

ORIGINAL ARTICLE

First results of metallographic analysis and absolute dating of iron finds from Nowe Brzesko, site 26, Proszowice District

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Abstract

This paper presents the results of analyses of metal artefacts discovered at a multiphase site in Nowe Brzesko, Małopolskie Voivodeship, Poland. Fieldwalking prospection led to the discovery of steely iron objects, ploughed out on the site among other numerous archeological finds. A prospecting sondage yielded more corroded metal artefacts, identified mainly as agricultural tools (e.g. sickles, scythes). These chronologically nondistinctive artefacts could be assigned to the third to fourth century AD using radiocarbon dating, complemented by metallurgical analysis. The results open up new research perspectives on the economy and society of the Przeworsk culture in the Late Roman period.

KEYWORDS

Archaeometallurgy, iron tools, Przeworsk culture, radiocarbon dating, Roman period

INTRODUCTION

This article deals with a collection of iron artefacts discovered in 2018 during a field survey of one of the largest Przeworsk culture settlement sites in southern Poland dated to the Roman period. It is located near Nowe Brzesko (50° 7' 11.91" N; 20° 22' 7.18" E), Proszowice District (Figure 1) and covers the edge of a steep Vistula terrace. The spread of surface material extends over an area of ca. 25 ha.

The site was originally identified by Władysław Morawski as a complex of several separate sites during works carried out under the Polish Archaeological Record project in 1983. At the beginning of the 21st century it became a subject of intensive and illegal activities of metal detectorists, indicating the existence of a site of high importance for the archaeology of the region. Due to the threat posed

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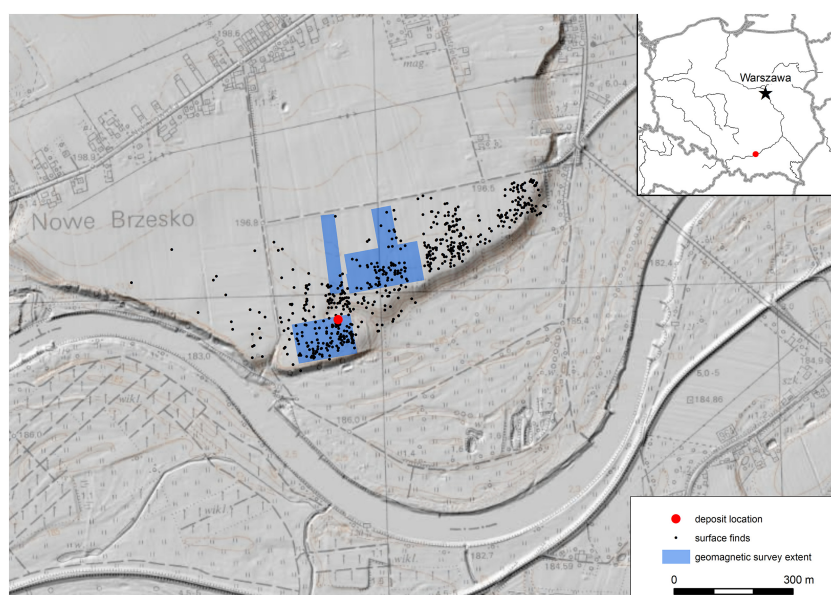


FIGURE 1 Location of the site 26 in Nowe Brzesko, Małopolskie Voivodeship, Poland. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com/doi/10.1111/jar.12358)]

both by the presence of the detectorists and intensive agricultural work, field-walking surveys of the site have been conducted by one of us (JB) since 2012. The field work conducted after 2012 included surface and metal detector surveys, and aerial photography using a drone. During this fieldwork, a large number of pottery sherds and metal objects were collected, which gave a first indication of the chronology of the whole site (Bodzek & Bulas, 2012; Bulas, 2015; Bulas & Kasiński, 2018; Bulas & Przybyła, 2018, 2021). In addition, a geomagnetic prospection was carried out at about 20% of this extensive site, giving important insights into the structure of the site and confirming a huge number of archaeological features. Among them are pits, pit houses, some linear structures and possibly even burned structures (Bulas & Przybyła, 2021). However, it did not confirm the presence of further iron artefacts at this location.

The collected finds indicate a sequence of occupational phases, dating from the Neolithic to the Middle Ages. Most of the finds are connected with the Przeworsk culture. It is one of the largest and long-lasting cultural units that developed in La Tène period, lasting through the Roman period up to Early Migration period (second century BCE–fifth century AD) in the area of present-day Poland (Andrzejowski, 2010).

The collection of metal artefacts from Nowe Brzesko

In spring 2018, a puzzling collection of iron objects was encountered during methodological fieldwalking survey periodically conducted at the site (Figure 1). The objects were scattered over a small area in the topsoil (about 20 cm) and were most likely brought to the surface during ploughing (Figure 2). An assemblage of approximately 50 corroded iron objects was recovered from the topsoil (Bulas & Kasiński, 2018; Bulas & Przybyła, 2018, 2021).

The excavated iron objects comprise of a variety of tools such as agricultural tools (sickles, scythes), carpentry and smithing tools (adze and tongs, respectively), objects with a multifunctional or general use (e.g., bucket handle, chain link, door bolt—sometimes connected also with the construction of carts), and a number of objects of undefined purpose. Notable among them is a massive iron bar with volutes (Figure 3, No 1).



