

Global Collaboration with CEMES, French National Center for Scientific Research

—Joint Project to Implement Aberration Corrector in High-end TEM—

INTRODUCTION

IN collaboration with the Center for Materials Elaboration and Structural Studies (CEMES), an institute of the French National Center for Scientific Research (CNRS), Hitachi High-Technologies Corporation has developed a high-end field emission-transmission electron microscope (FE-TEM) that incorporates a spherical aberration corrector. Delivered to CEMES in 2012, the instrument boasts the highest resolution in the world (spatial resolution of 0.5 nm in magnetic-field-free locations)*¹.

This article looks back at the events that led to the success of this collaboration.

REQUEST FROM CEMES

CEMES is a research institute attached to CNRS, and is known as one of Europe's leading materials research centers. CEMES is also renowned for its high level of electron microscope technology, and developed a proprietary ultra-high voltage electron microscope in 1958. It has recently developed a microscopy technique known as dark-field electron holography.

Around 2007, CEMES began a project to develop and install a new electron microscope designed to assist with the research and development of next-generation semiconductors. To provide support for strained silicon*² (a material that enhances semiconductor device performance), one of the project objectives was to develop technology and applications for electron holography-based observation and measurement of strain distributions within devices.

CEMES requested that Hitachi High-Technologies partner with them in the project. This was because Hitachi High-Technologies' cold-field emission (FE)

electron gun (cold cathode field emission electron gun) technology can produce highly coherent electron beams of high brightness and high-energy resolution, making it well suited to electron holography.

RESPONDING TO THE CEMES RESEARCHERS' PASSION

Hitachi High-Technologies had previously released a high-end transmission electron microscope (TEM), model HF-3300⁽¹⁾, an FE-TEM containing a cold FE electron gun.

CEMES wanted Hitachi High-Technologies to mount a spherical aberration corrector developed by Prof. Dr. Max Haider of the German manufacturer Corrected Electron Optical Systems GmbH (CEOS) in the HF-3300, however, there were many barriers to achieving this goal. Hitachi High-Technologies had previously partnered with CEOS to develop a scanning transmission electron microscope (STEM) containing a spherical aberration corrector (model HD-2700). However, with this request, CEMES wanted Hitachi High-Technologies to modify one of its existing products by mounting another company's device on it, and Hitachi High-Technologies' quality control standards dictated that performance could not be guaranteed after such a modification. Moreover, since the CEMES project would be handled as a custom order, there were that concerns it would affect the plant's production schedule and other products, and many in the company were against taking part in the project.

Ultimately, it was the enthusiasm of the CEMES researchers and the passion of Hitachi High-Technologies' marketing and design people to respond to that enthusiasm that overcame these obstacles.

The project represented Hitachi High-Technologies' first delivery to CEMES, although CEMES already had two Hitachi High-Technologies HF-2000 FE-TEMs. These HF-2000 microscopes had been obtained by project member Dr. Florent Houdellier in 2005 from universities in the UK and Switzerland. After being personally dismantled, transported, reassembled,

*1 Electron microscopes use a magnetic field in their electron lens, so the sample is usually affected by the field. Applications such as observing the magnetic domains of magnetic materials require the sample to be unaffected by the magnetic field, a state known as *magnetic-field-free*.

*2 A technology that improves the performance of semiconductor devices by straining the silicon crystal lattice to enhance electron/hole mobility. Measuring and controlling the strain on the silicon crystal lattice are important for semiconductor manufacturing process control.

and adjusted by Dr. Houdellier, the TEMs had been in use for various types of testing and research at CEMES. This hands-on experience with Hitachi High-Technologies' electron microscopes gave CEMES researchers thorough knowledge of their advanced core technologies, making CEMES eager to have the company take part in the project. Inspired by the spirit of discovery and the enthusiasm of Dr. Houdellier and the other CEMES researchers, Hitachi High-Technologies' marketing and design divisions became passionate about living up to their expectations.

