RESEARCH ARTICLE – VÝZKUMNÝ ČLÁNEK

Limonite deposit at the Mikniškiai settlement site (South Lithuania): A natural stratum or an exploited ore body?

Ložisko limonitu na sídlišti Mikniškiai (jižní Litva): přírodní vrstva nebo těžené rudní těleso?

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A cemented limonite deposit and sunken features recorded at the Mikniškiai settlement site (mid-1st century BC – early 3rd century AD) have been interpreted as potential signs of shallow opencast ore mining. Inhabitants of this area of Lithuania maintained close relations with the Bogaczewo culture, through which knowledge of iron metallurgy spread. However, the archaeometallurgical investigation revealed that the tested samples represent natural bog ore that was insufficiently Fe-enriched material for iron smelting. There were no attempts to increase the iron content by proper pre-processing of the ore. Moreover, the archaeological investigation revealed no traces of ore roasting and not a single piece of iron slag was found at the site. This implies that to assume the occurrence of ore-rich sediments in the vicinity of Iron Age settlements as an indication of iron bloomery might be premature and the circumstances of their discovery allow more than one alternative interpretation.

bog iron ore – limonite – archaeometallurgy – Iron Age settlement – Southern Lithuania

Ložisko stmeleného limonitu a zahloubené objekty zachycené v rámci sídliště Mikniškiai (polovina 1. století př. n. l. – počátek 3. století n. l.) byly interpretovány jako potenciální známky mělké povrchové těžby rudy. Obyvatelé této oblasti Litvy udržovali úzké vztahy s kulturou Bogaczewo, jejímž prostřednictvím se šířily znalosti o metalurgii železa. Archeometalurgický průzkum však ukázal, že testované vzorky představují přírodní bahenní rudu, která nebyla dostatečně obohacena pro tavení železa. Nebyly zaznamenány žádné pokusy o zvýšení obsahu železa vhodným předzpracováním rudy. Archeologický průzkum navíc neodhalil žádné stopy po pražení rudy a v lokalitě nebyl nalezen ani jeden kus železné strusky. Z toho vyplývá, že předpokládat výskyt sedimentů bohatých na rudu v okolí sídliště z doby železné jako známku hutnění železa by mohlo být předčasné a okolnosti nálezu umožňují více než jednu alternativní interpretaci.

bahenní ruda – limonit – archeometalurgie – sídliště doby železné – jižní Litva

Introduction

Iron bloomery is one of the most pronounced activities traceable through archaeological materials. Iron smelting produces a great deal of waste, namely iron slag that does not decompose and, therefore, iron bloomery sites contain an enormous amount of slag that has survived to this day and is abundantly found during archaeological excavations. Remains of furnaces, burnt soil, and charcoal found along with slag enable a precise identification of smelting sites and the specification of raw materials used in the smelting process.

Although iron was produced from local bog ore in prehistoric Lithuania, the bog ore itself is rarely found during archaeological excavations: its traces have been documented

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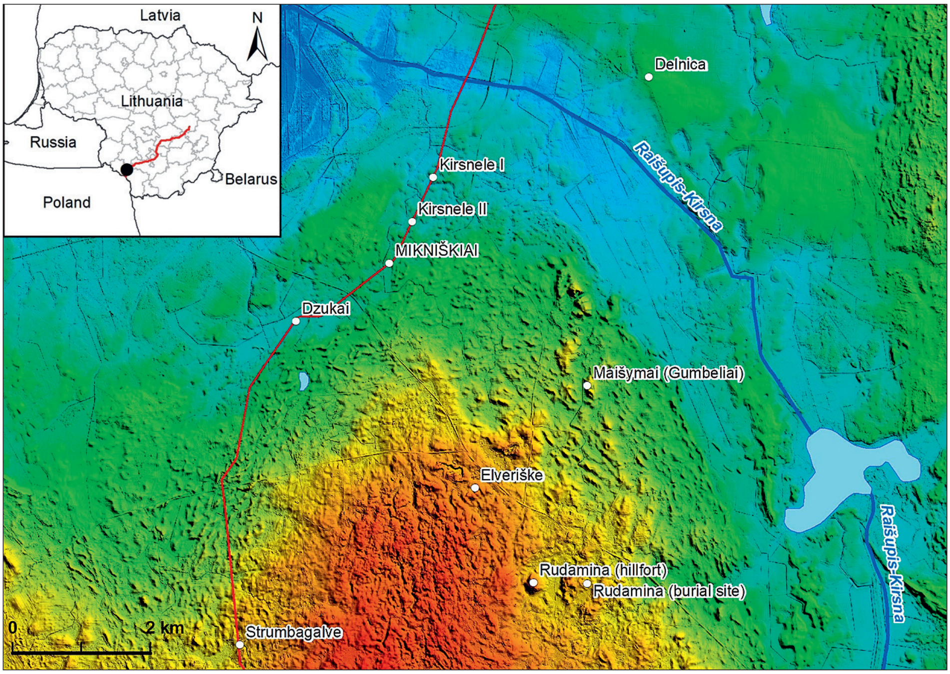

Fig. 1. Landscape setting of the Mikniškiai site with the nearest archaeological sites mentioned in the text. The red line marks the GIPL route (the terrain based on LiDAR data SEŽP_0.5LT © National Land Service under the Ministry of Agriculture of the Republic of Lithuania, 2009–2010).

only at approximately 7 % of the sites bearing features of ancient iron metallurgy (*Salatkienė 2009*). Therefore, we have very little evidence of bog iron ore in Lithuania and also lack an understanding of the bog ore occurrences in archaeological contexts and their interpretation.

Admittedly, this situation may be caused by the small size of the excavated areas or the poor preservability of the material; we can also assume that the ore was fully utilised in the smelting process. Nevertheless, the main limiting factor is an inadequate field research methodology that fails to meet contemporary standards. For many years, finds related to iron smelting have received little attention and that has impeded the ways such materials are recorded in the field. Ore evidence could potentially have gone unnoticed during field excavations or could have been neglected as worthless ecofacts. Therefore, collecting field data in compliance with the new standards of the contemporary methodology, as well as further laboratory testing of the acquired materials are of extreme importance, as they may open different perspectives for iron metallurgy research.

The Gas Interconnection Poland-Lithuania (GIPL) project carried out in 2020–2021 created a gas pipeline with a total length of 522 km, of which 165 km runs through Lithuania. During the survey research, many previously unknown archaeological sites dating back to different periods were discovered. Several new sites were identified and investigated in the historic Yotvingian-Sudovian Land of Kirsnava located in the territory of the current Lazdijai District Municipality (*Fig. 1*). Even before the excavations were finished, the settlements of Mikniškiai and Dzūkai received a great deal of attention from the mass media and the public, as mineral deposits of bog ore were found therein. Previously, such deposits had never been recorded or examined by archaeologists in Lithuania and, hence, their interpretation is not clear. Were they natural formations coinciding with the areas of ancient settlements just by accident or as ore bodies potentially exploited? This paper seeks to at least partly answer these questions. By archaeometallurgical analysis of samples from the Mikniškiai settlement, it aims to assess the probability of bog ore exploitation in the Iron Age.

