Can 2D trace elemental mapping of foraminifera help to resolve the presence of photosymbionts in the fossil record?

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Final Report

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1. Introduction

Planktonic foraminifera are single celled marine protists which secrete a shell (test) of calcium carbonate. They are useful in palaeoclimatic study because they are widespread globally and preserve the chemical signature of the seawater from which they calcify. Understanding the palaeobiology of deep time taxa in particular is integral to validating palaeoclimate proxy signals.

Many modern surface dwelling planktonic foraminifera possess algal photosymbionts, which live within a foraminifer's spines. However, tracing symbiont presence in the fossil record is difficult since they are soft-bodied and therefore not preserved. An enrichment in carbon isotope ($\delta^{13}C$) signatures with increasing test size generally indicates photosymbionts were present in extinct taxa (Spero and DeNiro 1987, Norris 1996, Norris and Corfield 1998). However, because this technique in resolving symbiont presence is dependent on size, the use of carbon isotopes in planktonic foraminifera is limited if several sizes are not present in an assemblage.

Modern symbiotic planktonic foraminifera taxa exhibit cyclic Mg/Ca banding in their shell walls. Various studies have attributed the banding as a result of photosymbiont presence (Eggins *et al.* 2004, Sadekov *et al.* 2005, 2008). This intra-shell feature is also independent of size, and could be complementary to carbon isotope data to resolve foraminiferal palaeoecology in deep time.

2. Project Aims

This project aims to:

- Detect changes in Mg/Ca in foraminiferal walls to identify whether cyclic banding occurs in deep time symbiotic taxa. This will first be tested on Miocene taxa.
- Compare trace element data with $\delta^{13}C$ data to determine whether both methods produce the same conclusions about foraminiferal palaeoecology.
- Perform further analysis on Eocene foraminifera (if Miocene analyses are successful) to identify whether the signal is preserved in older samples.

Electron microprobe analysis (EMPA) will be utilised to detect the Mg banding. This is a quantitative microanalytical technique capable of high spatial resolution (micron scale), and can also produce 2D trace element maps. Another advantage of EMPA is contaminant phases can be identified (Pena *et al.* 2008). Prior to EMPA analysis, scanning electron microscope imaging will be used to identify areas of interest in the foraminiferal test wall (see Fig. 2).

3. Samples

Samples in this study utilised Eocene and Miocene age sediments from ODP 174AX and IODP 320/321 respectively (Fig. 1).

