# **Jaspers of the Løkken ophiolite**

Jaspers are typically bedded cherts (silica-rich, microcrystalline rocks) rich in haematite  $\pm$  magnetite  $\pm$  Fe-silicate minerals. Stratiform occurrences of jasper with pillow basalts and volcanogenic massive sulphide deposits imply an origin from lithified hydrothermal precipitates  $^{21,27,29}$ , formed in submarine settings analogous to those of modern midocean ridges or back-arc sea-floor spreading centres, intra-plate seamounts, or flanks and calderas of arc volcanoes <sup>22,23,26</sup>. Si-Fe colloidal-gel protoliths of jaspers can be attributed to several processes, including *in situ* precipitation from low-temperature (20-100°C 60) fluid flow through oceanic crust  $^{61}$ , material derived from low-temperature (2-50°C) vent chimneys  $^{61}$ , and proximal or distal sedimentation from hydrothermal plumes  $^{62}$ .

The Early Ordovician (Tremadoc) Løkken ophiolite was deformed and emplaced during the Caledonian orogeny, reaching metamorphic grades of lower greenschist facies; the original setting was dominated by back-arc rifting in a marginal basin  $63$ . The ophiolite contains laterally extensive jaspilitic beds that cluster at stratigraphic levels equivalent to those of the Løkken and Høydal volcanogenic massive sulphide (VMS) deposits and are in places intimately associated with the Fe-Cu-Zn sulphide ore. The jasper, which formed originally as sea-floor gels, displays myriad exceptionally well-preserved primary textures, including haematitized bacteria remains, haematitic tubes, and a variety of textures attributed to gel maturation and more advanced diagenetic changes <sup>63</sup>. The gel was deposited by particle fallout from hydrothermal plumes, in which silica flocculation and rapid settling of colloidal particles was promoted by the bridging effect of Fe oxyhydroxide <sup>27</sup>. Fe was sourced from white smoker-type vents with high Fe/S ratios <sup>64</sup>, whereas silica is thought to be primarily of ambient seawater origin based on the interpretation of silica saturation or supersaturation in pre-Late Cretaceous oceans  $27$ . Trace element variations in the gel precursor were controlled by coprecipitation and/or adsorption by Fe oxyhydroxide particles within the plume(s), REE patterns (positive Eu and negative Ce anomalies) reflect mixing of hydrothermal solutions with seawater at dilution ratios of  $\approx 10^2$  to  $10^4$ .

# **The Nuvvuagittuq Supracrustal belt (NSB)**

The NSB is located in the Northeastern Superior Province, in northern Québec, Canada. It is surrounded and intruded by multiple generations of Eoarchaean to Palaeoarchaean tonalite-trondhjemite-granodiorite (TTG) gneisses dated at 3760 Myr, 3660 Myr, 3500 Myr, and 3350 Myr <sup>65,66</sup>, and forms a core enclave within the Neoarchaean TTG gneisses of the Inukjuak domain. The NSB is dominated by mafic amphibolites composed of cummingtonite-plagioclase-biotite±garnet<sup>17</sup>.

The amphibolite unit has been interpreted to represent mafic volcanic deposits, with compositions ranging from basaltic to basaltic andesite <sup>11</sup>. In some locations, pillow lava structures are preserved within the amphibolite <sup>11</sup>. These amphibolites are divided into three geochemical groups displaying distinct Al/Ti ratios and following a chemostratigraphy within the NSB  $^{11}$ . The base of the sequence is characterised by low Al/Ti

