



Petrographic and mineralogical characteristics of diagenetic overprinting in Neoproterozoic diamictites from Murchisonfjorden, Nordaustlandet, Svalbard

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Abstract

The diagenetic processes exhibited by the Neoproterozoic diamictites from Murchisonfjorden (Nordaustlandet, NE Svalbard) are presented. Diamictite samples from the Cryogene Polarisbreen Group - the Petrovreen Member of the Elbobreen Formation and the Wilsonbreen Formation were analysed. The mineralogical associations composed of chlorite group minerals (chamosite), albite, calcite, Fe-dolomite, clay minerals (illite) and quartz, recognized by integrated optical microscopy, scanning electron microscopy (SEM) and Raman spectroscopy techniques are typical for an advanced stage of mesogenetic diagenesis. Low-grade burial mineral associations were not identified. Our investigations indicate that during diagenesis increased heat flux accompanied fluid migration and remineralization. Identified Fe-Ti – oxides, anatase in thin sections and magnetite identified by rock-magnetic experiments are the products of the secondary diagenetic mineralization. The ferromagnetic carriers representing primary depositional remanence magnetization (DRM) were not preserved.

Keywords: diamictites, Neoproterozoic, Svalbard, diagenesis, Snowball Earth

1. Introduction

The Caledonian Hecla Hoek Series (Harland & Wilson, 1956) of north-eastern Svalbard hosts an almost continuous Neoproterozoic (850 Ma to 600 Ma) succession of carbonates to siliciclastics. Significant disconformities appear only in the upper part of the Neoproterozoic succession and are related to Cryogenian glaciations (e.g. Hoffman et al., 2012). According to Harland and Wright (1979) Svalbard can be divided into three crustal blocks or terranes that were assembled during Late Caledonian time along major sinistral strike-slip faults from locations along Eastern and North Greenland. Harland (1997) later proposed that

north-eastern Svalbard constituted the separate Eastern Svalbard Terrane (EST). Many workers, following Harland and Wright (1979), accept that before Caledonian time the EST was positioned along the east Greenland margin (e.g. Gee & Page, 1994; Harland, 1997; Gee & Tebenkov, 2004). However, direct paleomagnetic data has so far failed to quantify the paleogeographic position of the EST in relation to Laurentia (e.g. Denyszyn et al., 2009; Eyster et al., 2020; Macdonald & Swanson-Hysell, 2023). Indeed, there is an ongoing discussion concerning the primary character of the paleomagnetic record in the Tonian rocks of the EST (Maloof et al., 2006; Michalski et al., 2012, 2023). Simply, robust paleomagnetic data from the Cryogenian sequences of Svalbard are lacking.

The purpose of this research is to document the petrographic record of the diagenetic processes and the genesis of Fe-Ti oxides and sulphides, which are potential carriers of paleomagnetic signal in two stratigraphic units of the Cryogenian Polarisbreen Group diamictites sampled in the Murchisonfjorden area of Nordaustlandet, Svalbard (Fig. 1). This study is the first stage of ongoing paleomagnetic investigations of the EST Neoproterozoic succession.

This paper is the first to integrate optical microscopy techniques, scanning electron microscopy (SEM) and spectroscopic analyses to investigate the petrography and mineralogy of the Cryogenian EST diamictites.

2. Geological setting

The Neoproterozoic succession of EST is subdivided into the Tonian, Veteranen and Akademikerbreen Groups, succeeded by the Cryogenian to Ediacaran Polarisbreen Group (Harland, 1997). The target diamictites cropping out in Murchisonfjorden belong to two stratigraphic units: the lower unit belongs to the Petrovbreen Member of the Elbobreen Formation, the upper one represents the Wilsonbreen Formation (Halverson et al., 2004; Hoffman et al., 2012).

The first records of Svalbard's Late Precambrian diamictites were by Garwood & Gregory (1898). Kuling (1934) was the first to recognize the diamictites in Eastern Svalbard. They were described in detail by many authors (Krasil'shchikov, 1967; Chumakov, 1968; Flood et al., 1969; Edwards, 1976; Hambrey et al., 1981; Hambrey, 1982; Fairchild & Hambrey, 1984; Harland et al., 1993). Using transmitted light microscopy and cathodoluminescence, these authors focused on the sedimentology, composition and detailed clast descriptions, which allowed for lithostratigraphic correlation of Spitsbergen (Ny Friesland) and Nordaustlandet diamictite units (Fairchild & Hambrey, 1984; Harland et al., 1993).

As direct radiometric age constraints are lacking, the stratigraphic control of Svalbard's Neoproterozoic sections has been primarily on lithostratigraphic and chemo-stratigraphic correlations (Halverson et al., 2004, 2018a,b; Hoffman et al., 2012). Only recently, Halverson et al. (2022) calibrated the age of the Svalbard Tonian succession using carbon isotope chemostratigraphy correlation with other well-dated Tonian sections from Canada, Ethiopia, and Namibia. The most current published age by Zhang et al. (2023) combines a subsidence model with new radiometric Re – Os ages from Akademikerbreen and Polarisbreen Groups and U-Pb maximum depositional age obtained from Veteranen Group of Northeastern Svalbard (Millikin et al., 2022; Zhang et al., 2023). As a result of the significant hiatus between the Russøya Member of the Elbobreen Formation and the overlying Petrovbreen Member the age of the latter lacks precision (Halverson et al., 2018a,b, 2022; Millikin et al., 2022). Based on the isotopically diagnostic Ediacaran Dracoisen

Formation cap dolostone, deposited above upper diamictites, the Wilsonbreen Formation is interpreted as a representation of terminal Cryogenian (Marinoan) glaciation (Hoffman et al., 2012; Halverson et al., 2018a, b, 2022; Millikin et al., 2022).

