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High-quality flake graphite occurrences in a high-grade metamorphic region in Sortland, Vesterålen, northern Norway

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The aim of this study has been to determine the quality of flake graphite in high-grade metamorphic rocks in Sortland, Vesterålen, northern Norway. Flake graphite is present in graphite schist and samples were collected at three different locations, Hornvatnet, Lamarkvatnet and Vikeid. The host rocks of the graphite schist are dolomite or calcite marble, pyroxene gneiss and amphibolite. Granulite-facies metamorphism on sedimentary material led to the formation of the graphite schist. According to previous studies, the graphite content of the schists in the area varies between 5 and 30%. Sampled graphite schists were fragmented by selective fragmentation (selFrag) and analysed using scanning electron microscopy, Raman spectroscopy and X-ray diffraction. The grain size and texture of the sampled graphite schists vary macroscopically. Some contain abundant, visible, big (up to 5 mm in size) and ordered graphite flakes with layered structure whereas other schists contain smaller graphite flakes and a lower graphite content. Pictures from the SEM indicate that the graphite flakes consist of several, parallel, graphene layers with clean surfaces. The results from both Raman spectroscopy and XRD support the SEM analyses that the flake graphite consists of several layers and also imply that the flake graphite is almost free from defects.

Keywords: *graphite, flake graphite, high-grade metamorphism, northern Norway*

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Introduction

Graphite is formed in metamorphic rocks under reducing conditions and increasing temperature and pressure. The typical source of graphite in metamorphic rocks is sedimentary organic matter which, when subjected to increasing metamorphic grade, forms a flaky variant of graphite. In the industry, the biggest consumer of flake graphite is the refractories industry, because flake graphite improves the refractory products' resistance to heat and erosion.

Flake graphite is also used in the steel industry, in brakes, and as lubricants (Mitchell, 1993). Additionally, flake graphite can be used to produce graphene, as demonstrated in 2004 by Konstantin Novoselov and Andre Geim (Novoselov et al., 2004). Graphene is a one atom thick, two-dimensional, hexagonal lattice layer of carbon atoms with many properties desirable in developing better electronics, e.g., in batteries, conductors, transparent electrodes and supercapacitors (Singh et al., 2011). The high electrical conductivity of graphene is prominent (Geim & Kim, 2008) and could be utilised in many different electronic devices such as in smartphone displays.

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In this paper we describe flake graphite occurrences in Sortland, Vesterålen, northern Norway. They are located in a high-grade metamorphic domain on the island of Langøy. This work builds upon previous mapping, exploration and characterisation of graphite occurrences in Norway performed, e.g., by the Geological Survey of Norway in the 1990s (Gautneb & Tveten, 2000). The aim of this study has been to determine the abundance of high-grade flaky graphite showing a well-ordered crystal lattice free from contaminants, thus suitable for industrial and technological use.

Graphite

Graphite consists of multiple layers of graphene, which is a two-dimensional network of one-atom thick layers of carbon. The carbon atoms in graphene are connected by strong covalent bonds and the layers are connected by weak van der Waals bonds (Hazen et al., 2013). In oxidising environments, graphite decomposes at 600°C, whereas in non-oxidising environments it melts at 3550°C (Mitchell, 1993). Graphite occurs most commonly in metamorphic rocks, but also in magmatic rocks as an accessory mineral (Hazen et al., 2013). Graphite is a common mineral in Norwegian rocks; however, it is rarely found at levels of economic interest regarding grade and carbon content. Graphite in metamorphic rocks is formed from sediments rich in organic matter as a result of metamorphism. Initially, the organic matter goes through a process of

carbonisation during diagenesis, which results in the solid residue getting enriched in carbon while impurities escape. The remaining carbon can be subjected to further burial and metamorphism and thus become completely graphitised (Buseck & Beyssac, 2014).

Flake graphite is most commonly present in mica schist and quartzite, or schist rich in both mica and feldspar. Impurities in graphite ore are generally quartz, feldspar, mica and amphibole. Since carbon creates a reducing environment, any metals present are bound to sulphur as sulphides. A good graphite ore should contain as few sulphide minerals as possible. Flake graphite ores are usually stratabound, and the ore bodies commonly occur as lenses in the surrounding bedrock (Mitchell, 1993).

