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GLACIAL AND PERIGLACIAL TRANSFORMATION OF PALAEOKARST IN THE LUBLIN-VOLHYNIA REGION (SE POLAND, NW UKRAINE) ON THE BASE OF TL DATING

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Abstract: Distinctly diverse results of TL dating are obtained for the deposits with similar lithofacial features but filling morphologically differentiated karst palaeoforms (dolines, pipes, pockets). The infillings of dolines and pipes are mostly of the Saalian age. Based on sedimentological analysis, their formation conditions are related to sub- or/and terminoglacial environment. The age obtained for all infillings of pockets is underestimated in comparison with lithostratigraphic data. A close genetic relationship between these forms and periglacial conditions seems to indicate that the reduction of TL signal is mostly influenced by the disintegration of grains resulted from the repeated freezing and thawing of glaciogenic deposits. A considerable influence of frost weathering on the decrease of thermoluminescence intensity of mineral grains is indirectly confirmed by the results of experimental investigations consisting in the repeated TL measurements of pockets' infillings after successive freeze-thaw cycles.

Keywords: Thermoluminescence dating, palaeokarst, glacial transformation, periglacial transformation, Lublin-Volhynia karst region, Poland SE, Ukraine NW

1. INTRODUCTION

Thermoluminescence dating methods have been used in the Quaternary geology for more than fifty years. The results of TL dating, considered as an indicator of deposit age, are used for construction of local or regional stratigraphic schemes (among others Boguckyj and Łanczont, 2002; Dolecki, 1995; Frechen,1999; Frechen *et al.,* 2003; Harasimiuk *et al.,* 1988; Kukla, 1975; Lindner *et al.,* 1985, 1991; Maruszczak, 1986). Considerably more rarely they are used in litho- and morphogenetic discussions, and interpreted not as the age of deposit but of a process, which transformed this deposit (Dzierżek and Stańczuk, 2006; Raukas and Stankowski, 2005; Stankowski, 2001a, b). However, such approach requires evidence for postdepositional zeroing of the TL signal to residual level as a result of mechanical (friction effect), thermal, and/or insolar influences on mineral grains (Fedorowicz, 2006). Moreover, the TL ages divergent from stratigraphic schemes (i.e. incompatible with expectations) are rarely examined in terms of postdepositional transformation of deposits. They are most often omitted in palaeogeographic interpretations or treated as "measurement errors". In extreme cases, the divergences of the TL results with the "expected" age of the deposit (its lithostratigraphic situation determined on the basis of mineral-petrographic, sedimentologic and other features) can raise doubts about the reliability of thermoluminescence dating in general.

The authors of this paper encountered such a research problem when they tried to interpret the TL ages obtained for the series of samples taken from the infillings of palaeokarst forms occurring in the Lublin-Volhynia karst region (SE Poland, NW Ukraine). These forms and deposits are well examined in respect of geology and micromorphology (Dobrowolski, 2006). The obtained results of TL dating for morphometrically different forms (dolines, pipes, pockets) were distinctly differentiated in spite of similar lithofacial features of the infillings. All

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infillings of pockets gave the ages which are underestimated in comparison with lithostratigraphic data. Direct genetic relationship between these forms and periglacial conditions suggests that the reduction of TL signal was mostly caused by the disintegration of grains resulting from repeated freezing and thawing of glaciogenic deposits.

2. SETTING OF THE LUBLIN-VOLHYNIA KARST REGION

The Lublin-Volhynia karst region (**Fig. 1**) encompasses the Lublin-Volhynia uplands with the Polesie situated in their foreland, and is the largest area of the continuous occurrence of unique chalk karst in Europe (*vide* Rodet, 1991). Its morphological and hydrogeological identity is conditioned by lithological features of carbonate rocks developed as chalk or marl facies. Little resistance of these rocks to destruction results in the absence of underground water drainage typical of classic karst; relief differences are also important (Dobrowolski, 1998; Harasimiuk, 1975; Lomaev, 1979; Maruszczak, 1966).

The state of preservation of palaeokarst forms and deposits representing successive cycles of development is different. However, it is possible to distinguish several main stages of activity of karst processes (from Palaeogene to Holocene) related to the changes of climatic conditions and/or phases of tectonic activity of the area (Dobrowolski, 2006). The Neopleistocene dolines, pipes, and pockets with glaciogenic infillings of the Saalian age are the most frequently found and best-examined palaeokarst forms. In many cases, they have features of post- or syngenetic transformation (Dobrowolski, 2004, 2006).

