Novel use of cathodoluminescence to identify differences in source rocks for Late Paleoindian quartzite tools

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Abstract

Heterogeneous quartzite artefact finds from North America's Late Paleoindian period occur in several areas throughout the Northern Great Lakes region. Here standard petrographic analysis, back-scatter scanning electron microscopy (BSEM), and cathodoluminescence (CL) have been used to identify the properties of a regionally abundant, high-quality orthoquartzite stone as compared to high-quality Hixton silicified sandstone from the Silver Mound Archeological District (SMAD) in Jackson County Wisconsin. Results demonstrate the potential for reducing misidentification among material sources and also exhibit the acutely discerning tendencies of pre-contact peoples. Lithological interpretations of thin-sections identify the different properties of the Hixton material. Conversely, Mesnard quartzite, while it functions adequately as tool stone,

is fundamentally compromised by a tightly packed microstructure. This tightly packed microstructure produces a hard, less tractable material with erratic breakage, possibly explaining Mesnard quartzite's limited distribution prehistorically.

Introduction

Cryptocrystalline Silicate (CCS) endured as a primary tool stone throughout America's pre-contact period. Artefacts constructed from CCS materials offer us information about the culture, technology, trade, and economies of these early societies. Orthoquartzite CCS material from outcrops in the Marquette Mineral District (MMD) of the Lake Superior Iron Range, provided a substantial raw tool stone source during the Late Paleoindian / Early Archaic period. In the current research, we have evaluated the use of several petrographic techniques to examine Mesnard quartzite and identify the microstructural properties of this material and its suitability for flintknapping. For comparison, we ran the same analysis (standard petrography, back-scatter SEM, and cathodoluminescence) on all samples and, evaluated the properties of high-grade lithic material found elsewhere in the broader Midwest region.

Throughout North America's pre-contact period, people were keenly aware of CCS resources, and a careful selection of stone was essential. Selectively, high-grade tool stones that break predictably to form sharp edges were deemed suitable for flintknapping. When local tool stone was available, it was knapped and used locally within the area. In regions with limited stone resources, small cobbles were reduced using bipolar core reduction methods. Alternatively, people would likely have transported the necessary CCS resources or would have possibly traded for materials via intricate trade and exchange networks. Regional quarry locations were exploited as primary sources of the raw lithic material. Since tool stone underpinned everyday activities, known quarry locations were regularly worked, protected, and claimed within territories (Goodyear, 1979).

Within a cultural resource context, quarry locations are critical as they provide discernable focal points for past human activities. Often research data gleaned from quarries provides evidence of technology, artefacts, lithic scatters, and a range of prehistoric events that would have occurred within the immediate vicinity. The further refining study of quarried materials, spoil-hills of discarded materials, along with potential habitation encampments, yields rich cultural information about past peoples within these areas. Also, since quarries occur at fixed locations, they may have operated as regular stops within a seasonal round along known travel corridors between regions. Detailed information illuminating characteristic properties of CCS materials provides the foundational means to identify quarry sources and origins of dispersed lithic materials. With evidence of people visiting the MMD during the Late Paleoindian/Early Archaic, along with vast resources of CCS materials in this area, to what extent people from the broader region utilized this area as a source for their lithic materials remains unknown (Buckmaster and Paquette, 1988; Carr, 2008; Legg et al., 2017). In line with existing research that applies petrographical analysis to distinguish lithic raw materials (e.g. Dalpra and Pitblado, 2016; Moreau et al., 2016; Pitblado et al., 2008), here the current research aims to identify the characteristic properties of quartzite sourced from the Mesnard quarry (Figure 1) delineating the internal characteristics and signature properties of this tool stone. Furthermore, the research aims to evaluate how the composition and microstructure of Mesnard quartzite compares to high-quality tool stone from a neighbouring area within the broader Midwest. In addition, the work aims to build on previously applied techniques for the region (Julig et al., 1998) using improved imaging techniques. Ultimately the work identifies a reliable methodology for the proper identification of quartzite stone sources used in the manufacture of quartzite stone tools found on archaeological sites seeding ongoing studies about trade, exchange, and potential transportation of raw materials in a broader regional context.

The research builds on information gathered from material discovered at archaeological sites within the MMD, further expanding on a predictive archaeological model that identifies patterns associated with Late Paleoindian and Early Archaic settlement (Legg et al., 2017). In short, the research aims to perform the following: determine the mineral content and textural relations within Mt. Mesnard quartzite; characterize signature qualities of stone in both the MMD and Silver Mound Archaeological District (SMAD) and establish a systematic and repeatable methodology for evaluating the source of lithic assemblages.

