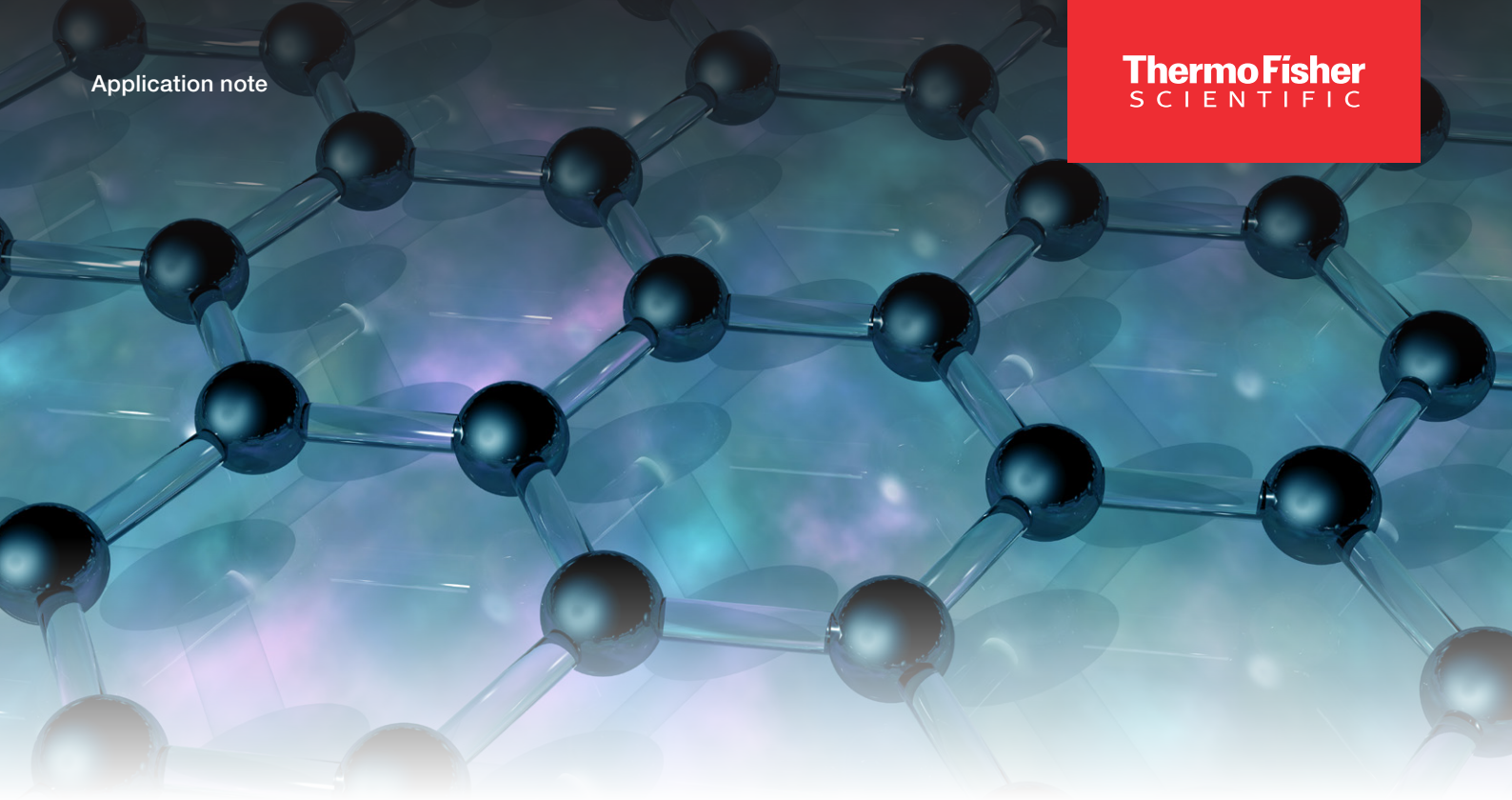
Application note



Scanning electron microscopy in a glovebox

Pioneering research of unstable 2D materials

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Innovative materials research at SuSTech

Two-dimensional materials are often unstable due to their high surface-to-volume ratios, which makes them highly sensitive to environmental factors such as moisture, oxygen, and contaminants. This sensitivity can cause them to degrade or undergo chemical changes under ambient conditions. To address these challenges, Professor Lin Junhao's team at Southern University of Science and Technology (SuSTech) has developed a unique Glovebox Interconnected System (GIS) for the study of these materials. By performing all their work in a contiguous, encapsulated, and inert environment, they are able to preserve their sensitive 2D materials through numerous analyses.

The benefits of an interconnected glovebox system

Typically, when sensitive 2D materials are analyzed, they have to be moved between multiple gloveboxes through an ambient environment, which can lead to contamination. GIS changes this by bringing all the necessary equipment into one controlled space. This seamless integration means that the entire process from material growth to preliminary characterization, modification, and transfer to high-resolution characterization can be performed without the sample exiting the inert glovebox environment. Analysis can include both TEM characterization as well as state-of-the-art techniques such as STM, MOKE, and PPMS, allowing the researchers to perform a range of high-resolution and ultra-clean structural characterization.