The Neopleistocene palaeokarst forms are mostly filled with the following deposits: (1) massive diamictic sands representing mass flows in sub- and/or terminoglacial environment or (2) horizontally laminated, sandy-silty rhythmite settled from suspension in ephemeral paraglaciolimnic water bodies. The occurrence of bottom massive clays, identified as residuum of weathered carbonate rocks, is a typical feature of all palaeokarst infillings. Deformation structures (involutions) occurring in the deposits filling the majority of shallow pockets prove cryogenic transformation of primary glaciogenic infillings (Dobrowolski, 2006).

3. TL DATING OF PALAEOKARST INFILLINGS

Methodology

TL dating was completed in Gdańsk University. The dose rate (D_r) was measured with the MAZAR 95 scintillation spectrometer. Dry deposit was placed in plastic Marinelli containers with a volume of 1.5 dm^3 and placed in the protective chamber of the spectrometer. 20 measurements were made, each of which lasted 2000 s. The sum of impulses in channels determined the concentration of the three nuclides: ^{226}Ra, ^{232}Th and ^{40}K. The values obtained were converted into dose rates for alpha, beta and gamma radiation. Corrections were made to account for earlier measurements of deposit humidity (Aitken and Xie, 1985. The cosmic radiation dose (d_c) was added to

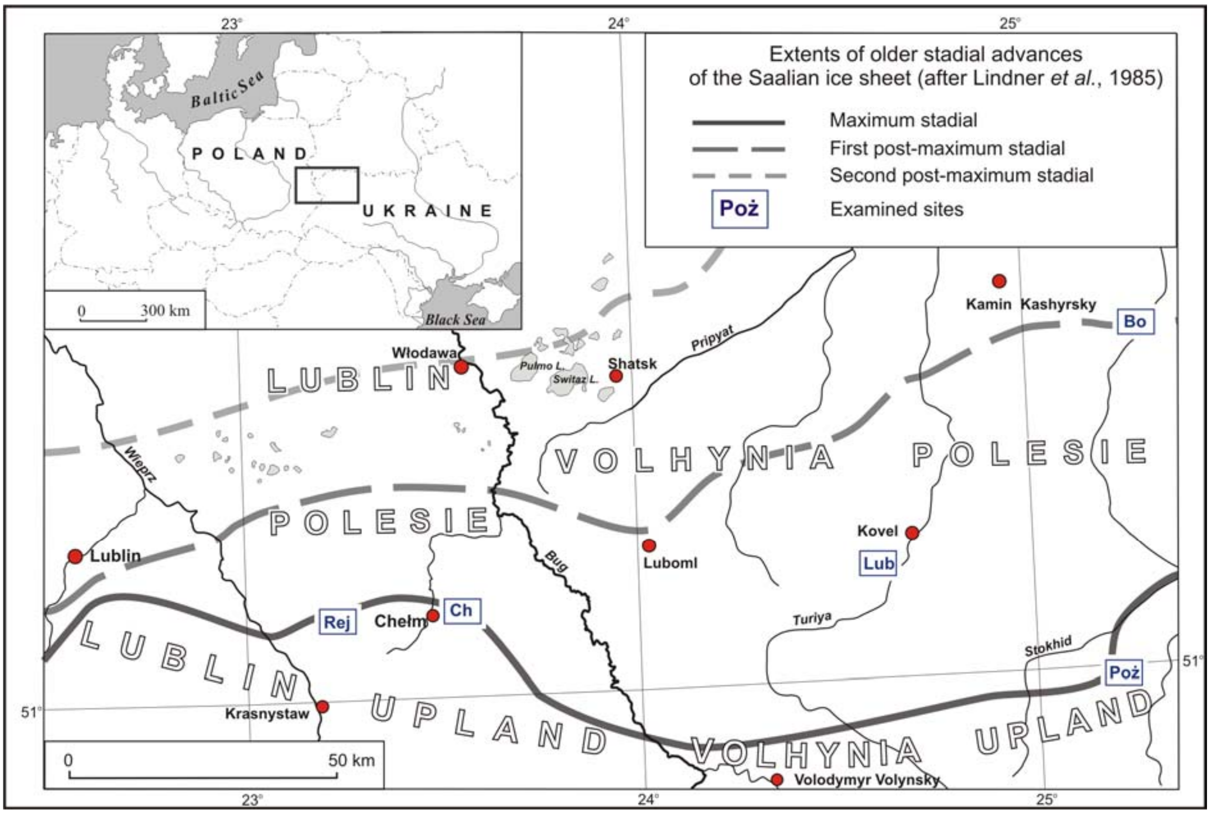

Fig. 1. *Location sketch of the studied area with the distribution of the examined sites: Rej – Rejowiec, Ch – Chełm, Lub – Lubliniec, Bo – Borki, Poż – Pożarki.*

