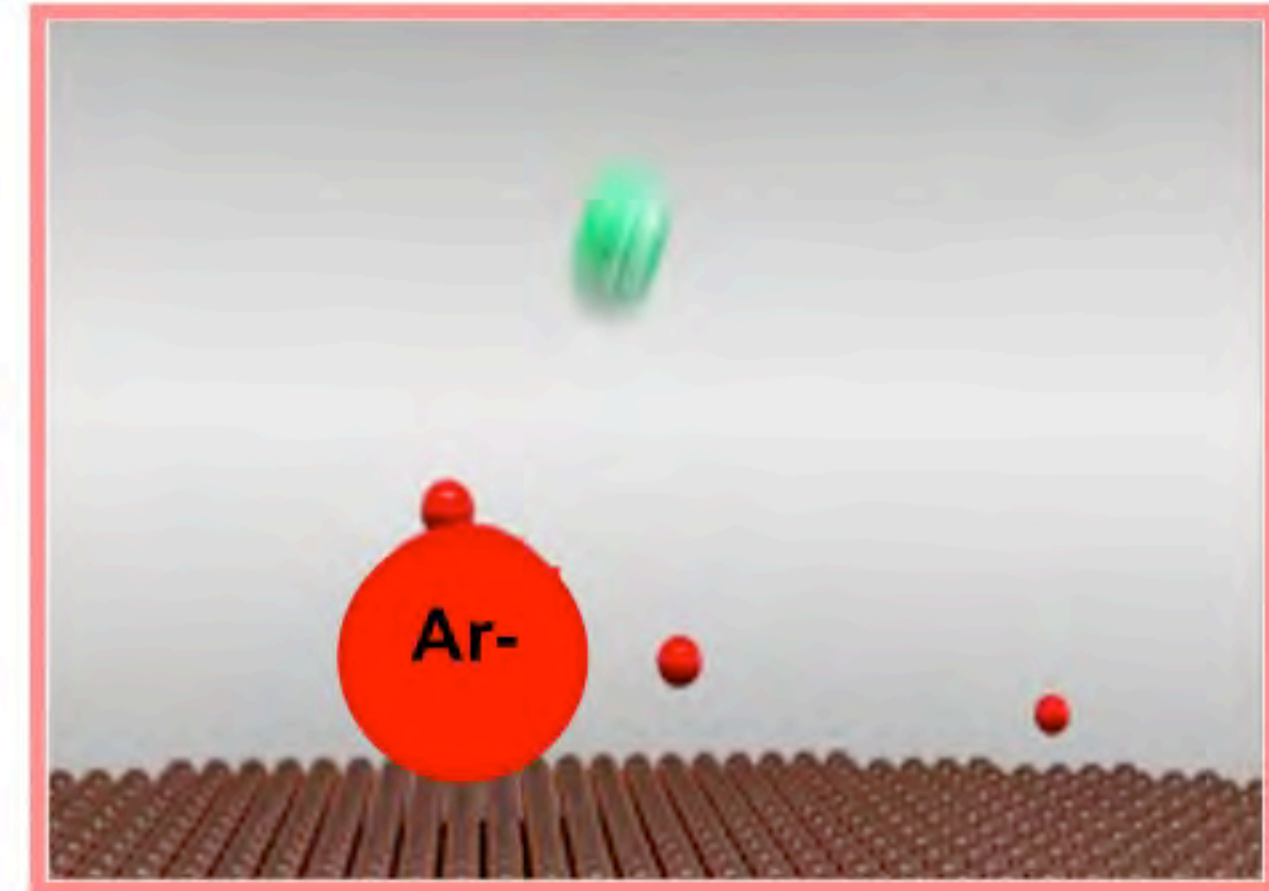
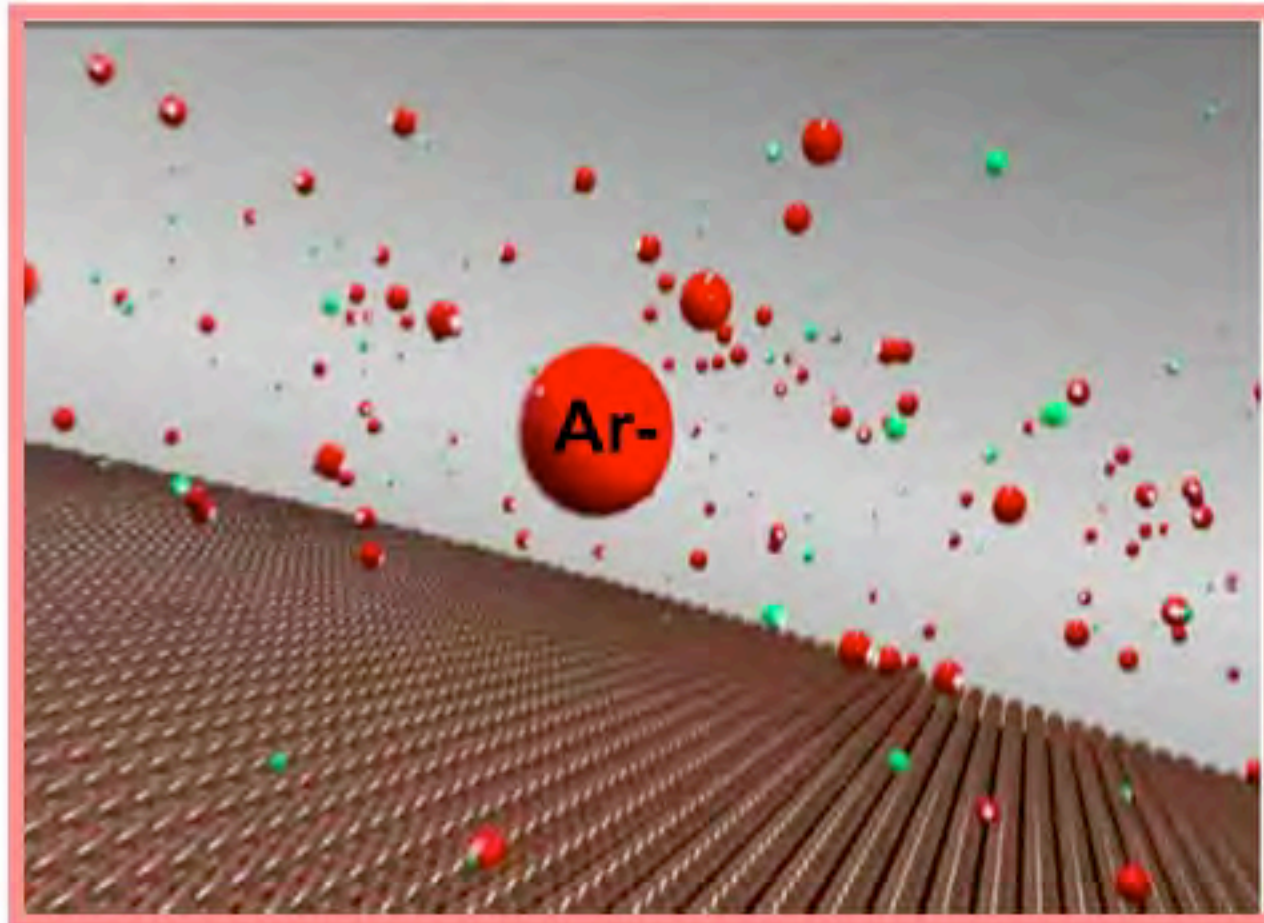


During a RF period electrons and ions accelerate towards the electrodes

RF is the right frequency (13.56 MHz) to preserve longer the ionisation process



ARGON PLASMA TREATMENT (Why?)

