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Bio-signatures in metabasaltic glass of a Caledonian ophiolite, West Norway

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Abstract – Evidence of bioalteration of natural basaltic rocks, presently receiving much attention, has so far been restricted to *in situ* oceanic crust and ophiolites in which fresh glass is still present. Here we present evidence of preserved bio-signatures in the chilled margin of pillow lavas of an old (443 Ma) ophiolite that has suffered pervasive lower greenschist facies metamorphism and deformation. X-ray mapping of initial alteration zones shows the remains of organic carbon associated with highly-concentrated Fe and S. Bioproduction of CO_2 is further reflected in the low $\delta^{13}C$ values of calcite extracted from pillow rims, compatible with microbe-induced fractionation during oxidation of organic matter. We attribute these effects to growth of sulphate-reducing bacteria at the early stage of ophiolite formation. During energy metabolism these bacteria reduce sulphate to H_2S and oxidize organic matter to CO_2. Hydrogen sulphide will eventually react with iron and form pyrite, and carbon dioxide is precipitated as calcium carbonate. The results of this study may thus trigger the search for bio-signatures in glassy volcanic rocks of any age.

Keywords: Caledonides, ophiolite, pillow lava, biogenic effects, stable isotopes.

1. Introduction

The alteration of basaltic glass from the upper 550 m of *in situ* oceanic crust has recently been suggested to be biomediated (Thorseth *et al.* 1995; Furnes *et al.* 1996; Fisk, Giovannoni & Thorseth, 1998; Torsvik *et al.* 1998; Furnes & Staudigel, 1999; Furnes *et al.* 1999; Furnes *et al.* 2001*a*,*c*). This has been documented by the alteration textures, the presence of DNA, carbon and nitrogen within alteration zones and carbon isotopes ($\delta^{13}C$) in disseminated carbonate. Even in the obducted oceanic crust of the Troodos ophiolite (Cyprus), Furnes *et al.* (2001*b*) found evidence that the pillow lavas had undergone bioalteration during an early stage of ocean-floor alteration.

In the undeformed and non- to low-grade metamorphic pillow lavas of *in situ* oceanic crust, as well as those of the Troodos ophiolite, undevitrified glass is abundant, and bio-generated alteration textures are easily visible. In this work we have examined volcanic material from a Late Ordovician ophiolite that has undergone lower greenschist facies metamorphism and polyphasal folding. During these events pervasive re-crystallization of the rocks largely obliterated the microscopic, bio-generated textures. This study was carried out in order to test whether any of the bio-signatures listed above have survived the effects of metamorphism and deformation in pillow lava of old oceanic crust, represented by an ophiolite.

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2. Material investigated

Samples were collected from the least deformed part of the Late Ordovician (443 ± 3 Ma) Solund–Stavfjord Ophiolite Complex in the western Norwegian Caledonides (see Furnes, Hellevang & Dilek, 2001, and references therein). The Solund–Stavfjord Ophiolite Complex displays a well-preserved volcanic sequence of basaltic pillow lavas, sheet flows and volcanic breccias, a sheeted dyke complex, and high-level gabbro. The volcanic sequence is overlain by siliciclastic rocks, which, in the lower part, contain metabasaltic intrusions of comparable composition to that of the underlying volcanic rocks. The Solund–Stavfjord Ophiolite Complex has suffered at least three phases of deformation, of which the first generation resulted in major isoclinal folds (Furnes, Hellevang & Dilek, 2001). However, the volcanic sequence in the southwestern part of the Solund–Stavfjord Ophiolite Complex (on Kattøy) is remarkably little deformed, and here pillow rims were sampled for the present investigation (Fig. 1). Seventeen samples were collected at different stratigraphic levels, extending to about 290 m stratigraphically below the sedimentary cover (Fig. 1). For isotope

