# SILICONE RUBBER CASTS OF SILICIFIED PLANTS FROM THE CRETACEOUS OF SUDAN

*by* JOAN WATSON *and* K. L. ALVIN

ABSTRACT. Silicified plant fragments, including the conifer *Frenelopsis* from the Lower Cretaceous of Sudan, are preserved as internal and external moulds. Low viscosity silicone rubber has been used to obtain casts showing fine details which may be studied by scanning electron microscopy.

WORK in progress on a revision of conifers belonging to the genus *Frenelopsis* Schenk has led us to re-examine some silicified material from the Nubian Sandstone of Eastern Darfur, Sudan. The material, which is in the collection of the British Museum (Natural History), was studied by Edwards (1926) and includes some shoots which he described and figured as *F. hoheneggeri* (Ettingshausen). The plants are entirely replaced by silica, none of the original plant material remaining. In the absence of a cuticle, we tried to obtain rubber impressions for use in the scanning electron microscope. The results obtained revealed exquisite details and showed the preservation to be of a most unusual and interesting kind. The taxonomy and morphology will be dealt with elsewhere. The purpose of this paper is to demonstrate our replica technique in conjunction with this unusual and exceptionally fine preservation.

## MATERIAL

The matrix is a silicified grit of highly variable grain size. There are nine pieces in the collection, some from loose weathered blocks, others collected *in situ*. Few details are given by Edwards (1926) about the geological occurrence of the specimens but the reported discussion following the original reading of his paper indicates that they occur in association with 'soda-rich volcanic rocks' of later age. One of the specimens (B.M. (N.H.) V.21708) is shown in Plate 96, fig. 1 at natural size.

Besides the *Frenelopsis* shoots there are cone remains, pieces of wood, and fragments of the fern *Weichselia reticulata* (Stokes and Webb). At first sight the shoots on the weathered surface appear to show certain details of the external surface of the leaves and internodes, such as rows of stomata and cell outlines, and these were figured by Edwards (1926, p. 96, fig. 4) using line drawings. However, when a piece of shoot from the rather crumbly unweathered material is mounted and examined under the SEM the result is rather surprising (Pl. 97, figs. 1 and 2). Clearly this is not the external surface of the shoot but silica infillings of the cells of the epidermis with gaps where the cell walls were. It can be seen in Plate 97, fig. 3 that the silica is in the form of euhedral quartz crystals up to about 10 $\mu$m in length.

## METHODS

We obtained casts of the exposed plant surfaces using a low-viscosity silicone rubber. The rubber, called 'Silflo', is a two-part impression material used in dentistry and

[Palaeontology, Vol. 19, Part 4, 1976, pp. 641–650, pls. 96–99.]

made by Flexico Developments Ltd. It is extremely easy and quick to use and the few problems which arise are easily overcome. The rubber is quickly mixed with its catalyst on a tile by means of a small spatula and then scooped onto the required area, again working fairly quickly. The rubber is white and the catalyst either colourless or dyed green; the green is preferred as it is much easier to see when mixing is complete. The amount of catalyst to use must be established by trial and error as it tends to deteriorate with age. Too much causes the rubber to start setting before it is put onto the rock surface and this results in loss of detail in the cast. Another result of too much catalyst is that it can come to the surface of the specimen during coating for the SEM and the gold mixes with it to form a sludge. We recommend always leaving the rubber impressions for twenty-four hours before coating, to ensure that the catalyst has entirely evaporated.

There is little trouble with air bubbles as they mostly rise to the surface quite quickly. However, in a particularly cavernous specimen such as the cone in Plate 99, fig. 1 gentle poking of the liquid rubber in the cavities with a needle helps the process.

We usually prefer to discard the first cast taken from a particular area, using it as a method of cleaning the surface. It is most satisfactory to work with small areas at a time; usually 1 $cm^2$ is sufficient for a SEM mount. Larger casts can be built up with several batches of freshly mixed Silflo as it bonds to itself completely. Such a large cast is shown in Plate 96, fig. 2, coated with aluminium to increase contrast. For SEM work there is the problem of sticking the smooth shiny surface to the metal stub. This was overcome by pressing a scrap of filter paper onto the surface of the unset rubber. The resulting specimen is easily cut to size with scissors and mounted by means of double-sided 'Sellotape'. The specimens were mostly coated with gold using a 'Rota Cota', but high relief specimens such as the cone-scale (Pl. 99, fig. 3) required sputter coating for satisfactory results.

