

## Innovative Air Protection Sample Holder for Ion Milling-SEM-SPM and Shared-Alignment Sample Holder for SEM-SPM

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Global issues concerning energy efficiency and the scarcity of natural resources have led to a trend towards the device miniaturization and material substitution to reduce material exploitation. Thus, these issues have prompted the need to invent high-performance materials and control the quality of materials with nanostructures and nanoproperties. As a result, the field of material science has gained more importance.

We developed an innovative air protection sample holder system for Hitachi's environment control high-vacuum Scanning Probe Microscope (SPM) unit AFM5300E to enable hermetically sealed sample transfer from the Ion Milling IM4000 and Field Emission Scanning Electron Microscopes (FE-SEM) to the SPM unit. In a previous study, we explained the advantages of this sample holder with regard to the analysis of cathode materials in a lithium-ion battery (Fig. 1) [1]. In this study, we explain the necessity to link all three systems using the hot-deformed Dy-free Nd<sub>2</sub>Fe<sub>14</sub>B magnet as a sample application [2]. After the Flatmilling<sup>®</sup> process, the topography and the magnetic force microscopy (MFM) signals of the c-plane surface of the magnet were observed with SPM AFM5300E. These images were overlaid and analyzed with the secondary electron (SE) and the backscattered electron (BSE) images of the same area. Using the SE-MFM overlay image, every crystal grain was classified into single or multi-domain magnetic grains. Furthermore, the overlay of the BSE with the MFM image allows us to correlate the element contrast distribution with magnetic properties (Fig. 3).

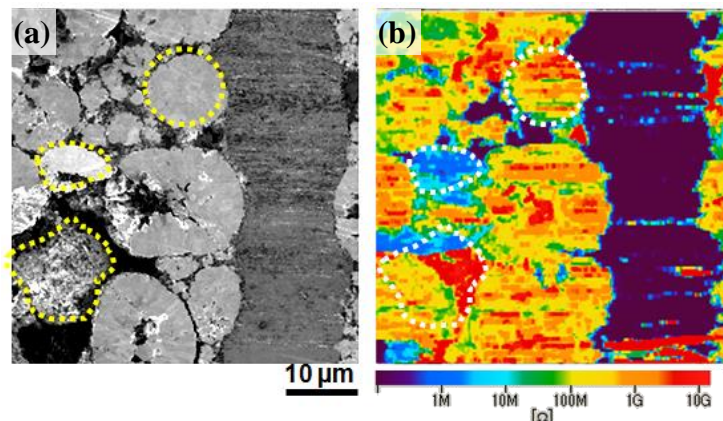
Our newly developed SEM-SPM linkage system with the shared-alignment sample holder enables a software-based alignment of the same measurement area for observations with the FE-SEM and AFM5500M. In this study, we used this linkage technology to analyze multilayer graphene on a SiO<sub>2</sub> substrate. We concluded from the topography, Kelvin Force Microscopy (KFM) image data, and the SE image data that the contrast in the SE image corresponds to the layer thickness and surface potential of graphene (Fig. 4).

As described above, both technologies allow a correlative observation with Hitachi's SPM and SEM to obtain more sample-specific properties that are important for the research and development of future nanomaterials with nanoproperties.

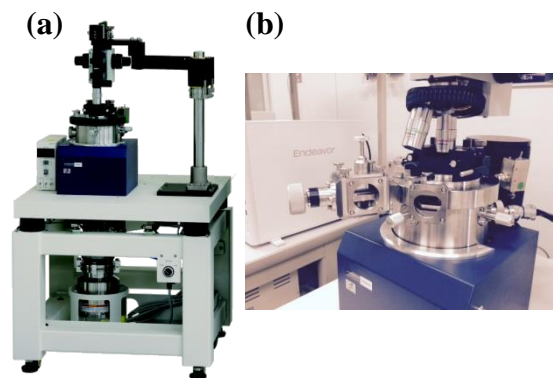
### References:

[1] T. Yamaoka *et al.*, The 34<sup>th</sup> Annual NANO Testing Symposium, **3** (2014), p.13-18.