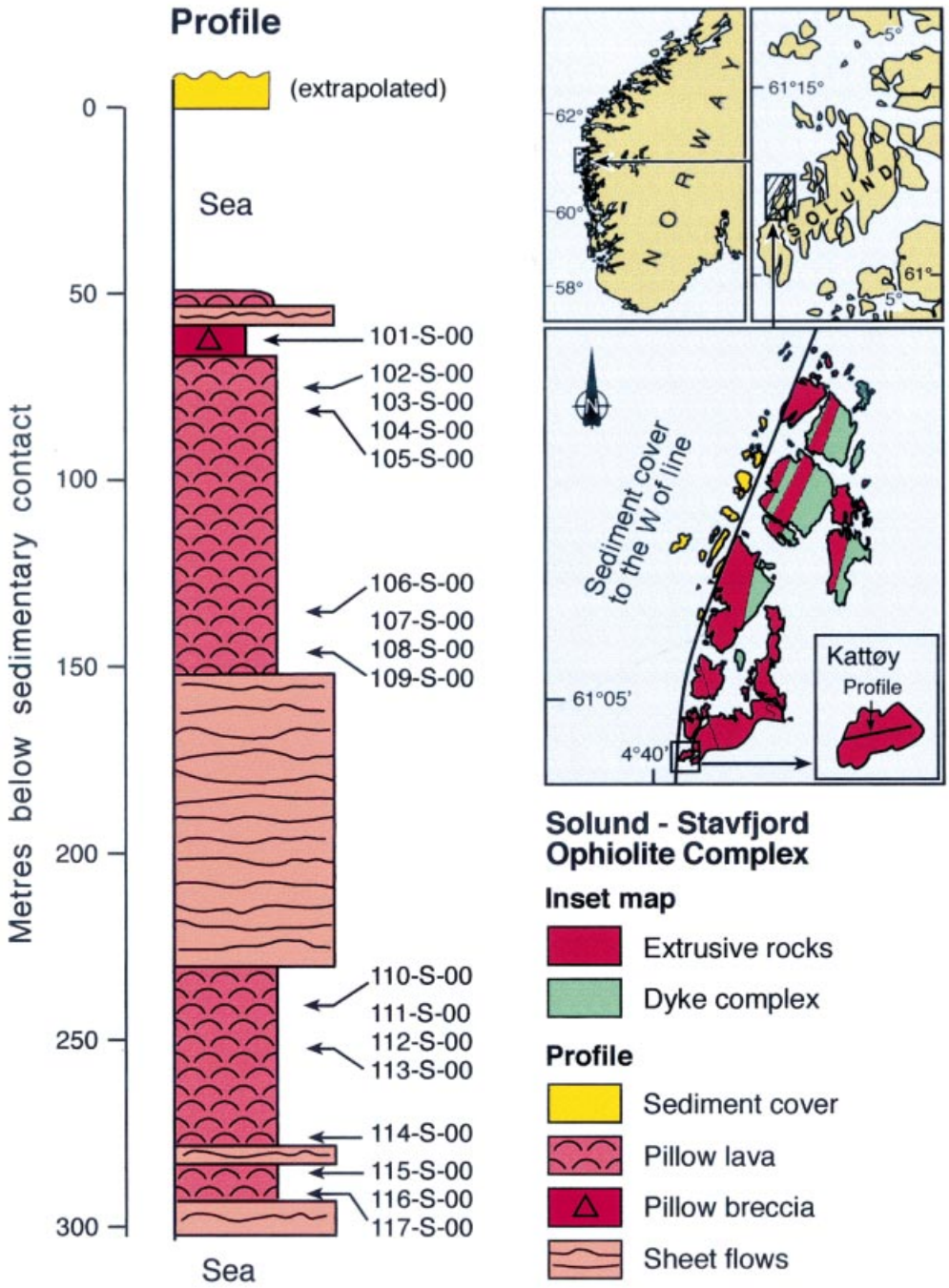

Figure 1. Geological map of part of the Solund–Stavfjord Ophiolite Complex showing location of the rocks investigated. Simplified after Furnes, Hellevang & Dilek (2001). The stratigraphic section shows the volcanological development of the sequence, and at which depth the samples investigated occur in the section.

analysis the samples were split into 'glassy' and 'crystalline' sub-samples. The 'glassy' samples represent the chilled glass margin of pillows, whereas the 'crystalline' samples represent material adjacent to and within the chilled margin. For this study, 33 sub-samples were analysed, of which 18 are 'glassy' and 15 crystalline (Table 1). The pillows consist of chlorite, epidote, albite and sphene, with minor amounts of apatite, quartz and calcite. All samples show well-preserved quench textures as invariably seen in the margins of fresh pillow lavas (e.g. Bryan, 1972). These include an outer 3–6 mm thick, originally glassy zone, a 5–10 mm thick variolitic zone that grades into the originally crystalline part (hereafter referred to as 'crystalline') of the pillows (Fig. 2a). The originally glassy zone (hereafter referred to as 'glassy') consists of fine-grained chlorite with strings of epidote crystals or single crystals of coarser-grained epidote. Individual varioles as small as 10 μm in diameter occur in the outer part of the variolitic zone, but inwards they