The only other minor problem encountered is that sometimes the rubber has a finely wrinkled surface as shown in Plate 99, fig. 6. It is only detected at magnifications around $\times 2000$ and does not seem to affect the useful detail but clearly we would prefer to eliminate it. It is certainly a feature of the Silflo, not the rock, as we have reproduced it in impressions of fingerprints, cardboard, and a wooden surface. However, we are not yet able consistently to reproduce or eliminate it.

---

## EXPLANATION OF PLATE 96

All British Museum (Natural History) specimen numbers.

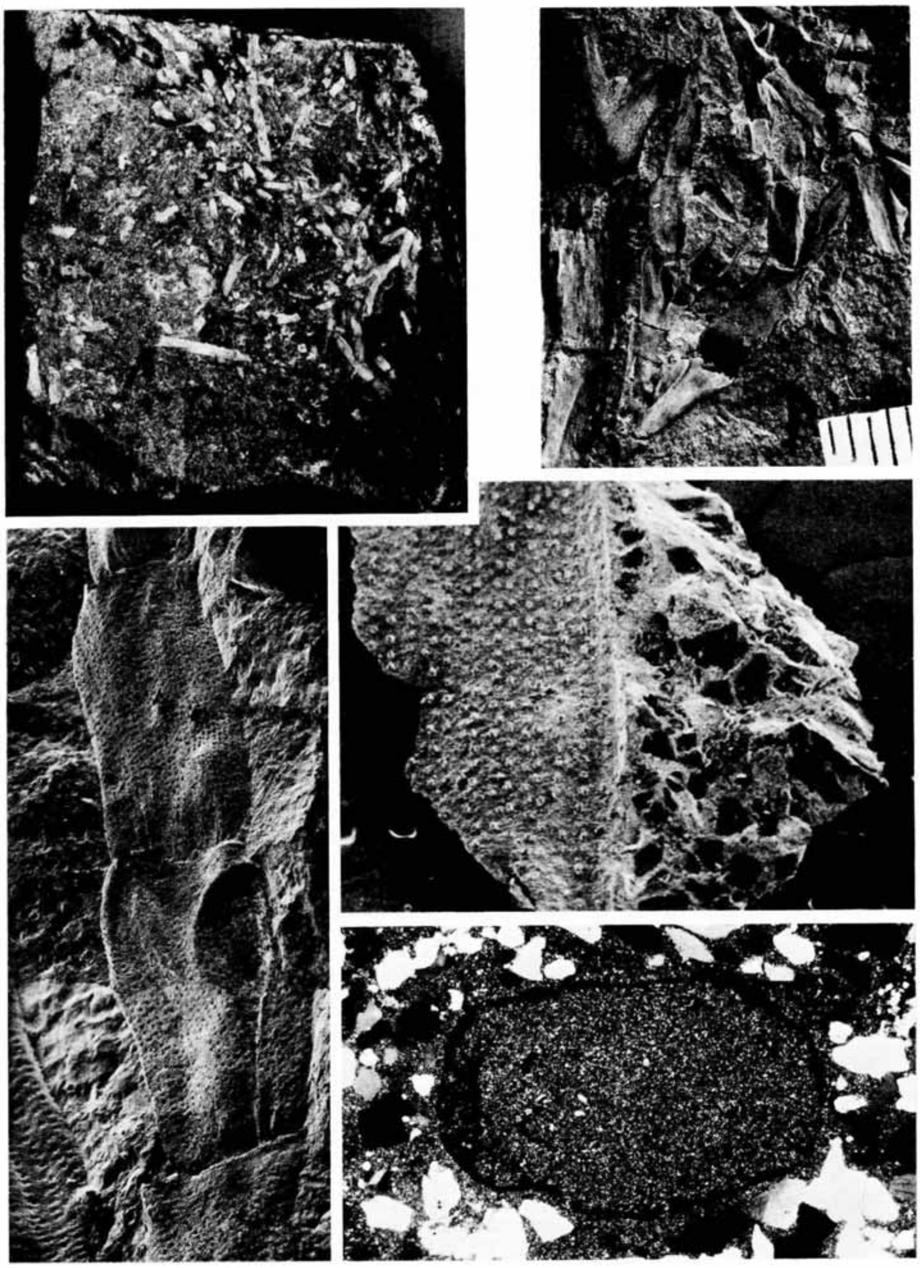
Fig. 1. Silicified specimen used to prepare several of the rubber casts figured in this paper, $\times 1$. V.21708.