Study areas and sample material

Here we examine stone from a lithic quarry and workshop at Mt. Mesnard. Mesnard quartzite exists in several places across the MMD. The MMD was the first Lake Superior Iron Range district to be discovered. Since the mid-19th century, the area has supported the continuous extraction of iron ore. The Entire Lake Superior Iron Range is rich in minerals and lies within the southern part of the Canadian Shield or Laurentian Plateau. Evidence of resource extraction underpins the placenames, industries, and societies throughout the region. The MMD features several variations of quartzite among metasediment outcrops; here, we focus on Mesnard quartzite. The particular stone can be found in several places throughout the district, with the primary formation delineated by a narrow outcrop spanning a distance of some 18 km between Teal Lake and the Mouth of the Carp River (Figure 1). Quarries from the extraction activities of precontact peoples are located at both ends of this outcrop, evidenced by the thousands of core reduction and bifacial thinning flakes; although broken bifaces recovered at these sites showing that fractured bifaces were only in production stage two (edged biface) of five, indicative of a quarry site (Andrefsky, 2011).

Another expanse of the material is found at a place called Pellisier Lake, and this formation arcs eastward some 8 km towards Lake Superior. Mesnard quartzite features mainly quartzite (metaquartzites) and is understood to originate from shallow-water marine deposition materials (Pettijohn et al., 1997).

Sample material of Mesnard quartzite was selected for further analysis due to evidence of the pre-contact quarrying activities at the base of the main outcrop. Further, recent excavations in the area by Demel led to the discovery of a diagnostic dart point within the vicinity of the quarry (Figure 2). This diagnostic chert point is similar to the Middle-to-Late Archaic period Brannon point morphology (Reber et al. 2017). Discovery of this diagnostic dart point among quartzite bifaces and flakes suggests that pre-contact peoples operated the quarry during the Middle to Late Archaic period (Reber et al., 2017). Physically, the Mesnard quartzite appears in variations of white to dark grey, and texturally as sugary quartz. The name derives from the location of the stone's primary source, which forms the bulk of the mass at Mt. Mesnard, located in the southeast corner of the district.

Mesnard quartzite is part of the Chocolay Group of the Marquette Range Supergroup (Cannon and Gair, 1970; Vallini et al., 2006; Van Schmus, 1976). The stone weathers to a greyish white and can feature varying shades of pink, light red, rusty red, or purple depending on amounts of iron ore present in its formation (Gair and Thaden, 1968; Pettijohn et al., 1997). Weathered quartzite can look very similar to Hixton silicified sandstone (SMAD, see below). In places, the quartzite interlayers with thin beds of grey slate, quartz-pebble conglomerate, and greywacke. The base of massive white-weathering vitreous Mesnard quartzite is located on top of the greywacke or sericite slate of the Enchantment Lake Formation of the Chocolay Group. It is either directly overlain by the Kona Dolomite of the Chocolay Group or by a slate, which then passes upwards into the Kona Dolomite (Vallini et al., 2006). The age of the material is thought to be middle Precambrian (early Animikie) (Gair and Thaden, 1968; Puffett, 1974). The Mesnard quartzite is considered a direct correlative of the middle Precambrian Sturgeon Quartzite of Iron and Dickinson Counties, Michigan. Due to the greater age range of the Animikie in northern Michigan, the Mesnard quartzite is at a lower horizon than the base of the type Animikie section found in the Thunder Bay area, Ontario, Canada (James, 1958). Throughout Michigan's Upper Peninsula, Precambrian quartzite outcrops are abundant.

Silver Mound Archaeological District (SMAD)

To provide a comparison of Mesnard quartzite with a source of high-quality lithic material, stone samples from the SMAD complex were selected for comparative analysis. Located in Jackson County, Wisconsin, the SMAD features a sandstone hill where pre-contact peoples extensively quarried Hixton orthoquartzite for the manufacture of stone tools (Carr and Boszhardt, 2014; Hill, 1994; Lang, 2004). The hill is located near the town of Hixton, northern Wisconsin, some 200 miles southwest of the MMD. Within the SMAD sandstone hill are layers of hard and brittle cemented silica or orthoquartzite. The material is formed from the glueing together of the sand grains with a fine quartz cement. The stone ranges in colour from orange, pinkish-red to a solid grey and nearly white. The silica-based rock, formed during the Upper Cambrian era (and therefore geologically younger than the Mesnard quartzite), is known by several names including Hixton Silicified Sandstone (HSS), "sugar quartz" or simply Hixton. The varying colours in the material are the result of varying degrees of hematite within the cement (Behm, 1984; Brown, 1984; DeRegnaucourt and Georgiady, 1988). Quartzite comprises the entire Silver Mound hill, but pre-contact peoples mined veins of quartzite of different qualities and colour.

