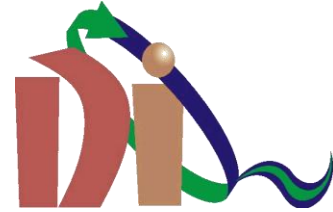


# CVD-GRAPHENE SYNTHESIS USING DIFFERENT TRANSITION METALS AS CATALYST

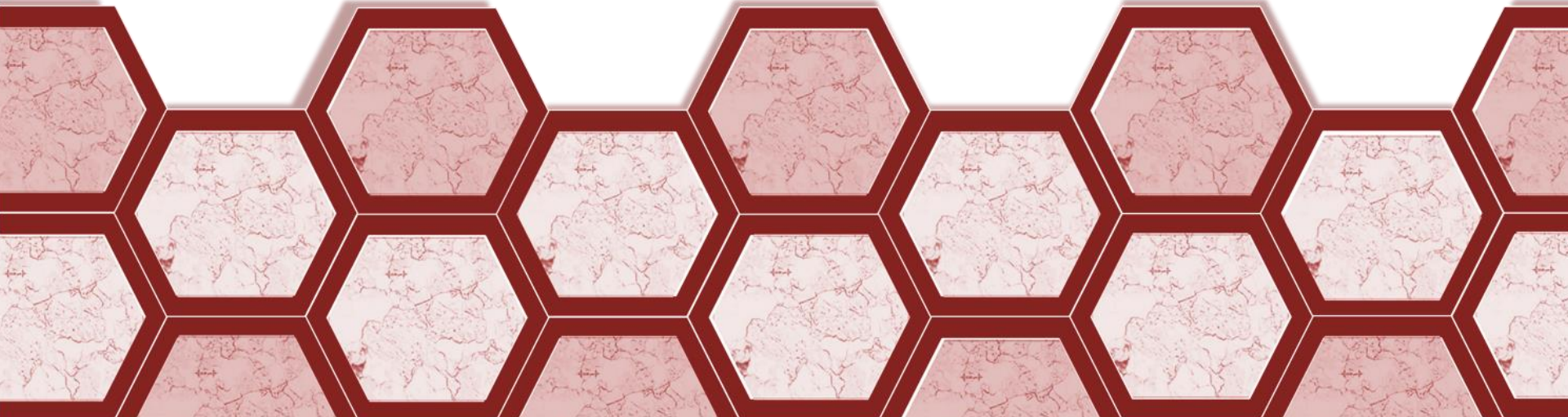


**M<sup>a</sup> del Prado Lavín López**

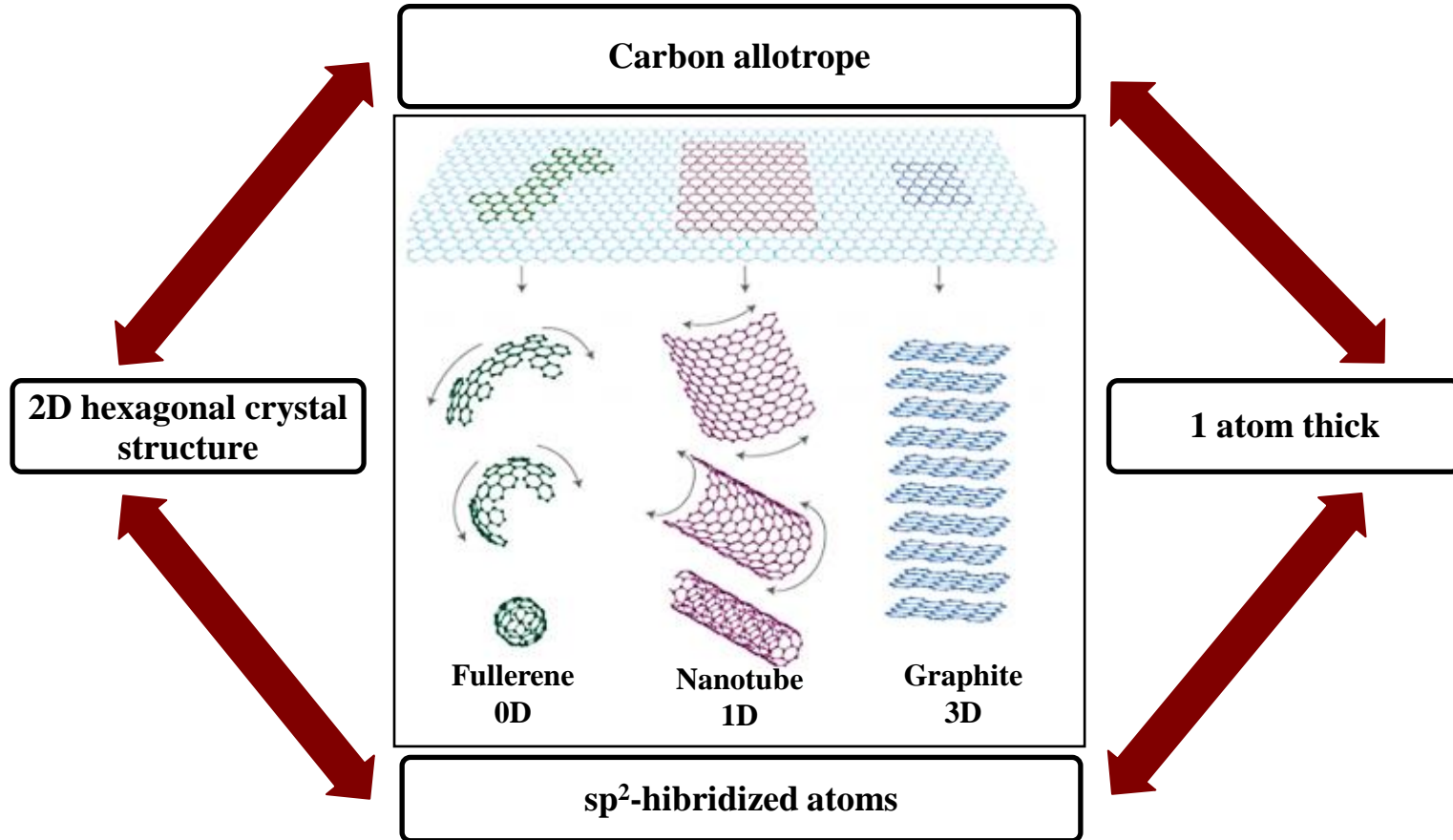
**Graphene  
Canada**

**October 14-16, 2015  
Montreal, Canada**

Graphene & 2D Materials International Conference and Exhibition

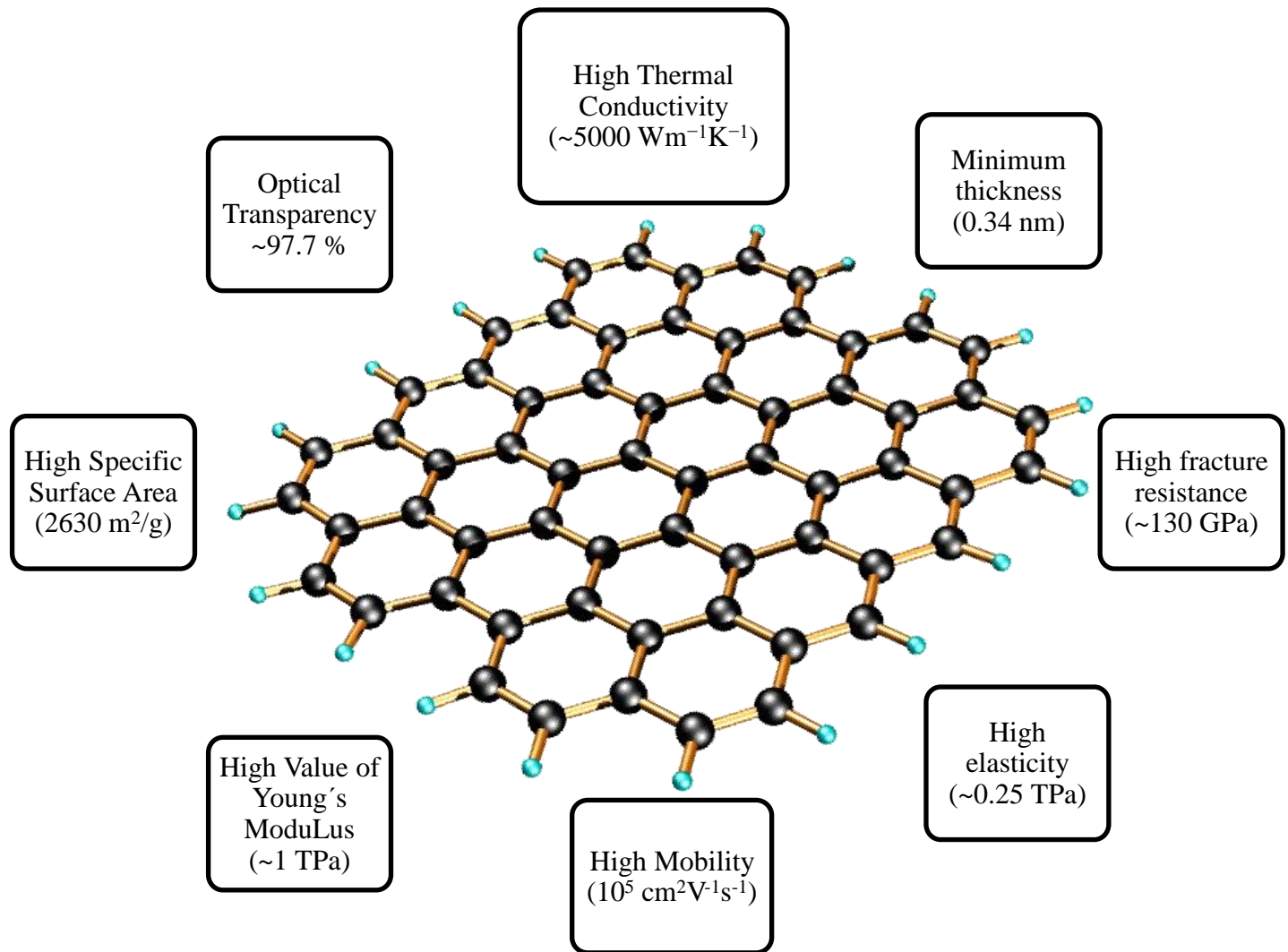


