

## STSM scientific report

**Host:** Ana Inés Fernández (DIOPMA – Universida de Barcelona)

**Applicant:** Vladimir Canseco Ruiz (Trefle – CNRS laboratory)

**Duration:** 5<sup>th</sup> – 16<sup>th</sup> of July 2010

**Reference:** Cost TU0802 - 6608

The present scientific report describes the main results obtained during the STSM hosted by Dr. Ana Inés Fernández at the Universidad de Barcelona – DIOPMA group (Centro de Diseño y Optimizacion de Procesos y Materiales).

### Purpose of the visit

The main purpose of the STSM was to perform a mechanical characterization of graphite – salt composite materials by using several experimental techniques such as nanoindentation and micro-compression tests. The experimental work carried out allowed us to compute the following parameters: Young modulus, hardness and yield strength. The mechanical characterization was completed with some microscope techniques (AFM and SEM observations).

### I Description of the work carried out during the visit

Before resuming the work, a brief description of the graphite – salt composite materials is presented. The composite is formed by a graphite porous foam (72% average porosity) infiltrated with salt (nitrates). All the tests performed during the STSM were done with three different types of samples, each one corresponding to a different type of graphite foam. The main differences among these graphite foams (named D1, L1 250, and L1) were their value of average porosity, thermal conductivity and density. Also, a series of micro-compression tests were performed with the three types of foams (with salt a no salt).

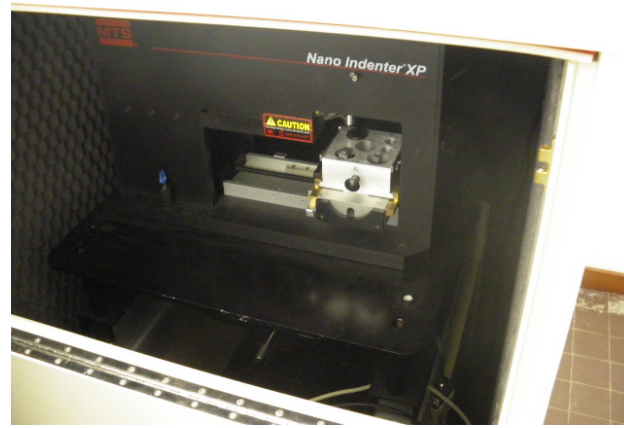
Finally it is important to mention that the samples (graphite + salt and graphite alone) were cut in two different orientations in order to verify the anisotropy of the material.

#### Nanoindentation experiences

The following steps describe the experimental procedure for the nanoindentation tests:

- Preparation of the samples: this step consists in fixating the samples to a cylindrical support and polishing the surface were the nanoindentation will be performed. Since the salt reacts with water and other solutions, we executed a dry polish *i.e.* without applying the typical colloidal suspension with diamond particles.
- Selection of the area: here the portion of the surface that will be indented is selected. In our case, we chose an area having salt and graphite. The area must be as flat as possible.
- Indentation test: for the tests, we used a Nano Indenter XP (figure 1). All the tests were performed with a Berkovich trihedral pyramid indenter and a depth of 200 nm was selected for all the indentations. Once the tests were done, the

obtained data allowed us to compute the Young modulus and Hardness of the samples. A Poisson's ratio of 0.3 (which corresponds to an anisotropic material) was chosen for computing these parameters.

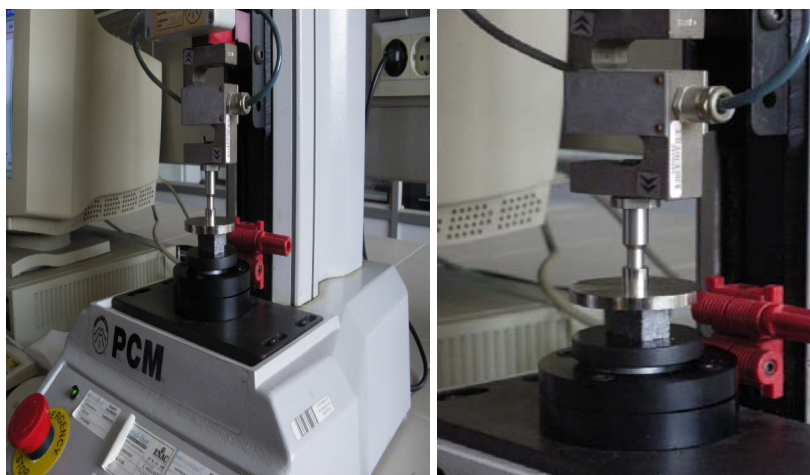


**Fig.1: Nano Indenter device used for testing the samples**

### Micro – compression tests

Beside the indentation tests, a series of micro-compression tests were carried out. The experimental procedure is:

- Preparation of the samples: as for the previous test, the sample must be polished in order to obtain a flat surface and it is important to have a sample with parallel surfaces since the tip that compress the sample is a flat disc.
- The equipment used for this test (presented in figure 2) has a maximum load of 2.5kN. During the experiments the compression rate was varied from 1mm/min to 10 mm/min.



