

GraphITA,
Laboratori Nazionali del
Gran Sasso, Assergi (AQ)
May 15, 2011

Effect of the synthesis parameters on the dc resistance of graphite nanoplatelets thick films

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Outline

- Synthesis of GNPs via Thermal Exfoliation
- Fabrication of GNPs thick films
- SEM and AFM characterizations
- FTIR spectroscopy of GNP-based foils
- DC conductivity measurements
- Conclusions

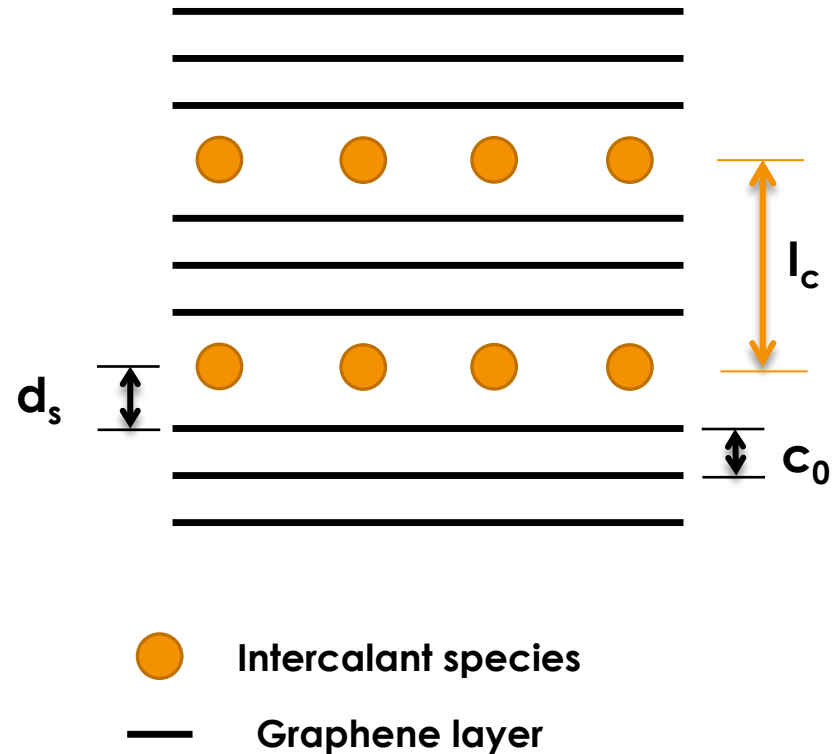
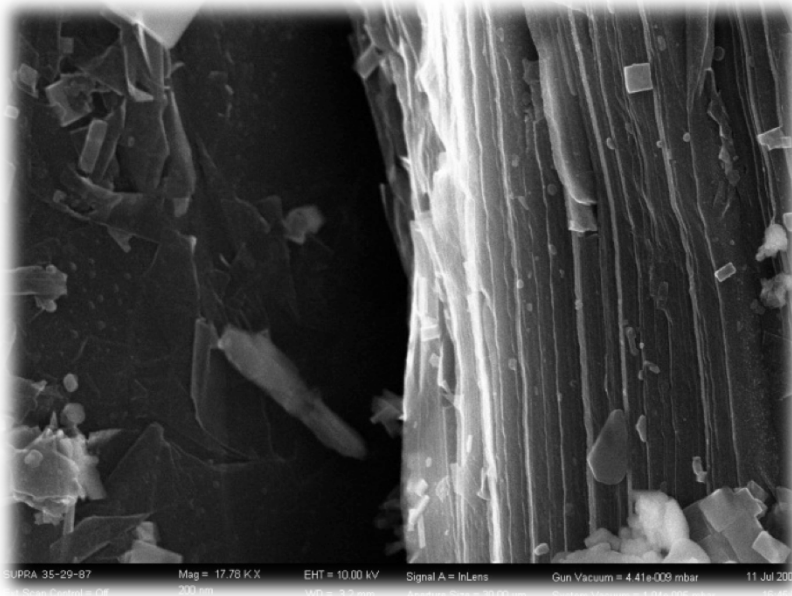
Graphite intercalation compounds (GIC)

- **1841** GICs first synthesized (with Potassium as the intercalant) [1]
- **1859** Brodie prepared the first graphitic acid (graphite oxide)
- **1898** Staudenmeier improved the Brodie process using sulfuric acid
- **1957** Hummers and Offemann further enhanced the technique efficiency, considerably reducing the time required for the whole process

[1] Schafhautl, 1841

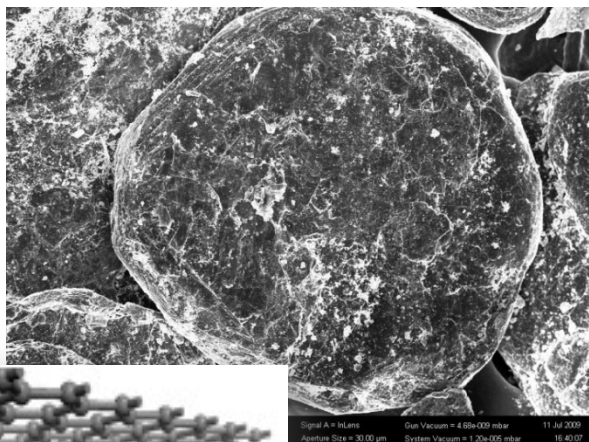
Graphite intercalation compounds are formed by the insertion of atomic or molecular layers of a different chemical species called the intercalant between layers in a graphite host material

GIC structure



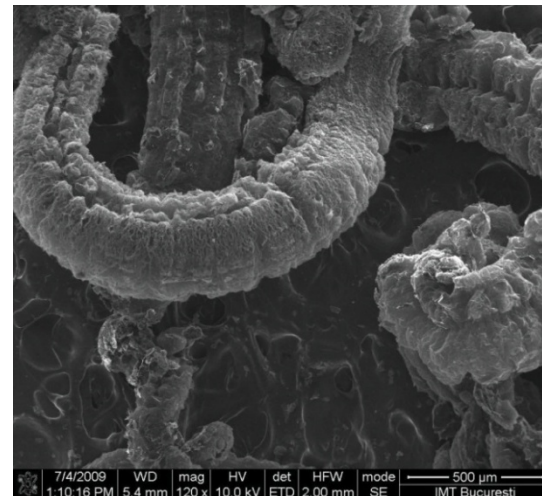
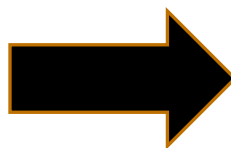
The stage index n can be extracted from the relation $l_c = (n-1)c_0 + d_s$

Thermo-chemical Exfoliation



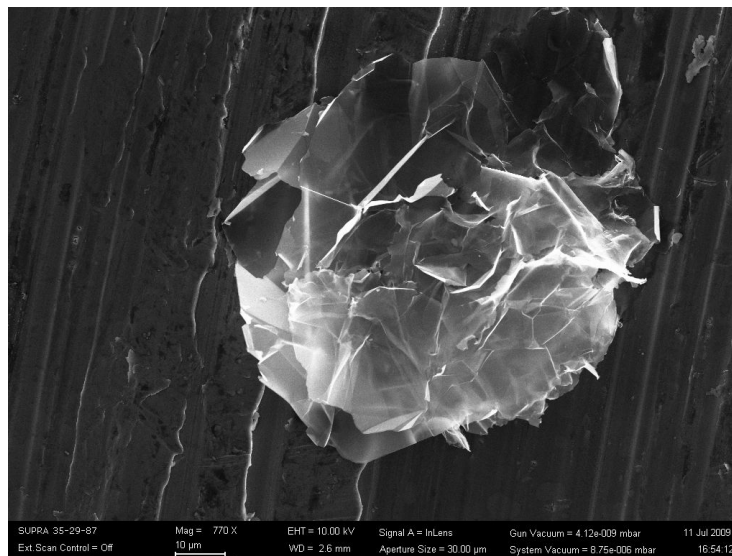
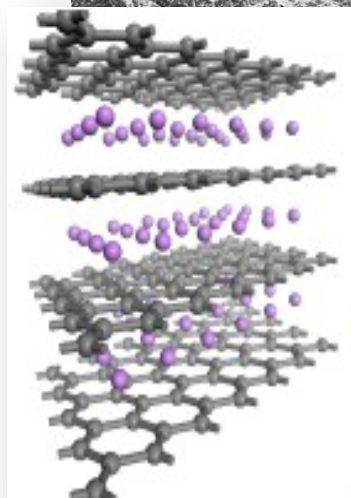
GIC flake