# INTRODUCTION



Geim, A.K. and K.S. Novoselov, *The rise of graphene*.  
Nature Materials, 2007. 6(3): p. 183-191.

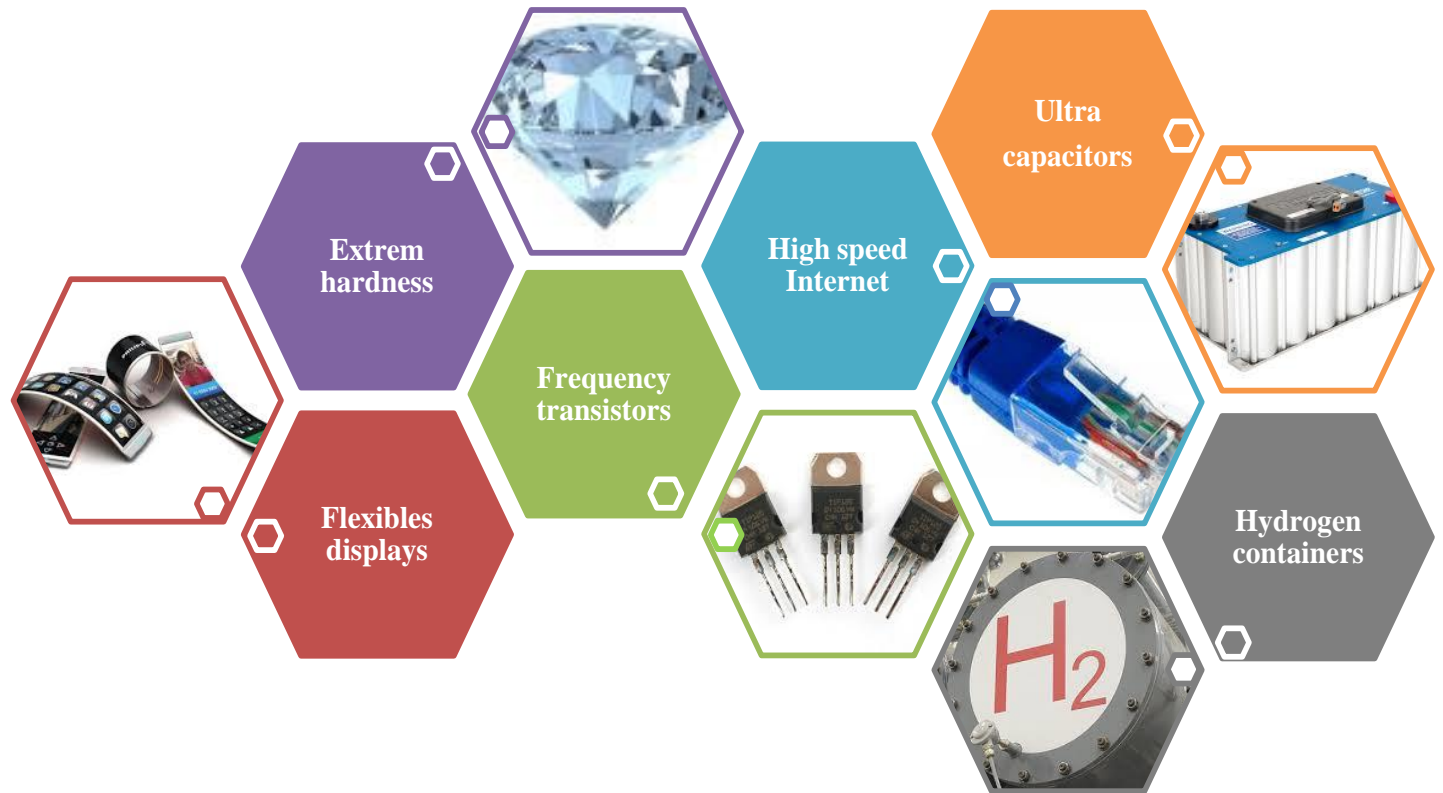
# INTRODUCTION



Zhu, Y., et al., *Graphene and graphene oxide: Synthesis, properties, and applications*. Advanced Materials, 2010. **22**(35): p. 3906-3924.

Dong, L.-X. and Q. Chen (2010). *Properties, synthesis, and characterization of graphene*. Frontiers of Materials Science in China **4**(1): 45-51.