Geological setting

The graphite schists investigated in this study are located on the island of Langøya close to Jennestad in Sortland, Lofoten–Vesterålen, northern Norway, at three different locations: Hornvatnet, Lamarkvatnet and Vikeid (Fig. 1). The graphite-bearing rocks belong to a suite of high-grade metasupracrustal rocks of Palaeoproterozoic age, probably deposited at around 1.95–1.87 Ga – the same time as the major depositional stage in arc environments in the Svecofennian domain (Lahtinen et al., 2002). Calcite and dolomite marbles, amphibolites and

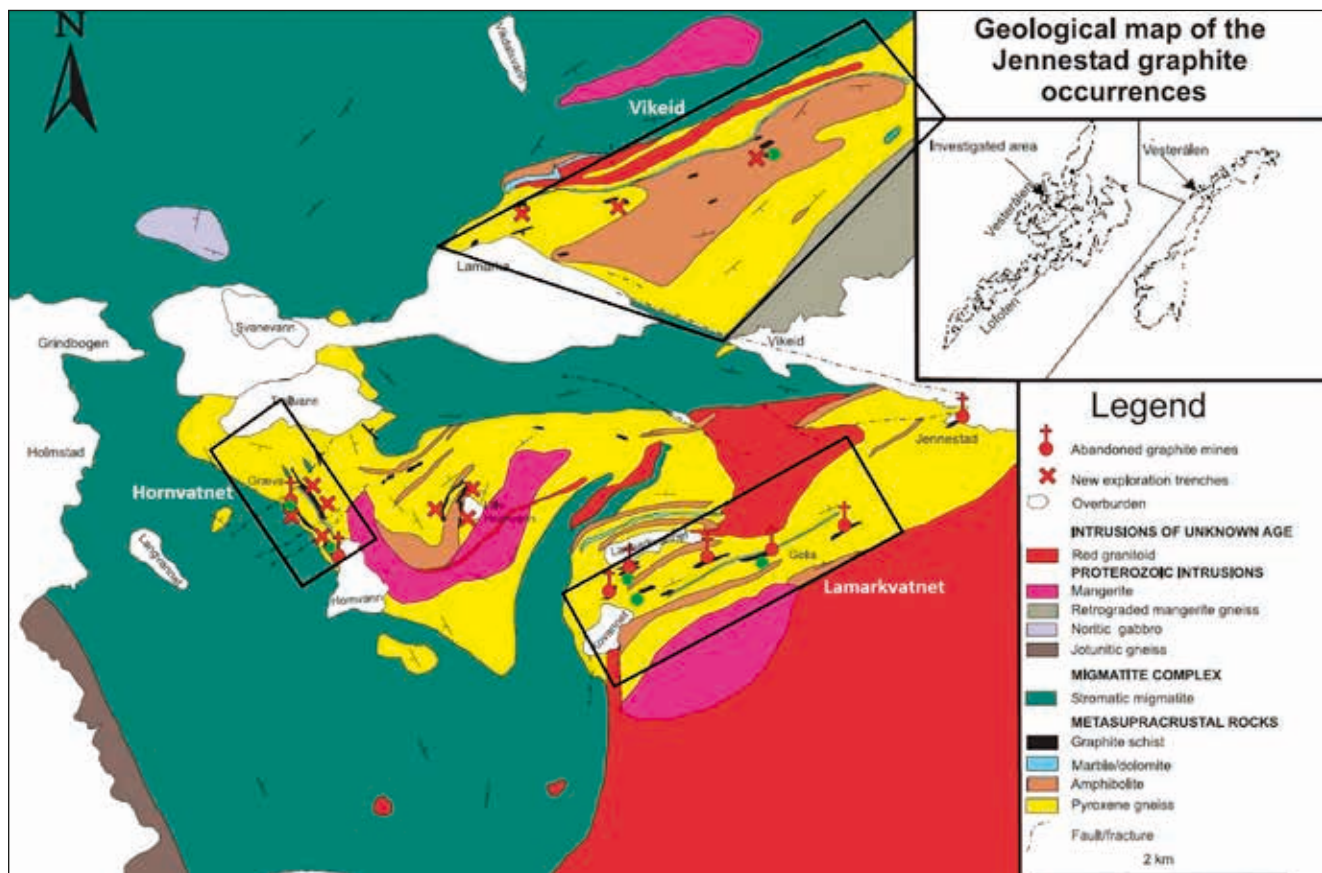


Figure 1. Geological map of the graphite occurrences (black veins) in Jennestad, Sortland (modified from Gautneb & Tveten, 2000). The investigated areas for this study are marked with black lines and the sample collection sites with green dots.

pyroxene gneisses are part of this suite. Based on their chemical composition, the protoliths of the metasedimentary rocks are of sedimentary and volcanic origin, probably from a shallow-marine environment (Griffin et al., 1978). The islands of Lofoten–Vesterålen can be divided into western and eastern segments. The western segment consists of gneisses in granulite facies and an intrusive anorthosite-mangerite-charnockite-granite (AMCG) complex, principally 1.8 Ga in age (Corfu, 2004), while the eastern part consists of rocks in amphibolite facies and felsic intrusive rocks which lack orthopyroxene. The transition zone also coincides with the eastern border of a high-magnetic anomaly (Corfu, 2007). All locations in this study lie in the transition zone between amphibolite and granulite facies. According to Ekholm (2015), there is some difference in metamorphic grade in the bedrock surrounding the graphite; orthopyroxene is found only in the Hornvatnet area, which is the westernmost of the investigated areas. This implies that the metamorphic grade in the area is increasing to the west. The bedrock in contact with the graphite schists consists of calcite and dolomite marble (hanging wall) and amphibolite (footwall), but pyroxene gneisses are also found close to the graphite schists. The rocks of the Lofoten–Vesterålen area have been subjected to granulite-facies metamorphism at several occasions during Archaean and Proterozoic time. Since evidence for all these metamorphisms is found on both sides of the transition zone between granulite and amphibolite facies, the zone could represent a tectonic boundary instead of a transition due to just one metamorphic event (Corfu, 2007).

