

Left: Fossilised foram, partially embedded in Kimmeridge Clay lamina. Right: A foram that has been cleaned of clay. Species to be determined.

**N**o, I am not referring to me and my collaborator, Chris. Rather, in this edition of SEM Diaries I describe the work that Chris is doing with some of the tiny fossils from Kimmeridge Clay, and how I am supporting him with my SEM.

I first came across Chris at a lecture he gave to our local U3A mineralogy group, as mentioned in SEM Diaries - 32. In that article I described imaging foraminifera (informally abbreviated as forams) he had dredged up from the Southern Ocean. In this one I shall describe working with material from much nearer home.

As many will know, Dorset has a very varied geology, and a history of fossil hunting going back all the way to Mary Anning. Among the various rock types, such as various sandstones, Portland stone, Inferior Oolite, Fullers' Earth rock, Forest Marble etc. there is something called the Kimmeridge Clay Formation of late Jurassic age (150-157 million years ago). While the strata of this formation extend well beyond the coastal village after which it is now named, or even beyond the county of Dorset, Kimmeridge is still just about the best place to observe this material and to collect fossils from it. Indeed, there has even been a modern museum erected (the "Etches Collection", a registered charity) at the top of the village, dedicated to the public display of the Kimmeridge Clay Formation fossils. The collection is curated by Steve Etches.

Steve is still active in adding to this collection, and while he spends his time preparing large specimens from great blocks of Kimmeridge Clay, Chris is allowed to take some samples of "off-cuts" and sets to work extracting microfossils from these, in collaboration with others. The specimens he collects are mainly foraminifera, but he has found other material such as a "leg" joint of a sea spider and the fossil hook from the tentacle of a Jurassic age squid.

At the time of his first visit with samples from Kimmeridge Clay Chris did not possess a microscope, so together we sorted through some of his material and succeeded in finding a small number of forams of different species. I then stuck these on stubs and imaged them on my SEM. After this, I presented Chris with a simple stereo microscope, and a little later I provided him with six stubs and some sticky tabs so that he could lay out his forams himself. (Delegation is good!) A short while later he returned with all six stubs laid out with anything up to about 30 forams on each. It was at this stage that I decided that I really needed to teach Chris how to use my SEM! Much to his surprise I sat him down at the desk and gave him some instruction. That day we worked on imaging together, but when he returned next I left him much to his own devices. Once the specimens are loaded and brought to the correct working



Squid hook



Juvenile oyster fossil



A mystery. Possibly a fossilised megaspore grain (perhaps *Horstisporites sp?*).

distance, there is only so much damage that a beginner can inflict on the SEM!

Opposite are three images made from non-foram samples on Chris's set of six stubs.

One day Chris emailed me, attaching a photo of a fragment of fossilised reptilian bones, probably ribs or phalanges (limb digit bones), and asking if I would like to try polishing them to see what a crossand longitudinal-section might look like. This sounded interesting, so of course I said yes, and a few days later he brought his sample round. This appeared to be two fossil bones, still partly encased in Kimmeridge clav. With minimal instruction from Chris I was left with the job of separating the fossils from the clay and each other.

Unlike Cornish China Clay, Kimmeridge clay is more like a rock. It does not soften in water - much - so it was up to me to discover how much brute force was needed to separate and preserve the fossilised bones, while removing the outer shell of clay. One trick well used by palaeontologists is to soak the specimen in water to which a small amount of Calgon has been added. I did this, and while I was at it I put the container in a water bath in my ultrasonic cleaner. After a few bursts of ultrasound I took out the specimen and used a dissection probe to scrape away any clay that had softened, before again immersing and insonifying the sample. Eventually, the two bones separated, and I cleaned the various parts that resulted. It was fortunate that the fossilised remains were a lot harder than the softened clay, so minimal damage was done to the wanted parts of the specimen.



Cleaned bone fragment prior to embedding. Field width approx 24 mm



End view of cleaned bone fragment. Field width approximately 22 mm

Having cleaned various fragments, I then cut cross-sections using my diamond saw, and also trimmed the longitudinal samples to a length compatible with my moulds, before embedding them in epoxy resin. The moulded specimens were then ground flat using a succession of abrasives of decreasing grit size, ending up with 1  $\mu$ m diamond paste.

In addition to the light micrographs of the sections, shown opposite and above, I also examined them under the SEM. As is



Embedded cross section of bone at two different scales. Scale bars 2.5 mm (top) and 750  $\mu m$  (bottom)



Embedded longitudinal section at two different scales. Scale bars 2.5 mm (top) and 750 µm (bottom)

common with flat sections, the SEM images were not very exciting, even using the backscattered electron detector. EDS analysis of the chemical composition did, however, confirm the likely presence of bone material by indicating a higher level of phosphorus in some areas than in others, as is shown in the "map" below. It's early days, but it feels like "real" research.



Map showing the distribution of phosphorus (light green) over the cross-section of a bone.