FIGURE 2 Ploughed out iron objects discovered during the field walking survey. [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com/doi/10.1111/jarcm.12858)]

Although the precise nature of the assemblage of artefacts discovered at Nowe Brzesko is not certain, it can be assumed that they form part of a single deposit, because the artefacts were discovered within a 1.5 m radius and the assemblage composition is analogous to that of other deposits from the Late Roman and Medieval periods (Poleski & Rodak, 2001, 49–74; Rodzińska-Nowak, 2012, 83–98). However, the lack of a clear context made it impossible to determine the intention behind their deposition. One may speculate that it could have been a collection of objects in use, representing farm equipment, or it could have been iron scrap intended for secondary processing, or it could have been an intentionally deposited collection of a hoarding or symbolic nature.

The design or shape of these objects does not answer one of the basic questions, namely the chronology of the collection. As utilitarian objects, their appearance is basically dictated by their purpose and, consequently, has varied little over time. As surface finds without a clear archaeological context, they cannot be dated by dating the context itself. Moreover, it should be emphasized that similar objects and deposits dated to prehistoric periods are known from the Vistula basin and that there was a village named Nenkanowice at this place in the 19th and perhaps also until the beginning of the 20th century. Considering the circumstances of the discovery, as well as the large assumed chronological range of pottery and metal finds at the site, there is a need to specify the chronology of the discovered iron objects.

A potential suitable tool is the radiocarbon dating of the carbon trapped within the iron lattice of the objects, originating from contemporaneous organic material (e.g. wood, charcoal) used during their manufacture (Van der Merwe & Stuiver, 1968). Metallurgical analyses are conducted to validate the origin of the carbon being dated and, in addition, to allow first, at this stage rather preliminary, assessments regarding their manufacture and relation to comparable objects.

MATERIALS AND METHODS

Four different iron objects were analyzed. The first, whose function is unknown, is a massive iron object with six coils (Object 55 – KIA-54786), measuring 376×101 mm (Figure 3, No 1). The main part of the object, to which the coils are attached, is rectangular in cross-section. The object is fully preserved and is not broken at any part. Four of the coils are corded in the manner similar to the many iron utensils known from the La Tène period and Roman period (Feugère, 2002; Pieta, 2010). A cross-section of the object was sampled at the (left) side (see Figure 3, No 1) for metallographic and isotopic analysis. The second is a half-sickle fragment (Object 47 – KIA-54785), measuring 200×35 mm. A cross-section of the object was sampled at the (upper) part (Figure 3, No 2). The third analyzed fragment is a fragment of a small bent object, possibly a chain link (Object 23 – KIA-54783), measuring 54×42 mm (Figure 3,

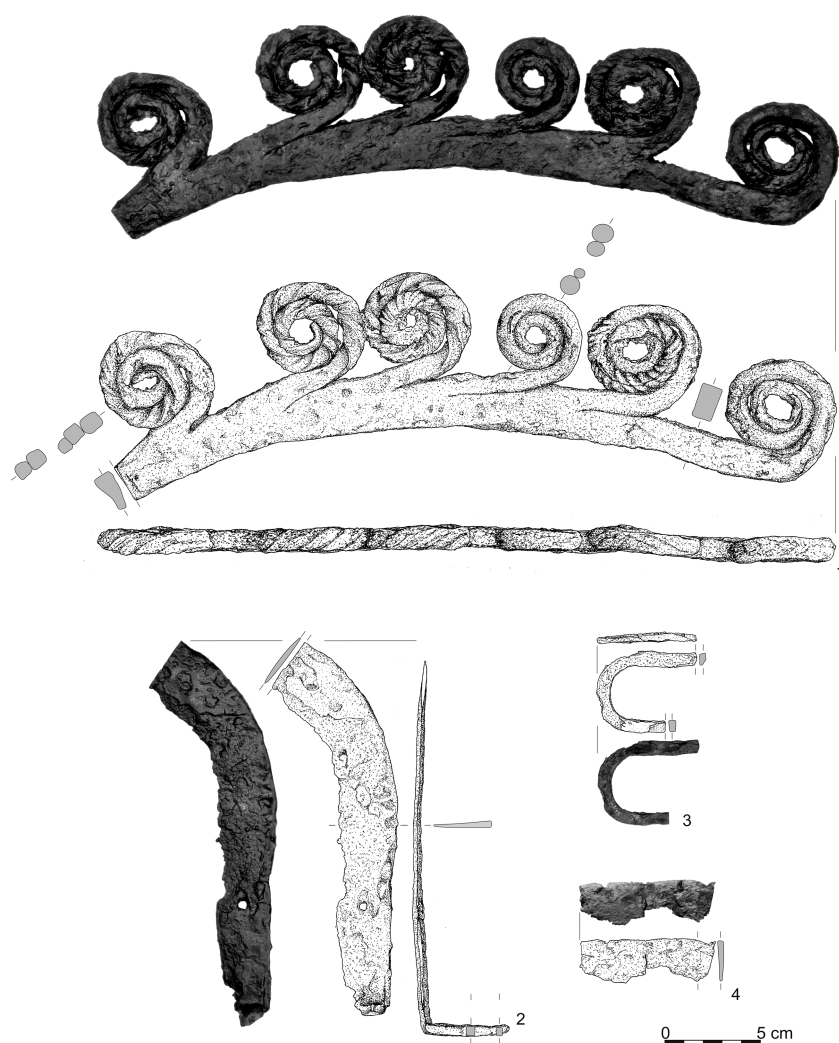


FIGURE 3 Selection of metal finds from the deposit. (1) object 55-KIA54786; (2) object 47-KIA54785; (3) object 22 - KIA 54783; (4) object 25 - KIA 54784.

