Examples of Focused Ion Beam (FIB) Milling for Analysing Samples

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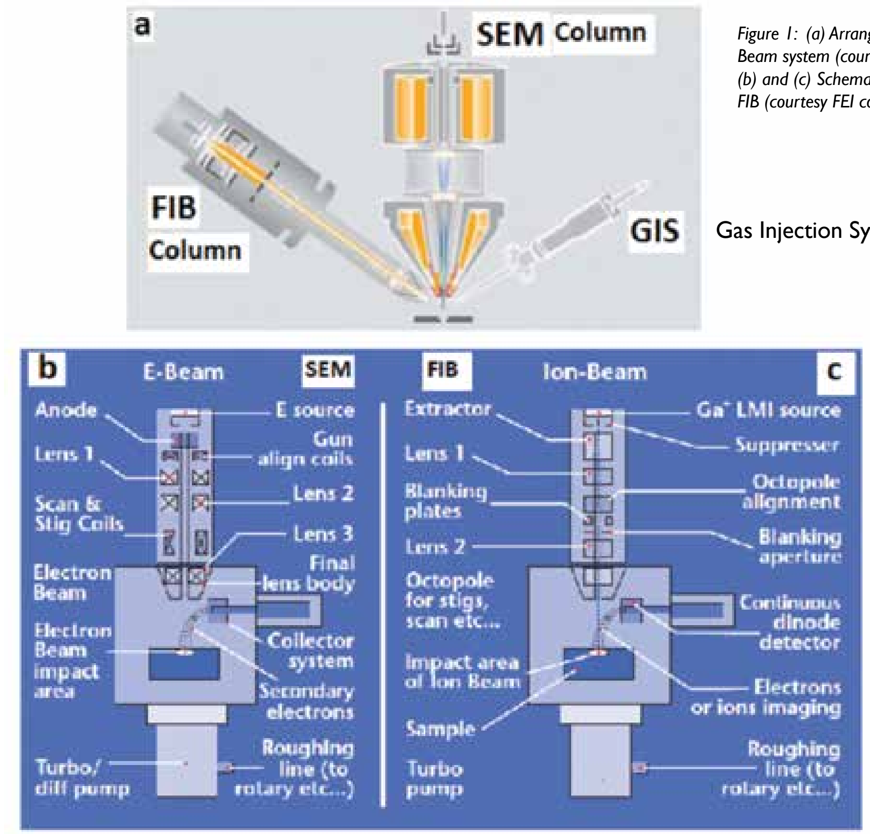
FIB systems have been produced commercially for approximately thirty years primarily for the semiconductor industry. Recently, earth science researchers have used FIB for studying various geological materials.

Introduction

FIB systems in principle operate in a similar fashion to a scanning electron microscope (SEM) except, rather than a beam of electrons, a beam of charged particles rastering across a selected region of the specimen and the ejected charged particles collect to construct a high resolution image of the surface. Two image modes are possible: secondary electron and secondary ions. These have different contrast properties. However, unlike SEM, the FIB microscope may produce a high current ion beam, which is used for 'in situ' sectioning of a selected volume.

FIB systems are provided with a liquid-metal ion source (LMIS), usually Ga* ions, that can be operated at low beam currents for imaging or at high beam currents for 'in situ' site-specific milling of materials from or depositing material onto a defined area whose dimensions are typically of the order of microns. For nonconductive samples the problem of charging up is resolved by using a low-energy electron gun for charge neutralisation. In this manner, by imaging with positive secondary ions - using the positive primary ion beam - even highly insulating samples may be imaged and milled without a conducting surface coating; as would be required in a SEM.

New generations of 'dual beam' FIB/SEM systems have been developed over the last two decades: these incorporate SEM for high resolution electron imaging and FIB for the 'in situ' milling of specific areas. The Ga* ions can then be used to mill (cut away) a specific site on the surface of a conductive or non conductive sample. A schematic diagram of the FIB-SEM column is illustrated in (Fig.1). The current paper is focused on demonstrating the uniqueness of FIB in cross-sectioning of samples on the micro-nano scale for transmission electron microscope (TEM), microelectronics and for geological applications.