the estimated alpha, beta and gamma radiation doses. The uncertainty of the dose rate estimation is about 3%. For the estimation of the equivalent dose from the total mass of the sample, a fraction of grains with a size of 80–100 µm was separated. The grains, isolated with the sieve method, were subjected to initial extraction to remove the external layer from the quartz grains and to clean their surface. The grains are then treated with 10% HCl acid and 40% HF acid within 24 hours. The samples were rinsed with distilled water and dried at room temperature. The absorbed dose ED was measured with the multi aliquot regeneration method (Fedorowicz, 2006). For each aliquot of extracted grains from 10 to 20 diagrams of the glow curve were prepared, as well as a plateau check test. Its results shed light on the accuracy of the initial TL value estimation and give some hints whether the laboratory time of sample bleaching was optimal (Bluszcz, 2000). The sensitivity of grains is also checked by way of complementary measurements according to the technique used in additive method. The uncertainty of ED determination is about 10%. Glow curves are done with the use of a reader-analyser type RA'94. The samples are heated up to 400°C with the heating speed of 8°C per second.

Results

In total, 25 TL ages were obtained using regeneration method for the typologically differentiated Neopleistocene palaeokarst forms (dolines, pipes and pockets) occurring at five sites (**Fig. 1**). In several cases, the equivalent dose for the same samples was determined by additive method, which gives additional opportunity to estimate a potential change of grain sensitivity (saturation effect – **Table 1**).

The obtained results indicate the occurrence of distinct regularities related to different genetic types of palaeokarst forms, and allow us to formulate the following generalizations:

1) Glaciogenic deposits (sandy-silty rhythmite or diamictic sands) filling relatively deep pipes and dolines (>2m) are of the Saalian age (≈ 250-300 ka BP).
2) The TL ages of the infillings of shallow pockets (<1m) are very young and indicate that the infillings were deposited during the Pleniglacial.

Table 1. *Results of thermoluminescence dating (R – regeneration method; A – additive method) of the materials filling the karst palaeoforms in the sites from Lublin-Volhynia karst region, after Dobrowolski (2006)*

Sample	Depth (m)	Lab.No UG -	d_α (Gy/ka)	d_β (Gy/ka)	d_γ (Gy/ka)	d_c (Gy/ka)	D_r (Gy/ka)		ED (Gy)	TL Age (ka BP)
POCKETS										
Ch-11/1	0.60	5650	0.03	0.50	0.22	0.14	0.89 ± 0.03	**R**	164.9 ± 15.4	185.3 ± 21.4
Ch-11/2	0.30	5651	0.03	0.62	0.40	0.15	1.19 ± 0.03	**R**	83.5 ± 7.9	70.0 ± 9.8
Ch-11/3	0.20	5652	0.03	0.71	0.44	0.15	1.33 ± 0.04	**R**	94.3 ± 9.8	70.9 ± 9.6
Ch-35/1	0.90	5657	0.01	0.44	0.16	0.14	0.75 ± 0.03	**R**	59.4 ± 6.9	79.2 ± 10.2
Ch-35/2	0.70	5658	0.02	0.39	0.20	0.14	0.75 ± 0.03	**R**	64.9 ± 8.6	86.5 ± 11.6
Poż- 2/1	0.50	5845	0.01	0.64	0.73	0.14	1.52 ± 0.05	**A**	59.0 ± 6.6	38.1 ± 4.4
								R	56.4 ± 6.4	37.1 ± 4.0
PIPES										
Ch-1/1	2.00	5750	2.06	2.65	1.56	0.12	6.61 ± 0.2	**R**	>650	>101.7
Ch-1/2	1.80	5751	0.99	1.96	0.98	0.12	4.04 ± 0.2	**R**	1020 ± 120	252.1 ± 39
Ch-1/3	1.20	5752	0.57	1.13	0.52	0.13	2.35 ± 0.1	**R**	660 ± 70	280.7 ± 42
Ch-1/4	0.50	5753	0.26	0.59	0.28	0.14	1.27 ± 0.1	**R**	314.0 ± 32	247.0 ± 37
Ch–2/1	2.00	5763	0.00	0.28	0.14	0.13	0.55 ± 0.03	**A**	129.0 ± 13	234.5 ± 25
								R	108.8 ± 11	192.8 ± 20
Ch–9/1	1.20	5762	0.01	0.53	0.36	0.14	1.04 ± 0.04	**A**	333.8 ± 32	321.0 ± 33
								R	279.8 ± 28	263.3 ± 29
Lub-1/1	2.00	5659	0.01	0.25	0.10	0.14	0.50 ± 0.03	**R**	178.0 ± 17	356.0 ± 47
Lub-1/2	1.00	5843	0.01	0.42	0.63	0.13	1.19 ± 0.03	**A**	528.7 ± 52	444.2 ± 66
								R	315.3 ± 32	264.9 ± 37
Bor-1/1	2.00	5870	0.08	1.39	0.86	0.10	2.51 ± 0.08	**R**	748.8 ± 62	298.3 ± 26
Rej-1/1	1.20	4201	1.45	2.37	1.31	0.13	5.26 ± 0.07	**R**	>650	>123.5
Rej-1/2	0.90	4202	0.40	1.11	0.47	0.14	2.11 ± 0.06	**R**	650 + 68	306.7 + 46
Rej-1/3	0.20	4203	1.73	1.61	1.31	0.15	4.79 ± 0.08	**R**	>650	>135.5
DOLINES										
Ch–24/1	1.00	5768	0.01	0.68	0.36	0.14	1.19 ± 0.04	**A**	>120.0	>100.8
								R	305.2 ± 30	256.5 ± 27
Ch–24/2	2.00	5769	0.01	1.05	0.67	0.13	1.86 ± 0.06	**A**	488.4 ± 49	262.6 ± 28
								R	508.3 ± 51	273.3 ± 29

