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2012 J. Geophys. Eng. 9 S118

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# Characterizing the hypersiliceous rocks of Belgium used in (pre-)history: a case study on sourcing sedimentary quartzites

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Received 28 January 2012

Accepted for publication 15 May 2012

Published 9 August 2012

Online at [stacks.iop.org/JGE/9/S118](http://stacks.iop.org/JGE/9/S118)

## Abstract

Tracking raw material back to its extraction source is a crucial step for archaeologists when trying to deduce migration patterns and trade contacts in (pre-)history. Regarding stone artefacts, the main rock types encountered in the archaeological record of Belgium are hypersiliceous rocks. This is a newly introduced category of rock types comprising those rocks made of at least 90% silica. These are strongly silicified quartz sands or sedimentary quartzites, siliceous rocks of chemical and biochemical origin (e.g. flint), very pure metamorphic quartzites and siliceous volcanic rocks (e.g. obsidian). To be able to distinguish between different extraction sources, ongoing research was started to locate possible extraction sources of hypersiliceous rocks and to characterize rocks collected from these sources. Characterization of these hypersiliceous rocks is executed with the aid of optical polarizing microscopy, optical cold cathodoluminescence and scanning-electron microscopy combined with energy-dispersive x-ray spectrometry and with back-scatter electron imaging. In this paper, we focus on various sedimentary quartzites of Paleogene stratigraphical level.

**Keywords:** siliceous rock, stone artefacts, extraction source, optical cathodoluminescence microscopy, electron microscopy, traceability, distinguishability, prehistory

(Some figures may appear in colour only in the online journal)

## 1. Introduction

The application in (pre-)history of rocks ranges from work tools to foundation and building material, millstones, refractory material, flintlocks and other objects of our cultural heritage. Generally, the assumptions concerning the extraction source of the stone material rely on macroscopic lithological characteristics (Di Modica 2005, Lan 2006, Collet and Woodbury 2007). Mostly, non-destructive techniques are preferred. Examples are scanning-electron microscopy (SEM) applied to both the surface of the raw rock sample and the stone artefact, and the minimally destructive technique of

laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) (Grégoire 2001, Bressy 2002). These techniques give no unequivocal results due to surface alteration of the stone artefact over time compared to the recently extracted rock sample. In the case of hypersiliceous rocks, this visual macro- and mesoscopic approach is often inadequate to differentiate between similarly looking hypersiliceous rocks of different extraction sources (e.g. Lan 2006: 209), as will also be shown in this paper. In those cases, it seems necessary to revert to destructive techniques such as the use of thin sections and powders of a stone sample for both petrological and geochemical studies (Akridge and Benoit 2001). Often

enough stone material of less aesthetic value is available at the site. Many methodological studies have been carried out to identify and source silica-rich rocks and are mainly focused on flint/chert, obsidian and metamorphic rocks (Akridge and Benoit 2001, Burke and Gauthier 2008). Only several studies specifically on sedimentary, detrital siliceous rocks have been carried out (Alexandre *et al* 2004, Ullyott and Nash 2006, Kelly *et al* 2007, Pitblado *et al* 2008). In Belgium, the identification of lithic artefacts is carried out occasionally by a geologist on demand, without a proper systematic approach (De Paepe and Pieters 1994, Pirson *et al* 2001). A thorough and methodological study of hypersiliceous rocks of Belgium is missing. The aim of this paper and our ongoing research is to locate the possible (pre-)historical extraction sources of hypersiliceous rocks in Belgium and to characterize the raw rock samples by petrographical and geochemical techniques. These extraction sources range from loose, individual blocks in superficial deposits, exhumed rock formations or outcrops, and mining sites, such as the Neolithic mining complex of Spiennes (Collet *et al* 2008). The results will be synthesized in a reference collection of hypersiliceous rocks containing the raw rock sample, thin sections and both the petrographical and geochemical descriptions obtained by optical microscopy, optical cold cathodoluminescence (CCL) microscopy and SEM. Tracking raw material back to its extraction source is a crucial step for archaeologists (Gendel 1982, Pirson *et al* 2001, Di Modica 2011). This reference collection will make it possible to compare stone artefacts with raw rock samples from known extraction sources, aiding the archaeologist in deducing human migration patterns and trade contacts within (pre-)history.

## 2. Definition of hypersiliceous rocks

In the literature, the term siliceous rocks typically refers to very fine-grained silica-rich rocks formed 'by the precipitation of silica at or near the site of deposition or by replacement of a pre-existing rock' (Middleton 2003). This definition excludes other high siliceous rocks such as the group of silicified quartz sand deposits, better known as sedimentary quartzites or orthoquartzites (Krynine 1941, 1948), in which the components are made up of quartz grains originating from weathered and transported pre-existing rock. Also excluded from this definition are very pure metamorphic quartzites and siliceous volcanic rocks such as obsidian. To unify all these high siliceous rock types, composed of at least 90% SiO<sub>2</sub>, into one generic reference, a new category is introduced: hypersiliceous rocks. An incentive for the introduction of hypersiliceous rocks is that it encompasses the most frequent rock types found in the archaeological record. In the literature, 'hypersiliceous' has been used as an adjective to indicate rocks, air particles or magma fluids with a high silica content (Racki 2000, Ausset *et al* 1998, Bard 1986). When it is used in the context of rock studies, it is often used as a synonym for siliceous rocks. To the authors' best knowledge, the term hypersiliceous rocks has never been strictly defined.

Hypersiliceous rocks are geographically widely distributed in Belgium and belong to different stratigraphical

levels from Cambrian to Neogene (Tertiary) in age. As can be concluded from Di Modica (2011), the main types of hypersiliceous rocks in the Belgian archaeological record are as follows: (1) flint from the Cretaceous, (2) sedimentary quartzites or strongly silicified quartz sand and silt deposits from Paleogene strata, (3) 'phtanites', a term used by archaeologists to refer to the carbonaceous radiolarites from the Cambrian Mousty Formation, (4) chert from the Carboniferous limestones, and finally (5) some pure Paleozoic quartzites.

## 3. Approach

It is the authors' opinion that research on sedimentary quartzites is rather scarce compared to studies of flint and other rock types in the archaeological records. Therefore, in this paper, we focus on the characteristics of sedimentary quartzites of the Paleogene Tienen Formation and St-Huibrechts-Hern Formation (figure 1). Sedimentary quartzites of the Tienen Formation are often encountered at archaeological sites in Belgium and neighbouring countries as work tools, e.g. scrapers and microliths (Van Neste 2009, Sergant *et al* 2009, Amkreutz and Verpoorte 2009).

