Image Credit: Lourdes Rodríguez, Communications Department, PUCMM.

LVEM 5 User Profile: Dr. Fabrice Piazza

We recently talked with **Dr. Fabrice Piazza**, Associate Professor at Pontificia Universidad Católica Madre y Maestra (PUCMM) in Santiago de los Caballeros, Dominican Republic, and Head of the Nanoscience Research Laboratory at the Santiago Campus. After receiving his Ph.D. in Physics in 2001 from the University Louis Pasteur, Strasbourg, France, he held Research Associate positions at the University of Cambridge (2001–2004) and at the University of Puerto Rico (2004–2006). At PUCMM, he founded the Functional Nanocarbon Materials Research Group, NANOCARBON, in 2007 and the Nanoscience Research Laboratory in 2009, and he continues to lead both since then. Dr. Piazza is a user of the Delong LVEM5, and has published work enabled by the low acceleration voltages of the instrument. The following interview has been edited for brevity and clarity.

Hi Dr. Piazza. I'm excited to learn more about you, your research, and how you've used the LVEM 5. To start, can you please tell our readers a little bit about yourself?

I am a physicist working in the field of nanocarbon material synthesis and characterization, with particular interest in the development of novel nanomaterials and new or improved synthesis, functionalization or integration processes. These materials are of great interest for a wide range of applications such as transistors, single photon emission, miniaturized biomedical devices, composites and hydrogen storage.

Can you share the story of what led to your purchase decision of an LVEM 5?

I pursued international collaboration for TEM at first because the Dominican Republic has many challenges to overcome to be ready for maintaining a conventional TEM running at 100 kV. I first learned about the existence of benchtop transmission electron microscopes (TEMs), in October, 2017 while visiting Dr. Marc Monthioux's group at CEMES-CNRS, Toulouse, France, to perform TEM experiments at 100 kV. I immediately searched the internet and found out about Delong instruments and LVEM models, and I was very excited.

I was invited for a demo on some of my samples by Delong America, at Pittcon Conference & Expo in Orlando in February 2018, and made the final decision at that moment. Of course, having an instrument available in our lab to analyze many samples daily is an enormous difference as far as scientific productivity is concerned, compared to previously sending selected samples abroad. The critical advantage of using such low accelerating voltage came after the purchase.

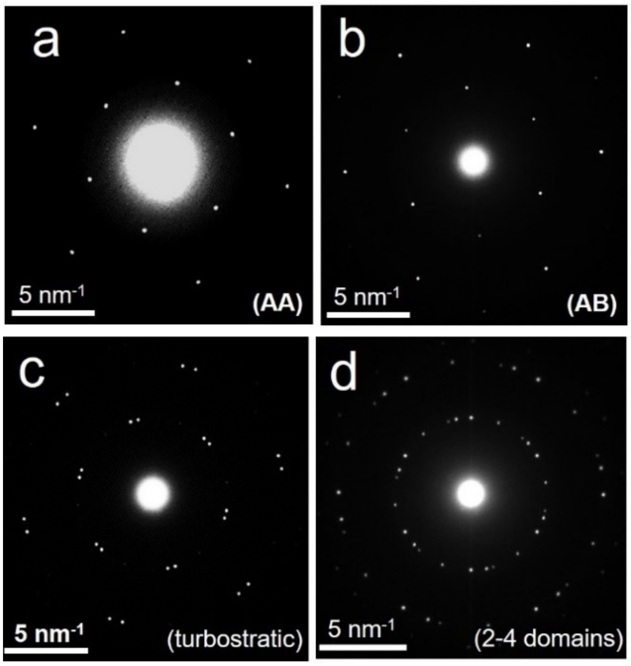
That's a wonderful journey. How did you raise the funding for the purchase?

We get a project funded in 2015 by the Ministry of Higher Education Science and Technology of the Dominican Republic, MESCyT. During the proposal review, we had to agree on cuts, including a sophisticated Fourier Transform Infrared (FTIR) microscope being purchased in a second phase of the project. After the second phase budget was reduced, in February 2018 I successfully asked for a modification of our instrument purchase strategy. By securing the purchase of a second hand FTIR microscope in Brazil, the savings enabled purchasing the LVEM5.

On March 1, 2019, we get our LVEM5 up and running in our laboratory, and within a few weeks later obtained results that are now published (Piazza, 2020; Piazza, 2021).