No 3), and the last is a flat ribbon-like object (Object 25 - KIA-54784) measuring 71×24 mm (Figure 3, No 4). Cross-sections were taken from the (right) side of Object 22 and 25, respectively.

The iron pieces were further sampled with a Dremel™ metal compound cutting disc (corundum and plastic fibers) for subsequent metallographic inspection and radiocarbon analysis in one or two cross-sections. The cutting surfaces as well as the circumference were ground with a corundum grinding tool to completely remove the visible corrosion layer.

Sample preparation for metallographic analysis

Metallographic analysis of Object 25 – KIA-54784, Object 47 – KIA-54785, and Object 55 – KIA-54786 was started first by one of us (MH) and was later completed by Dr. inż. Suliga (AGH Kraków), including also Object 22 – KIA-54783. Samples were prepared as follows:

- After cutting a cross-sections of each object, the material was embedded in plastic (StruersTM phenol resin with carbon filler for electric conductivity; 180°C, 290 bar) for subsequent scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX)
- Grinding with 320 standard silicon carbide grinding paper
- Polishing with
 - 9 μm diamond suspension (MasterMetTM)
 - 3 μm diamond suspension (MasterMetTM 2)
 - 0.05 μm Al_2O_3 suspension (MasterPrepTM)
- Etching with Nital (3% nitric acid $\{\text{HNO}_3\}$ + alcohol)
- Microscopic inspection of microstructure with Leica Reichert MeF3A
- Samples Object 25 – KIA-54784, Object 47 – KIA-54785, and Object 55 – KIA-54786, were inspected with SEM/EDX (Zeiss Gemini Ultra55plus, EDX-detector: Oxford X-act SD-detector. EHT: 15 kV, measuring time: 600,000 counts, calibration: standardless) for areal (between 1 and 180 μm^2) and point analysis of the chemical composition of the slag inclusions, observed in the samples during SEM and microscopic inspection. For each sample, several measurements (between 4 to 8) of random selected slag inclusions across the sampled cross-sections were carried out.
- In addition to the microscopic inspection, a quantitative analysis of geometric properties such as grain size and spacing of cementite lamellae in a number of randomly selected perlite crystals were performed in SEM images using the open-source software ImageJ 1.53a (<http://imagej.nih.gov/ij/>).
- Micro-hardness measurement (Object 25 – KIA-54784, Object 47 – KIA-54785, and Object 55 – KIA-54786) were done at randomly selected positions over the cross-sections using a Leica Reichert Micro-Duromat MD 4000E with prism-shaped diamond indenter (Vickers), a test force of 0.4 N, a convergence rate of about 0.4 N/s, and a dwell time of 20s, i.e. $\text{HV}_{0.04/20}$. After measuring the two visible axes (d_1 and d_2) of the indent prism, (micro) hardness values were determined $\text{HV}_{0.04/20} \approx 0.1891 \frac{F}{d^2} \left[\text{N}, /, \text{mm}^2 \right]$.

Iron sample preparation for radiocarbon measurements

Cut and grinded cross sections were further cut into 1–4 mm pieces with a metal shear, followed by grinding with a DremelTM corundum tool of all surfaces to remove possible abrasions from all tools used. Overall, about > 0.1 mm was removed from the solid metal surface prior to further handling. To remove lipids from handling and (possible) conservation substances, the metal pieces were treated with boiling tetrahydrofuran (THF), chloroform, petroleum-ether, acetone, methanol, and water in a computer-controlled “Soxhlet”-type series extractor (Bruhn et al., 2001), and finally dried at ~60°C in an oven.

The carbon trapped within the iron lattice was released by high-temperature oxygenation (Cook et al., 2001; Cresswell, 1992; Hüls et al., 2004; Van der Merwe, 1969; Van der Merwe & Stuiver, 1968). A step-combustion procedure with a low-temperature (LT, ~560–580°C, 4 h) step, followed by a high-temperature (HT, >970°C, 5 h) step, was applied (Hüls et al., 2019). Cut iron sample material was combusted with high-purity O_2 in an electric resistivity oven attached to a gas manifold for subsequent cryogenic CO_2 purification, selecting the HT- CO_2 -fraction for radiocarbon dating. The resulting sample HT- CO_2 was subsequently purified with a dry-ice/ethanol slurry (−70°C) to remove H_2O , a n-Pentane trap (−130°C) to remove SO_2 (Kusakabe, 2005), and was finally trapped with liquid nitrogen.

The obtained CO_2 was graphitized by the Bosch reaction with hydrogen and an iron catalyst (Nadeau et al., 1998; Vogel, 1992), and the resulting iron graphite mixture is pressed into aluminum target holders for AMS measurement.

The measurement is conducted at the Leibniz-Labor, using the type *HVE 3MV Tandetron 4,130* accelerator mass spectrometer (AMS). The $^{14}\text{C}/^{12}\text{C}$ and $^{13}\text{C}/^{12}\text{C}$ isotope ratios of the samples, simultaneously measured by the AMS, are compared to the oxalic acid II CO_2 measurement standard (NBS SRM 4990C) (Stuiver & Polach, 1977) and are corrected for effects of exposure to foreign carbon during the sample pretreatment, the process blank (Nadeau & Grootes, 2013). The resulting ^{14}C -content is reported in pMC (percent Modern Carbon) and used to calculate the radiocarbon age (Stuiver & Polach, 1977). The reported uncertainty of the ^{14}C result takes into account the uncertainty of the measured $^{14}\text{C}/^{12}\text{C}$ ratios of sample and measurement standard, the uncertainty of the fractionation correction, and the uncertainty of the applied blank correction. Radiocarbon ages are converted to calendar ages using the software package OxCal4 (Bronk Ramsey & Lee, 2013; Ramsey, 2009) and the Intcal20 dataset (Reimer et al., 2020).