Natural and cultural setting of the study area

The Mikniškiai settlement is located in Southwestern Lithuania, 9 km from the Lithuanian-Polish border (Lazdijai District Municipality, Šeštokai Eldership, WGS N 54° 19' 26.66", E 23° 24' 32.47"). The current landscape is non-urbanized, with predominant grasslands and arable fields. The soil is mainly sandy loam.

Geomorphologically, the Mikniškiai settlement was established in the northwestern part of the marginal moraine Sūduva Upland, specifically at the margin of the undulating Kalvarija plateau bordering here with the hilly Alytus Upland. The studied area was formed mostly by the glaciers of the final stage of the Baltic glaciation (Upper Nemunas; *Guobytė 2002*). After the glacier retreated, the sediments were washed away and small limnoglacial pools formed in the depressions occurring due to later thermokarst processes. They were filled with limnoglacial sands and, later, peat formed therein. Undulating flat morainic mounds were slightly dryer but were moistened by seasonal floods, thus having had a high groundwater table that used to be even higher before the melioration systems were built.

To date, visually expressive hillforts have had a prominent position among the registered archaeological sites in this area. The nearest hillforts of Elveriškė, Gumbeliai (Maišymai), and Rudamina are located 3–5 km SW from the Mikniškiai settlement (*Tautavičius 1975*; *Kulikauskas 1982*). As for earlier times, the literature mentions only several Roman Period burial sites in Rudamina and Delnica (*Rimantienė 1977*). Such a small number of known archaeological sites is likely the result of the lack of surveys rather than the sparsity of prehistoric occupation. This has been documented by discoveries of new Iron Age sites in the microregion during the GIPL project: along with Mikniškiai, they were found in Dzūkai (*Kvizikevičius – Čepelytė 2022*), Kirsnelė I and II (*Kiniulis – Kliaugaitė 2022*), and Strumbagalvė (*Kvizikevičius 2022*).

Archaeological traces of settlement in Mikniškiai were detected in a 60-m-long section of the gas pipeline in 2021 (*Fig. 2*). The researched section ran across a several-meter-high elevation – a short and narrow isthmus between two peaty depressions at its NE and SW foot. The altitude differences ranged from 117 to 119 meters above sea level. The margins of the wetlands matched with the boundaries of the anthropogenic sediments in the NE–SW direction. The settlement boundaries on the W–E axis remained unidentified during the survey, but the topography implies that the settlement could have stretched further both to the west and the east into the dryland widening at the ends of the 'land-bridge'. Some 100 m to the west, settlement may have been bordered by a ravine of the Maišymai Brook that flows into the Kirsna River. The SE boundary is less clear, but the settlement area was

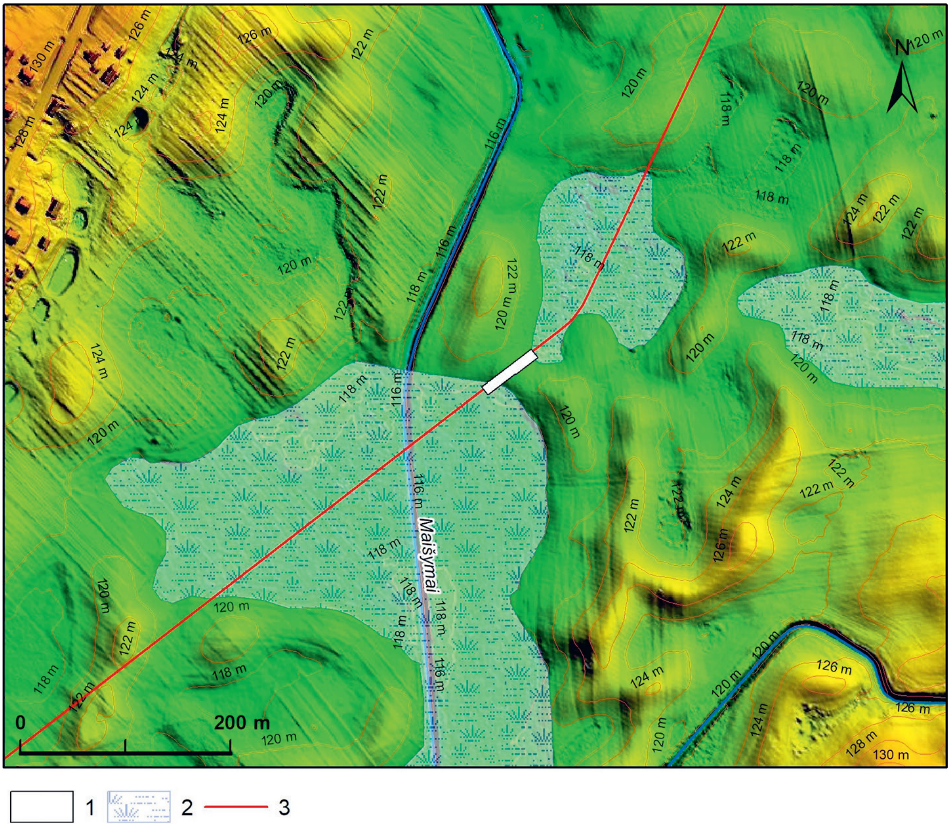

Fig. 2. Topography of the Mikniškiai settlement: 1 – area investigated in 2021; 2 – palaeolakes; 3 – the GIPL route (the terrain based on LiDAR data SEŽP_0.5LT © National Land Service under the Ministry of Agriculture of the Republic of Lithuania, 2009–2010).

probably limited to a single hillock and it is unlikely that it used to extend to the adjacent ones. Consequently, the settlement may have covered an area of approximately 2–2.5 ha.

The Mikniškiai site and its excavation

Archaeological excavation

The settlement was discovered on the constructed pipeline route after mechanically removing the turf and plant soil and surveying the unearthed sections for the pipeline construction. Initially, an area of 580 m^2 was surveyed and four test pits were investigated (*Kvizikevičius 2021*; *Kvizikevičius – Grabovska 2022*). Afterwards, full-scale excavations were carried out on an area of 600 m^2 divided into 17 trenches forming a zone 60 m long and 10 m wide. The archaeological layer was excavated in 15-cm-thick mechanical levels and the exposed surface, excavated by shovels and trowels, was recorded after each level. The sandy soil was sieved through a 5 mm mesh. The fill of sunken features was sieved

