TEM Sample Preparation: An Interdisciplinary Website

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Sample preparation is of central importance for the characterization of materials by transmission electron microscopy (TEM). As a guide to researchers seeking practical help on the use of all types of TEM sample preparation techniques, we have created an Internet website. This website has been designed in French and is now translated into English. The website is accessible free of charge.

The TEMSAMPREP website, figure 1 http://temsamprep.in2p3.fr/, is the result of a synergistic effort of an atypical team of five electron microscopy scientists having different research specialties in physics, mineralogy, material science, and biology. They shared five years of human adventure in creating the website to transmit their 30 years of TEM experience to the international community.

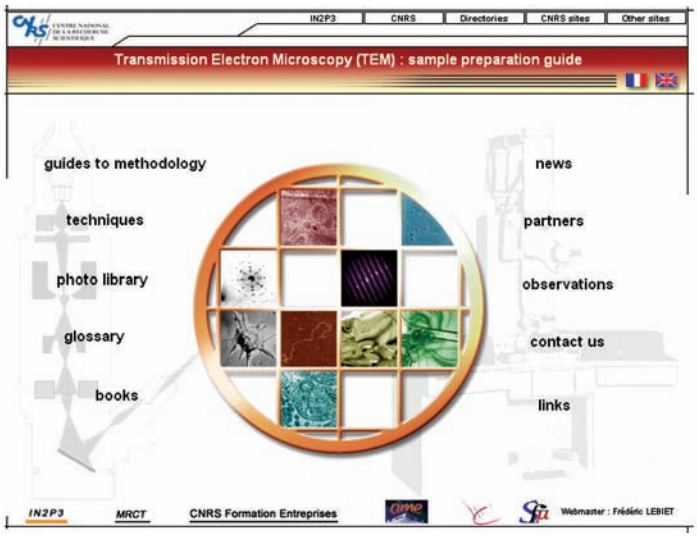

Figure 1. Website home page

The rationale for this interactive guide

What are the real questions concerning sample preparation in material science or biology? The goals of specimen preparation are to produce an electron transparent specimen representative of the bulk material in both structure and composition, to provide easy access to required information, and to make certain that the specimen enables the microstructure of the sample to be accurately studied and convincingly illustrated in reports and publications. In these terms, there is little difference between the various fields of research. The main differences are the water content in living materials, as well as the dynamics of these systems. In biology, we need to stop the dynamics and remove water from the sample or freeze the specimen. In material science, in order to study specimen dynamics, we need to increase the temperature of the sample. Materials science samples have high atomic numbers, generally, leading to high-contrast TEM images. Biological samples have low atomic numbers and produce low-contrast images via mass-thickness contrast effects. Accordingly, for TEM observation of biological specimens, one has to increase observable contrast by either staining the specimen, *i.e.* changing its chemistry, or freezing the specimen in its native conditions and observing the specimen using high-contrast TEM imaging modes.

Description

This website is a guide to researchers seeking practical and up-to-date information on TEM sample preparation. It contains all types of TEM sample preparation techniques in materials science, earth science, and biology. It is a work in progress. This website comprises:

1. Interactive Guides

This section contains a database of materials and provides an automated guide to the choice of the most suitable TEM sample preparation technique based on materials properties and type of analysis. This database has been designed to include new techniques developed by the user community. It includes:

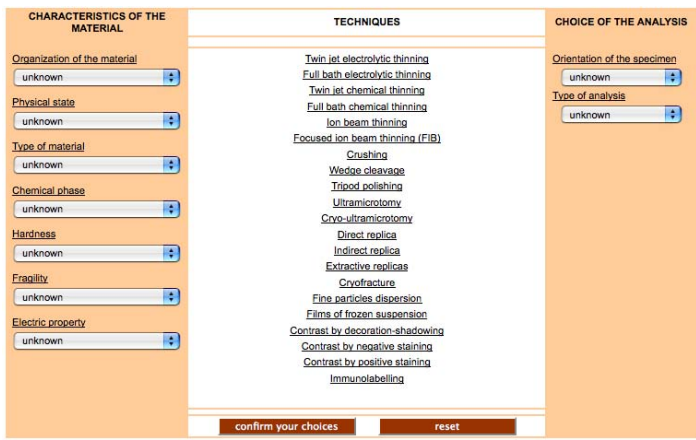

Figure 2. Simple Guide to select the appropriate method

a. A Simple Guide that automatically selects the sample preparation technique(s) that correspond to the best fit to the properties of the material being studied as well as to the type of TEM analysis to be performed (figure 2).