Fig. 2. Silicone rubber cast of *Frenelopsis* shoots, coated with aluminium to give good contrast, $\times 3$. Prepared from the bottom right-hand corner of V.21708.

Fig. 3. SEM montage of silicone rubber cast of *Frenelopsis* shoot, $\times 15$. Cast prepared from same area of V.21708 as fig. 2.

Fig. 4. SEM of silica specimen showing grit matrix on right and external mould of *Frenelopsis* on left, $\times 50$. From V.21708.

Fig. 5. Transverse section of little flattened *Frenelopsis* shoot seen under crossed polars, showing cryptocrystalline silica replacing plant material and cementing detrital grains. Powdery silica replacement of cuticle does not survive sectioning and now shows as a gap between external mould and steinkern (black ring), $\times 50$. Thin section taken from V.21708.



1 2 3 4 5

WATSON and ALVIN, *Frenelopsis*

It should also be mentioned in relation to SEM studies that the thickness of the rubber specimen may produce too short a working distance and to avoid this it is advisable to use stubs with thin tops (galvanized roofing nails are perfectly adequate).

## RESULTS

When a shoot surface such as shown in Plate 97, figs. 1-3 is used to obtain a rubber impression the result is as shown in Plate 97, figs. 5 and 6. This looks to us like a cast of the inner surface of the cuticle but may include additional portions of the anticlinal walls of the epidermis, possibly representing the cutinized parts of these walls. We have not seen guard cells in such an internal cast, possibly because they were too thinly cutinized.

A cast of the external surface of the shoot is obtained by taking an impression from the matrix in a place where the silica shoots have been removed as on the left-hand side of Plate 96, fig. 4. In contrast to the coarseness of the grit, with grains up to 1 cm across, the matrix immediately adjacent to each plant specimen is very fine grained and forms a crust which is a perfect external mould of the plant. All such surfaces show a pattern of protruberances (Pl. 98, fig. 1; Pl. 99, fig. 2) representing silica plugs which crystallized inside the stomatal pits and it is in specimens where these are intact that the finest details survive.

Thus, we have fine-grained silica moulds, both internal and external, of the outer part of the epidermis with a gap between them. This is a common enough situation with calcareous invertebrate fossils but in this case the cryptocrystalline silica has preserved details at the cellular level in both the steinkern and the external mould. The gap between the two is filled with a fine white powder (also crystalline silica) and can be seen on newly exposed surfaces but it does not survive handling or the making of a thin section. Thin sections (Pl. 96, fig. 5) show that the interstices between the detrital grains are filled with the same cryptocrystalline siliceous cement as that forming the internal and external moulds of the plants.

Text-fig. 1 is a diagrammatic representation of the state of preservation together with the relationships of the various surfaces of silica and Silflo. The reconstruction of the situation depicted in text-fig. 1 was greatly assisted when a portion of steinkern was pulled off partially embedded in the Silflo cast (Pl. 98, fig. 3). Thus where the Silflo has seeped into the gap between the two moulds we get an approximation to

---

### EXPLANATION OF PLATE 97

All scanning electron micrographs.

Fig. 1. Silica surface which forms internal mould of *Frenelopsis* cuticle, ×150. V.17224.