**Fig. 2: Micro – compression machine.**

- With the resulting data, the stress – strain curves were plotted.

### AFM – SEM samples observations

Once the indentation and micro – compression tests were executed, observations were done with AFM & SEM microscopes (figure 3).

For the AFM observations, no particular preparation is needed for the samples. In the case of the SEM, the sample needs to be placed in a special support designed for the microscope and also the silicon used for fixating the sample with the support has to be recovered with a colloidal suspension containing silver particles since all the elements need to be conductive materials. Both type of observations (AFM & SEM) were done at different scales and different sections of the samples with a particular interest for the interface zones.

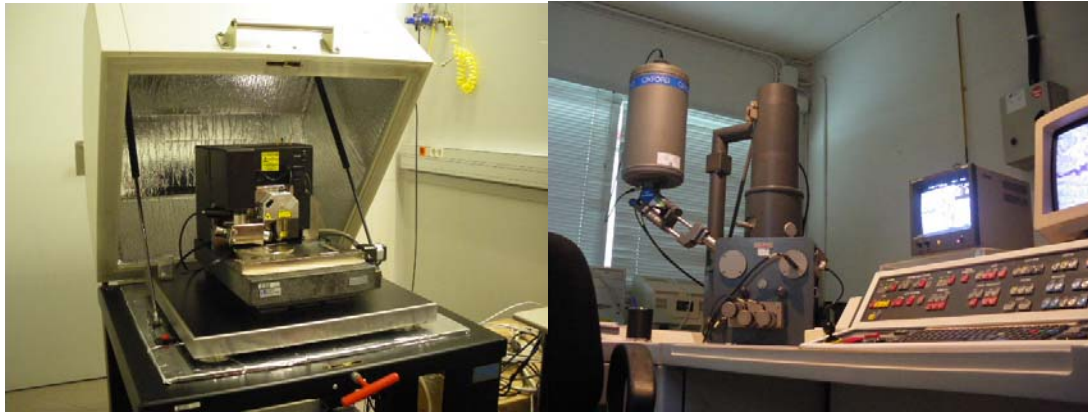


Fig. 3: AFM and SEM microscopes used for samples observations.

## II Description of the main results obtained

As mentioned in the previous section, the indentation and compression data was used to compute the Young modulus, the hardness and the yield strength.

In the case of the indentation experiments, a statistical method was applied for obtaining such parameters. This method consists in fitting the data with an error function (cumulative distribution function) which allows us to identify and quantify the equivalent Young modulus and hardness of the graphite, the salt and the graphite – salt interface. These results are illustrated in figure 4.

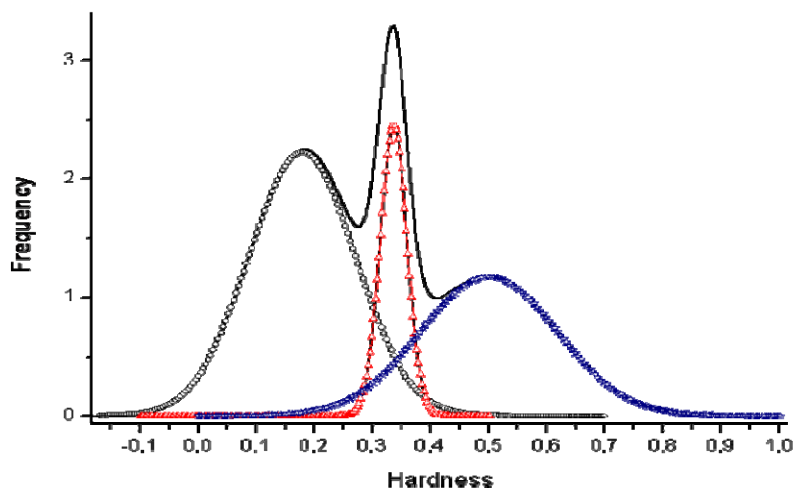


Fig. 4: Cumulative distribution function of hardness after fitting.

The cumulative distribution function has the following form:

$$CDF = 0.5f_1 \left( 1 + \operatorname{erf} \left[ \frac{(x - x_1)}{\sqrt{2}\sigma_1} \right] \right) + 0.5f_2 \left( 1 + \operatorname{erf} \left[ \frac{(x - x_2)}{\sqrt{2}\sigma_2} \right] \right) + 0.5(1 - f_1 - f_2) \left( 1 + \operatorname{erf} \left[ \frac{(x - x_3)}{\sqrt{2}\sigma_3} \right] \right)$$

where  $f_1$  and  $f_2$  correspond to the contribution of the graphite and salt phases (with  $1 - f_1 - f_2 = f_3$  been the contribution of the graphite – salt interface) to the hardness;  $\sigma_1$ ,  $\sigma_2$  and  $\sigma_3$  are the standard deviations (the width) of each peak and  $x_1, x_2$  and  $x_3$  are the hardness (or young modulus) of the corresponding phases.