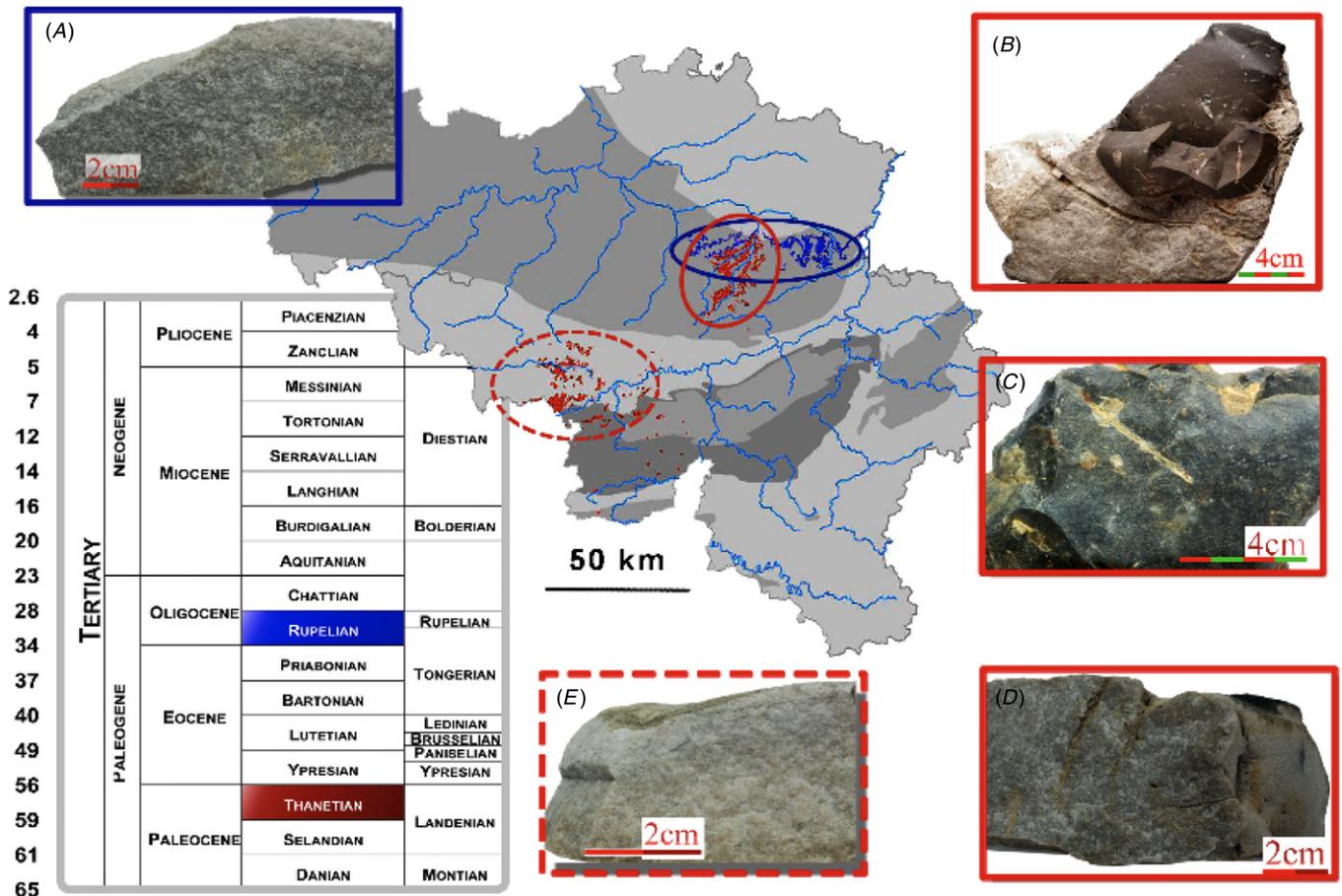
Inevitably, in traceability research, simply describing raw rocks from an extraction source is insufficient. A raw rock must also be described in a relative way, comparing it to raw rocks from other extraction sources. In this paper, the following terminology will be used to compare rocks.

The variability of a certain rock property within an extraction source is the amount rocks can differ in that property despite the fact that the rocks originate from the same extraction source (intra-source variability). The difference in a certain rock property between two extraction sources is the amount an average rock from the first extraction source can differ in that property from an average rock from the second extraction source. This difference in a certain rock property has to be studied taking into account both extraction sources' variability. In this paper the term distinguishability refers to the amount of difference in a certain rock property between two extraction sources relative to the amount of the variability in that property in each of the extraction sources. Large differences and small variabilities result in a good distinguishability.

Due to the scope and explorative nature of the ongoing research, insufficient samples were studied to allow for a thorough statistical analysis. However, a comparative study of relative rock properties of stone material from different extraction sources is performed using varying techniques to discover useful distinguishabilities. If a trend is found to be worthwhile to focus on, a more thorough statistical analysis can be performed later.

## 4. Terminology

The term quartzite usually refers to the mineralogy and origin of a rock, being the result of metamorphism in which a quartzarenite evolved into a quartzite as a result of increased pressure and temperature. The general distinction



**Figure 1.** Chronolithostratigraphical table with, in the furthest right column, the former ‘étage’ names. The Paleogene Formation of St-Huibrechts-Hern is of Rupelian age, which is highlighted (in blue) in the time table. Its distribution is indicated in blue with a blue circle on the map of Belgium. Within this formation, the *Alden-Biesen quartzite* (A) occurs. The Paleogene Formation of Tienen is of Thanetian age, which is highlighted (in red) in the time table. Its spatial distribution is indicated with both a continuous vertical oval and dashed horizontal oval (both in red) on the map of Belgium. The distribution of the Dormaal Member is situated in the Hesbaye region indicated with a continuous vertical oval (in red). In the Dormaal Member occur the *Wommersom quartzite* (B) (in the figure shown as a nodule within the *grey Wommersom quartzite*), the *black Tienen quartzite* (C) and the *Tienen quartzite* (D). The distribution of the Erquelinnes Member is situated in the Hainaut region, indicated with a dashed horizontal oval (in red), and contains the *quartzite of Bray* (E). Note the fossilized rootlets within all three quartzite samples of the Dormaal Member (B)–(D). Indicated on the map of Belgium in greyscale are the main geological structures (Brabant Massif, Stavelot Massif, Givonne Massif, Serpont Massif, Rocroi Massif, Dinant synclinorium and Namur synclinorium).

between a quartzite and a quartzarenite, its precursor, is the fracturing through the individual grains instead of along the grains, as is the case for its precursor. Nowadays, the term quartzite can refer to both a rock of sedimentary, detrital origin (quartzarenite), known as sedimentary quartzite, and of metamorphic origin (‘quartzite’), a metamorphic quartzite (Bates and Jackson 1987). The assignment of the term quartzite to a quartzarenite is due to the fact that this type of quartzarenite is so tightly cemented that it resembles physically and mineralogically a metamorphic quartzite, which it can easily be mistaken for. In a sedimentary quartzite, the grains are so thoroughly cemented that by fracturing, the grains act as a single crystal, while in a regular quartzarenite the fracture passes along the individual grains (Krynine 1948). To avoid confusion in terminology, Krynine (1941, 1948) introduced the terms orthoquartzite in contrast to metaquartzite, the former representing the strongly cemented quartzarenite or sedimentary quartzite, and the latter being the

result of recrystallization by metamorphism. This division is to underline the difference in their genetic origin. Because of the multiple misuse of the term orthoquartzite (Ireland 1974), the more general term ‘sedimentary quartzite’ is used throughout this paper to indicate these rocks’ sedimentary origin and strongly cemented nature resembling metamorphic quartzites.