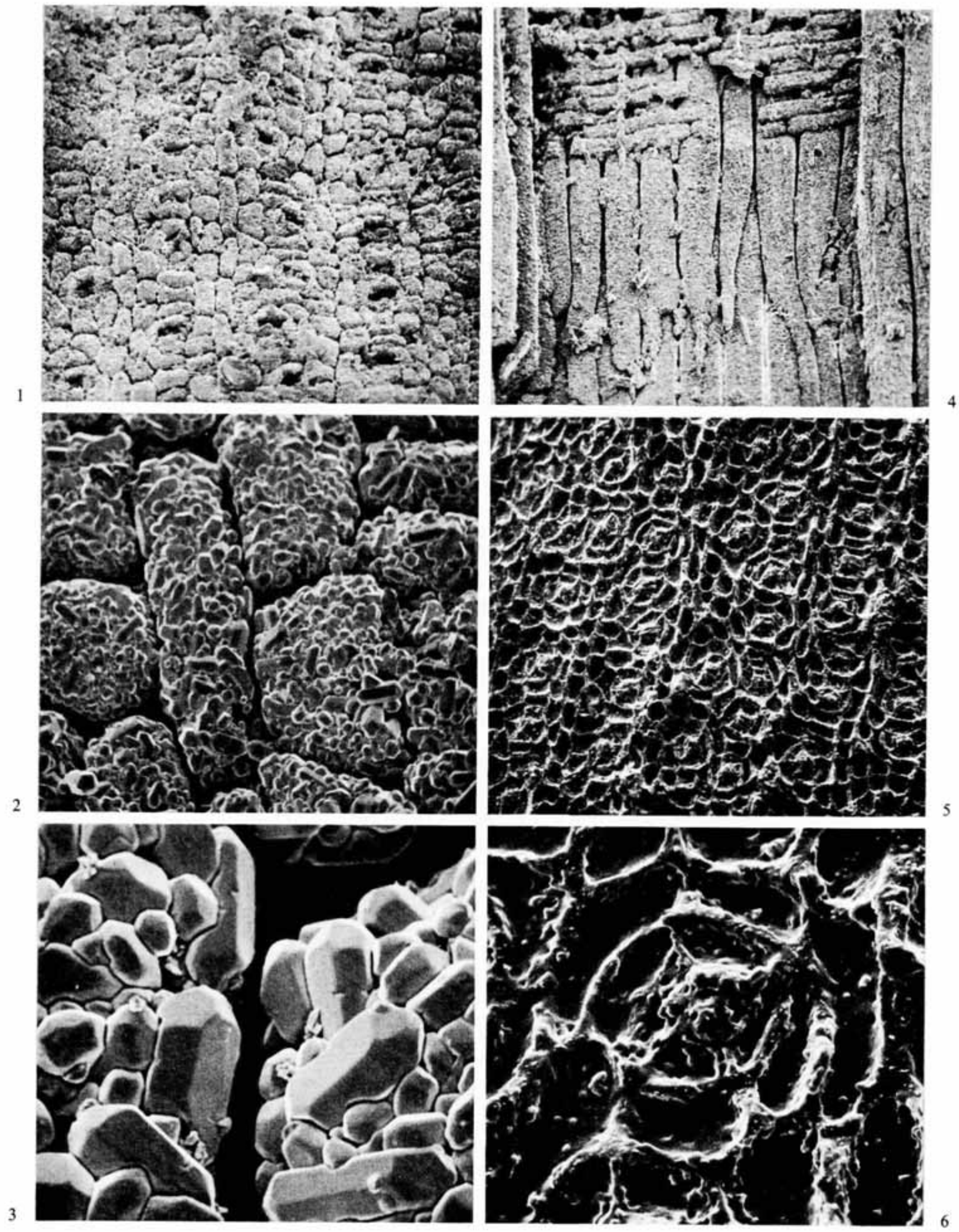
Fig. 2. Silica infillings of epidermal cell lumina, ×750. V.17224.

Fig. 3. Detail of same specimen as fig. 2 showing euhedral quartz crystals and gaps where cell walls were, ×3750.

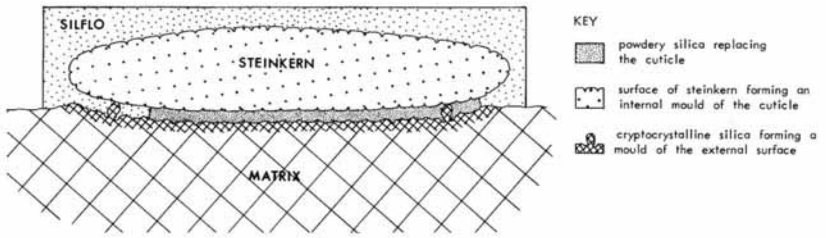
Fig. 4. Silica infillings of tracheids and ray cells, ×150. V.21706.

Fig. 5. Silicone rubber cast taken from surface such as seen in fig. 1, ×100. Prepared from V.21708.

Fig. 6. Silicone rubber cast of internal surface of single stoma, ×500. V.21708.




WATSON and ALVIN, *Frenelopsis*




TEXT-FIG. 1. Diagrammatic representation of transverse section of stem in relation to matrix and rubber casts.

the thickness of the cuticle (Pl. 98, fig. 2) disregarding any small degree of compression which may have occurred or additional cell wall material which may be represented.