metabasaltic rocks with tholeiitic affinities. They exhibit relatively flat trace element profiles and their chemistry is consistent with derivation from an undepleted mantle source and fractionation under relatively dry conditions  $11$ . The top of the stratigraphy comprises amphibolites having geochemical compositions consistent with derivation from a reenriched depleted mantle and fractionation under elevated water pressure <sup>11</sup>. They comprise high Al/Ti basaltic amphibolite relatively depleted in incompatible trace elements and displaying characteristic concave-up REE profiles typical of modern boninites. They are overlaid by intermediate Al/Ti amphibolite of basaltic to andesitic compositions with calcalkaline affinities displaying LREE-enrichments and flat HREE profiles. Chemical sedimentary rocks (BIF and chert-like silica rocks) mark the transition in the stratigraphy between the low Al/Ti tholeiitic amphibolite and the higher Al/Ti boninitic and calcalkaline amphibolite. This transition and chemo-stratigraphy led some to suggest that the NSB metavolcanic rocks were formed in subduction initiation settings similar to those of modern convergent tectonic settings <sup>67</sup>. Locally, the NSB amphibolite is composed of a cordierite-orthoamphibole mineral assemblage and is characterised by high Mg and K concentrations with depleted Ca, Na, and Si. This mineralogical assemblage and geochemical signature are consistent with seawater hydrothermal alteration of the oceanic crust <sup>68</sup>. Together with the presence of pillow lavas and BIF, this pattern suggests that the NSB formed through submarine volcanism with important hydrothermal activity.

On the basis of mineralogy, two broad types of BIF lithologies are recognized from the NSB including quartz + ferrous silicates (banded silicate formation (BSF) and quartz + magnetite + ferrous silicates (BIF) <sup>16</sup>. Other minor lithologies that have been identified in the belt are jasper-quartz-carbonate, quartz-biotite schists (with possible metaconglomerates), and chromite-bearing silica rock <sup>69, 12, 18, 19</sup>.

Outcrops of BIF vary from 5 to 30m in thickness, with the jasper forming thinner beds of 1 m thickness and less, commonly interbedded with layers of amphibolite. In places, small veins, typically a few cm wide of jasper infiltrate and cut the amphibolite <sup>19</sup>, suggesting Fe-Si gels infilled networks within the (interpreted) pyroclastics during deposition. The depositional depth of the NSB jaspers is uncertain from the associated lithologies or textures in rocks of the belt; however, the occurrence of fine-scale, ironoxide lamination (Fig. E3A) is similar to that in other younger jaspers and IFs suggestive of a calm depositional environment below wave base.

The NSB was mostly metamorphosed to upper amphibolite facies with temperatures reaching  $650^{\circ}$ C and  $4-5$  Kbar  $^{17, 20}$ . Sm-Nd isotopic compositions of garnets in the NSB amphibolites suggest that the peak metamorphic event occurred in the Neoarchaean  $14$ contemporaneous with intrusion of pegmatites dated at  $2688 \pm 2$  Myr<sup>66</sup>. This is also consistent with the regional metamorphism occurring at 2705–2680 Myr<sup>70</sup>. The amphibolites in the southwest and southeast corners of the NSB are characterised by lower metamorphic grade assemblages of chlorite+epidote+actinolite. To the southeast, chlorite preserves the shape of what appears to have been garnet crystals, suggesting that the lower greenschist assemblage is retrograde 17 . No evidence of retrogressed garnet is observed in the lower grade facies to the southwest, which may never have reached upper amphibolite facies. Very local layers in Fe-cherts preserve primary chert, calcite rhombohedra, and well-formed minnesotaite needles in jasper, suggesting some rare layers escaped severe recrystallisation during peak metamorphism. Furthermore, the crystallisation temperatures estimated for graphitic carbon in rocks from the southwest corner of the NSB do not exceed 550°C (Table S9-10), suggesting that the metamorphic facies did not exceed much past greenschist facies.

The geochronology of the NSB is highly debated and two ages have been proposed for the contained metavolcanic rocks: an Eoarchaean age of  $\sim$ 3750 Myr<sup>69</sup> and a Hadaean age of ~4280 Myr<sup>15</sup>. Here we present an overview of this geochronological debate.