RESULTS

Material analysis

For all metallographic analysis, the whole cross sections of samples are inspected under the microscope. Structures described below are summarizing the observations made over the complete cross sections (see also the Supplement). A more detailed metallurgical analysis will be given in a future separate publication.

Object 22 - KIA-54783

Metallographic (microstructural) analysis of the polished and etched cross section ($6\text{ mm} \times 4\text{ mm}$) show a hypoeutectoid mixture, that is, steely structure of ferrite and perlite (see Figure 4, a & b and Figures S1-S2). The carbon content can be estimated at 0.4 wt% by comparison with microstructures of known composition (e.g. Orlich & Pietrzeniuk, 1976; Orlich & Rose, 1973; Rose & Hougardy, 1972; Wever & Rose, 1954). Layers of low carbon iron (ferrite) are welded together with higher carbon iron (perlite) with elongated slag inclusions visible at the welding line. Within carbon enriched layers, ferrite grains appear within a perlite matrix, reminiscent of a Widmanstätten arrangement (Scott, 1991, 31).

Object 25 - KIA-54784

The metallographic inspection of the cross section (about $19\text{ mm} \times 2\text{ mm}$) revealed a hypoeutectoid mixture of ferrite and perlite grains with approximately 0.4 wt% carbon (s.o.) (Figure 4, c & d; Figures S3–S8; Table 1). At first sight, the amount of perlite seems to be higher than ferrite, that is, ferrite grains appear within a perlite matrix. Microscopic inspection of the nital-etched surface shows ferrite grain arrangements reminiscent of a Widmanstätten order (Scott, 1991, 31).

Within the steely metal matrix, several thin layers of elongated slag inclusions are visible, running along the long axis of the cross section, indicating welding (see Figure 4). The amount of perlite with Widmanstätten structure is more pronounced at both sides and along one edge than in the interior. Interestingly, the microstructure at both sides of the several welding lines does not show abrupt changes, which may indicate a rather homogeneous composition of welded material.

Ferrite and perlite grains are angular with sizes ranging from 15 to $30\text{ }\mu\text{m}$ and an aspect ratio of about 1.7. Cementite lamellar in [er]lite grains show a spacing of $0.6 \pm 0.2\text{ }\mu\text{m}$ (see Table 1). Several measurements gave microhardness values varying from $\text{HV}_{0.04/20} 170 - \text{HV}_{0.04/20} 270$, with larger values in the pearlite grains or along grain borders.