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Table 1. Analyses of carbon isotopes (from carbonate) and wt % calcite of analysed samples from the Solund–Stavfjord Ophiolite Complex

Sample no.	Character	MBSC	$CaCO_3$	$\delta^{13}C$
101-S-00-G	Hyaloclastite	65	0.0377	–6.92
101-S-00-C	Crystalline fragment	65	0.4284	–3.47
102-S-00-G	Chilled margin of pillow	73	0.0202	–11.24
102-S-00-C	Crystalline part of pillow	73	0.0206	–9.46
103-S-00-G	Chilled margin of pillow	77	0.0199	–10.50
103-S-00-C	Crystalline part of pillow	77	0.0710	–6.11
104-S-00-G	Chilled margin of pillow	81	0.0231	–11.60
104-S-00-C	Crystalline part of pillow	81	0.0422	–9.71
105-S-00-G	Chilled margin of pillow	85	0.0133	–15.52
105-S-00-C	Crystalline part of pillow	85	0.0312	–6.19
106-S-00-G	Chilled margin of pillow	137	0.0316	–8.87
106-S-00-C	Crystalline part of pillow	137	0.0185	–9.67
107-S-00-G	Chilled margin of pillow	141	0.0132	–9.99
107-S-00-C	Crystalline part of pillow	141	0.0976	–4.26
108-S-00-G	Chilled margin of pillow	145	0.0763	–5.98
108-S-00-C	Crystalline part of pillow	145	0.0138	–7.39
109-S-00-G	Chilled margin of pillow	148	0.0142	–11.33
109-S-00-C	Crystalline part of pillow	148	0.0131	–8.79
110-S-00-G	Chilled margin of pillow	242	0.0244	–11.38
111-S-00-G	Chilled margin of pillow	246	0.0215	–24.80
111-S-00-C	Crystalline part of pillow	246	0.1770	–3.48
112-S-00-G	Chilled margin of pillow	250	g.y.n.r.	–4.43
113-S-00-G	Chilled margin of pillow	254	0.0224	–14.03
113-S-00-C	Crystalline part of pillow	254	g.y.n.r.	–10.21
114A-S-00-G	Chilled margin of pillow	275	0.0464	–6.45
114B-S-00-G	Chilled margin of pillow	279	0.0719	–3.20
114A-S-00-C	Crystalline part of pillow	275	0.0405	–8.53
115-S-00-G	Chilled margin of pillow	283	0.0782	–2.53
115-S-00-C	Crystalline part of pillow	283	0.0657	–5.87
116-S-00-G	Chilled margin of pillow	287	0.0410	–6.55
116-S-00-C	Crystalline part of pillow	287	0.1664	–3.40
117-S-00-G	Chilled margin of pillow	291	0.0166	–5.02
117-S-00-C	Crystalline part of pillow	291	0.0543	–4.85

MBSC = metres below sedimentary contact; g.y.n.r. = gas yield not registered.

coalesce and become larger (up to *c.* 500 μm in diameter) (Fig. 2a). Beyond the variolitic zone the wholly crystalline part shows quench-generated features such as leaf-spherulitic and branching textures (Bryan, 1972).

3. Analytical methods

Carbonates were analysed for their C-isotopic composition by extracting CO_2 under vacuum from whole-rock powders (1 to 2 g) by treatment with 100 % H_3PO_4 (phosphoric acid) for 24 hours at 25.3 °C (McCrea, 1950). The exsolved CO_2 was analysed on a Finnegan MAT 252 mass spectrometer. Applying the method of McCrea (1950), quantities down to 0.05 mg of carbon can be measured. Hence, 0.05 mg extracted from 2000 mg rock gives a lower detection limit of 0.0025 %. The errors for CO_2 analyses range from about ± 1 % to ± 15 % for samples rich and poor in carbonate, respectively. The errors on isotopic analyses for carbon are about ± 0.2 ‰. The data are reported in the usual PDB delta notation with respect to carbon (Craig, 1957).

X-ray mapping of Cr-coated thin sections was carried out using a JEOL JXA-8900R electron microprobe. Scanning electron microscopy (SEM) of C-coated thin sections was carried out using a JSM-6400 instrument.