One of the challenges in precisely dating the formation of the NSB is the quasiabsence of zircon-bearing felsic rocks and their equivocal relationship to the mafic metavolcanic rocks. Rare bands of felsic rocks with trondhjemitic composition have yielded zircons dated by U-Pb between 3750 and 3774 Myr <sup>69, 66, 65, 13, 71</sup>. These trondjhemitic rocks consist of thin discontinuous bands 25-40 cm in width found only at the southern edge of the NSB <sup>17</sup>. Other interpretations placed these rocks as felsic intrusions due to cross-cutting relationships observed in the field  $69$ , thus giving a minimum age for the NSB. Other field evidence such as the presence of felsic intrusions within gabbro sills were used to reach to the same conclusion about their intrusive nature  $^{14,65}$ . Other works, however, interpreted these rocks as felsic volcanites <sup>71</sup>, hence providing the actual age for the NSB. Significantly, this is inconsistent with the cross-cutting relationships observed by <sup>69</sup>. Fuchsitic quartz-rich rocks in the NSB have been interpreted as detrital quartzites by some works  $^{12}$  which yielded 13 zircons with  $^{207}Pb^{/206}Pb$  ages ranging from 3718 Myr to 3780 Myr. These therefore put a maximum age for the NSB at 3780 Myr. However, this interpretation is inconsistent with the principle of inclusion, because the maximum age for the detrital quartzite should be constrained by the youngest 3718 Myr detrital zircon. This, however, is in conflict with the minimum age of 3770 Myr for the NSB supported by the age of the cross-cutting felsic rocks  $\frac{69}{2}$ . Other studies ascertained the same U-Pb age of  $3794 \pm 16$  Myr for the fuchsitic rocks, which they conversely interpreted as metasomatically altered felsic orthogneiss of intrusive origin <sup>13</sup>, hence providing a minimum age for the NSB. A Hadaean age for the NSB is supported by the  $142$ Nd isotopic composition of the mafic amphibolites.  $142$ Nd is the daughter product of <sup>146</sup>Sm, and because the parent isotope has a short half-life of 103 Myr, any deviation in

 $142$ Nd compared to modern terrestrial standards implies Sm-Nd fractionation before ca. 4000 Myr, prior to extinction of  $146$ Sm. A significant number of NSB amphibolites exhibit anomalous  $142$ Nd compositions compared to modern terrestrial standards. The correlation

between the  $\frac{142}{N d}$  Nd  $\frac{144}{N d}$  and Sm/Nd ratios for all three groups of NSB amphibolite is interpreted by some 14,15 to represent an isochron giving an igneous age of ca. 4290 Myr for the NSB metavolcanic rocks. This Hadaean age has been challenged by some who interpreted the  $\frac{^{142}Nd}{^{144}Nd}$  vs. Sm/Nd correlation to reflect mixing with a Hadaeanenriched reservoir <sup>72</sup>. However, this correlation is preserved between the mafic amphibolite and their ultramafic co-genetic cumulates, which can be used to argue against mixing as a cause of the  $\frac{142}{14}$ Nd<sup>144</sup>Nd vs. Sm/Nd correlation <sup>14</sup>. The composition of these ultramafic rocks is controlled by olivine fractionation and they are interpreted to be co-genetic cumulates to the mafic amphibolites  $11$ . The  $142$ Nd $144$ Nd vs. Sm/Nd correlation between the hypothesised mafic liquids and ultramafic cumulates can only be produced if the igneous fractionation occurs while <sup>146</sup>Sm was still decaying, i.e. prior to 4000 Myr. A Hadaean age for the NSB is also supported by the  $^{147}$ Sm- $^{144}$ Nd isotopic composition of intruding gabbro sills that yield an isochron age of ca.  $4100 \pm 100$  Myr, which would establish a minimum age for the NSB metavolcanic rocks <sup>14</sup>.

Hence, there is still no consensus on the exact age of the NSB, but nonetheless, it is at least 3770 Ma and possibly as old as 4290 Myr, which makes the NSB BIF among the oldest, if not the oldest, chemical sedimentary rocks known on Earth.

## **Jaspers and BIFs metamorphosed at the upper greenschist/ lower amphibolite facies**

Eoarchaean supracrustal belts worldwide are generally metamorphosed to at least upper amphibolite facies. The metamorphic mineral assemblages of BIFs are well-documented  $^{73}$ . Petrographic and quantitative analysis of elements in Fe-silicate minerals within BIFs can be used to constrain metamorphic facies. In the BIFs of the NSB, the highest-grade samples exhibit extensive recrystallization and dissemination of coarse magnetite layers, and contain fayalite and pyroxene that indicate amphibolite- to granulite-facies conditions. With decreasing grades, the BIFs lack fayalite and instead contain minor pyroxene, amphiboles (predominantly grunerite-cummingtonite), and retrograde phyllosilicates (clinochlore). The low-grade BIFs contain patches of fine and coarse grained quartz. The very lowest grade BIFs have acicular stilpnomelane, minnesotaite, and rhombohedral ferroan calcite (Fig. E3D). The stilpnomelane in lower grade BIF is likely prograde, because this mineral does not form massive pseudomorphs after higher grade minerals, but rather fine euhedral laths. An upper stability field of 430-470°C and 5-6 Kbar is proposed for stilpnomelane <sup>74</sup>. Furthermore, the presence of chert suggests that the P-T conditions of these prograde stilpnomelane--bearing samples did not exceed much past upper greenschist facies.