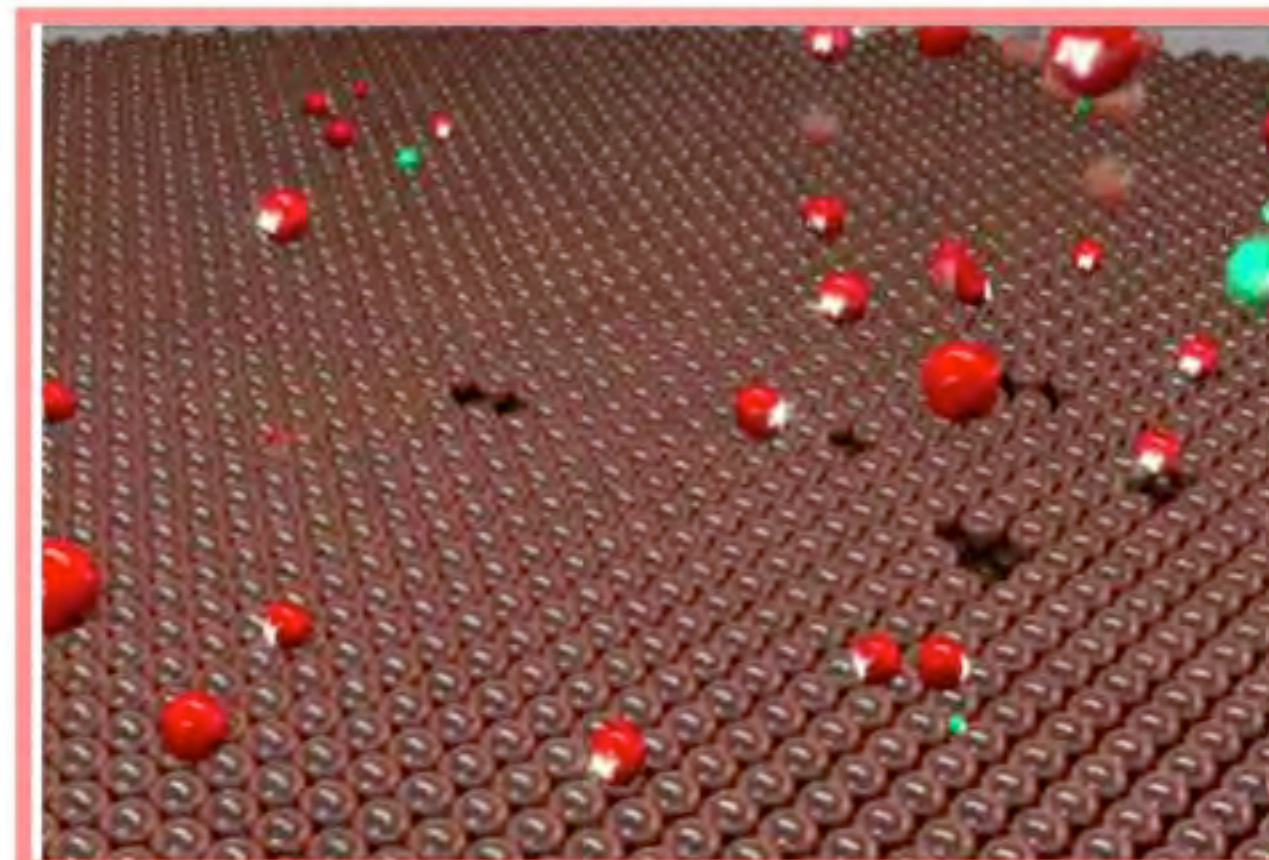


Pressure: 0,2 mTorr

Flow rate: 20 sccm

Power: 25 W

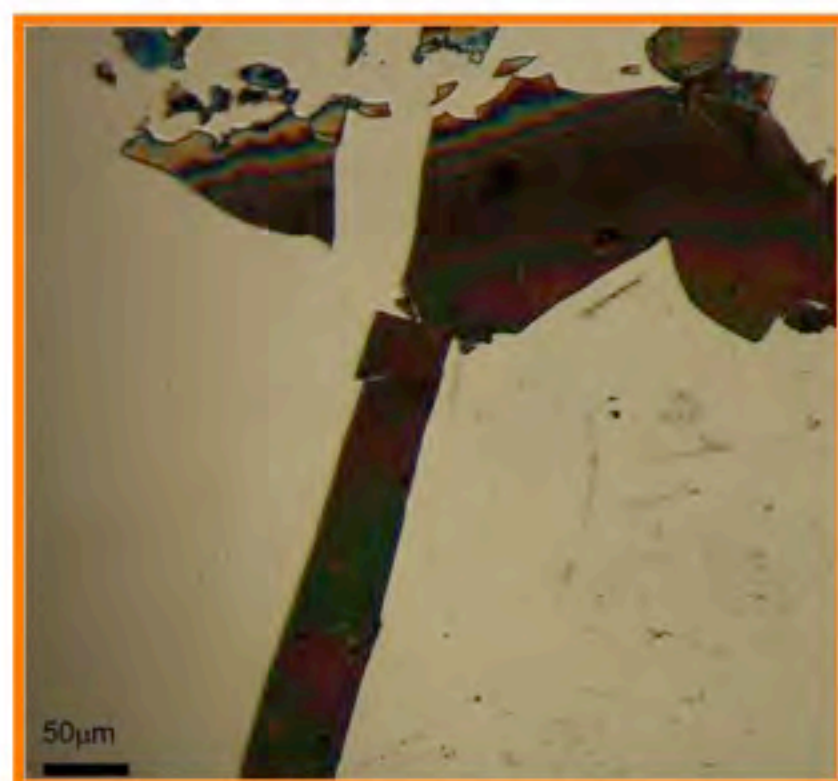
Bias: 200 V



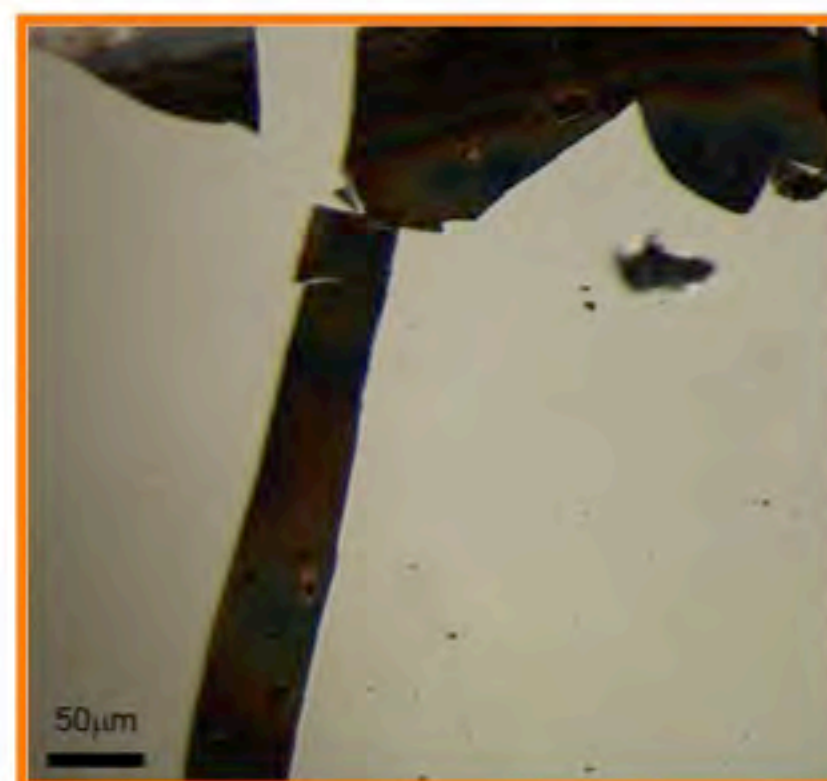
Time of treatment:

5', 20', 40'