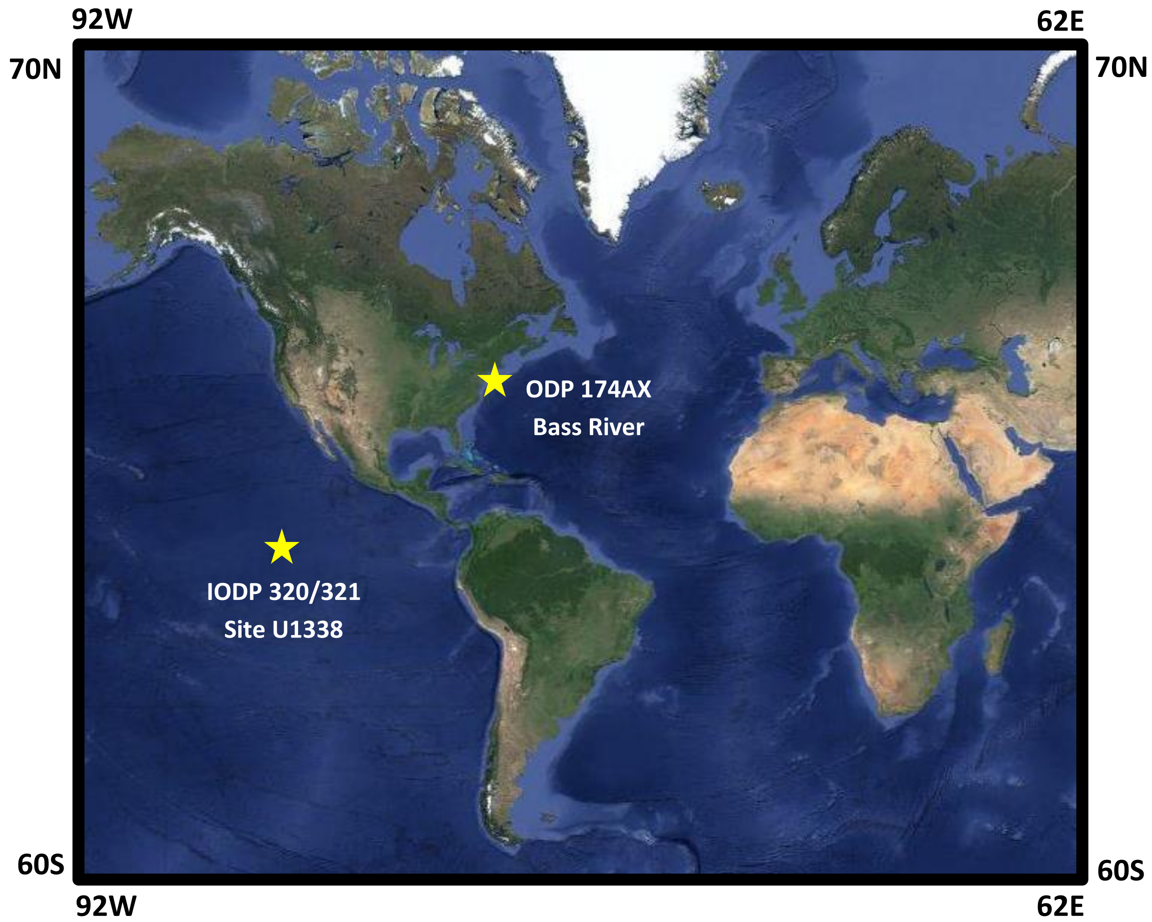

Figure 1: Map showing the locations of ODP 174AX and Site U1338

ODP 174AX is located in the north-west Atlantic. Samples are of Eocene age, and focus on the interval from 56 – 40 Ma, covering a time of dramatic climatic change from peak warmth during the early Eocene climatic optimum and subsequent temperature decline. The sediments contain extremely well preserved, glassy planktonic foraminifera with diverse assemblages, and are composed of calcareous marls, glauconitic silty clays with porcellanitic interbeds (Miller et al. 1998).

Miocene age samples are from Site U1338, located in the eastern equatorial Pacific. At this site, planktonic foraminifera are abundant, diverse and well preserved; pore spaces are open, spines are sometimes preserved and calcitic overgrowths are uncommon. This preservation has been attributed to the high sedimentation rate recorded at this site (30m/Ma) (Fox and Wade 2013).

Taxonomy

Taxonomy for Eocene foraminifera follows Pearson et al. (2006), and taxonomy for Site U1338 foraminifera follows Fox and Wade (2013). Surface dwelling species *Globigerinoides subquadratus* and *Acarinina praetopilensis* and deep dwelling species *Dentoglobigerina tripartita, Subbotina crociapertura* and *Parasubbotina hagni* were analysed. These species have previous $\delta^{13}C/\delta^{18}O$ derived palaeoecologies.