## 5. Geological background

The formation of Tienen, consisting of Late Paleocene continental-lagoonal deposits, corresponds to the former ‘étage Landénien supérieur’ (19th century geological maps: L2). The quartzites of the Tienen Formation crop out in two main regions in Belgium: in the eastern part of the country, more specifically the Hesbaye region, and in the western part of the country, more specifically the Hainaut region (figure 1). The quartzites of the Tienen Formation in the Hesbaye region belong to the Dormaal Member, whereas the

quartzites of the Hainaut region belong to the Erquelines Member (Laga *et al* 2001). The main sedimentary quartzite in the Hesbaye region is known as the *Tienen quartzite* (Gulinck and Tavernier 1947, De Geyter 1981, Di Modica 2011), whereas the most well-known quartzite of the Hesbaye region among archaeologists is the *Wommersom quartzite* (Di Modica 2011). Previously unrecognized quartzites in the region of Hesbaye are the *grey Wommersom quartzite* and *black Tienen quartzite*, which are defined and described here for the first time in the geological literature. The outcropping sedimentary quartzite in the Hainaut region is known as ‘*Grès de Bray*’ or *quartzite of Bray* synonymous with *quartzite of Binche*, which is the stratigraphical equivalent of the *Tienen quartzite* (Gulinck and Tavernier 1947). The formation of St-Huibrechts-Hern of early Oligocene age and outcropping in the Limburg province contains at the top of its Member of Neerrepn, a sedimentary quartzite level. This quartzite level, occurring within the very pure white sands of the Neerreperi Member, is known as ‘zoetwaterkwartsiet van Alden-Biesen’ or *quartzite of Alden-Biesen* (Gulinck 1968, Dreesen and Duser 2008). This unit corresponds to the lower part of the former ‘*étage Tongrien inférieur*’ (19th century geological maps: Tg1).

## 6. Methodology

*In situ* samples of the *Tienen quartzite* and the *Alden-Biesen quartzite* were taken from the lithological collection at the Geological Survey of Belgium (GSB), whereas the other quartzite samples were collected in the field. The *black Tienen quartzite* and *Wommersom quartzite* samples only occur as isolated, loose blocks in plowed fields. The *quartzite of Bray* occurs as large indurated blocks within the Erquelines sands, from which our samples are taken, or as loose blocks at the base of Quaternary deposits. The unique sample of the *grey Wommersom quartzite* enclosing a typical *Wommersom quartzite* nodule was taken from the archaeological raw rock collection of the Anthropology and Prehistory section at the Royal Belgian Institute of Natural Sciences (RBINS). A slice of each quartzite sample was used to make thin sections with a standard thickness of 30  $\mu\text{m}$ . A systematic research was performed on these thin sections using optical polarizing microscopy, optical CCL microscopy, energy-dispersive x-ray spectroscopy (EDS) and back-scatter electron imaging (BSE). The latter is also applicable to raw rock samples.

Optical polarizing microscopy was used to identify the mineralogy of the constituent grains and cement types, the micro-texture and micro-structures of the samples. This was executed both at the GSB with a Nikon Optiphot-Pol microscope and at the KULeuven with a Leica DMLP-microscope. In the studied quartzites, an outgrowth of secondary, syntaxial quartz overlying the original detrital quartz grains is often encountered. In most of these quartzites, a rim of impurities or dust rim, which delineates the original grain's morphology, is missing. Therefore, the estimation of the mean grain size in optical microscopy is based on the grain together with its secondary quartz overgrowths. Another difficulty due to the absence of a dust rim is deducing the degree

of grain compaction. Therefore, compaction is discussed using CCL.

Optical CCL was not only used to observe the morphology of the original quartz grains and the cement phases, but also to reveal accessory luminescent minerals such as zircon, apatite and kyanite. The minerals rutile and tourmaline are known to show no luminescence and could therefore not be observed in the CCL study. The CCL emission colour of quartz is known to change with electron irradiation time (Marshall 1988, Richter *et al* 2003). Usually, the blue component decreases while the red component increases with bombardment time. This property was used to highlight the cement, especially when it is composed of chalcedony. Optical cathodoluminescence microscopy was executed partly at the University of Mons with a CITL Mk5 CL unit fitted on a Zeiss Universal-R microscope (15 KeV–500  $\mu\text{A}$ ). Attached to this microscope was an optical spectrometer used for mineral identification, which allows one to record emission spectra from 380 to 1100 nm. Part of the study with CCL was performed at the KULeuven with a Technosyn model 8200 Mk2 CL unit fitted on a Nikon microscope (8 KeV–500  $\mu\text{A}$ ).

SEM in combination with EDS was used to determine accessory minerals by point analysis which was automated to map an area of a sample. Mapping was useful in gaining an insight into the spatial distribution of accessory minerals. In this research, it was found that hypersiliceous rocks required a high resolution to detect accessory minerals. However, this high resolution limited the area that could be mapped in order to keep measuring times feasible. Using the images obtained, a software tool calculated the area fraction of specific trace elements e.g. titanium and zirconium. This area fraction is obtained by pixel counting and is an approximate measure for the concentration of the trace element within a thin section sample. SEM in combination with BSE was used to study the morphology of the grains and the cement structures. The study was executed at the Mineralogy lab at the RBINS with a Philips ESEM XL30 scanning-electron microscope outfitted with a SEI detector and a combined BSE-EDS detector with a polymer window. The voltage used was between 23 and 25 kV in low vacuum mode, which did not require the application of a conductive coating on the sample.

## 7. Macro-petrographical description and terminology

The most common quartzite type within the Tienen Formation in the Hesbaye region is the *Tienen quartzite* (figure 1(D)). When geologists mention the *Tienen quartzite*, a light-grey coloured, massive, coarse-grained rock with a scaly fracture is meant (De Geyter 1981, Dreesen *et al* 2003). Often with sedimentary quartzites, a scaly fracture is the consequence of a specific micro-texture of well-interlocking detrital quartz grains by the outgrowth of secondary, syntaxial quartz (Cayeux 1906), as is the case for the *Tienen quartzite*. The *Tienen quartzite* often contains fossilized roots (De Geyter 1981). Archaeologists use the term ‘*grès-quartzite de Rommersom*’ or *quartzite of Rommersom* to indicate the *Tienen quartzite* as defined by geologists (Di Modica 2011), and is named

after its most productive outcrop area for the manufacture of cobblestones in the 19th century (Gulinck and Tavernier 1947). Compared to the other quartzites of the Tienen Formation in the Hesbaye region, it is the only *in situ* quartzite type described at outcrops (Rutot 1910). Its occurrence is not limited to one or several outcrop localities, but is, however, widespread.