Structurally, the Murchisonfjorden area exhibits many characteristics of thin-skinned fold and thrust belts. W-E field traverses Sparenestet to Roaldtoppen and Kinnvika to Floraberget and Celsiusberget, revealing that the prominent anticlinal folds display steep to overturned western forelimbs, often with evidence of thrust fault displacements. Cleavage development is prominent in the anticline cores. Cleavage development is lithology controlled in the associated more open synclinal folds. Subsidiary folds vary from upright (e.g. Kinnvika, south shore exposure of Celciusbergert anticline and NE Sore Russoya) to inclined structures, some associated with small-scale east-directed back thrusts. Conjugate shear faults related to brittle failure are noted on all scales. Major E-W trending transfer faults, common features of fold and thrust belts, were not evident. The investigated Polarisbreen diamictites and underlying Akademikerbreen Group rocks of Murchisonfjorden show no recognizable evidence of metamorphic alteration.

3. Sampling and analytical methods

During our 2022 field investigations, 53 independently oriented paleomagnetic samples were collected from eight sites in Murchisonfjorden (Fig. 1). Specifically, six to eight samples were taken from each site. Two sites were collected from the Petrovbreen Member of the Elbobreen Formation and six from the Wilsonbreen Formation (Tab. 1). Petrographic and mineralogical observations were conducted on ten polished-thin sections representing all sites (three from the Petrovbreen diamictites and seven from the Wilsonbreen diamictites).

The petrography and mineralogy of the target diamictites were documented using integrated optical microscopy techniques, SEM, and Raman spectroscopic analyses. Following equipment was used: the Keyence VHX-7000 and Nikon E600 optical microscopes and JEOL JSM-6380LA scanning electron microscope at the Inter-Institute Microanalytical Complex for Minerals and Synthetic Substances at Warsaw University (Faculty of Geology) and Field Emission Electron Probe Microanalyzer (JXA-8530F JEOL HyperProbe) at the Department of Earth Sciences, Uppsala University. Minerals were identified using their optical features in transmitted and reflected polarized light, Energy Dispersive X-ray Spectroscopy (EDS) and Raman spectroscopy data. Spectroscopic investigations were performed using a Renishaw inVia Qontor Raman spectrometer with 532 nm laser excitation, 50 mW laser power, 1800 lines/mm grating and a Peltier-cooled CCD detector. The spectra were recorded in the 100 – 4000 cm⁻¹ range and 10% of the laser power during the measurement experiments. Analytical points were selected under a Leica optical microscope with a 50x objective.

Saturation isothermal remanent magnetization (SIRM) experiments were conducted at the Laboratory of Palaeomagnetism, Institute of Geophysics, Polish Academy of Sciences using pulse magnetizer MMPM10 for sample magnetization up to 3T, non-magnetic furnace

MMTDSC for thermal demagnetization of the samples and superconducting quantum interface device (SQUID; 2G Enterprise, Model 755, USA) to measure natural remanent magnetization (NRM) and residual magnetization of the samples after each demagnetization step.