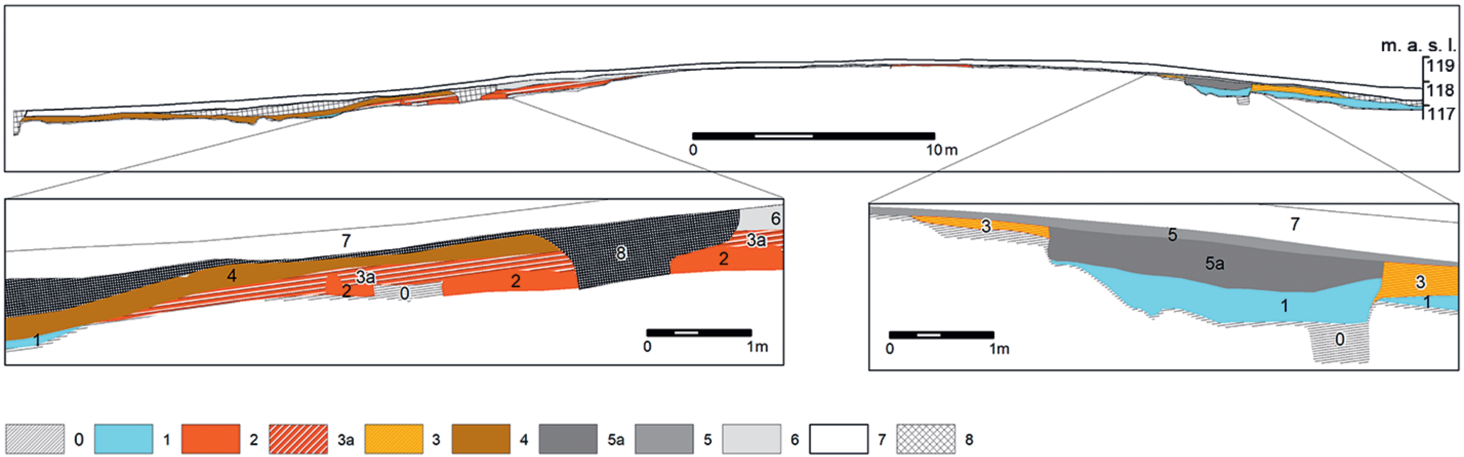

Fig. 3. Mikniškiai settlement, SE profile of the excavated zone: 0 – light greyish/ruddy clayey subsoil; 1 – greyish clay; 2 – cemented limonite; 3 – limonized clay loam; 3a – grey sandy loam with randomly spread iron ore concretions or limonized soil; 4 – peaty layer; 5a – blackish loamy soil with charcoal; 5 – grey loamy soil, mostly with charcoal; 6 – a plough-affected grey loamy soil with sparse charcoal; 7 – ploughsoil; 8 – truncated.

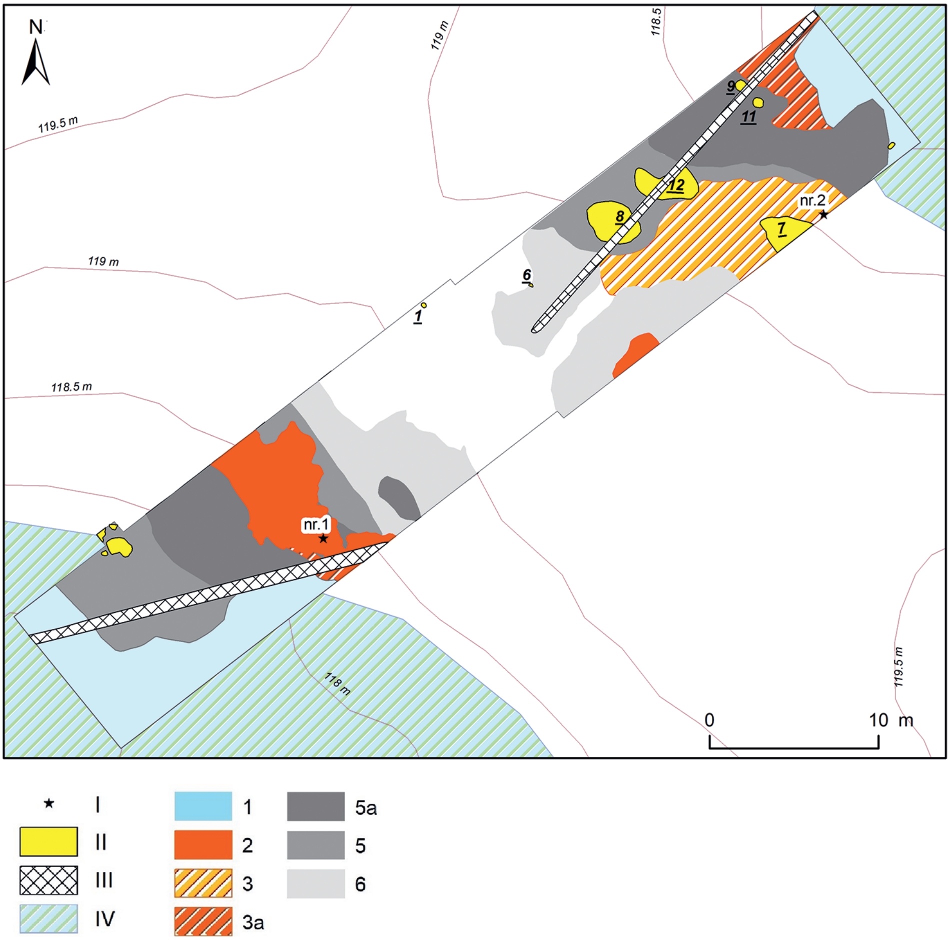

Fig. 4. Excavated area of the Mikniškiai settlement: I – locations of iron ore samples; II – sunken features (only anthropogenic features are numbered); III – drainage; IV – peaty wetlands. Areas of layers and horizons: 1 – greyish clay; 2 – cemented limonite; 3 – limonized clay loam; 3a – grey sandy loam with randomly spread iron ore concretions or limonized soil; 5a – blackish loamy soil with charcoal; 5 – grey loamy soil mostly with charcoal; 6 – a plough-affected grey loamy soil with sparse charcoal.

completely. Very clayey and peaty soils as well as limonized soil deposits were stripped off in thin layers without sieving.

The cross-section of the investigated site

In the central (highest) part of the excavated section (approximately 130 m^2) a clayey sterile soil was reached under the ploughsoil (layer 7) and plough-affected greyish sand containing sparse pieces of charcoal (layer 6) (*Fig. 3*; *Fig. 4*). Under the ploughed soil, the slopes revealed blackish (layer 5a) and greyish (layer 5) loamy soil, mostly with charcoal pieces. The stratigraphic relation of these layers is not clear. Some 5–37 cm large stones were also recorded therein: some of them were randomly scattered, while others formed loose concentrations.

Fig. 5. Continuous cemented limonite in the excavated area (photo by L. Kvizikevičius).

