"NO TITAN, NO EXCUSE" (T. J. KONNO, 1 APRIL 2008) A REPORT ON AN INTERNATIONAL WORKSHOP ON REMOTE ELECTRON MICROSCOPY FOR IN SITU STUDIES

R. Sinclair[a]*, R. Chin[c], A. L. Koh[a,d], and G. Solórzano[b]

[a] Department of Materials Science and Engineering, Stanford University. USA.
[b] Departamento de Ciência dos Materiais e Metalurgia, Pontifícia Universidade Católica do Rio de Janeiro. Brazil.
[c] Stanford Nanocharacterization Laboratory, Geballe Laboratory for Advanced Materials, Stanford University. USA.
[d] Department of Materials, Imperial College London. UK.

*Corresponding author e-mail: bobsinc@stanford.edu

Received: October 2007. Approved: December 2008
Published on-line: May 2009

ABSTRACT
This paper summarizes the presentations and discussions that took place at an international workshop concerning remote-access electron microscopy. This capability is rapidly expanding owing to improvements in the power of personal computers and ever-increasing speed and bandwidth of internet connections. It is also becoming more important as the cost of state-of-the-art equipment soars. The opportunities in both research and in educational outreach are identified and discussed, and the need for following meetings established. The importance of embracing established and emerging national scientific endeavors is also emphasized.

Keywords: Electron microscopy, remote access, in situ studies.

"LA FALTA DE TITAN NO ES DISCULPA" (T. J. KONNO, 1 de ABRIL 2008) INFORME DEL TALLER INTERNACIONAL SOBRE MICROSCOPIA ELECTRONICA POR CONTROL REMOTO PARA ESTUDIOS IN SITU

RESUMEN
El presente artículo resume las presentaciones y discusiones que tuvieron lugar durante el taller internacional sobre microscopia electrónica con acceso por control remoto. Esta nueva modalidad de operación remota se expande rápidamente debido al avance en la capacidad de computadoras personales y al creciente aumento en la velocidad y ancho de banda en las conexiones por Internet. También se vuelve más accesible é importante en la medida en que los precios de los instrumentos de microscopia electrónica de última generación llegan a valores cada vez más elevados. En este taller fueran identificadas y evaluadas las oportunidades tanto en el campo de la investigación como en la educación y también destacada la necesidad de establecer talleres y encuentros subsecuentes. Fue también enfatizada la importancia de incorporar en este esfuerzo la participación de existentes y nuevos emprendimientos científicos nacionales.

Palabras claves: Microscopía electrónica, acceso por control remoto, estudios in situ.

INTRODUCTION

A workshop was held at Stanford University on March 31, April 1, 2008 to discuss the current status, and future directions, of remote electron microscopy, especially for in situ studies [1].

The electron microscope is the most powerful analytical tool to study and to understand materials at the nano-scale. In fact, it was exactly such an instrument that revealed the details of carbon nanotubes [2] that led to the burgeoning field of nanotechnology. One of the major conundrums concerns the facts that as the electron microscopes become increasingly more powerful, their cost has risen so high that fewer scientific laboratories can afford to purchase them, a situation clearly exacerbated in the developing countries. One remedy to allow more widespread usage of these specialized instruments is by "remote access" whereby a researcher in a distant location can operate the microscope at the host laboratory and so obtain data not otherwise possible. With the ever increasing power of personal computers, and the available speed and bandwidth, this is now becoming an ever more

attractive option. Some laboratories have now set up these capabilities, mainly for educational outreach.

Participants were suggested and invited in order to represent a broad cross-section of remote access practitioners as well as those who might benefit from such a capability. The list was certainly not exhaustive nor complete, but was consistent with the funding and philosophy of the meeting. This paper is a short overview of the meeting. Further details, the complete program and the submitted abstracts can be found at the workshop website [1].

SUMMARY OF PROCEEDINGS

The first talk of the workshop was presented by Dr. John Mansfield (U. Michigan, USA) on the development of remote microscope for training groups of student users [3]. This followed up on his talks/seminars at recent conferences in South America, showing how essential this approach is for an institution-wide multi-user facility with a large constituency of users. Dr. Marc De Graef (Carnegie Mellon Univ., USA) continued along the same vein describing how their remote access capability had been designed to act as an interactive tool for teaching microscopy principles as well as its use in practice [4]. Both developments were recognized as being highly effective and innovative. Dr. Vicente Garibay (Instituto Mexicano del Petróleo, México) described the efforts of several institutions in Mexico to forge an active remote access for electron microscopes both for research and distance learning, and Dra. Gema Gonzalez (Instituto Venezolano de Investigaciones Científicas, Venezuela) outlined the increase in networking across the Americas, and ultimately to Europe, by way of the Internet II project.