Hixton lithic material was used extensively for stone tool production during the broader region's entire prehistoric record, beginning with the Early Paleoindians (Lang, 1987). Artefacts made from this tractable stone are found throughout Wisconsin and throughout the upper Midwest (Hill, 1994). In some instances, artefacts visually identified as Hixton have been found at distant locations, as far as 500 miles away (DeRegnaucourt and Georgiady, 1998). Indeed, discoveries of Late Paleoindian and Archaic spear or dart points in the MMD (e.g., the Gorto site) appear to be made with Hixton material (Buckmaster & Paquette, 1988). The similar visual appearance of the Mesnard and Hixton materials creates a high possibility for misidentification. Artefact discoveries made in the MMD were either made of HSS material or possibly material from ~~a~~ the more localized Mesnard source, but it is difficult to determine accurately through visual inspection.

Methods

This pilot study focused on a small number of samples (3) in order to establish whether various, relatively straight forward and accessible petrographic techniques could be used to characterise rock materials used to make stone tools and to identify possible sources. The analysis focused on petrographic work (University of Aberdeen, Scotland). Polished thin-sections of potential silicate tool stone materials were prepared and analyzed using standard petrographic, back-scatter scanning electron microscopy (BSEM), and cathodoluminescence (CL) techniques to determine their composition and texture. BSEM and CL were conducted in the Aberdeen Centre for Electron Microscopy, Analysis and Characterisation (ACEMAC) facility at the University of Aberdeen using a Carl Zeiss Gemini SEM 300 VP Field Emission instrument equipped with a Deben Centaurus CL detector, an Oxford Instruments NanoAnalysis Xmax80 Energy Dispersive Spectroscopy (EDS) detector and AZtec software suite. Prior to analysis, electron microscopy samples were sputter-coated with a thin coat of carbon under vacuum, to prevent electrical charging while the sample was in the electron microscope and prevent interference with the image. For both the BSEM and CL imaging, an accelerating voltage of 15 kV was used. A full explanation of the BSEM technique can be found in Krinsley (1998), but it is useful to note here that minerals containing elements with a high atomic number appear brighter in the image than elements with a low atomic number. Consequently, quartz (SiO_2) will appear much darker in a BSEM image than, e.g. calcite ($CaCO_3$).

When rocks are composed mainly of one mineral, however (e.g., quartz), it is necessary to use other techniques such as cathodoluminescence (e.g. Boggs and Krinsley, 2006), to observe subtle variations in the rock. In an SEM, the cathodoluminescence (CL) detector collects the light (or photons) emitted by the specimen when bombarded by electrons to produce an image of the luminescence (e.g., Bignall et al., 2004). The Deben Centaurus CL detector at the ACEMAC facility generates monochromatic images from light emitted in the wavelength range 185-850 nm (including ultraviolet but not infrared light). Spectral analysis, such as that conducted by Hunt, 2013 using a CL attached to a microprobe, is not possible using the ACEMAC facility.

The chemical and textural variations observed using CL, however, can be used to characterize individual samples and to look for differences between samples related to the formation of the rocks (e.g., Hunt, 2013). Although CL is a well-established technique for geological studies (e.g., Rice et al., 2016), it has only rarely been used in archaeological studies, usually for marbles (Corazza et al., 2001), but occasionally for quartzites (e.g., Julig et al., 1998) and ceramics (Chapoulie et al., 2005; Chapoulie and Floréal, 2004).

Results

Three samples (A-C) were studied, a sample of Upper Cambrian Hixton silicified sandstone (A), and two samples from the Marquette Mineral District (MMD B and C), samples A and B were source material whilst sample C was a quartzite artefact.

Standard Petrography (Figure 3)

Standard petrographic analysis shows that all three samples are composed mainly of quartz. The Hixton sandstone (Sample A) and MMD Sample B are composed of relatively large, rounded quartz grains (Figure 3A and 3B), while MMD Sample C is finer-grained, and grains are less rounded. The small sample size, however, limits characterizing inter outcrop variation.

