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Jerzy Nawrocki, Karol Standzikowski, Maria Łanczont,
Jan Gancarski, Mateusz Kłosowicz, Tomasz Leszczyński

Luminescence dating of ancient kilns and gothic to baroque bricks from SE Poland – a comparison with archaeomagnetic data

ABSTRACT

The TL and OSL ages obtained from two ancient kilns and gothic to baroque bricks in the SE of Poland, were compared to their presumed historical ages and discussed. The luminescence ages of ancient kilns were also matched with the results of archaeomagnetic dating. The OSL and archaeomagnetic data indicates that the ancient kilns were last used not later than ca. 280 AD. This age corresponds well to the lower limit of the postulated age of fragments of Roman type storage vessels found in the filling of kilns. The OSL data used alone can point even to the end of the phase B2 of Roman influence in Poland. The TL ages obtained from the kilns (5297 ± 256 BC, 7092 ± 423 BC) are significantly older. These bottom parts of kilns, from which samples were taken, most probably were heated to a relatively low temperature, insufficient to reset the TL signal. Most of TL ages obtained from gothic to baroque bricks do not correlate with their historical ages. Unknown and most probably varied brick moisture contents in sampled ground floor walls and cellars does not allow to precisely date these bricks using the TL signal.

KEYWORDS

luminescence dating, archaeomagnetism, kilns, bricks, SE Poland

I. INTRODUCTION

The application of thermoluminescence (TL) dating of ancient building materials is being increasingly developed for use in the field of building construction archeology (e.g. Blain *et al.* 2007; Chruścińska *et al.* 2008). In Poland the brick samples from the Castle of the Teutonic Order in Malbork were regarded as a very good material for TL dating, in spite of a certain age inaccuracy connected with the uncertainty of moisture content (op. cit.). Comparative luminescence and archaeomagnetic studies are not frequent. It was conducted for example on the Roman bricks from Italy (Thema *et al.* 2015).

The bricks and kilns are some of the best materials for archaeomagnetic studies. They usually contain a remanent magnetization that is very stable and intense and therefore suitable for defining the geomagnetic field parameters. The kilns because of their stable spatial position can provide all archaeomagnetic parameters also including archaeodeclination. It should be stressed, however, that ancient kilns inside the ground can have walls affected by high temperature in different degree. Especially their cooler bottom could acquire partial thermoremanent magnetization (pTRM) only and because of this is not useful for reliable reconstruction of palaeointensity of ancient geomagnetic field.

The aim of this study is to estimate precise ages of two ancient kilns from SE Poland using the luminescence and archaeomagnetic method. The TL method is also applied for dating of selected gothic to baroque brick building from the SE of Poland. Through the comparison of our data and historical information about the studied artifacts, we were going to define utility and constrains of such a comparative methodology of dating of the baked artifacts from this part of Poland. Samples from the brick buildings were taken from the basements where we expected original bricks i.e. not from any younger reconstruction phases. However, the moisture variable over time was also supposed in these parts of buildings. Bearing in mind all these constrains, we would like to answer the question: to what degree these unstable conditions expected in the basements of studied buildings (changing moisture, possible fires) can influence the results of TL dating.

II. STUDIED MATERIAL AND METHODS

The archaeological site with ancient kilns, located in SE Poland near the Lipnica Dolna village, 8 km NW of the city of Jasło (Fig. 1: A), was discovered in 1996 and recognized in details by means of magnetic survey in 2017. Two kilns, constructed one on top of the other Fig. 1b), were excavated and sampled. Fragments of Roman type storage vessels were found in the filling of kilns and are linked with phases C1b-C2 (210-310 AD) of Roman influence in Poland (Kłosowicz and Leszczyński 2017). The geographically oriented samples (25 mm in diameter and 22 mm long) used for the archaeomagnetic study were taken from the reddish silt cropping out in the bottom parts of the kilns using a steel cylindrical sampler. Natural remanent magnetization (NRM) was measured by means of a JR-6A spinner magnetometer (AGICO Brno). All samples were subjected to stepwise thermal demagnetization in the non-magnetic furnace MMTD1 of Magnetic Measurements. The NRM measurements and demagnetizations were carried out in the MMLFC shielded room of Magnetic Measurements. Demagnetization results were analysed using orthogonal vector plots (Zijderveld 1967), and the directions of linear segments were calculated using principal component analysis (Kirschvink 1980). Magnetic susceptibility and its anisotropy were measured by means of a MKF1-FB bridge (AGICO Brno). Isothermal remanent magnetization (IRM) with maximum amplitudes of 1.5 T was acquired using a Magnetic Measurements pulse magnetizer MMPM. As it was confirmed by unsuccessful TL dating (see below), the bottom parts of kilns were heated in medium temperatures only. Because of this we reconstructed the archaeodirection of geomagnetic field and resigned from its archaeointensity estimation. From the same reason, apart from the TL method, the optically stimulated luminescence (OSL) method was used additionally for dating of these kilns.