What are your favorite things about the instrument, now that you've owned it for some time?

First, the ease of training, use and operation. Of course, the possibility of quickly imaging at the micro- and nanoscale down to 1.2 nm resolution is great. And obtaining diffraction patterns from sample areas as small as 100 nm, provided the specimen is thin enough for being electron transparent at such a low electron energy (5 keV). The very low electron energy is another advantage, because our research is on highly hydrogenated 2D materials (graphene and diamane), which are quite electron sensitive. Quite recently, thanks to this low energy again, we discovered the possibility of discriminating between the various possible stacking sequences in bilayer and few-layer graphene (i.e. determining whether the graphenes involved are in A and/or B and/or C positions). This is something that electron diffraction using more common electron energy (60 keV and above) and much more expensive equipment cannot do alone.



Discriminating bilayer graphene with different stacking sequence is possible at very low accelerating voltages, a unique feature enabled by the LVEM5. (Piazza, 2021)

You shared three recent publications using LVEM, Piazza, et al., Carbon 169 (2020) 129–133, Piazza, et al., Carbon 156 (2020) 234–241, Piazza et al., C-Journal of Carbon Research, C. 2021 Jan 21; 7: 9, in which you work with a very interesting set of diamane materials. Can you briefly describe these materials?

Genuine diamane is a new member of the nanocarbon material family which includes fullerenes, nanodiamonds, carbon nanotubes and graphene. It consists of two crystalline sp^3–bonded carbon layers for which half of the carbon atoms are hydrogenated while the other half bond the two layers to each other. This is a wide band-gap semiconducting material, which is very attractive for nanoelectronics, nano-optics, quantum information processing, ultrathin protective coatings, composite materials, resonators and miniaturized electronics and biomedical devices. The material stability was computationally predicted in 2009. We are the first group to provide evidence of the successful synthesis of stable genuine diamane. Hot-filament-promoted hydrogenation process, at low pressure and at low temperature, is used to efficiently hydrogenate bilayer graphene and to subsequently convert them into stable diamane. Diamanoids are 2D wide band gap crystalline sp^3-bonded carbon materials as diamane, but they are composed of more than 2 layers. Their band gap is predicted to depend on the film thickness and crystalline structure.

Can you explain how the LVEM has helped in this work.

Genuine diamane can be produced by the chemisorption of hydrogen atoms on the "top" and "bottom" surfaces of bilayer graphene (2LG) and the subsequent interlayer bonding between sp^3-bonded carbon atoms, if the stacking sequence of the pristine 2LG is AA or AB. The number of layers can be increased, thereby generating that we called "diamanoids" with different properties (e.g., different gap values) if the stacking sequence is based on ABC or AAA. The use of graphene with the right number of layers and the right stacking sequence is therefore of the utmost importance when investigating genuine diamane and diamanoids.

Together with our collaborators in Toulouse, France, Dr. Pascal Puech and Dr. Marc Monthioux (CEMES-CNRS), and Dr. Iann Gerber (LPCNO-INSA), we have used the LVEM in its electron diffraction mode to determine both the number of layers and the stacking sequence of pristine 2LG films. Thanks to the use of an electron energy as low as 5 keV, we were able to obtain evidence, for the first time, of single 2LG domains made from either AA stacking, AB stacking, or from two randomly stacked single-layer graphene (1LG), otherwise designated as twisted 2LG. The 2LG-AB discriminates from 2LG-AA by the three-fold symmetry of the spot intensity distribution on the inner diffraction ring.

The 2LG-AA discriminates from 1LG by the significant lower intensity of the spot intensity on the outer ring. These differences are observable at 5 keV but not at 60–100 keV, as we confirmed by calculations. The reason is the following: As we are dealing with ultrathin crystallized materials (graphene, diamane, diamanoids), their images in the reciprocal space are made of reciprocal nodes which are elongated into rods in the z direction. For an electron energy of 100 keV, the Ewald sphere is large enough for its surface to be approximated as a plane. However, the lower the electron energy, the shorter the Ewald sphere radius, inducing the Ewald sphere surface to intersect the various reciprocal rods at different heights, thereby generating diffraction patterns exhibiting the same spots but with variable intensities, or even with some spots missing. Our work reveals that the distribution of spot intensities may vary dramatically with the electron energy. Our work was posted on ArXiv (https://arxiv.org/abs/2012.08869) and is currently under review. Our results will hopefully motivate the use of multiwavelength electron diffraction (definitely including energy as low as 5–10 keV) as a new tool for identifying stacking sequences in 2D materials.

