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OSL DATING AND LITHOLOGICAL CHARACTERISTICS OF LOESS DEPOSITS FROM BIAŁY KOŚCIÓŁ

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Abstract: Absolute dating using luminescence methods is widely applicable in geology, geomorphology, palaeogeography and in archaeology in establishing ages of sediments and archaeological artefacts. By creating absolute time scales for different events in the history of Earth scientists are able to reconstruct changes in climate and environment in the past, and the history of colonization and development of culture.

Grain size is the most important loess lithologic property. Grain size composition depends mainly on factors connected with depositional processes (i.e. variety of source areas, distance from source areas, frequency and intensity of dust transporting winds). The influence of post depositional processes on changes in grain size composition seems to be of less importance with the exceptions for warm and long periods of intensive pedogenesis, which are favorable for formation of clay minerals. Therefore the grain size differentiation within thick loess sections may be used as a proxy record of climate changes during loess cover development.

Here we present results for 12 samples dated at the profile at Biały Kościół. Obtained OSL results in some cases are quite different as compared with the OSL and TL dates obtained during last 10 years by other authors and presented in previous publications relating to this loess profile.

Keywords: luminescence dating, equivalent dose, grain size composition.

1. INTRODUCTION

Presence in almost any natural environment of quartz and feldspars and the development of the measurement equipment allows a wider use of luminescence techniques to determine absolute ages of sediments and archaeological artefacts. Luminescence methods are based on the assumption that these naturally occurring minerals can be used as dosimeters recording the amount of radiation to which they have been exposed, in the form of charge trapped in meta-stable states within the crystal lattice (Aitken, 1985; 1998). These states can have trapping lifetimes in excess of 10^8 years at ambient temperatures. This trapped-charge population increases with burial time; when the crystal is stimulated (e.g. with light) the electrons are able to recombine, with some giving up their stored energy in the form of visible photons (optically stimulated luminescence, or OSL, Aitken, 1998). As a result, the time elapsed since sediment grains were last exposed to light (e.g. by transport prior to burial) can be determined by measuring both the total OSL signal and the OSL sensitivity, and by estimating the energy flux from ionizing radiation to which it has been exposed since burial. Over the past 15 years, OSL techniques have become increasingly successful in estimating the burial

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dose (or more formally, the equivalent dose; e.g. Duller, 2004; Olley *et al*., 2006; Pawley *et al*., 2008).

Luminescence techniques provide an independent dating method which has very few constraints on the type of material to be dated, depending on the circumstances. They can be used to date sediments as young as a few years and as old as to several hundred thousand of years. Loess is very suitable for luminescence dating because the (long distance) aeolian transport of the dust particles ensures a complete zeroing of the latent luminescence signal at the time of deposition. Loess is a very homogeneous material with a relatively high radionuclide concentration (compared to more sandy sediments) – the annual dose rate in loess is quite high (about ~3 Gy/ka) compared to typical quartz-rich sands (~1 Gy/ka). As a consequence the upper dating limit of the method is lower in loess than in sands. It is widely accepted that quartz optically stimulated luminescence (OSL) dating using the single-aliquot regenerative dose (SAR) procedure gives reliable ages, but these signals suffer from saturation effects (usually at doses of about 250 Gy) imposing an upper dating limit of about 80-100 ka on quartz OSL dates from loess.

The use of proxy records to establish a chronology for a loess site relies on the assumption that stratigraphic boundaries or significant changes in the proxy record can be correlated with marine oxygen-isotope stage (MOIS) boundaries from the deep-sea record (e.g. Martinson *et al*., 1987). Age models for a site are often based on its pedostratigraphy. It is assumed that the Holocene soil correlates with MOIS 1 (0-12 ka), the underlying loess with MOIS 2 (12-24 ka), MOIS 3 (24-59 ka) and MOIS 4 (59-74 ka) and the last interglacial soil with MOIS 5 (74-130 ka). Weakly developed interstadial soil horizon can be observed also in loess and is assumed to correlate with MOIS 3. Soil boundaries are thus thought to correspond to the MOIS boundaries. However, this is an assumption. There is little evidence that soil forming processes began and ended synchronously across the whole Eastern Europe area, and that their time limits coincide with the time-boundaries between interglacial and interstadial periods (as deduced from the global marine oxygen-isotope record). It is also impossible to quantify the duration of soil forming processes from pedological and morphological characteristics alone. Finally, the use of pedostratigraphic observations to construct an age model for a loess site requires an assessment of how much of the soil consists of altered parent material (pedogenic overprinting) and how much is the result of dust accumulation during interglacial/interstadial periods; this is known to be extremely difficult, if not impossible (Sun *et al*., 2000).

Here we present results for 12 samples dated at the profile at Biały Kościół obtained in the Institute of Physics Laboratory at the Silesian Technical University. Institute of Geography and Regional Development at the University of Wroclaw made all lithological characteristics.

2. EXPERIMENTAL DETAILS

Samples from the 9 m long loess profile at Biały Kościół were chosen for this study from the most characteristic profile sections. The loess section in the vicinity of the village Biały Kościół is located in an old clay-pit, several meters from the road linking the Strzelin and Ziębice. The loess profile is situated on the western slope of Oława river valley at an altitude of approximately 180 m above sea level (**Fig. 1**).