The OSL and TL ages were determined for two samples taken from the same parts of kilns where archaeomagnetic samples were collected (Fig. 1: B). Samples of 600-800 g in weight were dried in a laboratory, placed in a Marinelli-type containers and then in a protective chamber of the three-channel, stationary gamma spectrometer of MAZAR-01 type (produced by Polon IZOT Warszawa) to measure the concentrations of Ra226, Th228 and K40 radionuclides. Each sample was analysed 40 times and each measurement lasted 2000 s. The concentrations of 226Ra, 228Th and 40K were converted into doses of α, β and γ ionizing radiation, taking into account the conversion factors of Adamiec and Aitken (1998). The TL and OSL dose values were calculated for the 45–63 μm grain fraction. The amount of cosmic radiation at the sampling site was calculated according to Prescott and Hutton (1994). The dose rate (dr) is a sum of doses from α, β, γ and cosmic radiation. Each sample was sieved to obtain a 45-63 μm fraction. Such a polymineral fraction was treated with 10%

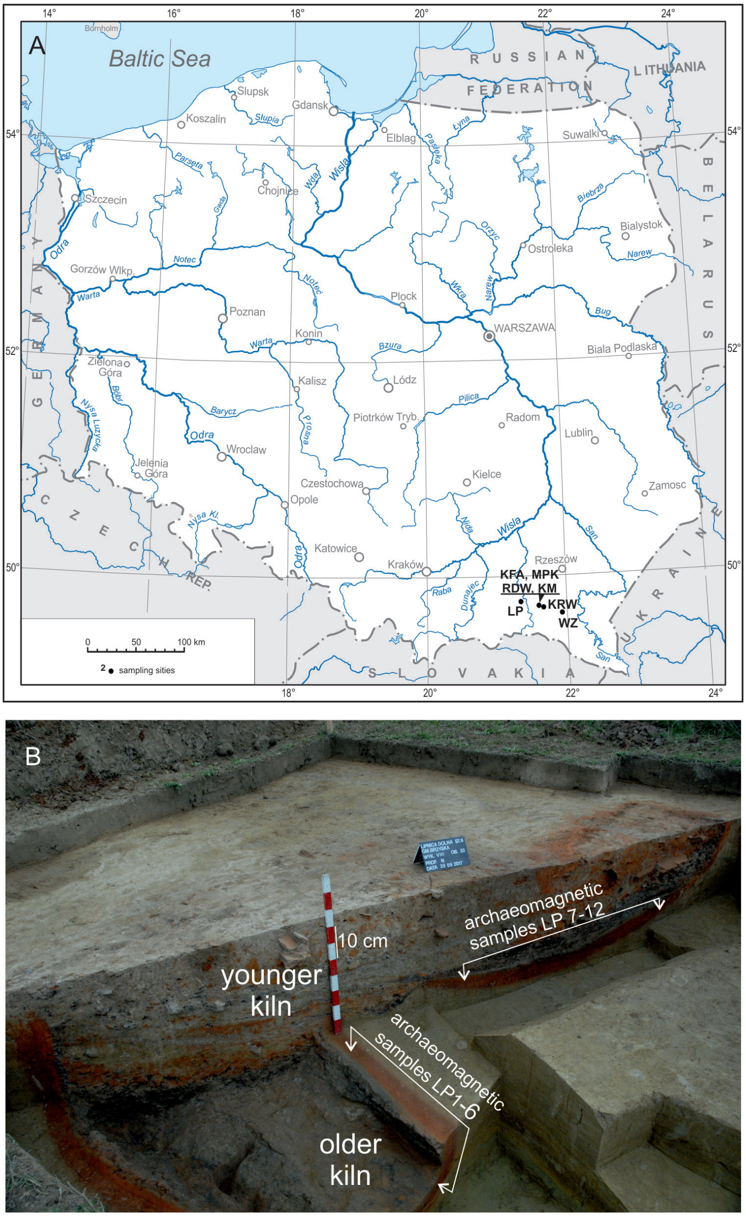

FIG. 1. A – Location of examined sites : LP – Lipnica Dolna kilns; MPK – Krosno, defensive wall in Subcarpathian Museum; KFA – Krosno, Franciscan church; wwwRDW – Krosno, Mayor's tenement house; KM-Krosno, defensive wall of Franciscan Monastery; KRW-Krościenko Wyżne, mansion ruins; WZ- Wzdów, palace (see Table 1). B – Bottom parts of kilns in Lipnica Dolna with sites of sampling marked

