

Mildred S. Dresselhaus (1930–2017)

Ado Jorio* 

This paper is an obituary in honor to Professor Mildred S. Dresselhaus, who passed away on 20 February 2017, letting behind a memorable contribution to the field of Raman spectroscopy applied to carbon materials. With 8 figures and 60 selected references, this text shortly overviews her contribution on paving the basis of our knowledge on graphite, graphite intercalated compounds, graphite ion-implantation, graphite fibers, fullerenes, carbon nanotubes, and two-dimensional systems, starting from graphene. Copyright © 2017 John Wiley & Sons, Ltd.

Raman spectroscopy lost one of its greatest contributors, Mildred Spiewak Dresselhaus, or just 'Millie' (Fig. 1). Institute Professor in the departments of Electrical Engineering and Physics at Massachusetts Institute of Technology (MIT), Millie passed away on 20 February 2017, at the age of 86. She co-authored about 1700 scientific papers, with at least 350 of those contributions carrying the word 'Raman' in the title, plus many others where the technique appears in the text, always applied to materials science, mostly focused on the new forms of graphene-related materials.

Millie and collaborators had significant contribution to pave the field of Raman spectroscopy applied to graphene-related materials: (1970s) graphite and graphite intercalated compounds (GICs), (1980s) graphite ion-implantation and fibers, (1990s) fullerenes, (2000s) carbon nanotubes, and (2010s) two-dimensional systems.

Millie's work on graphene-related materials started in the 1960, her early carrier, pioneering magneto-optic measurements to establish the proper identification of electron and hole states in the graphite Brillouin zone.^[1–3] She used to say that at that time of her career, it was very important not to work on crowded fields.

Millie's first contributions using Raman spectroscopy, in the 1970, addressed other materials,^[4,5] but graphite came soon after these initial works and paved the foundations of GICs (Fig. 2).^[6–12] Ion intercalation is a process where the intercalating atoms enter between every other graphene layers in graphite (stage 1), or every second layer (stage 2), every third (stage 3), and so on. It is a relatively mild interference in the graphene sp^2 -carbon structure, generating mostly charge transfer between graphene and ions. The use of Raman spectroscopy to characterize stage levels in GICs significantly impacted a field that is today the basis of lithium-ion batteries. One can also say that GICs triggered the study of truly two-dimensional materials, deepened later by the isolation of single-layer graphene.

In the 1980, Millie and collaborators moved Raman spectroscopy toward a stronger interference in the structure of the graphene sheet, by studying highly oriented pyrolytic graphite subjected to ion implantation (Fig. 3).^[13–15] Using different ions (Li, Be, B, C, P, As, among others) and ion fluencies, the foundations for the use of Raman spectroscopy to analyze amorphization of graphene-related materials were placed. These works discussed the defect-induced Raman band phenomena and the possibility of restoration of the graphite ordering by thermal annealing. The research in sp^2 -bonded carbons was consequently impacted in a very broad sense, including quality assessment of graphite fibers in the early

times,^[16–20] graphene growth, and the characterization of amorphous carbon in general,^[21,22] for example highly pure diamond-like carbon, utilized in surface technology; carbon black, used as pigment and reinforcing phase in automobile tires; biochar, for soil amelioration; among others.

Raman contribution in graphitic carbons kept growing and significantly impacted the field of nanoscale structures, starting in the 1990s with the emergence of fullerenes.^[23–28] A discovery that led to the Nobel Prize in chemistry in 1996, fullerenes are molecular structures formed by 60, 70, or as many possible C atoms, given that 12 pentagons are present in the graphene network to close a cage-like structure. They can be packed in different crystallographic structures to design three-dimensional solids. Such structures could carry doping atoms between cages, inside the cage, or even replacing C atoms in a cage wall, providing building blocks for molecular solids with a wide range of different properties, including unusually high T_C superconductors. Raman spectroscopy was crucial for understanding the very rich set of structural arrangements that were made possible with this newly discovered carbon allotrope and to study their properties (Fig. 4).^[23–28] Millie and collaborators were unquestionably among the leading scientists in this field.

