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NINETY DEGREE TILT MODULE AND DOUBLE-SIDED STUB FOR SCANNING ELECTRON MICROSCOPY

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Abstract

A simple inexpensive device to achieve ninety degree tilt in some scanning electron microscopes (SEM) not equipped with this facility. A new method of safely mounting and studying both sides of the same fragile flat specimen, without the hazards of remounting; used in conjunction with this device.

KEY WORDS: Stage module, special stubs, fragile material, chick embryo, scanning electron microscopy.

Introduction

The International Scientific Instruments (I.S.I.) stages, except those for the mini-sem, are made up of a base into which various modules (depending upon specimen size and working distance) are fitted, (Figs. 1 and 2). It is a very versatile system and allows rapid changes in work without major mechanical operations. The Leicester University SEM Unit provides a service for the entire university and it is necessary that the instrument must not be used in such a way as to preclude its availability to other users. The concept of modifying and building interchangeable modules is therefore of prime importance. Neither of the I.S.I. SEM's within the unit are equipped with ninety degree tilt stages.

Fragile material is difficult if not impossible to safely remount for viewing with a SEM. Specimens with complex curvature (e.g., rat embryos) need to be mounted in as stable a position as possible and it is imperative to be able to tilt and then rotate the stub to select the required view. The device described in this paper achieves this by acting as a double tilt stage and incidentally produces black backgrounds behind the specimen.

Materials and Methods

Description of module

The ninety degree tilt module device, assembled (Fig. 3) and broken down (Fig. 4a,b) consists of a 6 mm pin (a), which fits into the basic microscope stage (this pin is not clamped); a base plate (b); with a flange to stop the module rotating (c) and a flange which provides an electron shadow area (d). Mounted on the base is the main body (e) which consists of a square pillar of brass; this provides the weight to keep the device steady and also acts as the bearing for one axle (f) and main shaft (g). A rubber tyred wheel (h) is driven by the rotation of the base of the main stage of the microscope. This rubber tyred wheel drives, by means of the gear train, the geared driver (i) and the geared driven upper wheel (j) locked on by screw (k), the main shaft (g).

The specimen stub holder (l) with a socket

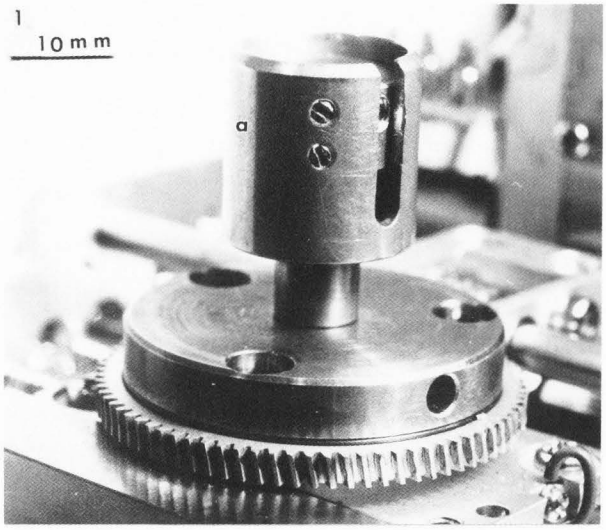


Fig. 1. Close up of lower stage of I.S.I. D.S.130 SEM with the standard stub holder for I.S.I. and Cambridge Scientific Instruments' stubs.(a)

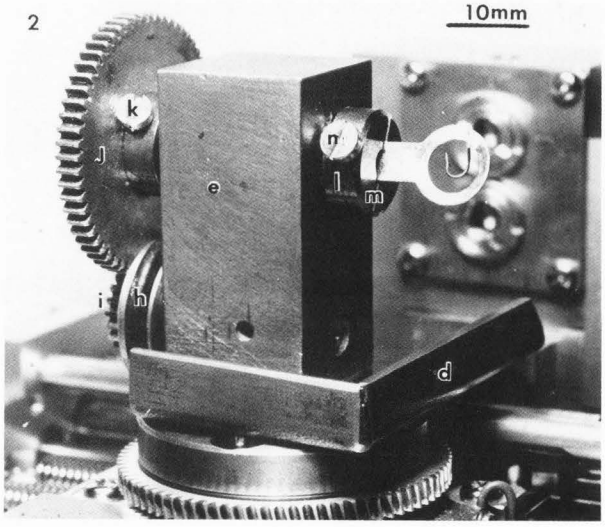


Fig. 2. Close up of lower stage of the I.S.I. D.S.130 SEM with ninety degree tilt module. Showing double sided stub in slot, specimen is a fish hook. Main body (e); electron shadow flange (d); rubber tyred wheel (h); geared driver (i); geared driven upper wheel (j) with lock screw (k); stub holder (l); stub lock screw (n); slot (m). The shanks of the Cambridge Scientific Instruments' stubs and the domed stubs fit into the central aperture.

for standard Cambridge stubs and a slot for double-sided stubs (m) and a lock screw (n), is attached to the main shaft (g). The axle (f) is held by a lock screw (o) and any slack and play is taken up by spacers (p). The rubber tyre is a vacuum "o" ring and the module is easily dismantled for cleaning and reassembling if required prior to insertion into the microscope.