4. Bio-signatures

4.a. Bio-generated textures

Along fractures within the glassy parts of the investigated pillows there are features that strongly resemble bio-generated textures adjacent to fractures in fresh, non-metamorphic pillows described from *in situ* oceanic crust elsewhere (Thorseth *et al.* 1995; Furnes *et al.* 1996). These features appear as sphene-rich aggregates developed in a symmetric or asymmetric arrangement around fractures, with an irregular front against the original glass, now replaced by chlorite (Fig. 2b). The asymmetric development of alteration products around fractures, as well as the irregular contact with the fresh glass, are typical textural features related to bio-generated alteration in the quenched zone of pillows of *in situ* oceanic crust (Fig. 2c). Below we will argue that the textural features shown in Figure 2b are due to bioalteration and have survived metamorphic recrystallization.

4.b. Element distribution within alteration zones

The concentration patterns of C, P, S, Ca, Mg, Fe, Ti, Si and Al at the intersection between two alteration zones, which on the basis of their textures resemble those of bioalteration, are shown in Figure 3. The

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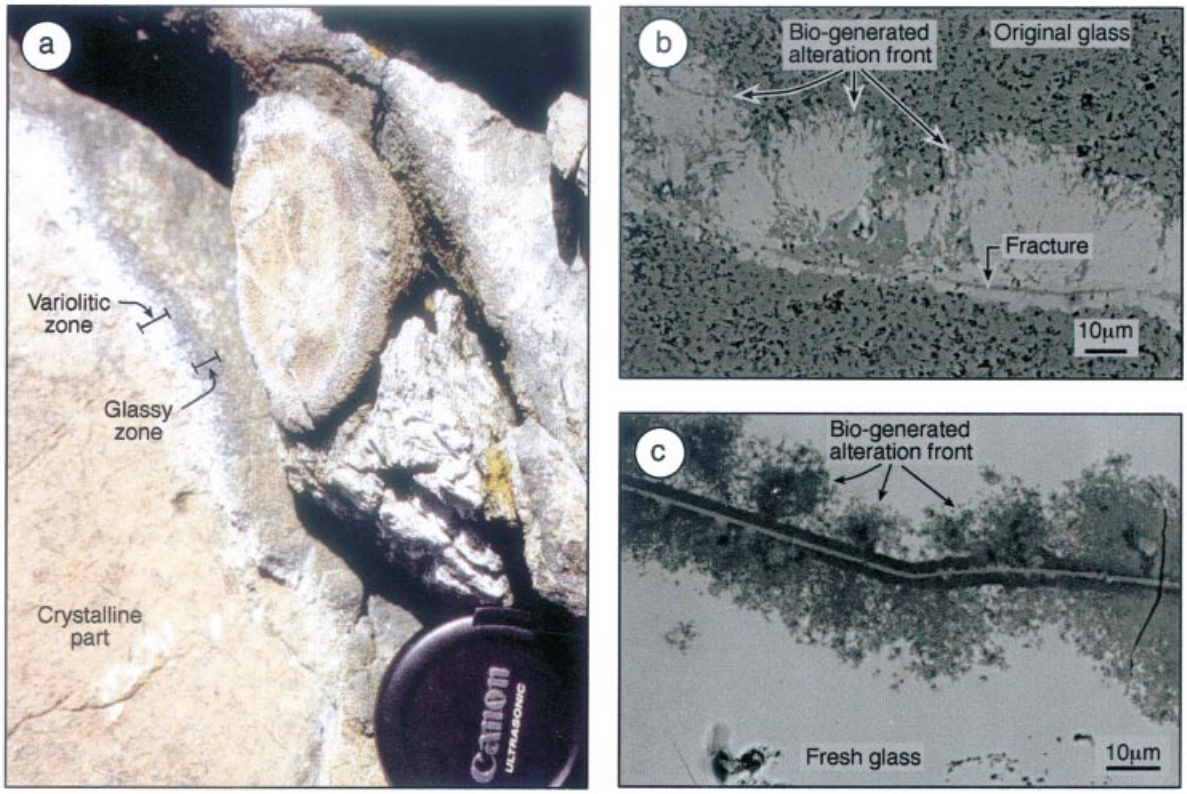