On the SW slope, a blackish loamy layer up to 0.4 m thick with charcoals (layer 5a) stretched throughout an approximately 10-m-long section. Down the slope, it transformed into a thinning greyish layer with charcoals (layer 5). The latter stretched down the depression and failed to reach only the lowest point of the investigated area. Here, a 30-cm-thick peaty layer (layer 4) was located just under the ploughsoil. Below was a c. 5-cm-thick layer of grey clay (layer 1) that was much thicker in some parts. The peaty layer (layer 4) stretched into a segment several dozen metres long in the direction of the rising terrain and marked the bank of a former body of water (*Fig. 2*). At the SE cross-section, another layer – greyish sand with bog ore concretions or traces of the limonized soil – was recorded in an approximately 12-m-long segment (layer 3a). The lower range of the layer 3a segment lay under the peaty layer (layer 4), whereas the upper one was just under the ploughsoil layer (layer 6). Within this intermediate area, an extremely dense limonite horizon with irregular contours covering the clayey subsoil was found in an area of about 40 m^2 (layer 2) (*Fig. 5*).

On the NE slope, layers with charcoal were recorded under the ploughsoil layer: grey sand (layer 5) and a blackish layer (layer 5a) were found slightly down the slope (*Fig. 3*; *Fig. 4*). Below was a 10–30-cm-thick limonized sand (layer 3a) that turned into yellowish limonized loam (layer 3) at the lowest point of the investigated area. Below was an up to 25–35-cm-thick dark-grey clay (layer 1) marking the edge of the depression. Deeper, an unweathered clay was reached in the entire investigated area.

Fig. 6. Sunken feature no. 7 (above) and no. 8 (below) regarded as ore mining pits (photo by L. Kvizikevičius).

Based on the number of finds in different layers (see below), the anthropogenic origin of layers 3a and 5a may be considered. Layers 1–4 represented natural sediments affected by post-depositional processes (soil formation, bioturbation, and erosion). The upper layers (layers 5, 6, and 7) were related to ploughing and erosion.

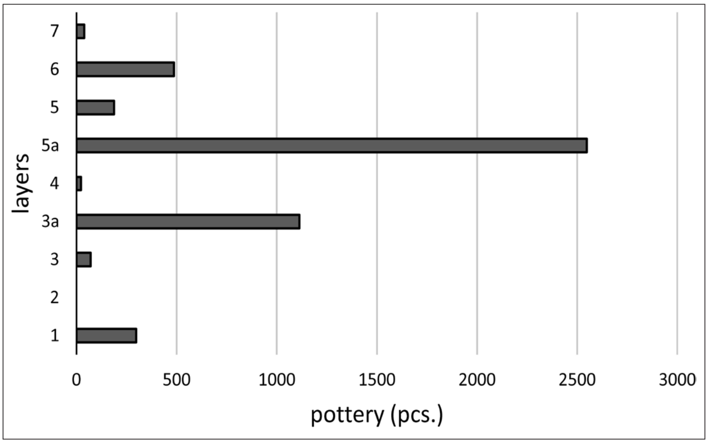

Fig. 7. Quantity of pottery in different layers.

Sunken features

Twelve sunken features were recorded in the investigated area of the Mikniškiai settlement site. Of these, seven may be interpreted as features of anthropogenic origin; some of them were damaged by the drainage ditch route (*Fig. 4*).

Two assumed postholes (features no. 1 and 6) were observed at the subsoil level on the ploughed top of the elevation. These features were 20–25 cm in diameter and 10 cm deep, irregular or close to rectangular in shape, and they were filled with brown loam or black soil without finds. Two other assumed postholes (no. 9 and 11) were identified next to each other in the limonized layer (layer 3a) on the NE slope of the elevation. Both features were approximately 60 cm wide circular pits with concave bottoms and steep sides; they were filled with black soil. Sunken feature no. 9 was 80 cm deep and contained five sherds of handmade pottery; sunken feature no. 11 was 30 cm deep and contained no finds.

In addition to the postholes, three features (no. 7, 8, 12) are classified as ore mining pits. The pits were found under the greyish layer with charcoals (layer 5) and they penetrated the limonized layers (layers 3 and 3a) all the way down to the clayey sterile soil. Pit no. 7 was irregular in shape and only a part of it fell into the uncovered area; it was 2.82 × 1.25 m and 60–87 cm deep. Its slopes were asymmetric and the bottom was uneven. The pit was filled with blackish soil (layer 5a) turning into grey clay at the bottom (*Fig. 6*). The pit contained 65 sherds of handmade pottery and nodules of the iron ore. Pit no. 8 was of an irregular shape, 3.25 × 2.5 m and 28 cm deep. At its bottom was a strip of solid cemented limonite (*Fig. 6*). The pit was filled with greyish sand (layer 5) and contained 15 sherds of handmade pottery. Pit no. 12 was irregular in shape, 3.68 × 1.35 m and 30 cm deep; it contained no artefacts.

Artefacts

Archaeological finds were concentrated in two 90–110 m^2 areas on the NE and SW slopes of the elevation. There were considerably fewer finds at the SW and NE ends of the investigated area and practically none were made in the central part. In total, 4 911 artefacts were recorded (*Kvizikevičius – Grabovska 2022*), nearly all of them sherds of handmade pottery (4 851). Most of the sherds were found in layer 5a (54 %) and layer 3a (23 %) (*Fig. 7*). The layers of natural origin, namely layers 1, 3, and 4, contained 6 %,

Fig. 8. Potsherds from the Mikniškiai settlement site: 1 – rim of kitchen vessel with a rough surface; 2 – rim of table vessel with smoothed surface (photo by A. Simniškytė).

1.5 % and 0.5 % of the sherds, respectively. Layer 2 contained no sherds. Artefacts found in the upper layers (layers 5, 6, and 7) made up 4 %, 10 % and 0.8 %, respectively, and were relevant to ploughing and erosion. Sherds found in different layers belonged basically to the same complex of pottery with rough and plain or smoothened surface (*Fig. 8*) dating back to the turn of the era. The collection of pottery found at Mikniškiai was typical to the Užnemunė (Trans-Nemunas) region and featured many similarities to the pottery of the sites found in the Suwałki and Masuria regions in modern Poland (*Szymanski 2003*; *Karczewska 2009*). In particular, there were many links to the pottery of the Wyszembork type typical for the early phase of the Bogaczowo culture (*Szymanski 2003*).

Five flint artefacts were recorded in the same layers that contained most of the potsherds. Based on the knapping technique, one blade was dated to the Upper Palaeolithic – Early Mesolithic, another artefact of an unspecified purpose most probably belonged to the Mesolithic, whereas other flint items were broadly dated to the Stone Age – Bronze Age range. Metal artefacts (37 pieces) were represented by nails of the modern era (23 pcs.) and fragments of unidentified artefacts. No metal artefacts dating back to the Iron Age were discovered. Almost all of these finds came from the ploughed upper layers (layers 6 and 7). Stone tools were not numerous. One sandstone (?) whetstone was found in the plough-affected layer (layer 6). Three granite rocks of an irregular form (two of them bearing traces of grinding) could have served as the base of a hand crusher. All of them were discovered in the layer of grey clay (layer l).