The second session started with Dr. David McComb (Imperial College, UK) explaining the "Global Lab" project under the umbrella of the AtlantICC Alliance [5, 6] jointly with Oak Ridge National Laboratory and Georgia Institute of Technology. Dr. Thomas Isabell (JEOL USA, Inc., USA) showed the development of the Sirius client/server platform for remote control of a TEM. Dr. Lancy Tsung (Gatan, Inc., USA) also elaborated on the industrial perspective, as particularly related to TV-rate CCD camera technology and its importance for both remote and in situ microscopy. Dr. Nestor Zaluzec (Argonne National Laboratory, USA), a long-time proponent and active developer of remote access, talked about his Tele-Presence Collaboratory that has been in operation for over a decade [7]. Finally, the morning session ended with Dr. Gary Brown (Exxon Mobil Chemical Company, USA) demonstrating how he and his colleagues utilize remotely from Baytown, Texas a TEM in the Exxon facility in Machelen, Belgium, during the daytime in the US, night-time in Europe. This TEM is equipped with a special multi-sample Autoloader. Dr. Brown further emphasized the importance of "people interactions" in facilitating remote microscopy experimentation.

The afternoon session started with Dr. Ulrich Dahmen (National Center for Electron Microscopy, USA) giving an update on the current status of the TEAM project (e.g., [8,9]) and the plans for its remote operation, due to be available to the scientific community in 2009. Dr. Larry Allard (Oak Ridge National Laboratory, USA) described, and actually demonstrated at a later time, the remote operation of an in situ heating experiment on Pt nano-particles at 1000ºC on his aberration-corrected JEOL TEM at his home laboratory in Tennessee [10]. Dr. Alfredo Tolley (Centro Atomico Bariloche, Argentina) talked about collaborations developed with electron microscope laboratories in Europe, USA and Japan mainly for physical metallurgy and Dr. Alejandro Zuniga (U. Santiago, Chile) described the interactions between laboratories in Chile and future possibilities with other facilities in Latin America.

Dr. Auke van Balen (FEI Company, The Netherlands) then discussed the philosophy behind the growing usage of remote microscopy and showed how it can become increasingly important not only in specialized research

centers but also for industrial applications. Dr. Norman Salmon (Hummingbird Scientific, Inc., USA) next described the development of various specimen holders for in situ TEM complementing those made by the microscope manufacturers themselves. Dr. Yoshio Bando (National Institute for Materials Science, Tsukuba, Japan) showed his group's most recent work combining a piezo-driven probing holder with electrical measurements on novel and metal-filled nanotubes [11]. Dr. Eva Olsson (Chalmers Univ. of Technology, Sweden) followed up with a comprehensive overview of her research using probes to mechanically and electrically stress nanotubes, discussing the role of future remote collaborative studies [12]. In the last paper of the first day, Dr. Daniel Ugarte (Laboratorio Nacional de Luz Sincrotron, Brazil) emphasized the importance of high quality basic research in Latin American countries, exemplifying this with their efforts on deformation of metal nanowires created in situ in noble metal gold thin films [13].

The second day started with Dr. Gustav Van Tendeloo (U. Antwerp, Belgium) outlining the Enabling Science and Technology through European Electron Microscopy (ESTEEM) project amongst eleven institutions in eight countries in Europe, which is clearly a most impressive international collaboration within the European Community [14]. It is anticipated that remote access to specialized instruments will become increasingly important, especially for the proposed second term for this project. Dr. Toyohiko Konno (Tohoku Univ., Japan) documented the evolution of his in situ TEM research, from studies on metal-induced crystallization to utilizing advanced imaging techniques for analyzing phase transformations in alloys [15]. He concluded with the most appropriate phrase for the workshop, which we have borrowed as the title of this paper, referring to the fact that it is not necessary to own the most advanced TEM's to carry out innovative research, especially with the advent of remote access and increasing global interactions. Dr. Maria Gomez (U. del Valle, Colombia) then reviewed her group's work on metallic superlattices in collaboration with researchers at the Universities of California at San Diego and at Berkeley [16], and the session was brought to a lively conclusion by Dr. Miguel Yacaman (U. Texas Austin, USA) not only describing his own work on catalytic nanoparticles (e.g., [17]) but putting it into the perspective of exciting the next generation of scientists, many of immigrant origin, in the local area of the University of Texas.

