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Low-loss Electron Energy-loss Spectroscopy in 2-D Materials and Liquids

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Thermal management and heat dissipation is an important limitation towards faster and more compact electronic devices. Specifically, interfaces, defects and surfaces can act as heat traps and sources of local strain due to a mismatch in the thermal expansion coefficient, which will lead to device failure or the formation of temperature induced defects. While TEM provides an excellent probe to measuring the local atomic and electronic structures, there is no direct approach to determine the temperature on a similar length scale. To date, nanoscale thermometry is carried out either through scanning probe microscopy-based (SPM-based) techniques like scanning thermal microscopy or non-contact optical methods, such as Raman, fluorescence, and luminescence thermometry.

In this contribution, we utilize atomic-resolution imaging and electron spectroscopy in an aberration-corrected scanning transmission electron microscope (STEM) to characterize the thermal properties of 2D materials, including pristine and allowed transition-metal dichalcogenides, as well as graphene liquid cells [1] during in-situ heating and cooling experiments. We use the aberration-corrected JEOL ARM200CF at the University of Illinois at Chicago, equipped with a cold-field emission electron source and a Gatan Continuum GIF. In-situ experiments will be carried out using the Protochips Aduro and Gatan cooling stages, which provide the stability to perform atomic-resolution analysis in the temperature range of 90 K - 1000 K.[2] The experimental data is then compared to our first-principles modeling results.[3]

Figure 1 show a HAADF image of $Mo_{0.3}W_{0.7}S_2$, where the heavier W atoms can be clearly identified as the brighter image contrast. The shift of the plasmon peak is then measured as a function of temperature in these materials. The right panel in Figure 1 shows a summary of the plasmon energy shifts for different sample compositions and number of layers. The results of alloyed $Mo_{1-x}W_xS_2$ are compared with pure MoS_2 and WS_2. We find that the measured plasmon peak shifts of the alloys are smaller than the range of values measured for MoS_2 and WS_2. This observation runs counter to the empirical Vegard's law, which is frequently followed by alloys, where a measured property of the alloy is a weighted average of the property of the end members.

While measuring the temperature and thermal expansion coefficient of solid state materials has become routine, such approaches have not yet be explored for liquids, especially in in-situ cells. In conventional SiN liquid cell, the plasmon peak of pure water can be identified, yet the presence of amorphous carbon ($E_{plasmon}$=22 eV) on the window layers poses a challenge to precisely track the plasmon peak of water at ~25 eV. Here, graphene liquid cells, which can be easily integrated with most in-situ heating and cooling stages, offer an ideal setup where the plasmon peak of water can be directly measured without much background from either carbon or SiN. Figure 2 shows initial data for in-situ cooling of graphene liquid cells, where a clear shift in the plasmon peak position is visible upon cooling from room temperature to LN_2. We will present a more complete dataset and analysis of the effects pf phase transitions on the plasmon peak, and examine the validity of this approach to measuring the temperature in in-situ liquid cells.[5]

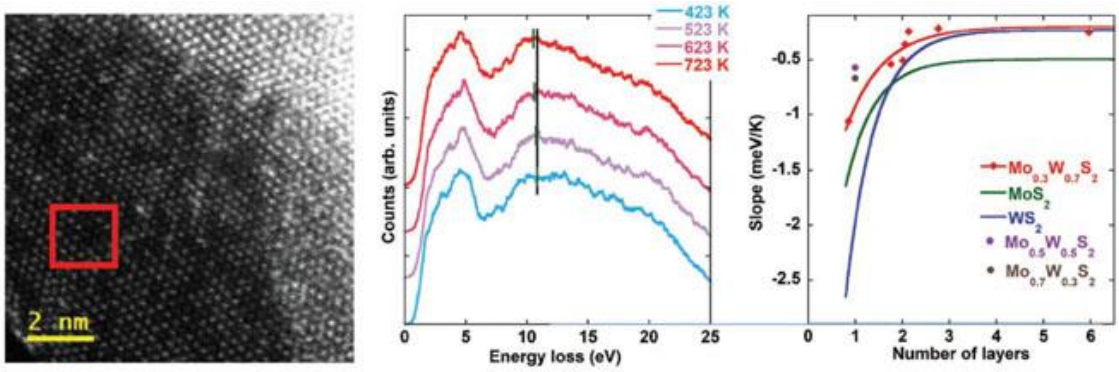

Figure 1. Left: HAADF image of $Mo_{0.3}W_{0.7}S_2$ and the corresponding low-loss EELS recorded in the red rectangle area at different temperatures (center). Right: The shift of the plasmon peak energy as a function of $Mo_{1-x}W_xS_2$ layers for $0.3 \leq x \leq 0.3$. The two endmembers are also plotted. Taken from [4].

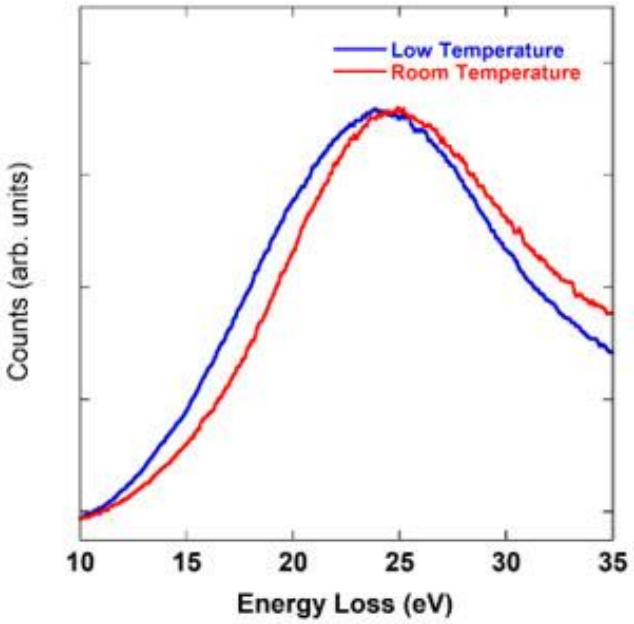

Figure 2. Plasmon peak EELS of H_2O in a graphene liquid cell as a function of temperature. A clear shift toward low energy is seen upon cooling the sample to 90 K from room temperature.

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