Gas Injection System

Figure 1: (a) Arrangement of FIB/SEM Dual Beam system (courtesy Bischoff)
(b) and (c) Schematic drawings of SEM and FIB (courtesy FEI company).

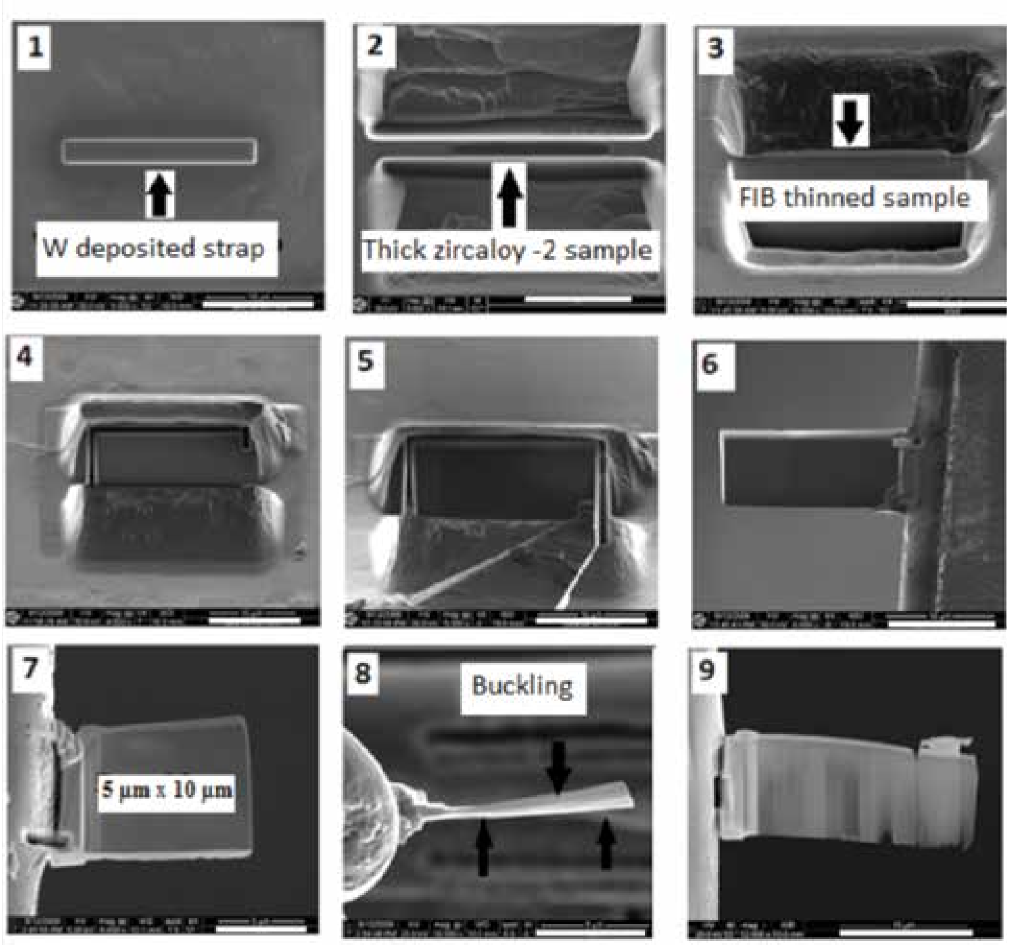


Figure 2: SEM micrographs showing the stages of preparing samples of zircaloy-2 by FIB machine: (1) Tungsten coating. (2 &3) Milling. (4) final stage of milling.(5) In-situ foil welding and extraction using the micromanipulator inside the FIB/SEM chamber. (6) Foil welding on copper lift-out-grid. (7,8,9) final stages of preparing TEM foil. Scale shown on micrographe 1-6 and 9 is 10 m . Scale shown on micrographs 7 and 8 is 5 m.