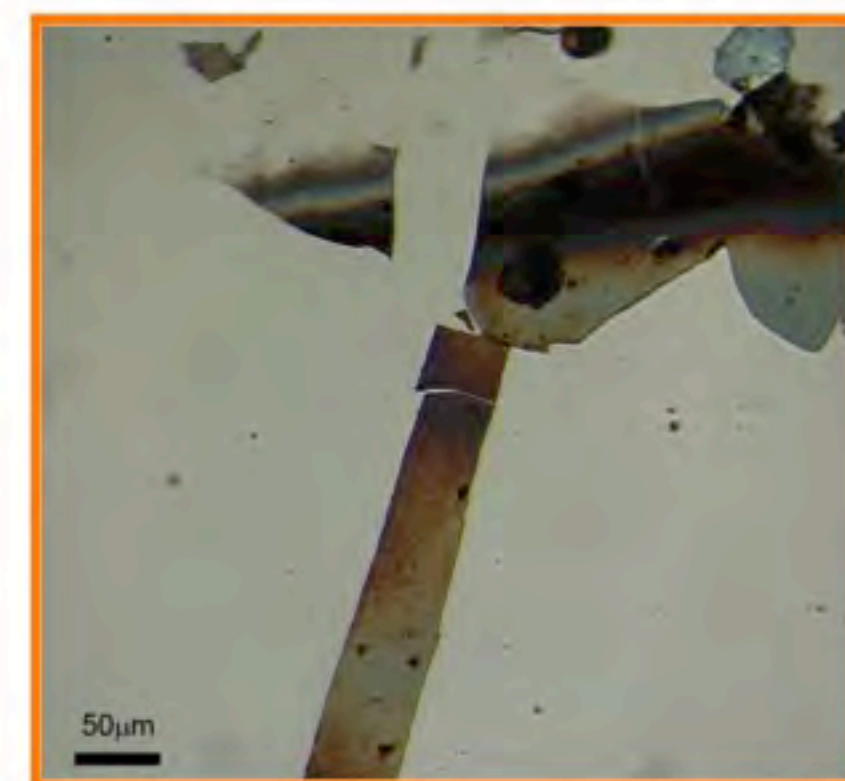
EXFOLIATION



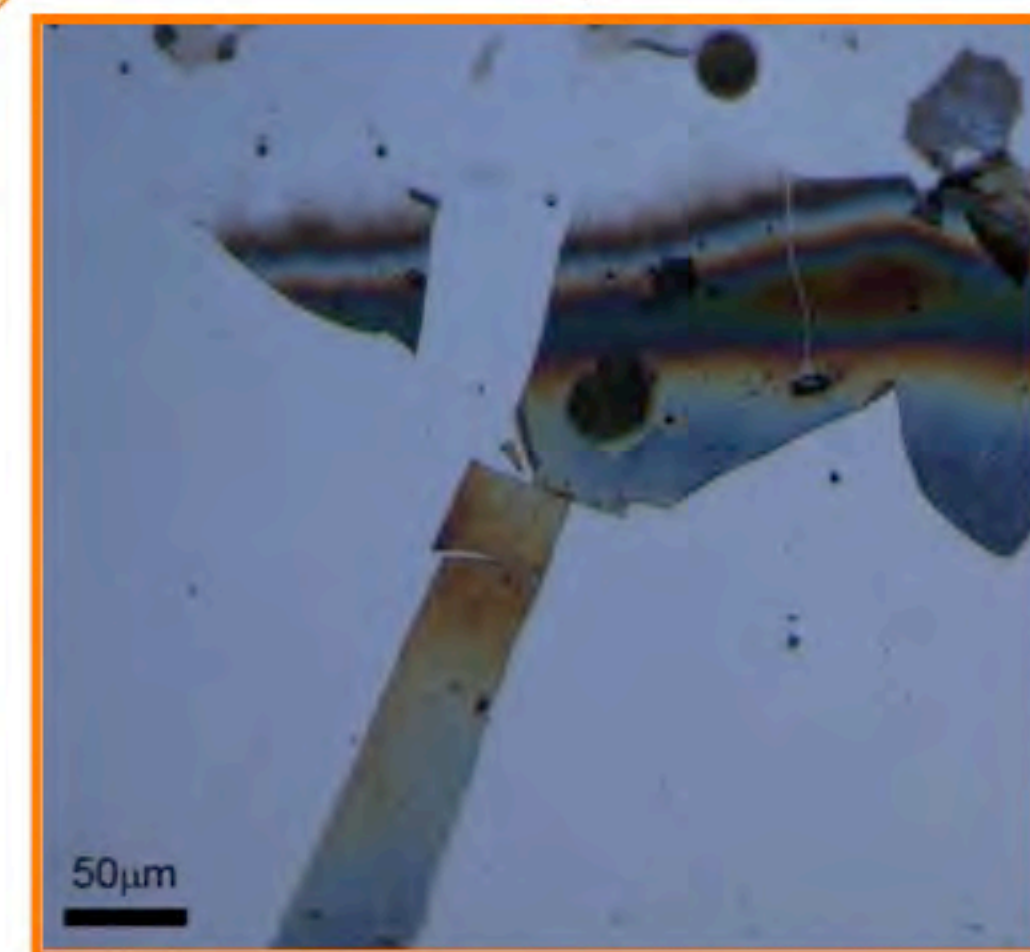
5'



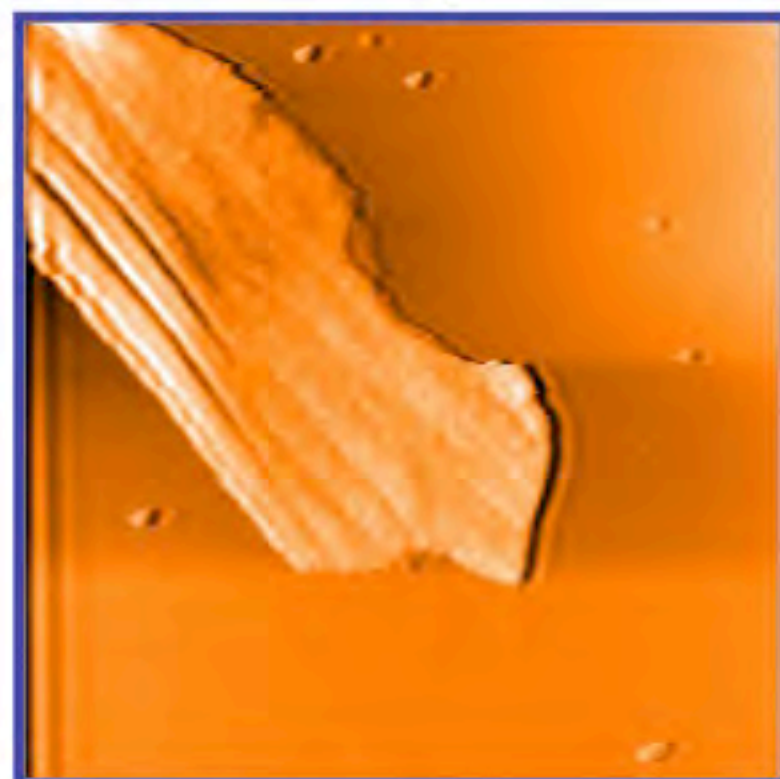
20'



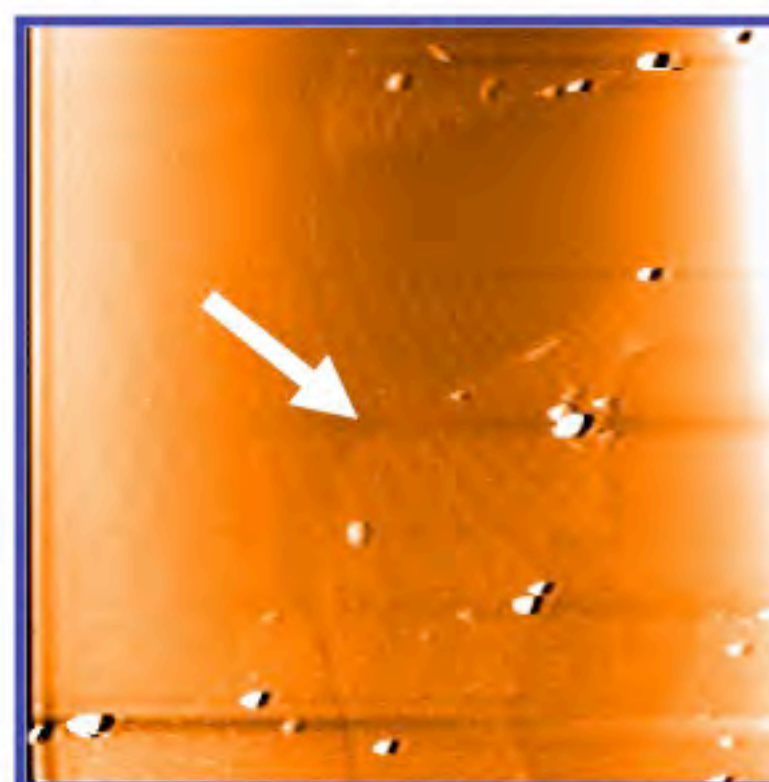
40'