+ thermal shock



+

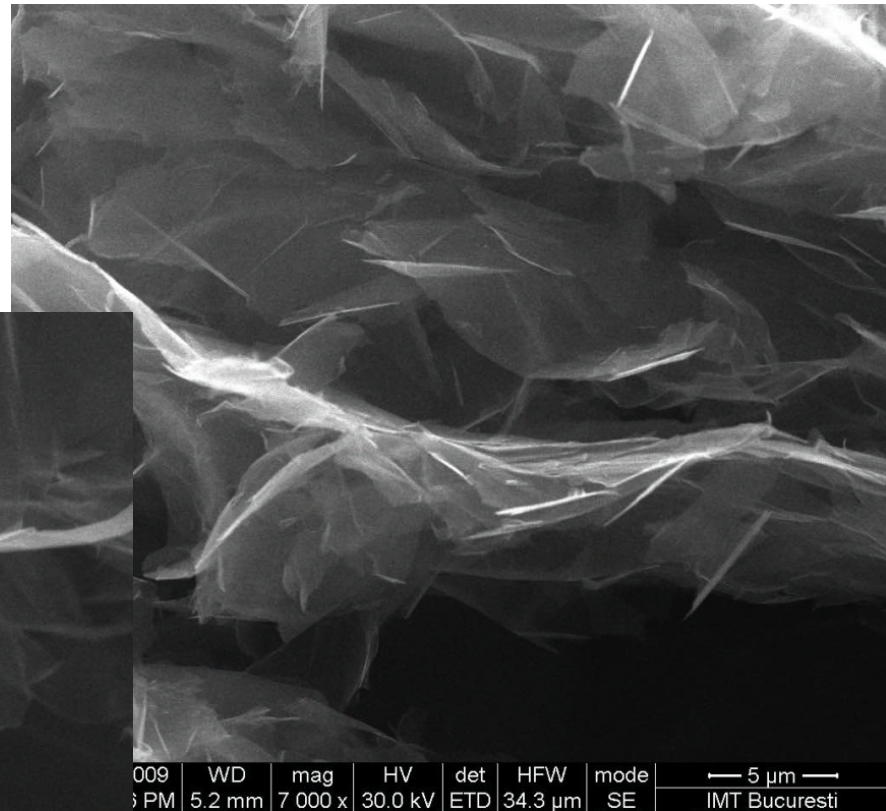
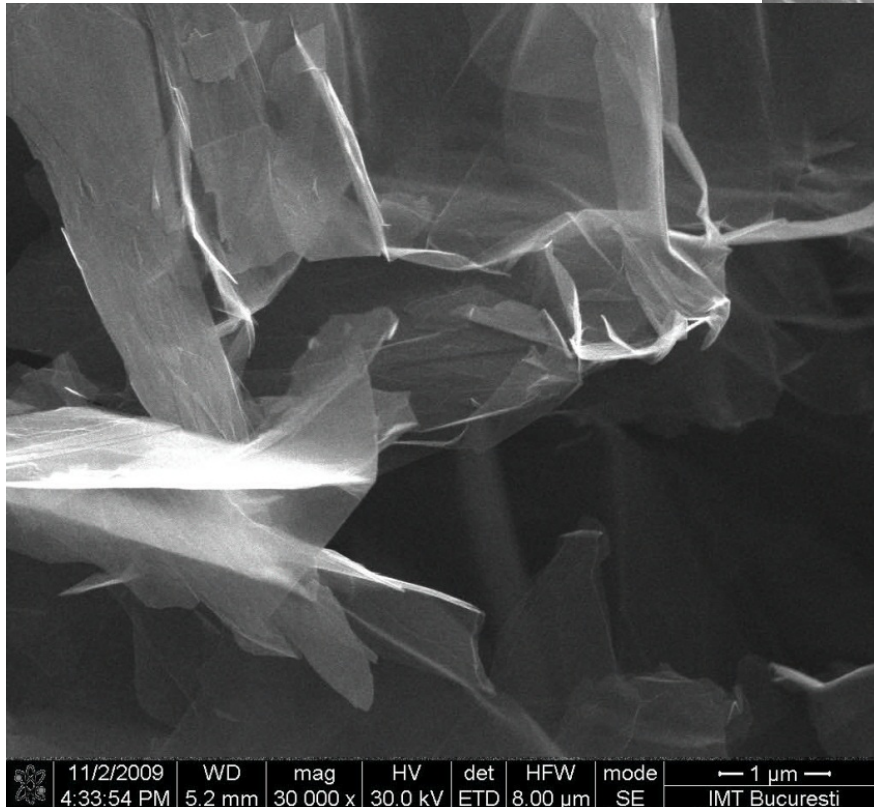
Ultrasonication