The Experiment

In the present work an FEI Quanta 3D FEG FIB-SEM was used. The SEM was operated at accelerating voltages from 0.2- 30kV; a probe current of up to 200nA was used for imaging. The FIB was operated at accelerating voltages from 2-30kV and a current of 1pA-65nA for fast material removal. It is worth mentioning that the FIB microscope is a fully automatic machine supplied with full software control of the sectioning stage. In the present work three samples were investigated:

1. A zircaloy-2 TEM sample; a small sheet of zircaloy-2 was mechanically polished and then, after a clean area was selected and coated with tungsten, milled with Ga* ions. The milling was monitored continuously by directly imaging the selected area by SEM. (Fig.2) shows the steps involved.(Fig.2-8) shows buckling of TEM foil due to beam heating and this effect was reduced by cutting off part of the foil. A sample thickness of less than 80 nm was achieved (Fig. 3).

2. An optical fiber with a diameter around 120µm was perforated with a square hole with dimension (30µm x 30µm) for use in a microelectronic experiment (Fig. 4).

3. Species of micro-fossil of **Benthonic Foraminifera** belongs to the upper cretaceous lower Paleocene age, from the Duhok region in the north of Iraq, were coated with carbon and

Figure 3: SEM micrograph of TEM foil (5 x 10 of zircaloy prepared by FIB. Thickness less than 80 nm was achieved.

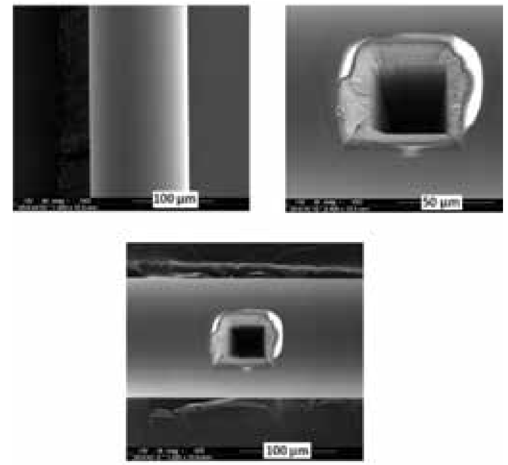
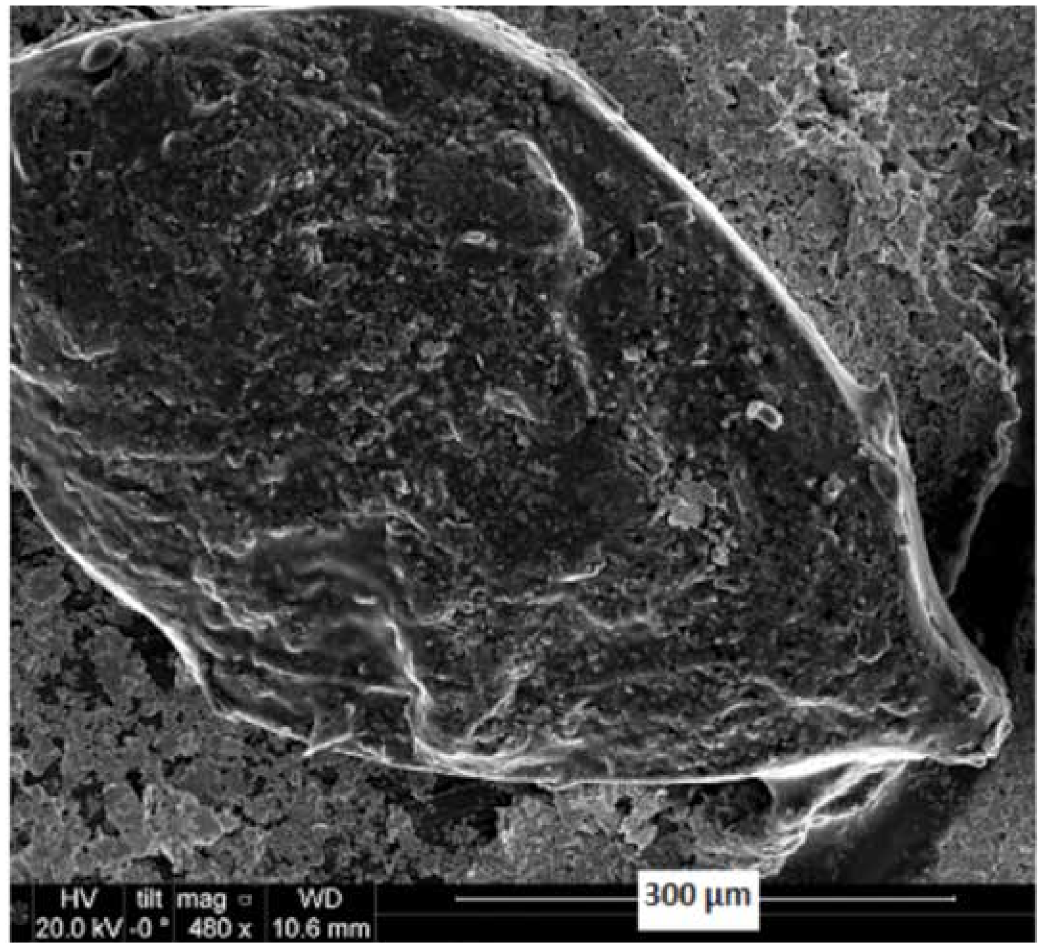