Figure 2. (a) Pillow lava from the Solund–Stavfjord Ophiolite Complex, showing the original glassy rim (grey), and variolitic texture (white spots adjacent to the dark glass rim). Sample 117-S-00 was taken from this pillow (the larger one). Diameter of lens cap is 5 cm. (b) Inferred bio-generated texture (light grey), in a dark chloritic matrix (originally glass) in pillow lava from the Solund–Stavfjord Ophiolite Complex (sample 101-S-00). (c) Bio-generated texture (dark grey) adjacent to a fracture in fresh glass (light grey) in pillow lava from the Hole 418A of the Atlantic Oceanic crust (sample 418A, 62-4, 64-70). Note the textural similarity between (b) and (c). (b) and (c) are both SEM images.

element maps clearly show the distribution of chlorite (high Mg, Fe and Al) and sphene (high Ti, Ca and Si), and the locally high carbon concentrations. By superimposing the element maps of C, Ca, Mg and Fe upon each other it is clear that the carbon is not bound in any kind of carbonates (calcite, magnesite, ankerite). The patches of high-concentrated carbon associated with chlorite, and carbon enclosed in pyrite, chlorite and sphene (Fig. 3, upper right corner) are most probably remnants of organic carbon. The highest concentrations of P and S coincide with high C contents. It should be noted that the signals for sulphur are weak compared with those for iron (Fig. 3), yet we have taken the combination of these two elements to reflect the presence of pyrite. The contrast in brightness of the S and Fe images, however, does not reflect the concentration of the elements, but rather a combination of technical features. These are the positioning of the probe detectors, as well as the topography of the analysed area.

4.c. Carbon isotopes

The $\delta^{13}C$ values of the CO_2 and the $CaCO_3$ concentrations in the glassy (chilled margin of pillows and hyaloclastite) and crystalline sub-samples are shown in Table 1. The range in the $\delta^{13}C$ values is considerably wider for the glassy than for the crystalline sub-samples (–2.5 to –24.8 and –3.4 to –10.21 respectively), and their average $\delta^{13}C$ values are –9.5 and –6.8, respectively. The relationship between $\delta^{13}C$ of the crystalline and glassy samples, plotted against stratigraphic depth, is shown in Figure 4. At stratigraphic levels higher than *c.* 250 m, the glassy sub-samples are lower in $\delta^{13}C$ than the crystalline sub-samples, whereas at the deepest levels (between 250 m and 300 m), this appears to be no longer the case (Fig. 4).

Figure 5 shows the relationship between $\delta^{13}C$ and CO_2 (recalculated to percentage calcite) for the glassy and crystalline sub-samples. Although the $\delta^{13}C$ values get poorer at progressively lower carbonate values, the data show a clear pattern. As $\delta^{13}C$ values increase from –7 to –3, there is a sharp increase in the amount of calcite.

5. Discussion and conclusions

Arguments in favour of bioalteration of basaltic glass are (1) the textural development of alteration zones, (2) the presence of typical bio-elements (C, S, P)