Expansion conditions

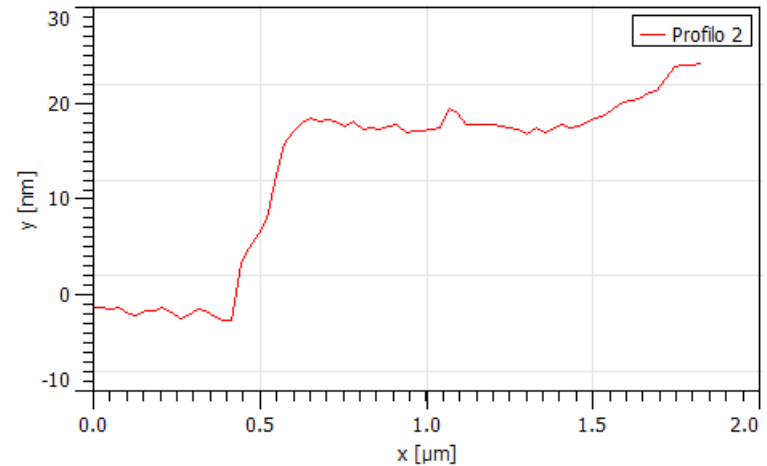
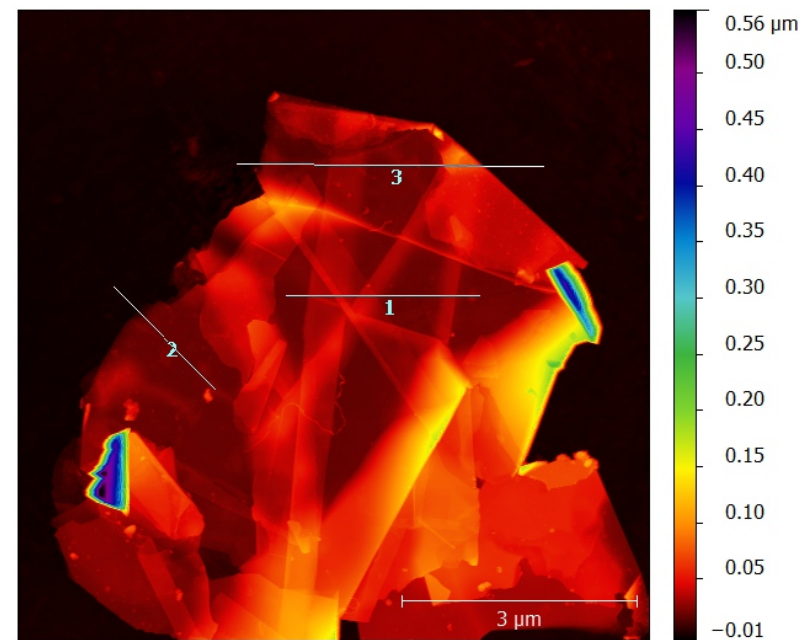
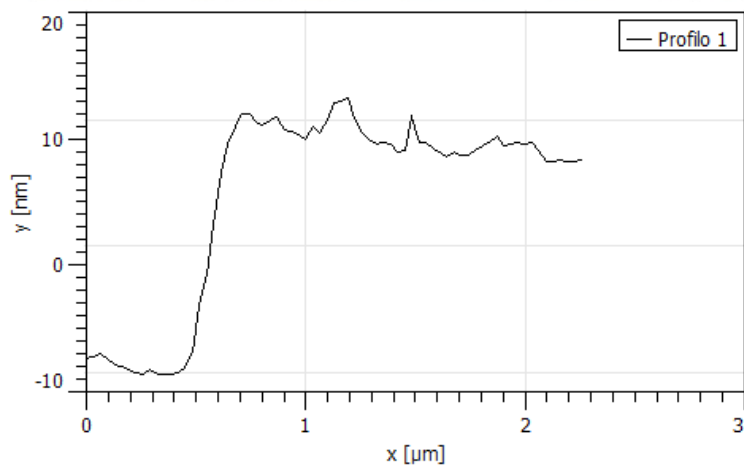
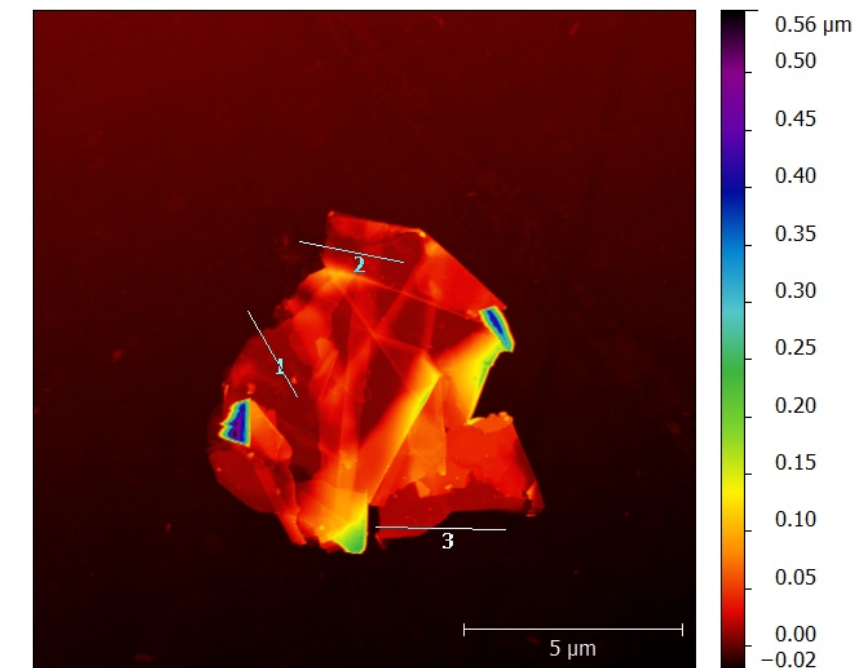
TEMPERATURE	EXPANSION TIME			
250°C		15s	30s	60s
500°C	5s	15s	30s	60s
750°C	5s	15s	30s	60s
1050°C	5s	15s	30s	50s
1150°C	5s	15s	30s	60s
1250°C	5s	15s	30s	

SEM imaging

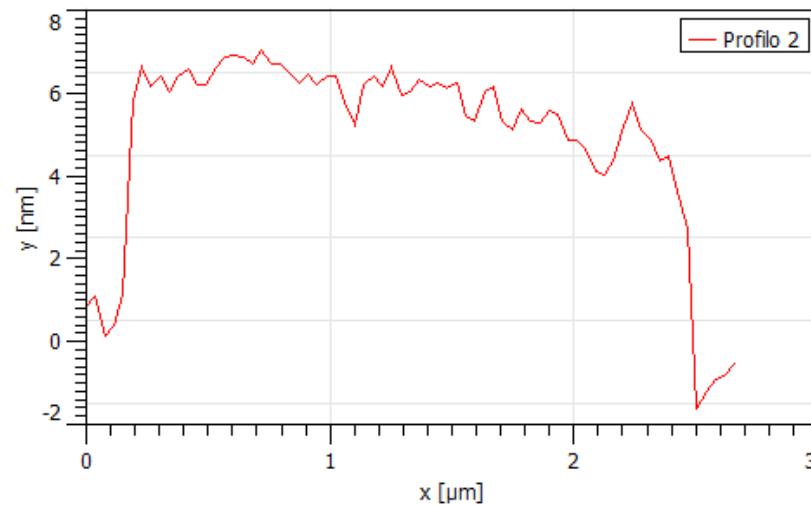
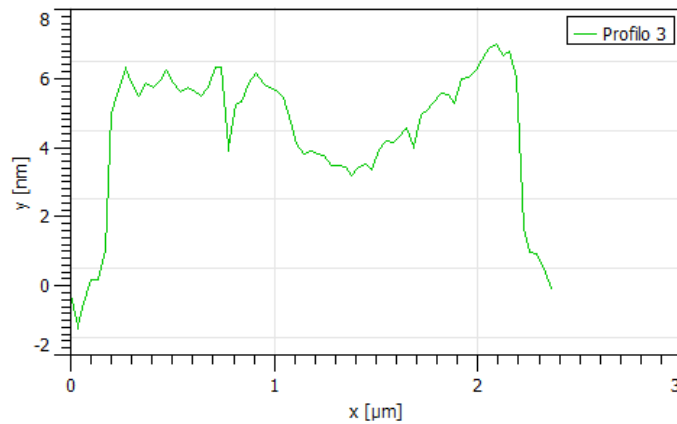
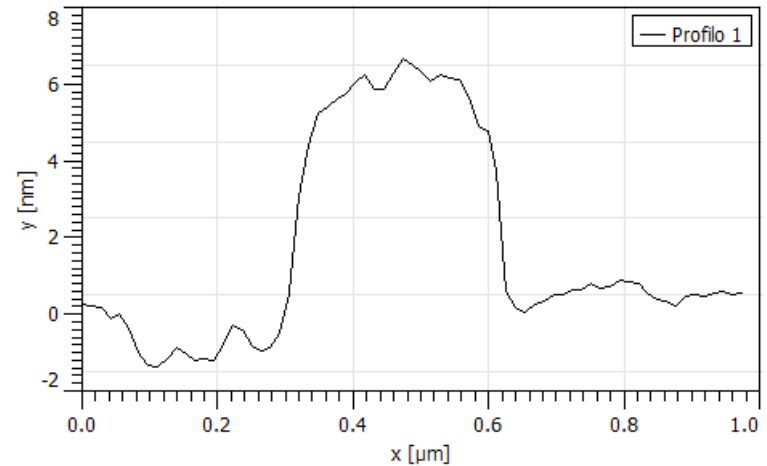
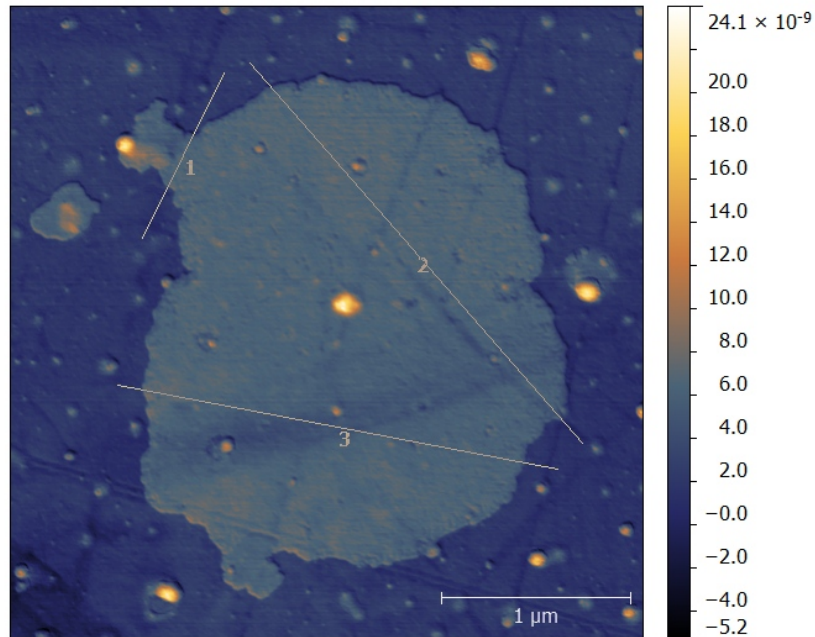