The casts of the external surface show numerous fine details and several examples are shown in Plates 96, 98, and 99. Plate 98, fig. 4 shows the stomatal arrangement and the papillate surface of the epidermis; Plate 98, fig. 5 shows what might possibly be the upper surface of the guard cells and stomatal aperture; Plate 99, fig. 6 shows the stomatal pit and subsidiary cell papillae. All the *Frenelopsis* shoots appear to be preserved in exactly the same way.

The cones are preserved in an essentially similar manner to the shoots but in the one large cone the steinkerns of individual scales are all broken or missing. Thus there are only external moulds of silica which crystallized around all the scales, penetrating deeply between them. These moulds therefore give accurate casts of the original cone scales showing beautiful details of the surface including delicate hairs along the margin (Pl. 99, fig. 4).

The wood fragments present in the material are preserved somewhat differently, being represented by silica infillings of the cell lumina. Plate 97, fig. 4 shows infillings of tracheids with tapered ends and medullary ray cells, everywhere with gaps where the cell walls were. Edwards (1926) figured growth rings in the wood and a pattern of pitting in the tracheids. We examined the thin sections used by Edwards and found

---

EXPLANATION OF PLATE 98

All scanning electron micrographs.

Fig. 1. Silica surface which forms external mould of *Frenelopsis* shoot showing protruberances which represent infillings of stomatal pits, ×750. V.21708.

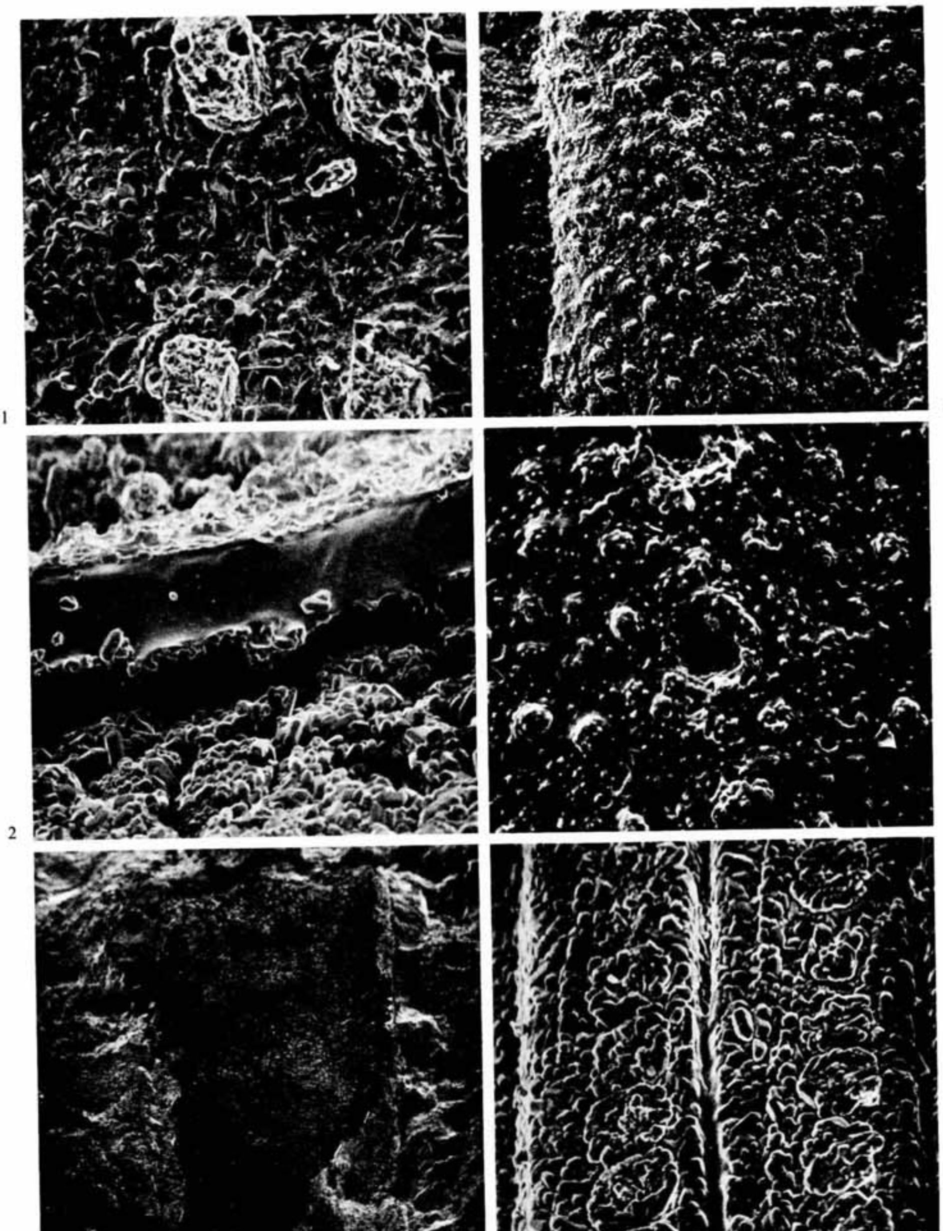
Fig. 2. Edge of rubber cast of *Frenelopsis* cuticle obtained when silicone rubber seeps between external mould and steinkern, ×750, tilted at 55°. V.17224.