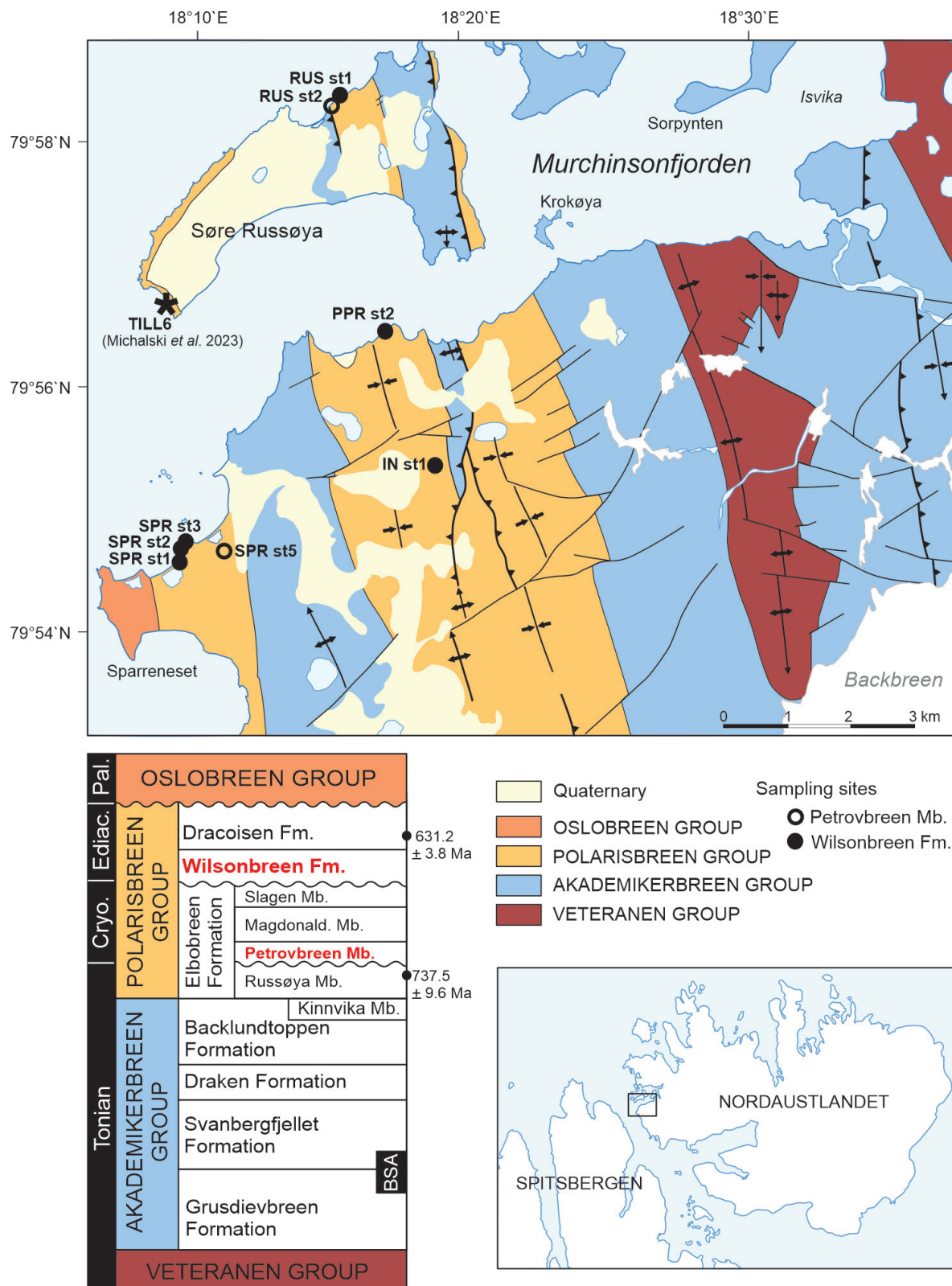


Figure 1. Geological map of southwestern Murchinsonfjorden modified after Hoffman et al. (2012) and online interactive geological map available at the NorskPolarinstitutt website (<https://www.npolar.no/>). Circles/dots represent the sampling locations of the Petrovreen Member (Mb.)/Wilsonbreen Formation (Fm.) diamictites, respectively. Paleomagnetic site of the Wilsonbreen Fm. diamictite (TILL6), sampled in 2013 and analysed by Michalski et al. (2023), is marked by a star. Stratigraphic column after Halverson et al. (2022) with Re-Os ages after Millikin et al. (2022). BSA – Bittel Spring Anomaly.

Table 1. Geographic coordinates (WGS84 reference system) of the diamictite sites sampled in Murchisonfjorden (this study).

Site	Latitude	Longitude	Stratigraphic unit
IN st1	79.91927 N	18.32933 E	Wilsonbreen Fm.
RUS st1	79.97208 N	18.24177 E	Wilsonbreen Fm.
RUS st2	79.96948 N	18.23032 E	Petrovbreen Mb.
PPR st2	79.94028 N	18.28775 E	Wilsonbreen Fm.
SPR st1	79.90663 N	18.13743 E	Wilsonbreen Fm.
SPR st2	79.90897 N	18.13895 E	Wilsonbreen Fm.
SPR st3	79.90950 N	18.14060 E	Wilsonbreen Fm.
SPR st5	79.90877 N	18.17152 E	Petrovbreen Mb.