The loess cover in the surrounding area was previously described by Raczkowski (1969) and Ciszek (1997). The complete loess-soil sequence at Biały Kościół was excavated and characterized by Ciszek *et al*, (2001) and Jary *et al*, (2004a, 2004b, 2008). In those studies the profile was sampled in vertical continuous sections at close intervals (5 cm) and documented in respect of its sedimentology, palaeopedology and stratigraphy. The following analyses were conducted: grain-size distribution (laser diffraction method), magnetic susceptibility, contents of $CaCO_3$ and organic carbon. TL (11 samples)

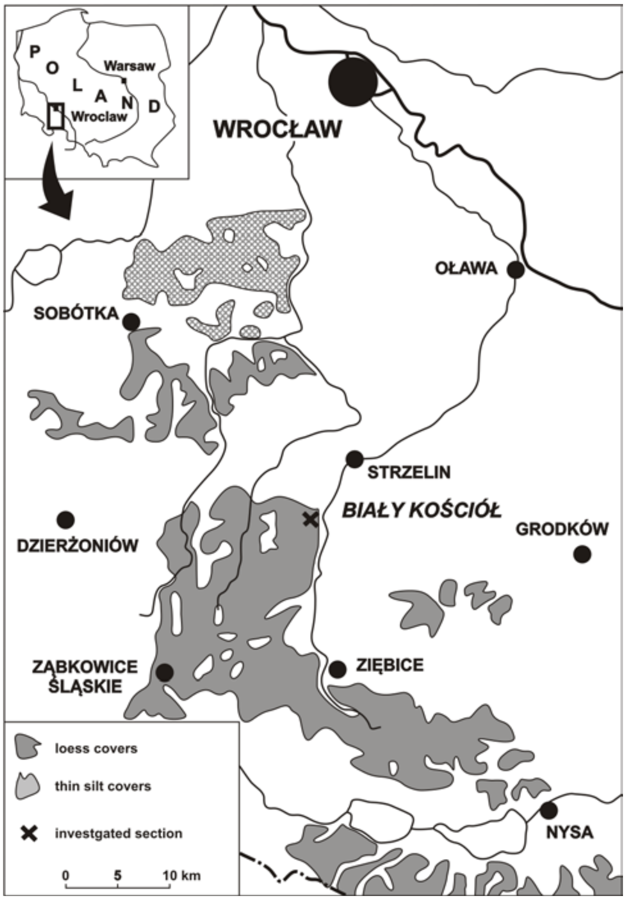

Fig. 1. *Loess distribution in western Poland.*

and OSL (2 samples) dating was performed in the past (Fedorowicz, 2006). Combined characteristics of lithostratigraphic units (**Fig. 6a**) and interpretation of periglacial phenomena were presented and discussed by Jary (2007, 2009; **Figs. 6a, 6b**). In 2009, on the basis of preliminary results of palynologic investigations presented by Komar *et al*, (2004), simplified pollen diagram of the Biały Kościół loess section was published by Komar, Łanczont and Madeyska (2009). Although the loess section was exactly the same, the results of pollen analysis suggest completely different positions of loess and soil units within the profile in comparison with previous papers of Jary *et al*, (2004a, 2004b, 2008) and Jary (2007).

The loess profile at Biały Kościół was presented several times to participants of International Symposiums (11th Polish-Ukrainian Seminar "Stratigraphic Correlation of loess and glacial deposits in Poland and Ukraine" – Wrocław, September 2001; 4th International Loess Seminar - Strzelin, October 2004; 5th International Loess Seminar – Wrocław- Srebrna Góra, September 2008). In subsequent years the loess section was cleaned and deepened. The research material has been supplemented by new results of laboratory examinations and extended field observations.

Up to now there was no possibility of finding any pieces of charcoal large enough to collect a sample for radiocarbon AMS dating, so there is no independent age control for our OSL dating results.

Bulk samples with mass ~800 g were prepared for gamma spectrometry. The samples were stored for about 3 weeks to ensure equilibrium between gaseous ^{222}Rn and ^{226}Ra in the ^{238}U decay chain. The concentrations of the radionuclides in the U, Th and K series decay chains were measured using a high-resolution gamma HPGe detector manufactured by Canberra. The samples were counted for 24 hours. The cosmic rays dose-rate to the site follows the calculations suggested by Prescott and Hutton (1994). The dry dose rates (Adamiec and Aitken, 1998) were adjusted for water content, following Aitken (1985). We assumed that the average water content was no higher than 15% and consequently used a value of 10±5% for further calculations. Based on these data, the average dose rates were calculated and are presented in **Table 1**.

The sample tubes for equivalent dose determination were processed under subdued orange light conditions. Material in the outer part of the tube, potentially exposed to daylight during sampling was removed. The remaining ~300-400 g of material was used for quartz extraction using conventional sample preparation techniques (e.g. Aitken, 1998). The samples were first sieved to obtain the 90-125 µm fraction; this fraction was then treated with hydrochloric acid (20%) and H_2O_2 (20%) to remove carbonates and organic material, respectively. Between every step the samples were washed with distilled water. Finally the grains were etched for 60 min in a 40% hydrofluoric acid to remove the outer ~10 µm layer which absorbed a dose from alpha radiation (Aitken, 1985, 1998) and to completely remove any feldspar grains. After etching they were washed in HCl (20%) to remove any precipitated fluorides and the purified quartz extracts were mounted on stainless steel discs using silicone oil.