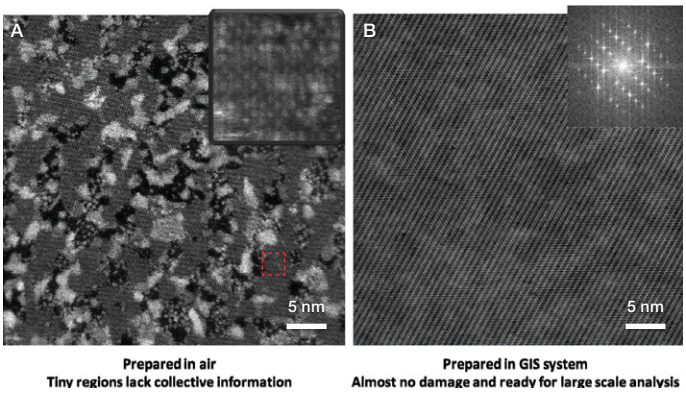

Figure 2. Scanning transmission electron microscopy images of a tungsten ditelluride (WTe_2) monolayer produced with chemical vapor deposition. When exposed to air, the sample shows extensive damage that is not present in the GIS-prepared sample.[1]

The role of the Phenom Pharos Desktop SEM

One of the key components of the GIS setup is the Thermo Scientific™ Phenom™ Pharos Desktop Field Emission Scanning Electron Microscope (FEG SEM), which provides a resolution of 2.0 nm at 20 kV, along with integrated energy-dispersive X-ray spectroscopy (EDS). It is the first of its kind to be housed directly in a glovebox. The Phenom Pharos Desktop SEM quickly captures images, inspects the surface, and analyzes the composition of the 2D materials right after they are created. Its compact design is perfect for the confined spaces of a glovebox, and its anti-vibration and insert-sample holder features make it easy to use and stable to run. This is quite crucial when screening samples produced with chemical vapor deposition (CVD), which often contain multiple unknown phases of the target material. The Lin team also developed an electron-beam evaporator that can be connected with the GIS for electron-beam lithography. Using the Phenom Programing Interface (PPI) of the Phenom Pharos Desktop SEM, they could perform the entire nano-device fabrication process within the GIS, without air exposure.

Figure 3. The Thermo Scientific Phenom Pharos Desktop SEM in the GIS.

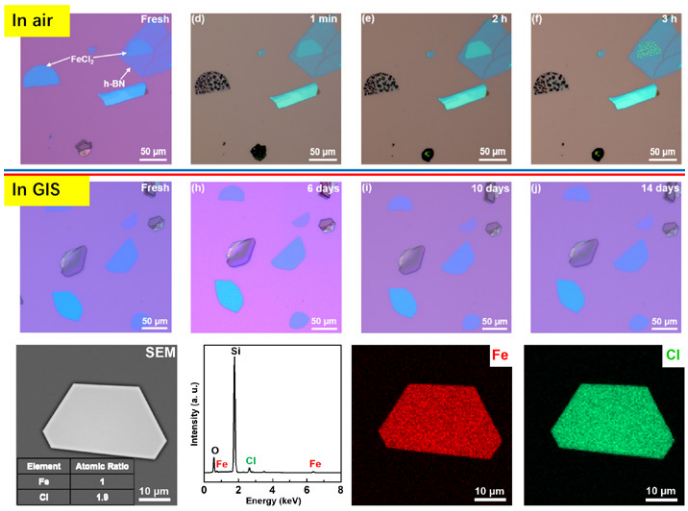

Figure 4. Extremely sensitive CVD-grown ferrous chloride ($FeCl_2$) flakes characterized with the Phenom Pharos Desktop SEM within the GIS. Once exposed to air, the material deliquesced in less than 10 seconds.[2]

Applications of the GIS for 2D materials analysis

Figure 4 shows ferrous chloride ($FeCl_2$), a sensitive 2D magnetic transition metal dihalide. It was prepared through the reduction of trihalide $FeCl_3$, and subsequently preserved intact for extended periods in the nitrogen atmosphere of the GIS. The final row of images, captured using the Phenom Pharos Desktop SEM, shows both SEM images and EDS maps of the ferrous chloride. A uniform elemental distribution of iron (red) and chlorine (green) is seen, with a Fe/Cl atomic ratio of 1:1.9. This serves as a critical indicator of successful 2D $FeCl_2$ flake synthesis by $FeCl_3$ reduction. Lin's team notes that only the GIS-SEM can effectively handle such delicate materials.[2]

Enhance your 2D materials research

Integration of a scanning electron microscope, like the Phenom Pharos Desktop SEM, directly into a glovebox environment is a significant innovation in the study of unstable materials. For a deeper understanding of GIS and potential collaborations, visit Professor Lin's team website.

References

1. Niu, K, *et al.* Direct Visualization of Large-Scale Intrinsic Atomic Lattice Structure and Its Collective Anisotropy in Air-Sensitive Monolayer 1T'-WTe_2. *Advanced Science* 8:20, 2101563. doi: 10.1002/advs.202101563
2. Jiang, S, *et al.* General Synthesis of 2D Magnetic Transition Metal Dihalides via Trihalide Reduction. ACS Nano 17:1, p363-371 (2023). doi: 10.1021/acsnano.2c08693

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