Type of presentation: Oral

IT-6-O-2397 Raman Spectroscopy coupled with environmental scanning transmission electron microscope

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In recent years the environmental transmission scanning electron microscope (ESTEM), has been successfully employed to reveal and understand the structural and chemical changes occurring in the nanoparticles under reactive environments [1,2]. The lack of statistical information available from TEM measurements is generally balanced by using other, ensemble measurement techniques such as x-ray or neutron diffraction, x-ray photoelectron spectroscopy, infrared spectroscopy, Raman spectroscopy etc. However, it is almost impossible to create identical experimental conditions in two separate instruments to make measurements that can be directly compared. Moreover, ambiguities in ESTEM studies may arise from the unknown effects of the incident electron beam and uncertainty of the sample temperature. Here, we present a unique platform that allows us to concurrently measure atomic-scale and micro-scale changes occurring in samples subjected to same reactive environmental conditions by incorporating a Raman Spectrometer on the ESTEM.

We use a parabolic mirror, attached at the end of a hollow rod that can be inserted between the sample holder and the lower pole piece of the microscope (Fig. 1-2a). The mirror focuses the incoming laser on the sample and collects the scattered Raman photons. A set of optics then carries the Raman signal up to the spectrometer. Fig. 2.b,c show the Raman D and G band as well as the radial breathing modes of single walled carbon nanotubes (SWCNT) formed in the ETEM and an atomic-resolution still image extracted from a video sequence, respectively. We can monitor the growth rates using the G-band intensity under different growth conditions (Fig. 2d). This versatile optical setup can also be used i) to measure the temperature using Raman shifts, ii) to investigate light/matter interactions iii) as a heating source, iii) for general spectroscopy such as cathodoluminescence. Details of the design, function, and capabilities will be illustrated with results obtained from experiments on the *in situ* synthesis of carbon nanotubes.

Reference:

[1] Sharma, R., J. Mat. Res. 2005, 20, 1695

[2] Hansen et al., Science 2001, 294, 1508

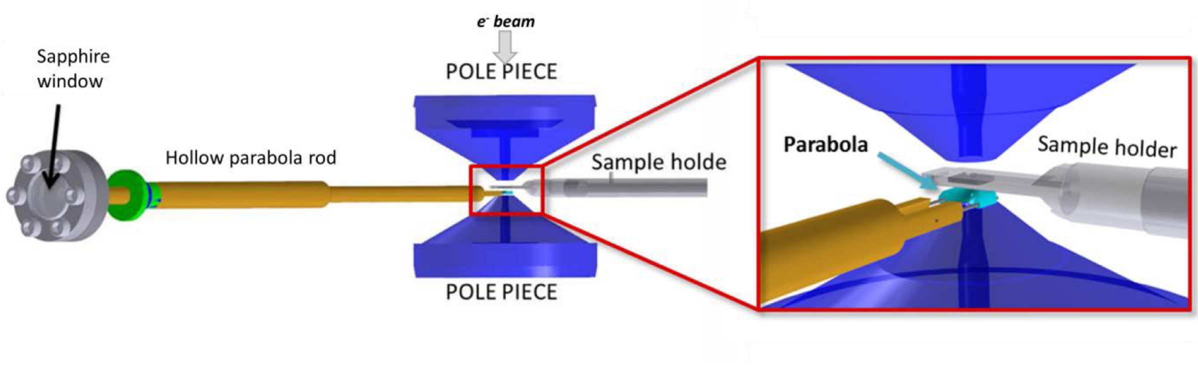

Fig. 1: Schematic representation of the Raman data collection system: the laser passes through the hollow parabola holder, and is then focused on the sample by the parabola. The parabola collects the Raman signal and directs is back to the spectrometer.

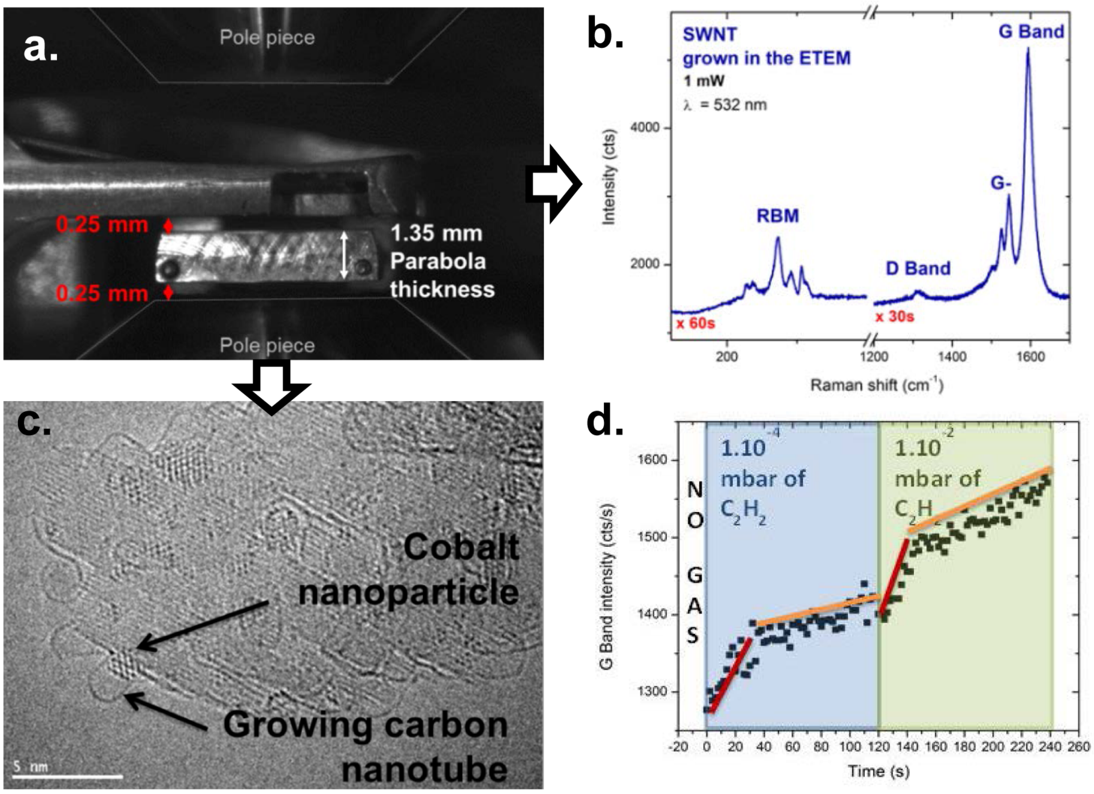

Fig. 2: (a) Location of the parabolic mirror that collects the Raman signal, is located between the sample holder and the lower pole piece. (b) Raman spectrum collected from SWCNTs grown in the ESTEM. (c) Atomic Resolution image showing as grown SWCNTs (d) Time resolved evolution of the G band intensity (SWNT growth rate) at 650 °C under two C_2H_2 pressure.