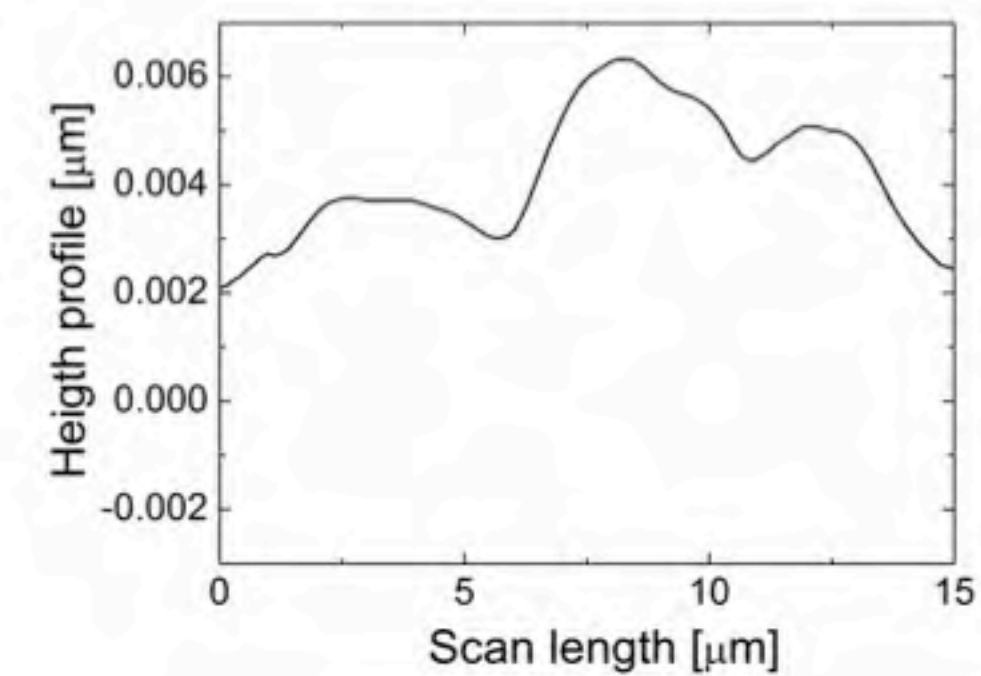
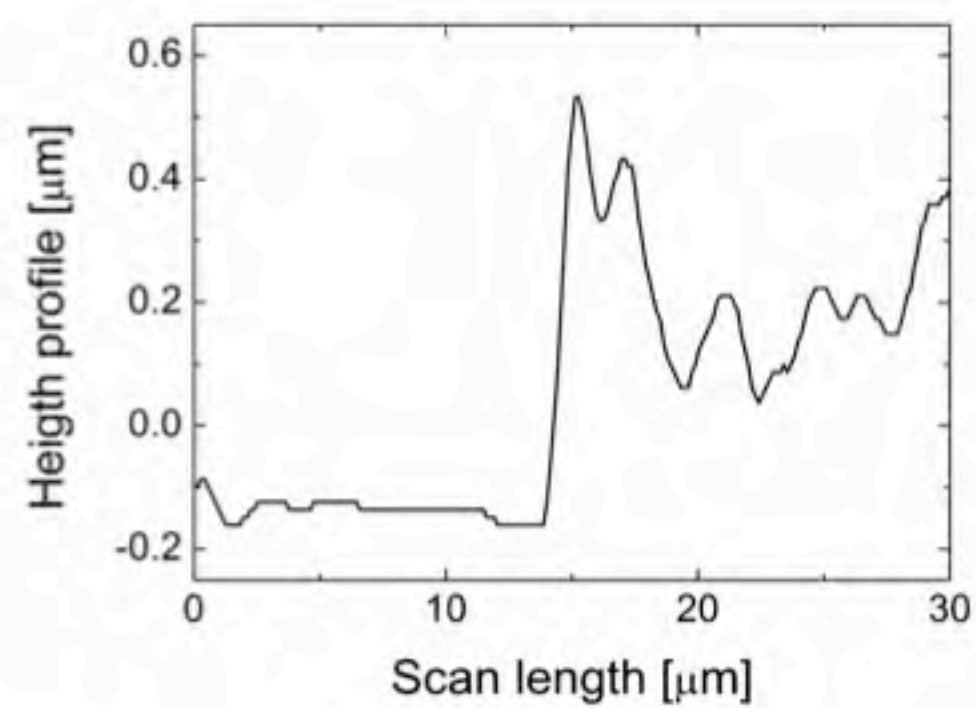
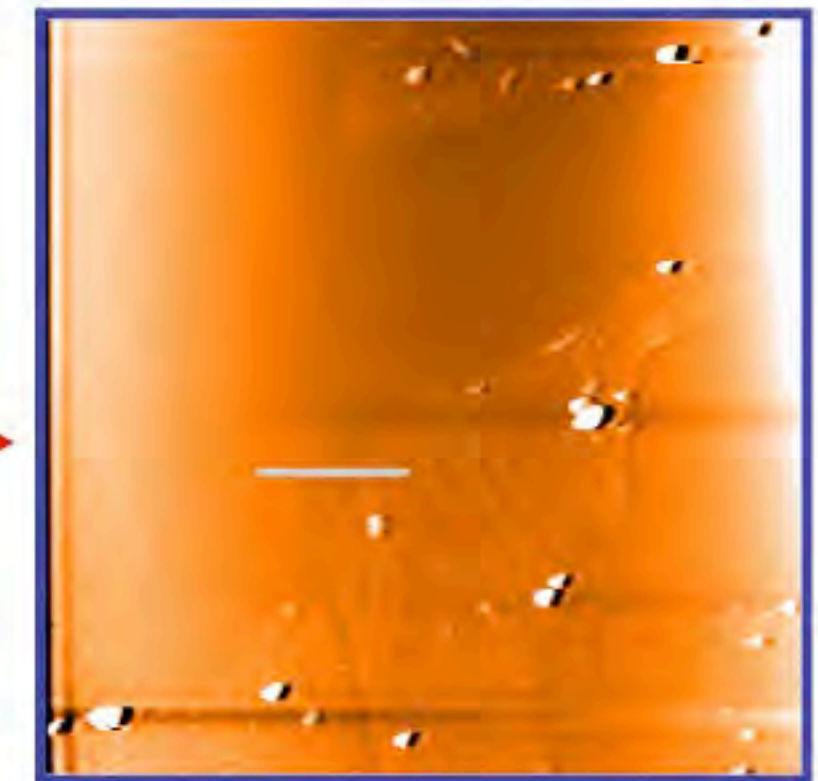
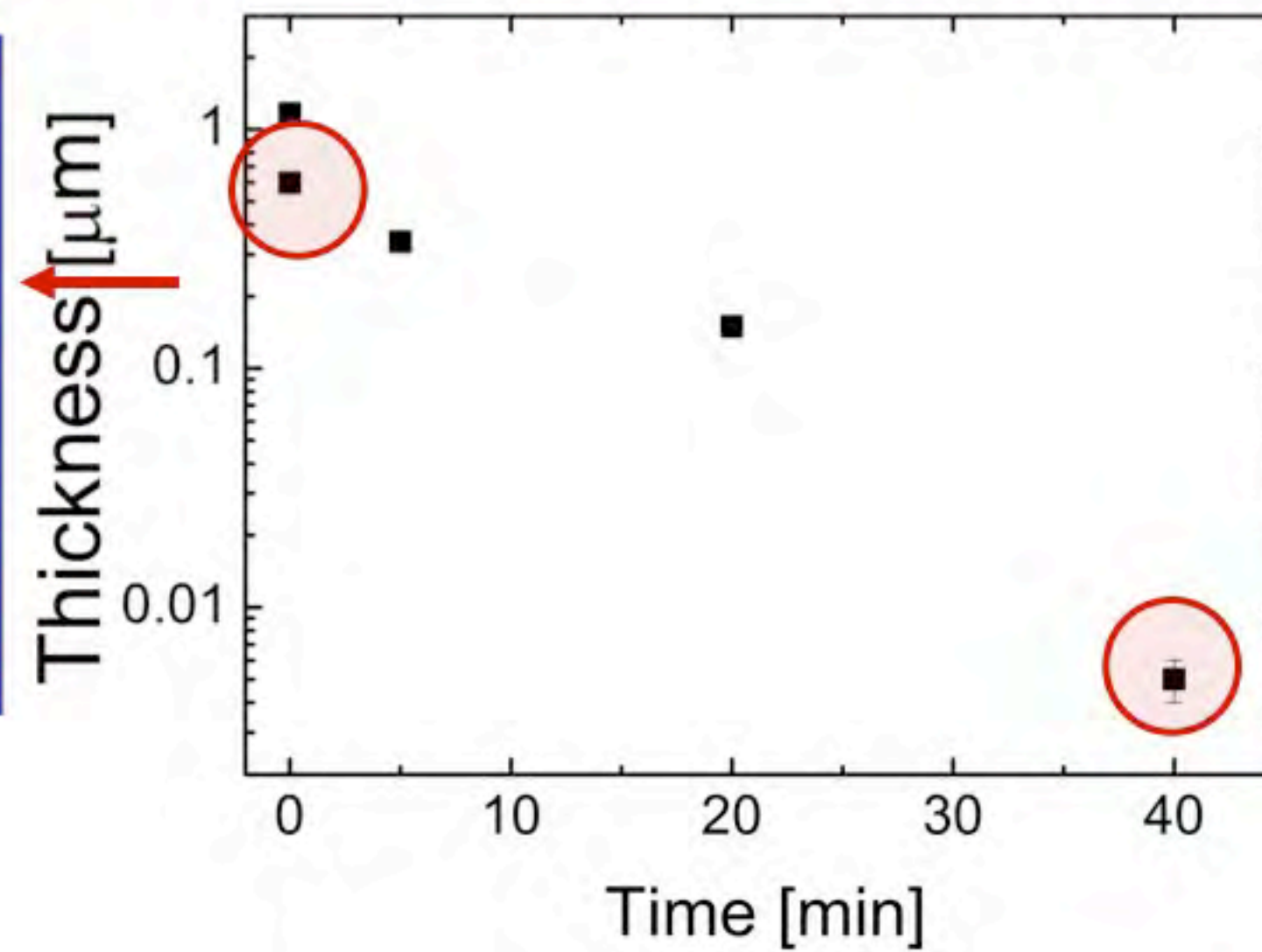
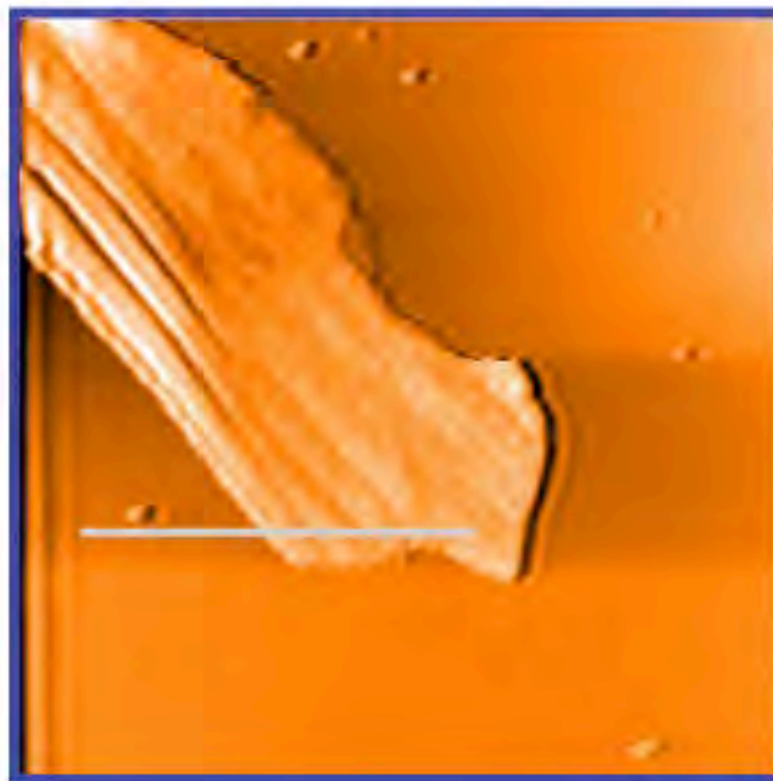
Plasma effects on
morphology and thickness