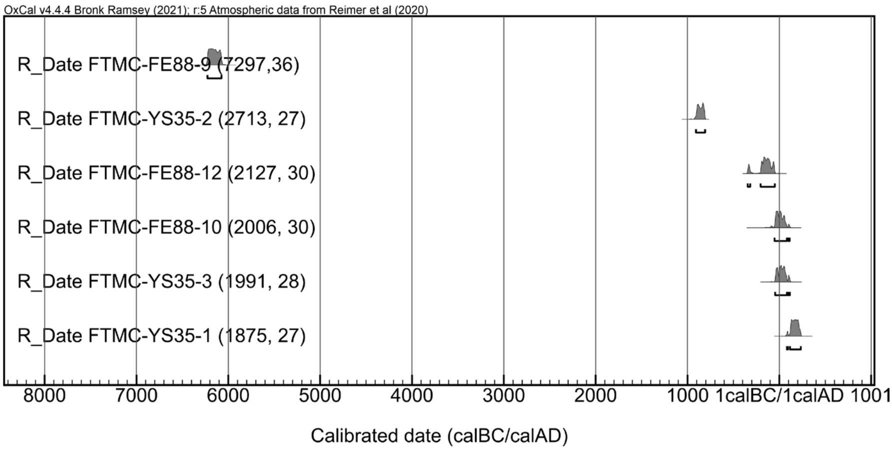

Fig. 9. A calibration plot of AMS radiocarbon dates obtained from charcoal samples from Mikniškiai. The resulting dates were calibrated with OxCal v4.4.4 software and the IntCal20 atmospheric curve (*Bronk Ramsey 2009*; *Reimer et al. 2020*). The calibrated dates are presented at 95.4% probability.

The typology of the finds allowed us to identify two main phases in the occupation of the Mikniškiai settlement, the first taking place during the Stone and Bronze Age, the second one at the beginning of the 1st millennium AD. The chronology of these occupation phases was refined by radiocarbon dating (*Fig. 9*). The dating was carried out at the Mass Spectrometry Laboratory of the Centre for Physical and Technological Sciences in Vilnius. Standard acid-alkali-acid (AAA) pre-treatment was used for all samples. Six dates were obtained from charcoal samples. Two samples were taken from feature no. 7 and two samples each were also taken from both layers 3a and 5. The acquired results indicated that the limonized layer (layer 3a) was the surface exposed to human activities from the Stone Age to the Iron Age. The layers with charcoals (layers 5a and 5) formed on top of that. Although the stratigraphy implies certain changes in the sedimentary settings, no differences in the typology of the pottery were observed. It is evident that these strata were very close chronologically and, speaking of the Iron Age, they cover the span from the late pre-Roman Iron Age to the Early Roman Iron Age.

Materials and methods

Bog iron ore samples

Bog iron ore is a sedimentary rock of the Holocene period consisting of iron oxides and oxide-hydroxide minerals collectively known as *limonite* ($Fe_2O_3 \cdot nH_2O$). Quartz, silicates, phosphates, and other minerals also tend to be mixed therein. Bog ore forms when parent rock minerals are affected by atmospheric humidity and the hydrosphere. When penetrating cracks and pores in rocks, groundwater dissolves various minerals, including those containing Fe. This process accelerates at pH<7; it is stimulated by carbon dioxide in the water, which increases the solubility of iron compounds (Fe^{+2}) under anaerobic conditions, as well as by various micro-organisms. When such a solution reaches the surface, carbon dioxide evaporates and iron hydro-oxides precipitate as sediments. Limonite ore (ancient Greek *leimon* – wetland) is found in wet meadows, in the areas of former swamps, peat fields, watered lowlands, and springy banks of rivers and lakes. Limonite-rich layers of sand, sandy loam or siltstone are usually rather thin, ranging from centimetres to several dozen centimetres, and their layers tend to lie shallow, only 0.3–0.8 m deep under the turf or topsoil (*Linčius 1972*, 117; *Anteins 1976*, 70–72; *Malinauskas – Linčius 1999*, 112–113, *Navasaitis 2003*, 17, 20; *Peets 2003*, 31; *Kulbickas 2006*). Bog iron ores are divided into three different macromorphological types: (a) a soft unstable form, (b) randomly spread, nest-like distributed concretions, blocks, or nodules, and (c) bog iron fragments and continuous cemented horizons. These types often represent different development phases, with bog iron soils being the first phase, cemented bog iron horizons the very last. Variable contents of Fe_2O_3 are to be expected in different types, with <25 mass% Fe_2O_3 for the initial and >25 mass% Fe_2O_3 for the developed bog iron ores (*Rzepa et al. 2016*; *Thelemann et al. 2017*, 6; *Brenko et al. 2021*).

At Mikniškiai, bog iron ore sediments formed on the slopes of the elevation, where it was surrounded by bodies of water or swamps. Groundwater used to rise seasonally and protrude to the surface. When the iron-containing groundwater subsided, the iron precipitated into sediment due to contact with oxygen. As the process repeated itself year after year, deposits rich with bog iron ore formed. At the SW foot of the elevation, hard limonite (layer 2) was recorded in an area of 40 m^2, from which sample no. 1 was collected (*Fig. 4*; *Fig. 5*). On the opposite slope, sample no. 2 in the form of the iron ore concretion was collected from the yellow limonized sandy loam (layer 3).

Chemical composition

For element analysis of the bog iron ore samples, an X-ray spectrometer with a wavelength dispersive detector Axios mAX (PANalytical Netherlands. 2010) was used. Five grams of each milled sample were mixed with 1 g Hoechst wax C micro-powder and compressed into tablets. The acquired quantities of the elements (Si, Al, Fe, Mg, Ca, Na, K, Mn, P, Ti, Ba) were converted to oxides and normalized including loss on ignition (LOI). The detected iron was recalculated stoichiometrically as Fe_2O_3.

Loss on ignition

The loss on ignition (LOI) in the temperature ranges 105–500 °C and 500–1000 °C was determined. Five grams of sample were placed in a heated and weighed ceramic crucible and subsequently heated for 16 h at 105 °C and for 2 h each at 500 °C and 1000 °C. After each heating procedure, the sample was cooled down in the oven to 100 °C. Following this, the sample was capped and placed in a desiccator until cooled to room temperature. After that, it was weighed and the loss on ignition data was calculated.

Mineralogical composition

The mineralogical composition of samples was determined using powder X-ray diffraction (XRD). XRD measurements were carried out using a SmartLab (Rigaku) X-ray diffractometer equipped with a 9kW rotating Cu anode X-ray tube (l = 0.154183 nm). The

XRD patterns were measured using the Bragg-Brentano method and a DtexUltra linear detector. Discrimination of the diffracted beam was applied to prevent background from fluorescence. The phase identification was performed using PDXL2 software and the ICDD database PDF 4+ (2022 release).

Microstructure

To identify the microstructure, cross-sections of the bog iron ore samples were prepared using the standard mounting, grinding and polishing procedure with Tegramin-25 equipment (Struers). The microstructures of the samples were investigated using an FEI Helios Nanolab 650 scanning electron microscope (SEM) in the secondary electron (SE) imaging mode.