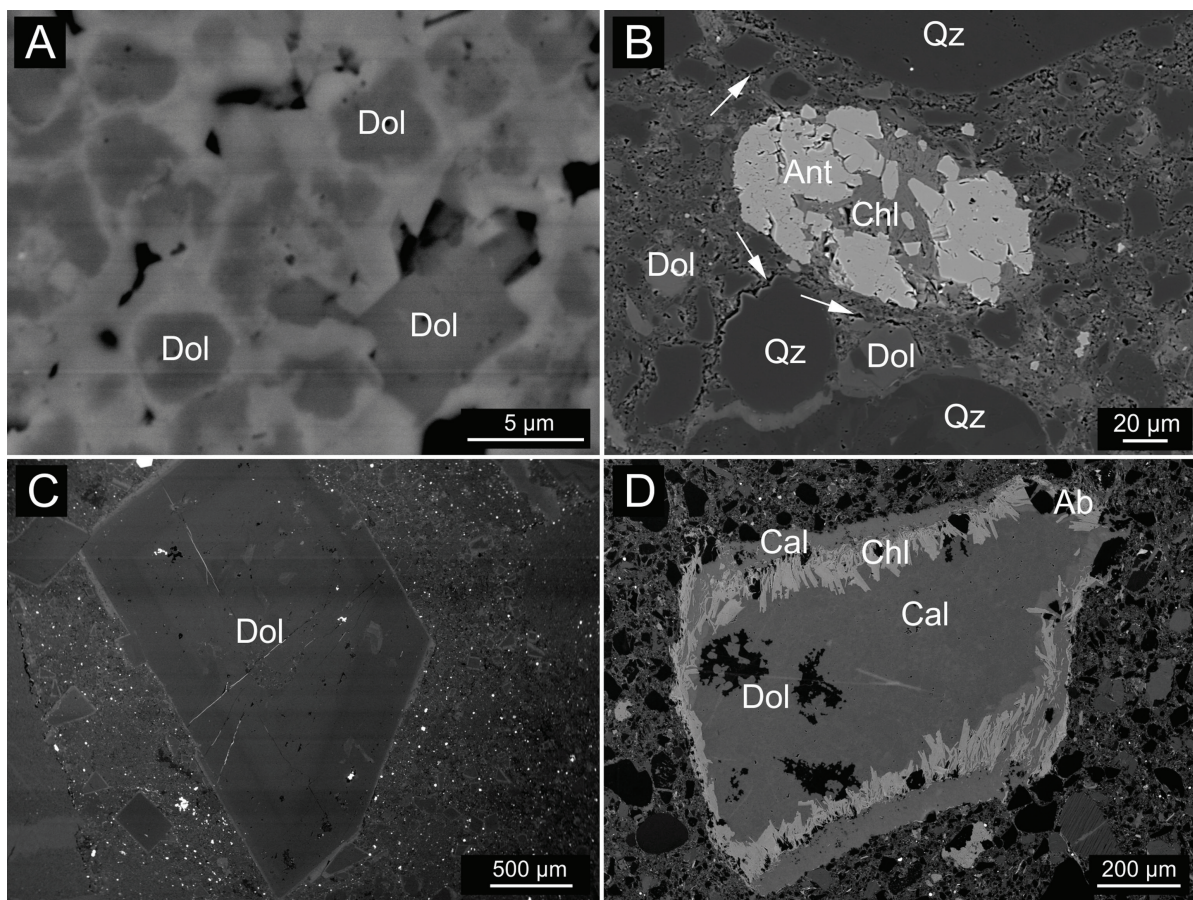


Figure 2. Backscattered electron (BSE) images of the diamictite samples from the Petrovbreen Mb. and the Wilsonbreen Fm.; A - Porosity and overgrowing of Fe-rich chlomite on dolomite (RUS st2); B - Porosity in the matrix (marked with an arrow) with secondary diagenetic overprinting of the clast by chlorite and anatase. Detrital dolomite and quartz with visible etched edges (IN st1); C - Euhedral, compositionally zoned dolomite with secondary Fe-rich outer rim, tiny grains of scattered pyrite and anatase (both white in colour) visible in the matrix (RUS st2); D - Micrite limestone clast being partly replaced by dolomite, overgrown first by chlorite (light-grey, crystals arranged in the form of a palisade) and then albite (dark), grey most outer rim consists of new calcite (SPR st1); Ab - albite, Ant - anatase, Cal - calcite, Chl - chlorite, Dol - dolomite, Qz - quartz.

4. Results

4.1. Petrography of the diamictites from the Murchisonfjorden

The two targeted diamictite units differ substantially. In the Petrovbreen Mb., the clasts are the dominant component of the rock (ca. 80 vol%), while the Wilsonbreen Fm. is matrix-dominated. A yellowish

weathering of both sequences suggests a common dolomitization. The Supplementary Materials present extensive macroscopic and microscopic documentation of investigated diamictites.

The Petrovbreen Mb. diamictites consist of poorly sorted, angular to sub-rounded, pebble-sized (up to 5 cm in diameter) clasts supported by a micrite/carbonate mud matrix. Dolomicrite clasts and crystalline

carbonate clasts dominate. Minor chert and individual grains of quartz and feldspar were noted. Partly clasts can be matched with the underlying carbonates of the Russoya Mb. (compare Harland et al., 1993). The matrix of diamictites is composed of fine-grained carbonate rock fragments with recognizable grains of apatite, pyrite, quartz and chalcedony. Fairchild and Hambrey (1984) described this matrix as a “rock flour” composed of dolomicrite with muscovite and quartz. In some specimens, carbonate veins cross the clasts and the matrix.

In the majority, the Wilsonbreen Fm. clasts are rarely larger than 4 mm and are well-rounded to sub-rounded; sub-angular clasts are rare. They are sporadically dispersed throughout the dark grey matrix, forming less than 10% of the rock. The diamictites incorporate clasts dominated by quartz, feldspars, and magmatic rock fragments. The population comprises various precursor rock types, including igneous (doleritic, granitoid), metamorphic and sedimentary (carbonates, mudstones, cherts, siltstones, sandstones) lithologies (compare Kulling, 1934; Krasil'shchikov, 1967; Flood et al., 1969; Edwards, 1976; Hambrey et al., 1981). Clast sizes are highly variable, and individual clasts may reach 7–8 cm. Microscopically, the diamictites matrix comprises a clay-mica-carbonate mixture with fine-grained sand- and silt-sized grains of quartz, plagioclase and K-feldspar

(compare Edwards, 1976), with grains of apatite, barite, zircon, Ti-oxides and chlorite minerals as well as small amounts of pyrite and chalcopryrite.

Optical, SEM and Raman spectroscopic observations have revealed that all the sampled diamictites display significant diagenetic mineralogical changes. Multiple overgrowths (Fig. 2A,B) on some grains and evidence of diagenetic mineralogical alteration indicate significant fluid flow through the sediment pore spaces during compaction and lithification. The carbonate clasts have diagenetic overgrowths of chamosite with albite (Fig. 2D; 3A) and later calcite, while the magmatic clasts are chloritized to varying degrees. Some clasts also show iron and clay mineral enrichments (Fig. 2C; 3B-D). As none of the sampled rocks exhibits low-grade metamorphic mineral assemblages, we conclude that the diamictites were not subject to such elevated temperature/pressure conditions.

4.2. Mineralogy of Fe-Ti bearing minerals

The Fe-Ti mineral assemblage in both diamictite sequences is dominated by single crystals and aggregates of framboidal pyrite (Fig. 4), which occur in the zone of carbonate-clay types of cement connecting detrital grains or different mineral compositions. The size of pyrite crystals varies from sub-microscopic to grains in the 70–100 µm in diameter range.