The quartz extracts were checked for purity by means of an IR-test. For every sample a natural aliquot went through a cycle of preheating at 260°C for 10 s followed by IR-stimulation and stimulation with blue-light both at 125°C for 40 s. Then a regenerative dose of ~30 Gy was given and the preheating-stimulation cycle repeated. The samples were considered sufficiently pure when the natural and regenerated signal ratios of the IRSL to the blue-light stimulated luminescence were $\leq$ 10%. If a sample failed the IR-test, the 40 min etching in 40% HF and the test were repeated.

An automated Daybreak 2200 TL/OSL reader (Bortolot, 2000) was used for the OSL measurements of multi-grain aliquots, each of ca. 1 mg. The grains were attached to stainless steel discs by silkospray. Daybreak 2200 uses blue diodes (470±4 nm) delivering about 60 mW/cm^2 at the sample position after passing through BG39 filters. This reader for infrared stimulation used red diodes (880±4 nm) delivering about 68 mW/cm^2 at the sample position. Laboratory irradiations were made using a calibrated ^{90}Sr/^{90}Y beta source mounted onto the reader

Table 1. *Specific radiation of concentrations of radionuclides measured in samples from Biały Kościół and dose rates calculations for these samples.*

Depth (m)	Sample name	U-238 (Bq/kg)	Th-232 (Bq/kg)	K-40 (Bq/kg)	Dose rate (Gy/ka)
9	BK1	22.4±0.6	37.5±0.9	485±14	2.44±0.07
8	BK3	23.8±0.8	34.2±1.3	498±17	2.46±0.07
7.4	BK5	28.0±0.9	42.6±1.5	534±19	2.77±0.08
6	BK6	30.1±0.5	44.9±0.7	586±15	3.00±0.08
5.7	BK7	28.1±0.9	44.0±1.5	581±20	2.93±0.09
4.9	BK9	29.6±0.6	41.5±1.0	561±16	2.87±0.08
6.5	BK11	29.0±0.6	43.8±1.0	572±16	2.92±0.08
4	BK13	29.5±0.6	40.6±1.0	580±16	2.91±0.08
3	BK16	30.1±1.0	40.6±1.6	532±19	2.81±0.08
2	BK18	30.0±1.0	38.1±1.6	492±17	2.67±0.08
1	BK20	30.9±0.9	40.5±1.5	526±19	2.85±0.08
0.5	BK21	32.1±0.6	42.2±1.0	541±16	2.97±0.08

with dose 5.27 Gy/min.

For the samples, equivalent doses were determined using a single-aliquot regenerative-dose (SAR) protocol (Murray and Roberts, 1998; Murray and Wintle, 2000).

The OSL SAR protocol which was used in our measurements contained following steps:

1) Irradiation with the regenerative beta dose D_i
2) Preheat at the temperature 260°C for 10 s
3) Blue light stimulation at the temperature 125°C for 30 s
4) Irradiation with the test dose D_t (10% of the natural dose. but not less than 5 Gy)
5) Preheat at the temperature 220°C for 0 s
6) Blue light stimulation at the temperature 125°C for 30 s

A preheat plateau test was performed to establish the most appropriate preheat temperature. Preheat temperatures were varied from 180°C to 300°C in 20° steps. No systematic variation in D_e with preheat temperature was observed (**Fig. 2**). This is good evidence that thermal transfer from incompletely emptied traps should not be a problem, and therefore a 260°C, preheat was chosen for all subsequent measurements.

Preheating the sample can also cause recuperation of the OSL signal (Aitken, 1985; Aitken and Smith, 1988). To test for this a 0 Gy regenerative dose step was incorporated into the SAR protocol (Murray and Wintle, 2000). The luminescence signal should then be zero (this is known as the 'recuperated' luminescence signal). Any grains for which this sensitivity-corrected recuperated signal was >5% of the corresponding natural signal were rejected (Murray and Olley, 2002). The recuperation for all measured aliquots was lower than 5%. Typical results for one of our samples are presented in **Fig. 3**.

In the SAR protocol, sensitivity changes which may occur from one measurement cycle to another are measured by the OSL response to a small test dose (Murray and Wintle, 2000). The corrected OSL ratio (regenerated OSL response divided by the OSL response to a fixed test dose) should be independent of prior dose or thermal treatment. This is tested by repeating a particular regenerative dose after various larger values have been used, and comparing the ratio of the two regenerated sensitivity-corrected OSL responses (known as a recycling ratio); this ratio should ideally be close to unity (Murray and Wintle, 2000). The recycling ratios for all measured aliquots were close to unity, results for one of our samples are shown in **Fig. 4**. To ensure our subsequent analyses were reliable, we only accepted results from aliquots for which the differences in luminescence response to test doses during the SAR protocol were not greater than 10%.