Fullerenes inspired the discovery of one of the most intensively studied carbon materials, the carbon nanotubes. A carbon nanotube is a hollow cylinder made from adding more and more carbons to extend the fullerene into a one-dimensional structure. Different atomic arrangements can generate tubes of different diameters and chiral angles; carbon nanotubes can be single-wall, double-wall, or multi-wall; they can exist isolated, in solution, in the solid form; the carbon nanotube-based materials can be doped with atoms inside the tubes, between tubes in a bundle, or inserted in the tube walls. Millie's work on the Raman spectroscopy of carbon nanotube started in the early 1990 but achieved significant impact in the new century,^[29–45] when Millie's contribution to Raman spectroscopy boomed (Fig. 5(a)) due to the richness of the

* Correspondence to: Ado Jorio, Departamento de Física, Universidade Federal de Minas Gerais. Av. Antônio Carlos, 6627, Campus Pampulha, Belo Horizonte, MG, Brazil, 31.270-901.
E-mail: adojorio@fisica.ufmg.br

Departamento de Física, Universidade Federal de Minas Gerais (UFMG), Belo Horizonte, MG, BRAZIL, 31.270-901



Figure 1. (top left) Millie setting a magneto-optic experiment at the MIT Lincoln Labs. (bottom left) Millie teaching Raman spectroscopy at MIT (courtesy from MIT). (right) Millie looking at a carbon nanotube sample in the micro-Raman laboratory at Universidade Federal de Minas Gerais (UFMG), Brazil (courtesy from M. A. Pimenta).

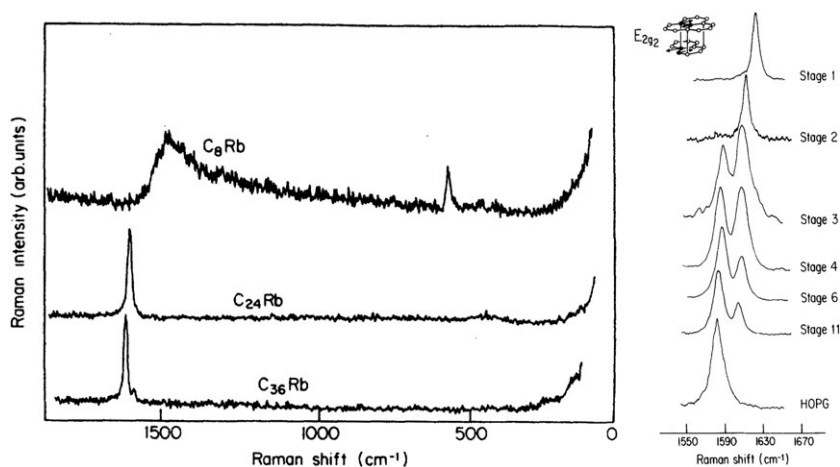


Figure 2. (left) Raman spectra of C_8Rb (stage 1), $C_{24}Rb$ (stage 2), and $C_{36}Rb$ (stage 3) graphite intercalated compounds. Reproduced from Phys. Rev. B 16, 1977, 3330.^[7] (Right) Raman spectra for stage n ($n = 1, 2, 3, 4, 6,$ and 11) graphite ferric chloride compounds and for pristine graphite [highly oriented pyrolytic graphite (HOPG)]. The upper wavenumber component is identified with the bounding layer mode and the lower component with the interior graphite mode. Reproduced from Solid State Comm. 29, 1979, 769.^[10] It was possible to differentiate the intercalation stages, allowing the development of modeling the atomic and electronic structure of graphite intercalated compounds.

nanotube photophysics. It is relevant to mention that in 2000, Millie held the position of Secretary of the US Department of Energy. She would spend the week in Washington and come to MIT every Saturday and Sunday. She held many of those service positions during her career, but she never lost the focus on science.