The stratigraphically equivalent *quartzite of Bray* (synonymous with *quartzite of Binche*), outcropping near the community of Binche, is macroscopically identical to the *Tienen quartzite*, but is often less well cemented, exhibits a more whitish colour and contains fewer fossilized roots (Nijs and De Geyter 1984) (figure 1(E)). Archaeologists, however, use the term ‘Tienen quartzite’ to indicate a specific dark type of quartzite that is known to occur at just one locality, on a field in the commune of Hoegaarden, 7 km south of Tienen (E Robinson, personal communication). It is a fine-grained quartzite, still exhibiting a certain rugosity, with a black colour which alternates discretely with lighter patches. The fracture is conchoidal and non-lustrous, but sparkles slightly. Fossilized rootlets are common in these samples. In this paper we refer to this quartzite type as the *black Tienen quartzite* (figure 1(C)). It has never been described in the geological literature, unless it coincides with a single description by Rutot in 1899. He observed in one of the many quarries in Overlaar, together with fossil bones and rolled flint, some voluminous blocks of black quartzite lying in between white and grey, lobed quartzite blocks. The latter presumably corresponds to the *Tienen quartzite*. Some splintery fragments of the blocks showed characteristics of knapping by man (Rutot 1899). According to Rutot (1899), the black quartzite resembled the *Wommersom quartzite* of Steenberg, but differed from it by its coarser granulometry. The colour and granulometry description of Rutot suggests that this is the archaeologically defined ‘Tienen quartzite’. In this research, it is also assumed that our *black Tienen quartzite* samples are from the same extraction source as the samples named ‘Hoegaarden quartzite’ in the simultaneous and cooperative study of Blomme (2011).

The *Wommersom quartzite* is another much finer-grained quartzite type within the Tienen Formation (figure 1(B)). The *Wommersom quartzite*’s outcrop is limited to the area of a narrow hill, the Steenberg, in the village of Wommersom, 6 km northeast of Tienen. This quartzite type was named by archaeologists, and later studied by geologists (De Loë and Raeymaekers 1902, De Geyter 1981, Bassleer, 1985, Blomme 2011). The *Wommersom quartzite* is also known by different names in archaeology, being ‘grès-quartzite de Wommersom’ and ‘silex de Wommersom’ (Di Modica 2011). In geology, different authors (De Geyter 1981, Bassleer 1985, Vandenberghe and Gullentops 2001, Dreesen *et al* 2003) assign the term microquartzite to the *Wommersom quartzite* following the classification system of Michot (1958). The *Wommersom quartzite* has a brownish dark-grey colour and is as smooth as flint, exhibiting an aphanitic macro-texture. The fracture is typically conchoidal with a shiny to waxy luster, making it easy to confuse with flint. Rutot (1902) described the appearance of the *Wommersom quartzite* as nodules in a light-grey, lobed quartzite, generally interpreted as the ‘Tienen quartzite’ by geologists from that moment on. The enveloping material

around the *Wommersom quartzite* nodules was labelled as ‘Tienen quartzite’ simply due to its scaly fracture and lighter colour, and therefore macroscopically easy to misinterpret as the geologically defined *Tienen quartzite*. In general, a scaly fracture is often, if not always, interpreted as the result of a microscopically quartzitic texture of well-interlocking quartz grains visible with classical microscopy. This assumption is based on studies by Cayeux (1906). No microscopic study was done on these samples in which a nodule of *Wommersom quartzite* appears within an apparent ‘Tienen quartzite’. As later described, our research contradicts the assignment of the term ‘Tienen quartzite’ to the rock in which the *Wommersom* nodules appear as described by Rutot (1902). This is a first indication of the misleading determination on the macroscopic scale to identify a stone piece. We refer to this grey rock type, which encases the *Wommersom quartzite* nodule, as the *grey Wommersom quartzite* (figure 1(B)).

A macroscopic identical quartzite to the *Tienen quartzite* occurs in the Limburg province, but with a younger geological age. It concerns the *Alden-Biesen quartzite* located at the top of the Neerrepen Member in the St-Huibrechts-Hern Formation (figure 1(A)). Similar to the *Tienen quartzite*, the *Alden-Biesen quartzite* exhibits a lobed surface and is composed of quartz grains cemented by secondary quartz overgrowths. The *quartzite of Alden-Biesen* is only described twice (Gulinck 1968, Dreesen and Duser 2008) in the surroundings of the Castle of Alden-Biesen in the community of Rijckhoven (Limburg Province, Hesbaye region). Due to its low outcrop frequency, it has not been described by archaeologists.

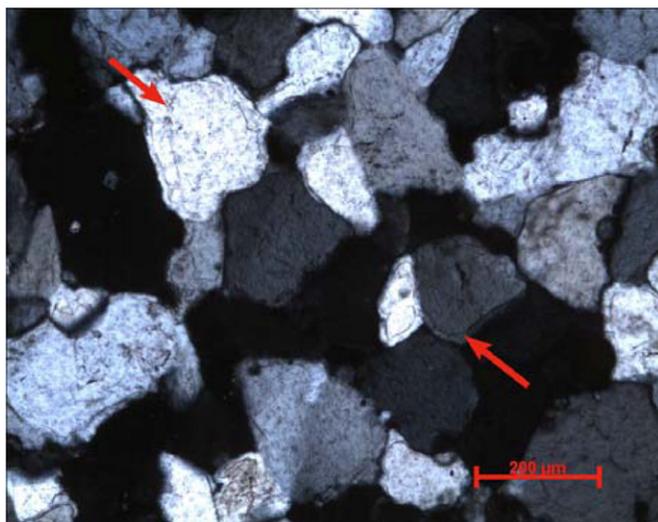
A common macroscopic feature of all the previously described sedimentary quartzites of both the Tienen and St-Huibrechts-Hern Formations is their lobed surface, also described as ‘surface mamelonnée’ in French (Gulinck 1968, Dreesen and Duser 2008).