A dose recovery test (Wallinga *et al.*, 2000) was run on 175 aliquots from 5 samples (35 aliquots for each measured sample). The aliquots used in this test were first bleached with blue light for 100 s (at a room temperature) and after a pause of 10000 s were bleached for another

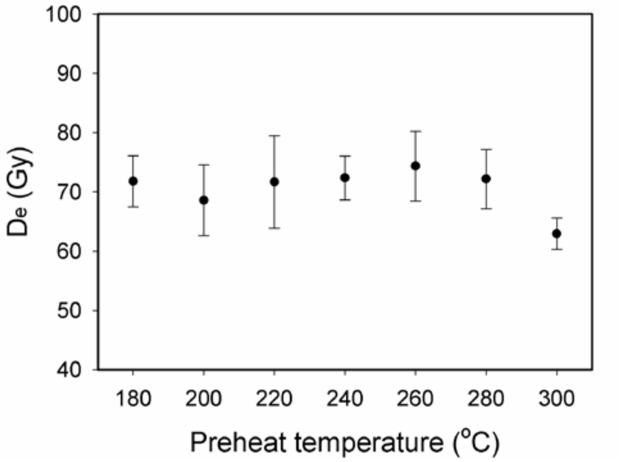

Fig. 2. *The preheat plateau test for sample BK16. The excitation time was 30 s and the excitation temperature was 125ºC. Five aliquots were used for each point.*

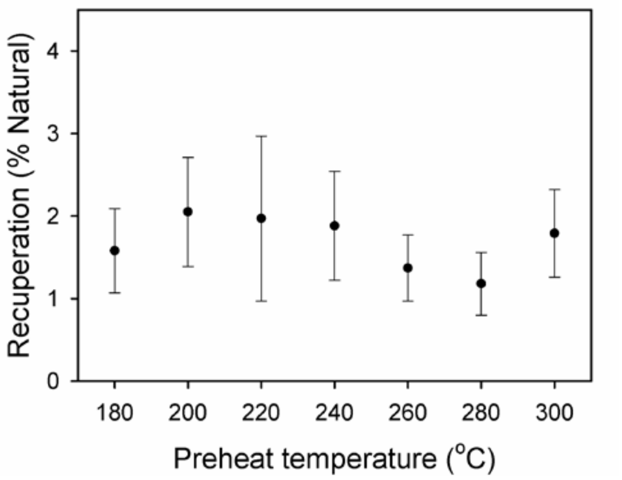

Fig. 3. *The recuperation measurements test for sample BK16. Excitation time was 30 s and excitation temperature was 125ºC. Five aliquots were used for each point.*

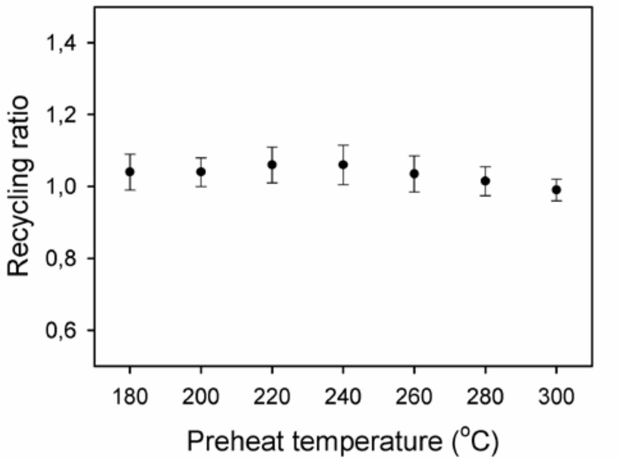

Fig. 4. *The recycling ratio measurements test for sample BK16. Excitation time was 30 s and excitation temperature was 125ºC. Five aliquots were used for each point.*

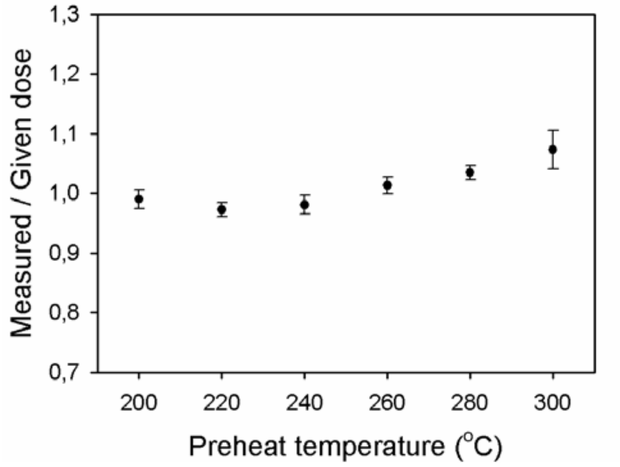

Fig. 5. *The dose recovery test for sample BK20. Excitation time was 30 s, excitation temperature was 125ºC. Five aliquots were used for each point.*

100 s. Then a laboratory dose of value similar to the equivalent dose for each sample was given and measured using the SAR protocol. The examples of the ratios between the given and measured doses are shown in Fig. 5 as a function of preheat temperature. The measured dose reproduces the known given dose at all preheat temperatures from 180ºC to 300ºC. For younger samples the recovery doses were close to unity, but for older samples (BK5, BK6) this ratios were about 0.93, for the oldest sample BK1 this ratio was 0.80. Based on the results of the preheat plateau and dose recovery tests a preheat at 260°C for 10 s and a cut-heat at 220°C were chosen for all further analyses.