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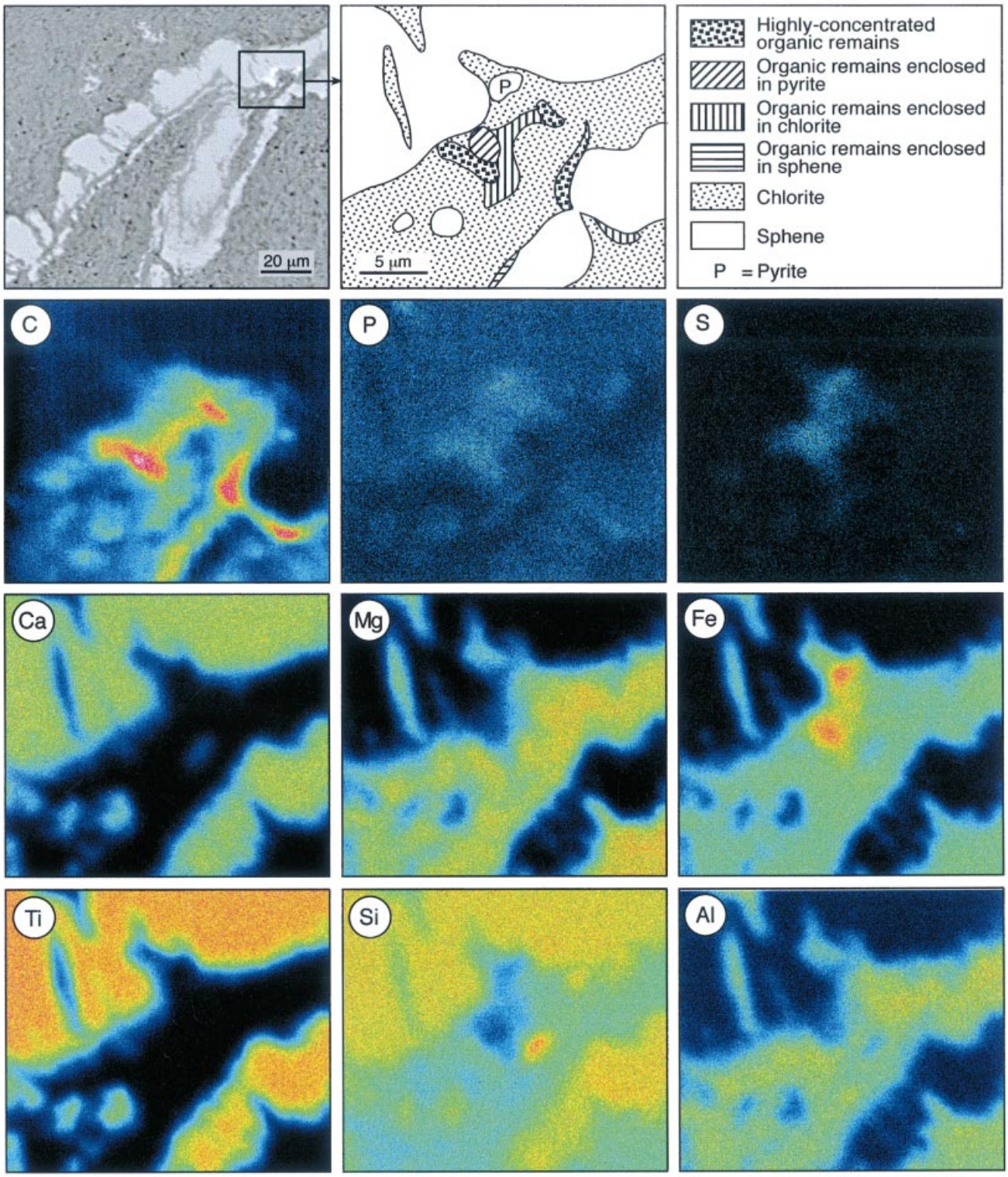

Figure 3. Illustration showing SEM image and X-ray maps (of C, P, S, Ca, Mg and Fe) from a bioaltered area of sample 114-S-00-G.

within alteration zones, and (3) carbon isotope signatures that can be attributed to microbial fractionation.

The development of irregular patterns of alteration zones adjacent to fractures in the glass, the internal granular and/or tubular textures of the alteration zones, as well as the irregular character of the alteration front, are features that have been regarded as typical bio-signatures (e.g. Thorseth *et al.* 1995; Furnes *et al.* 1996; Fisk, Giovannoni & Thorseth, 1998; Furnes *et al.* 2001*c*). The alteration zones along fractures in the original glass, expressed as the sphene-rich zones adjacent to chlorite, have irregular alteration fronts to the chlorite, as well as asymmetric development of the alteration zones (Fig. 2b). In these metamorphosed samples, we have not been able to detect the details of the internal structure of the alteration

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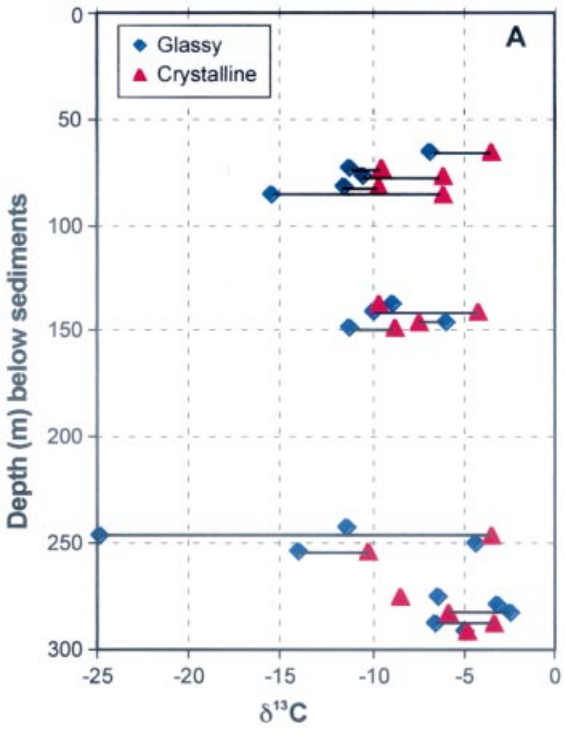