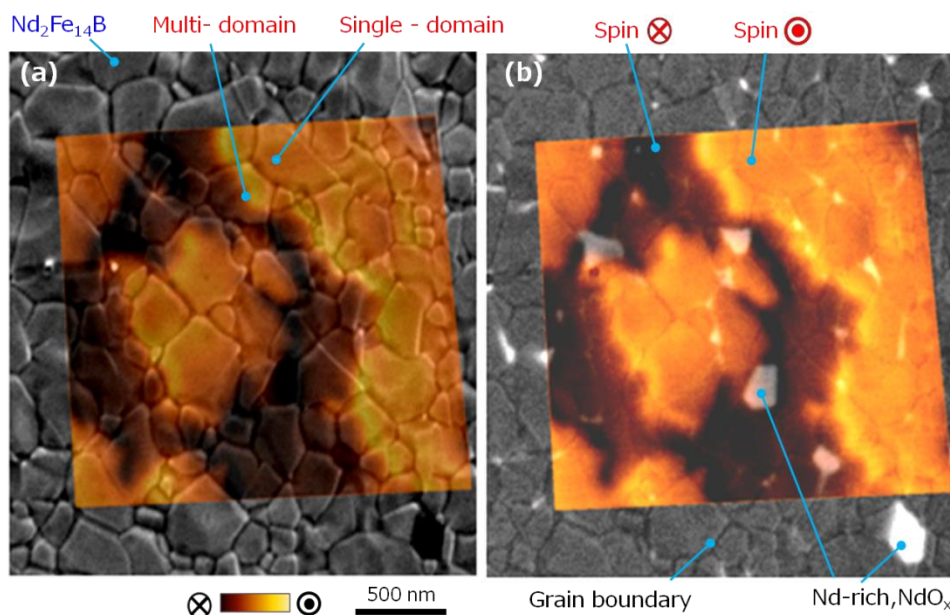
[2] T. Yamaoka *et al.*, Microscopy Today **22** (2014), p.12-16.



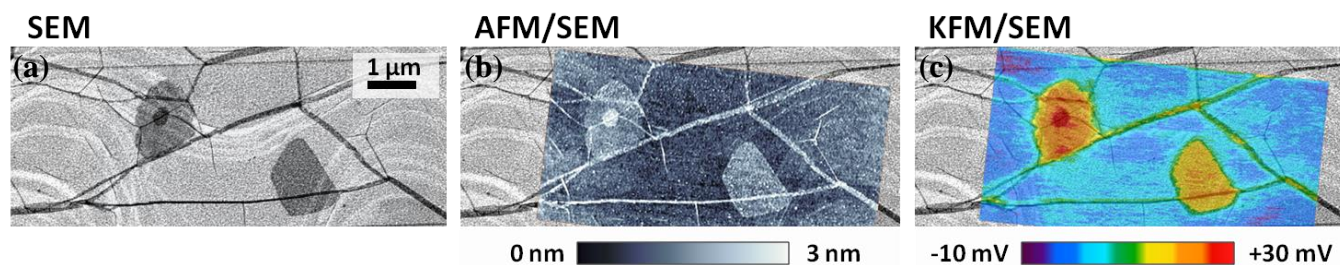
**Figure 1.** Ion-milled cross-section of lithium ion battery cathode materials: (a) SE image, (b) Scanning spread resistance microscopy (SSRM) image.



**Figure 2.** High-vacuum environment control unit AFM5300E (a) with the Air Protection Sample transfer unit (b).



**Figure 3.** Hot-deformed  $\text{Nd}_2\text{Fe}_{14}\text{B}$  magnet: (a) Overlay image of MFM on SE, (b) Overlay image of MFM on BSE for magnetic grain classification.



**Figure 4.** Multilayer graphene on  $\text{SiO}_2$ : (a) SE image, (b) Topography (AFM) on SE overlay image, (c) Surface potential (KFM) on SE overlay image allow us to correlate SE contrasts with surface properties.