SEM Images courtesy of
Dr A.Dinescu, IMT Bucharest

AFM imaging 750°C-30s

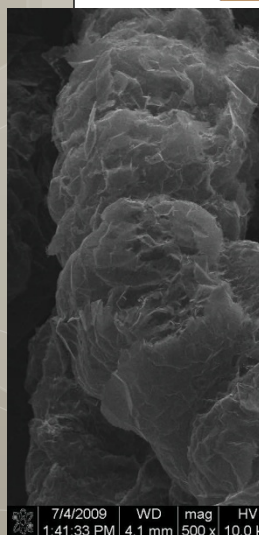
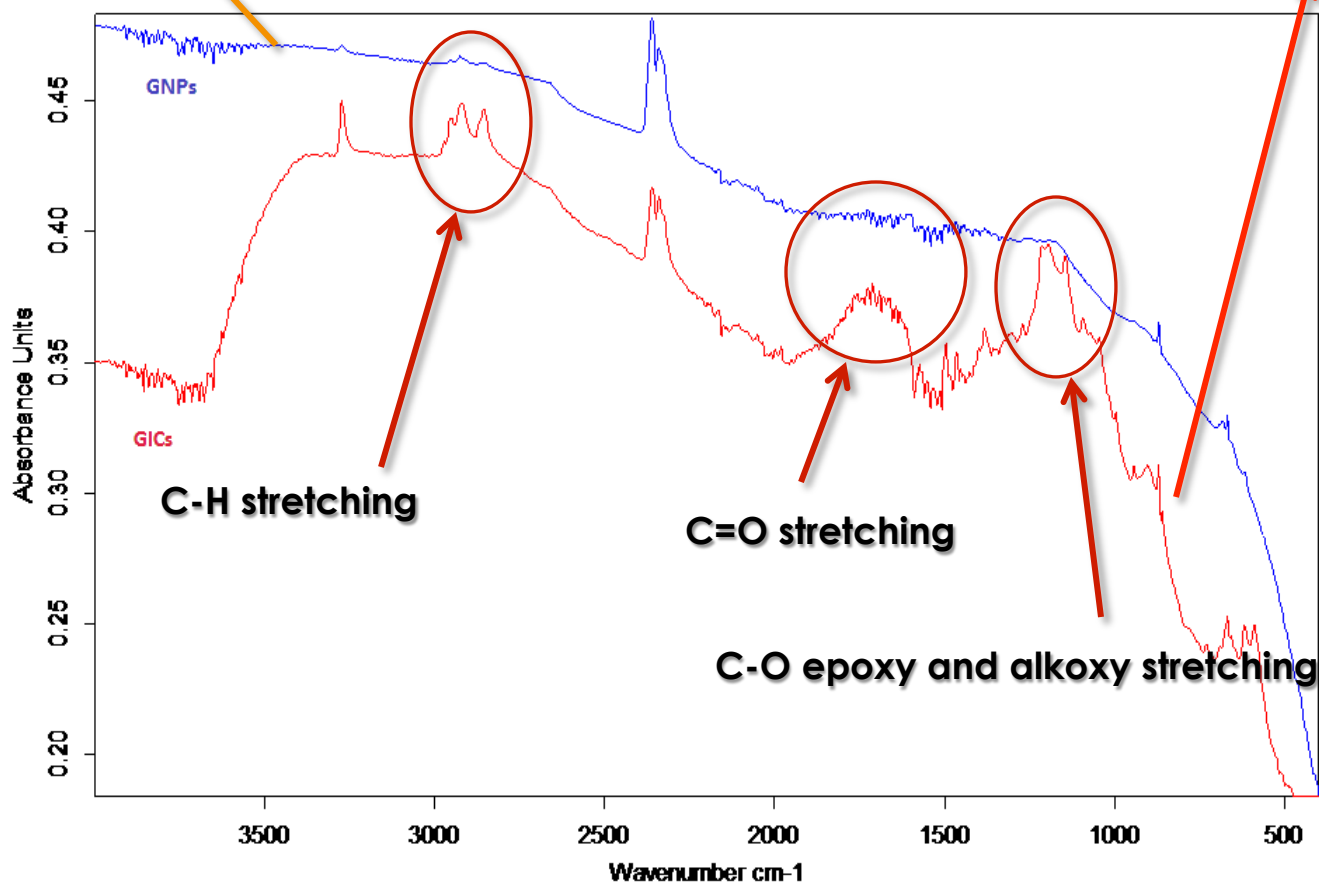
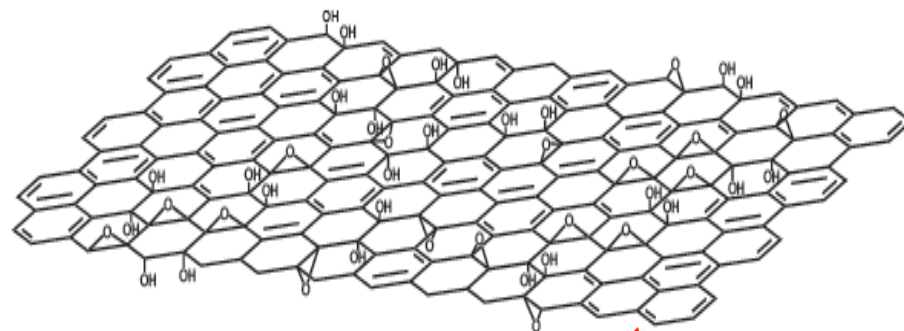
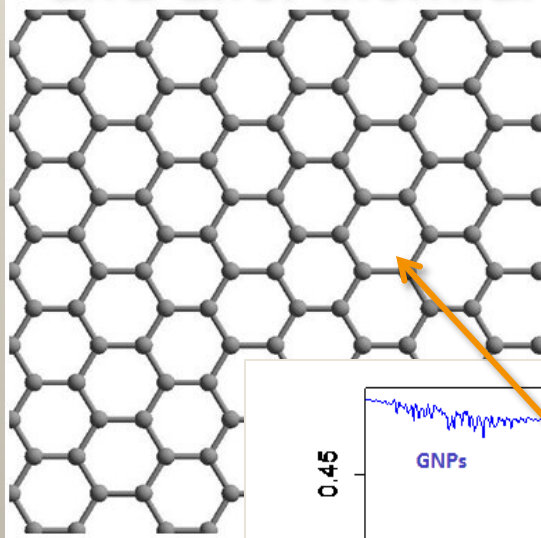