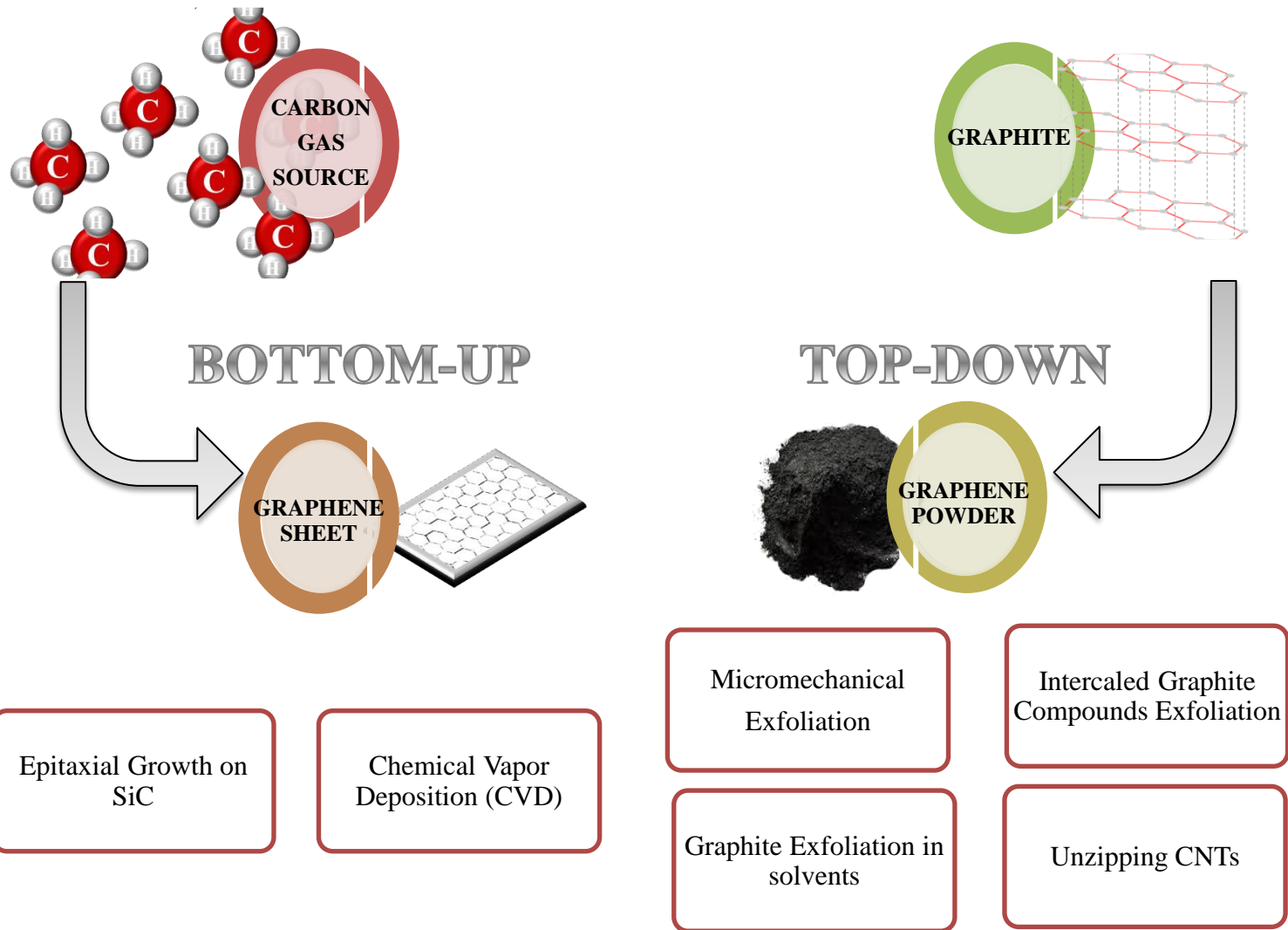
# INTRODUCTION



Zhu, Y., et al., *Graphene and graphene oxide: Synthesis, properties, and applications*. *Advanced Materials*, 2010. **22**(35): p. 3906-3924.

Dong, L.-X. and Q. Chen (2010). *Properties, synthesis, and characterization of graphene*. *Frontiers of Materials Science in China* **4**(1): 45-51.

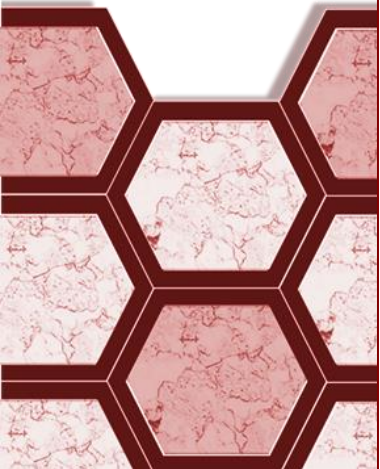
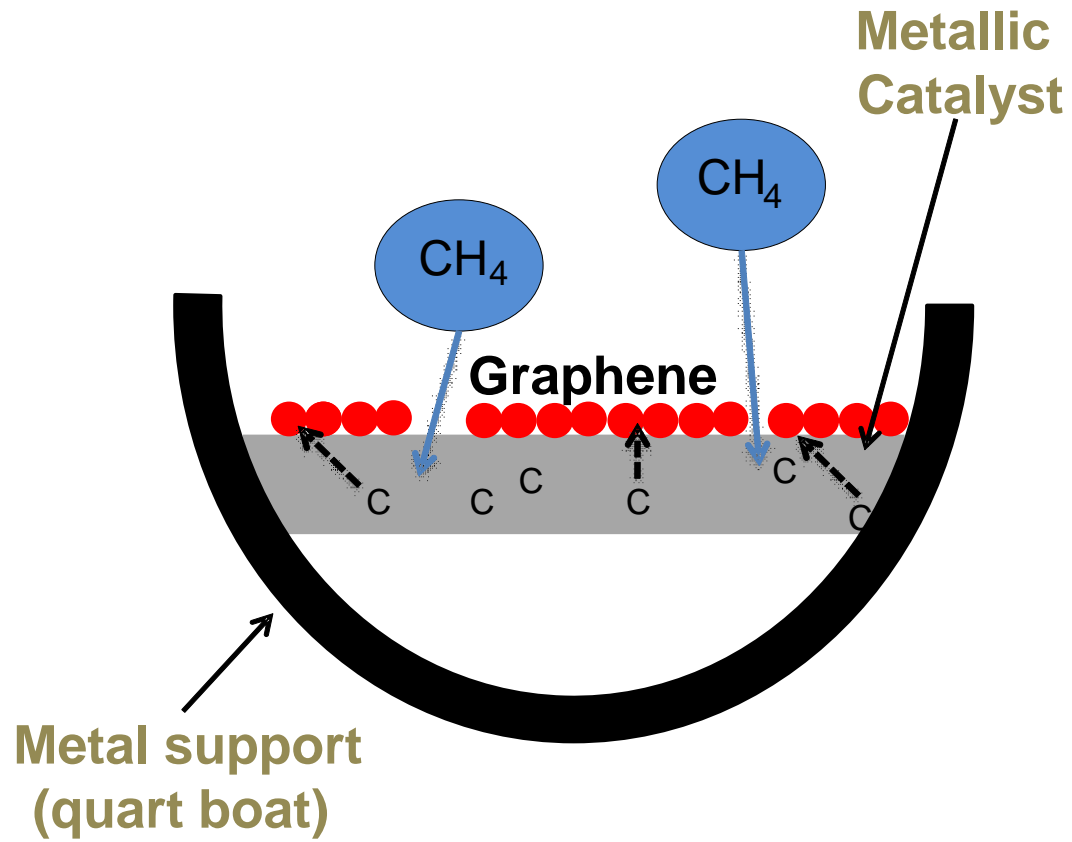
# INTRODUCTION