### **Null hypothesis and abiotic formation of haematite tubes and filaments**

The null hypothesis in the context of early life studies requires one to assess the likelihood of plausible abiotic explanations for observations purporting to support a biological phenomenon. If all plausible abiotic explanations are considered and assessed to be significantly unlikely, then one can reject the null hypothesis leaving a biological interpretation for the observed features. Below we consider the possible abiogenic mechanisms that could have created haematite tubes and filaments akin to those documented in the NSB.

First, the NSB tubes rarely branch, are commonly straight, and exhibit parallel orientation that may reflect preferred mineral growth. If iron was remobilised along crystal boundaries of acicular minerals as they grew during diagenesis and metamorphism, tube-like structures of haematite could have formed. However, if the precursor was not opal then the mineral would need to have been replaced completely, because only quartz is found inside the tubes. Secondly, acicular mineral growth in rocks is not uniform in shape, in contrast to the consistent morphology observed for the tubes.

Likewise, metamorphic processes may elongate quartz-hosted haematite to produce aligned, tubular structures like the tubes in the NSB, through fluctuating volume change in the rock during silica remobilisation or metamorphic stretching. Such processes would lead to the structures and minerals in the local environment being preferentially and consistently aligned, yet the orientation of ribbons and tubes of nanoscopic haematite (Fig. 1C) in the NSB jasper is highly variable over scales from hundreds of microns to millimetres. However, the presence of 500-µm gaps in iron-oxide layers (Fig. E3B) showing strong alignment of tubes and ribbons of haematite raises the possibility that during metamorphism elevated strain pressure pried apart these iron-oxide layers, which subsequently were infilled with silica and haematite that nucleated on the iron-oxide layers and were elongated during continued layer separation. In addition, layers and pre-existing amorphous structures of haematite may have, under pressure, undergone a process similar to neurulation, whereby layers of haematite are pressed together to form tubes. The two mechanisms above could, in theory, form consistently sized tubes in the range of  $16-30 \mu m$ by reaching a critical size limit, but it is highly unlikely that such mechanisms can facilitate the growth of multiple tubes from a single haematite knob at varying angles (Fig. 1A, E4), both horizontally and vertically during prograde metamorphism, together with the formation of internal filaments and similarly coiled, branched, and twisted filaments (Fig. E4) with delicate haematite envelopes (Fig. 1C). In addition, their close spatial association with carbonate and graphite (Fig. E5D) is most compelling and consistent with a biogenic origin for these tubes and filaments.

Because formation of the NSB tubes via the above metamorphic processes fails to fully explain the NSB tubular structures as metamorphic products, a primary origin of the tubular structures is considered more probable. Nonetheless, there still exist exotic processes that may produce pseudo-microfossils under certain conditions <sup>75</sup>. However, these known exotic processes form filaments of silica and barium carbonate and form under very alkaline conditions, which are unlikely to be relevant for the sea-floor environment of deposition of the NSB jaspers (which lack barite) and formation of the contained tubes. Similar iron-oxide tubes like those present in the NSB have been synthesised using iron-ammonium-sulphate solutions and electric currents, or via mixing

of hydrogen with ammonia bubble streams <sup>76</sup>. Notably, these synthesised tubes are demonstrably different from those in the NSB and younger jaspers. Firstly, the experimental tubes have solid walls of iron oxide and consist of a complex mixture of Fe minerals that vary depending on the nature of the bubble solution, whereas the NSB tube walls are composed only of dispersed, individual, nanoscale grains of haematite, consistent with the recrystallization of primary Fe-oxyhydroxide precipitates from seawater. Secondly, the experimental tubes are variable in size, which contrasts with the generally consistent size of the NSB tubes; yet, a number of tubes in the NSB jasper are strongly deformed and display irregular forms and sizes (Fig. 2D). Finally, the synthesised tubes lack internal chains of platy haemat ite or any other phase, unlike the NSB tubes.