Back-scattered electron imagery (BSEM, Figure 4)

The Hixton sandstone (Sample A) shows significant amounts of space (termed porosity, c.10%) between the individual quartz grains (Figure 4A). Very little porosity is observed in MMD Sample

B (Figure 4B) and is absent in the MMD Sample C (Figure 4C). EDX analysis shows that the very limited, white areas that occur between grains in both MMD Sample B (Figure 4B) and MMD Sample C (Figure 4C) contain aluminium (Figure 6A), which is likely to be residual aluminium oxide polishing powder.

Cathodoluminescence (CL, Figure 5)

Under CL, the Hixton sandstone (Sample A) shows well-rounded quartz grains (various shades of grey) surrounded by a black, non-luminescent quartz 'cement' rim (Figure 5A). Cement is a geological term for crystals grown after the original sediments were deposited. This cement (Figure 6A) consists of repeated layers of finely crystalline quartz (chalcedony), and EDS shows that is composed of pure silica (Figure 6B). Using CL, space between the grains (porosity) is shown in white rather than the black seen using BSEM. MMD Sample B (Figure 5B) is also composed of large quartz grains, although these are not as well rounded as those in the Hixton sandstone and also show more evidence of cracking.

The space between grains in the MMD Sample B has been filled by black, non-luminescent quartz cement. MMD Sample C (Figure 5C) is composed of quartz grains of various sizes. The original grains are also relatively rounded and fractured but, like MMD Sample B, have been cemented by black, non-luminescent quartz. The very bright spots in MMD Samples B and C are residual aluminium oxide polishing powder (Figure 6A).

Discussion

The results show that CL imaging can be an appropriate technique to distinguish between quartz samples (both artefacts and potential lithic sources). Using standard petrographic and BSEM imaging techniques, it proved difficult to find features that were characteristic of individual samples. Figure 5, however, shows that using a CL attached to a BSEM with EDS (termed 'hot' CL), the differences between the samples are clearly visible. Hunt (2013) took this technique further using a CL detector attached to a microprobe where spectra of the light emitted could be collected. This approach was done using a similar wavelength range (200-800 nm) as this study and enabled her to determine the origin of quartz used in ancient ceramics (e.g. metamorphic, volcanic, hydrothermal). Unfortunately, this facility is not currently available at ACEMAC, but this pilot study shows the possibilities of using relatively routine CL analysis to screen potential source lithic sources and artefacts. Other researchers have used 'cold' CL techniques (where the CL gun is attached to a standard petrographic microscope) to analyse quartz grains in ceramics successfully (Ammari et al., 2017) as well as the Paleoindian tool material (Julig et al., 1998).

The petrographic results gathered suggest that all three samples had a different origin and would, therefore, have different properties. The Hixton silicified sandstone had undergone minimal alteration from when the original sediments were deposited in the Upper Cambrian. The porosity means that it would be relatively lightweight, and its relatively uncemented nature would also mean that it would have been easy to work into various tools. Due to its brittle, homogeneous, isotropic nature, the Hixton material is the most suitable for flintknapping as it fractures predicably. Less homogeneous materials like MMD quartzite may display less predictable characteristics (Andrefsky, 2011).

CL observation has shown that although MMD Sample C is much finer-grained than MMD Sample B, they have similarities, e.g., fractured quartz grains suggesting that they may be from the much older Precambrian Mesnard quartzite. Because of the dense, cemented nature of MMD (Samples B and C), the material is less homogeneous, and microfractures are visible in many near-surface outcrops. The MMD quartzite is heavier, more difficult to work, and less predictable, making it less prized than the Hixton sandstone (Sample A). Demel's experience with MMD quartzite in experimental archaeology course activities confirms that the local Mesnard quartzite is difficult to knap. In fact, during experimental coursework trials, the Mesnard quartzite often broke igneous and metamorphic rock hammerstones during attempts to shape the material. The Hixton material, on the other hand, is highly suitable for flintknapping, as evidenced by its extensive usage for the production of chipped tools throughout prehistory (Carr and Boszhardt, 2010; Reber et al., 2017).

Conclusion

In the Upper Great Lakes, artefacts made from CCS quartzite material are found regularly at pre-contact archaeological sites throughout the region. A large quarry and workshop site at Mt. Mesnard in the MMD, for example, features thousands of flakes littered across portions of the mountainside. Visual characteristics of this stone and the sheer number of flakes are evidence that the quarry functioned as a critical reservoir of tool stone for pre-contact peoples living in the area, and possibly even further afield. However, the visual properties of this stone are similar to high-grade material from the SMAD near Hixton Wisconsin, and there is the possibility of source misidentification.