That sounds like a wonderful advancement. Are there other ways you utilize the LVEM in your research?

We inspect materials to detect any metallic contamination and damages, such as cracks. Contrast variation is used to observe graphene grain size and shape, and to determine the number of layers. We also detect the presence of twisted bilayer graphene in diamanoid/graphene hybrid materials by using electron diffraction and imaging moirés in bright-field mode.

Where did you decide to place the instrument, in your lab or a core facility?

In our lab. This is what is great also with this piece of equipment: it is table-top and small enough to be put in a regular laboratory-like room.

How hard was site prep, and how did installation go?

Very easy. In our characterization room, we had some space remaining on top of an optical table equipped with an active isolation system where our Raman spectroscope system sits. Electrical connection to power backup was already available. Installation was very straightforward.

Is this a shared resource for other researchers at your institution?

As other researchers gain interest, it may become shared between various groups and even other institutions. Until then, we are analyzing micro- and nano-plastics for a project led by the PUCMM Nanobiology Lab.

How many users in your group (and in others if applicable) are trained on the instrument?

At the moment, I am the lucky and sole user but a new member of our group who recently received her Ph.D. will be trained during the next few weeks.

What is the most exciting moment you've had using the LVEM so far?

The most exciting moment was to find diffraction patterns of single bilayer graphene domain with AB stacking. The single bilayer graphene domain with AB stacking discriminates from AA counterpart by the three-fold symmetry of the spot intensity distribution on the inner ring of the diffraction patterns. This cannot be observed at 60–100 keV. Those observations confirmed the calculations of one of our collaborator at CEMES, Dr. Pascal Puech. Definitively, one of the greatest moments in my 22-year-long career.

What is something you would like to say to someone considering purchasing a LVEM?

Until now, we tended to think that the main reasons for purchasing a LVEM were related to relatively low cost of purchase, installation, maintenance and operation, and easy access. I say "relatively" because a lab such as mine, located in a country such as Dominican Republic, does not have access to the same amount of budget as laboratories from other Western countries for instance. Hence, the budget effort is higher for us, and cost was a main motivation for buying a LVEM instead of a regular TEM. But more and more, we have found that the advantages of using a LVEM go beyond cost issues. Indeed, by using LVEM to analyze 2D materials, in many cases, one can quickly obtain the number of layers and stacking sequence. Also, as we demonstrated the methodology is useful for materials other than graphene, such as transition metal dichalcogenides (TMD) which are nowadays very popular worldwide. Analyzing these materials in these ways is not possible using a conventional TEM operating at 60–100 keV.

Is there a piece of leadership advice you'd like to share for anyone pursuing a STEM career?

Identify your passion and go for it. Find a balance between your personal and professional life. Do not get frustrated if you cannot get a permanent position in a globally recognized institution. There are excellent alternatives to enjoy your job and to successfully carry out exciting research, for instance in some institutions which are very committed in increasing their research quality and production.

That is wonderful advice! Do you have any final thoughts you'd like to share?

Post-sale customer service support is of course of the utmost importance. We are very pleased to receive excellent post-sale support from Delong Instruments representatives, who have always answered very quickly to our questions, from Canada or the Czech Republic. Since March 2019, we have not experienced any serious technical difficulty with our LVEM5. In October 2020, after a 7-month lab shutdown, the microscope was slightly misaligned. However, we easily recovered the alignment by following the instructions in the manual, which is very well written.

That is wonderful to hear! Thank you Dr. Piazza for taking the time to share your stories about your exciting research and how the LVEM 5 is a powerful enabling tool.

References:

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About the author:

Robert I. MacCuspie, Ph.D., has over twenty years of experience in nanotechnology and materials characterization. Career highlights include leading the team that developed the silver nanoparticle reference materials at the National Institute of Standards and Technology, the first faculty and Director of Nanotechnology and Multifunctional Materials Program at Florida Polytechnic University, and over five years of consulting at the business-science interface from MacCuspie Innovations, helping companies commercialize and educate on technologies to improve human health.