Raman spectroscopy was shown to depend significantly on the excitation laser energy (Fig. 5(b)), when measuring the recently found one-dimensional carbon nanotubes, demonstrating that their rich electronic structure generates interesting resonance effects.^[30,31] Those resonances were sufficient to allow single-nanotube Raman spectroscopy (Fig. 6),^[34] an observation that significantly impacted the field of molecular spectroscopy in the early 2000s. It was amazing that the Raman intensities observed

from single isolated carbon nanotubes were equivalent to the Raman intensity of the Si substrate where they were lying, despite the fact that there were million more Si atoms under illumination. This is all due to the great resonance effect in the one-dimensional carbon nanotubes, allowing in depth study of the rich optical phenomena, which depends on specific carbon nanotube structure. Resonance Raman spectroscopy made it possible to study how carbon nanotube properties change for different tube structures, functionalizations, and environmental conditions, important for the different applications. Again, Millie and collaborators were unquestionably among the leading scientists in this field.

When graphene appeared in 2004, the field was mature to fully address all the wonders that were made possible grateful to the

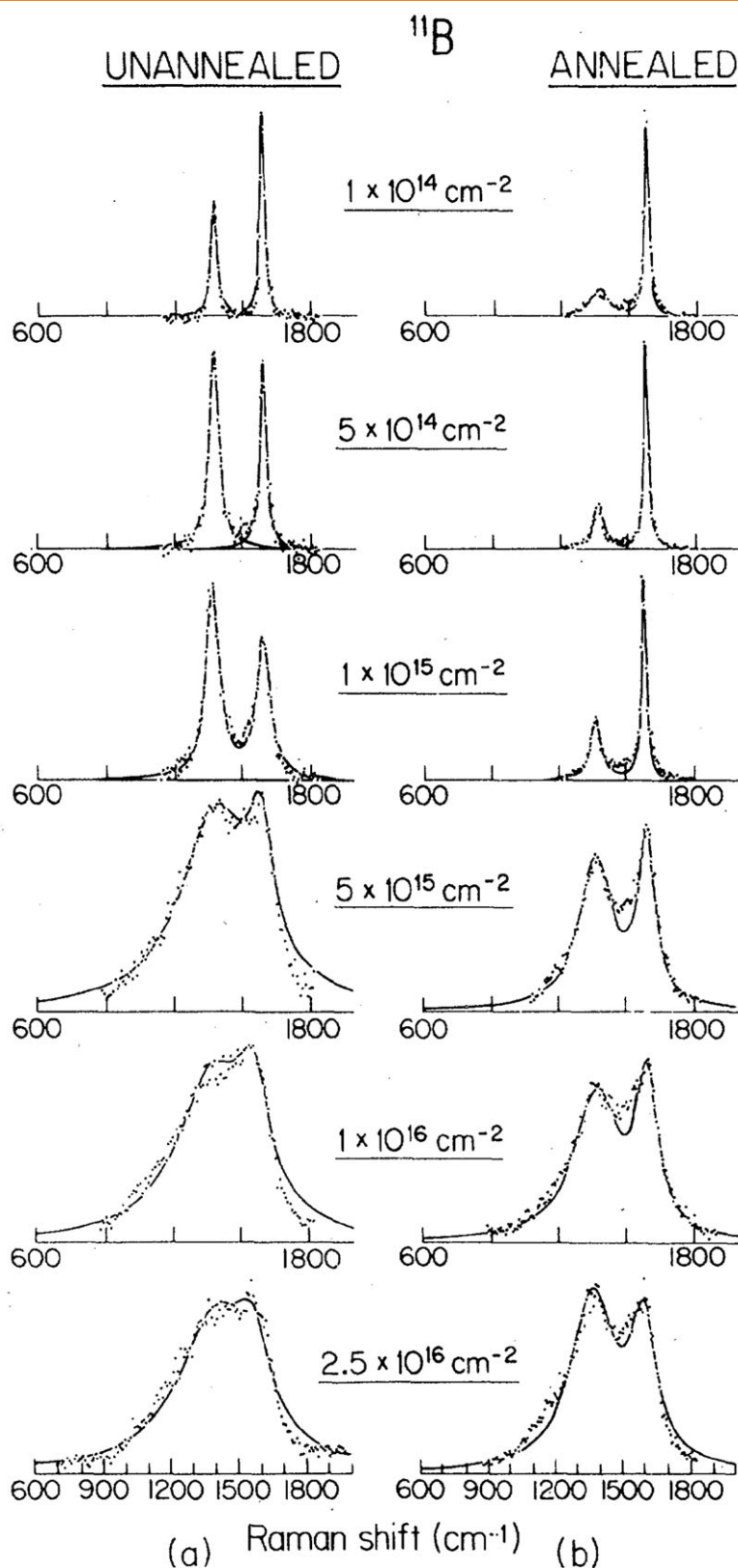


Figure 3. Raman spectra for various ^{11}B fluencies of ions implanted into six HOPG samples. The spectra on the left are for the unannealed samples, and on the right are for the same samples annealed at 950°C for 0.5 h. Pristine graphite exhibits a relatively sharp peak at 1584 cm^{-1} (G band), and the break of the sp^2 bonds activates a defect-induced peak at 1350 cm^{-1} (D band). The more defects on the lattice, the larger the D band intensity and the wider the D and G peaks. Reproduced from Phys. Rev. B 24, 1981, 1027. ^[13]

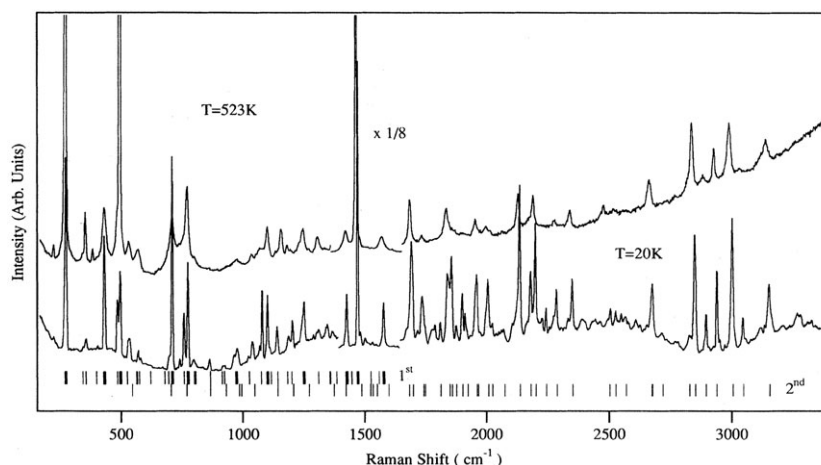


Figure 4. Raman spectra for solid C_{60} films (7000 Å thick) taken at temperatures $T = 523$ K and 20 K. The mode frequencies shown schematically in the figure indicate theoretically predicted Raman-allowed modes (darker ticks), overtones, and combinations (lower, marked second). This type of analysis was broadly used to access the structural composition of fullerene-based materials. Reproduced from Phys. Rev. B 48, 1993, 2862.^[26]