HCl for 24 hours and with 30% H_2O_2 for 8 hours, washed with distilled water several times after each phase of treatment, and then dried in a temperature of 50°C for 24 hours. OSL and TL measurement cycles were carried out using the RISO TL/OSL DA-20 reader with the U340 filter. Each sample was stimulated with a blue LED with a wavelength of 470 nm at ~ 60 mW/cm2, and then irradiated using a 90Sr/90Y beta source. For the OSL method the equivalent dose was determined using the SAR procedure (Murray and Wintle 2000), and for the TL method – the TL SAR procedure (Honga *et al.* 2006). The ^{238}U, ^{232}Th decay chains, and ^{40}K contents were converted to dose rates with conversion factors provided by Cresswell *et al.* (2018). The correction on dose calculations as presented by Autzen *et al.* (2022) was also applied.

Samples for TL studies were also taken from the ground floor walls and cellars of six historical brick buildings constructed in SE Poland (Fig. 1: A; Table 1). The historical ages of these buildings enclosed between 1405 and 1790 AD were adopted from existing written sources (Table 1). However, the accuracy of them was defined arbitrarily to be not less than 5 years, even if historical sources provide more precise ages. This is due to a possible time gap between brick production and their use in the construction of buildings. In cases when the information about the age of a particular building was not direct, e.g. referred to its funding only, we assumed an age accuracy of ca. 15 years. The TL ages were determined for twelve bricks.

The moisture content of samples from Lipnica Dolna was measured in the laboratory and was 12%. The same value was assumed for the bricks.

TABLE 1. Brick buildings in SE Poland sampled for TL studies

No	Site, brick building	Geographic coordinates		Age, references	N
1.	Krosno, defensive wall in Subcarpathian Museum	49.6956	21.7637	1350 ±10, Proksa, 1990	2
2.	Krosno, Franciscan church	49.6937	21.7667	1405 ±5, Łopatkiewicz, 1993	2
3.	Krosno, Mayor's tenement house	49.6936	21.7659	1490 ±10, Cynarski, 1960	1
4.	Krosno, defensive wall of Franciscan Monastery	49.6935	21.7664	1505 ±5, Łopatkiewicz, 1993	3
5.	Krościenko Wyżne, mansion ruins	49.6810	21.8076	1650 ±10, Arłamowski *et al.* 1970	3
6.	Wzdów, palace	49.6430	22.0011	1790 ±5, Budziński, 1998	1

N- number of bricks used for TL studies

III. RESULTS

Lipnica kilns - TL and OSL ages

OSL ages determined for both kilns constructed directly in the ground are close to each other (Table 2). A certain difference reflects the stratigraphic superposition of studied objects. Sample from the lower kiln yielded OSL age of 122 ± 6 AD whereas sample taken from the upper kiln gave OSL age of 100 ± 6 AD. TL ages from the same samples are completely different. The older kiln provided a TL age of 5297 ± 256 ka and the younger one overlying it yielded a TL age of 7092 ± 423 ka (Table 2).

TABLE 2. Results of TL and OSL dating of two ancient kilns from Lipnica Dolna (SE Poland)

Site (depth) [m]	Lab. No LUB	^{40}K [Bq/kg]	^{226}Ra [Bq/kg]	^{228}Th [Bq/kg]	Dose rate d_r [Gy/ka]	OSL Equivalent dose d_e [Gy]	TL Equivalent dose d_e [Gy]	OSL age [ka]	TL age [ka]	Date [years]
Upper kiln (1.00)	**6463**	726±10	34.6±2.3	56.3±1.2	4.13 ± 0.21	7.92 ± 0.22		**1.92 ± 0.11**		**100 ± 6 AD**
Bottom kiln (1.50)	**6464**	860±11	39.2±2.3	53.7±1.0	4.56 ± 0.22	8.65 ± 0.22		**1.90 ± 0.10**		**122 ± 6 AD**
Upper kiln (1.00)	**6463**	726±10	34.6±2.3	56.3±1.2	4.13 ± 0.21		37.63 ± 1.07		**9.11 ± 0.53**	**7092 ± 423 BC**
Bottom kiln (1.50)	**6464**	860±11	39.2±2.3	53.7±1.0	4.56 ± 0.22		33.36 ± 0.97		**7.32 ± 0.35**	**5297 ± 256 BC**