3. RESULTS AND DISCUSSION

Description and interpretation of the main lithostatigraphic units (Fig. 6a and Fig. 6b)

The Biały Kościół loess-soil sequence consists of four lithostratigraphic units developed during the last interglacial-glacial cycle (Eemian plus Weichselian): two polygenetic fossil soils (fossil soil sets) and two calcareous loess units. In the top of the younger loess unit recent soil has developed.

I lithostratigraphic unit

In the lower part of the investigated sequence. polygenetic pedocomplex (fossil soil set) with well-developed Bt (illuvial) horizon was formed on a heterogenic sandy-clay substrate. The upper part of the pedocomplex consists of an accumulation (A) and elluvial (E) horizons and a clearly visible transitional horizon EA with charcoal clusters. There is evidence of periglacial and other deformational processes within the palaeosol set (e.g. cryo-desiccation cracks, desert pavements) which demonstrate the complex history of the pedocomplex. In the final phase of the development the top of pedocomplex has been transformed by gley and gelifluction processes.

The palaeosol set was probably formed during three intensive soil formation stages: Eemian Interglacial

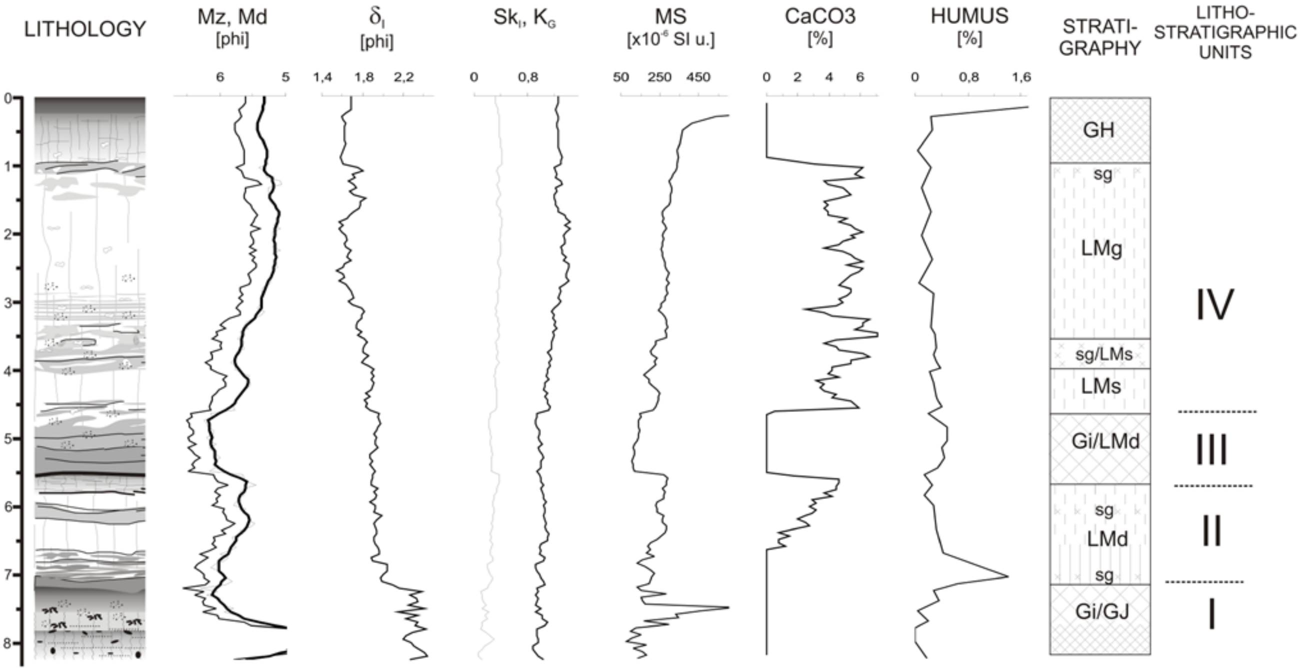

Fig. 6a. *Late Pleistocene loess section at Biały Kościół (modified after Jary, 2007). Md acc. to Inman (1952); Mz, δI, SkI, KG acc. to Folk and Ward (1957); MS magnetic susceptibility. Explanations of lithologic and stratigraphic signatures as in figure 6b.*

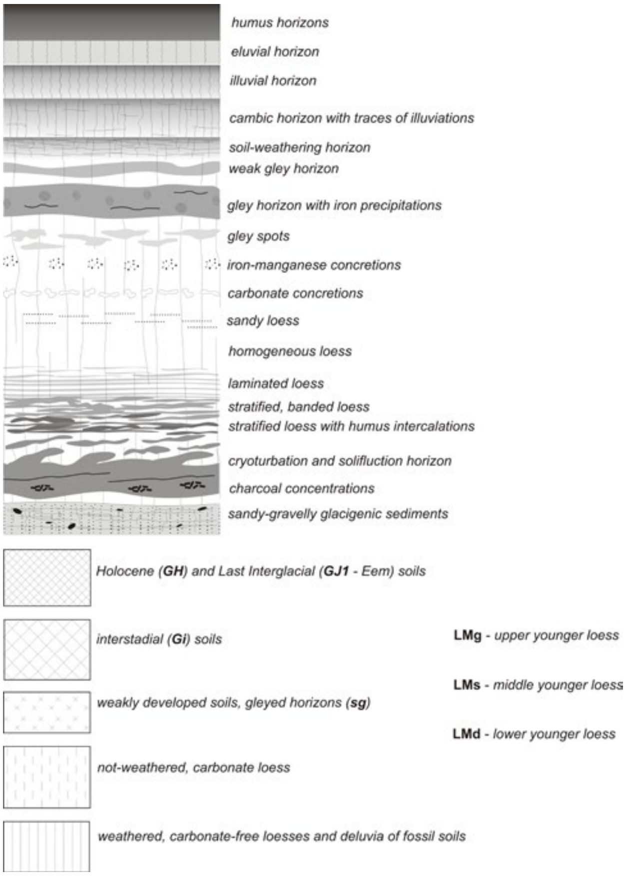

Fig. 6b. *Explanations of lithologic and stratigraphic signatures used in geological profiles.*