b. An Educational Guide This interdisciplinary guide allows one to understand and learn about the applicability of the selected criteria for each technique and each type of material combination (figure 3). Among the selected techniques, it provides an online list of advantages, artefacts, drawbacks, and limitations of candidate specimen preparation methods *vs.* protocols that allow the user to refine the final choice of methods.

For both guides, the user can enter through two pathways: 1) by the choice of TEM analyses to be done; 2) by the properties of the material to be analyzed. In either case, the output of the guide provides input for the researcher to make a final choice of a sample preparation protocol. In the educational guide, an information table is given that speaks to the relative criteria chosen as a function of sample properties, type of TEM analysis, and preparation technique employed. The table will be active any time during the research, in order to give the user all the necessary knowledge on what is, or is not, possible for each of the criteria.

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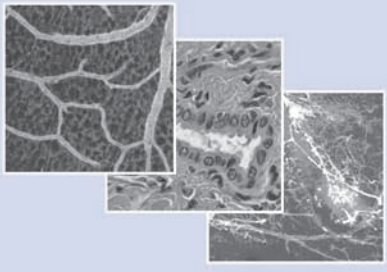

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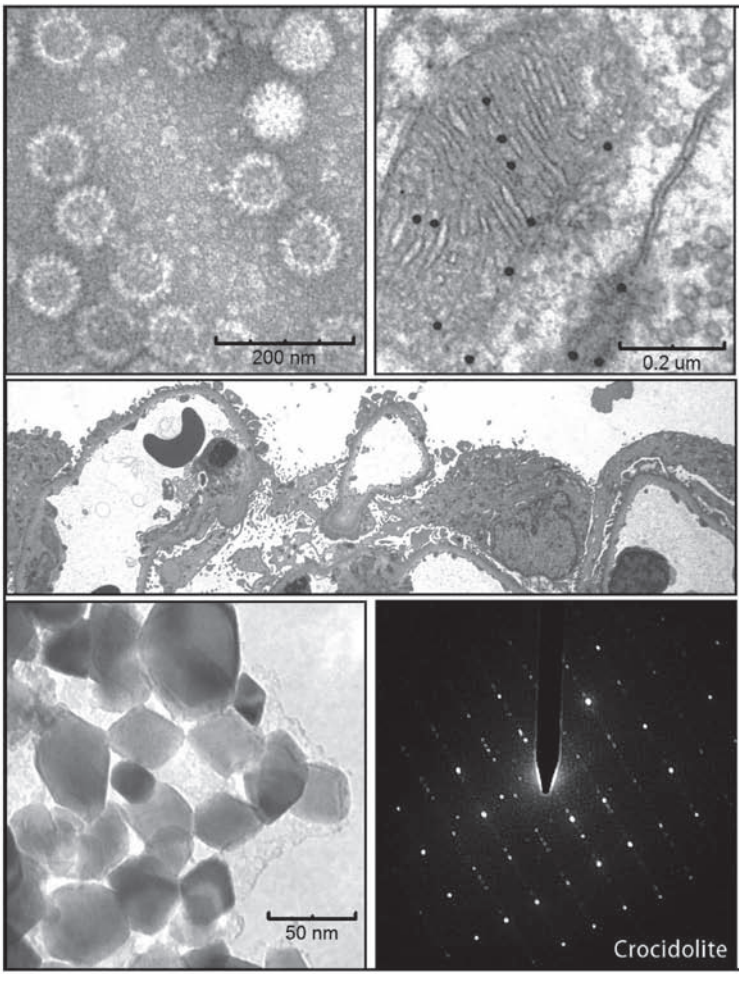