AFM imaging 1250°C-30s



FTIR spectroscopy on GICs before and after thermal expansion

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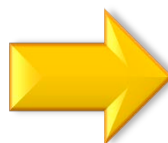
7/4/2009 WD mag HV
1.41 33 PM 4.1 mm 500 x 10.0 kV



GNP films preparation



Worm-like
graphite



Ultrasonication



GNP film

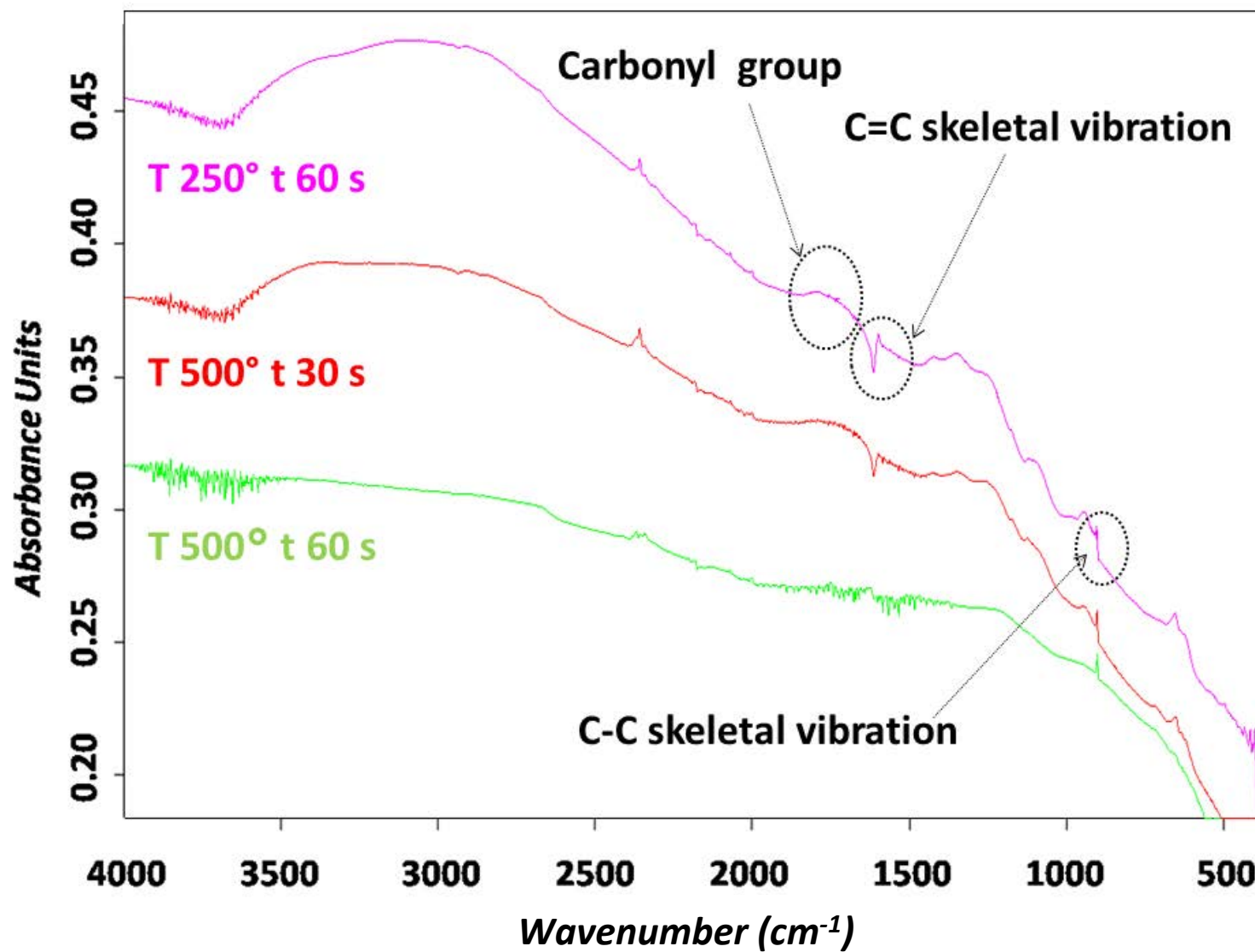


Vacuum
filtration