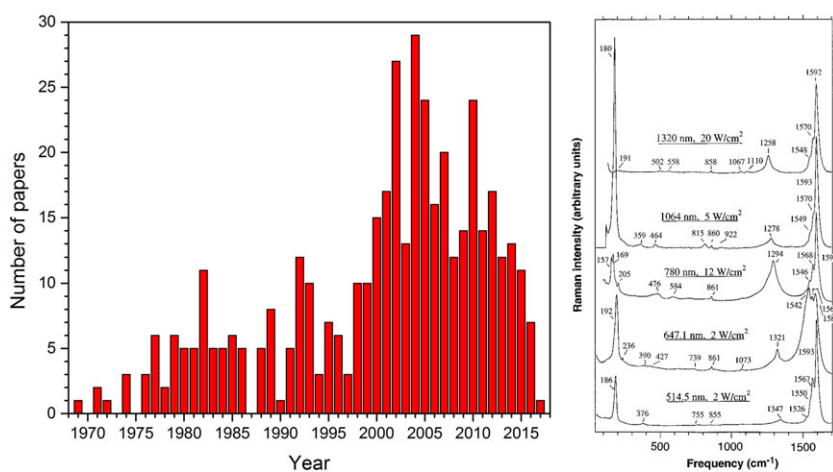


Figure 5. (left) Number of papers authored by Dresselhaus, using the word ‘Raman’ as searching word (Scopus, 28 April 2017). (Right) Raman spectra of carbon nanotubes measured with different excitation laser energies, published in 1997, the year when intensive Raman research on carbon nanotubes began. Reproduced from Science 275, 1997, 187.^[30] Besides the usually known D and G bands, carbon nanotubes exhibit a strong contribution at lower frequencies (100–200 cm^{-1}), the so-called radial breathing modes (RBMs). By changing the excitation energy, different carbon nanotubes are in resonance with the laser, thus dominating the spectra. This explains the differences in peak frequencies and lineshapes observed.

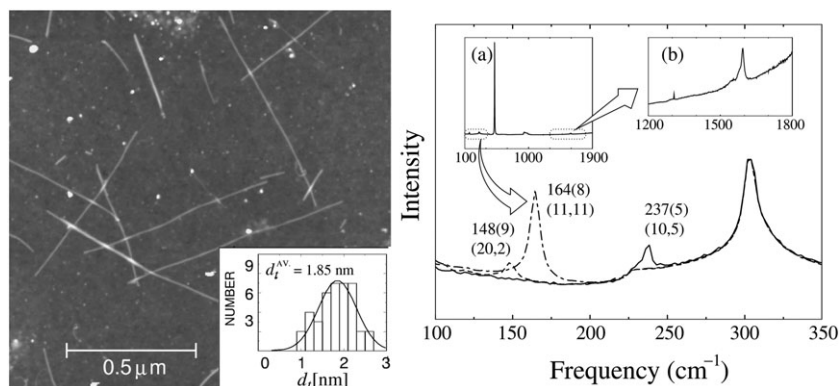


Figure 6. (left) Atomic force microscopy image of isolated single-walled carbon nanotubes (SWNTs) deposited on a Si substrate. The inset is the SWNT diameter distribution. (Right) Raman spectra from three different spots on the Si substrate, showing only one resonant nanotube and one RBM wavenumber for each of three spots. The RBM wavenumber (width) and (n,m) assignment for each resonant SWNT is displayed. The 303 cm^{-1} feature comes from the Si substrate (a), and a zoom of the G-band region (b). Reproduced from Phys. Rev. Lett. 86, 2001, 1118.^[34]

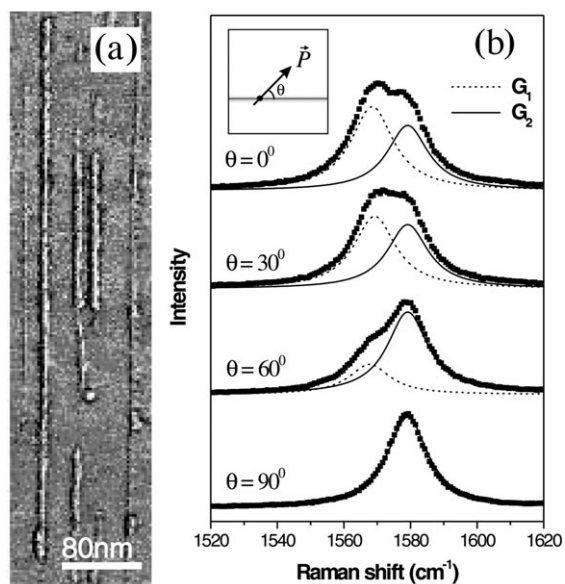


Figure 7. (a) Atomic force microscopy image of graphene ribbons parallel to each other. (b) Raman spectra obtained for light incident with different polarization angles (θ) with respect to the ribbon direction (see inset). Reproduced from *Phys. Rev. Lett.*, 93, 2004, 047403.^[46] Peaks G_1 and G_2 come from the graphene ribbon and HOPG substrate, respectively, as evidenced by the polarization dependence.