Lipnica kilns - archaeomagnetic data

The anisotropy of magnetic susceptibility points to preservation of sedimentary magnetic fabricks in most of samples which is documented by the nearly vertical location of minimum susceptibility axes K3 and positive value of the shape parameter T (Fig. 2) that implies the dominance of magnetic foliation in most of samples and oblate character of the AMS ellipsoid. The mean value of the corrected anisotropy factor Pj is 1.036. Apart from two samples, the rest display magnetic susceptibility values enclosed between 309 and 712 x 10^{-6} SI

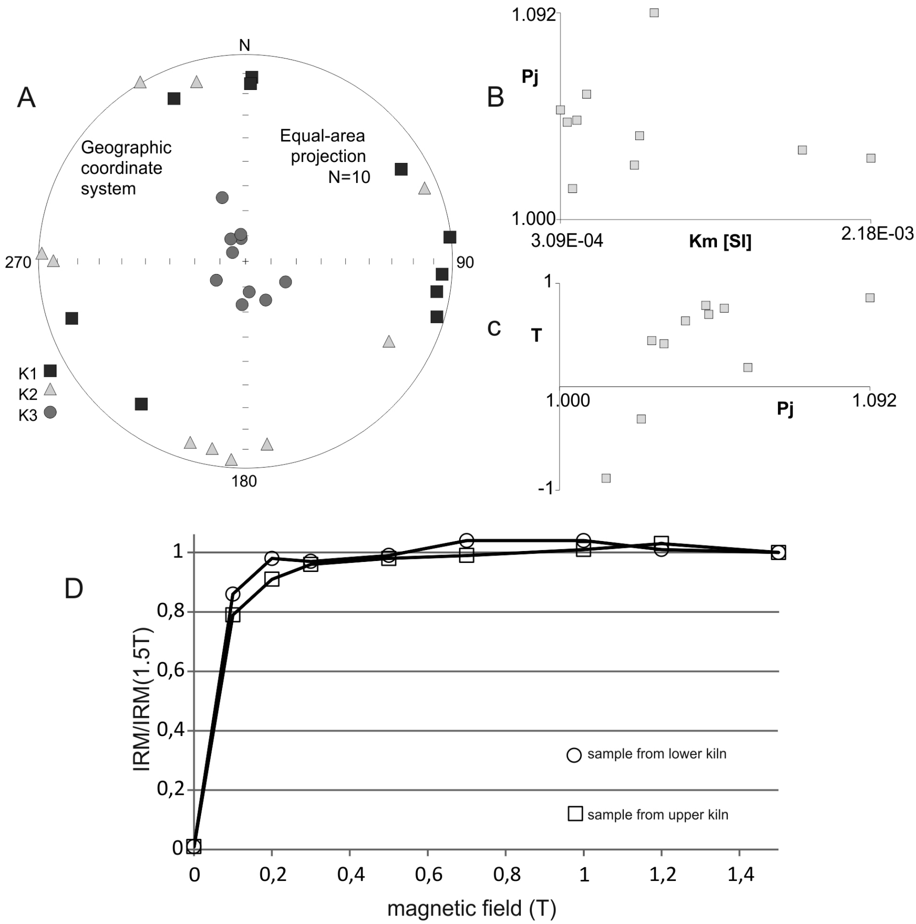

FIG. 2. A – Equal area projection of the anisotropy of magnetic susceptibility (AMS) principal axes defined for samples from kilns in Lipnica Dolna (K1 – maximum susceptibility axes, K2 – intermediate susceptibility axes, K3 – minimum susceptibility axes). B – The relationships between the AMS parameter Pj (corrected anisotropy factor) and magnetic susceptibility, and between the shape parameter T and corrected anisotropy factor (C) presented for the same sample set. D – Isothermal remanent magnetization (IRM) curves prepared for two samples taken from the lower and upper kilns that were exposed in Lipnica Dolna

units that are not significantly higher than those observed in unaffected by heating natural loess-like deposits that usually display 100 – 300 x 10^{-6} SI units of the volume magnetic susceptibility (see e.g. Nawrocki *et al.* 1999).

All the above mentioned magnetic features indicate that the bottom parts of kilns constructed directly in the surrounding sedimentary rocks could not be heated to temperatures as high as these changing substantially the primary structure and composition of magnetic fabrics. The IRM acquisition curves (Fig. 2: D) and the results of thermal demagnetization (Fig. 3) indicate that partial thermoremanent magnetization of bottom parts of kilns is carried by the low coercivity mineral with unblocking temperatures enclosed between 500 and 550 °C, most probably detrital magnetite with a small content of titanium (e.g. Dunlop and Özdemir 1997).