Without the assistance of CL and petrographic analysis, appearances of CCS stones can be similar, but differences are more dramatic at microscopic scales. With the application of back-scatter scanning electron microscopy (BSEM) and cathodoluminescence (CL), we elucidate the underlying microstructure of stone samples. The results of the CL study of the MMD and HSS are similar to those of Julig et al. (1998) and confirm that CL, coupled with EDS, is a repeatable technique to determine composition and textures at high resolution and could possibly be used to identify the source material for pre-historic tools. Improvements in imaging techniques over the past 20 years have also made the technique more reliable.

Here our resulting analysis indicates that because of the different origins of these stones, each has different properties. While MMD stone is suitable for the construction of lithic tools, due to its dense cemented nature, the stone is hard, cumbersome, and from experimental archaeology flint knapping trials, is challenging to work. High-grade Hixton material from the Silver Mound Archaeological District, on the other hand, features more rounded quartz grains and minimal alteration from when the original sediments were deposited. The stone is relatively porous, comparatively lightweight, and relatively uncemented, with an underlying structure desirable for flintknapping.

The differences among these materials suggest that the structure of the MMD materials may have encouraged mostly local applications from peoples with limited access to the higher-quality sources. Despite difficulties with flintknapping, pre-contact people were successful in using this material as evidenced by the thousands of core reduction and bifacial thinning flakes. Given this nature and only limited quantities of tool grade stones in other areas, it is likely that some amount of this material was transported out of the area, traded and used further afield. On a small scale, the material was exploited, exchanged and potentially sought after by various groups. Just as the MMD raw materials are mined in the present day, prehistoric evidence of resource extraction suggests that people have exploited the MMD for resources at the outset of their arrival.

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Figures:

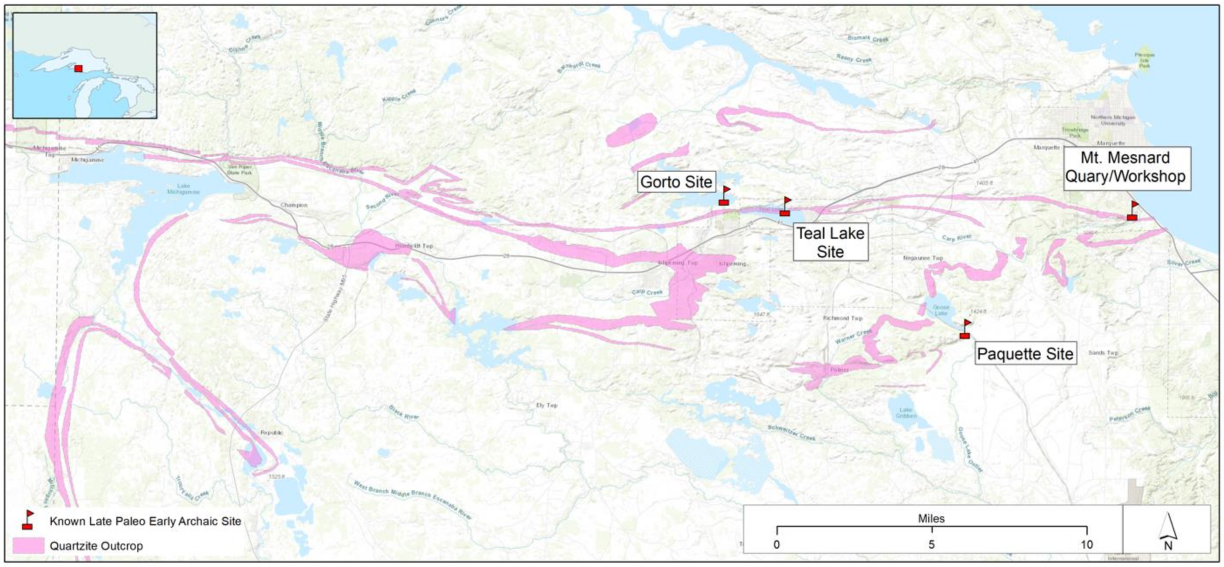


Figure 1: Late Paleoindian/Early Archaic sites and quartzite distribution, compiled from publications from the U.S. Geologic Survey, Michigan Department of Natural Resources - Geology Division, Michigan Technological University, and Cleveland- Cliffs Iron Company and Callahan Mining Corporation.

Figure 2: A diagnostic chert projectile point was found at the Mt. Mesnard Quarry/Workshop site in Fall 2016, supporting evidence to activities in the area dating to the Archaic period. Samples of material from the Mt. Mesnard site in the MMD.