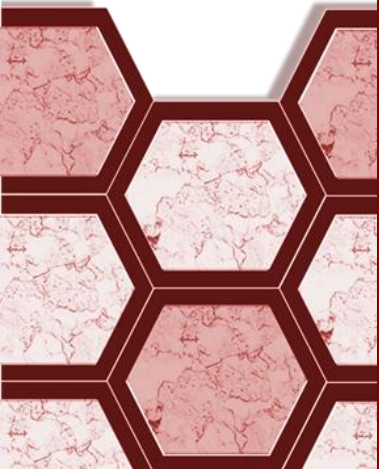
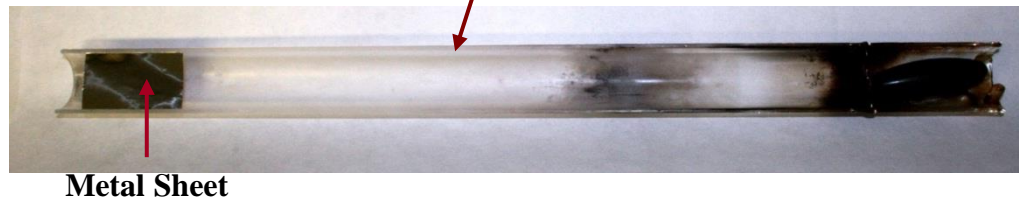
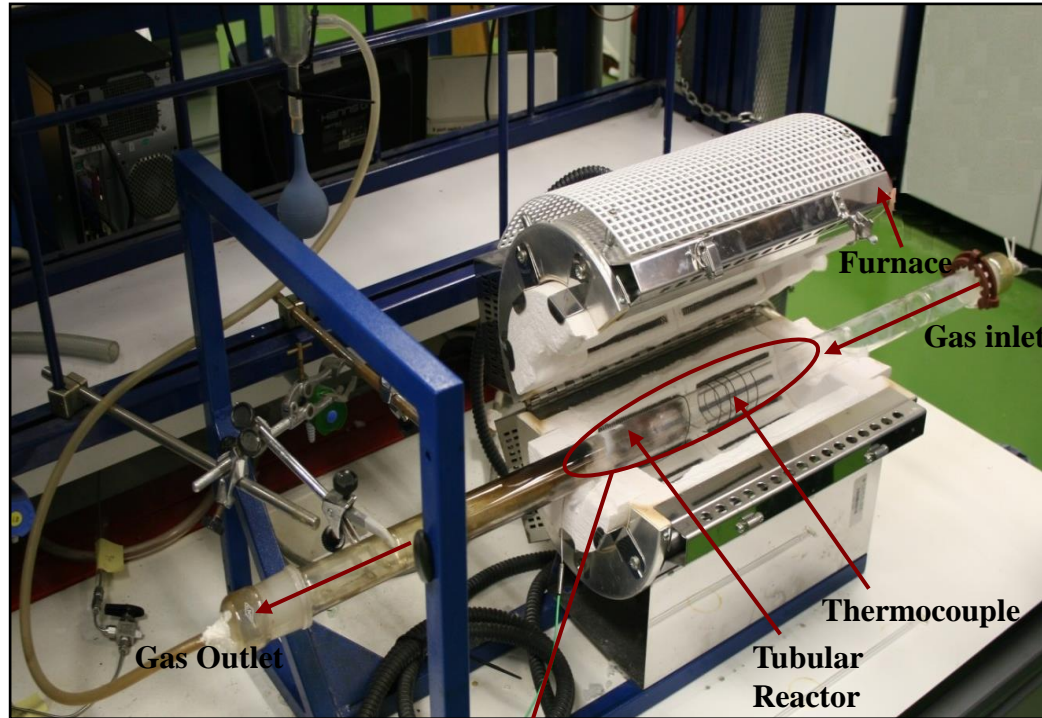
# INTRODUCTION

## Chemical Vapor Deposition (CVD)

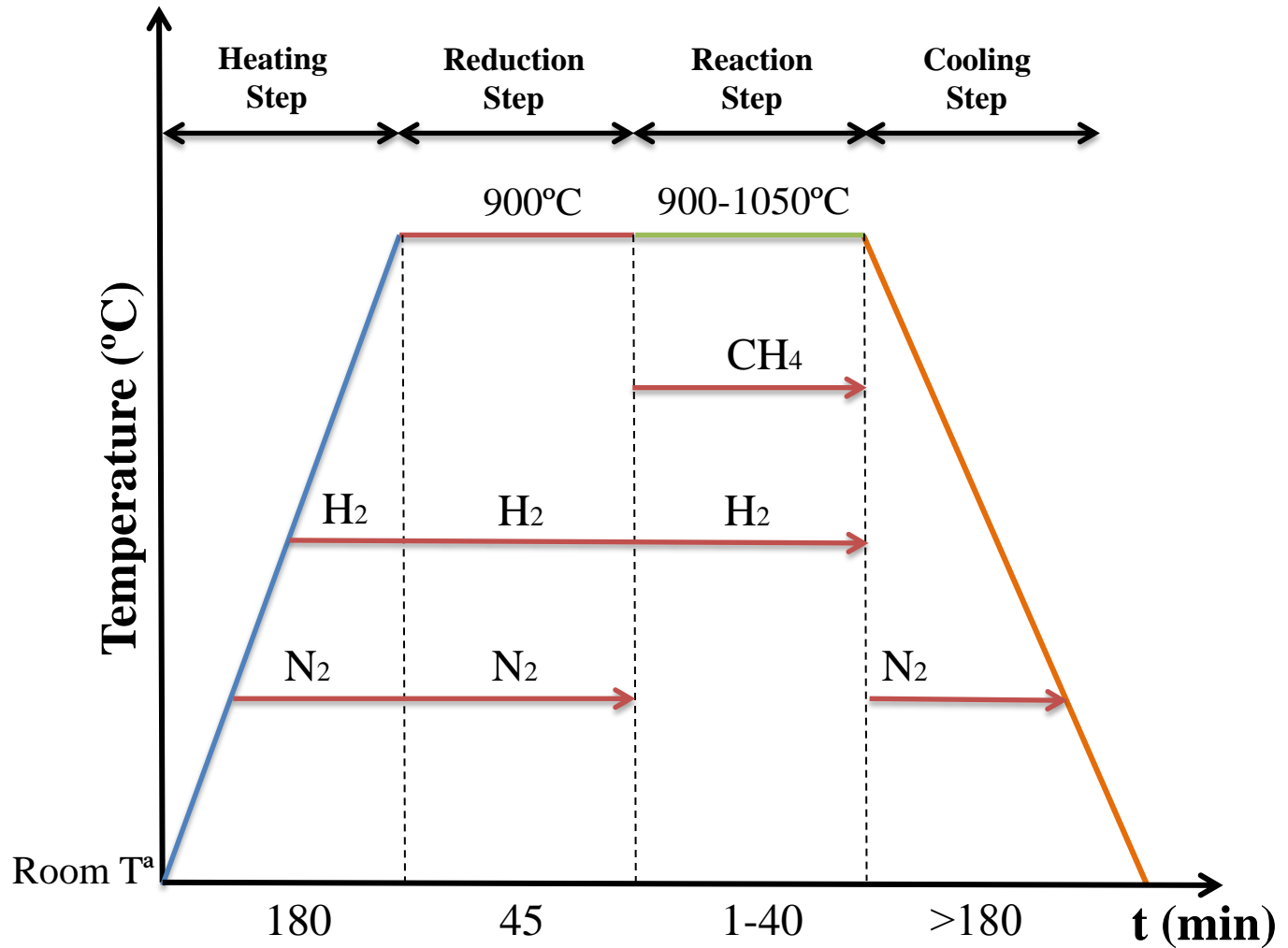
Chen, Z., et al., *Bulk growth of mono- to few-layer graphene on nickel particles by chemical vapor deposition from methane*. Carbon, 2010. **48**(12): p. 3543-3550.



# EXPERIMENTAL



# EXPERIMENTAL





# EXPERIMENTAL

CHARACTERIZATION  
TECHNIQUES

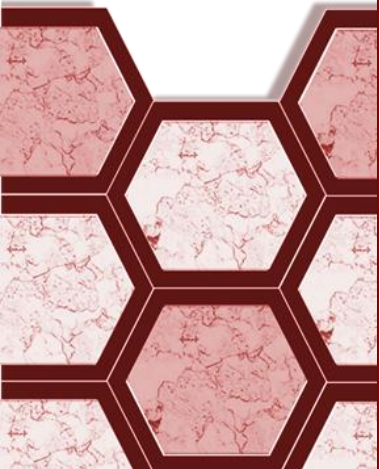
```
graph TD; A[CHARACTERIZATION TECHNIQUES] --> B[OPTICAL MICROSCOPY]; A --> C[RAMAN SPECTROSCOPY]; D[EXCEL-VBA APPLICATION]
```

The diagram is a flowchart with a hierarchical structure. At the top level is a rounded rectangular box containing the text 'CHARACTERIZATION TECHNIQUES'. A vertical line descends from the bottom center of this box and connects to a horizontal line. From this horizontal line, two vertical lines extend downwards to the top centers of two separate rounded rectangular boxes: 'OPTICAL MICROSCOPY' on the left and 'RAMAN SPECTROSCOPY' on the right. Below these two boxes, centered horizontally, is a third rounded rectangular box containing the text 'EXCEL-VBA APPLICATION'. All boxes have a dark red border and white background. The text is in a bold, black, sans-serif font.