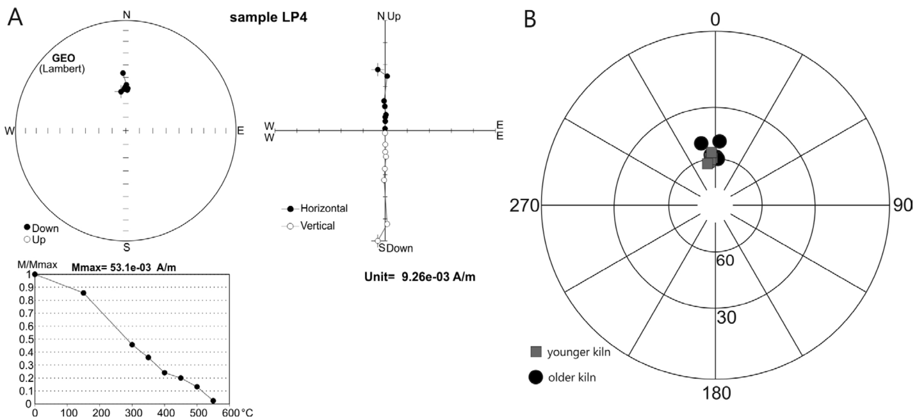

FIG. 3. A – Results of thermal demagnetization of representative samples from kilns in Lipnica Dolna (stereographic projection of demagnetization path, intensity decay curve and orthogonal plot; M – intensity of remanent magnetization). B – Stereographic projection of characteristic archaeomagnetic directions isolated from kilns in Lipnica Dolna

The structure of NRM in all samples is very simple. After removal of unstable, most probably viscous magnetization at a temperature of 150°C, only one component of NRM remained. This characteristic magnetization with a northward declination and positive moderate inclination was completely removed at a temperature of about 550°C (Fig. 3). The standard deviation angular error calculated during the determination of characteristic directions by the best-fit line method was less than 3° in all studied samples. The mean palaeomagnetic directions determined at sample level for both kilns are close each other (Table 3).

TABLE 3. Archaeomagnetic directions and poles obtained from two kilns in Lipnica Dolna (SE Poland: 49.784278oN, 21.381202oE)

Site	D (°)	I (°)	α 95(°)	K	N	PLAT(°)	PLONG(°)	dp (°)	dm(°)
Older kiln	359	58	4.8	196.2	6	78 N	206 E	5.1	7.2
Younger kiln	352	61	2.5	745.4	6	80 N	238 E	3.2	4.2
Both kilns	356	59	2.7	262.4	12	80 N	221 E	3.0	4.1

Brick buildings - TL ages

TL ages obtained from particular buildings, where more than one brick was sampled, are not consistent. Two bricks from the defensive wall of Subcarpathian Museum in Krosno, that was constructed ca. 1350 AD, gave TL ages of 1248 ± 60 AD and 1526 ± 32 AD (Table 4). Two other bricks taken from the Franciscan Church (c.a. 1405 AD) in the same city yielded TL ages 1309 ± 46 AD and 1408 ± 41 AD. TL ages from the defensive wall surrounding this building and constructed ca. 1500 AD are older but convergent (1312 ± 47 AD and 1315 ± 47 AD). The third TL age from the same site is significantly older (1188 ± 57 AD). Three bricks from the mansion ruins in Krościenko Wyżne

TABLE. 4. Results of TL dating of gothic to baroque bricks from SE Poland

No.	Site and historical age (AD)	Sample	lab. No. LUB	Dose rate dr [Gy/ka]	TL equivalent dose de [Gy]	TL age (A.D.)
1.	Krosno – Franciscan church, 1395 - 1405	KFA1	6445	3.50 ± 0.21	2.48 ± 0.05	1309 ± 46
2.		KFA2	6446	3.28 ± 0.18	2.00 ± 0.07	1408 ± 41
3.	Wzdów, palace, 1785-1795	WZ1	6501	3.22 ± 0.21	0.94 ± 0.03	1729 ± 21
4.	Krościenko Wyżne – mansion ruins, 1640-1660	KRW3	6503	3.92 ± 0.23	1.98 ± 0.06	1513 ± 33
5.		KRW1	6514	3.95 ± 0.24	1.41 ± 0.06	1593 ± 33
6.		KRW2	6515	3.75 ± 0.24	1.20 ± 0.03	1657 ± 24
7.	Krosno – defensive wall in Subcarpathian Museum, 1340-1360	MPK1	6504	3.60 ± 0.21	1.77 ± 0.05	1526 ± 32
8.		MPK2	6505	3.16 ± 0.24	2.44 ± 0.06	1248 ± 60
9.	Krosno – Mayor's tenement house, 1480-1500	RDW	6507	3.40 ± 0.18	2.47 ± 1.88	1273 ± 49
10.	Krosno – defensive wall of Franciscan Monastery, 1500-1510	KM1	6511	3.58 ± 0.20	2.75 ± 0.08	1188 ± 57
11.		KM2	6512	3.48 ± 0.21	2.34 ± 0.05	1312 ± 47
12.		KM3	6513	3.55 ± 0.20	2.32 ±0.06	1315 ± 47