Figure 4. Relationship between $\delta^{13}C$ for the pillow lavas of the Solund–Stavfjord Ophiolite Complex and stratigraphic position. 'Glassy' and crystalline sub-samples from the same pillow rim are connected by tie-lines.

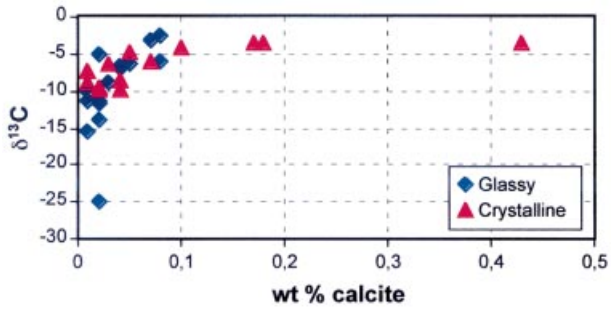

Figure 5. Relationship between $\delta^{13}C$ and wt % calcite for the 'glassy' and crystalline rocks of the Solund–Stavfjord Ophiolite Complex.

zones as seen in non-metamorphosed samples, that is, the presence of spherical bodies about 0.2–0.6 μm in diameter that agglomerate to give a typical granular texture (Furnes *et al.* 2001*c*) as seen in Figure 2c. However, the features of the metamorphic samples, in particular the alteration front (Fig. 2b), are most reminiscent of bio-alteration textures developed in glassy, non-metamorphic basalt, and may have been produced by microbial activity. Textural indication of bioalteration has not, to our best knowledge, previously been reported from deformed and metamorphosed Palaeozoic rocks. In an extended search for bioalteration of volcanic rocks back to Archaean time, investigation of any possible bio-signature is important.

Further evidence for the bio-activity within the alteration zones is provided by the presence of organic carbon as shown in Figure 3. In this case, where the metamorphic rock complex has been subject to a temperature of *c.* 350–400 °C, organic remains were most probably preserved by having been enclosed by the crystals, or becoming part of the crystalline structure. As to the first alternative, this has been proposed for the preservation of organic matter in fracture fillings of authigenic minerals of the Columbia River Basalts (McKinley, Stevens & Westal, 2000). As for the latter, it is well known that organic molecules may become part of crystals during diagenesis and recrystallization (Collins, Bishop & Farrimond, 1995; van Lith *et al.* 2001). Since the carbon can be shown not to be in the form of carbonates, we propose that it may represent one or both of the two above-mentioned alternative modes of carbon preservation.

The strongly negative $\delta^{13}C$ values of the carbonates of the glassy samples (average –9.5) are much lower than those found in mantle carbonates (–5 to –7 according to Alt *et al.* (1996) and Hoefs (1997)). The wide range in the $\delta^{13}C$ values of the glassy sub-samples (between –5 and –25) occurs at near constant and extremely low carbonate values (*c.* 0.02 wt %) for the majority of the samples (Fig. 5). Thus, Rayleigh fractionation, which may alter carbon isotope ratios during basalt degassing and subsequent alteration (Hoefs, 1997), can hardly be responsible for the large spread in the $\delta^{13}C$ values of the glassy samples. The crystalline sub-samples, however, show a larger spread in the calcite content with a much smaller $\delta^{13}C$ range than that of the glassy sub-samples, and the two parameters show a weak positive correlation. The average $\delta^{13}C$ value of the crystalline samples (–6.8) is comparable to magmatic values. The lowest $\delta^{13}C$ values at the highest $CaCO_3$ values (Fig. 5), however, may be due to the presence of thin veins of inorganically precipitated calcite with a $\delta^{13}C$ value of *c.* 0 (Alt *et al.* 1996).