In the area, the graphite schists and the other associated metasedimentary rocks are mostly covered by superficial deposits. The best locality to study the setting of the graphite schists is at the entrance of the abandoned graphite mine Golia, south of Lamarkvatnet (Fig. 1). There, the graphite-bearing bodies are commonly 2–4 m thick (Gautneb & Tveten, 2000). The graphite schist strikes NW–SE at Hornvatnet, N–S and NE/E–SW/W at Lamarkvatnet and NE–SW at Vikeid. The graphite schists at Hornvatnet are steeply dipping, nearly vertical, towards southwest. At Lamarkvatnet and the Golia mine, the graphite schist strikes from west to east and dips c. 60° towards north (Gautneb & Tveten, 1992). The amount of graphite in the graphite schists in the area varies a lot between c. 10% and 30%, but the average graphite content is approximately 19 wt.% (Gautneb & Tveten, 1992).

Materials and methods

Of the samples collected, 7 samples of graphite schists were prepared for further study (Table 1). Macroscopically, the graphite schists differ slightly from each other concerning grain size and graphite content. The grain sizes in the schists vary from fine to medium, but some of the schists are even coarse grained. To determine the quality of the flake graphite, flakes extracted by selective fragmentation (selFrag) were subjected to study by scanning electron microscopy (SEM), X-ray diffraction (XRD) and Raman spectroscopy.

Petrography

The petrography of the material was documented from thin-sections and SEM. With SEM, the principal composition of some minerals was measured. No detailed metamorphic study was undertaken, because of the uncertainties in the chemical composition obtained by SEM. However, we present some general data of the mineral chemistry. Depending on the host rock, graphite appears in different ways. Graphite was found in two main rock types in the sampling area: pyroxene gneiss and metapelite. The pyroxene gneiss (Fig. 2A) consists mainly of diopside ($wo_{53.43}$, $en_{42.04}$, $fs_{4.54}$, $(Fe/Fe+Mg) = 0.10$ and Al_2O_3 around 3 wt.%), graphite and poikiloblasts of spinel ($(Fe/Fe+Mg) = 0.331$, $ZnO = 2.5$ wt.%) and some garnet ($pyr_{81.35}$, $alm_{15.20}$, $gross_{3.45}$ and $and_{1.07}$). In the pyroxene gneiss, black veins are identified as graphite and brown veins as iron oxides together with minor amounts of Mg, Si and Al. The graphite grains have 120° borders and no foliation is visible in the pyroxene gneiss. The foliated metapelite (Fig. 2B) contains K-feldspar, quartz, plagioclase (oligoclase), graphite and biotite, while clinopyroxene ($wo_{49.97}$, $en_{36.74}$, $fs_{3.28}$, $Fe/Fe+Mg = 0.27$), orthopyroxene, apatite and pyrite are present as minor phases in the sample. The Ti content of the biotite was measured to approximately 6 wt.%. The metapelite samples are mainly evenly grained and medium to fine grained in size. The graphite appears deformed together with the biotite. The majority of the graphite flakes in the samples are stubby, elongated and 0.5–1 mm long, and some flakes reach up to 1.2 mm in length.

Table 1. Processing and analytical methods applied to the Sortland graphite schist samples.

Sample	300220.1	300247.1	300247.2	300251.1	300266.3	300289.1	300295.1
Coordinates (UTM33)	X: 507563 Y: 7625339	X: 509899 Y: 7625179	X: 509899 Y: 7625179	X: 509989 Y: 7625014	X: 510968 Y: 7625359	X: 510983 Y: 7628548	X: 507235 Y: 7625664
Location	Hornvatnet	Lamarkvatnet	Lamarkvatnet	Lamarkvatnet	Lamarkvatnet	Vikeid	Hornvatnet
Selective fragmentation	X			X	X	X	X
SEM	X	X	X	X	X	X	X
XRD			X			X	X
Raman			X			X	X