In the final session of presentations, Dr. Jon McCarthy (U. Wisconsin – Madison, USA) showed how it is now feasible to develop remote access without the need for either proprietary hardware and with minimal (but important) input from the instrument manufacturer, and showed how their collaboration with nearby institutions was so being established. Dr. Xiaofeng Zhang (Hitachi High Technologies America, Inc., USA) presented some of the latest developments from Hitachi using a specialized specimen holder (developed by Dr. Takeo Kamino) for in situ environmental TEM. Dr. Nobuo Tanaka (Nagoya Univ., Japan) outlined his group's recent in situ work on phase changes in photocatalytic titanium oxide materials [18] and Dr. Renu Sharma (Arizona State Univ., USA) showed her work on environmental in situ studies of carbon nanotube growth [19]. The formal presentations were concluded by Dr. Ann Marshall (Stanford Univ., USA) showing the sub-freezing point solidification of Au-Ge catalyst particles for germanium nanowire growth and the recent analysis of chirally-branched PbSe nanowires associated with the Eschelby twist by the core growth screw dislocation [20].

The afternoon sessions involved a division of the participants into two breakout groups, one concerned with remote microscopy for research (chaired by Dr. Ulrich Dahmen) and the other focusing on the educational aspects of remote access (chaired by Dr. Marc De Graef). The respective chairs reported to the whole group the major discussion points that can be summarized as follows:

Research

In order to make remote electron microscopy an enabling research tool, it is necessary to understand the needs and expectations of users. Four levels of remote microscopy were defined by the group as: passive microscopy, collaborative microscopy, remote diagnostics and active telepresence, in increasing order of complexity. Each level requires a different set of specifications in terms of host and client servers, bandwidth etc. Protocols will need to be established for each category so that the users can have access to expertise and documentation when performing their experiments from a remote location.

The main action item identified, and currently in-progress, is the launch of a TelePresence Collaboratory & Remote Access Microscopy/Microanalysis Database by Dr. Nestor Zaluzec (Argonne National Laboratory, USA) [21]. This database is publicly accessible and can be used by potential remote access users worldwide to determine the characteristics of facilities or hardware and software developed for telepresence/remote access protocols. In the long term, it is envisioned that researchers will be able to submit proposals and make the "connections" by identifying the national labs and research institutions that have the required microscope capabilities. A Steering Committee with structure similar to that of the ESTEEM Project, or U.S. Department of Energy (DOE) funded projects, can be established to approve the proposals.

Potential issues were also identified during the session. These include difficulties associated with specimen preparation and transportation to the host site. Furthermore, as many state-of-the-art electron microscopes are currently located in the U.S. National Laboratories, regulations governing such DOE-based research laboratories must be considered before embarking on such research projects.

Education

While the advances in electron microscopes are great, the question of how to guarantee that there will be a sufficient number of qualified users arises. Further, it was noted that good scientific practices and methodology are independent of the sophistication of the instrument, and that there is, and always will be, a need for people with basic microscopy skills, reiterating Dr. Konno's statement of "No Titan is no excuse". As such, in thinking of remote microscopy, there are two "faces" to consider: instructional remote microscopy (to facilitate teaching) and operational remote microscopy (to carry out real research). Present educational resources include: tabletop SEMs, textbooks, recorded lectures (i.e., MSA and RMS videotape and DVD libraries), and courses taught at college and university facilities. Several action items were identified, including: initiating conversation with microscope manufacturers to donate tabletop SEMs to universities with the understanding that these instruments are to be used for outreach activities in high schools and such; updating the MSA and RMS instructional DVDs; exploring the possibility of using existing courses on SEM and TEM to (i) broadcast or (ii) allow remote control, as per the Carnegie Mellon University setup; addressing what kind of "delivery system" works well in undergraduate and graduate education, perhaps also working with assessment and education experts to ensure efficacy; and describing the job of a modern microscopy facility technician/professional, including aspects of sample preparation and what in-house services would be needed, and indeed possible, with the modern microscopes.

Fig. 1. Participants in the International Workshop on Remote Electron Microscopy for In Situ Studies, Stanford University, March 31 – April 1, 2008.

The overall conclusion of the meeting was that remote access is both a reality and necessity for future research and education involving advanced microscopy studies. In order to develop this avenue further it was decided to have, in the first instance, two further meetings in the forthcoming years. The first is to be held at Chalmers University of Technology, Gothenberg, Sweden in April 2009 (please contact Dr. Eva Olsson or the present authors for details) and the following meeting will be at Carnegie Mellon University, Pittsburg, USA organized by Dr. Marc De Graef.

ACKNOWLEDGMENTS

The core funding for this workshop was kindly provided by the Tinker Foundation through the Center for Latin American Studies, Stanford University. We would like to thank Dr. Herbert Klein, Ms. Megan Gorman, Ms. Geraldine Slean, Mr. Omar Ochoa and Dr. Adrian Lew for their enthusiastic support and assistance for this project. Additional funding was also generously donated by several equipment manufacturers, most notably JEOL Ltd., Gatan Inc., FEI Co., Hitachi Inc., Carl Zeiss SMT Inc. and Hummingbird Scientific Inc. The use of facilities at Stanford University and support for our research through the Cancer Center for Nanotechnology Excellence – Therapeutic Response program are also very much appreciated. We would also like to thank Mr. Fernando Novoa for discussions concerning the translations.