(MOIS 5e). Brörup and Odderade Interstadials (MOIS 5c and 5a). Cold periods, which interrupted three stages of pedogenesis are correlated with Herning (MOIS 5d) and Rederstall (MOIS 5b) Stadials. Eemian-Early Weichselian soil complex at Biały Kościół was formed probably during 2-3 stages of forest-type pedogenesis. The final steppe soil forming phase, characteristic for eastern part of Poland, has been weakly developed. This pedocomplex is usually correlated with Lommatzscher Komplex in Germany (Zöller and Semmel, 2001), PK III+PK II in Czechia (Kukla, 1975), Nietulisko I (Jersak, 1973) and Gi+GJ1 (Maruszczak, 1991; 2001) in Poland. This pedocomplex also can be correlated with Horohiv Pedocomplex in western part of Ukraine (Bogutsky, 1986; 1987) and Mezin Complex in Russia (Velichko, 1990).

II lithostratigraphic unit

Above the pedocomplex lower loess unit (LMd – lower younger loess accumulation Maruszczak, 1991, 2001) was deposited probably during the Lower Pleniweichselian (MOIS 4). This calcareous loess is about 1.5 m deep. Some weak tundra-gley horizons can be distinguished within this lithostratigraphic unit.

III lithostratigraphic unit

In the top of LMd successive fossil soil complex (Gi/LMd accumulation Maruszczak, 1991, 2001) was developed with strong evidence of tundra-gley soil processes formation.

Gi/LMd soil is the most important fossil soil (soil complex) within the Weichselian loess succession. It separates two main stages of loess accumulation during the Last Glacial. Morphological and genetic differentiation and varied preservation of this fossil soil is the major feature of this unit. Therefore it is sometimes difficult to make stratigraphic correlations between the particular sections. It is the only carbonate free soil in the Last Glacial loess sequence at Biały Kościół (**Fig. 6a**). Substrate of this soil is characterized by high indexes of chemical weathering indicating a considerable role of transformation by soil processes. However, morphological characteristic and presence of periglacial phenomena suggest tundra-gley type of pedogenesis. Chronostratigraphic position of Gi/LMd soil is usually correlated with final phases of MOIS 3 (Hengelo and Denekamp Interstadials), contrary to some Polish authors (e.g. Maruszczak, 1991, 2001) who correlate this soil with the onset of MOIS 3 (Oerel and Glinde Interstadials). Jary (2007) suggests that chronostratigraphic position of Gi/LMd soil complex should be correlated with the whole MOIS 3. Climatic changes during MOIS 3 on investigated loess areas in SW Poland were probably not rapid and/or significant enough to evoke effective processes of loess accumulation. The top of tundra-gley soil (Gi/LMd) was deformed by gelifluction, frost heave and other periglacial processes. The Gi/LMd pedocomplex is usually correlated with Lohner Boden in Germany (Zöller and Semmel, 2001). PK I in Czech (Kukla, 1975). Komorniki (Jersak, 1973) in Poland. Dubno in western part of Ukraine (Bogutsky, 1986, 1987) and Bryansk in Russia (Velichko, 1990).

IV lithostratigraphic unit

Over the gelifluction horizon the younger middle loess (LMs acc. Maruszczak, 1991, 2001) occurs (about 0.4 m depth). This loess subunit was probably deposited in the initial phase of Upper Pleniweichselian (MOIS 2). The weak tundra-gley soil with numerous ferruginous concretions was developed in the top of LMs. Above the LMs soil younger upper loess LMg (ca. 3.5 m depth) subunit occurs.

Middle younger loess (LMs) and upper younger loess (LMg) together with calcareous gley soil in the top of LMs form upper loess lithostratigraphic unit. This loess was deposited during Upper Pleniweichselian (MOIS 2). There are few other weak tundra-gley horizons (besides the mentioned gley soil in the top of LMs) within Upper Pleniweichselian loess sequence, which are evidence for short climate variations in the time of loess accumulation. Nevertheless its recognition is not unambiguous. It can be related to climate change of the loess sedimentary environment. Rapid changes of grain size composition and magnetic susceptibility (MS) within upper loess unit suggests sudden changes of environmental conditions. In the top of upper loess unit the modern brown soil has developed.

Changes in intensity in the winter winds are reflected in proxy records related to the transport process. During relatively cold periods the dust-bearing winter winds will be stronger and the deposited loess will be coarser and thicker. Thus the grain size distribution of the loess-palaeosol sequences can be used as a proxy for winter changes; several parameters have been put forward such as the median grain size (An *et al*., 1991), the median diameter of the quartz grains (Xiao *et al*., 1995) and the U ratio (mass ratio of 44-16 µm to 16-5 µm grains; Vandenberghe *et al*., 1997).