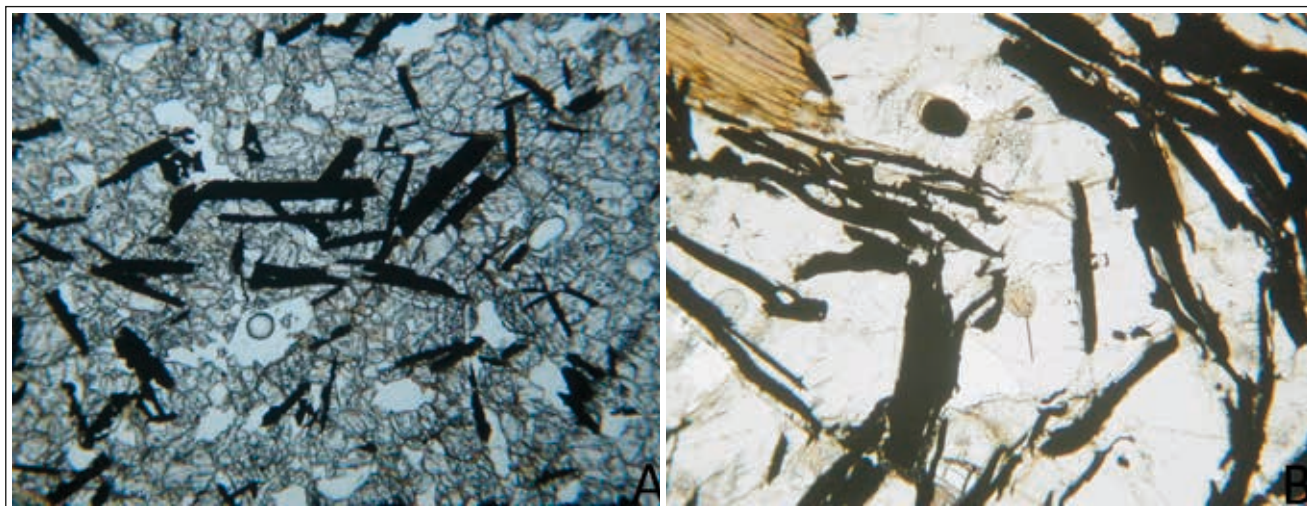


Figure 2. (A) Graphite-bearing pyroxene gneiss and (B) metapelite as seen in a polarisation microscope. The black nails are graphite flakes. The pictures are 1.88 mm across.

Selective fragmentation

Selective fragmentation (selFrag) was performed at the research laboratory of the Geological Survey of Finland in Espoo. This method was used to test if it is suitable for separating graphite from other minerals and separating the graphite flakes from each other. The method uses short (2–5 Hz), high-voltage pulses (150–200 kV) directed on the sample in a container filled with water and gaseous nitrogen. The goal is to obtain mineral aggregates fragmented at mineral grain boundaries (Wang et al., 2011).

The first round of fragmentation for sample 300289.1 was performed with 25 pulses at 100 kV with a frequency of 3 Hz, which took 2–3 minutes. For the second round of fragmentation the pulses were increased to 50 at 100 kV and for the third round to 100 at 130 kV. The settings were then changed because the material did not fragment as desired. Samples 300220.1, 300251.1, 300266.3 and 300295.1 were fragmented using the same settings for three rounds, i.e., 50 pulses at 130 kV with 3 Hz frequency, except on round two for sample 300251.1 with 30 pulses at 130 kV and 3 Hz frequency. After fragmentation, the samples were left to dry in an oven at 60°C for four days. The material was sieved into different fractions after fragmentation: >2 mm; 1–2 mm; 0.59–1 mm; 0.5–0.59 mm; 0.25–0.5 mm; and <0.25 mm.

Scanning electron microscopy

The scanning electron microscope (SEM), a LEO Gemini 1530 FEG–SEM instrument with a ThermoNoran X-ray detector, at the Laboratory of Inorganic Chemistry, Åbo Akademi University was used to examine two thin-sections and the graphite flakes obtained by selective fragmentation. Sample 300247.2 consisted of graphite flakes that were manually extracted from the graphite schist. As the graphite sample is conductive, no additional conductive layer was needed. The samples from the selective fragmentation were also examined with the SEM. Energy dispersive X-ray analysis (EDXA, Ultradry) was used for elemental analysis. The goal was to determine if the graphite schists differ from each other in flake size, if

there are any impurities in the schists or in the graphite flakes, and how the selective fragmentation might have affected the samples.

X-ray diffraction

X-ray diffractometry (XRD) was used to determine the distance between the graphene layers in the flake graphite and to check for potential impurities. The analysed samples were 300247.2, 300289.1 and 300295.1 (Table 1). The XRD used was a Bruker D8 Discover, located at the Laboratory of Physical Chemistry at Åbo Akademi University, and the wavelength used was Cu–K α (1.5406 Å). The measurements with the X-ray diffractometer were performed with a point detector. The samples were measured on a single spot and scanned in the range 20° to 80° several times for a better signal-to-noise ratio. Samples 300289.1 and 300295.1 were ground to powder (grinding can contribute to better statistics obtained from the analysis) while 300247.2 was a single \varnothing 2 mm grain with a thickness of 0.5 mm.