REFERENCES

[1] http://snl.stanford.edu/remote/workshop.htm

[2] Iijima S. (1991) “Helical microtubules of graphitic carbon”, *Nature* 354:56-58.

[3] Mansfield J.F., Adamson A., Coffman K. (2000) “Development of a System to Provide Full, Real-time Remote Control of a Scanning Electron Microscope

across the Second Generation Internet: The Teaching SEM", *Microsc. Microanal.* 6:31-41.

[4] De Graef M., Nuhfer N.T., Cleary N. (2002) "The Digital Microscopy Teaching Environment: a Hard-Wired Microscopy Classroom", *Microsc. Microanal.* 8:176-181.

[5] Perkins J.M., Blom D.A., McComb D.W., Allard L.F. (2007) "Functional Remote Microscopy via the AtlanTICC Alliance", *Microsc. Microanal.* 13 (Suppl 2):1702CD.

[6] http://www.atlanticcalliance.org/

[7] Zaluzec N.J. (1995) "The Development Of An Electronic Microscopy Journal On The World Wide Web", *Proc. Microsc. Microanal. 1995* 658-659.

[8] http://ncem.lbl.gov/TEAM-project/

[9] Kisielowski C., Freitag B., Bischoff M., van Lin H., Lazar S., Knippels G., Tiemeijer P., van der Stam M., von Harrach S., Stekelenburg M., Haider M., Uhlemann S., Müller H., Hartel P., Kabius B., Miller D., Petrov I., Olson E.A., Donchev T., Kenik E.A., Lupini A.R., Bentley J., Pennycook S.J., Anderson I.M., Minor A.M., Schmid A.K., Duden T., Radmilovic V., Ramasse Q.M., Watanabe M., Erni R., Stach E.A., Denes P., Dahmen U. (2008) "Detection of Single Atoms and Buried Defects in Three Dimensions by Aberration-Corrected Electron Microscopy with 0.5-Å Information Limit", *Microsc. Microanal.* 14:469-477.

[10] Allard L.F., Bigelow W.C., Nackashi D., Damiano J., Mick S.E. (2008) "A New Paradigm for Ultra-High Resolution Imaging at Elevated Temperatures", *Microc. Microanal.* 14:792-793CD.

[11] Costa P.M.F.J., Golberg D., Shen G., Mitome M., Bando Y. (2008) "ZnO low-dimensional structures: electrical properties measured inside a transmission electron microscope", *J. Mater. Sci.* 43:1460-1470.

[12] Strand H., Svensson K., Olsson E. (2008) "Measuring electrical properties of carbon nanotubes using liquid metal immersion, an in situ scanning electron microscopy study", in EMC 2008, Vol. 2: Materials Science, S. Richter, A. Schwedt (Eds.) pp. 313–314, DOI: 10.1007/978-3-540-85226-1_157.

[13] Ugarte D. (2007) "Real-time atomic resolution study of atomic-size metal nanowires", *Acta Microsc.* 16 (1-2, Supp. 2): 7-8.

[14] http://esteem.ua.ac.be

[15] Konno T.J., Nishio R., Semboshi S., Ohsuna T., Okunishi E. (2008) "Aging behavior of Cu–Ti–Al alloy observed by transmission electron microscopy", *J. Mater. Sci.* 42 (11):3761-3768.

[16] Santamaria J., Gómez M.E., Vicent J.L., Krishnan K.M., Schuller I.K. (2003) "Scaling of the Interface Roughness in Fe-Cr Superlattices: Self-Affine versus Non-Self-Affine", *Phys. Rev. Lett.* 89:190601-190604.

[17] Mejía-Rosales S.J., Fernandez-Navarro C., Pérez-Tijerina E., Montehano-Carrizales J.M., José-Yacamán M. (2006) "Two-Stage Melting of Au-Pd Nanoparticles", *J. Phys. Chem. B* 110(26):12884-12889.

[18] Yoshida K., Nozaki T., Hirayama T., Tanaka N. (2007) "*In situ* high-resolution transmission electron microscopy of photocatalytic reactions by excited electrons in ionic liquid", *J. Electron Microsc.* 56(5):177-180.

[19] Sharma R., Rez P., Brown M., Du G., Treacy M.M.J. (2007) "Dynamic observations of the effect of pressure and temperature conditions on the selective synthesis of carbon nanotubes" *Nanotechnology* 18:125602-125609.

[20] Zhu J., Peng H., Marshall A.F., Barnett D.M., Nix W.D., Cui Y. (2008) "Formation of chiral branched nanowires by the Eshelby Twist", *Nature Nanotechnology* 3: 477-481.

[21] http://telepresencecollaboratory.org/