Fig. 3. Steinkern of a *Frenelopsis* internode partially enclosed in silicone rubber impression; 'rubber cuticle' overlapping it is that shown in fig. 2, ×40. V.17224.

Fig. 4. Surface of rubber cast of *Frenelopsis* internode showing stomata and papillae, ×200. V.17224.

Fig. 5. Detail of specimen shown in fig. 4, ×500.

Fig. 6. Silica infillings of two tracheids showing pits, ×750. V.21706.




WATSON and ALVIN, *Frenelopsis*

that both the cells and the pits are clearly outlined by 'Carborundum' (or other abrasive powder) filling the gaps mentioned above. We have also located pits with the SEM (Pl. 98, fig. 6) but the exact state of their preservation is unclear. They appear lens-shaped and we presume that each represents a silica infilling of the pit chambers of a pit pair attached to the infilling of one of the tracheids. However, we have been unable to find corresponding depressions in other tracheids.

## DISCUSSION

The sequence of preservation can only be surmised but clearly in the case of the *Frenelopsis* shoots the fine-grained secondary silica, both internal and external, must have crystallized whilst the cuticle was still intact, the cuticle itself being removed at a later stage. We know, from our studies of compressed specimens of *Frenelopsis* from elsewhere, that the cuticle is often leathery and resistant, and it is easy to picture a stage in decay where the shoot consisted only of cylinders of cuticle, joined together at the nodes, trapped in the detrital matrix. Thin sections show detrital grains inside a few shoots. This would accord with a damaged cylinder of cuticle allowing entry of grains from the unconsolidated sediment before becoming entombed in the crystallizing secondary silica.

The use of low-viscosity silicone rubber casts of fossil plants for SEM studies has been briefly reported (Dilcher 1974) as has the use of latex rubber (Chaloner and Gay 1973). We find that Silflo has several distinct advantages over latex rubber. It is very quick to use; there is no water in the mixture and therefore no damage even to a clay matrix; there is no detectable shrinkage even over a period of months; air-bubble problems are reduced to a minimum.

*Acknowledgements*. The scanning electron microscopy was carried out partly on a Cambridge 'Stereoscan' in the Department of Textile Technology, University of Manchester Institute of Science and Technology, and partly on a similar instrument in the Department of Botany, Imperial College, London.

---

### EXPLANATION OF PLATE 99

Fig. 1. Silica mould of female cone, ×5. V.21704.