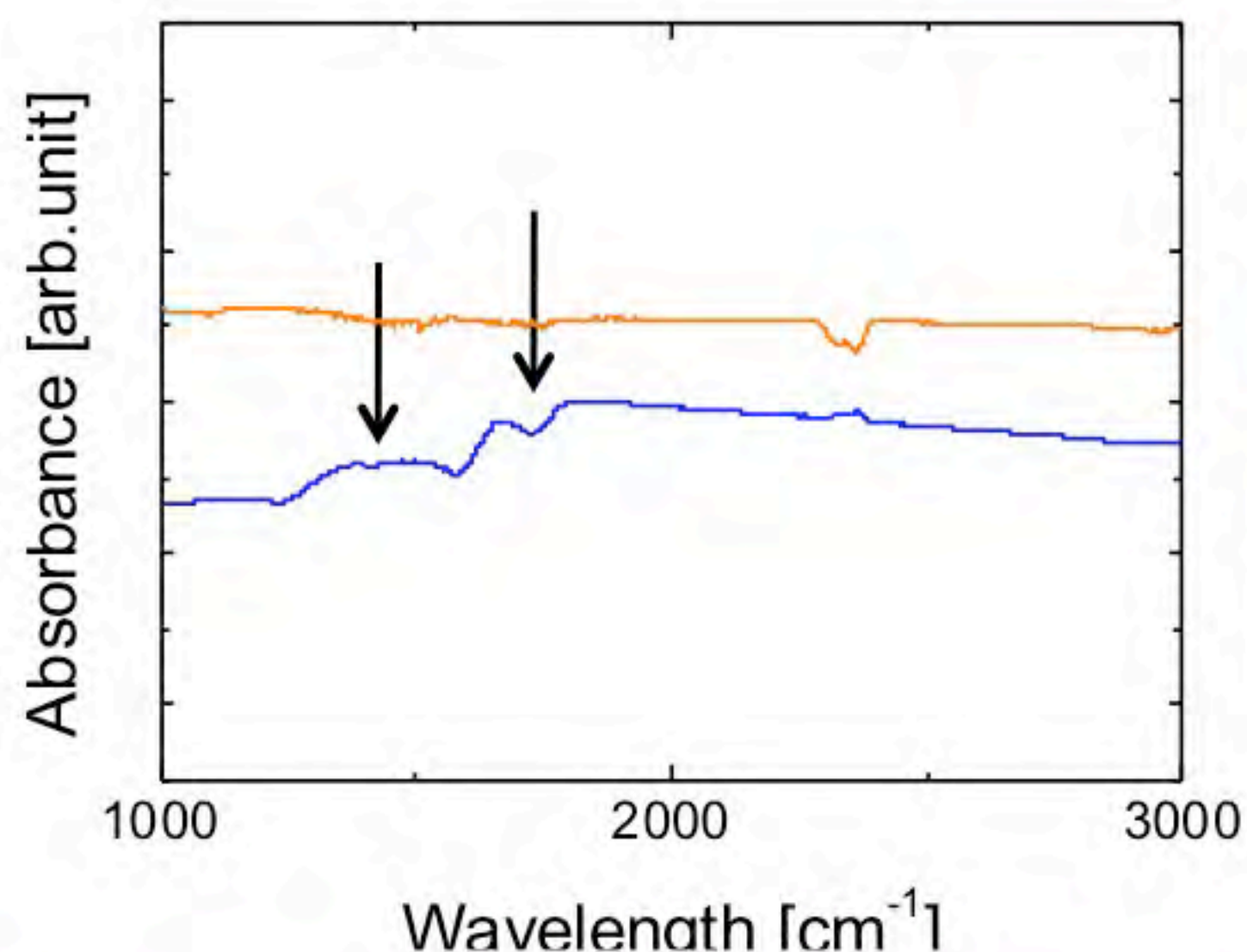
AFM images
(70 μm X 70 μm)



