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High Resolution *In Situ* and Transmission Environmental Electron Microscopy of Material Reactions

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There has been a steady growth in the applications and breadth of *in situ* transmission electron microscopy (TEM) since the 1980's [1]. At that time, the procedures to carry out meaningful experiments were described (e.g. [2]) but it was thought that high voltage TEM and thick specimens were required to reproduce bulk behavior. However, in a series of studies, we established that this was not necessarily the case and that even high resolution TEM recordings could be made in real time, *in situ* and that the atomic behavior associated with materials reactions at interfaces could be deduced (e.g. [3, 4]). Moreover, with the advent of thin film and nanotechnology, the investigation of thin and nano-scale materials became a necessity (e.g. [5]). In recent years, there has been an additional proliferation, most notably from *in situ* TEM in controlled environments such as in gases and liquids (e.g. [1], [6]).

This paper reviews the application of *in situ* high resolution TEM to investigate material reactions. An overarching theme of our work has been to ensure that the *in situ* studies are truly representative of the real behavior of the material system, and we have advanced a number of guidelines to ensure this. [3 ,7] Moreover, we have also expanded our approach to environmental material-gas reactions such as carbon nanotube (CNT) oxidation [8], hydrogen reactions with molybdenum sulphide catalysts [9] (e.g. Fig.1), oxygen vacancy formation in ceria thin films [10] etc. The influence of the imaging electron beam is more important for the gaseous reactions, as the beam ionizes the reacting gas species, and it is necessary to develop protocols to take this into account, especially monitoring the electron beam dose and dose rate. In some cases this phenomenon can be used to good effect [11, 12]. The procedures we have adopted to do this will be described, [13].

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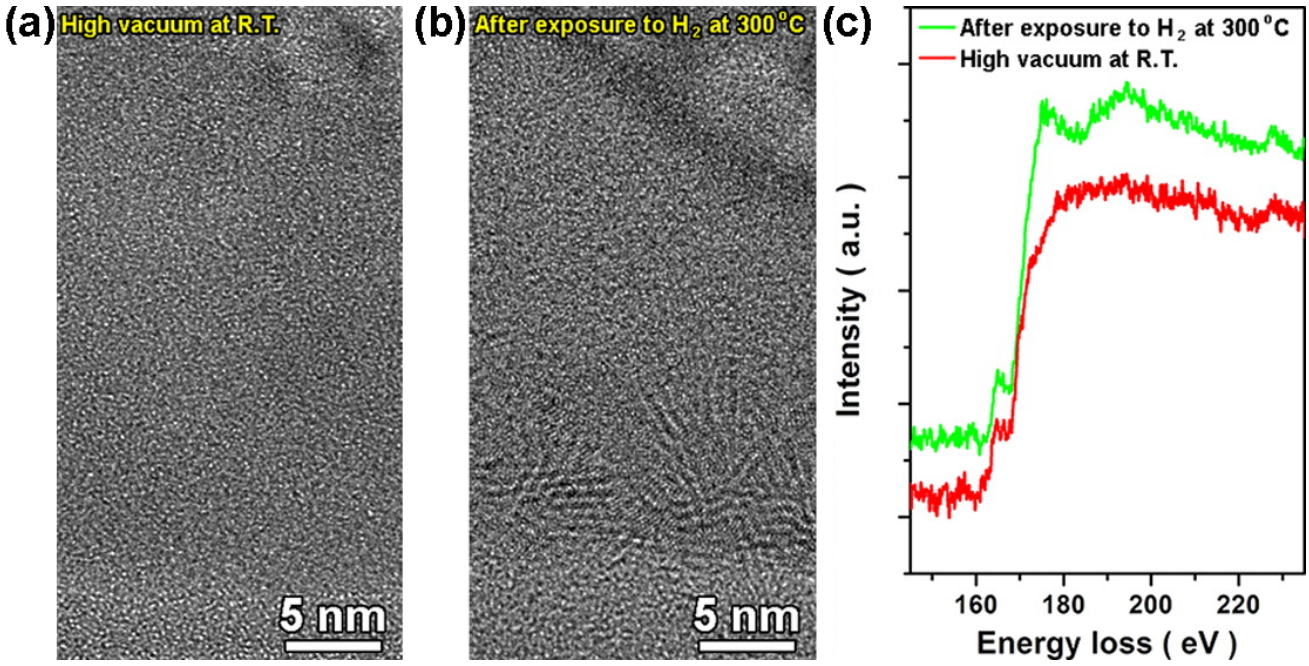

Figure 1. High resolution TEM images of amorphous molybdenum sulphide before (a) and after (b) hydrogenation in the environmental TEM, showing the formation of crystalline disulphide regions. (c) Electron energy loss spectra showing the characteristic sulphur edge for crystalline MoS_2 in the latter.

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