Whereas tubular structures in modern and lithified hydrothermal precipitates are interpreted almost exclusively as microfossils, some branching filaments have been attributed to inorganic, self-organized mineral growth of Fe-oxyhydroxide, controlled by redox fronts in a silica gel<sup>77</sup>. However, such structures are highly branching and are not reported to occur as tubes, but instead as solid Fe-oxyhydroxide filaments, and hence are debated in terms of biological origin  $^{28}$ . Similarly, Fe- oxyhydroxide filaments could form abiogenically in a Fe-Si gel and then be coated subsequently by colloidal silica and Feoxyhydroxide to form tubes with internal filaments, like those observed in the NSB. However, this process fails to explain the tubes that lack internal filaments, because silica has never been proven to replace haematite, and fails to explain their associations with carbonate and CM; thus, the tubes are unlikely to have formed via this process. Rather, in our interpretation, silica precipitated on and replaced bacterial filaments, which were then coated with nanoscale Fe-oxyhydroxide to form the tubes, while minor amounts of microbial organic matter from the filaments was oxidised to carbonate that are now preserved as carbonate-graphite associations with the filaments (Fig. E5). In summary, the NSB haematite tubes and filaments cannot be explained fully by any known abiotic mechanism, and in light of their association with multiple, independent lines of evidence supporting biological activity, the simple uniting explanation is that these tubes and filaments represent fossilised microbial remains.

# **Table S1 Laser Ablation parameters**



LA-ICP-MS instrument parameters

RF power 1340 W Ar Carrier Gas ow 1.04 l/min He gas ow 950 ml/min Sweep Time 400 / 900 ms (U-Pb / trace elements)

## **Table S2 Acquired masses for U-Pb dating. 235U was ommitted, natural 238U/235U ratio of 137.88 is assumed.**



# **Table S3 Acquired masses for trace elements**.

 $\overline{\phantom{0}}$ 



#### LA-Samples locations General rock Optical Raman SEM/ Formation Samples Age  ${\sf FIB}$ ICPtype microscopy microscopy EPMA MS Northings Eastings **НО6А, НО7В,** Løkken ophiolite, TG-82-S6, TG-82-Høydal jaspers, 0.485 Ga Jasper X X X Mine tailings H19 and H16, Norway Jah-1 and 2 Animike, Mesabi Stromatolitic range, Biwabik Iron MARY-ELLEN1  $1.8<sub>Ga</sub>$ Χ X Mine tailings jasper Formation, USA Brockman formation, Dales BIF/Fe-DGH1\_273'1" 2.5 Ga  $\mathsf{x}$ DGH1 drill core X  $\mathsf X$ gorge group, cherts Australia PC0822, PC0823, 58 16 50.9 77 44 14.7 PC0824 58 17 08.7 77 44 12.2 PC0825 **NSB** >3.77 Ga BIF/jasper  $\mathsf X$  $\mathsf X$ X  $\mathsf X$ X 58 17 09.1 77 44 10.8 PC028/A 58 16 53.8 77 44 14.1 PC0844

#### Table. S4 List of samples and their locations and methods used in this study

# Table S5. Iron-oxide tubes and filaments



## Table S6. Carbonate rosettes in IFs



## Table S7. Iron-oxide rosettes



#### Table S8 Wavelength-dispersive spectroscopy (WDS) analysis of various minerals in the NSB BIF and jaspers.



\*Sample makes reference to thin sections in Figure E3. Low totals attributed to no analysis for REE, hydroxyl and carbon.

#### Table S9 Raman parameters of graphitic carbon in carbonate rosettes (Fig. E6C).



\* Grains starting with G- are from the Raman scan in Fig 3D and grains starting with SG- are from the Raman scan in Fig E6.

#### Table S10 Raman parameters of graphitic carbon from NSB jasper sample (Fig. E8E).



#### Table S11 Raman parameters of poorly crystalline graphite from NSB. NR – Not resolvable (Fig. E8F).



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