## 8. Microscopical description and comparison

### 8.1. Optical polarizing microscopy

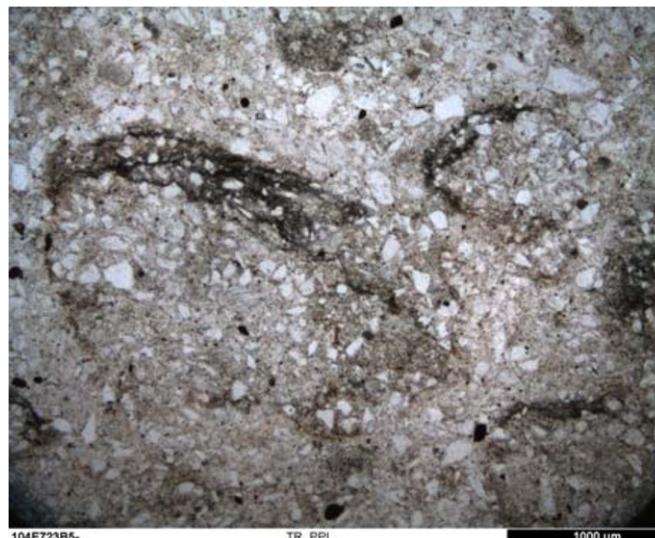
All six presented quartzites consist almost entirely of the mineral quartz, exhibiting a mosaic texture of interlocking quartz grains. This texture is commonly referred to as quartzitic texture. The grains are mainly monocrystalline and are surrounded by the outgrowth of secondary, syntaxial quartz. Exceptions to this quartzitic texture are the *Wommersom quartzite* and *grey Wommersom quartzite* with rather distinct textural features and the *black Tienen quartzite* showing textural characteristics intermediate between the *Tienen quartzite* and *Wommersom quartzite*. Feldspars or other mineral grains were not observed, resulting in mineralogically very mature rocks. Common accessory minerals in all samples are Ti oxides (rutile, anatase, brookite and ilmenite) and zircons, and to a lesser extent Al-rich silicate minerals such as tourmaline, kyanite and staurolite.

The *Tienen quartzite* and *Alden-Biesen quartzite* are microscopically identical. Both have a grain size ranging from about 100 to 250  $\mu\text{m}$ . Both miss a distinct dust rim and show frequently smaller sub- to euhedral quartz grains (20–60  $\mu\text{m}$ )



**Figure 2.** A mosaic of well-interlocking, monocrystalline quartz grains cemented by syntaxial, secondary quartz overgrowths. A distinct dust rim (arrows) surrounds each detrital quartz grain and delineates its original rounded to well-rounded grain form. Optical polarizing microscope—crossed polars—*quartzite of Bray*—scale 200  $\mu\text{m}$ .

at contacts between larger quartz grains. The main differences of the *quartzite of Bray* compared to the *Tienen* and *Alden-Biesen quartzites* are as follows: firstly, the slightly larger average grain size ( $> 200 \mu\text{m}$ ), secondly the rare occurrence to even absence of heavy accessory minerals, thirdly the absence of smaller quartz grains in-between the larger quartz grains, and lastly the observation of a very distinct dust rim showing the original grain morphologies of sub-rounded to well-rounded grain edges (figure 2). On some grains of the *quartzite of Bray*, a very thin opal coating can be recognized and other grains can show a second dust rim. The *Wommersom quartzite* can easily be distinguished from the previous quartzites by its unique texture. It consists of quartz grains in the range of about 30–150  $\mu\text{m}$  floating within a granular micro-quartzitic matrix ( $< 20 \mu\text{m}$ ). Some of the quartz grains exhibit fretted boundaries which locally show embayments. The grains commonly lack secondary, syntaxial quartz overgrowths, but when present, they are irregular and discontinuous. Opaque, clay-sized material is common and organized in wavy, discontinuous patterns, sometimes even concentrically embracing otherwise invisible nodular structures, resembling glaebules. The *grey Wommersom quartzite*, embracing nodules of the *Wommersom quartzite*, is texturally and mineralogically identical to the nodule and also contains wavy, discontinuous laminae of opaque, clay-sized material. The only difference is the existence of very small intergranular porosity compared to the nodule. The *black Tienen quartzite* can be described as an intermediate form between the *Tienen quartzite* and *Wommersom quartzite*. Its fabric varies between a texture of floating quartz grains (50–200  $\mu\text{m}$ ) in a micro-quartzitic matrix, mostly lacking secondary, syntaxial quartz overgrowths, and a mosaic texture of well-interlocking quartz grains. Locally, the matrix is replaced by chalcedonic cement. As in the *grey Wommersom* and *Wommersom quartzites*, nodular structures (around 1–2 mm) also occur within the



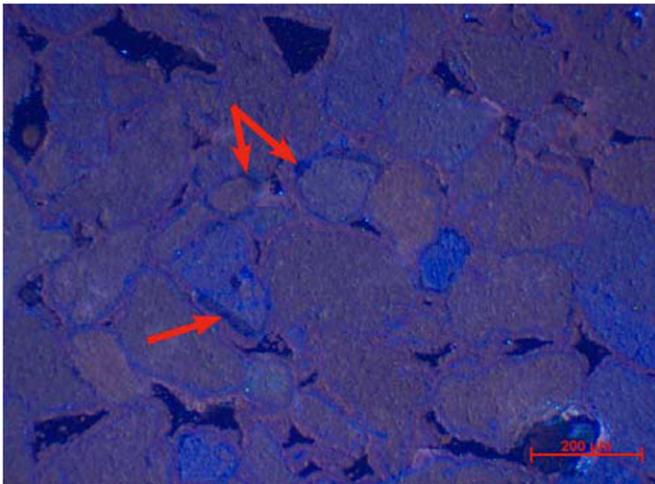
**Figure 3.** Nodular structures resembling glaebules recognized on the hand of a finely stratified opaque, clay-sized coating. The figure shows a micro-texture of floating quartz grains within a much finer-grained micro-quartzitic matrix. Optical polarizing microscope—plane polarized light—*black Tienen quartzite*—scale 1000  $\mu\text{m}$ .

*black Tienen quartzite* and are surrounded by a finely stratified coating of opaque, clay-sized material (figure 3). A test with Raman spectroscopy of this opaque, clay-sized material revealed that it consists of a mixture of organic material with Ti oxides, predominantly anatase. Cap-like structures, glaebules and wavy concentrations or discrete laminae of this clay-sized material are common in clay-rich sands according to Webb and Golding (1998) and can refer to pedogenetic silcretes (Thiry 1999, Nash and Ullyott 2007). Intergranular porosity is absent within the *black Tienen quartzite* and *Wommersom quartzite* but appears slightly within the *grey Wommersom quartzite*. A relatively higher, but still low degree of intergranular porosity is observed within the *Tienen*, *Alden-Biesen* and *Bray quartzites* and increases progressively towards the edges of the stone samples. This can be the result of recent weathering or moving silicification fronts during the formation of the quartzite.