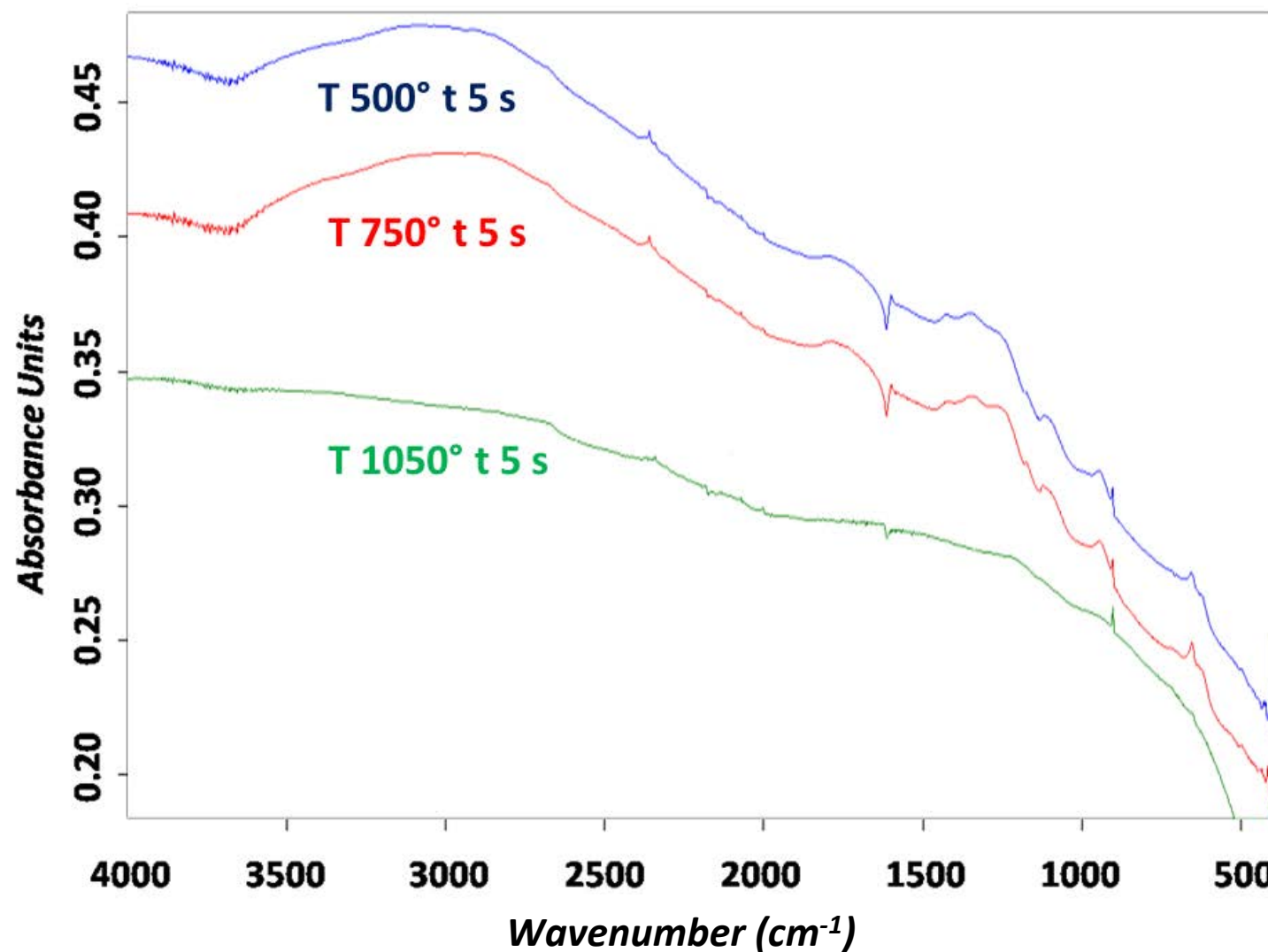


FTIR spectroscopy



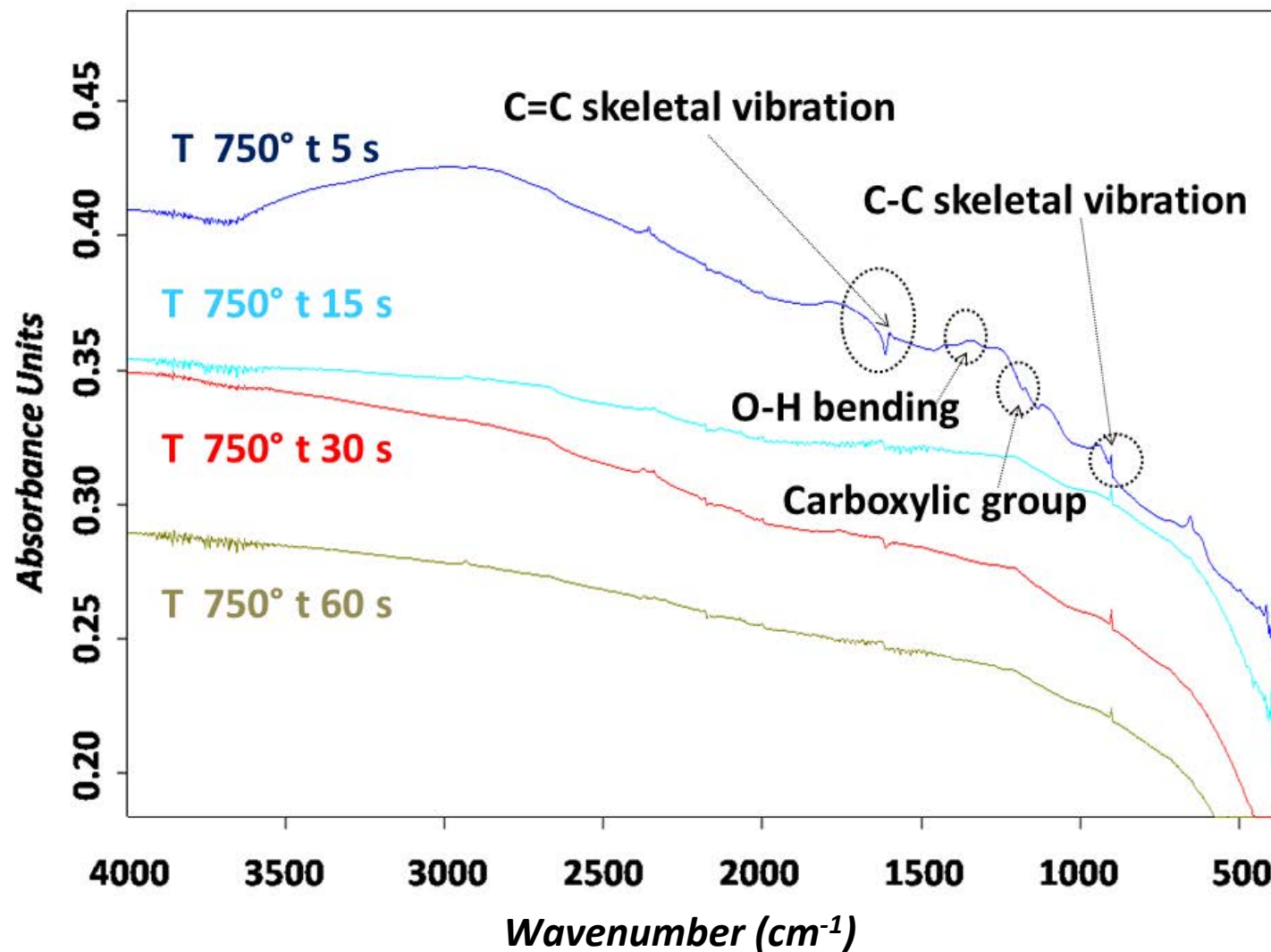


Effect of the expansion temperature



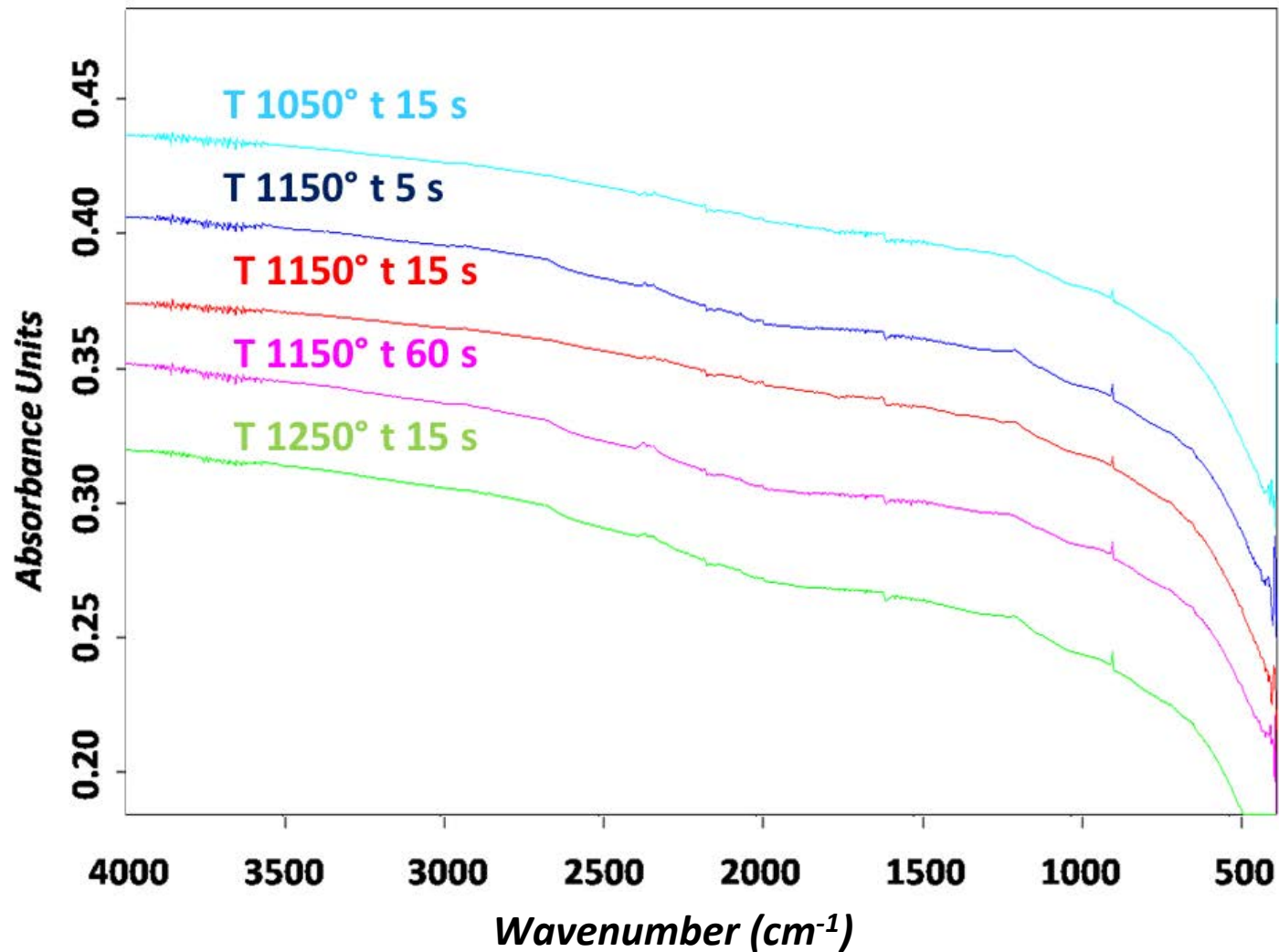


Effect of the expansion time





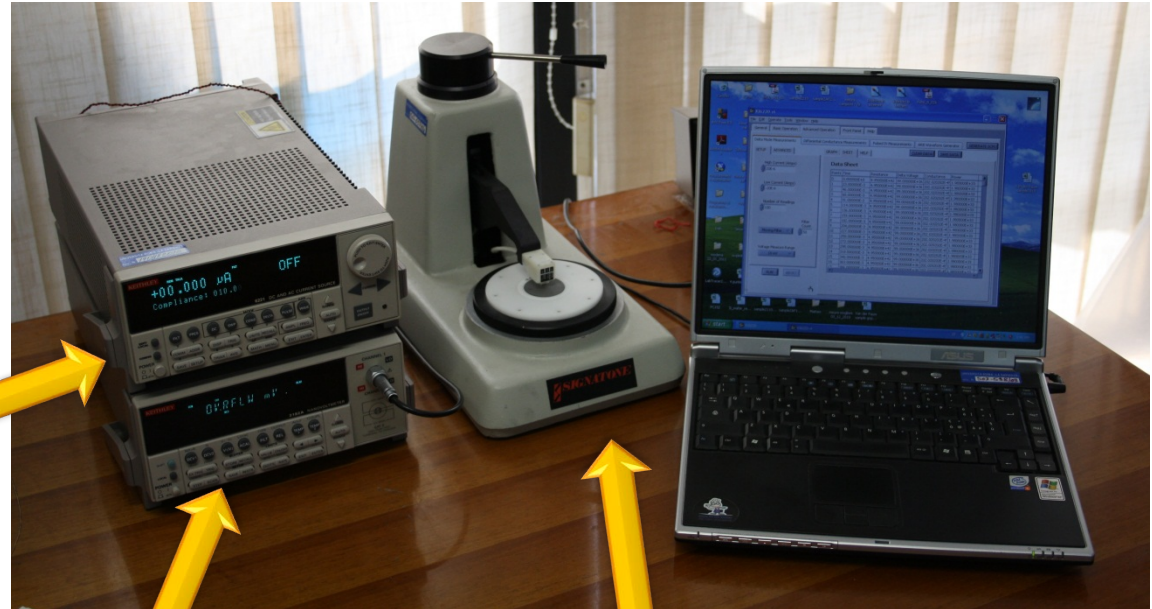
High temperature expansion



DC measurements experimental setup



Source meter



Nano Voltmeter

Four probe station