Results

The iron content was comparatively low, 21–25 % Fe, equivalent to 30–35 % Fe_2O_3. The rest was mostly SiO_2 (41–46 %). The samples had a slightly different composition: sample no. 1 contained slightly more MgO, whereas sample no. 2 had slightly more P_2O_5 and MnO (*Tab. 1*).

Sample no.	(Fe)	Fe_2O_3	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	MnO	TiO_2	BaO	LOI (105–1000 °C)	Σ
1	20.88	29.85	45.84	6.09	3.67	2.06	2.18	0.37	2.60	0.38	0.31	0.10	6.55	100
2	24.62	35.20	41.35	5.28	3.40	1.38	1.85	0.34	3.62	1.36	0.23	0.25	5.74	100

Tab. 1. Chemical composition of the bog iron ore (wt.%).

The XRD measurements of the samples revealed that the main iron-containing minerals were goethite and probably ferrihydrite (*Fig. 10*). Besides that, the samples also contained carbonates (dolomite and calcite), as well as minerals from the feldspar group (albite, microcline, and orthoclase), chlorite group (clinochlore), and quartz. Sample no. 1 contained more dolomite (this was also related to a higher amount of MgO) and slightly more calcite.

LOI at temperatures of 105–500 °C revealed that organic matter (OM) burned and most of the iron oxides-hydroxides decomposed (*Tab. 2*). Sample no. 2 had slightly more iron and therefore naturally had higher values. LOI (500–1000 °C) revealed that carbonates and clay minerals decomposed and that iron compounds could have finished dehydrating (*Rzepa et al. 2016*). Sample no. 1 contained more carbonates, so LOI (500–1000 °C) also had higher values.

Sample no.	105 °C /16h	500 °C /2h	1000 °C /2h	Σ
1	3.15	2.90	3.65	9.70
2	4.34	3.45	2.29	10.07

Tab. 2. Loss on ignition results for the bog iron ore samples (wt.%).

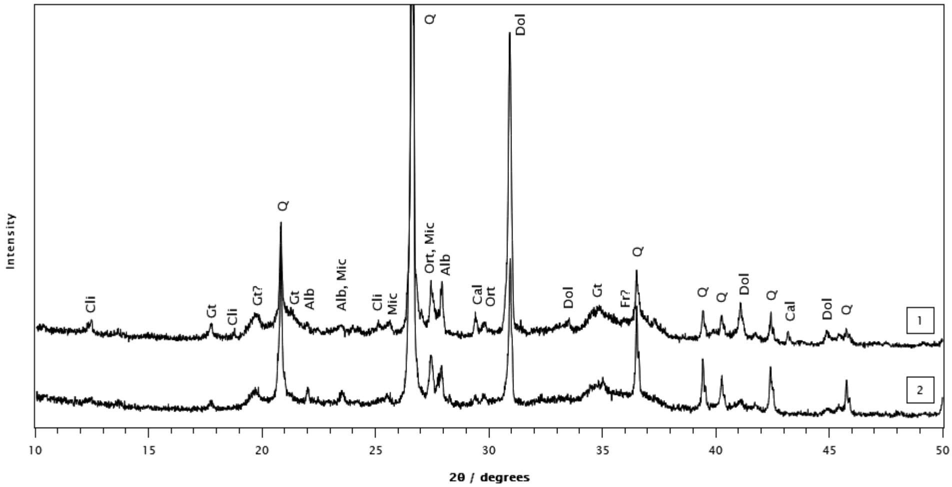

Fig. 10. XRD patterns of ore samples. Explanations: Alb – albite; Cal – calcite; Cli – Clinochlore; Dol – dolomite; Fh – ferrihydrite; Gt – goethite; Mic – microcline; Ort – orthoclase; Q – quartz.

The investigation of the microstructure of both samples also proved that they were natural compounds: unevenly distributed mineral (mostly quartz) grains of various sizes were bound together by an iron-containing mass (*Fig. 11*). The microstructure did not resemble iron slag or roasted ore.

Therefore, we assume that the investigated samples represent natural bog iron ore, which was not rich enough for iron bloomery smelting. Minor differences in the chemical and mineral composition of the samples revealed that the composition of the deposit was not the same everywhere, as is often the case.

Discussion

Regarding the Mikniškiai settlement structure, it must be noted that no remains of residential buildings or features (hearths or contours of building walls) were identified. Most probably, this was because the excavations took place within a narrow 'land-bridge' connecting areas more suitable for dwelling. As the traces of several postholes of an unidentified period imply, there could have been some buildings at the centre of the elevation, but long years of ploughing and erosion have erased them. Two zones of archaeological sediments detected in the excavated area and containing almost exclusively pottery did not correlate with any of the recorded sunken features; only one of the zones on the NE slope of the elevation bordered with a construction of an unidentified purpose represented by two posts.

On the NE slope, further from the epicentre of the artefact concentration, three pits (no. 7, 8, 12) dug all the way through the limonized layer were identified. Judging from their position and form, they could have been dug during prospection for bog iron ore. A strip of not fully extracted (?) limonite was recorded at the bottom of one of these features. Later, the pits could have been used for waste disposal, filled and levelled manually