OPTICAL  
MICROSCOPY

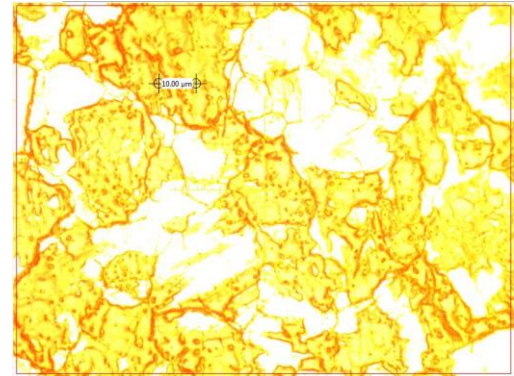
RAMAN  
SPECTROSCOPY

EXCEL-VBA  
APPLICATION

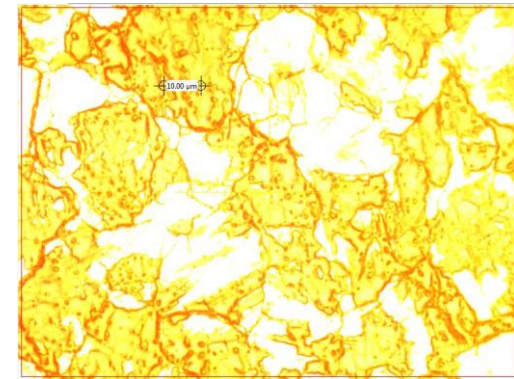


# EXPERIMENTAL

OPTICAL  
MICROSCOPY



EXCEL-VBA  
APPLICATION

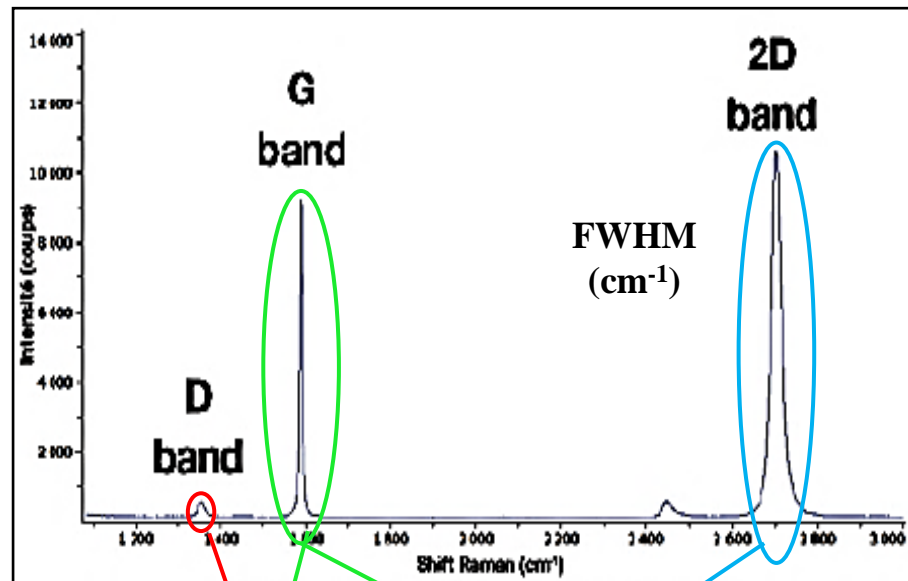


**QUALITY: 1 – 1000**

# EXPERIMENTAL

## RAMAN SPECTROSCOPY

Lavin-Lopez, M.P., et al., *Synthesis and characterization of graphene: Influence of synthesis variables*. Physical Chemistry Chemical Physics, 2014. **16**(7): p. 2962-2970.



Ferrari, A. C., J. C. Meyer, y col. (2006). *Raman Spectrum of Graphene and Graphene Layers*. Physical Review Letters **97**(18): 187401.

# RESULTS AND DISCUSSION

Reaction  
Temperature

• 900 - 1050°C

Reaction  
Time

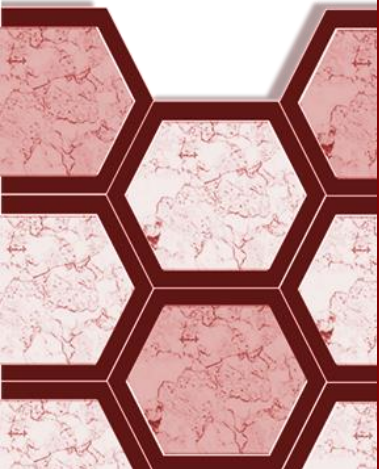
• 1 - 40 min

CH<sub>4</sub>/H<sub>2</sub>  
Flow Rate  
Ratio

• 0.07 - 0.4 v/v

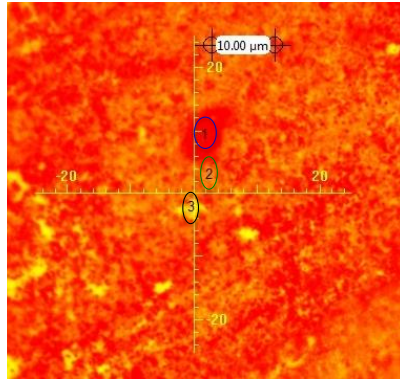
Q<sub>T</sub> during  
reaction step

• 60 - 130 Nml/min



# RESULTS AND DISCUSSION

## Influence of the reaction $T^a$ at different reaction times



Lavin-Lopez, M.P., et al., *Synthesis and characterization of graphene: Influence of synthesis variables*. Physical Chemistry Chemical Physics, 2014. **16**(7): p. 2962-2970.