gave TL ages of 1513 ± 33 AD, 1593 ± 33 AD and 1657 ± 24 AD. This building was constructed c.a. 1650 AD (Table 1). Single bricks from the Wzdów palace (ca. 1790 AD) and the Mayor's tenement house in Krosno (ca. 1490 AD) gave TL ages of 1729 ± 21 AD and 1273 ± 49 AD, respectively (Table 4).

IV. DISCUSSION

Archaeomagnetic and luminescence ages of kilns

A comparison of the mean characteristic declinations and inclinations obtained for the younger and older kilns with the reference curves of fluctuation of these parameters between 0 and 1200 AD (Schnepp *et al.* 2020) recalculated for geographic coordinates of the sampled site is presented in Fig. 4. Bearing in mind the analytical errors of the reference curve and the data from Lipnica, it can be noticed that the mean inclination indicates the younger kiln was heated for the last time ca. 0 – 425 AD and the older one ca. 0 -520 AD. These wide age intervals are evidently narrower when characteristic declination obtained from the younger kiln is compared with the reference declination data. This comparison indicates univocally that both kilns were heated for the last time between ca. 20 and 280 AD. This age interval is in agreement with the OSL ages (100 ± 6 Ad and 120 ± 6 AD) (Table 2) and refers well to presumed historical age of storage vessels found in the filling of kilns. These vessels are linked with the C1b-C2 phases of Roman influence in Poland dated at ca. 210-310 AD (Kłosowicz and Leszczyński 2017). The OSL data used alone can point even to the end of the phase B2 of Roman influence in Poland.

TL ages obtained from the both kilns are significantly older and inverted. They do not refer at all to the historical ages of studied artefacts. It cannot be excluded that these ages are close to the time of deposition of the silt in which the kilns' chambers were dug. Evidently, the OSL method gives much more credible results at least in this case, despite the fact that it is most often used for dating of unfired sediments. This difference may result from the fact that the material studied was not subjected to a sufficiently high temperature, therefore the TL signal was not reset or was reset only partly. The OSL signal is, due to its higher light sensitivity, of particular interest when dating archaeological sediments and may have experienced only a little light during deposition that is not sufficient to reset the TL signal (e.g. Wagner 1995). It is very likely that the OSL signal is also more sensitive for the temperature resetting.