Each of the three gaussian peaks in figure 4 correspond to one of the terms of the cumulated distribution function and thus to a phase in the sample. In the plot, the first gaussian peak (from left to right) corresponds to the graphite, the second one to the salt – graphite interface and the third one to the salt. As expected the graphite phase has the lowest hardness and the salt phase the highest with the interface in the middle. The same cumulative distribution function was obtained for the Young modulus and this statistical method treatment was repeated for each of the samples data.

From the compression tests, the stress – strain curves were plotted and the Young modulus were determined for the elastic regime as illustrated in figure 5. All the curves correspond to graphite salt samples, the same type of curve were obtained for the pure graphite samples. In some cases for the graphite salt samples one compression test wasn't enough to crush the samples, thus a series of cycles were performed. At the end a few samples didn't collapse at all, nevertheless some fractures appeared in those samples.

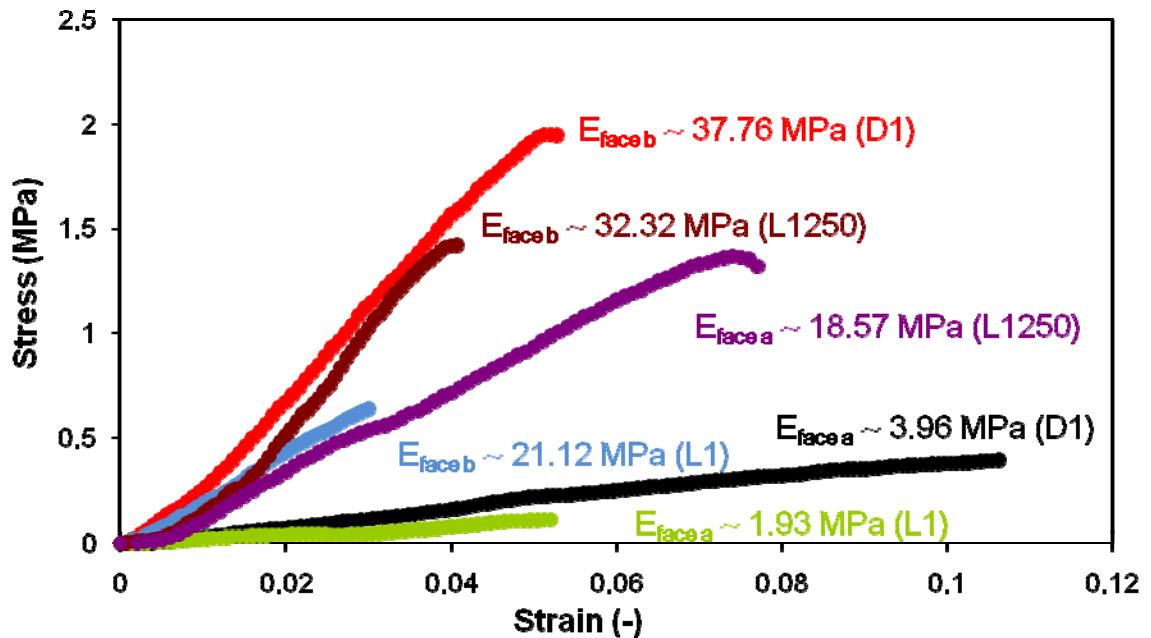
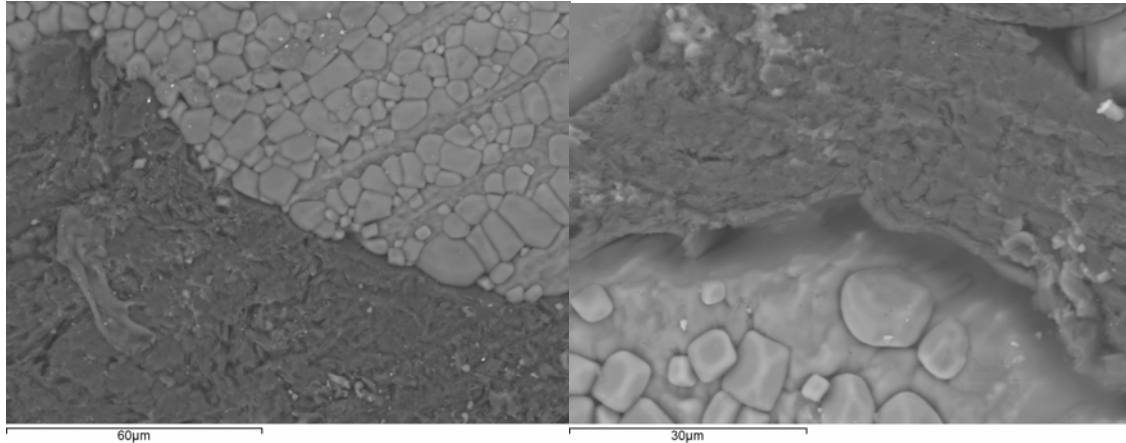