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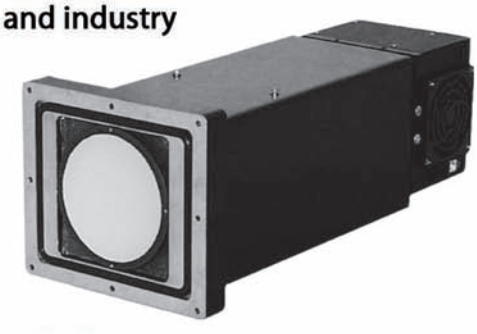

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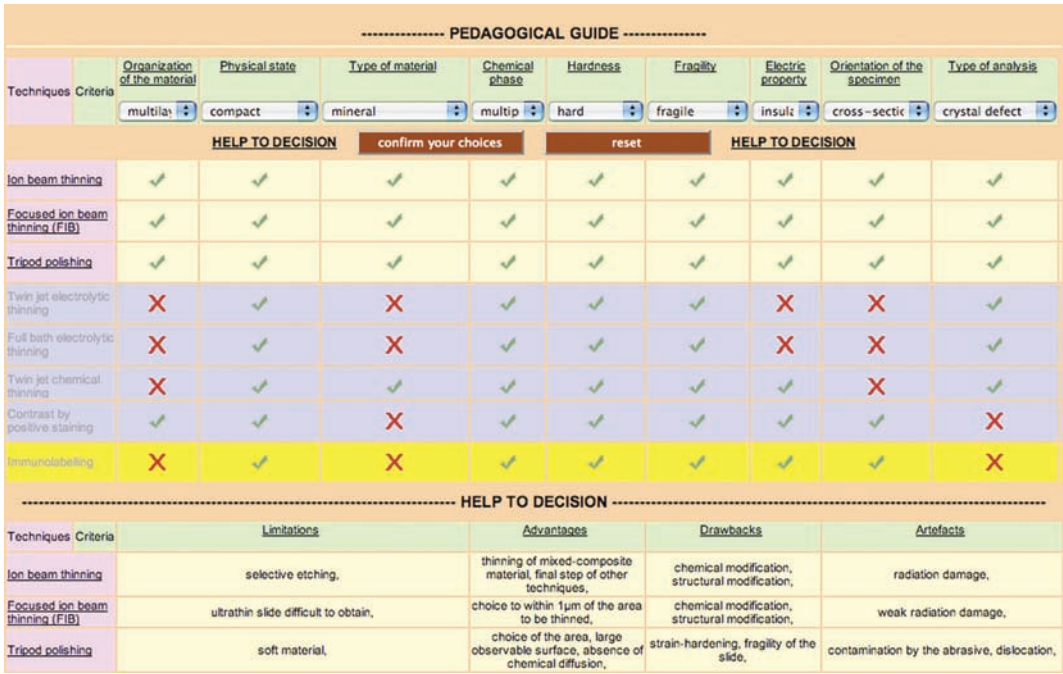

Figure 3. Educational guide

2. Techniques

At this writing, the technique section contains 36 sample preparation techniques with important information on the basic principle, materials, procedure, artefacts, advantages, drawbacks, and risks (figure 4). Also included is the specific equipment required for each technique, the domain of its application, the possible variants of each technique, prior and/or complementary techniques, and the types of TEM analyses made possible. The new microscopist user will be helped, for each of the 36 technique files, to find within each file the company names for choices of specific equipment needed for each technique. Each file window gives access to all the possible complementary techniques to perform before thinning or after thinning to improve TEM observation. All information on key words of the database are activated and linked to the lexicon to provide needed definitions. In addition, new techniques coming from the user community can be added to this database from the "News" window.

3. Photo library

The website contains a database with images of different types of materials and of artefacts produced by the sample preparation techniques (figure 5). This database is enriched by a link to the biological French data base (RCCM) (Reseau des Centres Communs de Microscopie of MRCT unit of CNRS) that uses the same criteria for the description of organization and type of material. This image database has been designed to be interactive and will be augmented by new images of various materials from the international user community either from the photo library or from the "News" windows.