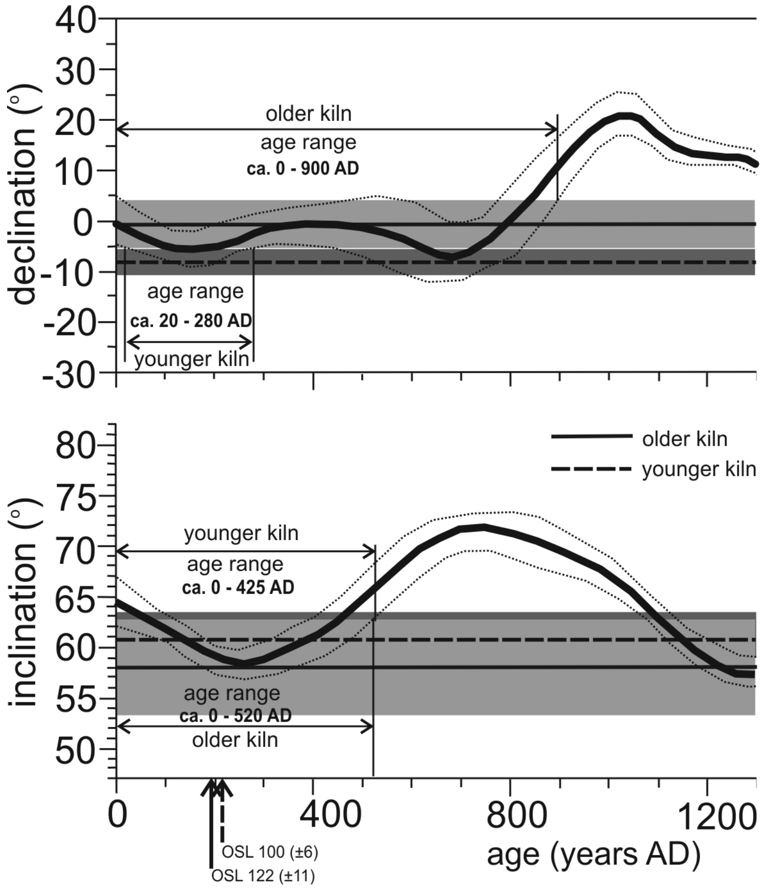

FIG. 4. Mean characteristic inclination and declination obtained from the kilns in Lipnica Dolna (with α95 errors marked by shaded areas, see Tab. 3) on the background of archaeomagnetic reference curves for central Europe (Schnepp et al. 2020) recalculated for geographic coordinates of sampled site. Limits of error are presented by dotted line

Thermoluminescence ages of bricks

TL ages obtained from the sites where more than one brick was analysed are not consistent (Table 4). They are older by ca. 200 years than historical ages in the case of bricks from the defensive wall of the Franciscan Church. The age obtained from the single brick taken in the cellar of the Mayor's tenement house in Krosno differs even more than 200 years from its historical age.

On the other hand the youngest ages obtained from the mansion ruins in Krościenko Wyżne and from the Franciscan Church in Krosno are close to the historical ages of these buildings (Table 1). The age determined for the youngest studied site i.e. the Wzdów palace is ca. 50 years older than the historical age of this building. Apart from the TL age obtained for one brick from the defensive wall of the Subcarpathian Museum in Krosno, all the remaining TL ages, which are distant from the historical ages of buildings, are older than expected. The TL dose ration in the bricks with these overestimated ages most probably was underestimated in different degree. The moisture content applied here (12%) and usually used successfully as the lowest value for the loess, from which the studied bricks were produced (Fedorowicz *et al.* 2018), seems to be overestimated for most of the bricks studied here. This overestimation of moisture content implies a decrease of dose ration and in consequence an older age than the time of brick firing (see e.g. Gueli *et al.* 2018). Unfortunately, it is impossible to reconstruct the moisture content conditions in which particular brick existed over hundreds of years of its history. It seems that the moisture content conditions were different for most of sampled bricks. The moisture content value of ca. 12% is most probably close the upper limit occurred in these bricks. Apart from one sample, the ages younger than expected were not obtained. The utility of the TL method in dating of ancient bricks from the SE of Poland that were affected by varied and unable for reconstruction moisture content conditions, is therefore very limited.

One of the problem related to the use of bricks in the TL and archaeomagnetic studies is the risk of a possible historical age inaccuracy. Younger bricks could be incorporated in the building in the case of renovation. On the other hand, bricks could be reused from former building and the historical date of the studied building can be more recent than the date of the brick production. The mean values of archaeointensities and archaeoinclinations obtained for the same sites and bricks (Nawrocki *et al.* 2023) are generally convergent with the reference curves (Fig. 5). This fact supports the correctness of their historical ages. A clearly divergent archaeointensity value before and after correction for anisotropy and cooling rate was obtained for the location of the "Krosno – Franciscan church" (location no. 2, 1405 ±5 AD) only. It is most probably natural phenomenon because a similar drop of archaeointensity around 1400 AD is characteristic also for the data from Bulgaria (Kovacheva *et al.* 2014).