## COPPER

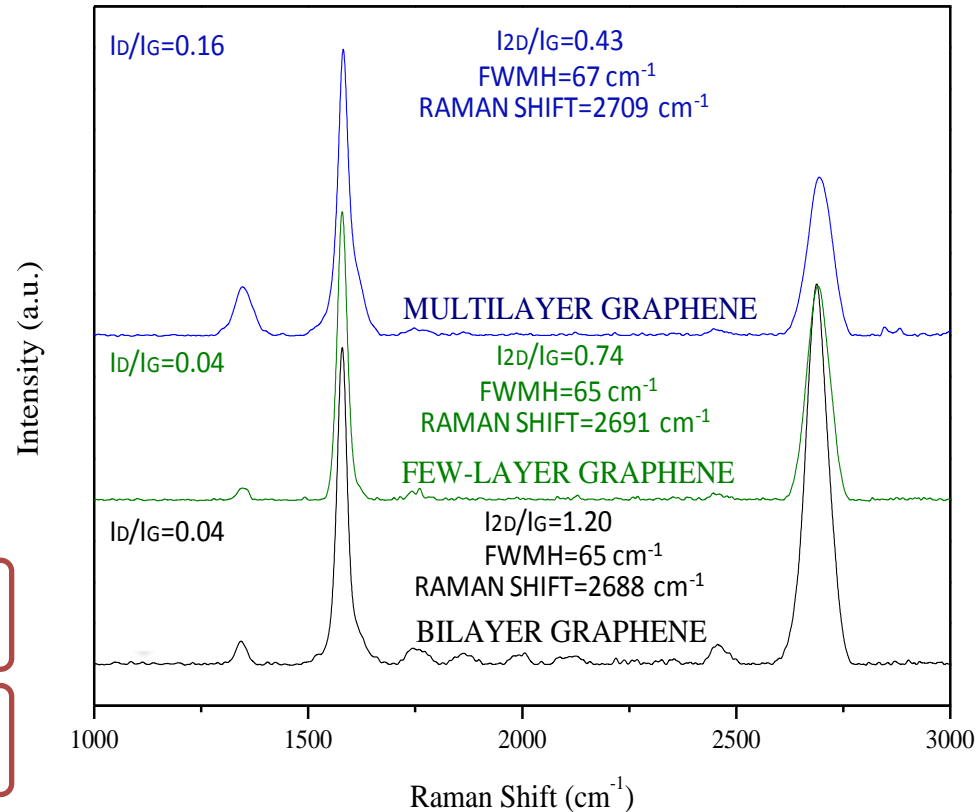
OPTIMUM  
CONDITIONS

$T^a=1050^\circ\text{C}$

Time=10 min

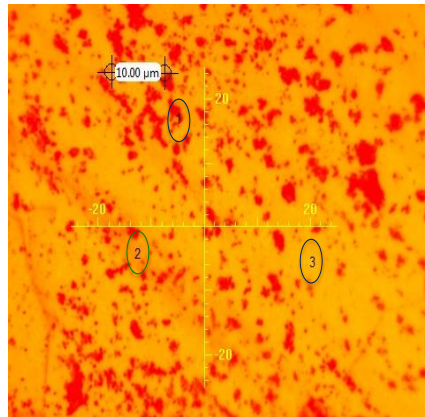
$T^a_{\text{reaction}}=1050^\circ\text{C}$ ,  $\text{CH}_4/\text{H}_2=0.3$  v/v,  $\text{time}_{\text{reaction}}=10$  min,  $Q_T=130$  Nml/min

% MULTILAYER GRAPHENE	% FEW-LAYERS GRAPHENE	% BILAYER GRAPHENE	QUALITY
81.34	17.00	1.66	4.2



# RESULTS AND DISCUSSION

## Influence of the CH<sub>4</sub>/H<sub>2</sub> Flow Rate Ratio



Lavin-Lopez, M.P., et al., *Synthesis and characterization of graphene: Influence of synthesis variables*. Physical Chemistry Chemical Physics, 2014. 16(7): p. 2962-2970.

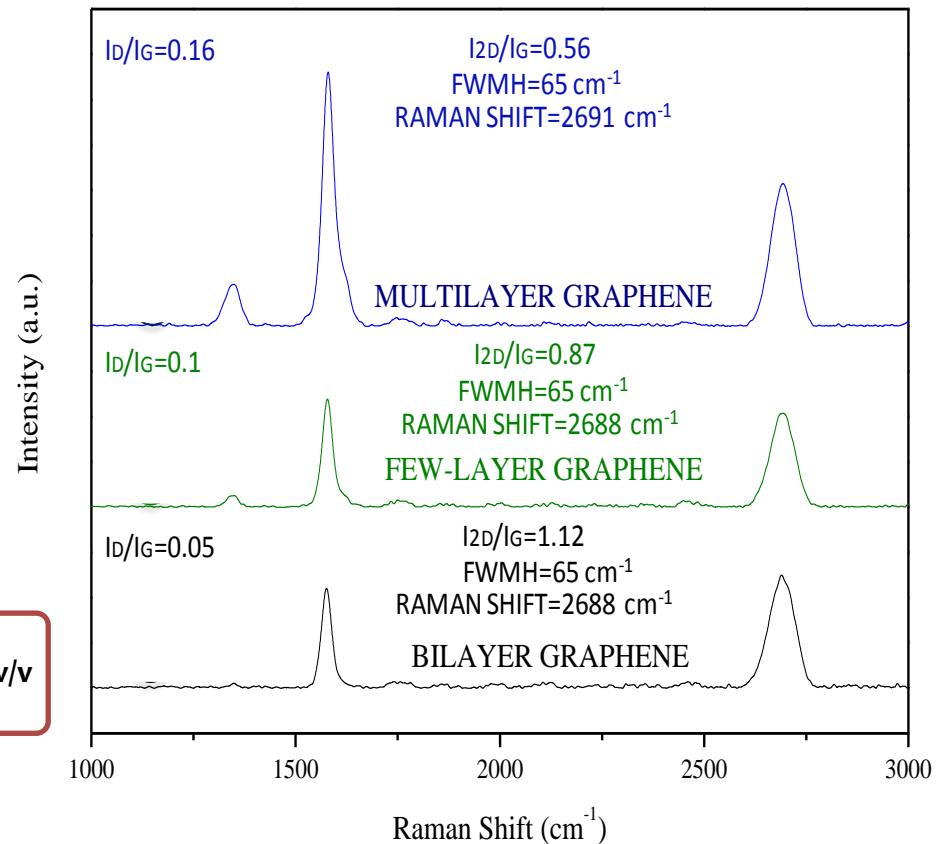
# COPPER

OPTIMUM  
CONDITIONS

CH<sub>4</sub>/H<sub>2</sub>=0,07 v/v

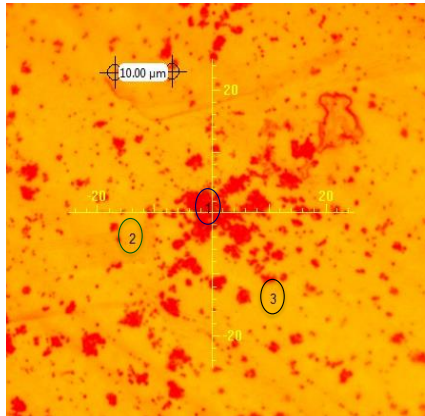
T<sup>a</sup><sub>reaction</sub>=1050°C, CH<sub>4</sub>/H<sub>2</sub>=0.07 v/v, time<sub>reaction</sub>=10 min, Q<sub>T</sub>=130 Nml/min

% MULTILAYER GRAPHENE	% FEW-LAYERS GRAPHENE	% BILAYER GRAPHENE	QUALITY
19.67	50.92	29.41	34.7



# RESULTS AND DISCUSSION

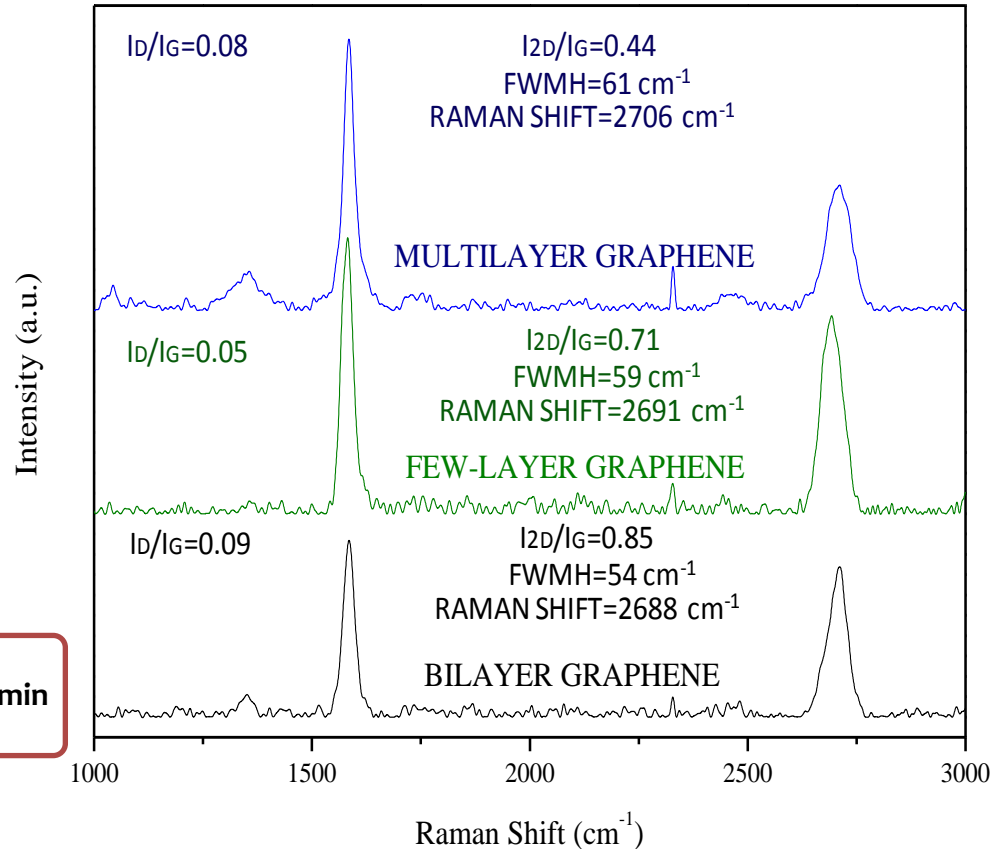
## Influence of the $Q_T$ during the reaction step



Lavin-Lopez, M.P., et al., *Synthesis and characterization of graphene: Influence of synthesis variables*. Physical Chemistry Chemical Physics, 2014. **16**(7): p. 2962-2970.