## 8.2. Optical CCL microscopy

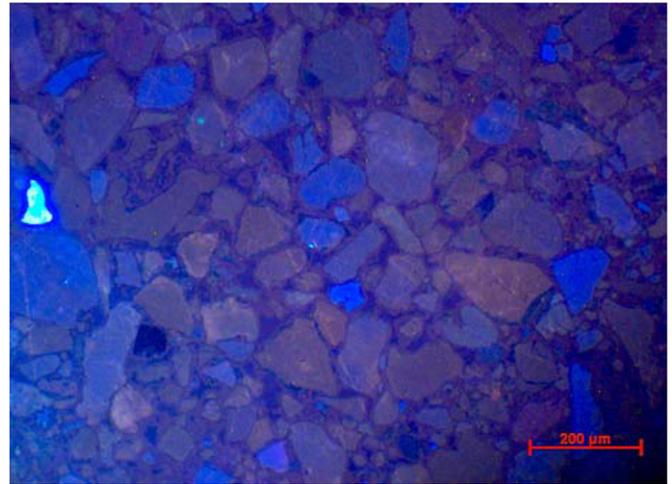
In general, the quartz grains of the six quartzite types exhibit a blue to violet or brownish luminescence with low to moderate intensity. The CL colour of the quartz also gradually changes (often becomes more reddish) over electron irradiation time, with a variable magnitude from grain to grain. No significant difference was noted between the samples, suggesting no important variations in detrital sources (Richter *et al* 2003). In some quartzites, the cement is clearly multi-staged and may relate to changes in pore fluid composition as the silica cement was deposited. This cement succession is sometimes preceded by a non-luminescent, discontinuous cement rim of varying thickness, and is interpreted as a relict of the rock it is derived from.

The *quartzites of Alden-Biesen* and *Tienen* contain a higher amount of accessory minerals, mainly zircon and in



**Figure 4.** A multi-staged cement, grown onto rounded to well-rounded quartz grains, starts with a very thin blue luminescent coating and is succeeded by a thin dull red to weakly luminescent rim. A dull-brownish luminescent rim fills up the remaining intergranular space. Grains in the centre show an irregular, discontinuous non-luminescent cement phase (arrows), presumably a relict of the rock the detrital grain is derived from. Note the intergranular porosity (black), indicative of the less well-cemented phase of the *quartzite of Bray*, near the edges of the sample. Optical cold cathodoluminescence—*quartzite of Bray*—scale 200  $\mu\text{m}$ .

a lesser extent kyanite together with a few small apatite grains and kaolinite flakes, compared to the *quartzite of Bray*. The *Wommersom quartzite*, *grey Wommersom quartzite* and *black Tienen quartzite* also contain zircons, but generally of smaller size. Within the *Tienen quartzite* and *Alden-Biesen quartzite*, the original grain morphology differs from elongated to spherical forms with varying angular, sub-rounded to rounded edges. In contrast, the grains of the *quartzite of Bray* are in general sub- to well rounded. The continuous secondary, syntaxial quartz overgrowths within the *Tienen quartzite* and *Alden-Biesen quartzite* show a weak brownish luminescence and fill up the pre-existing intergranular pore space. The quartz overgrowths in the *quartzite of Bray* consist of a thin dull-red to weakly luminescent rim succeeded by a dull-brownish luminescent rim with a seemingly fibrous character. Locally, in the *quartzite of Bray*, a very thin, blue luminescent coating ( $<10 \mu\text{m}$ ) is recognized and interpreted as the thin opal coating observed in optical microscopy (figure 4). The grains are moderately to well compacted, often resulting in the occurrence of pressure solution along grain contacts. The compaction degree within the *Tienen quartzite* and *Alden-Biesen quartzite* does not differ from the *quartzite of Bray* and grain contacts range from tangential and slightly concave–convex to long straight contact boundaries. In CCL, the *Wommersom quartzite* displays a pattern of very small dull-brownish luminescent grains ( $<20 \mu\text{m}$ ) cemented by a non-luminescent cement, and make up the micro-quartzitic matrix as observed in optical polarizing microscopy. In this matrix, larger quartz grains float, rarely showing secondary quartz overgrowths. When present, they consist of an irregular, discontinuous non-luminescent cement. The opaque, clay-sized patches exhibit a slightly more

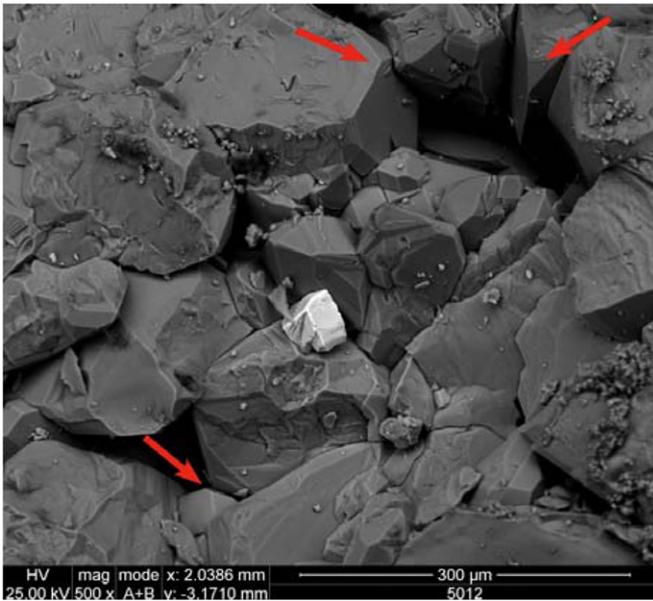


**Figure 5.** A dull-red luminescent cement rim surrounds and interconnects all quartz grains. The remaining pore space is filled by a non-luminescent cement. Grains are often angular and can have very irregular boundaries with embayments. Note the bright, blue luminescent zircon on the left. Optical cold cathodoluminescence—*black Tienen quartzite*—scale 200  $\mu\text{m}$ .

intense dull-yellow luminescence. The cement's luminescent character of the micro-quartzitic matrix is different within the *grey Wommersom quartzite* and shows a bright orange luminescence in contrast to the non-luminescent cement of the *Wommersom quartzite*. The *black Tienen quartzite* is characterized by a dull-red luminescent rim (5  $\mu\text{m}$  thick) surrounding and interconnecting all grains (figure 5). The remaining pore space is filled by a non-luminescent cement. The compaction degree is low and tangential contacts occur with rare pressure solution at grain contacts. As well as in the *black Tienen quartzite* as in the *grey Wommersom quartzite* and *Wommersom quartzite*, the grains are generally not rounded and have rather irregular forms showing fretted boundaries locally with embayments.