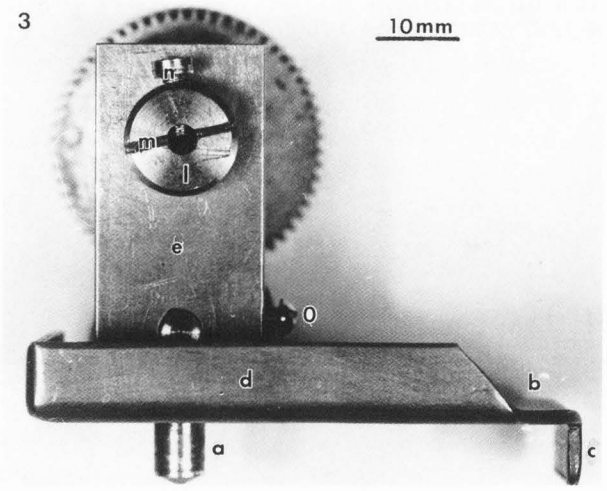


Fig. 3. The assembled ninety degree tilt module showing:- 6 mm pin (a); base plate (b); locking flange (c); electron shadow flange (d); main body (e); lock screw (k); geared driven upper wheel(j); stub holder (**O**) stub lock screw (n) slot (m).

A scale drawing has not been included because the module must be modified to fit each model of microscope.

Description of double-sided stub

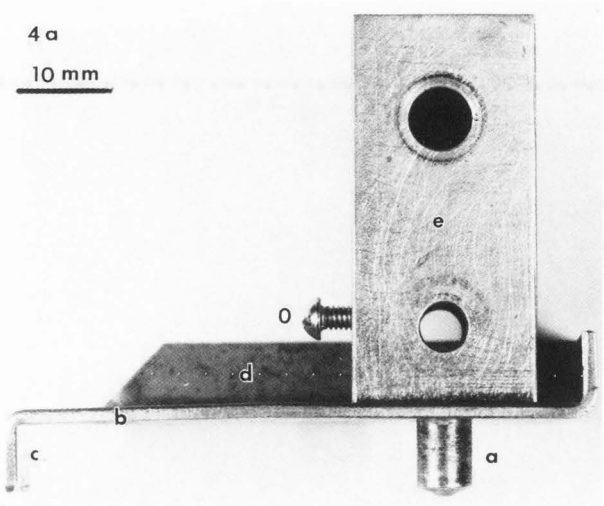
There are two types of double-sided stubs; a simple washer and a solder tag (Fig. 5a,b,c,d). The simple washer of required dimensions used in some cases (McTurk and Summerton, 1986), is the best method as the available fractured transverse section for profile study can be selected after mounting. This method was particularly useful for looking at both sides of the same flat specimen (i.e., chicken embryos) (Figs. 5 and 6). The embryos are flat, very fragile sheets and in transverse sections structures can be traced from one side of the embryo to the other side.

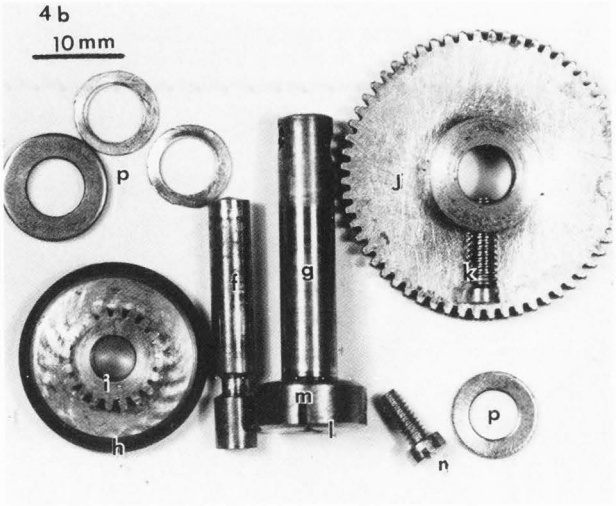
Although specimens cannot be rotated about an axis parallel to the beam while in the microscope, scan rotation can be used to correct image orientation prior to micro-photography.

The solder tag stub is more suitable for very fragile and smaller specimens. Solder Tags are obtainable in four sizes from: RS, Lammas Road, Weldon Industrial Estate, Corby,Northants. NN179RS, U.K.