Fig. 5: Stress – strain curves as a function of the sample's orientation.

Each curve corresponds to a different sample orientation (named face *a* or face *b*). We can see that face *b* (for the three samples D1, L1 and L1250) presents highest values of

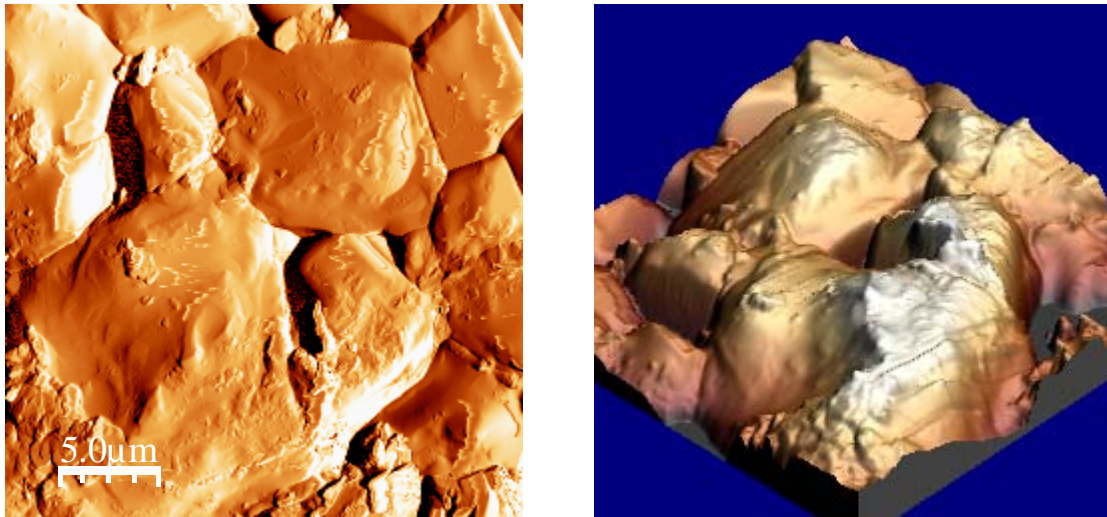
Young modulus than face  $a$  and thus we can confirm the hypothesis that we are working with a high anisotropic material.

The following figures correspond to the observations made with the AFM and SEM microscopes.



**Fig. 6: SEM images for two different samples (graphite + salt).**

The salt grains are clearly visible in these series of images. The sample structure is particularly anisotropic and graphite salt interface presents some interesting concepts. For the L1250 samples, the interface shows a good cohesion between the salt and the graphite (left image in figure 6) but for the L1 sample (right image) we can see some gaps between the graphite and the salt. This gap may be linked to the type of foam and its affinity with the salt, or to the contraction of the salt when it crystallizes. Nevertheless the gap can be the result of the polishing process since a stress is applied to the sample in order to obtain a good polished surface.



**Fig. 7: AFM images for a graphite + salt sample.**

Finally I would like to thanks Ana Inés Fernández for hosting this STSM, Joan Josep Roa Rovira for helping me and all the DIOPMA team.

**Future collaboration with the host institution:**

The work carried out at the DIOPMA institute as well as the obtained results proved to be very useful and interesting. Many concepts about mechanical characterization at the micro and nano scale were learned and discussed. In order to push such characterization farther and share more knowledge between our two research teams, a future collaboration with the host institution is being setup.

**Foreseen publications/articles resulting or to result from the STSM:**

The collected data obtained from the experiments will be analyzed and completed with a thermal analysis. After this, an article will be written and submitted to a journal.

**Confirmation by the host institute of the successful execution of the mission**

The 2 weeks STSM performed by Vladimir Canseco Ruiz from Trefle – CNRS UMR 8508 Laboratory was successfully accomplished since important data was obtained from the mechanical tests that were executed. The resulting data was completed with some AFM – SEM microscope observations that showed some interesting concepts about the structure of the samples. More details can be found in the host report.



---

Ana Inés Fernández  
DIOPMA Depto de ciencias de los materiales  
e Ingeniería Metalúrgica Universidad de Barcelona