$T^a_{\text{reaction}}=1050^\circ\text{C}$ ,  $\text{CH}_4/\text{H}_2=0.3$  v/v,  $\text{time}_{\text{reaction}}=10$  min,  $Q_T=130$  Nml/min

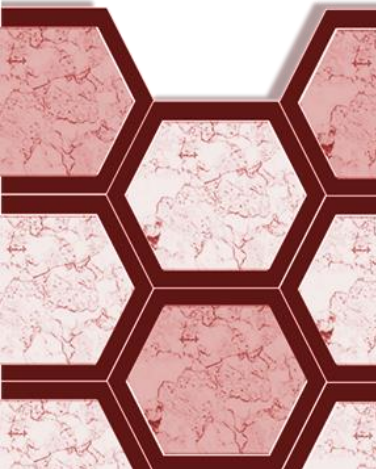
% MULTILAYER GRAPHENE	% FEW-LAYERS GRAPHENE	% BILAYER GRAPHENE	QUALITY
10.87	33.27	55.86	59.3



# COPPER

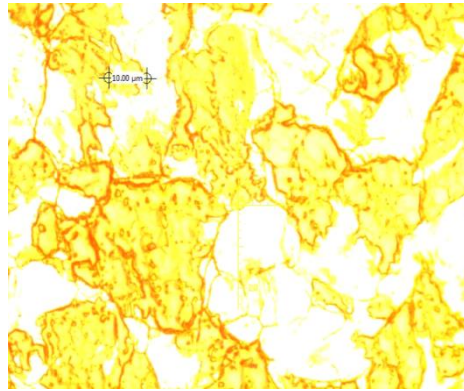
OPTIMUM  
CONDITIONS

$Q_T=60$  Nml/min



# RESULTS AND DISCUSSION

## Influence of the reaction temperature



Lavin-Lopez, M.P., et al., *Thickness control of graphene deposited over polycrystalline nickel*. New Journal of Chemistry, 2015.

# NICKEL

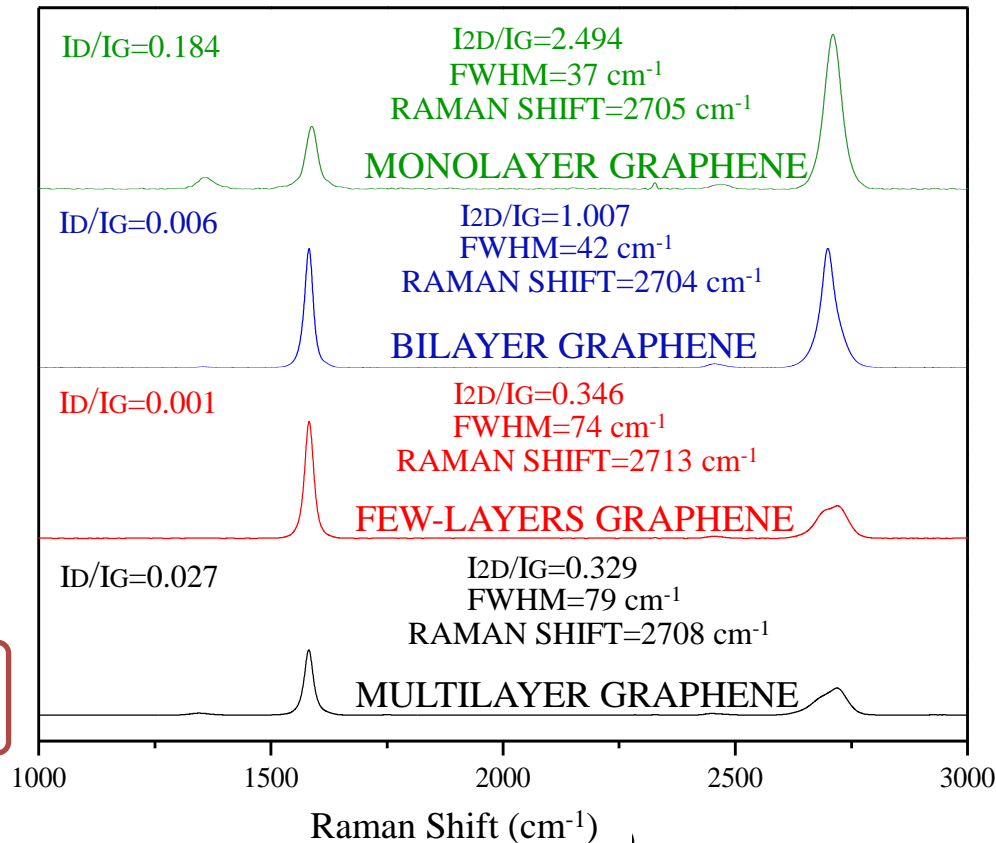
Intensity (a.u.)

OPTIMUM  
CONDITIONS

$T^a=980^\circ\text{C}$

$T^a_{\text{reaction}}=980^\circ\text{C}$ ,  $\text{CH}_4/\text{H}_2=0.3$  v/v,  $\text{time}_{\text{reaction}}=10$  min,  $Q_T=130$  Nml/min

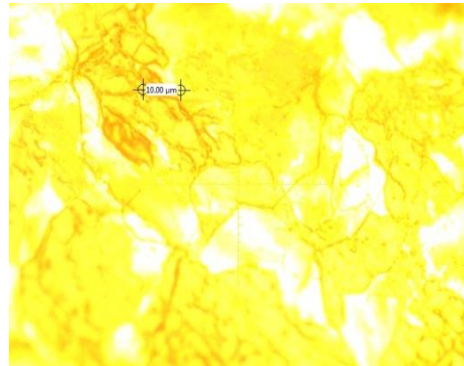
% MULTILAYER GRAPHENE	% FEW-LAYERS GRAPHENE	% BILAYER GRAPHENE	% MONOLAYER GRAPHENE	QUALITY
0.87	40.20	21.80	37.13	397





# RESULTS AND DISCUSSION

## Influence of the CH<sub>4</sub>/H<sub>2</sub> Flow Rate Ratio



Lavin-Lopez, M.P., et al.,  
*Thickness control of graphene deposited over polycrystalline nickel.* New Journal of Chemistry, 2015.