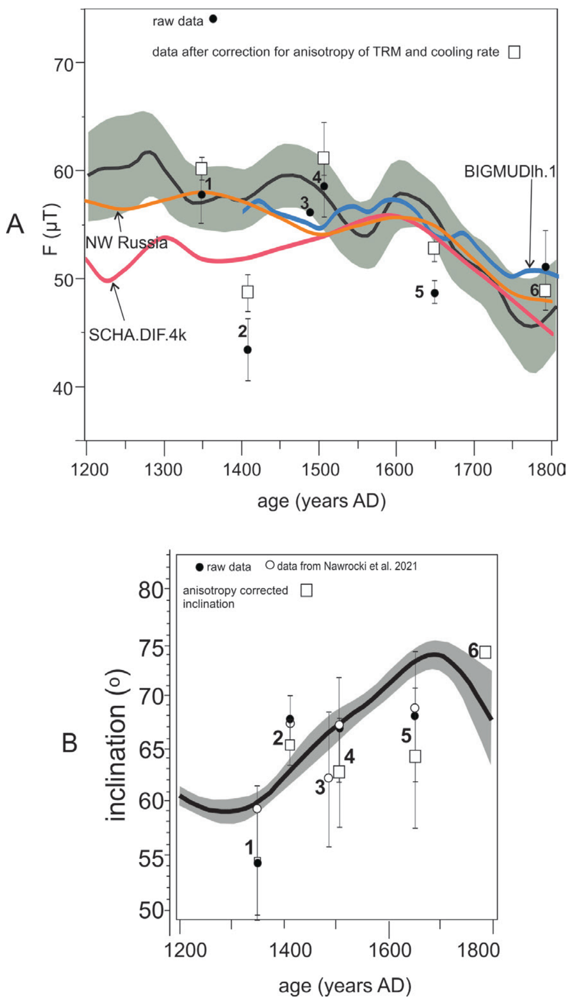

FIG. 5. A – Mean archaeointensities obtained by Nawrocki *et al.* (2023) from studied brick buildings (numbers of sites as in Table 1) presented for the raw data and after correction for the anisotropy of TRM and cooling rate on the background of Bayesian curve with error envelope (grey area) for western-central Europe (Schnepp *et al.*, 2009). The paleosecular variations curves for models BIGMUIh.1 (Arneitz *et al.*, 2021) and SCHA.DIF.4k (Pavón-Carasco *et al.*, 2021), and for NW Russia (Salnaia *et al.*, 2017) are also presented. B – Mean archaeoinclinations defined by Nawrocki *et al.* (2023) for studied here historical brick buildings from SE Poland (raw and corrected for the anisotropy of TRM data) (Nawrocki *et al.*, 2023) on the background of archaeoinclination curve (with its error limit) defined for Austria and Germany (Schnepp *et al.*, 2020). The archaeoinclinations obtained for the same sites by Nawrocki *et al.* (2021) are also presented. All data are recalculated for geographic coordinates of the city of Rzeszów. Vertical bars define standard deviation errors

V. CONCLUSIONS

1. The archaeomagnetic and OSL data indicate that two ancient kilns found near Jasło (SE Poland) were last used not later than 280 AD. This age is close to the estimated age of the Roman type storage vessels (210-310 AD) found in the filling of kilns that were dug directly in the Quaternary loess-like deposits. The OSL data used alone can point even to the end of the phase B2 of Roman influence in Poland.
2. The TL measurements conducted for these kilns gave ages significantly older (ca. 5 -7 ka BP). It is likely that the OSL signal is not only more sensitive in this case than the TL one to being reset by light but by increased temperature as well.
3. Most of the TL ages obtained from the bricks sampled in six historical buildings from the SE of Poland do not correlate with their historical ages. Apart from one estimation, they are older than the time of construction of the studied buildings.
4. Unknown and most probably varied in time moisture content in the places of brick sampling makes impossible to precisely date the bricks using the TL method. Our TL data indicates that assumed 12% of moisture content could be close to the upper limit of this parameter which characterized the surroundings of studied bricks.

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Author contribution

JN conducted and interpreted archaeomagnetic measurements and was major contributor in interpretation of all data and writing the manuscript. KS performed the OSL and TL analyses. MŁ took part in sampling and collected historical data. JG defined historical ages of bricks and obtained permission for sampling. MK analyzed and interpreted data defining historical age of kilns, TL took part in sampling and interpretation of data from kilns. All authors read and approved the final manuscript.

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ADDRESSES OF THE AUTHORS

Jerzy Nawrocki
Maria Curie-Skłodowska University,
Faculty of Earth Sciences and Spatial Management,
Lublin, Kraśnicka 2cd,
20-718, Poland
jerzy.nawrocki@pgi.gov.pl
ORCID 0000-0002-9644-0224

Karol Standzikowski
Maria Curie-Skłodowska University,
Faculty of Earth Sciences and Spatial Management,
Lublin, Kraśnicka 2cd,
20-718, Poland
ORCID 0000-0002-1215-0415

Maria Łanczont
Maria Curie-Skłodowska University
Faculty of Earth Sciences and Spatial Management
Lublin, Kraśnicka 2cd,
20-718, Poland
ORCID 0000-0002-0459-8658

Jan Gancarski
Subcarpathian Museum in Krosno
Piłsudskiego 16,
38-400 Krosno, Poland

Mateusz Kłosowicz
Subcarpathian Museum in Krosno
Piłsudskiego 16,
38-400 Krosno, Poland

Tomasz Leszczyński
Subcarpathian Museum in Krosno
Piłsudskiego 16,
38-400 Krosno, Poland