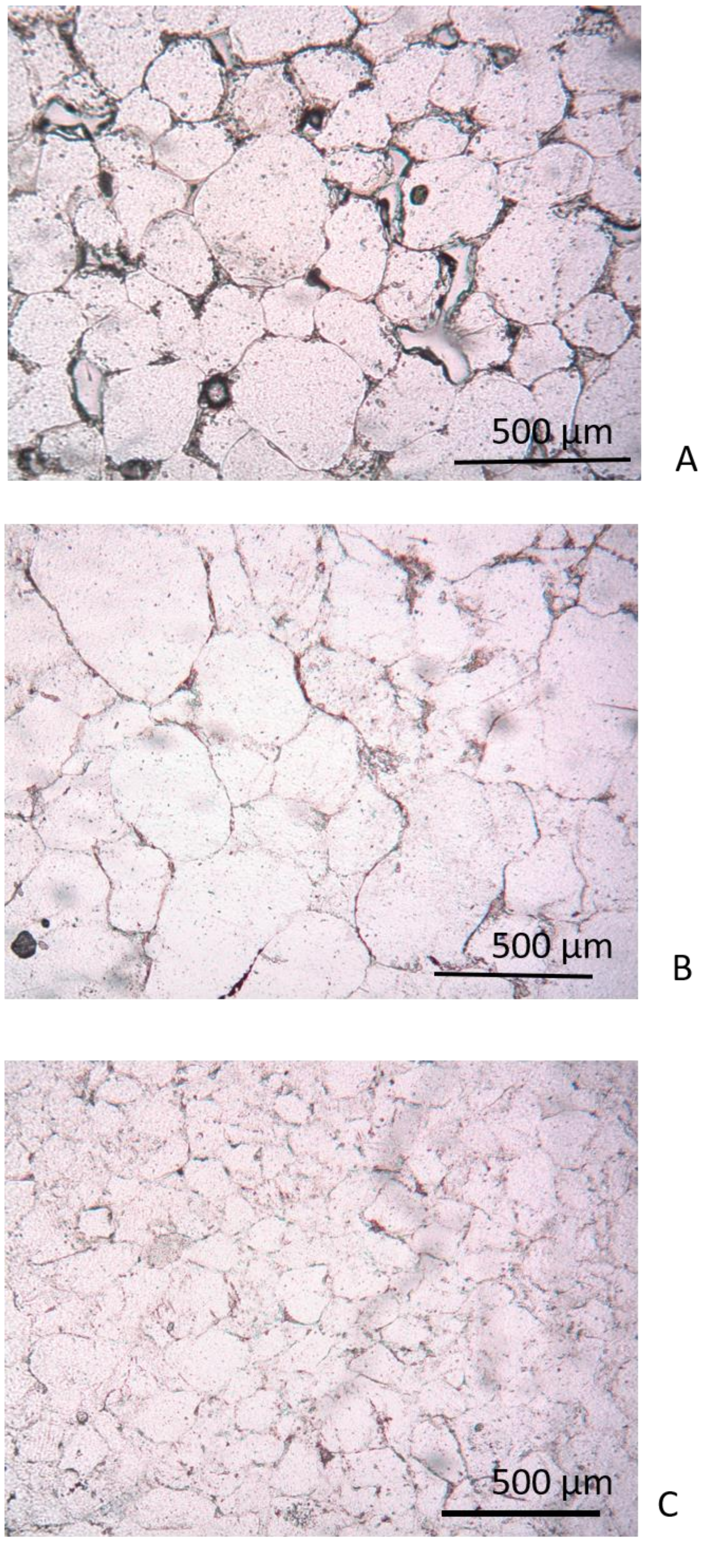


Figure 3: A) Hixton Sandstone (Sample A) and B) MMD Sample B both composed of large, rounded quartz grains. C) MMD Sample C – finer grained with smaller, less rounded grains which are tightly packed.