Figure 4: SEM micrograph: (a) Optical fiber with diameter about 120 . (b) and (c) A perforated square hole (30 x 30) made by FIB milling.

Figure 5: SEM micrograph of Benthonic Foraminifere belongs to the upper cretaceous lower paleocene age.



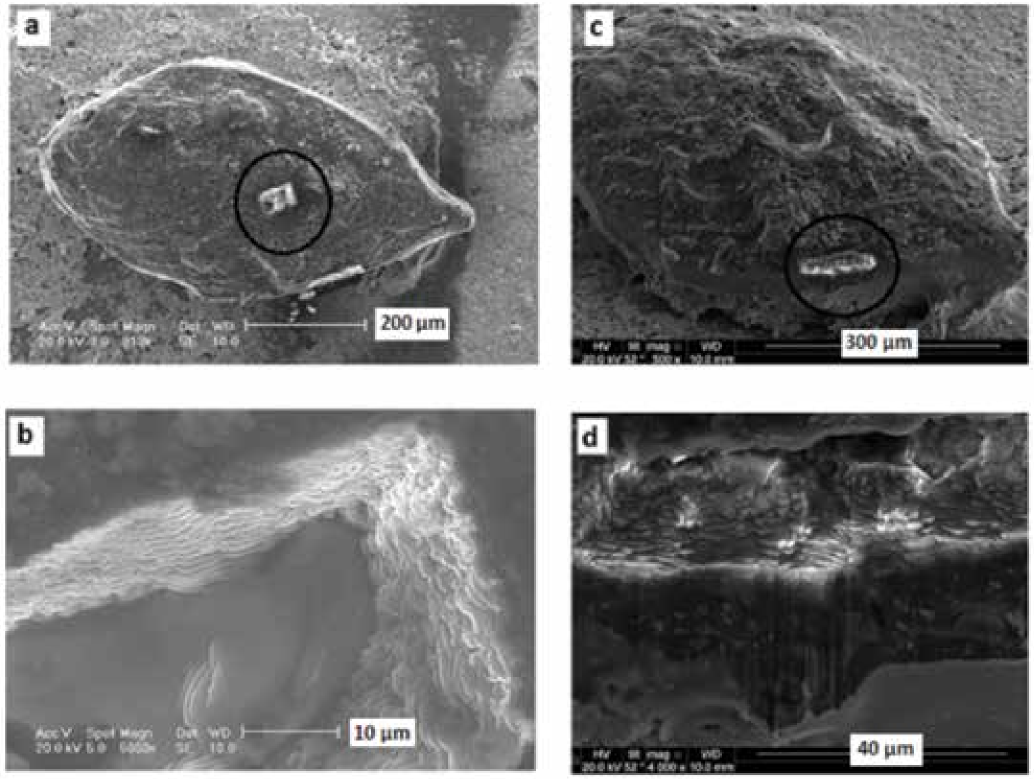


Figure 6: (a) and (c) SEM micrographs of in-situ sectioning of two regions in a microfossil (Benthonic Foraminifera) made by FIB/SEM machine. (b) and (d) Micrographs of the magnified images of the encircled cross sections show a dilation with a scale factors of about 16 and 8, respectively . The texture of the inner part of the fossil revealing the formation of multilayers with an average layer thickness of about 1.

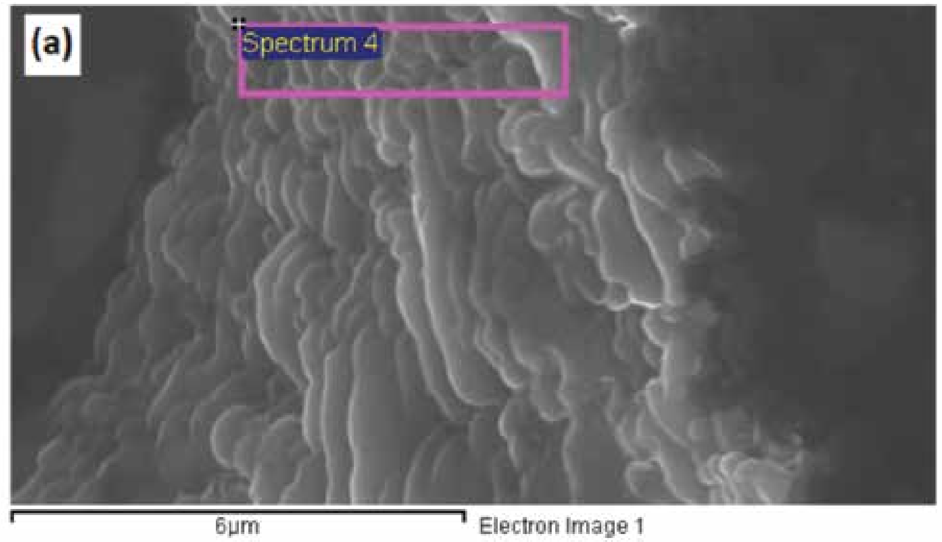
then milled using the FIB machine (Figs. 5 and 6). An elemental analysis of the exterior and interior parts of the fossil used EDS on a XL-30 (LaB_6) (Fig. 7).

Results

The present paper is aimed at three special problems: (i) sample preparation of a µm-sized foil of zircaloy-2 suitable for TEM, (ii) drilling a perforated hole in an optical fibre and (iii) sectioning of less than mm-sized micro fossils. The results are shown in (Fig.'s 1-6).

(i). Several techniques are available for preparing electron-transparent foils for TEM. The two most widely used techniques are electro-polishing (manual and automatic twin-jet) and PIPS (precise ion polishing system), which is conventional argon milling with a special mounting technique which is involves a certain amount of argon beam focusing. These two techniques have certain limitations such as (a) Difficulties in preparing thin foils of thickness in the nano scale required for certain objectives like EELS (electron energy loss spectroscopy) study and (b) The type of material and whether it is conductive or nonconductive.