### 8.3. Scanning-electron microscopy–energy-dispersive x-ray spectrometry (SEM-EDS)

The six quartzite types consist predominantly of quartz with a minor amount of accessory minerals, frequently zircon and Ti oxides, and in a lesser extent Al-rich silicates interpreted as tourmaline, kyanite, staurolite, kaolinite and monazite. Within the *Tienen* and *Alden-Biesen quartzites*, Ti oxides are more abundant than zircon grains. Some rare grains of kyanite or andalusite as well as tourmaline are observed. In contrast, the *quartzite of Bray* contains relatively much less the amount of accessory minerals. The *black Tienen quartzite* shows mineralogically the same features as the *Tienen quartzite* with the exception of the rare presence of barium sulfate crystals. These mineralogical properties of the *black Tienen quartzite* are both supported and contradicted by results of ICP-OES analysis performed on samples of the *Tienen quartzite*, *black Tienen quartzite* and *Wommersom quartzite* (Blomme 2011). This analysis demonstrates the higher amount of the element Ba within the *black Tienen quartzite* compared to the *Tienen quartzite* and *Wommersom quartzite*, but also

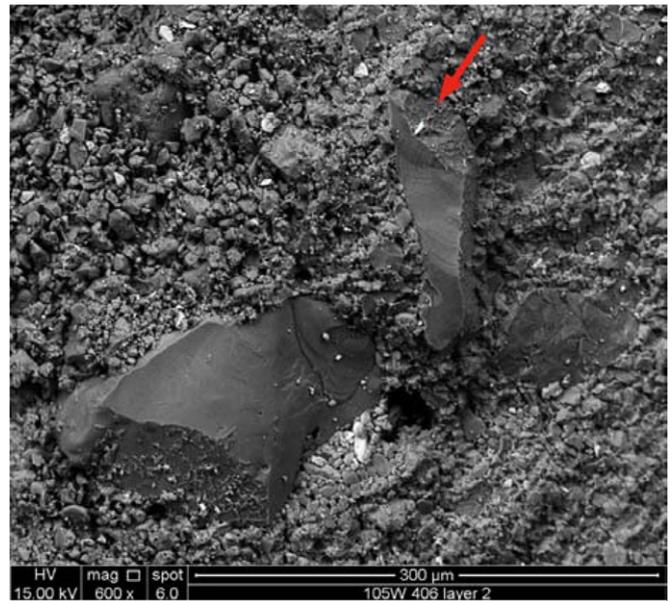


**Figure 6.** Angular quartz grains as the result of secondary quartz overgrowths which give the detrital quartz grains an euhedral crystal shape (arrows). Note the well-formed zircon mineral in the centre. SEM-BSE image—*black Tienen quartzite*—scale 300  $\mu\text{m}$ .

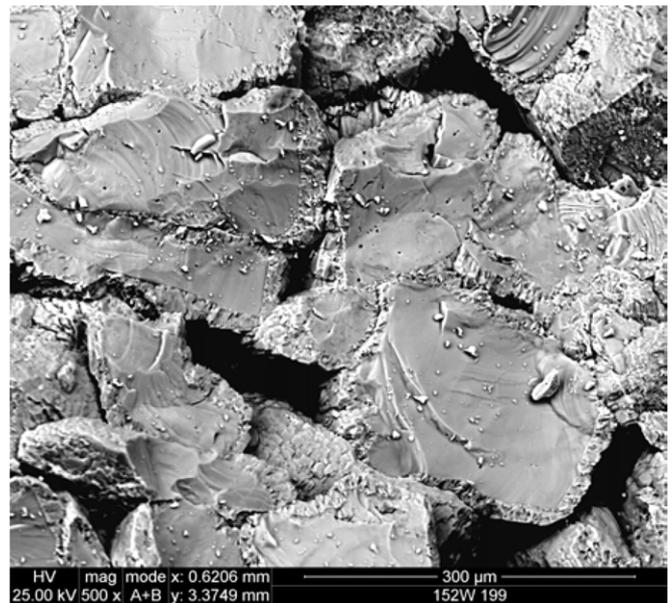
shows an increased concentration of Zr compared to the *Tienen quartzite* and *Wommersom quartzite*, which was not noted in our study with SEM-EDS. If the *black Tienen quartzite* can be considered as a pedogenetic silcrete, then the barium sulfate crystals may be the result of an evaporation process in relation to the formation of this sedimentary quartzite (Thiry 1999). The *Wommersom quartzite* and *grey Wommersom quartzite* are easily differentiated from all other quartzites by their high enrichment in Ti oxides (figure 9). These Ti oxides are locally concentrated in patches constituting wavy patterns or surrounding larger, detrital quartz grains and coincide with the dark, clay-sized patches in optical microscopy (figure 3). Similar fine-grained Ti oxides of authigenic origin are common in pedogenetic silcretes (Thiry 1999). Al-rich accessory minerals also occur regularly within the *grey Wommersom quartzite* and *Wommersom quartzite* and are mostly interpreted as tourmaline. Rarely, a monazite mineral is observed within the *Wommersom quartzite* types. The remarkably higher amount of the element Ti in the *Wommersom quartzite* is also supported by the ICP-OES analysis (Blomme 2011).

#### 8.4. Scanning-electron microscopy—back-scatter electron imaging (SEM-BSE)

The *quartzites of Alden-Biesen* and *Tienen* display a rough fracture surface and are constituted of well-interlocking quartz grains exhibiting euhedral crystal shapes resulting from the outgrowth of secondary, syntaxial quartz (figure 6). Locally within the inter-granular micro-space, smaller sub-to euhedral quartz grains occur. In contrast, the quartz grains constituting the *quartzite of Bray* are rimmed by a thick fringe of micro-quartz consisting of elongated and sharp pointed crystals grown perpendicular to the original grains' edges (figure 8). Locally, a very thin cryptocrystalline coating of

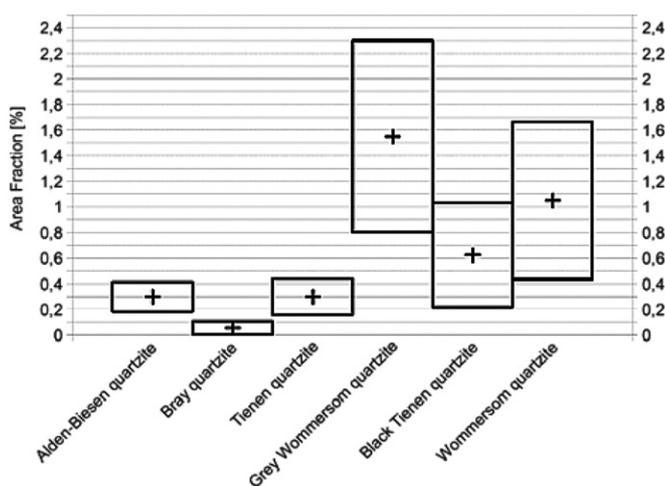


**Figure 7.** Angular quartz grains embedded in a very fine-grained, granular matrix. This matrix consists of detrital quartz grains with an angular appearance which is considered the result of the outgrowth of secondary quartz. Note the 'casts' on the large quartz grains of smaller 'matrix' grains which have fallen out due to the fracturing of the sample (arrow). SEM-BSE image—*grey Wommersom quartzite*—scale 300  $\mu\text{m}$ .