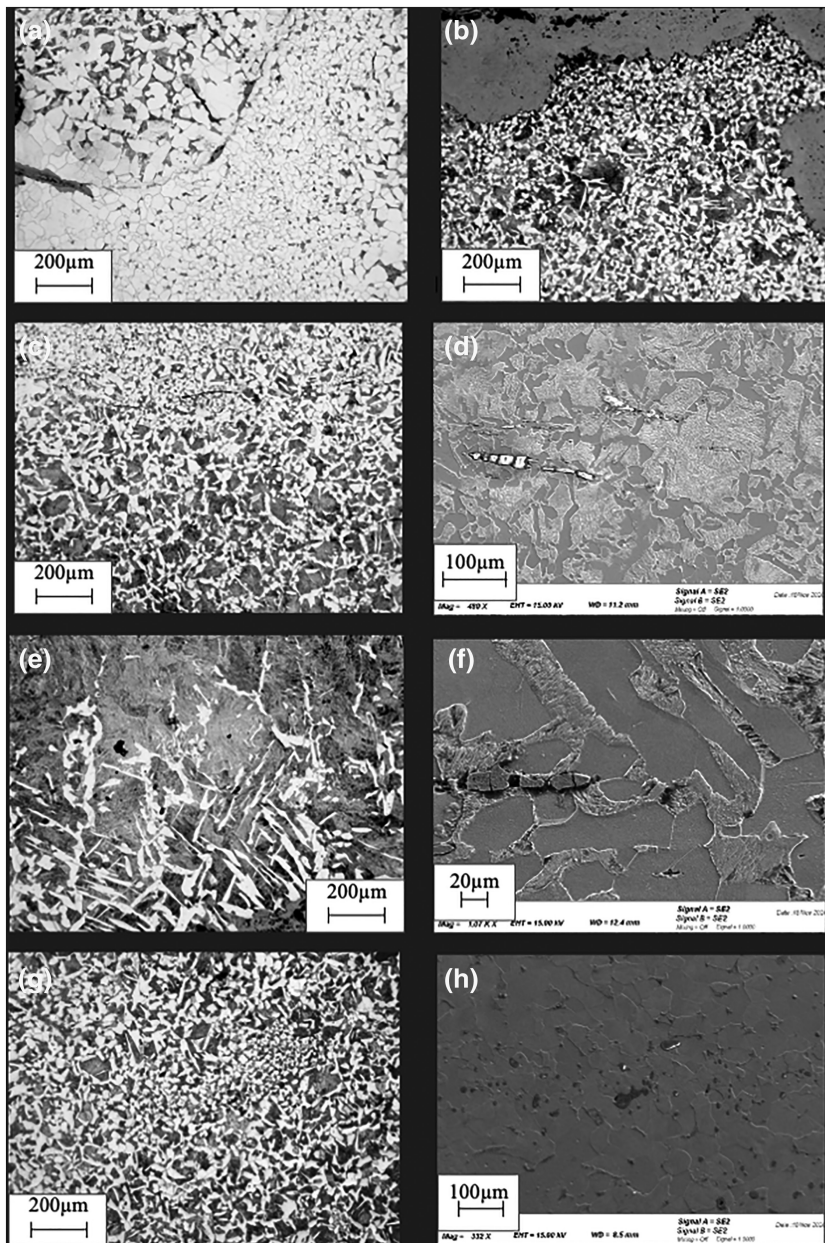


FIGURE 4 Nital etched microstructure of samples under light microscope (Suliga, pers. comm January 2022) and SEM microstructure inspection. a, b: Microscope photo of etched surface of object 22 -KIA 54783; c: Microscope photo of etched surface of object 25 – KIA 54784; d: SEM picture of object 25 – KIA 54784; e: Microscope photo of etched surface of object 47 – KIA 54785; f: SEM picture of object 47 – KIA 54785; g: Microscope photo of etched surface of object 55 – KIA 54786; h: SEM picture of object 55 –KIA 54786.

The mean chemical composition of eight slag inclusions of Object 25 – KIA-54784 is summarized in Table 1. The measured slags contain the least amount of Fe of the samples for which we conducted SEM/EDX analysis. Microscopic microstructure (s.o.), the amount of iron measured and calculated using indices such as the glass index ($G: \frac{(\%CaO + \%Al_2O_3 + \%K_2O + \%MgO) * 100}{\%FeO + \%MnO}$, Buchwald, 2005) or reduc-

TABLE 1 Results for microstructure analysis (grain size [largest diameter d , aspect ratio], cementite lamellar spacing in pearlite, microhardness; amount of measurements is given in parentheses), chemical composition of SI by EDX, radiocarbon measurement results (carbon amount extracted, ^{14}C concentration and ages, calibrated calendar ages).

| | Object 22 - KIA-54783 | Object 25 - KIA-54784 | Object 47 -KIA-54785 | Object 55 -KIA-54786 |
|---|--------------------------|-----------------------------------|------------------------------------|------------------------------------|
| <i>Microstructure analysis</i> | | | | |
| Crystall size largest diameter (μm) /aspect ratio (no. analyses) | n.m. | 24.0 \pm 8.9/1.7 \pm 0.7 (29) | 59.9 \pm 48.9/1.7 \pm 0.7 (13) | 66.4 \pm 26.2/1.7 \pm 0.7 (13) |
| Cementite lamella spacing (μm) (no. analyses) | n.m. | 0.6 \pm 0.2 (12) | 1.0 \pm 0.2 (9) | 0.5 \pm 0.1 (20) |
| Micro hardness (HV _{0.04/20}) (no. analyses) | n.m. | 263.5 \pm 62.7 (22) | 222.9 \pm 41.0 (21) | 190.1 \pm 73.4 (14) |
| <i>Chemical composition of SI measured by EDX</i> | | | | |
| No. of measurements | | 8 | 6 | 4 |
| Na ₂ O | n.m. | n.d. | n.d. | n.d. |
| MgO | n.m. | 1.4 \pm 0.3 | 2.0 \pm 2.0 | 0.3 \pm 0.6 |
| Al ₂ O ₃ | n.m. | 4.3 \pm 0.8 | 5.3 \pm 4.1 | 4.7 \pm 2.4 |
| SiO ₂ | n.m. | 53.1 \pm 7.2 | 16.9 \pm 13.9 | 21.5 \pm 12.0 |
| P ₂ O ₅ | n.m. | n.d. | 0.1 \pm 0.2 | n.d. |
| SO ₃ | n.m. | n.d. | n.d. | n.d. |
| K ₂ O | n.m. | 2.6 \pm 0.5 | 1.0 \pm 0.9 | 1.8 \pm 1.1 |
| CaO | n.m. | 4.5 \pm 1.0 | 1.4 \pm 1.2 | 1.1 \pm 0.8 |
| TiO ₂ | n.m. | 0.3 \pm 0.2 | 0.3 \pm 0.3 | n.d. |
| MnO | n.m. | 16.8 \pm 3.3 | 7.1 \pm 6.1 | 3.5 \pm 3.9 |
| FeO | n.m. | 17.0 \pm 10.9 | 66.0 \pm 28.1 | 68.2 \pm 18.1 |
| G | n.m. | 37.9 | 13.3 | 11.1 |
| F | n.m. | 12.5 | 3.2 | 4.6 |
| SiO ₂ /FeO | n.m. | 3.1 | 0.3 | 0.6 |
| (RII) | n.m. | 3.8 | 0.6 | 0.7 |
| <i>Radiocarbon measurements</i> | | | | |
| Mass combusted (mg) | 970.5 | 613.2 | 915.2 | 1160.8 |
| Carbon (mg) | 2.8 | 2.1 | 5.3 | 3.9 |
| C (wt%) [‡] | 0.3 | 0.3 | 0.6 | 0.3 |
| ^{14}C (pMC) [†] | 80.74 \pm 0.26 | 80.49 \pm 0.24 | 80.19 \pm 0.23 | 80.04 \pm 0.23 |
| Conventional radiocarbon age BP | 1720 \pm 25 BP | 1745 \pm 25 BP | 1775 \pm 25 BP | 1790 \pm 25 BP |
| Calibrated calendar ages (2 σ -range) [‡] | AD 251 - AD 412 | AD 245 – AD 384 | AD 231 – AD 348 | AD 217 – AD 337 |

Abbreviations: n.d. = not detected; n.m. = not measured.