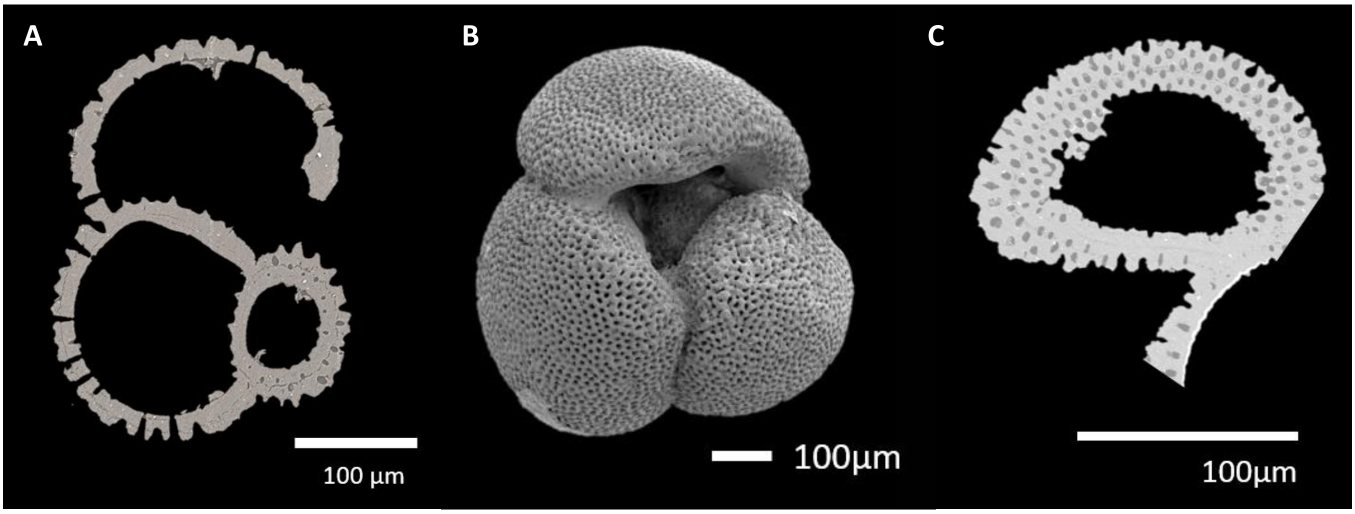

Figure 2: Scanning Electron Microscope (SEM) images of Miocene taxa analysed.
A: Backscattered electron image of *G. subquadratus* **B:** SEM image of *D. tripartita*
C: Backscattered electron image of part of a *D. tripartita* specimen

4. Analytical methods

Samples were embedded in Streurs Specifix-20 epoxy resin blocks, polished to expose the foraminiferal walls, and coated with 15 nm of carbon. Electron microprobe analyses were performed using a wavelength-dispersive Cameca SX100 electron microprobe at the Natural History Museum, London. Operating parameters used were a 10 kV accelerating voltage, 15 nA beam current and 1 µm spot size. A low accelerating voltage and beam current were chosen to create a small excitation volume. A standard of crystalline calcite was used to correct counts per second (cps) data into weight percent (wt%). Step size, dwell time and grid size chosen were 0.25 µm, 100-200 ms and 256 x 256 µm respectively. Maps took between 3 hours – 7 hours to produce, depending on dwell time. 3-5 specimens of each species were analysed.

5. Preliminary results

Preliminary EMPA maps produced (in draft so not included) show that the surface dwelling species *Globigerinoides subquadratus* & *Acarinina praetopilensis* exhibit Mg banding, whilst the deeper dwelling species *Dentoglobigerina tripartita*, *Subbotina crociapertura* and *Parasubbotina hagni* do not exhibit Mg banding (and little to no variability in Mg concentration in the test). Inferring that Mg bands indicate symbiont presence this shows that *Globigerinoides subquadratus* and *Acarinina praetopilensis* are symbiotic and

Dentoglobigerina. tripartita, *Subbotina crociapertura* and *Parasubbotina hagni* are asymbiotic. These relationships are consistent to findings in modern taxa by Sadekov et al. (2005). There was also no difference in the frequency of bands in different chambers in *Globigerinoides subquadratus* which would also suggest the mean Mg/Ca in the final chamber is not much different from that in the other chambers, which agrees with Fehrenbacher and Martin (2014), but contrasts Sadekov et al. (2005) (both studies performed on the symbiotic species *Globigerinoides ruber*). Magnesium also seems to increase at the primary organic membrane in the symbiotic species but not in the asymbiotic species, which agrees with a study on the planktonic foraminiferal species *Pulleniatina obliquiloculata* by Kunioka et al. (2006).

Mn, Fe and Al maps show no correlation to Mg band positioning within the foraminiferal walls, indicating that they are not an artefact of contaminants (e.g. clays).

6. Future work

Future analyses will target more Eocene and Miocene taxa to ensure that the Mg/Ca presence/absence is consistent. Spine presence-depth-symbiont relationships will also be explored further. Carbon isotope data will also be compared to the trace element maps. Results from this study are expected to be presented at the International Conference on Paleoceanography in August/September 2016.

7. Lab expenses

An ECORD grant of 2000€ (£1575.04) was awarded. It was used as follows for the sample preparation and analyses at the Natural History Museum, London**.**

	GBP	**EUR**
Sample preparation (resin mounting & carbon coating, @ £70/mount+coating (inc. VAT)	£420	€533.32
NHM Microprobe analysis @ £766.91/day (3 days, inc. VAT)	£2300.73	€2921.49
Total	£2720.73	€3454.81

SEM images were obtained at UCL using funds from my NERC studentship grant

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