**Figure 8.** Rounded to well-rounded quartz grains are rimmed by a thick fringe of micro-quartz consisting of elongated and sharp pointed crystals grown perpendicular to the original grains' edges. SEM-BSE image—*quartzite of Bray*—scale 300  $\mu\text{m}$ .

a few microns is observed on the grains of the *quartzite of Bray*. The *Wommersom quartzite* displays a rather smooth fracture surface consisting of recognizable angular quartz grains floating within a much finer grained, granular matrix of very small detrital quartz grains (< 20  $\mu\text{m}$ ) cemented by a cryptocrystalline cement. In contrast, the *grey Wommersom quartzite* lacks this cryptocrystalline cement and the quartz granules appear angular by the outgrowth of secondary,



**Figure 9.** The average area fractions of EDS mappings of the element titanium of the six sedimentary quartzites discussed in this paper. The titanium area fraction of the *grey Wommersom quartzite* and *Wommersom quartzite* is considerably higher than the other four sedimentary quartzites. The *quartzite of Bray* has the lowest area fraction of all six sedimentary quartzites for the element Ti. Legend: + average area fraction, bar delineates one standard deviation above/below average.

syntaxial quartz resulting in a certain inter-granular porosity which is absent in the *Wommersom quartzite* (figure 7). The *black Tienen quartzite* exhibits the same characteristics as the *Wommersom quartzite*, but with a larger mean grain size of the floating quartz grains and locally with a concentration of grains in tangential contact. Noteworthy is the rare presence of fanning barium sulfate crystals between quartz grains, as individual massive grains or as inclusions in accessory minerals, such as zircon within the *black Tienen quartzite* as previously observed with SEM-EDS.

### 9. Discussion and conclusion

The Tienen Formation contains quartzite types which could be distinguished with optical polarizing microscopy, optical cold cathodoluminescence microscopy and scanning-electron microscopy equipped with EDS and BSE detectors (figure 10).

Using optical polarizing microscopy, the *Tienen quartzite* and *quartzite of Bray* can be distinguished on the basis of quartz cementation type, the relative amount of accessory minerals and the difference in grain size. Optical cathodoluminescence microscopy is useful in displaying significant differences in the grains' roundness and luminescence properties of the cement phases between the *quartzite of Bray* and *Tienen quartzite*. The scanning-electron microscope using back-scatter electron images of the fracture surface confirms this distinction of the cement types by displaying the crystal structure of the quartz cementation on the original grains of the *quartzite of Bray* which is distinct from the syntaxial quartz overgrowths of the *Tienen quartzite*. The cementation types of the *Tienen quartzite* and *Bray quartzite* allow for a good distinguishability between both quartzites and is best observed with CCL and SEM-BSE. The *Wommersom quartzite* and the *grey Wommersom quartzite* can be distinguished macroscopically on the basis of colour and fracture behaviour, but on the microscopic scale they unexpectedly show an identical texture and mineralogy. The difference in colour and fracture behaviour between these two quartzites is presumably influenced by the degree of cementation, and by defects and impurities within the cement. The latter is concluded from the different luminescence properties of the cements of both quartzites in CCL. Macroscopically, the *Tienen quartzite* and *grey Wommersom quartzite* are similar by their colour and scaly fracture pattern. At first sight, they can easily be mistaken for one and other, but optical microscopy or SEM-BSE makes it possible to immediately distinguish them on the basis of their different micro-textures. The *black Tienen quartzite* and the *Wommersom quartzite* can be differentiated macroscopically by their distinct colour and surface rugosity. An additional study with CCL reveals their different luminescence properties. These luminescence properties can be used as a good microscopic criterion for distinguishability between both quartzites.

The distinguishability between the *Tienen quartzite* and the *Alden-Biesen quartzite* is very low to non-existent using optical polarizing microscopy, CCL, SEM-EDS and SEM-BSE. These quartzites are macroscopically identical, and exhibit the same mineralogy and microscopic quartzitic

Sample	Method	Bray quartzite ↔ Tienen quartzite ↔ Grey Wommersom quartzite	Black Tienen quartzite ↔ Wommersom quartzite
Thin section	Optical polarizing microscopy	grain size dust rim	
	CCL	cement phases grains' roundness	
	SEM-EDS		Ba-concentration Ti-concentration
Fracture surface	SEM-BSE	cement morphology	
			micro-texture

**Figure 10.** The main criteria to easily distinguish similarly looking sedimentary quartzites are summarized. These criteria are placed in the row of the measurement technique required to determine the specific criteria. The measurement techniques themselves are grouped into two groups depending on whether they require a fracture surface or a standard thin section. The criteria with a grey background are to the authors' opinion the strongest criteria to differentiate between the corresponding sedimentary quartzites. Other criteria using different methods should also be taken into account to ensure a correct determination. These criteria are indicated with a white background.

texture. A suggested method for future research is  $^{18}\text{O}$ -isotope-analysis performed on silica cements of different quartzite types, which can reveal differences in fluid conditions at the time of silica precipitation (Kelly *et al* 2007).

For a potential increased accuracy in detecting trace elements, the application of laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) could prove to be useful on all these quartzites (Pitblado *et al* 2008). Moreover, the use of a mass spectrometer would make it possible to measure isotope ratios of specific elements (Tykot and Young 1995).

An additional interesting result of this research is that evidence was found to question the general assumption, which was based on the observations by Rutot (1902), that the *Wommersom quartzite* occurs as nodules within the *Tienen quartzite*. Studying the so-called 'Tienen quartzite' in which the nodules occur, revealed the misinterpretation of this *grey Wommersom* as *Tienen quartzite*. More samples should be studied to confirm this claim.

Taking into account the aforementioned distinguishabilities of hypersiliceous rocks and the demand for a proper reference collection (Di Modica 2011), this research should be continued, using even more research techniques. The research presented in this paper is part of wider, ongoing research on hypersiliceous rocks. At the time of writing, a start is made on a reference collection with rock samples, thin sections and associated descriptive sheets, ready to be both expanded in scope with more rock types, and to be extended with complementary techniques.

## Acknowledgments

Thin sections were made at the University of Liège and the University of Mons. Herman Goethals and Tom Thys are thanked for assisting in the use of the SEM at the Mineralogy Lab of the RBINS. Tom Thys is also thanked for the Raman-spectroscopy test. We thank Professor Dr Rudy Swennen for the use of the optical polarizing microscope and optical cold cathodoluminescence microscope at the KULeuven. Erick Robinson is thanked for guiding us to the site of the *black Tienen quartzite*. We thank Eric Goemaere for providing the sample of the *Alden-Biesen quartzite*.

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