In a similar study of C-isotopes in altered basaltic glass from *in situ* oceanic crust, Furnes *et al.* (2001*a*) proposed that organic material had been oxidized to carbon dioxide by biological processes during which isotopic discrimination of ^{13}C has taken place. A similar process can be attributed to the samples in this study. The observation that organic carbon is found associated with pyrite (Fig. 3) indicates that the microbiological process has been sulphate reduction. A tentative hypothesis for the microbial process would be: organic-C + $SO_4^{2-} \Rightarrow CO_2 + H_2S$. The carbon dioxide would eventually precipitate as calcium carbonate and the hydrogen sulphide would react with iron and form pyrite.

Three of the glassy samples (112-S-00-G, 114-S-00-G and 115-S-00-G) have $\delta^{13}C$ values greater than –5 (–4.43 to –2.53, Table 1), that is, within the range of combined $\delta^{13}C$ values resulting from mixtures of mag-

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matic CO_2 and inorganically precipitated calcite. These 'high' values, apparently non-indicative of bio-influence upon the glass, could have resulted at least two different ways, both partly bio-generated. All the samples would, prior to alteration and metamorphism, have initial magmatic $\delta^{13}C$ values in the range –5 to –7 (Alt *et al.* 1996; Hoefs, 1997). If the $\delta^{13}C$ from inorganically precipitated calcite (with $\delta^{13}C \sim 0$) exceeds that resulting from microbial oxidation of organic carbon, the $\delta^{13}C$ value of a sample may be greater than –5. Alternatively, if methane-producing organisms were operative during alteration, ^{12}C-enriched methane is lost, and the remaining carbonate will be ^{13}C-enriched. Thus, if the glassy rock was processed by both chemo-organotrophic *Bacteria* and methanogenic *Archaea*, a phenomenon which was demonstrated in a glassy rock from the Costa Rica Rift (Torsvik *et al.* 1998), the resulting $\delta^{13}C$ value could also be greater than –5. This phenomenon was proposed to explain the large range in $\delta^{13}C$ values of pillow lava samples from *in situ* oceanic crust, in which H_2 production during serpentinization of ultramafic rocks would provide favourable conditions for *Archaea*, resulting in high $\delta^{13}C$ values (Furnes *et al.* 2001*a*).

As to the timing of bioalteration of the investigated volcanic rocks, we will argue that this process most probably occurred prior to the regional greenschist metamorphism. Evidence for this supposition is given by (1) organic remains encrusted by sphene, chlorite and pyrite (Fig. 3), and (2) the difference in the $\delta^{13}C$ values of the chilled margin of pillows and samples within the chilled margin (Fig. 4). As to the first point, the textural relationships would indicate that the organic remains were present in the margins of the pillow lavas prior to the formation of the metamorphic minerals. Concerning the second point, experimental data have shown that microbes prefer lodgement on glass rather than crystalline material (I. Brekke, unpub. Cand. Scient. thesis, Univ. Bergen, 1998). The $\delta^{13}C$ data shown in Figure 4 thus strongly favor bio-activity within the pillow rims while they were in a glassy stage, that is, prior to the formation of the metamorphic minerals. If the bio-process was post-metamorphic, any difference in the $\delta^{13}C$ values between the 'glassy' and 'crystalline' samples, as demonstrated in Figure 4, should not be expected.

The most active bioalteration process of the oceanic crust generally occurs during the hydrothermal stage (Furnes *et al.* 2001*c*). This implies that bioalteration takes place when a crustal segment is near the active spreading ridge, that is, at a time when water circulation through the crust was high and the availability of nutrients (carbon, energy, mineral nutrients) was at a maximum. By applying similar arguments for the bio-signatures of the glassy volcanic rocks of the Solund–Stavfjord Ophiolite Complex, we consider it most likely that they date from the early stage of the crust, approximately 443 Ma ago.

This study, demonstrating the presence of bio-signatures in metamorphosed and deformed pillow rims of the 443 Ma Solund–Stavfjord Ophiolite Complex, may have important implications in the search for evidence of life in much older glassy volcanic rocks. All palaeontological evidence of fossilized micro-organisms has so far been discovered in rocks of sedimentary origin. However, the birthplace of life may have been in the volcanic environment, for example, in hydrothermal vents of a spreading ridge (e.g. Russel & Hall, 1997). Thus, the observation that bio-signatures can be preserved in old ophiolites, like the Solund–Stavfjord Ophiolite Complex, provides an impetus for the search for fossilized micro-organisms in the oldest known terrestrial ophiolites, or other old volcanic rocks.

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