Chick embryo

Early chick embryos were incubated at 37^{o}C for 18 - 20 hours until stage 4 - 6, (Hamburger and Hamilton, 1951). They were then removed from the shells and fixed in a glutaraldehyde-formaldehyde solution buffered with cacodylate. The embryos were then placed in a cacodylate buffer followed by 1% solution of OsO_4 for 30 minutes and dehydrated in an ascending series of ethanol/water to 100% ethanol. They were transferred to 100% acetone, critical point dried using acetone replacement and mounted on the stub using a colloidal silver paint. The specimens were coated with 15 nm of gold and examined in an I.S.I. DS130 SEM.





Figs. 4a and 4b. The components stripped for cleaning. 6 mm pin (a); base plate (b); locking flange (c); electron shadow flange (d); main body (e); lock screw (k); axle (f); rubber tyred wheel (h); geared driver (i); geared driven upper wheel (j); shaft lock screw (o); shaft (g); stub holder (l); stub lock screw (n); slot (m); spacers (p).

Discussion

The advantages of the module over the standard stub holders are numerous. Besides the ability to tilt the stub +/- 30 degrees from 90 degrees to the beam, it is possible to rotate it through 360 degrees. This allows any desired position to be achieved and it is also possible to get high contrast relief of the specimen's profile. The built-in flange (d) (Fig. 4a) provides an electron shadow area for small specimens; for large or awkwardly mounted specimens the same effect is achieved by adding an adhesive copper foil (source is also R.S. as previously given) at a distance below the specimen. The foil is bent to produce a vertical shield out of the scanned view which produces an electron shadow. Because the smooth copper foil is flexible it can be moulded to give the required results and can be renewed with ease when the surface ceases to be smooth. Both the above features are used to produce plates of standard views of Ostracods. This is especially useful when the specimen is part of a shaped and mounted block of rock. The ease of handling the double-sided stub is also an advantage. Eucentric double tilt would be possible using this device but would require complicated and expensive apparatus.

The working distances of the specimen shown varies between 15 and 20 mm. The range of possible working distances within the DS130 SEM is 8mm to 53 mm and the most suitable is chosen. The double-sided stub can be mounted in a modified standard holder and one side studied in the normal way for high resolution results. It should be possible to mount small double-sided stubs in the very high resolution top stage.

Standard type stubs are also used with the 90 degrees tilt module and if the stub used has a curved (domed) type instead of a flat surface, the foreground and background produced by the stub can be removed from the view and foreign body interference (e.g. hairs, glue, or dust) is reduced. The domed stubs of varying size are mainly used for Ostracods and other micro-palaeontology specimens. It is recommended that only one specimen be mounted per stub of this type in order to obtain micrographs of publication quality.

Specimens are mounted using silver colloidal paint. Coating is carried out with the specimen vertically mounted and this is sufficient for the I.S.I. DS 130. Double coating is also used but takes longer.

Acknowledgements

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References

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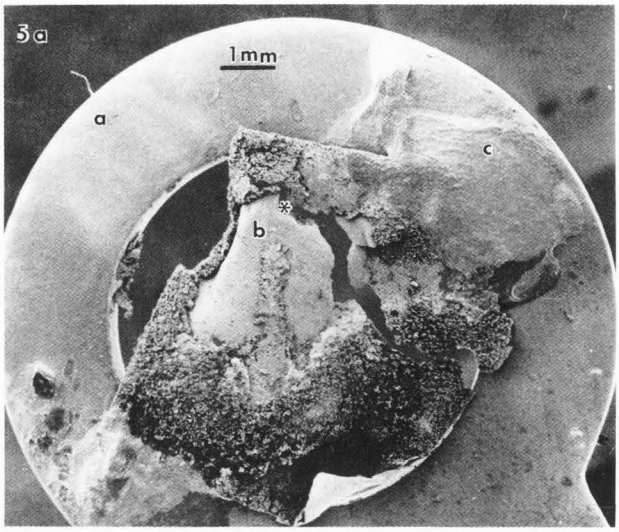
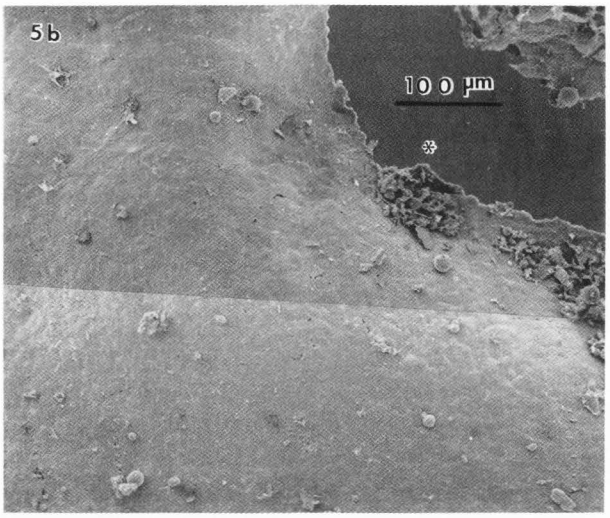
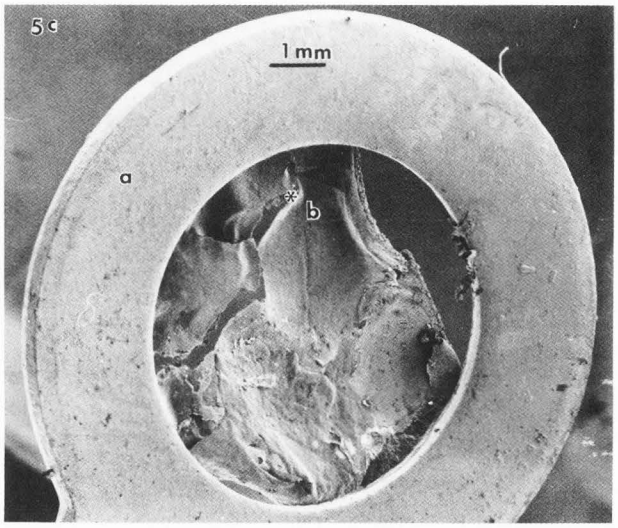
2. McTurk GLC, Summerton DJ. (1986). A new double-sided stub for scanning electron microscopy. J. Anatomy, 146, 267-268.