Raman spectroscopy

Raman spectroscopy can reveal defects in the sample and how thick the layers are in, e.g., flake graphite. The peak metamorphic conditions can also be defined from Raman spectroscopy measurements, since the variations in the metamorphic grade affect the degree of organisation of the carbonaceous material (Beysac et al., 2002). The tested samples were 300247.2, 300289.1 and 300295.1 (Table 1) and the measurements were made with the Raman spectrometer at the Laboratory of Analytical Chemistry, Åbo Akademi University. The Raman spectra were recorded using a Renishaw Ramascope (system 1000) equipped with a Leica DMLM microscope connected to a CCD camera. The measurements were made by using an Ar-ion laser (LaserPhysics) with the excitation wavelength of 514 nm on a c. \varnothing 2 μ m surface area with a laser power of 20 mW. The spectrometer was calibrated against a Si standard (520.0 cm⁻¹).

Results

The mineralogy of the pyroxene gneiss and metapelite was identified in thin-sections and with SEM. According to the results from the analyses, the pyroxene gneiss consists mostly of diopside and graphite and the metapelite of K-feldspar, quartz, plagioclase, biotite and graphite. The graphite flakes vary in size (Fig. 2A & B), and are up to 1.2 mm in length. The flakes are both evenly distributed

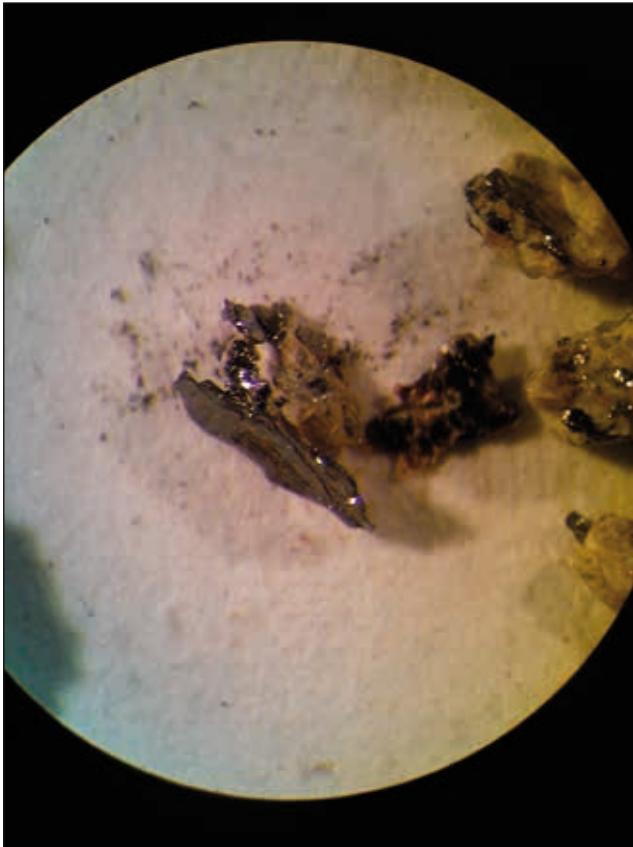


Figure 3. Distinct graphite flakes after fragmentation in a selFrag device (sample 300266.3), fraction 0.5–1 mm. The big flake is approximately 1 mm in length.

in the rock and concentrated in fractures and along the foliation. Some graphite flakes seem to be folded and broken, which may imply that the rock has been affected by tectonic stresses after the crystallisation of the graphite flakes. The flakes are situated in possible pressure shadows and also associated with metamorphic triple-junction textures (seen in Fig. 2A) throughout the sample.

Selective fragmentation

After three sets of high-voltage pulses the graphite schists were fragmented, but not completely. Many mineral grains were still attached to each other on a microscopic scale. In an optical microscope, graphite, feldspars, quartz and some rusty unidentified minerals were observed in the sample. Some graphite flakes were still attached to other minerals, while others were not (Fig. 3). The results from fragmentation of the samples obtained with selective fragmentation are promising, but the method needs to be optimised and investigated further in order to establish the optimal parameters for fragmentation and for not breaking the graphite flakes.

Scanning electron microscopy on grains

The different size fractions of graphite flakes obtained after fragmentation were examined with a scanning electron microscope (Table 1). Almost all graphite fragmented by selFrag is deformed. This could be an effect of the high voltage electrical pulses passing through the samples in the selFrag, but could also be a result of tectonic deformation. In some places, the layers of the graphite flakes seem to have slipped relative to each other and space has formed between the layers in microscopic folds (Fig. 4A). These structures must have formed during the secondary handling of the sample, i.e., the selFrag treatment. The SEM images also show that all the samples consist of flaky graphite, i.e., that one graphite flake consists of several layers, with regular and irregular flake edges and clean flake surfaces. Sample 300247.2 differs from the other samples in that the graphite flakes do not appear to be as intensely deformed as in the other samples (Fig. 4B).