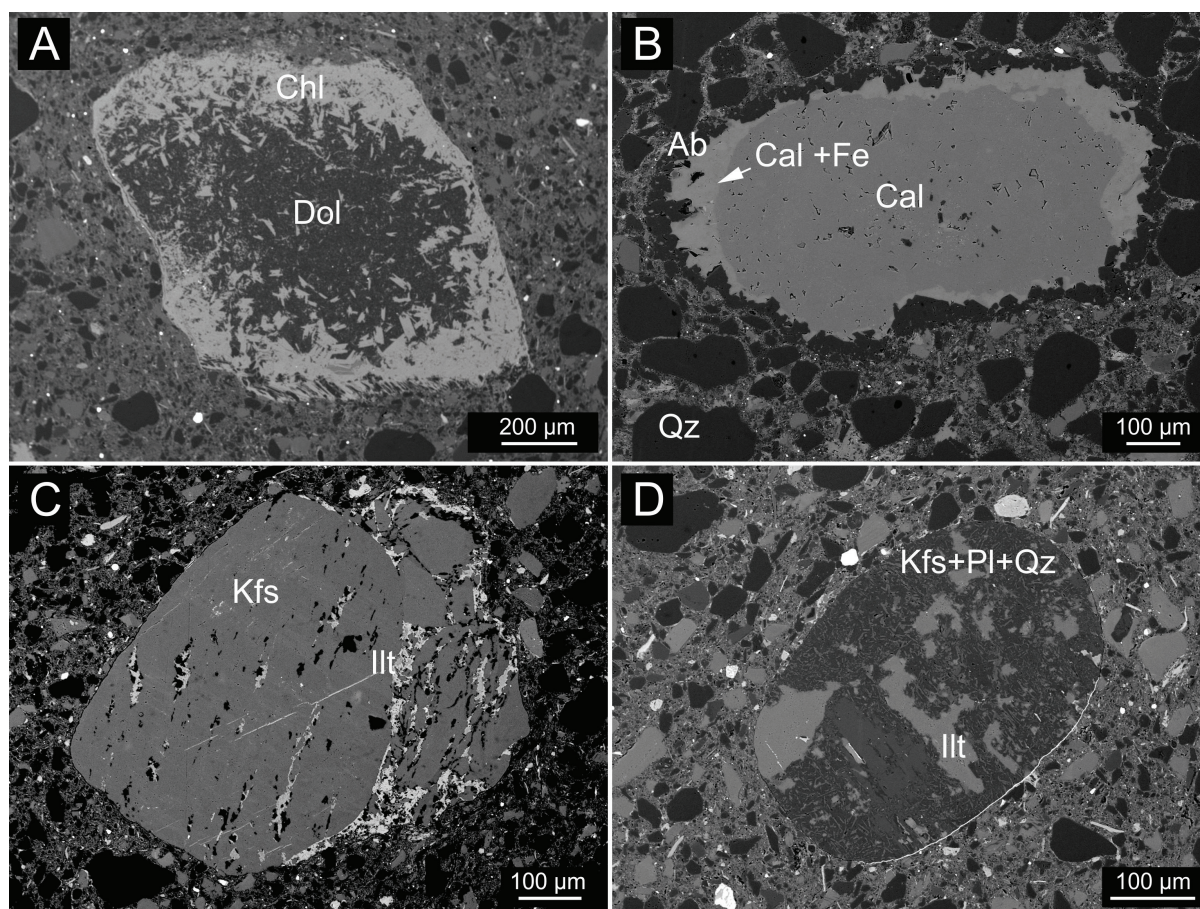


Figure 3. Backscattered electron (BSE) images of the diamictite samples from the Petrovbreen Mb. and the Wilsonbreen Fm.; A - Dolomitic clast with outer rim of chlorite (SPR st1); B - Clast of micrite limestone rimmed by late Fe-rich calcite and albite (SPR st2); C - K-feldspar with diagenetic illite distributed along cleavage plains (PPR st2); D - Magmatic clast (granite) with K-feldspar, quartz and plagioclase, partly replaced by clay minerals (PPR st2); Ab - albite, Cal - calcite, Chl - chlorite, Dol - dolomite, Ill - illite (clay minerals), Kfs - K-feldspar, Pl - plagioclase, Qz - quartz.