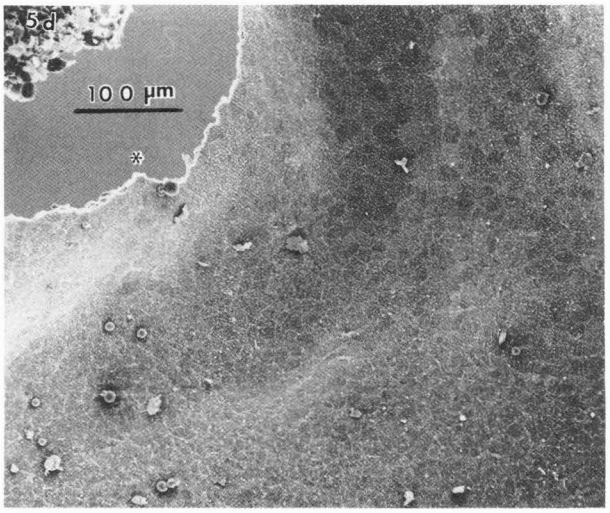
Discussion with Reviewers

T. Johnson: What is the dimension between the sample surface and the ISI stage platform? If it can be maintained at 25 mm the inherent eucentricity of the stage will be unimpaired.
Author: The height between the centre of the stub and the base of the microscope in this study is 30 mm. The height of the specimen will vary on orientation and mounting position on the stub.









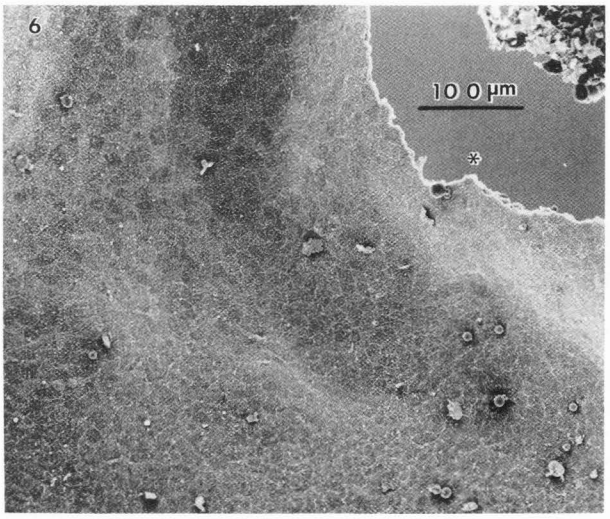


Fig. 5. Views of both sides of dissected stage 4 chicken embryo, mounted on washers: a) metal washer, b) chick embryo, c) silver dag mounting media.
Fig. 5a. Endoderm.
Fig. 5b. A higher magnification of Fig. 5a.*
Fig. 5c. The same metal stub and embryo viewed from the reverse, Ectoderm, side.
Fig. 5d. A higher magnification of Fig. 5c.*

Fig. 6. Same frame as Fig. 5d printed with the photographic negative reversed in the enlarger. This makes the direct comparison of each side of the specimen easier. Compare Fig. 6 with Fig. 5b. The * is the same structure on both sides of the specimen.