‡resulting from combusted amount of iron.

†pMC give percentage of modern carbon, related to the hypothetical atmospheric value of 1950, corrected for $\delta^{13}\text{C}$ (–25‰) using the $\delta^{13}\text{C}$ value measured by AMS.

‡calibrated using OxCal4 and the Intcal20 dataset (see main text).

ible iron index ($RII = 2.39 \cdot \frac{\text{SiO}_2}{\text{FeO} + \text{MnO}}$; Charlton et al., 2010; Thelemann et al., 2017), the silicate to aluminium oxide ratio, and the silicate to iron ratio all indicate the origin of this material from the (direct) bloomery (smelting) process. More advanced production methods such as blast furnaces,

delivering higher temperatures, and a more reducing environment during the conversion from ore to iron do produce lower quantities of slags (or even none in the case of resulting cast iron), which also contain less iron oxide in the slags due to a higher reducing efficiency. In addition, the resulting material will also show different metal phases in the microstructure analysis, for example, ledeburite, as shown, for example, in experimental smelting studies (e.g. Crew et al., 2011) or in archaeological material, for example, iron from China coming from the blast furnace process (e.g. Liu, 2021).

From all three analyzed samples, the chemical composition of the slag inclusions of Object 25 – KIA-54784 seem to indicate a higher efficiency during the iron making process (i.e. the smelting), as is shown by a low Fe content within the slag inclusions, a larger F value, and a high reducible iron index of 3.8 (RII values ≥ 1 retain SiO_2 , which was not fluxed with FeO ; Charlton et al., 2010).

The slag inclusions of this sample contain a considerable amount of MnO , 16.8 ± 3.8 wt%, indicating that this object may have been smelted from a high Mn ore (L'Héritier et al., 2013).

Object 47 - KIA-54785

Microscopic and SEM inspection of the about $30\text{ mm} \times 1\text{--}2\text{ mm}$ cross section show a mixture of angular, elongated ferrite and slightly more perlite crystals, indicating a carbon content around 0.5–0.6 wt% (s.o.), that is, steel. Comparable to sample Object 25, ferrite crystals are embedded in perlite grains with apparent Widmanstätten structure (see Figure 4, e and f; Figures S9–S15). An unfavorable thermal handling is indicated by conifer-like ordering of ferritic crystals (see supplement) in some part of the inspected cross section.

Several layers of flat, elongated slag inclusions are visible, running along the long axis of the analyzed cross-section. Grain sizes vary around $25\text{--}60\text{ }\mu\text{m}$ with an aspect ratio of about 1.6. Cementite lamellae spacing within the perlite grain is $1 \pm 0.2\text{ }\mu\text{m}$. Multiple measurements of the microhardness vary between HV_{0.04/20140–280} (Table 1).

The comparable large amount of Fe within the slag inclusions (66 wt%), the low SiO_2 and Al_2O_3 content, as well as a reducible iron index < 1 (Table 1) indicate an inefficient smelting process (Buchwald, 2005; Charlton et al., 2010; Thelemann et al., 2017), considerably lower than for Object No.25.

Object 55 - KIA-54786

Inspection of the microstructure of the polished and surface-etched cross-section (about $15\text{ mm} \times 6\text{ mm}$) of sample KIA-54786 reveals a mixture of perlite and ferrite crystals, varying in size from around $26\text{ }\mu\text{m}$ to $120\text{ }\mu\text{m}$, with an aspect ratio around 1.7 (see Figure 4; Figures and Table 1, Figures S16–S21). In contrast to the other two samples analyzed, some voids are observed within the material. In addition, the many slag inclusions appear more angular, indicating that less mechanical force was applied during the smithing process. Cementite lamellae in pearlite grains show a spacing around $0.5 \pm 0.1\text{ }\mu\text{m}$. The distribution of pearlite and ferrite grains indicate a carbon content around 0.3 wt% (s.o.), that is, a low carbon steel.

As in the other samples, some Widmanstätten structure of ferrite within perlite grain is observed. Measured mean micro hardness is around HV_{0.04/20190.1}.

As in sample Object 47, the slag inclusions have a high Fe content (68 wt%, Table 1). Likewise, low SiO_2 and Al_2O_3 contents indicate an inefficient smelting process (s.o.).

To sum up the metallurgic analysis, the microstructure analysis of the four metal objects reveals low to medium steely iron, containing a significant number of slag inclusions, characteristic for the bloomery iron smelting technique. Welding zones indicate the forging of slightly different carbonized iron with gradual transitions. Further analysis would be needed (e.g. slag inclusion analysis) to detail about the origin of forged iron parts.

Furthermore, the microstructure may indicate a heat treatment, even if cooling rates were not high enough for the formation of martensite. The analyses of the chemical composition of the slag inclusions for three samples show some variability with respect to the iron production. Results of the from Object 25 – KIA-54784 could indicate a more efficient smelting process as compared to Objects 47 and 55. In addition, the considerable higher MnO content of slag inclusions within Object 25 in comparison to Objects 47 and 55 exceed concentrations measured in archaeological slags from Silesia characteristic for bog-iron ore origin (Thelemann et al., 2017), which could indicate other, high MnO-rich iron ore source.