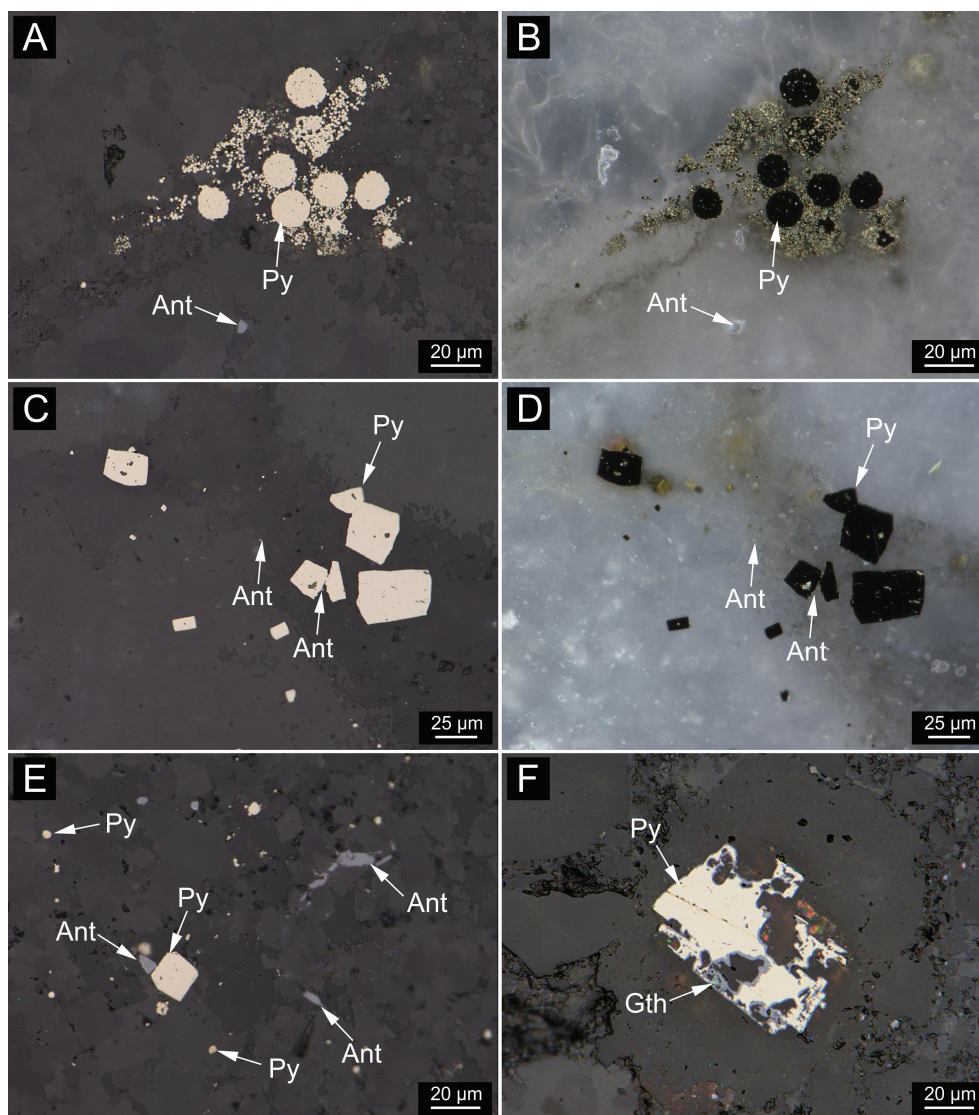


Figure 4. Pyrite and coexisting minerals from the TiO_2 group (anatase). A-B - Framboidal pyrite aggregates, partly modified by syntaxial growth of late diagenetic pyrite. Polarized reflected light, one Nicol (A) and crossed Nicols (B), sample SPR st5; C-D - Euhedral pyrite crystals in the carbonate matrix. Tiny grains of anatase co-occur with pyrite. Polarized reflected light, one Nicol (C) and crossed Nicols (D), sample SPR st5; E - Single euhedral pyrite crystal associated with numerous small oval pyrite aggregates that probably represent partly recrystallized framboidal pyrite aggregates. Tiny anatase intergrowths some of which occur as euhedral grains in the carbonate matrix. Polarized reflected light, one Nicol, sample RUS st2; F - Euhedral pyrite partly replaced by late goethite. Polarized reflected light, one Nicol, sample RUS st2; Ant - anatase, Gth - goethite, Py - pyrite.

Occasionally, chalcopyrite intergrowths and aggregates were documented in higher pyrite concentration zones. Their textural relations suggest the simultaneous crystallization of pyrite and chalcopyrite (Fig. 4A,B). Small oval pyrite grains with destroyed framboidal structure (Fig. 4E) are common in all samples. This is a consequence of the advanced degree of syntaxial pyrite growth processes occurring around pre-existing tiny pyrite crystals. These processes lead to the formation of massive spherical pyrite aggregates (Fig. 4A), in which the original framboidal structure is gradually replaced.

The second common textural variety of pyrite grains found in the samples exhibit euhedral habits (Fig. 4C-F). This type of pyrite occurs as single grains or clusters of crystals in clay-carbonate cement. These crystals have no visible zonal texture (Fig. 4C) and no inclusions of other ore minerals (e.g. pyrrhotite, chalcopyrite, magnetite). Some pyrite crystals underwent secondary

oxidation processes, being replaced by secondary Fe oxyhydroxides (Fig. 4F).

The Fe-Ti bearing phases of the diamictite samples reveal an extreme degree of alteration of Fe-Ti oxides. Anatase is the only phase identified in the samples (Fig. 4E; 5) and is a significant component pseudomorphing ilmenite (Fig. 5E) and Ti-bearing magnetite with oxy-exsolved ilmenite (Fig. 5C,D). Anatase also occurs in small-sized grains that partly represent detrital grains and newly crystallized diagenetic crystals (Fig. 4E; 5A,C,E). Optically, anatase exhibits intense white internal reflections (Fig. 5B,D) that have aided the identification of sub-micrometer grains. Systematic studies using the *in-situ* Raman spectroscopy method confirmed the common presence of anatase in the diamictite samples (Fig. 6A). In addition to the presence of dust-like anatase crystals dispersed within the matrix (Fig. 5C,E; 6B), it also forms intergrowths with euhedral pyrite. The