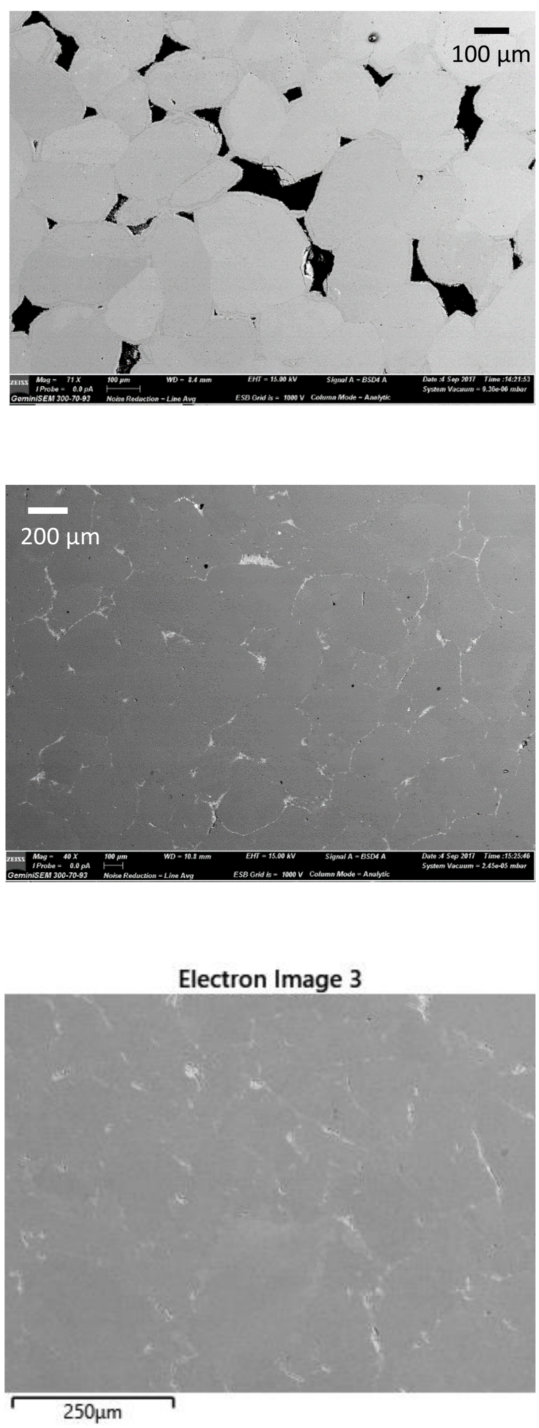
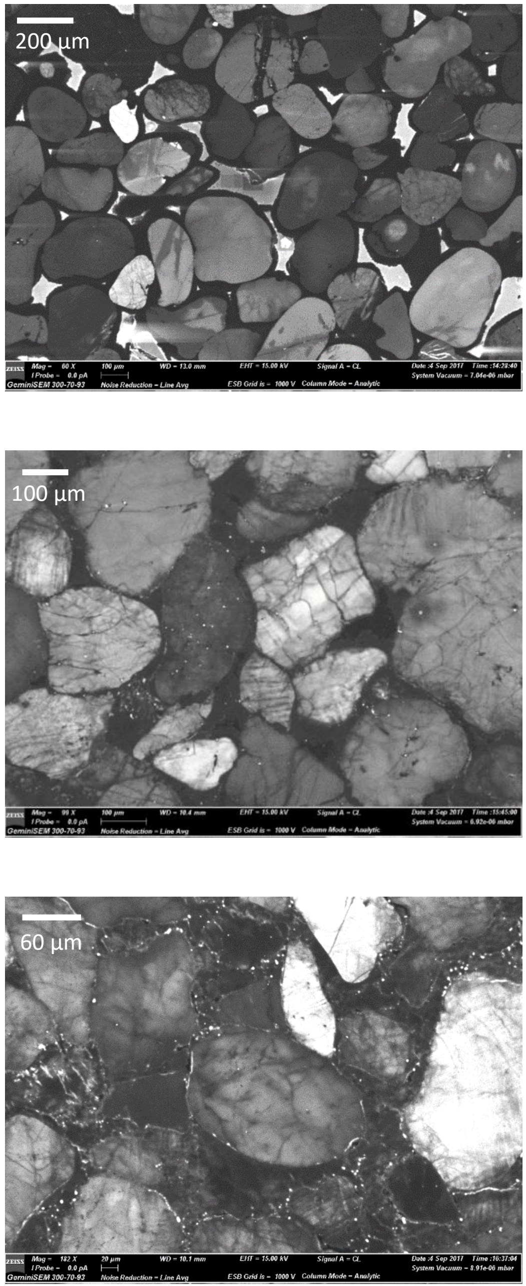


Figure 4: BSEM images, scale bars shown. A) Hixton sandstone (Sample A) showing space (porosity c.10%, black) between quartz grains (grey). This porosity is much reduced in MMD Sample B (B) and absent in MMD Sample C (C).



A

B

C

Figure 5: Cathodoluminescence image, scale bars shown. A) Hixton sandstone (Sample A) showing well rounded quartz grains (various shades of grey) surrounded by a black, non-luminescent quartz cement rim. Space between grains (porosity) is shown in white. B) MMD Sample B also shows large, rounded grains, but these are more cracked. The space between grains has been filled by black, non-luminescent quartz with very small amounts of clay (very bright spots). C) MMD Sample C - quartz grains of various sizes. The space between grains has been filled by black non-luminescent quartz (black) and clay (very bright spots).