We cannot discuss the accuracy of the optical ages from the Biały Kościół site, because we have no independent age control at this location. As can be seen in **Fig. 7**, the ages obtained for the individual aliquots usually cover a large range. This may indicate the presence of grains of varying ages, as it is a loess profile it is not expected that partial bleaching is a problem here. In addition, given the homogeneity of annual dose (**Fig. 2**), along the profile, microdosimetry is considered unlikely to be the cause of the large spread of dates. There remains the question whether bioturbation might be responsible for such a state of affairs.

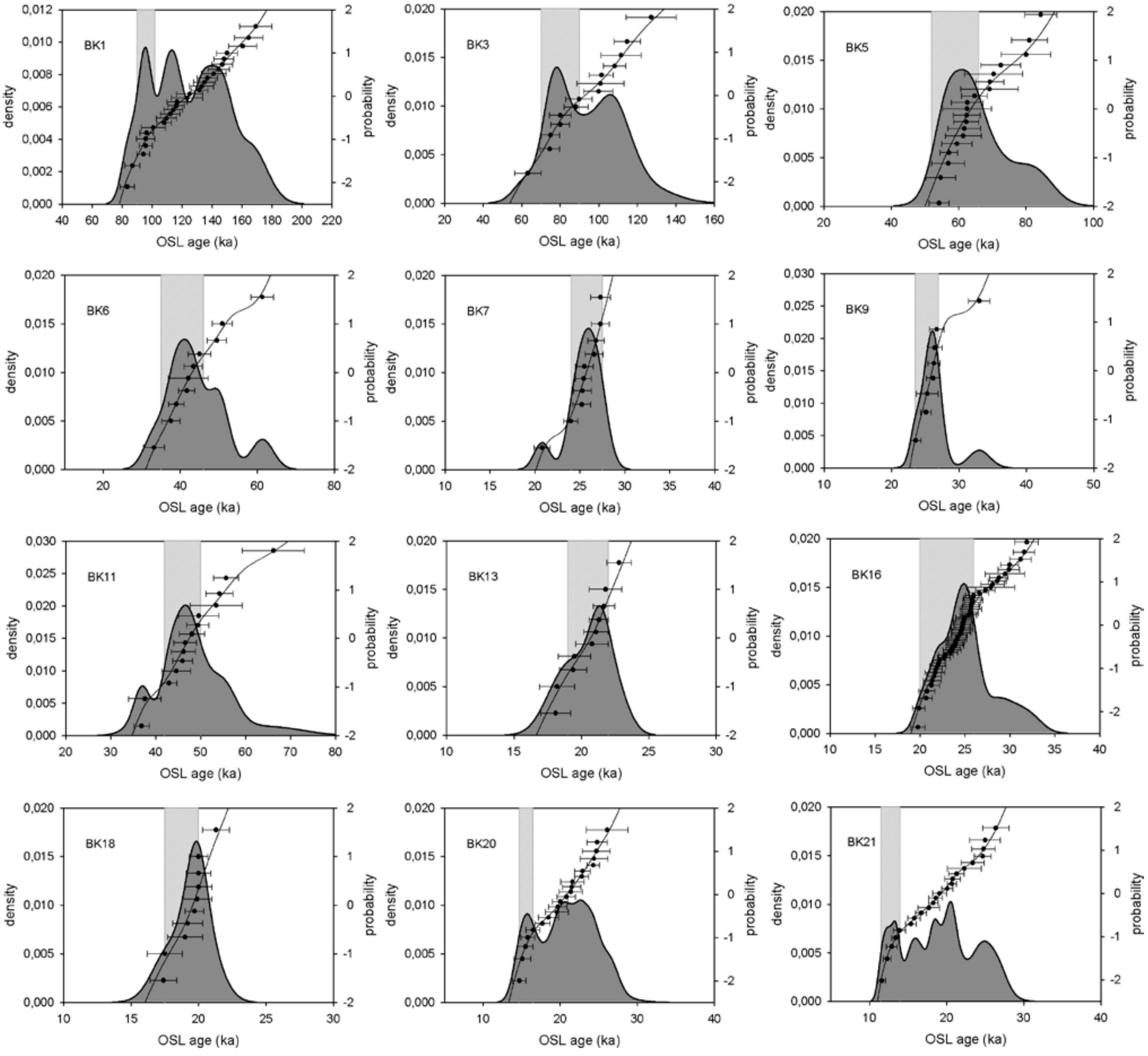

Fig. 7. *OSL age results and probability density functions for all 12 samples presented in this study.*

In order to interpret the results, we decided to use the group of the lowest ages for each sample as the one representing the grains that are certainly well bleached. The grey areas in **Fig. 7** indicate the aliquots that were taken into account when calculating the age of the individual samples. These ages are thought to define the low age limit of the samples.