One should note, though, that the inspected cross section of each metal object only covers a fraction of the whole. Also, the considerable low amount of measured slag inclusions (< 10 analysis) in comparison to studies such as Dillmann and L'Héritier (2007) or Buchwald and Wivel (1998), question the representativity of calculated average values. A more in-depth microstructure and geochemical slag inclusion analysis as well as a larger sample base for comparison, ideally a complete investigation of all the Nowe-Brzesko metal finds, would be needed to confirm above interpretation regarding the iron technology used with respect to production and handling. Nevertheless, we think that even the low number of measurements of randomly selected slag inclusions and metallographic inspections, distributed over the complete inspected cross-section of samples Object 22, 25, 47, and 55, still give a preliminary indication of techniques used to produce the objects.

Radiocarbon measurement

After surface grinding, all samples reveal a considerable amount of solid, uncorroded metal for subsequent thermal carbon extraction. During HT-combustion, all samples delivered a more than sufficient amount of carbon (Table 1), trapped within the lattice mostly as cementite (Fe_3C), as indicated by microstructure analysis. Measured radiocarbon ages vary between 1720 to 1790 ^{14}C years BP, corresponding to calibrated ages around calAD217–calAD412 (Table 1 and Figure 5). Note that the rather broad calibrated age range is caused by a larger variability within the IntCal20 calibration data (Bronk Ramsey & Lee, 2013; Ramsey, 2009).

A prerequisite for a reliable radiocarbon age determination is the use of contemporaneous carbon sources during the production process. Ancient metal production utilized mainly charcoal, made from wood, to provide the heat necessary for the reduction of the ore and for smithing of the resulting bloom, which can be assumed to have an intrinsic age (old wood effect) of up to a few decades (Buchwald, 2005; Rehder, 1991). An indirect confirmation may come from the study of the iron production site in Joldelund, Northern Germany, from the third century AD, where anthracological analysis of associated charcoal finds indicated wood age offsets up to only a few decades (Dörfler

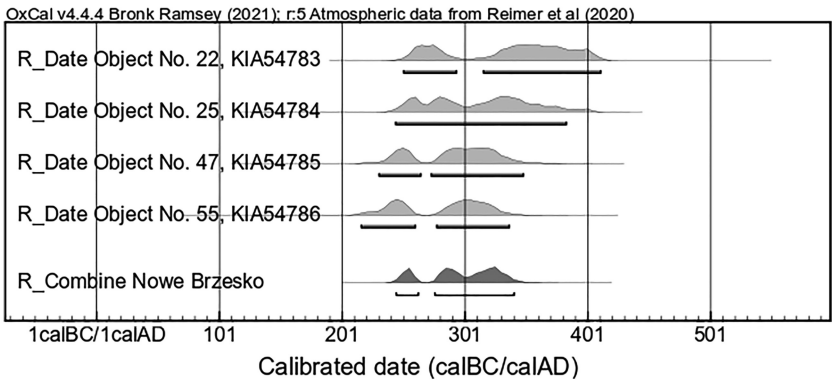


FIGURE 5 Radiocarbon dates of iron samples.

& Wiethold, 2000). This heat source was used in Europe until the early 18th century AD, when the coking process using fossil coal was invented and applied within iron production (Hyde, 1973).

Nevertheless, the radiocarbon age estimate could be influenced by contamination with fossil carbon, as discussed in Craddock et al. (2002), for example, by the use of fossil carbon containing calcite or lime as a flux ingredient (for increasing reduction efficiency and Fe yield from the ore), resulting in much older ($>> 10,000$ years) age estimates, as shown by Park et al., 2008. Experimental smelting by Oinonen et al. (2009) of roasted iron ore, contemporary charcoal, and calcite gave an iron bloom which showed an apparent ^{14}C age of the extracted carbon that was about 2000 yrs too old. Unfortunately, Oinonen et al. (2009) measured Ca concentrations in bulk metal, which are not comparable to the analyses of slag inclusions in this study. Another contamination source may come from used iron ores itself such as geological (i.e. fossil) carbon in siderite (FeCO_3). However, the low thermal stability of this phase (siderite decomposes $< 550^\circ\text{C}$; Gallagher & Warne, 1981) will result in a removal of the fossil carbon either during the roasting of the ore or during the low temperature, oxidising upper part of smelting ovens, as was also confirmed by experimental iron smelting studies (e.g. Leroy et al., 2012).

Considering that the microstructure analysis indicates that our objects were made by the bloomery process, and that the remaining Fe concentration in the slag inclusions indicates a less efficient smelting process compared to results documented by Buchwald (2005) for archaeological iron produced by the blast-furnace technique, the use of flux ingredients may be questioned.

Another potential source of contamination is addition of fossil carbon from modern steel tools used to sample the objects (Hüls et al., 2011; Scharf et al., 2004). This risk, however, was minimized using coarser cut iron pieces and a thorough surface cleansing by grinding cut edges. The efficacy of these steps was confirmed by newer studies with iron and steel samples with a known modern ^{14}C composition (Hüls et al., 2019).

