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ORNL

FOREIGN TRIP REPORT

ORNL/FTR-3589

DATE: April 30, 1990

SUBJECT: Report of Foreign Travel of L. F. Allard and T. A. Nolan, Materials Analysis User Center, Metals and Ceramics Division

TO: Alvin W. Trivelpiece

FROM: L. F. Allard and T. A. Nolan

PURPOSE: To present concepts for future developments in electron microscopy and to gather information on the Japanese manufacturers' plans for new electron microscopy instrumentation.

SITES VISITED:

03/26/90	JEOL Ltd. Factory Akishima, Tokyo, Japan	J. Suzuki
03/27/90	Ginza Dai Ichi Hotel Tokyo, Japan	Y. Yoda
03/28/90	Hitachi Central Research Laboratory, Kokubunji, Tokyo, Japan	A. Tonomura
03/28-29/90	Hitachi Naka Works Katsuta, Japan	Y. Yoda

ABSTRACT: At the request of Hitachi, Ltd. and JEOL, Ltd. and primarily at their expense, the travelers visited the JEOL factory and the Hitachi Central Research Laboratory in Tokyo, and the Hitachi Naka Works Factory in Katsuta. At these three facilities the travelers presented seminars explaining both ORNL's significance in materials research and electron microscopy and concepts relating to transmission electron microscopy resolution improvements, instrument automation, and electronic image acquisition. In return they received information on new instruments and concepts that the manufacturers are developing. Additionally, they had the opportunity to assess the performance of the recently introduced Hitachi 200-kV Field Emission Gun TEM. The visit provided the travelers a unique opportunity to influence the directions for future instrumental developments by two major electron microscope manufacturers.

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Comprehensive Trip Report

The travelers were invited jointly by JEOL Ltd. and Hitachi Ltd. at JEOL's and Hitachi's expense to visit their laboratories and factories in Japan for technical discussions related primarily to newly developing technologies in electron microscopy, and to present the travelers' ideas on directions for future development of advanced capabilities in analytical and high resolution electron microscopy (AEM and HREM). Our primary motivation for the visit was to gather information on the development of the intermediate voltage field emission electron gun technology which is of current interest as the method that will provide the most attractive combination of capabilities for both AEM and HREM. Such an instrument has been proposed for FY-1991 funding for the Materials Analysis User Center of the High Temperature Materials Laboratory.

March 26, 1990: JEOL Ltd. factory (Host: J. Suzuki)

The first day was spent at the JEOL factory in Akishima (Tokyo), where about 6 h of discussions were held with several of the top instrument design engineers (Y. Ishida, Y. Kokubo, and T. Taira). The JEOL field emission instrument (a 200-kV model) is still in the design and prototyping stage of development, but JEOL was able to give details of the performance of the electron gun. We were interested in obtaining from both JEOL and Hitachi engineers as much information as possible about their designs of field emission electron guns, based respectively on the "thermally assisted" and "cold" field emission technologies. The JEOL engineers contrasted both gun technologies, and justified their choice of the thermally assisted field emission gun for their forthcoming microscope. We were shown a previously confidential comparison chart with the parameters for operation of several types of field emission guns, based on information developed at JEOL.

The discussions at JEOL included a seminar presented by the travelers entitled "Future Directions in Electron Microscopy." This seminar (presented also at both Hitachi sites) was intended to generate interest by the manufacturers in the development of advanced capabilities for AEM and HREM, with an emphasis placed on extending the capabilities of today's high resolution TEM's by incorporating new ideas for the design of stages and pole pieces. For example, the JEOL engineers were surprised at the suggestion that users would gladly (we think) sacrifice the longstanding 3-mm specimen size in order to permit smaller gap pole pieces and stages to be designed that would allow for large (~30°) specimen tilt angles with significantly smaller aberrations. We pointed out that the 3-mm grid size was established in the 1940s by RCA as a standard in competition with the 2.3-mm grids then in use by Siemens Company and neither of these pioneering manufacturers was still in the electron microscopy business. The possibility of achieving a point resolution of better than 1.4 Å with an improved pole piece for our JEOL 4000EX was discussed.

At JEOL we also had the opportunity to demonstrate a number of Macintosh computer programs of interest to electron microscopists, which we have developed in or acquired for our own laboratory. Programs for high resolution image simulation and for calculation of the transfer function of the objective lens were particularly interesting to the JEOL engineers. We used these demonstrations as a basis for considering the details of changes in lens design and the consequent effects on the high resolution image.