4. Scientific News

This part is interactive and concerns points of interest to both the microscopist community and companies: calendars of events with announcements of workshops, meeting dates, special events on TEM and sample preparation techniques, as well as general TEM news. This part has been designed to be fed by the microscopy community, including event organizers.

5. Lexicon

In order to take into account the interdisciplinarity of the TEM community, definitions of technical words have been included to be sure that the same meaning is given for each important key word. All information concerning the criteria definitions in the guides is linked to the lexicon. These key words, when present in the technique files, are also linked to the lexicon to give the definitions at any time.

6. Books:

We have authored a book series in two volumes addressed to researchers, engineers, and technicians in the fields of materials science (chemistry and physics), earth science (mineralogy and geology), and biology, to whom transmission electron microscopy

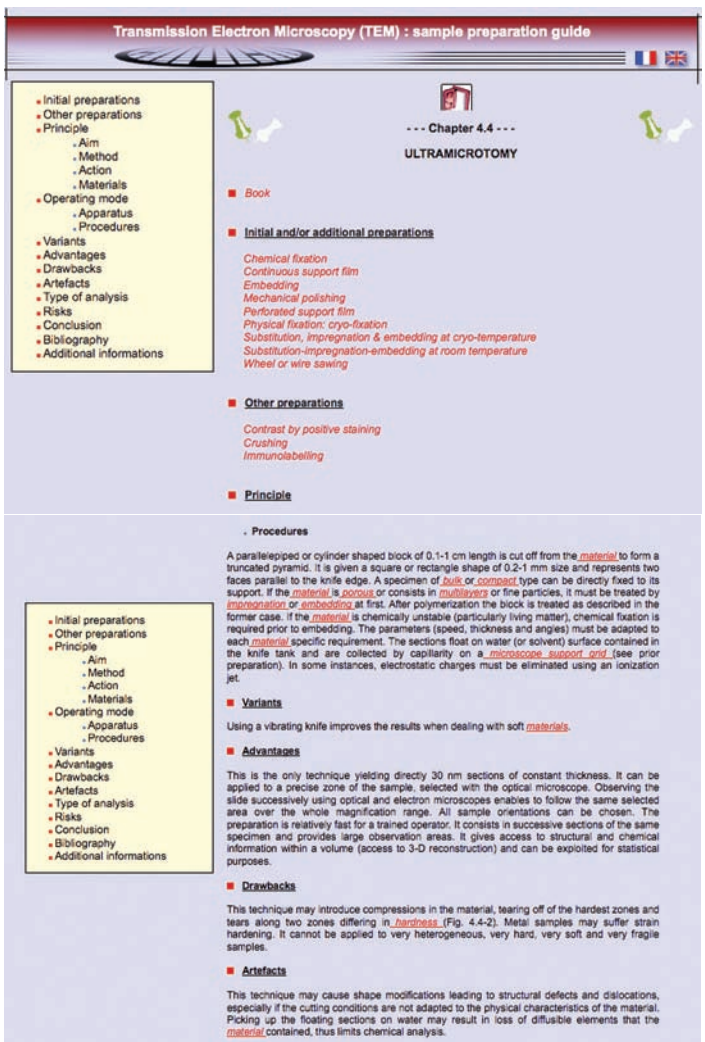

Figure 4. Technique files

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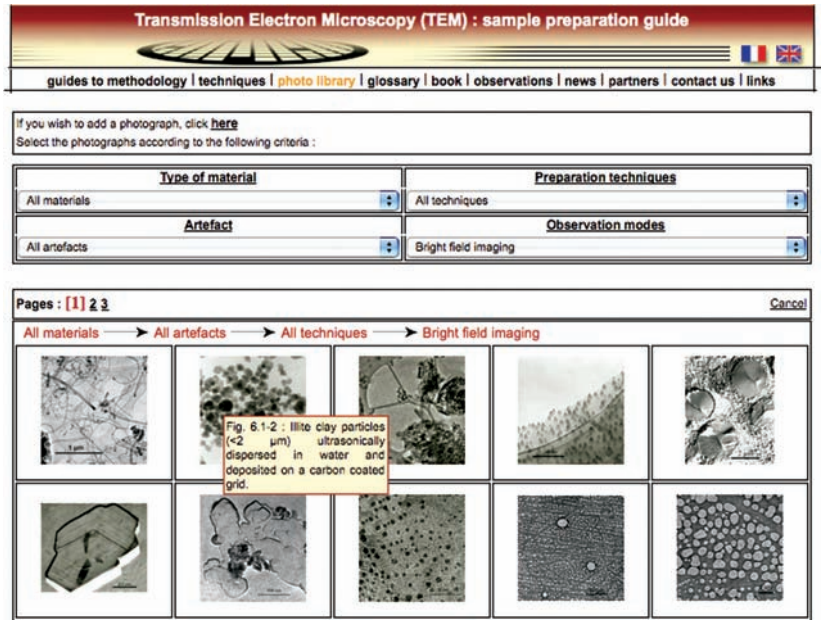
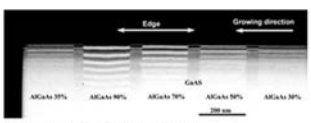


Figure 5. page of the photo library.



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Caption : Fig. 4.2-2 : AlGaAs/GaAs multilayer. The TEM image (combined to a simulation program) gives access to semi quantitative chemical composition analysis.

Experimental conditions : TEM bright field image. Microscope: Philips EM 430, 300kV

Laboratory : Centre Interdisciplinaire de Microscopie Electronique (CIME)

Laboratory address : EPFL-CIME, Bï¿½timent MXC, Station 12, 1015, Lausanne, CH

Preparation technique : Wedge cleavage

Observation mode :

Scientific research domains and/or scientific themes : Material Science

Keywords : semiconductor,

Link : http://cime.epfl.ch

Figure 6. Example of an image found in the photo library.

analysis is being used to understand both structural characteristics and the properties and specific functions of the materials. The books are companions to the website.