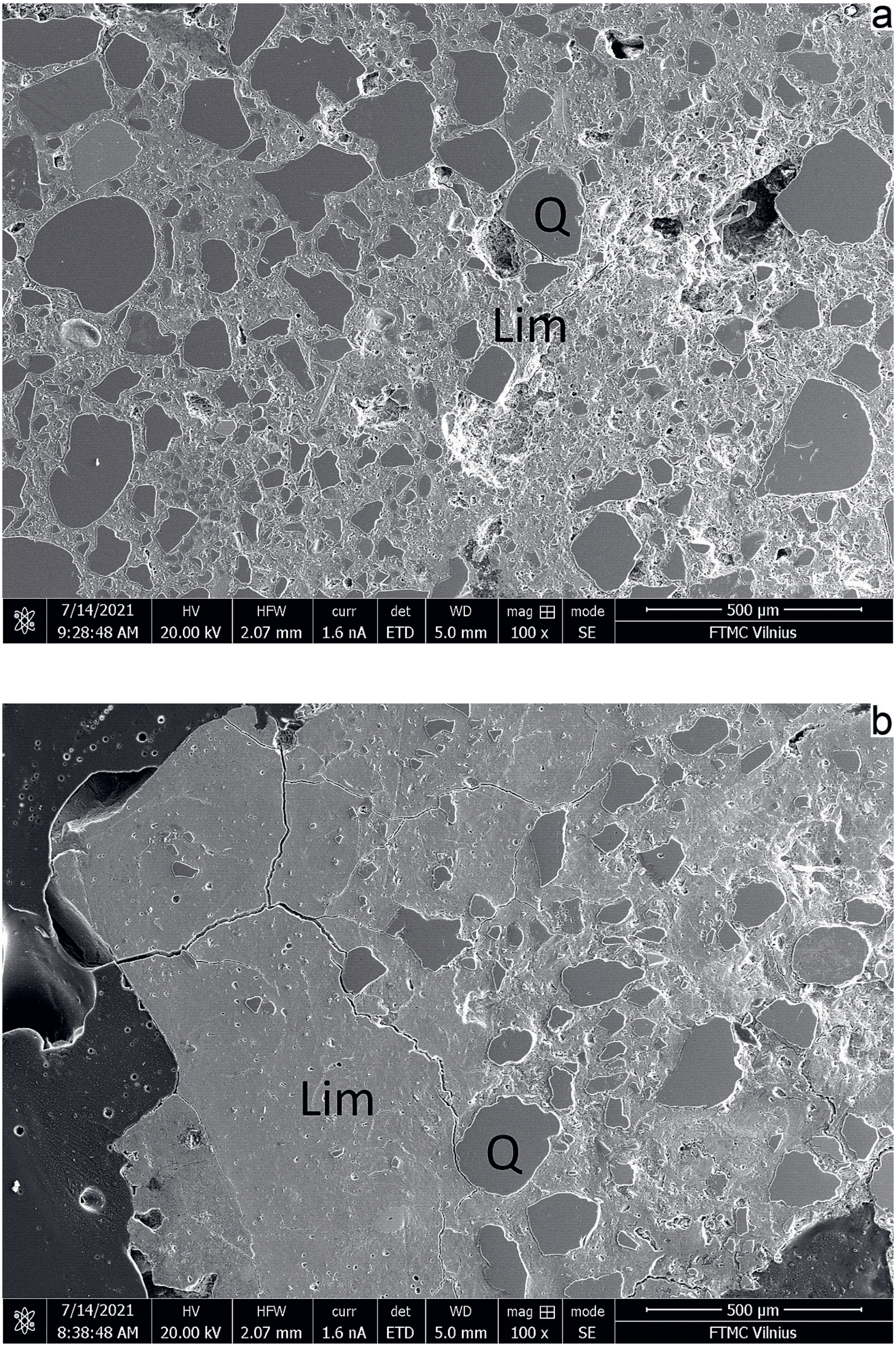

Fig. 11. Structure of ore sample no. 1 (a) and sample no. 2 (b) (SEM). Q – quartz; Lim – limonite matrix.

or naturally. To date, more thorough data on the bog iron ore mining pits in Lithuania have been available only from the Lieporiai settlement, where 18 such pits were found. The pits were discovered in a sandy lowland where iron ore clusters were present. They were of an irregular shape, 0.6 × 0.8 to 2 × 2.5 m and 0.1–0.6 m deep, with uneven bottoms (*Salatkienė 2009*, 57–61).

No pits have been discovered on the SW slope of the elevation. However, the dense limonite horizon that was found there indicates the potential location of the ore source. Also, the irregular contours of this horizon imply that the ore could have been mined.

Areas of iron ore deposits tended to feature infertile soil and poor vegetation that animals did not eat (*Lyngstrøm 2011*, 414). Therefore, it was rational to use such areas for residential purposes, leaving more fertile areas for agricultural activities or other forms of land use. Nevertheless, it was not convenient to live in the quarry area during the mining of iron ore. Dwelling in the quarry area, expanding the residential area (if previously living nearby) or turning it into a sanitary zone was possible only after the ore deposit was exhausted or the mining activities terminated. At the aforementioned Lieporiai settlement, it has been argued that a small iron ore deposit was initially mined and iron was smelted on the site. People built houses and started living there only later after the ore was exhausted (*Salatkienė 2008*, 67; *2009*, 57–71).

At the Mikniškiai site, several chronologically close occupation phases (disregarding the Stone and Bronze Age relics) could also have taken place. The mining and occupation/ waste disposal stages merged into one segment in the archaeological record. The mining episode was typologically indistinct, and it is assumed that ore mining could have been attempted here at some moment in the late pre-Roman Iron Age. Mineral iron ore deposits could have been the main resource that attracted people to these areas. After terminating the ore exploration, people did not abandon these areas and settled there for a longer period. The settlement existed between the mid-1st century BC to the early 3rd century AD.

A very significant attribute of the Mikniškiai site material is that not a single piece of iron slag has been found here. During the excavation, 14 finds were originally labelled as pieces of iron slag but a later review of the finds revealed that some of them were unidentifiable iron oxides, whereas others were hollow cylindrical colloidal compounds, namely mineral soil cemented with limonite grains that used to cover decayed plant roots. This is very interesting considering that ore-rich surroundings of Iron Age settlements are often regarded as an indication of iron bloomery smelting. Nevertheless, not all the bog iron ore was suitable for bloomery smelting. *Pleiner* (*2000*, 87) established an empirical boundary value for iron bloomery at 55–60 % Fe (equivalent to 79–86 % Fe_2O_3). Experimental direct iron smelting undertaken in the last decades has shown that bog iron ores with much lower iron contents of up to 34 % Fe (49 % Fe_2O_3) or even lower, if in combination with higher graded ores, could have been used for bloomery iron production (*Brenko et al. 2021*, 9). According to various researchers, the minimal iron concentration in the limonite ore used for iron bloomery smelting was supposed to be 30 % Fe (43 % Fe_2O_3) (*Endzinas 1969*, 95), 35–42 % Fe (50–60 % Fe_2O_3) (*Stankus 2001*, 171), 35–47 % Fe (50–67% Fe_2O_3) (*Navasaitis 2003*, 20), 35–40 % Fe (50–57 % Fe_2O_3) (*Anteins 1976*, 71) or 40–60 % Fe (57–86 % Fe_2O_3) (*Orzechowski – Przychodni 2014*, 252).

The iron concentration reached only 21–25 % (30–35 % Fe_2O_3) in Mikniškiai samples. Limonite ores used for iron smelting usually contain approximately 15–23 % SiO_2. At Mikniškiai, the SiO_2 content was as high as 41–46 %. The ratio of iron and silicon oxides (1:1.2–1.5) was quite far from the optimum of 2:1 (*Navasaitis 2003*, 22, 31). When a certain amount of quartz reacts with iron oxide, fayalite is produced (Fe_2SiO_4), which is necessary for the iron smelting process. However, when there is too much quartz, iron may be waste in the formation of excess fayalite. The ore at Mikniškiai had too many other unwanted impurities as well. The amounts of Al_2O_3, K_2O and MgO were several times higher than in ores from other sites (e.g., *Navasaitis 2003*, 22, Table 2.3). This is typical for deposits where the cementation process and substitution of the total alumosilicate content with a Fe-enriched matrix has not yet progressed (*Brenko et al. 2021*, 6). The mineral raw material of such a composition is not suitable for iron smelting. It could have been used only in the case of eliminating the excessive amount of gangue material. To reduce the gangue and increase the relative content of iron in the ore, it had to go through a series of preparatory procedures: drying, sluicing, roasting, and crushing, which helped to get rid of sand, other minerals, and organic matter. This way, the iron content could have been increased considerably, reducing the amount of impurities. For instance, after the roasting of the ore from the village of Rudnia, the Fe_2O_3 concentration increased from 67.47 to 78.1 % (see *Navasaitis 2003*, 22, Table 2.3 and 2.5). The roasted ore of the Lieporiai ancient settlement featured similar values (*Navasaitis 2003*, Table 2.5). It should be noted that archaeological features related to ore exploitation and enrichment were found at this site. There were ore mining pits, sluicing facilities, shallow ore roasting pits, stone ore crushers, as well as finds related to iron smelting itself (18 bloomeries and over 400 kg of iron slag).