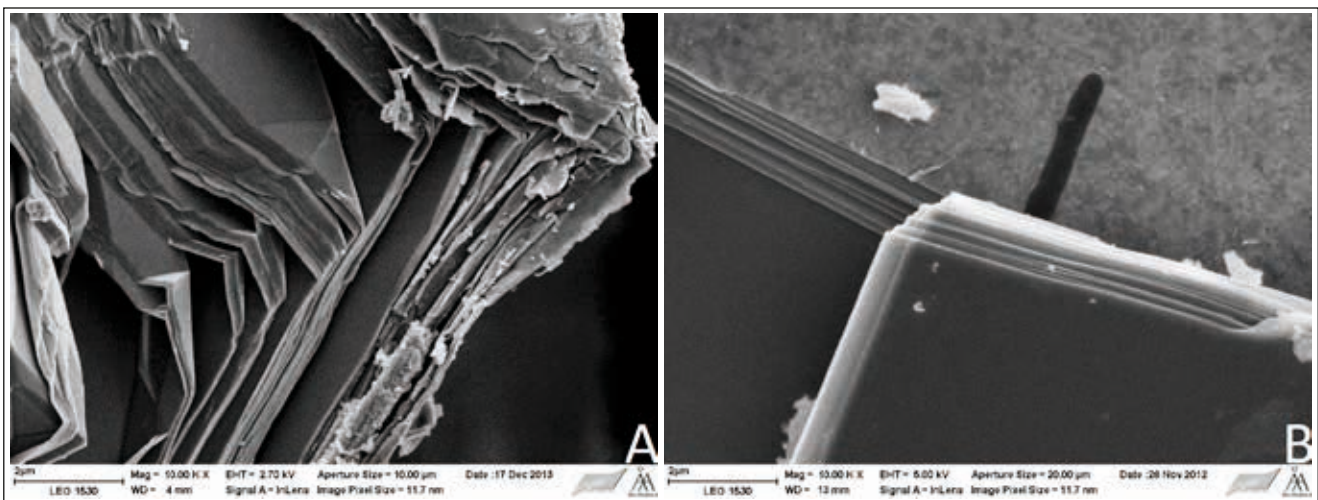


Figure 4. (A) Selectively fragmented graphite flakes (sample 300220.1) as seen in SEM; and (B) manually fragmented flakes from graphite schist (sample 300247.2). The graphite flakes fragmented by selFrag are folded.

X-ray diffraction

The obtained XRD results on graphite, both single grain and powder, are presented in Fig. 5. For the 300247.2 sample (single grain), only two sharp reflections related to graphite are visible, indexed as (002) and (004), and indicate a long-range ordering in the atomic structure of the flake graphite. For the 300289.1 and 300295.1

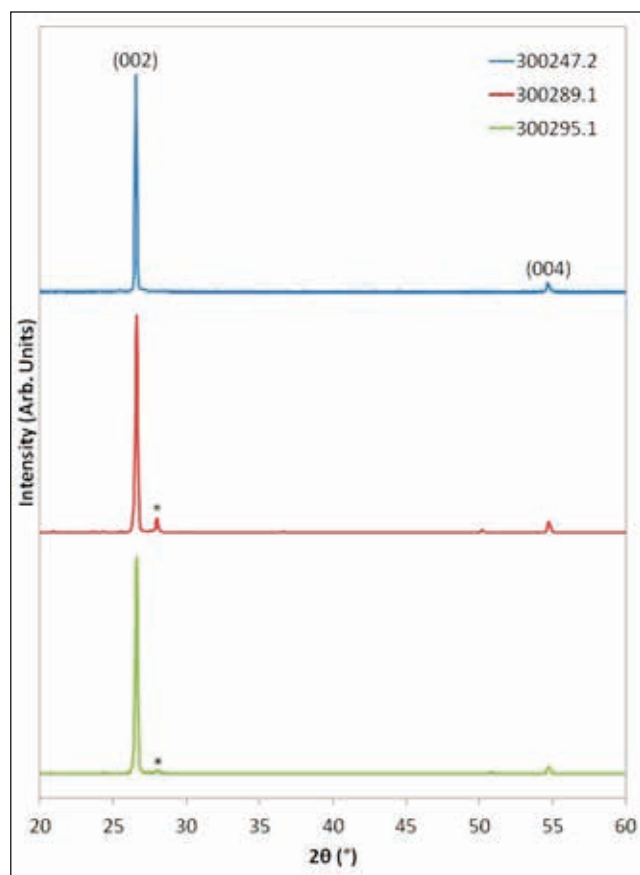


Figure 5. The XRD results for samples 300247.2 (blue), and 300289.1 (red) and 300295.1 (green). The plots have been offset for clarity.

samples (powder), traces of quartz and albite were also observed in addition to the (002) and (004) graphite reflections (e.g., albite is indicated with an asterisk in Fig. 5). The slight peak broadening of the (002) reflection for the latter samples indicates that the graphite crystals are smaller or slightly less ordered. There appear not to be any differences between the (002) position of the samples, which indicates that the distances between the graphene layers are similar. The d spacing between the graphene layers was analysed from the (002) reflection to be 0.335–0.336 nm, which is the expected distance between the graphene layers in graphite (Pierison, 1993).