discovery of graphene isolation.^[46–50] Already in 2004, Millie and collaborators had published the measurement of the Raman spectra of a single graphene-ribbon grown on graphite (Fig. 7).^[46] Like for carbon nanotubes, the signals from isolated graphene ribbons were equivalent to the signal of the graphite substrate. These resonance effects allowed single graphene Raman spectroscopy, which became the preferable technique for graphene characterization.

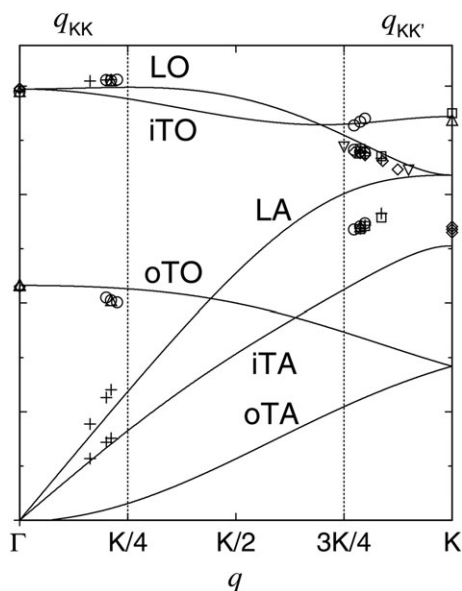


Figure 8. The six graphite phonon dispersion curves (lines) and experimental Raman frequencies (symbols) placed in the interior of the phonon dispersion according to the double resonance theory. Reproduced from *Phys. Rev. Lett.* 88, 2001, 027401.^[55] This work triggered a significant advance on the theoretical understanding necessary to properly describe the vibrational properties of carbon atoms in graphene-related materials.

Besides the contributions to graphite, GICs, graphite fibers, fullerenes, carbon nanotubes, and graphene, Millie also utilized Raman spectroscopy to reveal other intriguing carbon nanostructures, such as activated carbon aerogels^[51] and linear carbon chains,^[52] to advance novel Raman techniques, such as fast optics,^[53] and novel graphene-related phenomena, such as graphene-enhanced Raman spectroscopy.^[54] A very unusual aspect of Raman spectroscopy applied to graphene-related materials is the possibility to access the interior of the Brillouin Zone, measuring the phonon dispersion relation with unprecedented accuracy (Fig. 8).^[55] Millie and collaborators demonstrated that internal resonances in electron–phonon scattering involving different phonons make it possible. The phonon dispersion relation of graphite was unsolved until the discovery of this phenomenon, and large advances came from this novel understanding, including the possibility of measuring, with Raman spectroscopy, changes in the material's Fermi level under doping. Millie was awarded with the Kavly prize in 2012 'for her pioneering contributions to the study of phonons, electron-phonon interactions, and thermal transport in nanostructures'.

The rise of graphene generated, from the years 2010 onward, a broad interest on lamellar two-dimensional systems, including transition metal dichalcogenides and graphene-like structures made of different atoms, such as phosphorene. Millie rapidly moved into these new materials and made important contributions using Raman spectroscopy.^[56–60] She used to advise fast moves toward new materials, and she left us with clear perspectives for new directions in the Raman spectroscopy of materials science.

Besides the outstanding scientific contribution, Millie has earned undeniable respect for her willingness to help people, including the defense of minorities, women in science, and science in developing countries. In my 17 years of collaboration with Millie, only once I saw Millie's eyes wet, when I asked why Raman spectroscopy was so special for her. She said that it was special because it was discovered in India, despite so many richer and scientifically more powerful nations. She was an example of scientist and human being, having enriched us with science and inspiration.

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