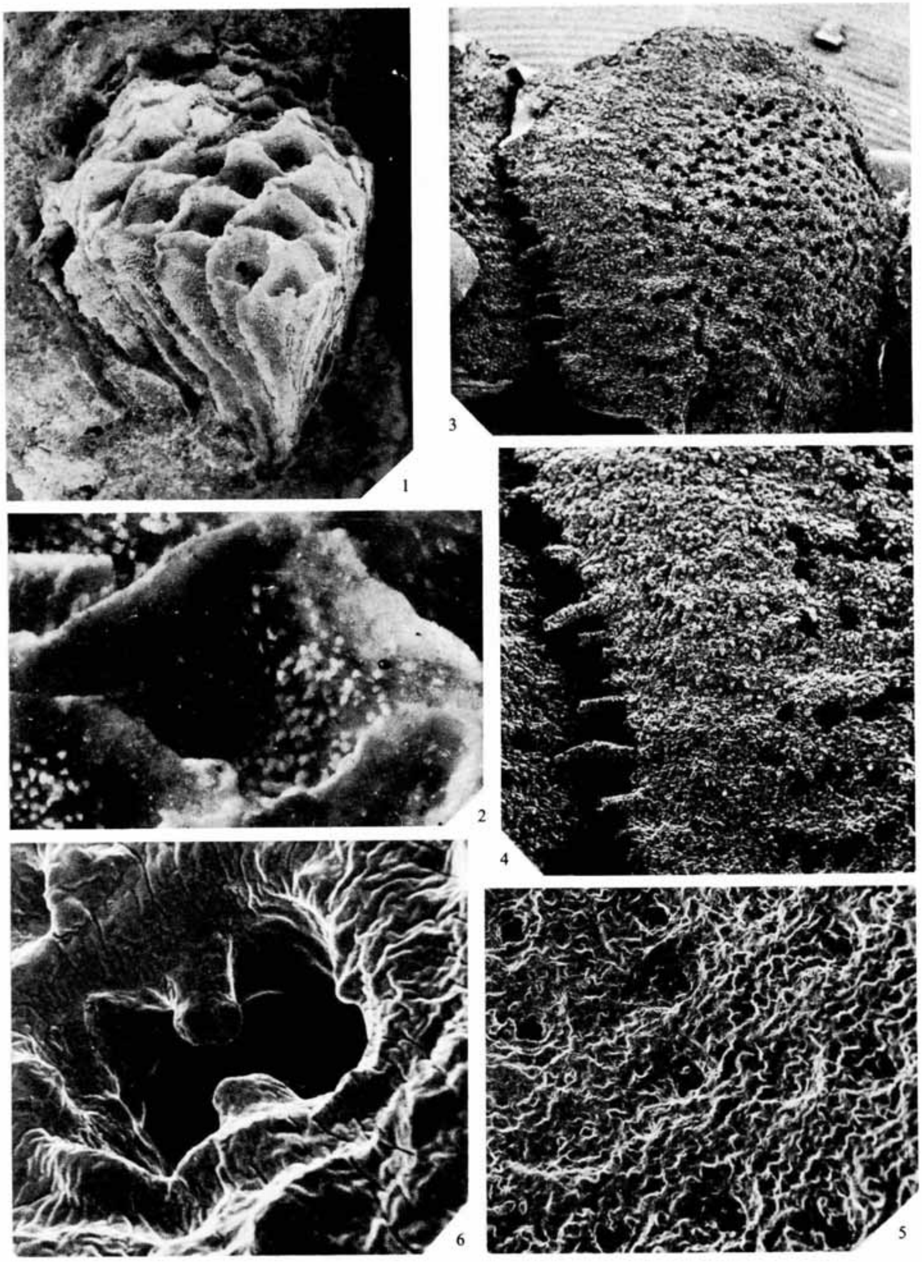
Fig. 2. External mould of single cone scale showing pegs which are infillings of stomatal pits, ×27. V.21704.

Fig. 3. Scanning electron micrograph of rubber cast of cone scale obtained from mould seen in figs. 1 and 2, ×40.

Fig. 4. Detail of same cone scale showing fringe of hairs on margin, ×80.

Fig. 5. Scanning electron micrograph of rubber cast of *Frenelopsis* internode surface. This is an example of specimens with wrinkled surface mentioned in text; wrinkling not detected at this low magnification (see fig. 6), ×225. Prepared from V.21708.

Fig. 6. Single stoma from specimen seen in fig. 5, showing well-preserved papillae; fine wrinkling seen at this higher magnification, ×2250.



WATSON and ALVIN, *Frenelopsis*

## REFERENCES

CHALONER, W. G. and GAY, M. M. 1973. Scanning electron microscopy of latex casts of fossil plant impressions. *Palaeontology*, **16**, 645–649.

DILCHER, D. L. 1974. Approaches to identification of angiosperm leaf remains. *Bot. Rev.* **40**, 1–157.

EDWARDS, W. N. 1926. Fossil plants from the Nubian Sandstone of Eastern Darfur. *Q. Jl geol. Soc. Lond.* **82**, 94–100.

JOAN WATSON

Department of Geology
The University
Manchester M13 9PL

K. L. ALVIN

Department of Botany and Plant Technology
Imperial College of Science and Technology
London SW7 2AZ

Typescript received 27 August 1975
Revised typescript received 19 November 1975