GRADUAL THINNING



REDUCTION



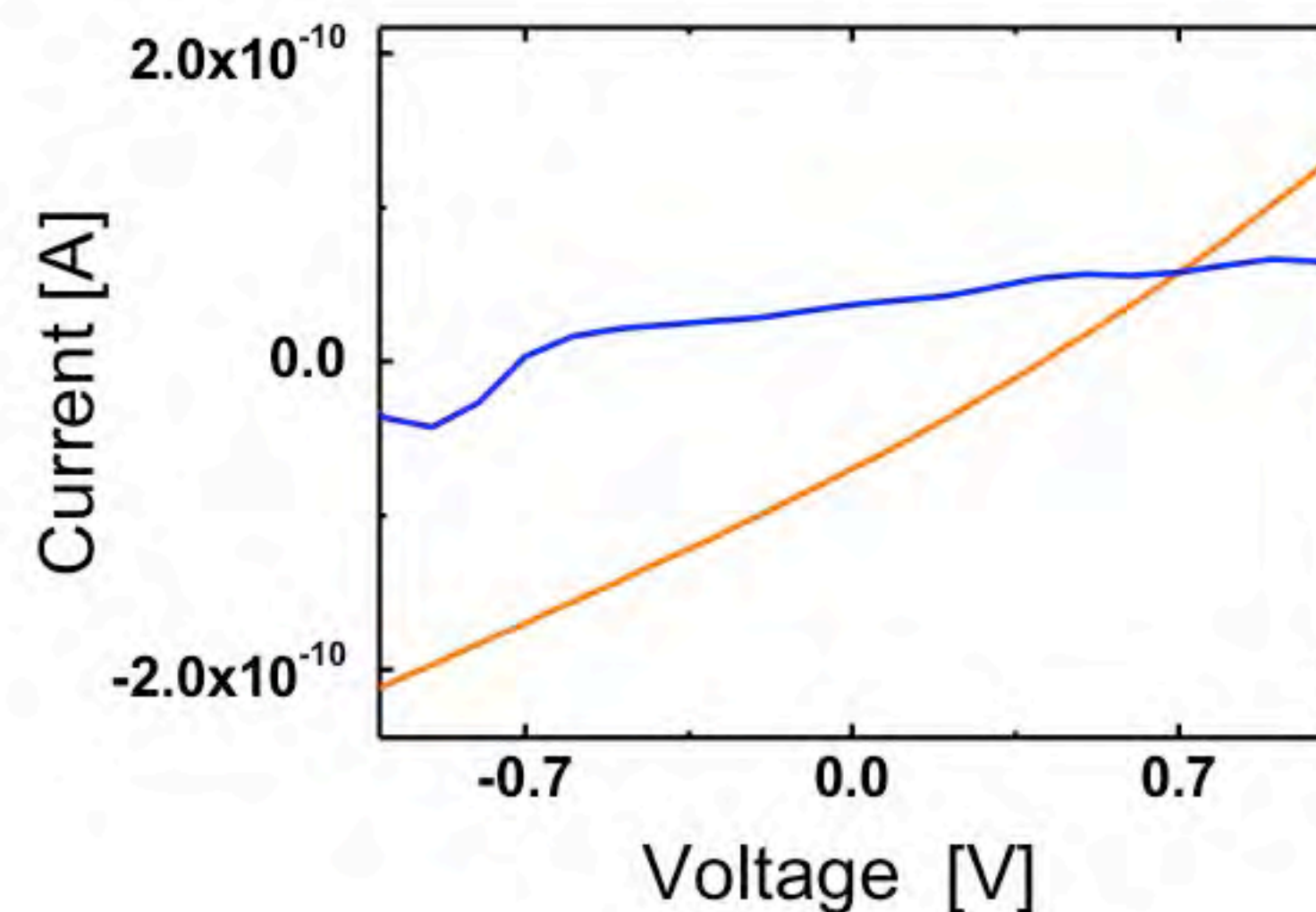
GOPs after 40' of Ar PLASMA

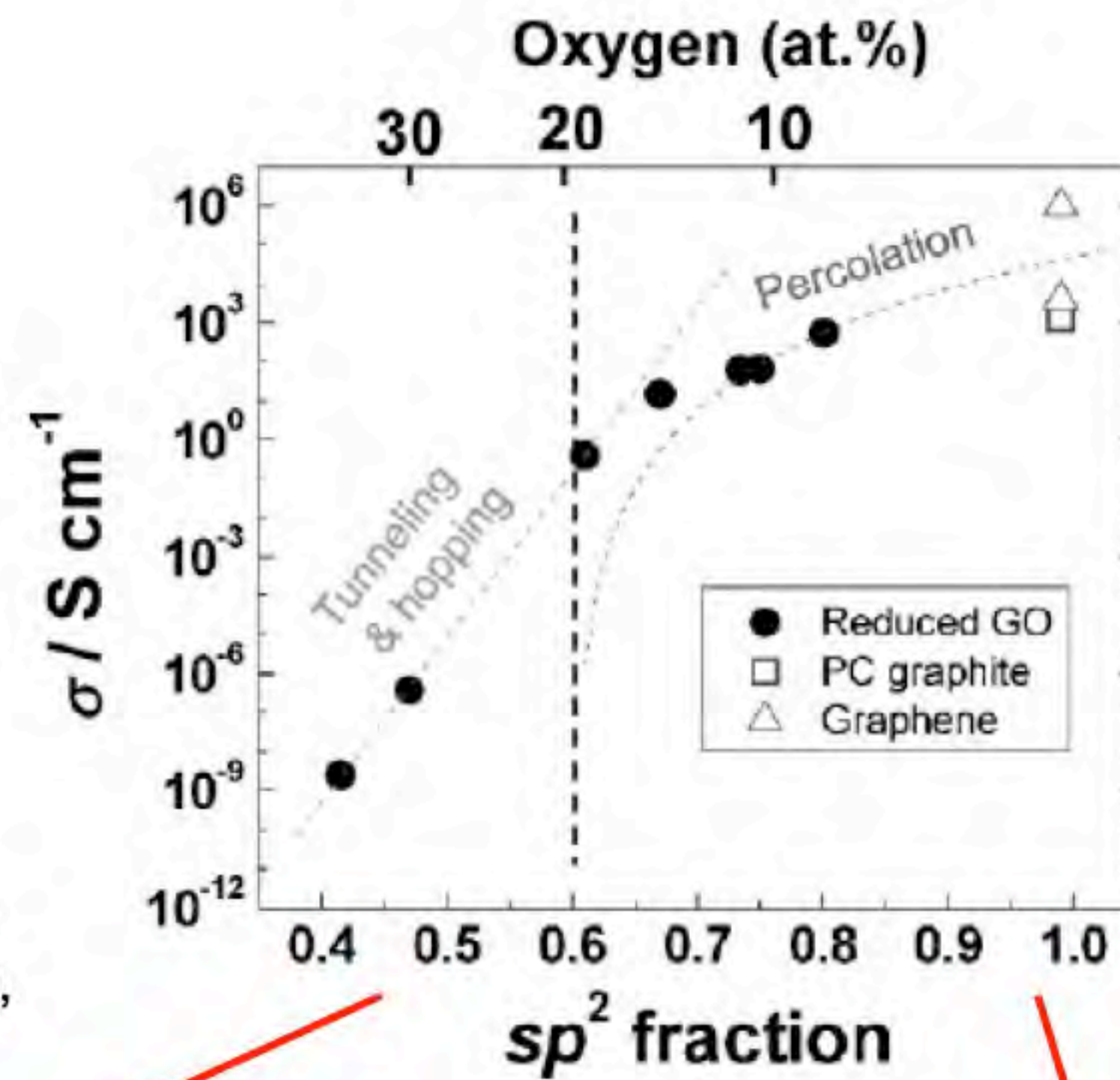
UNTREATED GOPs

1733 cm^{-1} C=O carbonyl stretching

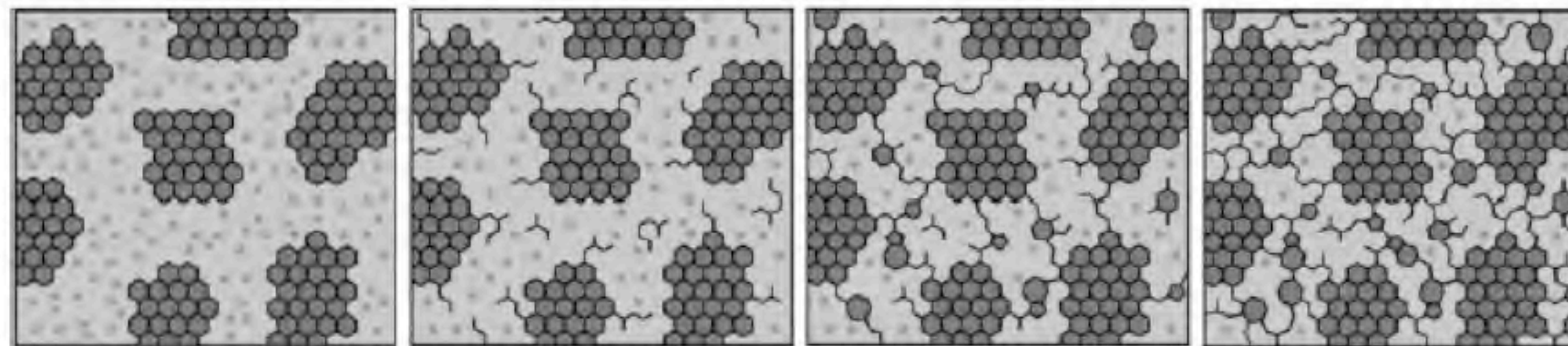
1412 cm^{-1} O-H deformation vibration