The problematic issue of guaranteeing performance was resolved by meticulously defining each area of responsibility over a series of meetings among CEMES, Hitachi High-Technologies, and others involved with the project. It was determined that maximizing the aberration correction performance would require improvements to the electron microscope's mechanical and electrical stability, and the aberration corrector would be mounted on the HF-3300S after these improvements had been achieved. A solid relationship of mutual trust was gradually built up, with Hitachi High-Technologies' design division working hard on solving problems through uncompromising and frank back-and-forth discussions with CEMES about the technology. To respond to concerns about the effect of the project on Hitachi High-Technologies' production schedule, the advantages and expected benefits of partnering with CEMES were presented within the company, and participation in the project was eventually approved through support from related organizations. CEMES and Hitachi High-Technologies signed an official agreement in January 2011.

However, two months later, Hitachi High-Technologies' production center in the Naka Division of Ibaraki prefecture suffered major damage from the Great East Japan Earthquake. It was forced to close for about a month, and the scheduled delivery date was scrapped. A sales representative posted to Hitachi High-Technologies Europe GmbH immediately

visited CEMES to apologize for the delivery delay. He made an effort to determine exactly when and how the plant would be repaired and ready for operation again, and to faithfully report the findings to CEMES. Eventually these challenges were overcome, and a new delivery plan was submitted, leading to the project's completion with the September 2012 installation and handover of the equipment.

The custom FE-TEM delivered to CEMES is both a holography electron microscope, enabling interference fringe observation, and a Lorentz microscope^{*3}, enabling kinetic in situ observation of changes to materials at the atomic level. It was therefore named I2TEM, in reference to the two initial i's of *interference* and *in situ* (see Fig. 1).

I2TEM WORKSHOP

In June 2013, CEMES held a two-day event consisting of an I2TEM completion ceremony and a workshop. The event served as an opportunity to showcase the anticipated results from I2TEM. Researchers presented data obtained after the equipment's delivery, such as semiconductor strain distributions observed using dark-field electron holography, and magnetic field distributions in magnetic nanowire.

The event was attended by about 100 researchers and others from CEMES, CNRS, and other organizations in Europe. It was also attended by researchers from Hitachi's Central Research Laboratory in Japan. Congratulatory remarks were delivered by the Director of the CNRS Institute of Physics, an acting mayor, a provincial governor, and the heads of scientific research divisions. The event was covered by the French newspaper *Le Monde*, which reported that I2TEM was the result of a collaboration between CEMES and Hitachi High-Technologies.

*3 An electron microscope configured so that the position of the sample prevents it from being affected by the magnetic field of the objective lens. Used for applications such as observing the magnetic domains of magnetic materials.



CEMES: Center for Materials Elaboration and Structural Studies
 CEOS: Corrected Electron Optical Systems GmbH
 TEM: transmission electron microscope

Fig. 1—I2TEM, Developed and Delivered in Collaboration with CEMES.

I2TEM was created by mounting a CEOS aberration corrector in Hitachi High-Technologies' HF-3300S TEM. I2TEM can perform both interference fringe observation and atomic-level kinetic observation (in situ observation), and so it was named I2TEM in reference to the two initial i's of interference and in situ. An inscription reading '愛²TEM' (愛 is pronounced 'I' and means 'love') was added to the main body to commemorate the passion of the project participants.

A signing ceremony for the partnership agreement between CEMES and Hitachi High-Technologies took place in the presence of the CNRS chief executives in September 2014. The agreement officially outlines how CEMES and Hitachi High-Technologies will collaborate on electron microscope R&D.

I2TEM APPLICATIONS

CEMES foresees I2TEM being used for measuring the magnetic fields of the permanent magnets that drive high-performance hybrid and electric vehicle motors. I2TEM can make high-resolution observations

of magnetic materials in a magnetic-field-free state (a state in which the materials are unaffected by the field). CEMES is planning to take advantage of this feature to measure the boundary magnetic forces of rare earth materials, and apply the results to explicating magnetic force mechanisms and to materials research.

Another I2TEM use anticipated by CEMES is in measuring the magnetic fields of the magnetic heads used in hard disk drives (HDDs), to help increase HDD density and read/write speed. The organization is also looking into the use of I2TEM for cancer treatments and other medical applications, and anticipates its use in the development of new materials and in pioneering new industrial and academic areas.

REFERENCE

- (1) T. Sato et al., "Hitachi's High-end Analytical Electron Microscope: HF-3300," *Hitachi Review* **57**, pp. 132–135 (Jun. 2008).

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