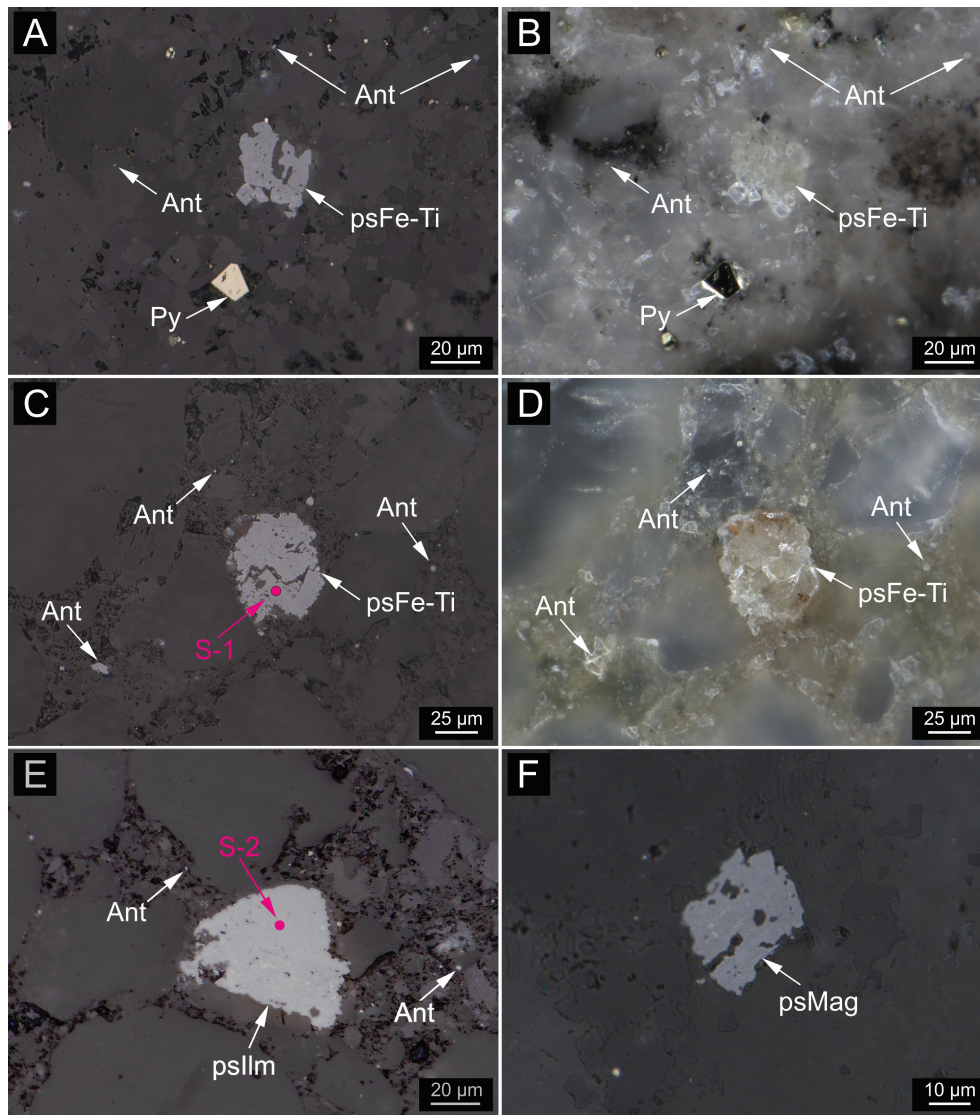


Figure 5. Highly altered detrital grains of Fe-Ti oxides and their alteration products (pseudomorphs after Fe-Ti oxide consists of anatase). Please note the diagnostic white internal reflections visible in (B) and (D). A-B - Anatase pseudomorph after detrital Fe-Ti oxide co-occurred with euhedral pyrite and fine-grained diagenetic anatase. Polarized reflected light, one Nicol (A) and crossed Nicols (B), sample RUS st2; C-D - Anatase pseudomorph after detrital oxy-exsolved titanomagnetite grain. Polarized reflected light, one Nicol (C) and crossed Nicols (D), sample IN st1; E - Anatase pseudomorph after ilmenite, numerous, small anatase grains occur in the carbonate-clay matrix. Polarized reflected light, one Nicol, sample SPR st5; F - Anatase pseudomorph after Ti-magnetite, sample SPR st5. Ant - anatase, psFe-Ti - pseudomorph after Fe-Ti oxide, psIlm - pseudo ilmenite, psMag - pseudo magnetite, Py - pyrite. Red dots (in C and E) indicate points where the S-1 and S-2 Raman spectra were collected. The interpretation of the spectra is given in Fig. 6A.

textural relationships indicate that euhedral pyrite and anatase (Fig. 4E) as well as anatase and chamosite (Fig. 2B) crystallized simultaneously.

None of the samples examined incorporate any ferro-magnetic phases. However, the presence of ferromagnetic (*sensu lato*) mineral, characterized by a maximum unblocking temperature around 580°C typical for magnetite (Lowrie, 1990; Dunlop & Özdemir, 1997), was indicated by results of the three-component isothermal remanent magnetization (Michalski et al., 2023) and SIRM experiments (Fig. 7).

5. Discussion and conclusions

1. From a petrographic point of view, the studied sites represent typical examples of the Petrovbrein Member and the Wilsonbrein Formation

diamictites. Although petrographically investigated stratigraphic units significantly differ, they could share a similar thermal history.

2. The lack of low-grade metamorphic mineral associations and the presence of chlorite group minerals (chamosite), albite, calcite, Fe-dolomite, clay minerals (illite) and quartz indicate that diamictites were subjected to diagenetic processes, below 200–250°C (Blatt et al., 2006), and did not reach anchi-metamorphic grade. The diagenetic conditions, theoretically, do not reset completely thermally the syn-depositional DRM, which could survive in ferromagnetic grains characterized by unblocking temperatures above 200–250°C. However, our observations indicate that during diagenesis increased heat flux was accompanied by fluid migration and remineralization, which could have resulted in the complete removal of the