$$R_{\square} = \pi / \ln(2) V / I \text{ } [\Omega / \text{square}]$$

$$\sigma = 1 / R_{\square} t \text{ } [S / m]$$



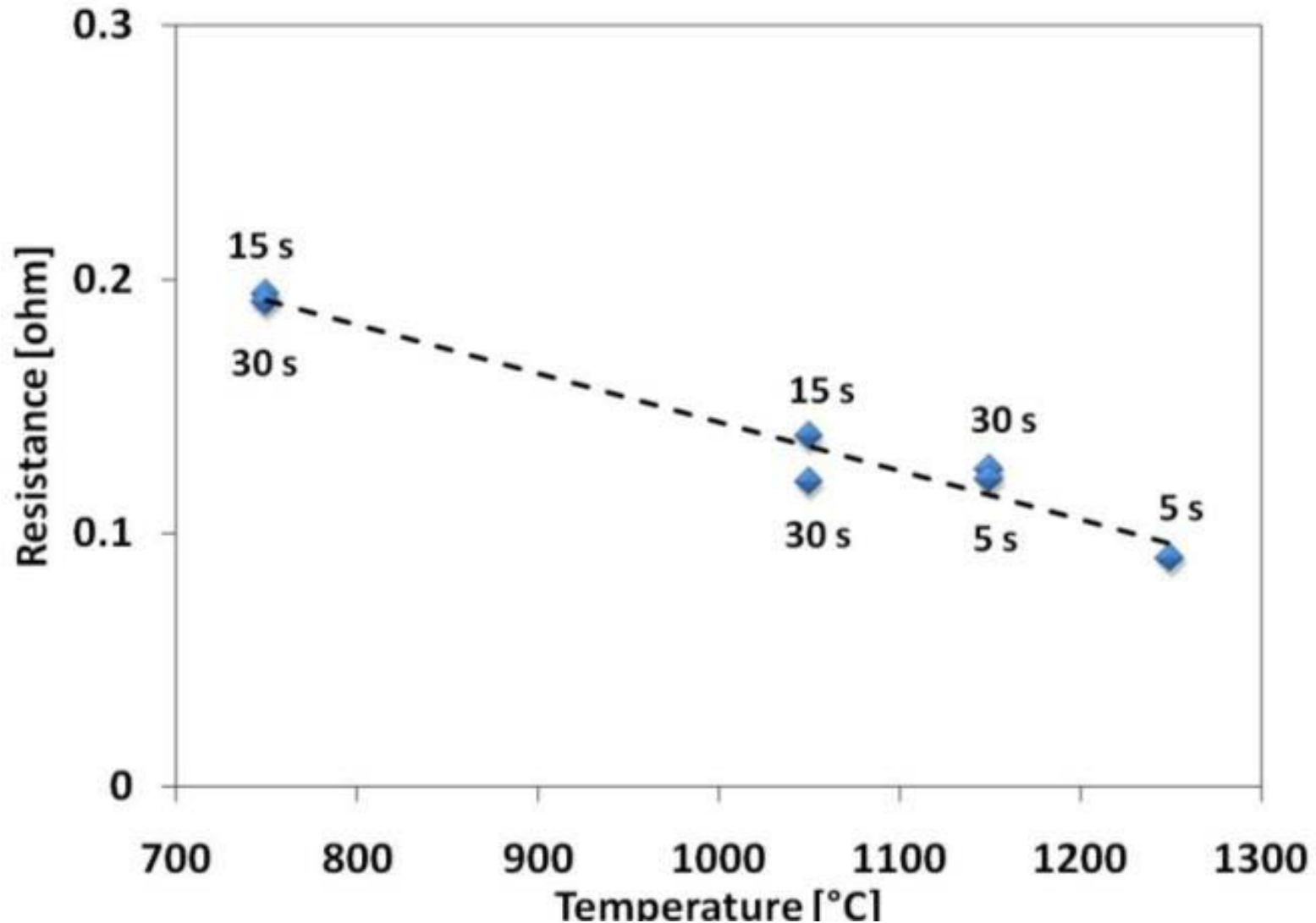
DC measurements results

Expansion conditions	Resistance (V/I) [Ω]	Conductivity* [S/m]
T 750° † 15s	0.194	5808
T 750° † 30s	0.191	5919
T 1050° † 15s	0.138	8063
T 1050° † 30s	0.120	9273
T 1150° † 5s	0.125	9627
T 1150° † 30s	0.121	9196
T 1250° † 5s	0.090	12393