**0% from original GOP: 10%
Wt (from Cheaptubes)**

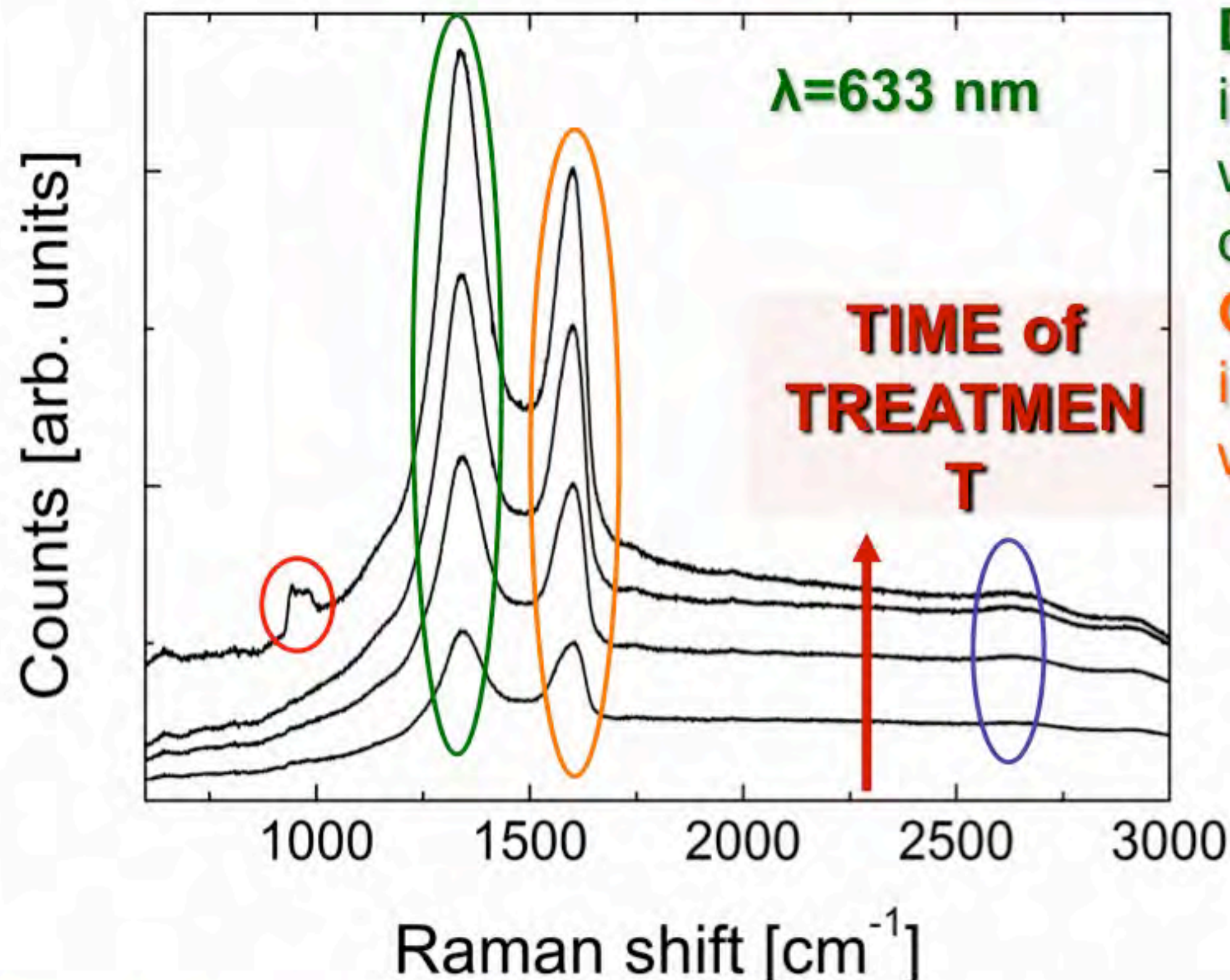




C. Mattevi et al., Adv.
Funct. Mater. 2009, 19,
2577



RAMAN

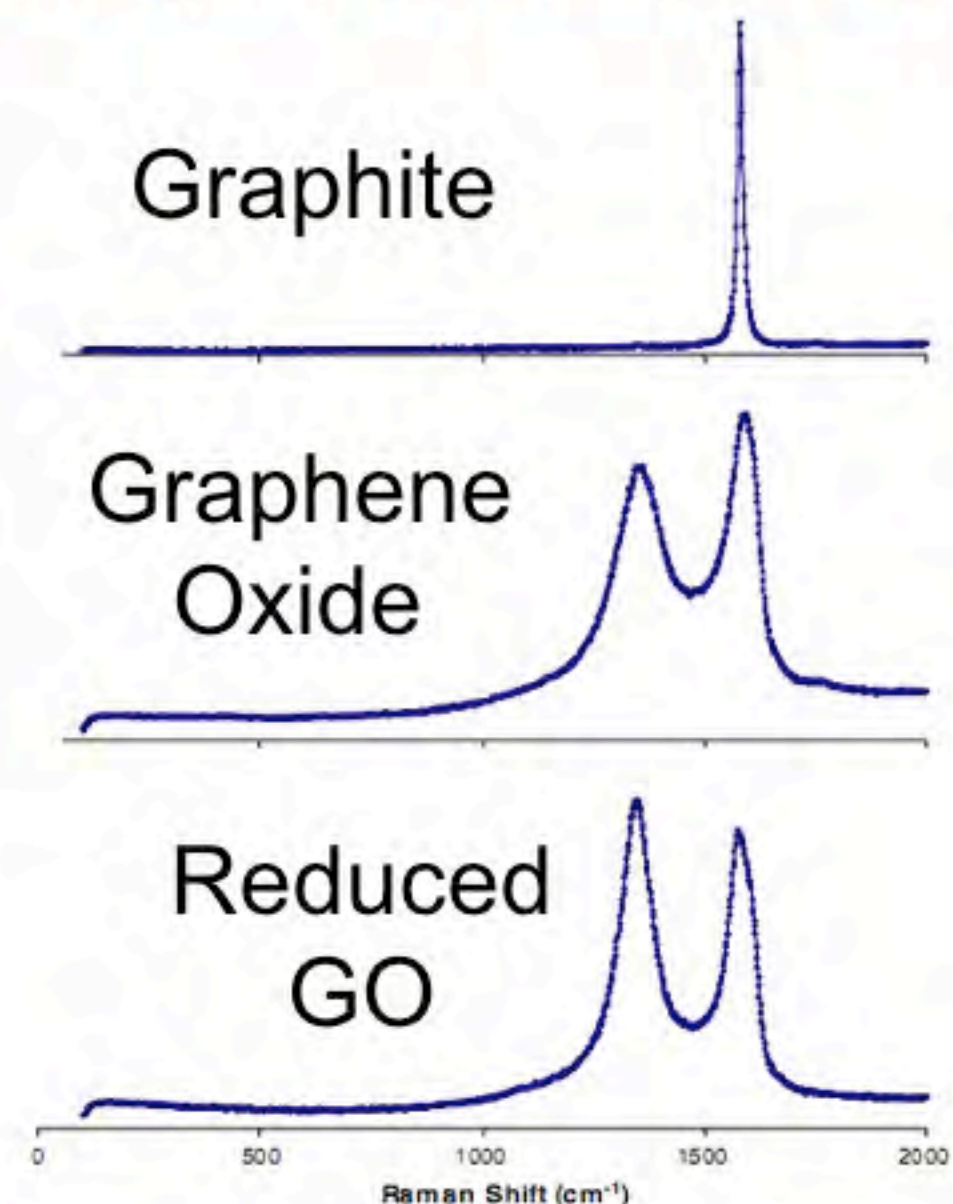


D peak (~1340 cm⁻¹):
increases with disorder (i. e. with the plasma treatment),
decrease of FWHM