March 27, 1990: Ginza Dai Ichi Hotel, Tokyo (Host: Y. Yoda)

We met with Hitachi representatives (Y. Tsugane, M. Miwa and Y. Yoda) on Tuesday evening, March 27, to initiate discussions that provided information to us on the directions Hitachi was planning to proceed in the development of their recently announced field emission TEM. We also discussed the potential for location of their instrument in the Oak Ridge area in the near future for demonstration purposes and to begin technique development.

March 28, 1990: Hitachi Central Research Laboratory, Kokubunji (Tokyo)
(Host: A. Tonomura)

At the Hitachi Central Research Laboratory (HCRL), we were hosted by Dr. A. Tonomura, Chief Researcher at HCRL and the scientist most identified with the historical development of techniques of electron holography using field emission sources. We again presented the seminar noted above, which was attended by about 30 Hitachi scientists and visiting scientists involved in Hitachi-sponsored research programs. We met Dr. K. Ishizuka, well-known to microscopists for introduction of Fast Fourier Transform techniques that have greatly speeded the calculation of crystal structure images by the multislice method. Dr. Ishizuka was in residence at Hitachi as a participant in the recently started Tonomura Electron Wavefront Project, a five-year effort sponsored by the Japanese government to further development of capabilities for coherent beam imaging in the transmission electron microscope. He was, of course, particularly interested in our image simulation program, MacTempas®, and commented favorably on the ease of use of the program that was provided by its Macintosh environment. We also met Dr. Komoda of Hitachi, who was instrumental in developing the field emission gun, and who would join us at the Naka Works to brief us in detail on the operation of the Hitachi FEG.

The HCRL visit also included a brief tour of the facilities, that included a chance to see a FEG TEM on which the commercial instrument was based. Dr. H. Kakibayashi described in detail the construction and use of the electron biprism used for acquiring an electron hologram.

March 28-29, 1990: Hitachi Naka Works, Katsuta (Host: Y. Yoda)

We travelled from the HCRL to Mito City on March 27 to permit two full days of discussions and instrument demonstrations at the Hitachi factory in nearby Katsuta. On the first day we met with S. Isakozawa, T. Kamino, and Y. Ueki in the morning to be briefed on the construction and operation of the cold field emission gun employed by Hitachi in their recently introduced 200-kV instrument. Several interesting differences were evident between the JEOL and Hitachi views on gun operating parameters and the physics of field emission guns in general. We conducted another seminar on future directions as at the two other locations visited, and finished with another demonstration to a large group of development engineers of our suite of Macintosh electron microscopy programs. It was interesting to note that several Macintosh computers were in use by scientists and engineers in both of the Hitachi facilities, even though Hitachi manufactures competitive computers (sold primarily in Japan).

We spent a large fraction of the afternoon on March 29 working with Y. Ueki on the new 200-kV FEG instrument, analyzing specimens from ORNL that were chosen to test specific advanced capabilities of the instrument. For example, we first analyzed a grain boundary in a Si_3N_4 ceramic that had a few percent of Y_2O_3 added as a sintering aid. The boundary was shown

to be less than 1 nm wide in a high resolution image, and yttrium was clearly detected in the boundary but not seen in the adjacent Si_3N_4 grains. Such a result has not heretofore been possible using our standard 200-kV AEM in the High Temperature Materials Laboratory. We also obtained information on the nature of systems peaks in the energy dispersive spectrum to resolve a question that arose from an earlier set of experiments conducted at the request of E. A. Kenik from ORNL during his visit to the Naka Works in November 1989 (see ORNL/FTR-3480). This information has been communicated to Dr. Kenik.

The instrumental tests were continued on March 30, when we concentrated on obtaining a series of high resolution images of gold particles on an amorphous substrate designed to demonstrate the contrast transfer characteristics of the objective lens of the field emission instrument. We were particularly impressed with the stability of the image at direct magnifications of up to 1 million times, and the high contrast in the image for observation of phase contrast detail. It was very easy to observe 2-Å interplanar spacings in the gold particles directly on the fluorescent screen of the instrument, with axial illumination. However, no TV pickup system was available for the more critical image adjustments, so it was particularly difficult for the operator (and the travelers in turn) to choose the exact operating parameters for the many high resolution images that were required by the experiment. Furthermore, when the plates were developed we found that the sample had been tilted relative to the electron beam so that the image exhibited a range of focusses from one side of the negative to the other. This made accurate interpretation impossible, and it was decided that another full set of images should be taken and provided to us at a later date when an appropriate TV system could be installed on the instrument.