# NICKEL

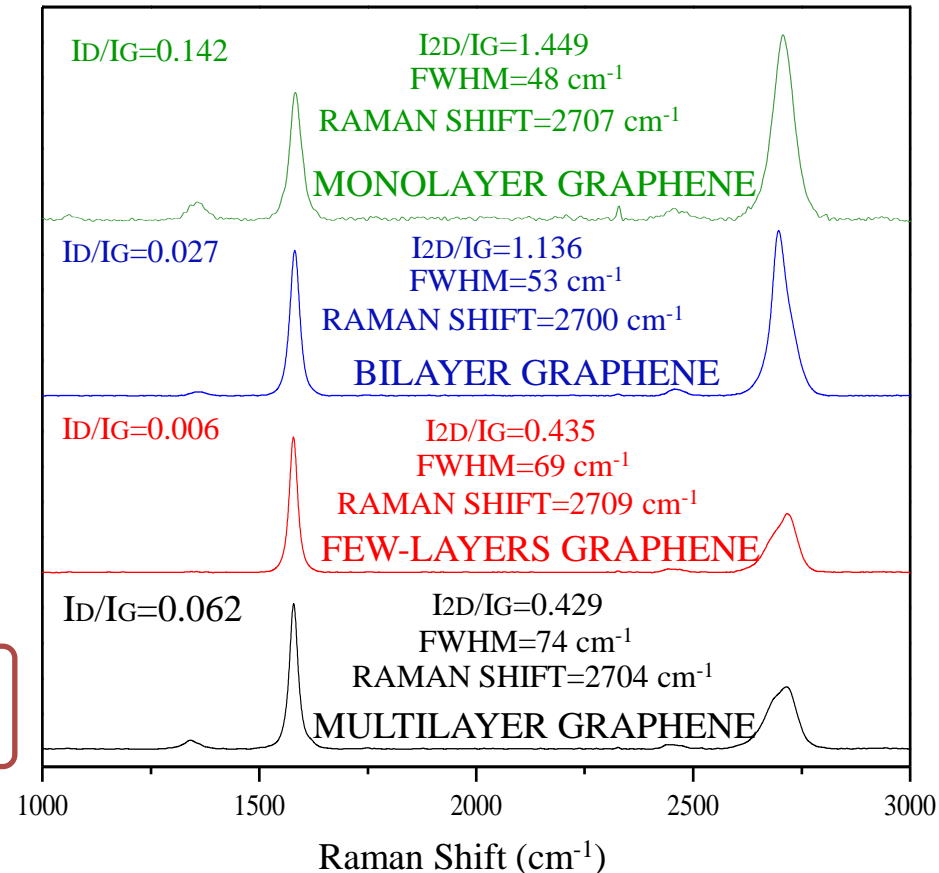
OPTIMUM  
 CONDITIONS

CH<sub>4</sub>/H<sub>2</sub>=0,07 v/v

T<sub>reaction</sub>=980°C, CH<sub>4</sub>/H<sub>2</sub>=0.07 v/v, time<sub>reaction</sub>=10 min, Q<sub>T</sub>=130 Nm<sup>3</sup>/min

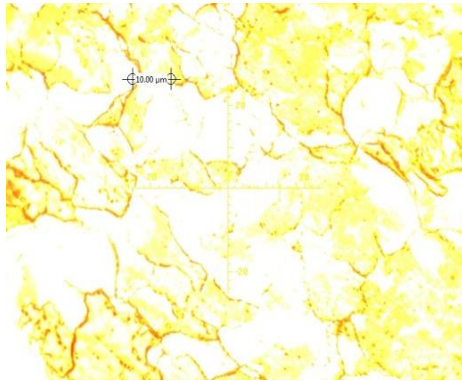
% MULTILAYER GRAPHENE	% FEW-LAYERS GRAPHENE	% BILAYER GRAPHENE	% MONOLAYER GRAPHENE	QUALITY
0.45	27.15	20.39	51.99	536

Intensity (a.u.)



# RESULTS AND DISCUSSION

## Influence of the QT during the reaction step at different times



Lavin-Lopez, M.P., et al., *Thickness control of graphene deposited over polycrystalline nickel*. New Journal of Chemistry, 2015.

### NICKEL

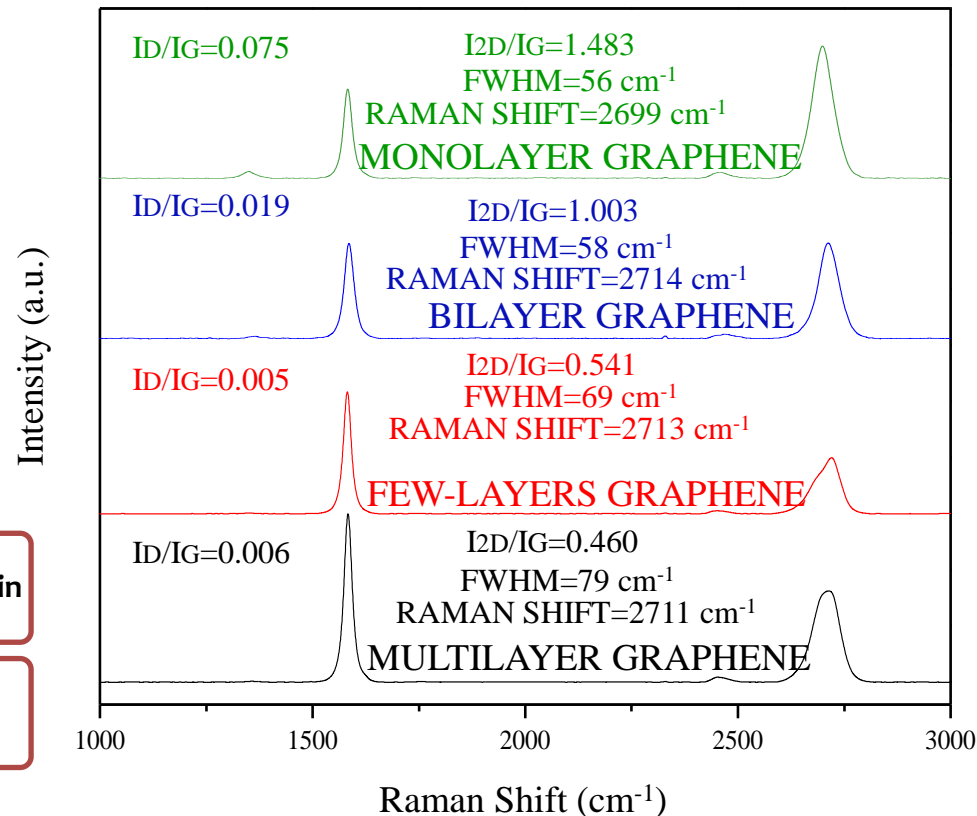
OPTIMUM  
CONDITIONS

$Q_T=80\text{Nml/min}$

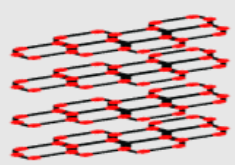
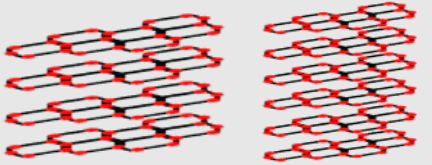
Time=1 min

$T^a_{\text{reaction}}=980^\circ\text{C}$ ,  $\text{CH}_4/\text{H}_2=0.07\text{ v/v}$ ,  $\text{time}_{\text{reaction}}=1\text{ min}$ ,  $Q_T=80\text{ Nml/min}$

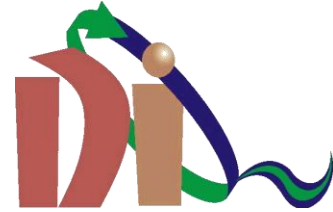
% MULTILAYER GRAPHENE	% FEW-LAYERS GRAPHENE	% BILAYER GRAPHENE	% MONOLAYER GRAPHENE	QUALITY
0.33	11.15	8.41	80.12	810



# CONCLUSIONS

VARIABLE	COPPER	NICKEL
REACTION T <sup>a</sup>	1050 °C	980 °C
CH <sub>4</sub> /H <sub>2</sub> FLOW RATE RATIO	0.07 v/v	0.07 v/v
Q <sub>T</sub> DURING REACTION STEP	60 Nml/min	80 Nml/min
REACTION TIME	10 min	1 min
GRAPHENE		
QUALITY	59	810

# CVD-GRAPHENE SYNTHESIS USING DIFFERENT TRANSITION METALS AS CATALYST



**M<sup>a</sup> del Prado Lavín López**

**Graphene  
Canada**

**October 14-16, 2015  
Montreal, Canada**

Graphene & 2D Materials International Conference and Exhibition

