High-resolution compact TEM for the characterization of nano-composites

Yaguchi, T.[1], Tamura, K.[2], Nagakubo, Y.[2], Wakui, A.[2], Wayama, M.[2], Mise, H.[2] and Konomi, M.[2]

[1] Hitachi High-Technologies Corporation, Japan, [2] Hitachi High-Technologies Corporation, Japan

The performance of high functionality nano-composites is heavily impacted by fine morphology and crystal structure of the utilized materials. Therefore, characterizing the nano-composites and the dynamic processes during degradation or synthesis is a necessity. Low voltage transmission electron microscope (TEM) is now one of the most powerful instruments in the study of nano-composites, which includes electron beam sensitive materials such as carbon-based and polymer compounds. In order to specifically address this growing area of research, we developed the HT7830 20-120 kV TEM with a high-resolution objective lens demonstrating on-axis lattice resolution of 0.19 nm and STEM resolution of 1 nm. Additionally, the HT7830 can accommodate a high solid angle silicon drift detector for energy dispersive X-ray (EDX) analysis.

In order to effectively analyze the crystalline structure of nanomaterials, we employed a selected-area electron beam diffraction (SAED) technique utilizing a Focused Ion Beam (FIB)-fabricated selected-area (SA) aperture with an inner diameter of 1 micron [1]. This approach is more appropriate for such nano-composites due to the damage caused by an electron beam irradiation being much less than that of nano-probe based electron diffraction. The smallest selected area diameter at the specimen plane is calculated to be 18 nm making fine ROI selection possible. To improve efficiency and throughput for acquiring the SAED patterns, a new automatic operation function, deemed "nano analysis function", has been developed. This function enables the acquisition of SAED patterns automatically at multiple positions pre-designated by a user.

Additionally, to observe solid-gas reactions at elevated temperatures, *in situ* TEM techniques can be performed with the HT7830 by utilizing a gas injection specimen heating holder [2]. To aide in such analyses, a high-speed turbo molecular pump with pumping speed of 300 l/s and an oil free scroll pump are configured along with a gun airlock valve. Depending on the required gas pressure level at the specimen area, the electron source can equipped with a LaB6 single crystal emitter or a tungsten hair-pin filament. Gas pressure of the specimen area can be increased up to 0.1 Pa when the tungsten hair filament is employed. By utilizing a compact gas supply system with a short gas path, the time for replacing the gas environment surrounding a specimen is drastically shorten compared to conventional environmental TEMs.

The implementation of these features and more present a robust, reliable, and efficient system to characterize nano-composite materials.

[1] Kamino T., et al, Proc.of IMC 18, Prague, Czech Republic (2014) IT-6-P-1552.
[2] Kamino T, et al., J. Electron Microsc. 54, (2005) pp.497 - 503.