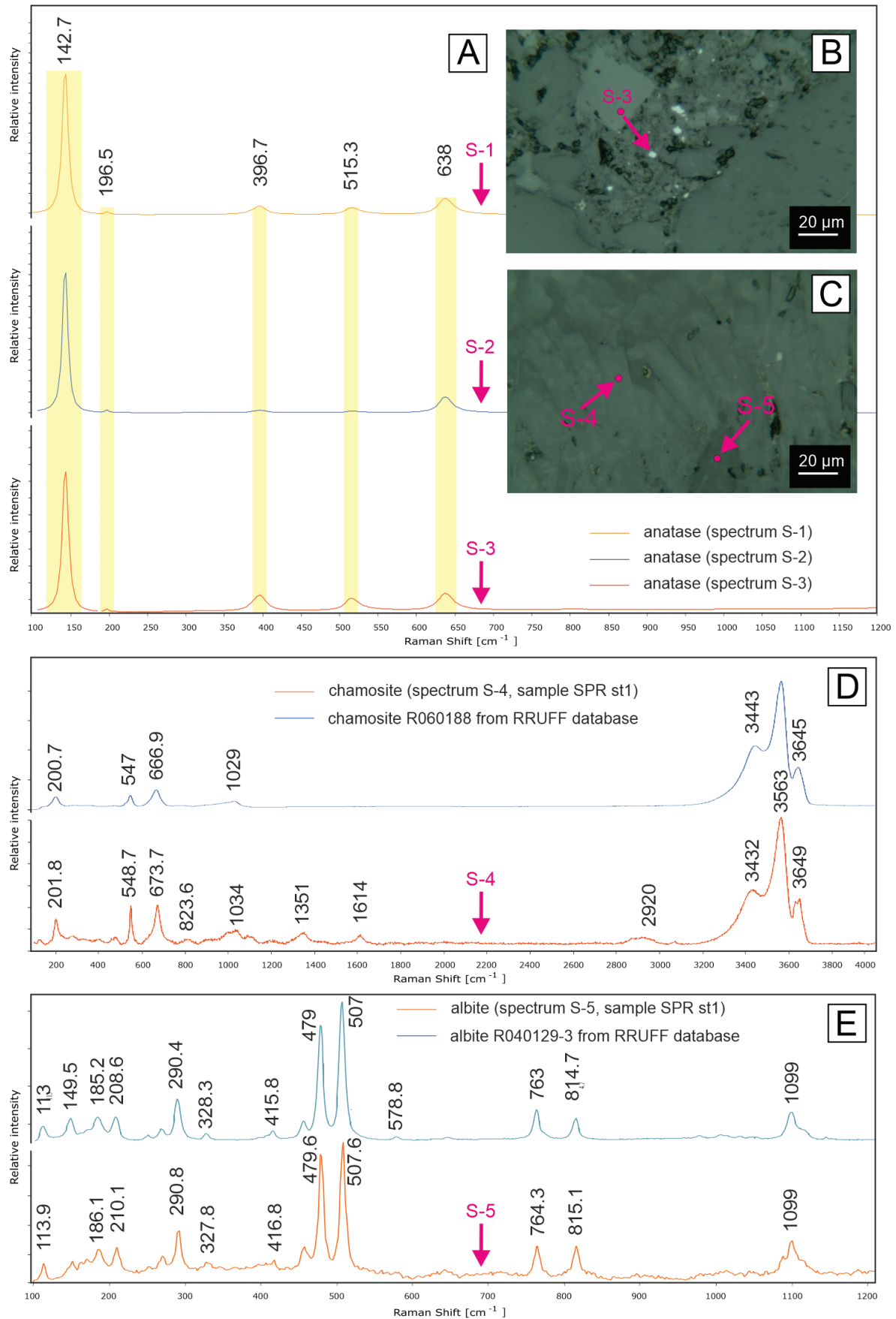


Figure 6. Identification of diagenetic minerals using the Raman spectroscopy method. A - Raman spectra of anatase recorded from pseudo-morphs after detrital magnetite (spectrum S-1, see Fig. 5C), ilmenite (spectrum S-2, see Fig. 5E), and refined dust-like anatase grains in a carbonate-clay matrix (spectrum S-3); B - Carbonate-clay matrix with fine diagenetic anatase crystals. The red dot marks where the anatase spectrum S-3 were recorded, and a reflected light image was taken from the IN st1 sample; C - Fe-Mg chlorite (chamosite) in paragenetic association with albite. Red dots indicate the Raman analytical points of spectrum S-4 (chamosite) and spectrum S-5 (albite). The image was taken from the SPR st1 sample; D-E - Representative spectra of Fe-Mg chlorite (chamosite) and albite were recorded from the alteration zone surrounding the carbonate clast from the SPR st1 sample.

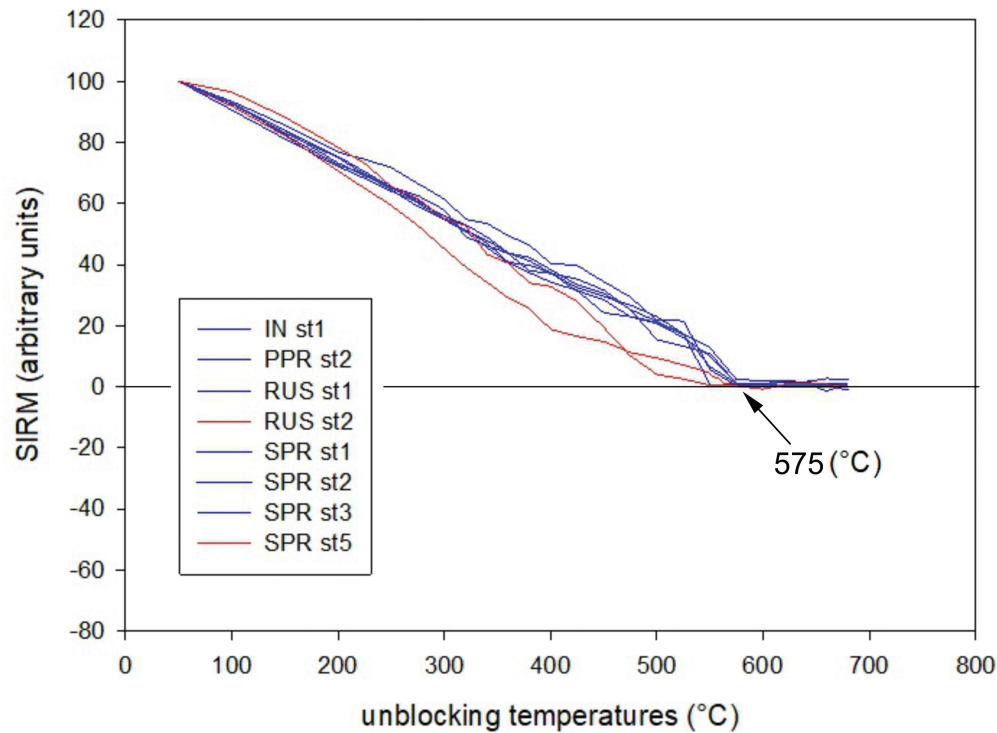


Figure 7. Saturation isothermal remanent magnetization (SIRM) results with the visible presence of magnetite (maximum blocking temperature for magnetite - 575°C) in diamictites; red lines - Petrovbrene Mb., blue lines - Wilsonbrene Fm.

primary ferromagnetic mineral assemblage and imposing the carriers of the secondary chemical remanent magnetization (CRM).

3. In carbonate rocks primary DRM can be carried by magnetite with very low or no titanium admixtures and/or texturally diverse intergrowths of oxy-exsolved ilmenite (Haggerty, 1976, 1991). Early diagenetic magnetic remanence is often related to a magnetotactic bacteria's activity (Devoard et al., 1998), which produces small grains of biogenic magnetite in a 20–120 nm size range. Oxidation usually occurs during interactions of pore waters with magnetite and Fe-Ti oxides, forming secondary maghemite, hematite, titanohematite, and anatase. Microscopically, Fe – phases in the diamictites are dominated by pyrite and Fe-dolomite. Fe-Ti oxides are represented exclusively by secondary diagenetic anatase. No ferromagnetic (*sensu lato*) minerals were detected during integrated SEM-EDS analyses. The presence of magnetite was, however, undoubtedly confirmed by thermomagnetic experiments (this study Fig. 7, Michalski et al., 2023). Sub-microscopic magnetite (< 100 nm) can form intergrowths in diagenetic chlorite and/or pyrite. Further research will focus on precisely locating the paleomagnetic carriers.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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