Book editions: Title : "Guide de Préparation des échantillons pour la Microscopie électronique en transmission". French version, Edited by "Les publications de l'Université de Saint Etienne" (April 2007), The English version titled: "Sample Preparation Guide for Transmission Electron Microscopy". Authors: J. Ayache, L. Beaunier, D. Boumendil, G. Ehret and, D. Laub. Edited by Springer N.Y. will be available ~November 2009.

VOLUME 1: Methodology covers theoretical and practical aspects of sample preparation for TEM analysis. It brings the reader tools for preparation and observation techniques. This volume also gives directions to the best preparative techniques to implement by taking into account material types, material structures, and their properties. This technical handbook identifies the main artefacts arising from the preparation techniques (mechanical, physical and chemical techniques) employed. It covers a wide range of TEM analysis and observation modes and gives a comprehensive comparison among techniques.

VOLUME 2: Techniques is dedicated to technical hints related to 14 prior- and post-preparation techniques and 22 thin slice detailed protocols for TEM analysis. It considers theoretical sidelights, experimental conditions and guidelines, options and variations, advantages and constraints, and common artefacts induced by given sample/methodology combinations. Application fields for the main techniques are developed with particular considerations of the type of materials, conditioning, and compatibility of analysis with a given preparation protocol and the risks of the technique.

Conclusion

Because of its conception, this website is unique in its kind. It provides help to the user in learning the criteria for the best choice for sample preparation technique depending on the type of TEM analysis to be conducted for materials science or biological problems.

The website will be closely linked to the international microscopy centers for TEM and SEM sample preparation. It is now referenced in the many research centers and appears at the present time on the website of national and international electron microscopy scientific research centers such as CNRS (France), NCEM (Berkeley, USA), CIME (Lausanne, Switzerland), and RUCA (Anvers, Antwerpen). It is listed with electron microscopy societies as SFC, SFµ, GUMP, *etc.*, in France, as well as EMS, EMC in Europe. It is expected to extend to other international microscopy labs and universities. ■

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