*Estimated assuming an average film thickness of 200 μm

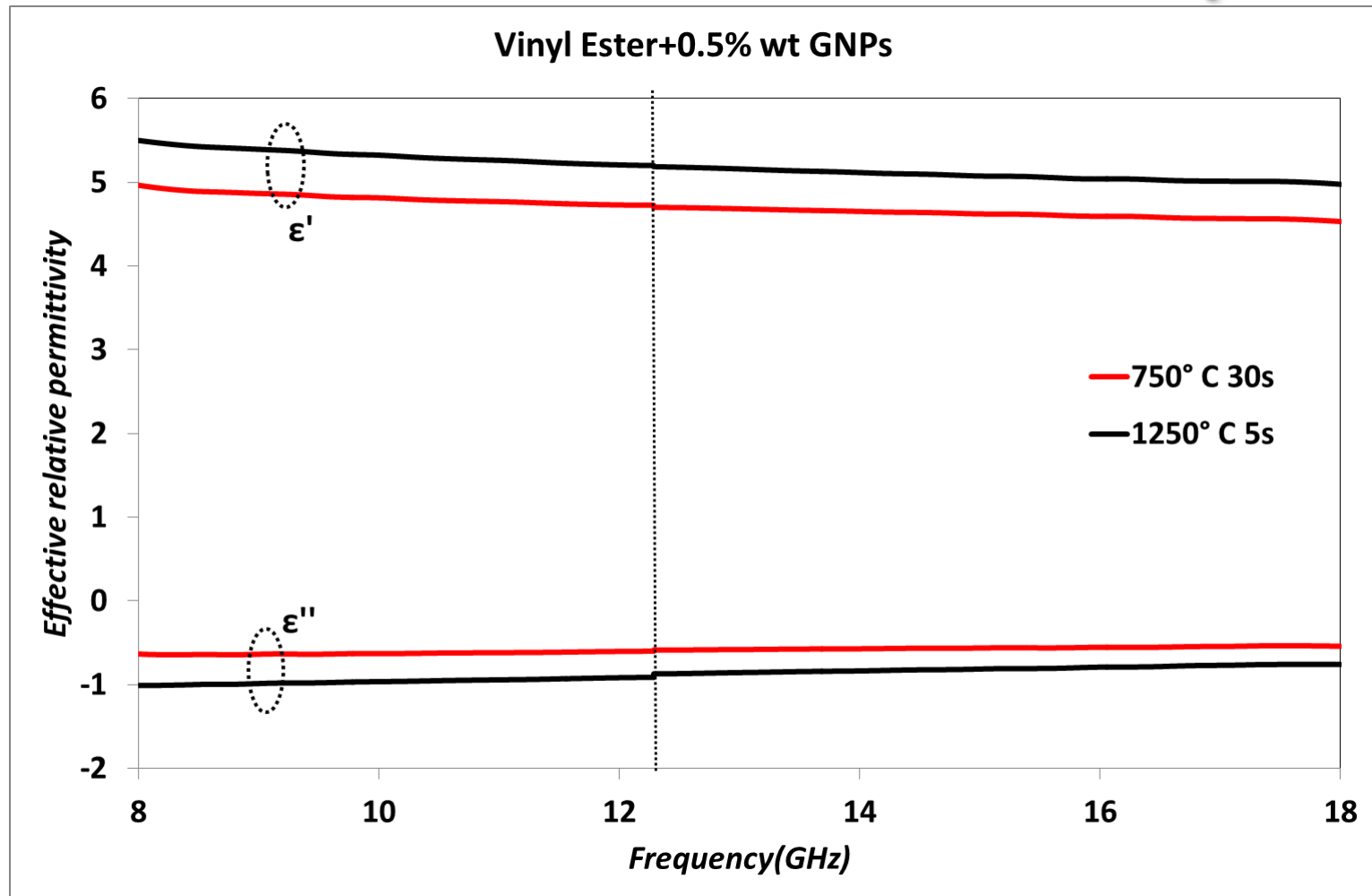


Resistance vs expansion temperature



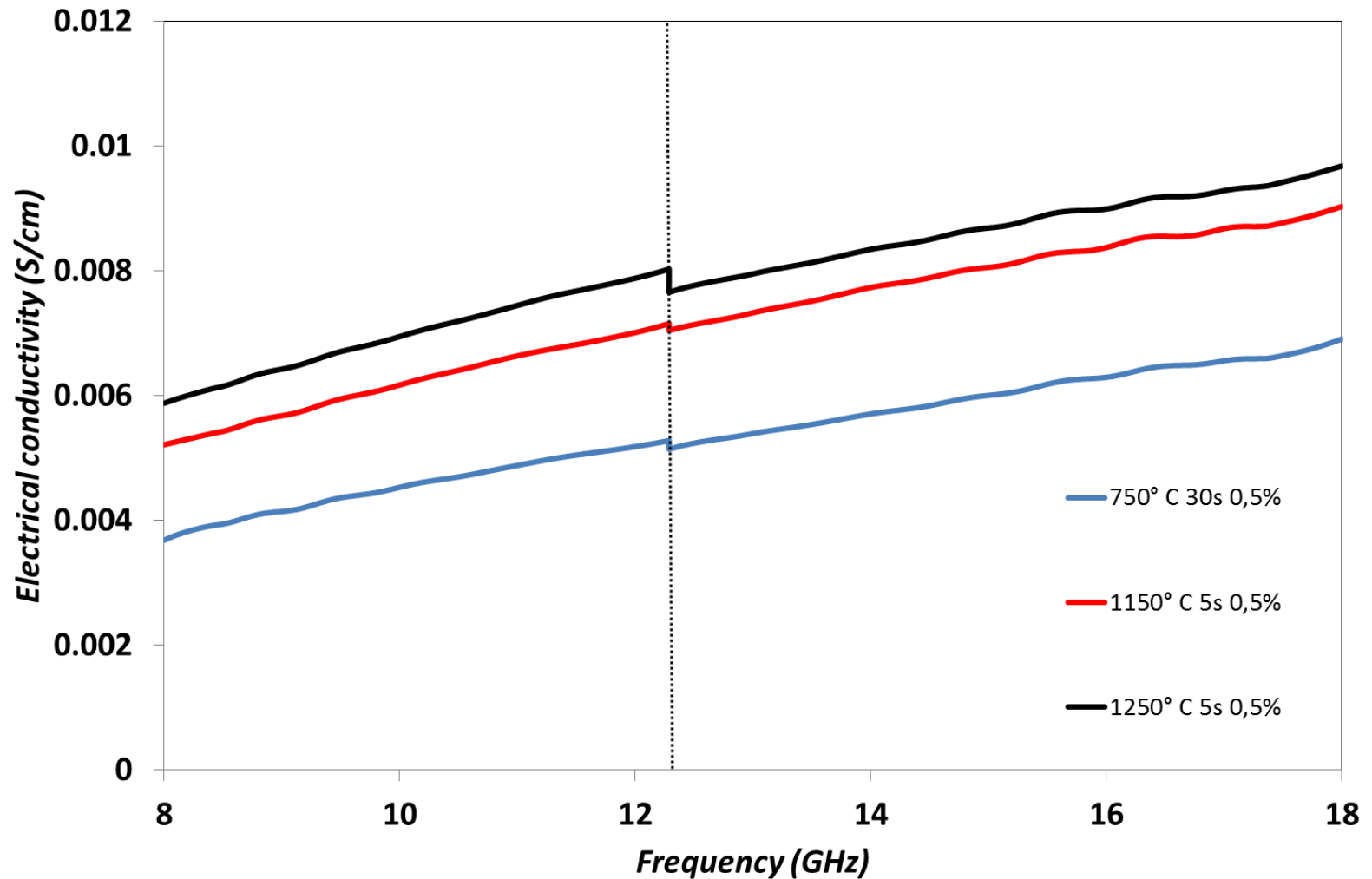


Effect of the synthesis conditions on the EM performance of GNP-based nanocomposites





Electrical conductivity of nanocomposites loaded with 0.5% wt GNPs





Concluding remarks

- The morphological and physical properties of GNPs produced by TCE of GICs are strongly affected by expansion temperature (T) and time (t)
- FTIR spectra are nearly independent from those parameters for $T > 1000^{\circ}\text{C}$
- Proper choice of the processing conditions enables tailoring of the electrical properties
- GNP-filled composites with tailored EM properties can be realized by properly choosing the process parameters for the GNP synthesis



**Thanks for your
attention**