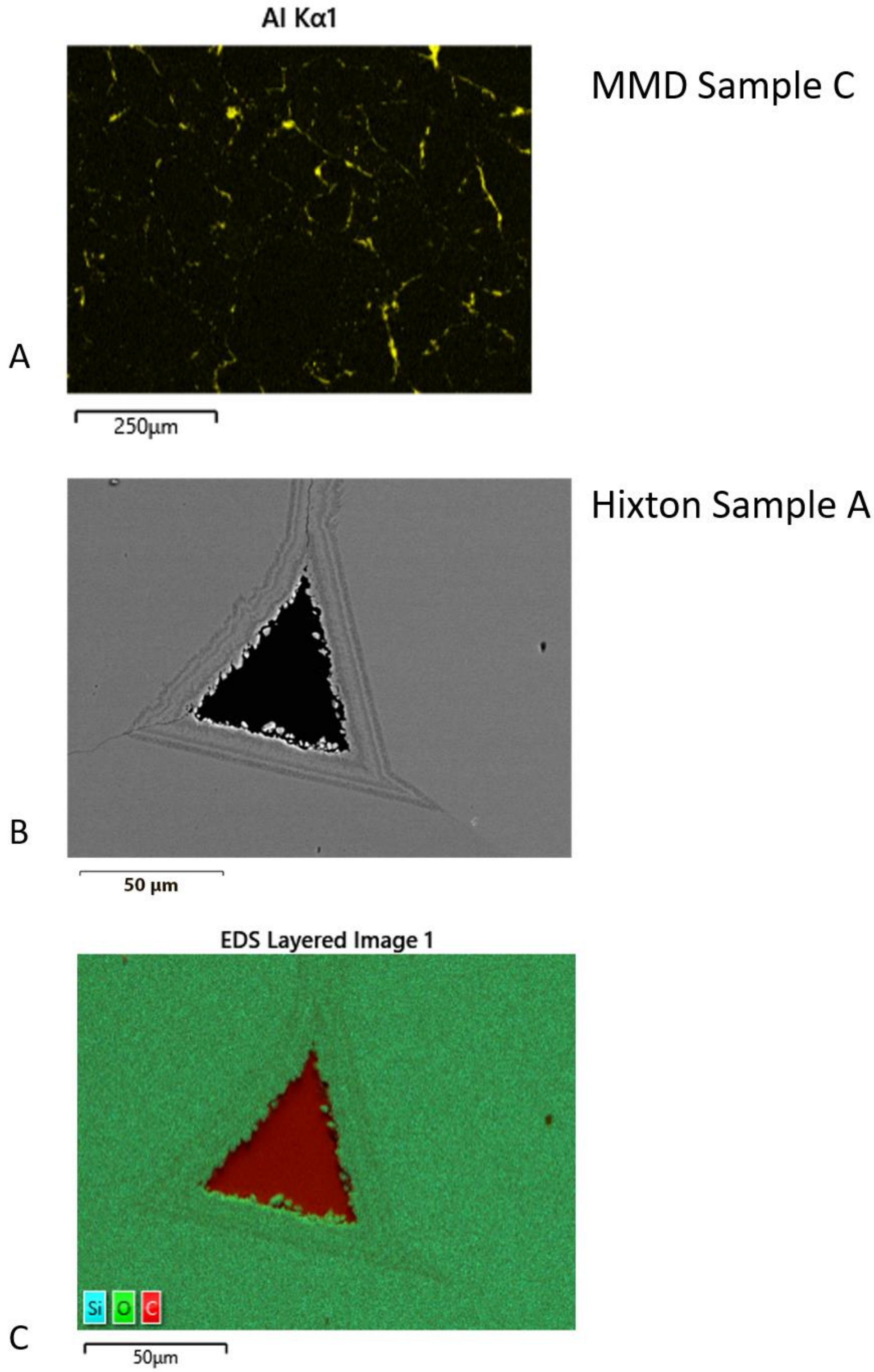


Figure 6: A) MMD Sample C - EDS image showing the distribution of aluminium between grains. B) BSEM image showing the finely layered and crystalline nature of the non-luminescent cement that surrounds rounded grains in the Hixton Sandstone (Sample A). C) EDS analysis shows that this cement is of the same composition as the grains (pure quartz).