In the present study using FIB, foils of zircaloy-2 have been prepared with a thickness less than 80 nm (Fig.3) suitable for EELS. A major drawback of FIB milling of TEM samples is the damage caused by Ga^+ ion beam bombardment near the foil surface; this can take the form of sample amorphization, point defect creation and dislocation formation. Buckling (Fig.2-8) of foil due to beam heating has been observed in some samples but this can be avoided by reducing the size of the foil. Radiation damage was not clearly observed in the zircaloy-2 sample though the energy transferred to a zirconium atom by the 30keV Ga^+ ion is higher than the threshold displacement energy of zirconium, E_d = 25 eV. This may be attributed to the affinity of zirconium



(b)

Element	Weight%	Atomic%
C K	5.56	10.11
O K	46.42	63.42
Al K	0.37	0.30
Si K	0.81	0.63
Cl K	0.90	0.55
K K	0.32	0.18
Ca K	45.14	24.62
Fe K	0.49	0.19
Totals	100.00	

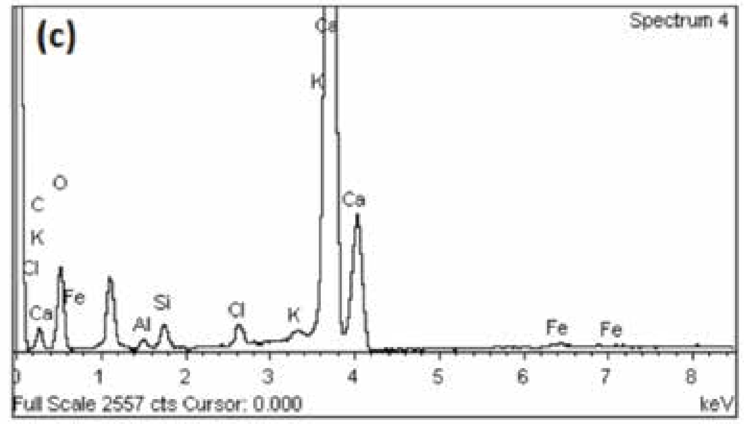


Figure 7: (a) Micrograph of a cross section in the inner part of a micro fossil (Benthonic Foraminifera). (b) and (c) EDS elemental analysis of the selected region in (a) showed that calcium and oxygen are the dominant elements in the composition of the fossil.

to interact and dissolve oxygen and nitrogen (present inside the FIB/SEM chamber) which are accommodated by successive occupation of octahedral holes in the HCP crystal and eventually suppressing radiation damage by preventing point defects mobility.

(ii). Drilling a µm-sized hole through an optical fiber enables experiments in the field of microelectronics. (Fig.4) shows a square hole (30 µm x 30 µm) made for passing a beam of laser light across the fiber. However, any shape of a hole with smaller dimensions is also achievable.

(iii). Sectioning of microfossils: to our knowledge no attempt has been made in the past to section and reveal the texture of the interiors of microfossils. In the present work ,we claim that, for the first time, FIB–SEM has been used for sectioning microfossils (**Benthonic Foraminifera**)(Fig.5). The multilayered texture of the interior was clearly observed, as shown in (Fig.6), this texture was formed and preserved since the upper cretaceous lower Paleocene age (from 55 to 65 million years ago). Elemental analysis, using EDS, of these fossils showed that the chemical composition of the interior part is dominated, as one would expect, by the presence of the two elements calcium and oxygen.

General conclusions

FIB-SEM, in particular a dual beam system, is a modern tool for µm and nm structure analysis, sectioning and sample preparation due to its flexibility.

A FIB is a fully automatic machine supplied with full software control of the sectioning stage.

FIB can be applied to many problems, but due to its operational cost and serial operation it is not suited to mass production.

FIB can handle most conductive and nonconductive materials without significant beam damage to samples. Compared with hard materials, soft materials take less time to mill.

Compared with electro-polishing and PIPS, FIB minimises mechanical and chemical damage of the material and allows preparation of a TEM foil at any area of interest within any polished section.

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In the summer of 2010 A.Z AzZubaydi joined the CEM group for another three months training (UNESCO-Qatari grant). During these two periods his work concentrated on the investigation of hydrides in zircaloys using electron microscopy. He is currently involved with FIB microscopy and its applications in nanotechnology.