Raman spectroscopy measurements

Both manually fragmented (300247.2) and selectively fragmented (300289.1 and 300295.1) samples were examined with Raman spectroscopy. The spectrum of the manually fragmented sample is shown in Fig. 6A, measured at two different positions on the crystal shown on the microscope image in the inset. The Raman spectra of the samples fragmented by the selfFrag method are shown in Fig. 6B & C. The most intense features of Raman spectra of graphite are the so-called G band and 2D band (also called G' band) (Ferrari et al., 2006; Eckmann et al., 2012; Wall, 2012). The G band is characteristic for the in-plane vibrational mode involving sp^2 -hybridised carbon atoms which comprise the graphene sheets in graphite. The position of the G band is highly sensitive to the number of graphene layers and in graphite it is at c. 1580 cm^{-1} . The G band position shifts to a higher energy (higher wave numbers) location as the layer thickness decreases (Wall, 2012).

The narrow G band in the manually and selectively fragmented samples is present at c. 1579 cm^{-1} (Fig. 6), which implies that the material is graphite formed at high-grade metamorphic conditions (Beyssac et al., 2002). If the G band were to be present at lower wavenumbers, the material would possibly be amorphous graphite (Ferrari et al., 2006). The 2D band is supposedly unrelated to the G band; rather, it is an overtone of the so-called D band. The D

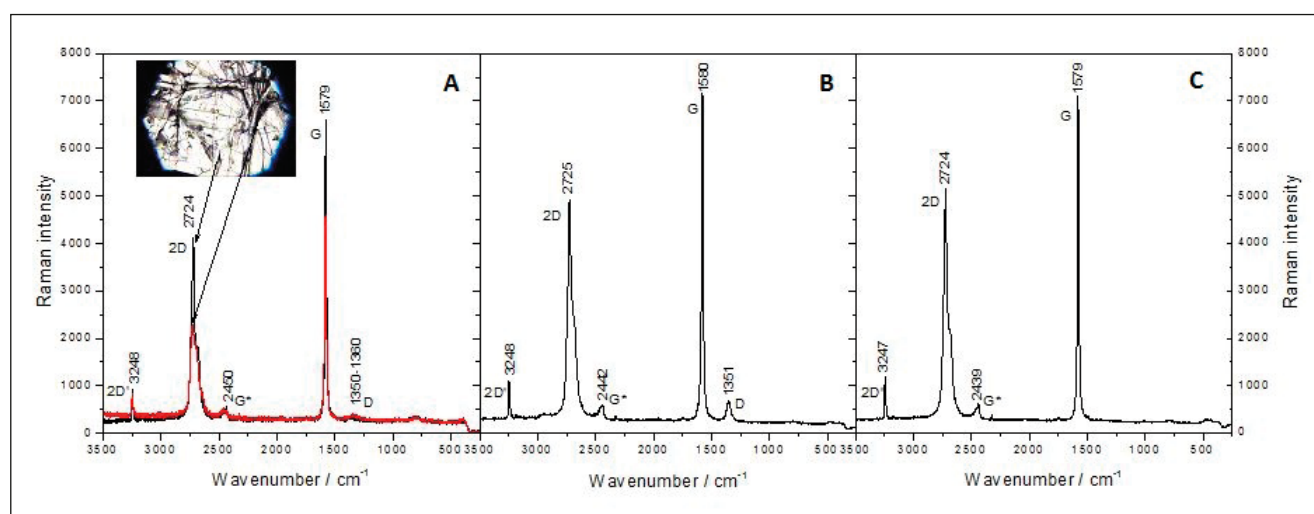


Figure 6. Raman spectra of (A) sample 300247.2, (B) sample 300289.1 and (C) sample 300295.1. The measuring points are marked with arrows in (A). The laser excitation wavelength used is 514 nm.

band, located at c. 1350 cm^{-1} , is characteristic for unorganised carbon. It is also known as a disorder or defect band and represents a mode from sp^3 -bound carbon (Ferrari, 2007; Wall, 2012). The D band intensity decreases in relation to the G band due to stiffening of aromatic planes. On the other hand, the intensity of the D band increases with a decrease in the graphite crystal size and therefore it can be said that the presence of the D band indicates that the crystallite size is smaller than a few microns. A low-intensity D band is visible at 1350–1360 cm^{-1} in the manually fragmented sample shown in Fig. 6A and at 1351 cm^{-1} in one of the selectively fragmented samples (300289.1) shown in Fig. 6B. The other selectively fragmented sample (sample 300295.1, Fig. 6C) did not show any D band at all.