We finished our visit to the Naka Works with a tour of the factory led by M. Matsushita and a tour of the nearly completed new 1500-kV ultrahigh resolution TEM, which is ready to be shipped to a university in Japan. This instrument is guaranteed to give 1-Å point-to-point resolution, and is available from Hitachi for a mere $7M. The high resolution performance is obtained by the reduction of wavelength provided by the increased accelerating voltage, but also suffers from the potential problem of sample damage that can occur at high voltages. In discussions with Mr. T. Honda, chief engineer on the ultrahigh resolution project, we learned that Hitachi has been working toward an advanced operational capability for future electron microscopes that was outlined in detail in our seminar. They plan to introduce remote operation for future high voltage microscopes in order to reduce the radiation shielding requirements. It was somewhat gratifying to see firsthand that our ideas are being echoed by at least one manufacturer.

Summary Comments:

Our consensus opinion was that the performance features of the 200-kV field emission gun microscope represent a significant advance in analytical capabilities over present AEMs. The combination of good resolution, high brightness, and fine probe size would give at least an order of magnitude improvement in the ability to characterize fine details of a material's microstructure over that which we presently have in our laboratory. The modern FEG AEM clearly overcomes some of the limitations of older, lower-voltage FEG instruments for routine microscopy.

APPENDIX A

ITINERARY FOR L. F. ALLARD AND T. A. NOLAN

1990

03/24-25	Travel from Knoxville, TN, to Tokyo, Japan
03/26	Visit JEOL, Ltd., factory in Akishima to discuss Field Emission Electron Microscopy and High Voltage Electron Microscopy
03/27	Meet with Hitachi representatives at Ginza Dai Ichi Hotel
03/28	Visit Hitachi Central Research Laboratory in Kokubunji to discuss Field Emission Electron Microscopy and attend Electron Holography workshop
03/28	Travel from Tokyo, Japan, to Katsuta, Japan
03/29-30	Visit Hitachi Naka Works in Katsuta to discuss Field Emission Electron Microscopy and Holography Instrumentation
03/31	Travel from Katsuta, Japan, to Tokyo, Japan, to Knoxville, TN

APPENDIX B

LIST OF PERSONS CONTACTED TO A SIGNIFICANT EXTENT

JEOL Ltd.	
Mr. A. Mori	Managing Director Overseas Operations
Mr. M. Ebina	Assistant General Manager Overseas Operations
Mr. J. Suzuki	Assistant Manager, USA Section Overseas Operations
Mr. Y. Ishida	General Manager R&D Department Electron Optics Division
Mr. Y. Kokubo	Assistant General Manager R&D Department Electron Optics Division
Hitachi Central Research Laboratory	
Dr. A. Tonomura	Chief Researcher Advanced Research Laboratory
Dr. H. Kakibayashi	Researcher Electron Optics Central Research Laboratory
Mr. Y. Tsugane	Vice President & General Manager Scientific Instruments Division
Mr. Y. Yoda	Manager Demonstration Laboratory, USA
Mr. Y. Tsukada	Manager & Senior Engineer Scientific Instruments Division
Dr. K. Ishizuka	Visiting Researcher Electron Wavefront Project Advanced Research Laboratory
Dr. J. Ezot	Visiting Professor (Stuttgart University)
Mr. M. Miwa	Manager Scientific Instruments International
Hitachi Naka Works	
Mr. S. Isakozawa	Senior Engineer Electron Beam System Design Department
Dr. T. Komoda	Chief Engineer Electron Beam System Design Department
Dr. F. Nazata	Chief Researcher Application Technology Department

Mr. Y. Ueki	Engineer Application Technology Department
Mr. T. Kamino	Engineer Application Technology Department
Mr. M. Matsushita	Chief International Operations
Mr. T. Akiba	Scientific Instruments International
Mr. T. Honda	Chief Engineer Electron Beam System Design Department

APPENDIX C

LITERATURE ACQUIRED

1. A. E. Bell and L. W. Swanson, "Total Energy Distribution of Field Emitted Electrons at High Current Density," *Phys. Rev. B* 19(7) 3353-64 (1979).

2. Assorted Hitachi brochures of instruments and research operations.

APPENDIX D

DISTRIBUTION LIST

1. John J. Easton, Jr., Assistant Secretary for International Affairs and Energy Emergencies, DOE, Washington
2. J. M. Davis, Assistant Secretary, Conservation and Renewable Energy, DOE, Washington
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5. D. J. Cook, Director, Safeguards and Security Division, DOE/ORO
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24. W. Fulkerson
25. L. L. Horton
26. C. R. Hubbard
27. D. R. Johnson
28. E. A. Kenik
29. M. C. Matthews

30-34. T. A. Nolan

35. P. L. Rittenhouse
36. A. C. Schaffhauser
37. G. M. Slaughter
38. V. J. Tennery
39. J. O. Stiegler
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