At the Mikniškiai settlement, ore exploration could be attributed to three pits on the NE slope, as well as to the limonite continuous horizon. However, no traces of further ore processing have been identified. Iron oxides/hydroxides (goethite and probably ferrihydrite) identified in both samples collected from Mikniškiai are the sole Fe-bearing minerals. The samples contained no other minerals (e.g. hematite, maghemite, and especially magnetite) that could form when heating the material to a temperature of >300 °C, for instance, in a bonfire used for roasting purposes. Also, their microstructure was not like that of roasted ore or slag. Due to pre-processing (washing) and roasting, LOI in pretreated ores are lower in comparison to unpretreated ores (*Rzepa et al. 2016*; *Brenko et al. 2021*, 9). Organic matter is combusted when exposed to temperatures of 500 °C, iron oxides and hydroxides also decompose and, at higher temperatures, clay and other minerals decompose as well. The ore of Mikniškiai featured rather high heating losses (9.9 %) and high concentrations of other minerals. This indicates that the samples were not pretreated thermally or otherwise pre-processed. Therefore, we argue that these were natural formations of hydrated limonite.

No traces of ore roasting pits or bonfires were found during the archaeological excavations, nor were any traces of iron smelting. It cannot be ruled out that a smelting workshop was located outside the surveyed area, because the total area of the settlement could have covered several hectares. However, slag pieces are not only scattered in the vicinity of the iron smelting bloomeries, but are also abundantly dispersed throughout the whole settlement area. Such patterns observed at other settlements suggest that if a sufficiently large area of the Mikniškiai settlement failed to reveal a single slag find, it is likely that its inhabitants did not engage in iron smelting and exploitation of the limonite deposit was never attempted. The only question is whether that was caused by the unsatisfactory quality of the ore or was the result of other factors.

Iron metallurgy requires not only natural resources; cultural and technological conditions must also be met. Smelting iron from local bog ore cannot be considered in the entire Baltic region before the beginning of the Roman Period (*Navasaitis 2003*; *Peets 2003*, 47–48; *Salatkienė 2008*; *2009*, 124; *Rundberget et al. 2018*). The Early Iron Age in Lithuania (400–1 BC) is perceived as a transitional period during which iron artefacts gradually became available, although no archaeological traces of iron bloomery smelting have been discovered (or they are very questionable). The latest segment of the late pre-Roman Period could have been a time of experimenting and finding the required and appropriate resources, as well as adopting and mastering iron smelting skills. The knowledge of iron and incentives to indigenous metallurgy reached South Lithuania from the heart of Central Europe through the lands of present-day Poland. Here, the beginnings of indigenous metallurgy, as a separate and fully developed branch of economy, could be associated with the Przeworsk culture (the turn of the 2nd century BC–mid-5th century AD). Beside the main centres of the specialized metallurgy in the Swietokrzyskie Mountains, Masovia, and Silesia (*Orzechowski 2018*; *2020*), smelting was performed in some larger and several smaller regions, both in the Przeworsk culture and outside of its distribution area. Among them, there was a small iron smelting complex associated with the Bogaczewo culture in Masuria (*Orzechowski 2018*, 392; *Szymański – Orzechowski 2021*), of which the pottery of the Mikniškiai settlement, as well as many other Southern Lithuanian sites (*Grižas – Bitner-Wróblewska 2007*) shares cultural attributes. Also, several burial sites near Mikniškiai (namely Delnica and Rudamina) were apparently influenced by the West Balts that disseminated from the S–SW, particularly from the Suwałki-Augustów region since the beginning of 1st millennium AD (*Banytė 2007*).

The aforementioned evidence indicates that communities of SW Lithuania were to have met the cultural and technological preconditions for iron metallurgy. The fact that no iron slag was found at the Mikniškiai settlement may be interpreted in a variety of ways. It cannot be ruled out that the local ore was not meant to be smelted and the excavated sunken features were misinterpreted as ore exploration pits. Nevertheless, in such case the acquired data also raises questions regarding the start of iron bloomery smelting: could iron smelting in Lithuania become widespread later than assumed thus far? In this respect, it is also notable that no slag has been found in the nearest well-researched Roman Iron Age settlements in Užnemunė (Trans-Nemunas region): for instance, Dzūkai (excavated area of 920 m^2) (*Kvizikevičius – Čepelytė 2022*), Zubriai (excavated area of over 5349 m^2) (*Baubonis et al. 2012*; *2013*) or Birsčiai (excavated area of 172 m^2) (*Piličiauskienė et al. 2022*), or the Skudeniai settlement in Eastern Lithuania (excavated area of 431 m^2) (*Vengalis et al. 2022*).

Conclusion

Ore-rich sediments in the vicinity of Iron Age settlements are often interpreted by default as evidence of iron bloomery smelting. However, proper research of bog iron ore evidence in the archaeological context has not yet been performed and it is not clear how such evidence should be interpreted – as natural formations untouched by humans or potentially exploited ore mines.

Excavations at the Mikniškiai settlement site opened new perspectives for the investigation of the early iron smelting. Sunken features and a continuous limonite deposit were initially interpreted as relics of shallow opencast mining of the bog ore, which were later turned into a zone for household waste disposal. These episodes were claimed to be not simultaneous, but chronologically rather close to each other, covering the period from approximately the mid-1st century BC to the early 3rd century AD. It was assumed that the mineral iron ore deposits might have been the main resource that attracted people to these areas, especially considering that this part of Lithuania maintained close relations with the Bogaczewo culture, from which and through which knowledge about iron metallurgy spread.

Nevertheless, the archaeometallurgical investigation has revealed that the tested samples represented natural bog ore that was not rich enough for iron smelting and that there were no attempts to increase its iron contents by proper pre-processing. The archaeological survey revealed no traces of ore roasting pits or bonfires and not a single piece of iron slag was found here. This can be interpreted in different ways: the quality of the ore was found unsatisfactory by the smelters or the ore-rich environment and the excavated pits were not related to iron production, as the iron smelting know-how might not have reached the community that inhabited the settlement. However, even if this was the case, it raises questions concerning the beginning of iron metallurgy. Could it be that iron smelting in Lithuania became widespread later than assumed until recently? These questions cannot yet be answered reasonably since they require more evidence and a specialised study regarding the temporal and spatial organization of different ironworking stages and dedicated spaces, like extraction and preparation of ore, fuel and clay, the actual iron smelting, smithing, etc. A comparative analysis of the material from other settlements discovered in similar ore-rich environments must now be addressed.

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