To sum up, we take the amount of slag inclusions observed in the microstructure analysis, and the geochemical signature (s.o.), as evidence for the bloomery iron production process of these objects. Furthermore, the metallurgical analysis does not indicate the use and presence of fossil carbon containing processes. That said, we believe the measured radiocarbon ages of extracted carbon mirrors the age of the iron production process within reasonable limits coming from analytical uncertainties, inherent age effects such as old-wood effects, and errors introduced by calibrating to calendar ages. A possible magnitude of these uncertainties up to 60 yr is shown in radiocarbon ages of known-age archaeological samples, such as the Nydam iron finds (Hüls et al., 2019).

DISCUSSION

The practice of depositing hoards including metal tools has been encountered in Europe since the Bronze Age, although this phenomenon occurs with varying intensity at different times and in different areas of the continent. In the case of the Vistula basin, they have been known since the earliest phases of the Bronze Age and are recorded up to historical periods. In Bronze Age hoards, tools constitute the most numerous category of artefacts (among which axes and sickles predominate), and their share decreases significantly only in the early phases of the Iron Age, when costume ornaments become more common (Blajer, 2001). It should be noted, however, that in addition to their function as tools, these objects most likely also functioned as currency used in trade (Sommerfeld, 1994). The appearance of the next wave of tool deposits, in this case iron tools, occurs during the La Tène period and the Roman period. In the La Tène period, it is associated with the customs of depositing this type of object by the Celtic population (Kurz, 1995; Pieta, 2010; Schönfelder, 2006). Very often these deposits are composed of tools of different types, linked to different zones of craft and agricultural activity. They include agricultural tools, blacksmith's tools, carpentry tools, and parts of carts. From southern Poland, similar group finds are known from the site in Kraków-Wyciąże (Buratyński, 1953) and Pakoszówka, Sanok District (Bochnak et al., 2016).

The number of metal artefacts discovered, often testifying long-distance cultural contacts, including a collection of Roman imports, allows us to hypothesize the centuries-long significance of this site as a port of trade. Indirectly, this hypothesis may also be supported by the size of the site in Nowe Brzesko and the number of the above-mentioned metal objects found until now. In the area of present-day Poland, several sites of this type are known which date to the Roman period and the early phase of Migration Period of people. Among them is Jakuszowice (Bursche et al., 2000; Godłowski, 1991). These evidences led us to assume that the site in Nowe Brzesko occupies a unique position in the local, or perhaps even supralocal, settlement network. The collection of iron objects described here was discovered in the western part of the site, where artefacts from all the above-mentioned periods are recorded.

Deposits of iron tools are also found in the area occupied by a population linked with the Przeworsk culture, a cultural unit that was developing in central and southern Poland between second century BCE to fifth century AD (Andrzejowski, 2010). These assemblages are dated mainly to third–fourth century AD, and they contain variable categories of accessories, including agricultural, blacksmithing, and carpentry tools (Kolenda & Chrzan, 2012; Rodzińska-Nowak, 2012; Żygadło, 2002). Finds of this type have been discovered both at the settlements and, though much less frequently, in burials. It should be stressed, however, that the Nowe Brzesko collection discussed here is the largest collection of this type so far.

We re-emphasize that many of these objects exhibit little variation in form, which makes it difficult to assess their chronology by typology, and that the assemblage, discovered on the surface of the site, was displaced from original deposition location. Therefore, the results of the scientific analyses prove how useful these methods are already at this stage of the investigation. Importantly, we want to stress the fact that the obtained dates are quite similar and consistent, and support our idea that the discovered group of metal artefacts is a dispersed hoard of objects of about the same date. This allows us to state that we are dealing with one of the largest iron tool deposits of that kind, from this part of Barbaricum dated to the Roman Period.

As mentioned above, the methods applied also offer wider analytical possibilities than just the possibility of precise dating of individual objects. Taking into account the regionalisation of iron production within the Przeworsk culture (Orzechowski, 2018, 2019), and the importance of known production centres for the economy of the entire area of the Przeworsk culture, the possibility to study the provenance of individual iron objects taking into account their dating opens up completely new research areas. Their development over time can significantly help to understand economic and social relations in the Vistula basin.

CONCLUSION

Here we present preliminary results of metallurgical analyses of a number of iron artefacts from a displaced hoard discovered at a multiphase site in Nowe Brzesko, Małopolskie Voivodeship, Poland. Absolute ages were estimated using the radiocarbon dating method on carbon extracted from the metal lattice. Radiocarbon analysis was complemented by metallurgical analyses such as microstructure investigations and determination of the chemical composition of slag inclusions.

The AMS measurements gave consistent ^{14}C ages, which would correspond to production dates in the third–fourth century AD. The four analyzed iron objects show a structure typical for the bloomery iron production technique, and the slag inclusions contain significant amounts of iron, which is also characteristic of the comparatively inefficient bloomery smelting technique with respect to the more efficient blast-furnace technology used in modern times. The chemical composition of the slag inclusions of three objects may also indicates a variability in the production process (smelting efficiency) and possible differences in the origin of the ore used, but these interpretations need further verification by expanding this investigation to (a) increase in geochemical analysis of slag inclusions of single samples, and (b) to an increase of analysis of more objects in the assemblage. The metallurgical analysis does not indicate the use of substances containing fossil carbon, which might compromise

the radiocarbon measurements. We thus believe that the results presented here could open up new research avenues on the economy and society of the Przeworsk culture in the Late Roman period.

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DATA AVAILABILITY STATEMENT

All the data discussed in the manuscript is shown and documented in the manuscript and in the additional online available supplement.

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