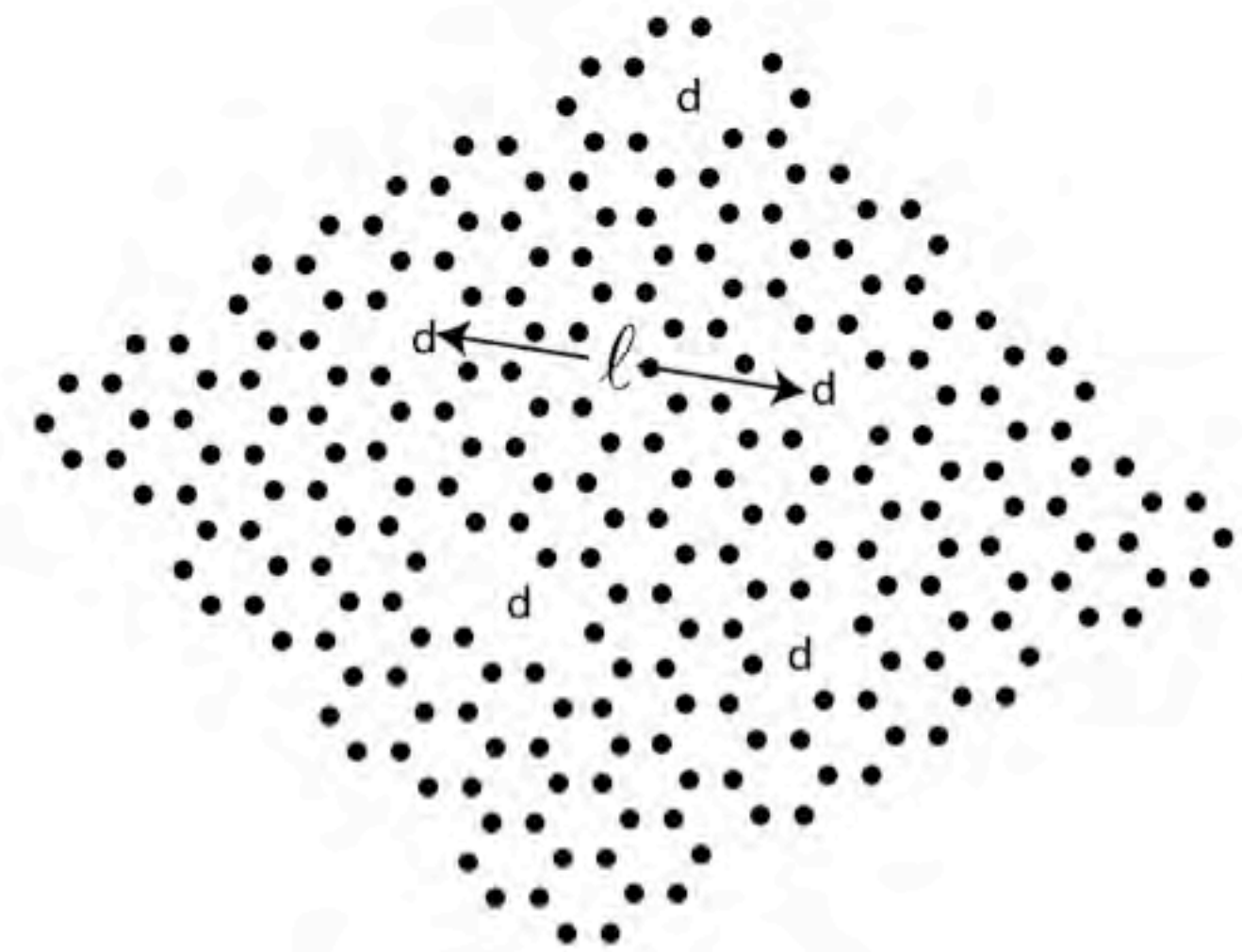
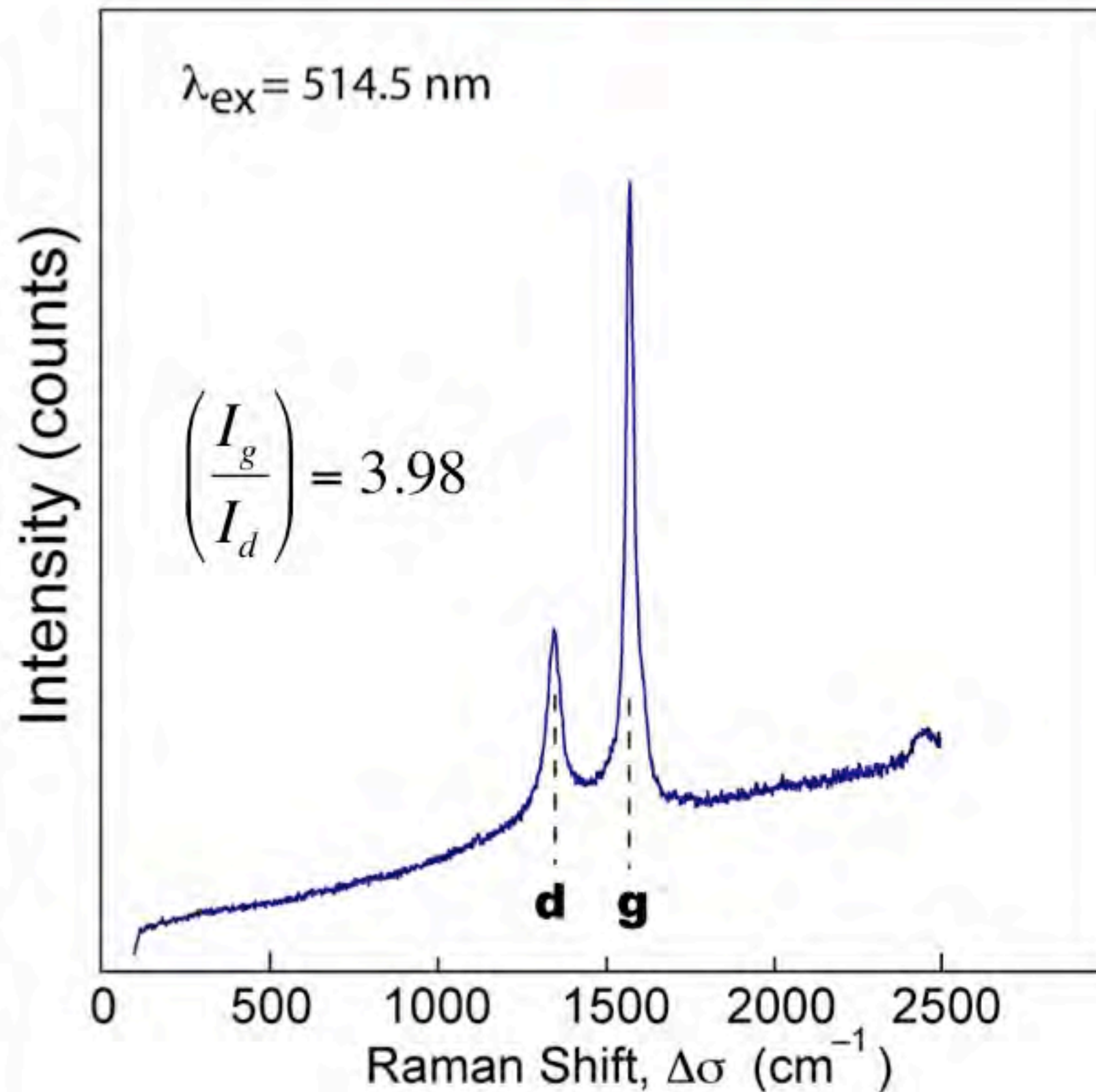
G peak (~1590 cm⁻¹):
increases with disorder (i. e. with the plasma treatment)

2D peak (~2646 cm⁻¹): induced layering and lattice disorder

Si peak (~960 cm⁻¹): thinning



Graphite



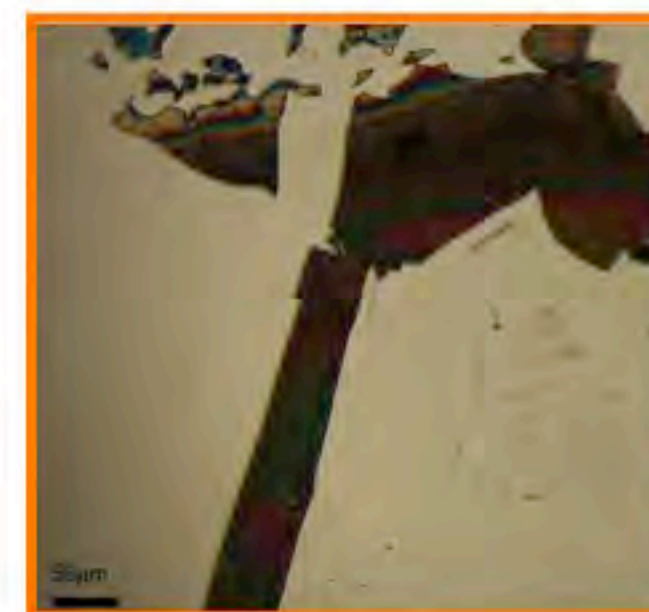
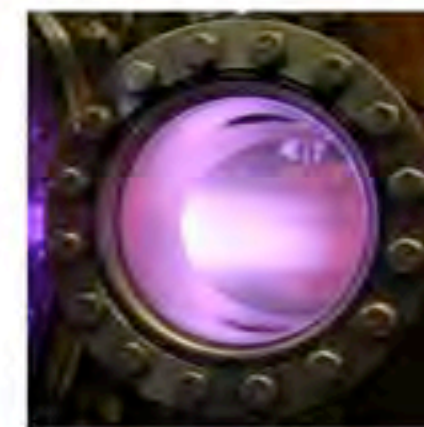
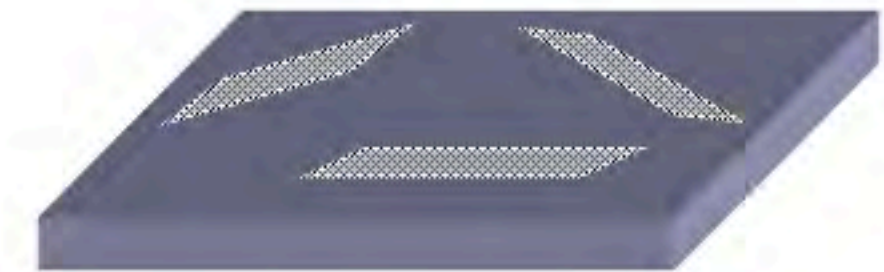
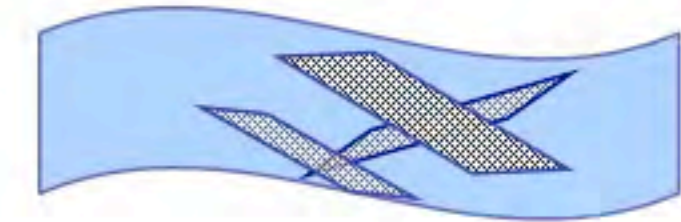
Nanocrystalline graphite has graphitic (**g**) and disorder (**d**) peaks. The characteristic dimension of graphitic domains is given by:

$$\ell(\text{\AA}) = 44 \left(\frac{I_g}{I_d} \right) = 175 \text{\AA}$$

Tuinstra and Koenig, J. Chem Phys. 53 (1970) 1126.

CONCLUSIONS

- Combination of different techniques to produce large GOPs
- Using and tuning of **RF Argon plasma** to exfoliate **GOPs**
- Realization of a procedure to **thin** GOPs



ACKNOWLEDGEMENTS

- **Dr. Paola Fabbri** (Dep. of Material and Environmental Engineering, University of Modena and Reggio Emilia; Raman facility)
- **Dr. Fabio Bergamini** (CIGS-Centro Interdipartimentale Grandi Strumenti, University of Modena and Reggio Emilia; Raman facility)
- **Dr. Pierluigi Rondoni** (Centro Universitario di Microscopia Elettronica, University of Perugia; TEM analysis)

AND YOU FOR THE ATTENTION!!!