OSL ages determined in such a way (**Fig. 8**) show a clearly defined increase with depth, but the OSL ages for the soil samples at the bottom part of the loess profile are slightly younger than expected from the pedostratigraphy (compare **Figs. 6a**, **6b** and **Fig. 8**) since the last interglacial soil is expected to belong to the MOIS 5 (74-130 ka, Martinson *et al*., 1987). The OSL dates obtained for the younger deposits from Biały Kościół are coincident with pedostratigraphy and indicate a high loess deposition in a relatively short period of time.

It is clear that the OSL ages obtained for quartz from the oldest samples in this loess profile still require interpretation (especially sample BK1) because of the multimodal distribution (**Fig. 7**) and lower than normal dose recovery ratio.

4. CONCLUSIONS

It is generally assumed that sediments on the Polish Loess provide continuous and detailed records of climate changes in Middle East Europe during the whole Quaternary period. A wide variety of proxy records can be used to infer climate changes and the similarity between some of these and the marine-oxygen isotope ($\delta^{18}O$) records has prompted many researchers to use such proxies as dating tools. However, to prove these assumptions through providing an absolute time scale, we have applied optically stimulated luminescence to Polish loess. Such approach would allow detection of instances where the climate records are incomplete and/or have been significantly altered by erosional, diagenetic and pedogenic processes.

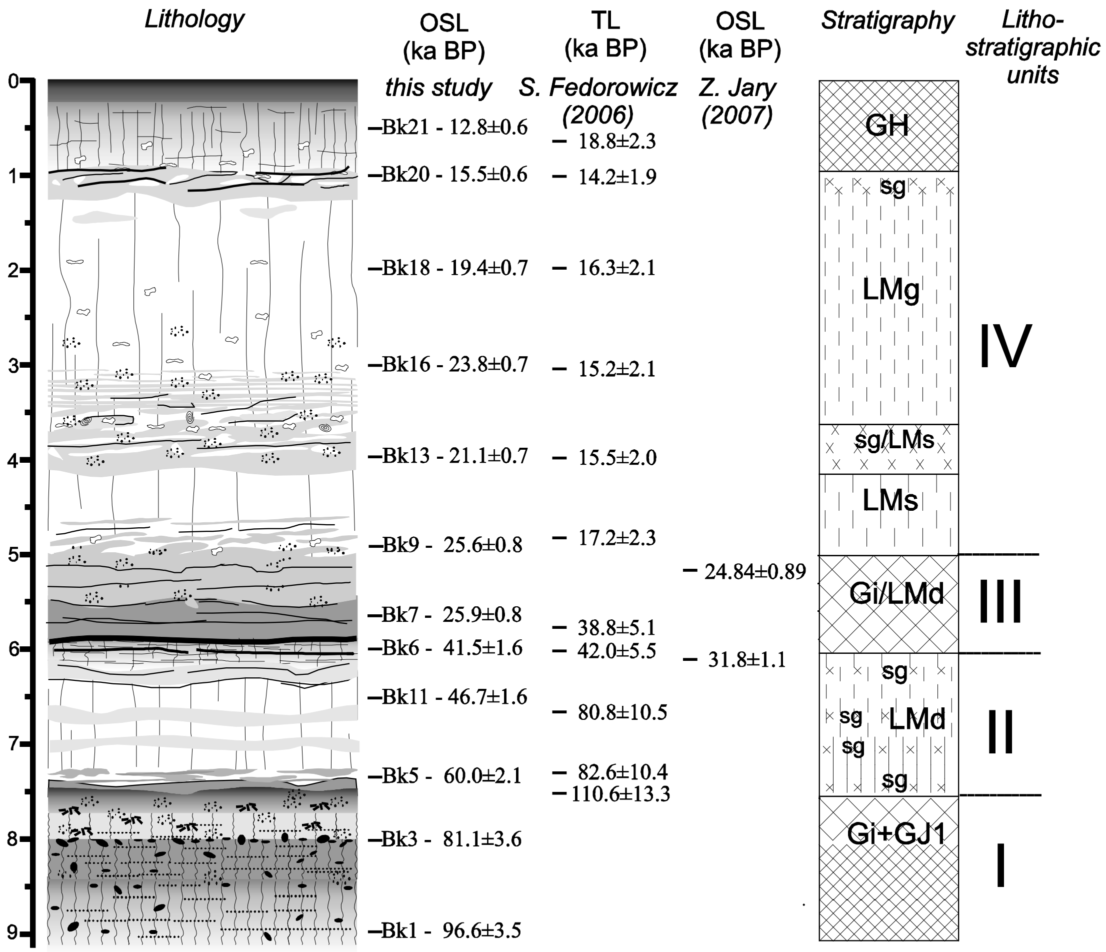

Fig. 8. *Comparison all OSL dates with previous results. Obtained OSL results in some cases are quite different as compare with the OSL (Jary, 2007) and TL (Fedorowicz, 2006) dates obtained during last 10 years by other authors and presented in previous publications relating to this loess profile.*

At present, the only technique that is able to detect disturbances in the loess record and to place the sediment sequences in an absolute time frame without the need for assumptions concerning proxy records is luminescence dating. Over the past few years, several successful studies have been reported but the full potential of the application of luminescence dating techniques on loess (mainly Chinese loess), especially in high-resolution studies, has not yet been reached. This is one of the first comprehensive studies using the OSL signals from sand-sized quartz grains extracted from Polish loess. In this way it was hoped that a contribution to the development of improved absolute chronologies for Polish loess can be made.

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