In contrast to a single-layer graphene having a symmetrical 2D band with a high intensity at c. 2700 cm^{-1} , the 2D band in bulk graphite is composed of four components, two of which have higher relative intensities than the other two and shown as a shoulder towards lower intensities (Ferrari, 2007; Malard et al., 2009; Wall, 2012). The maximum of the 2D band in the manually fragmented sample is located at 2749 cm^{-1} and in the selectively fragmented samples at 2725 cm^{-1} and 2724 cm^{-1} , respectively (Fig. 6). The asymmetric 2D band implies that there are many parallel layers of graphene in the samples. The fourth characteristic band visible in the graphite Raman spectra is called the 2D' band at c. 3250 cm^{-1} . It is the second order of the intra-valley peak called D' at c. 1620 cm^{-1} . The D' band becomes visible when the laser is shot on a boundary edge, formed by cutting the graphene planes at different angles (Malard et al., 2009). The 2D' band is visible in all the graphite spectra shown in Fig. 6. The fifth characteristic band in graphite Raman spectra is visible at c. 2450 cm^{-1} and is called as G*. Several assignments exist for this band, but it is believed to originate from an inter-valley process as an overtone of in-plane modes (Nemanich & Solin, 1979; Ferrari & Basko, 2013).

The degree of organisation of carbonaceous material is found to be highly sensitive to variations in the metamorphic grade. It has been shown by comparing Raman spectra from several reference samples from different metamorphic belts that Raman spectroscopy can be used as an indicator of the metamorphic conditions (Wopenka & Pasteris, 1993; Beyssac et al., 2002). The ratio between the peak area of the D band and the sum of the areas of the G, D and D' bands (R) has been used as a reliable parameter to follow the graphitisation process. A linear relationship between R and temperature has shown R values <0.2 at temperatures between 500 and 650°C. In contrast, no clear trend has been shown between R values and pressure. The R values for the manually fragmented sample shown in Fig. 6A are 0.05 and 0.15 for the spectra measured at different locations on the sample surface. The corresponding R value for the selectively fragmented sample (Fig. 6B) is 0.13. According to the geothermometer based on Raman spectroscopy, the flake graphite deposits in Sortland in Vesterålen were formed at temperatures between 575 and 620°C. The metamorphic pressure during the graphitisation process, on the other hand, is more difficult to determine by Raman spectroscopy, but could have been between 5 and 20 kbar.

Discussion

The transformation of carbonaceous material to fully ordered graphite is dependent on the temperatures during diagenesis and metamorphism (Buseck & Beyssac, 2014). At lower grades of metamorphism, organic matter transforms into graphite. With increasing metamorphic conditions, annealing processes purify the graphite layers and initiate growth of the flakes. During these processes, the lattice structure converts to more perfect graphene layers (Buseck & Huang, 1985). The grade of metamorphism is thus essential for the quality of flake graphite. In Sortland, the maximum grade of metamorphism is lower granulite facies, since garnet is not present, while the observed paragenesis (ortho- and clinopyroxene, biotite, hornblende and minor quartz) indicates granulite facies (Best, 2003).

Our petrographical and SEM studies have shown how the graphite flakes are evenly distributed in the graphite schist and are up to >1 mm in size. Selective fragmentation (selFrag) has turned out to be effective for obtaining graphite flakes from the graphite schist. By using SEM, Raman spectroscopy and XRD we could establish that the graphite flakes consist of several layers of graphene, that the crystallographic ordering within the flakes is high, and that there are no remarkable defects in the crystal structure. Raman spectroscopy was the most suitable method for analysis of the quality of the flake graphite. The quality varies between the examined graphite schists: the samples with visible graphite flakes (sample 300247.2) and/or high carbon contents (samples 300219.1, 300230.2 and 300295.1) are of high quality, whereas the samples of lower quality are those with low carbon content (300266.3 and 300251.1), and the sample (300289.1) that indicated the presence of sulphur during fragmentation.

Future studies should focus on exfoliation of flake graphite, i.e., how to separate graphite flakes from the host rock and the flakes from each other. Furthermore, as flake graphite is of substantial economic value, a non-destructive exfoliation process should be developed to obtain as large flakes as possible from the host rock.

Conclusions

Flake graphite from Sortland, northern Norway, has been examined. The bedrock in Sortland consists of pyroxene gneiss, amphibolite, calcite and dolomite marble with serpentinised olivine, and graphite-bearing metapelite metamorphosed to amphibolite and lower granulite facies. Several metamorphic events have been recorded in the rocks in the Lofoten–Vesterålen region, which have led to the formation of flake graphite from sedimentary carbonaceous material. According to our SEM, XRD and Raman spectroscopy results, flake graphite in Sortland is of high quality and does not contain any significant impurities. The Raman spectroscopy measurements show that the flake graphite has formed at temperatures between 575 and 620°C, temperatures indicating amphibolite facies. However, these numbers should be treated with caution, since they are not in accordance with the general petrography of the rocks of the area that indicates lower

granulite facies. There does not appear to be any difference in the quality of the flake graphite between the rocks in the three investigated areas. The results indicate that the Sortland flake graphite is of high quality and excludes the presence of any significant number of defects in the graphite deposits. The selfFrag method also seems to be useful for cleaning and fragmentation purposes for extraction of the graphite.

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