d_α, d_β, d_γ, d_c – α, β, γ, cosmic dose rate, D_r – dose rate, ED – Equivalent Dose

4. DISCUSSION

TL ages of infillings of the dolines and pipes vs. sedimentological analysis

The defined age of dated samples, indicating the Saalian age of deposits, is consistent with lithostratigraphic interpretation (Dobrowolski, 2006; Dobrowolski *et al.,* 1995; Harasimiuk, 1975; Harasimiuk *et. al.,* 1981; Rzechowski, 1962). The exceptions to the principle, found in several cases, require a comment giving the basis for morphogenetic interpretation. The following facts draw attention:

1) The undefined age of the bottom massive clays (Ch-1/1, Rej-1/1). Taking into account weathering origin of the clay (= karst crust), the undefined age seems to be the only acceptable result from the methodic point of view because the aggradation of this material occurred without contact with sunlight. Exposure to sunlight is necessary for the reduction of energy contained in grains, which is measured during luminescence measurements.
2) Divergence of the ages obtained for the sample Lub-1/2 by different methods (the ED value obtained by additive method is almost two times higher).
3) Very low dose rate (D_r) of the sample Lub-1/1. Determination of the equivalent dose accumulated by this sample, reaching 900 Gy, indicates that the growth curve is linear.

The most probable explanation of the problems mentioned in point (2) and (3) is the occurrence of mixed-age (Tertiary and Quaternary) grains in the samples. Such an interpretation seems to be indirectly confirmed by the high content of local Palaeogene and Neogene material commonly found in glaciogenic deposits of this area (among others Harasimiuk *et al.*, in press; Lomaev, 1979; Mojski, 1968; Rühle, 1933). Moreover, the admixture of older (Palaeogene?) grains in the sample from a depth of 2.0 m (Lub-1/1) is probably greater than in the sample from a depth of 1.0 m (Lub-1/2). Great divergence between the TL ages obtained using additive and regeneration methods (sample Lub-1/2) indicates that sensitivity of the examined grains changed.

The interpretation of TL ages of the deposits filling the Neopleistocene pipes and dolines presented above is consistent with palaeogeographic reconstruction based on sedimentologic and micromorphologic data (Dobrowolski, 2006). Both types of palaeoforms bear traces of intensive glacial transformation (*ibid.*). Pipes were probably originally formed as evorsion hollows in chalk surface in subglacial environment. With the decreasing energy of meltwater flows the following phenomena could have occurred: (i) rapid disappearance of water flow, and appearance of mass flows of *flow till* type (= infillings composed of massive diamictic sand), or (ii) development of small water bodies of paraglaciolimnic accumulation in the dispersed drainage zones (= infillings composed of horizontally laminated sands and silts). High energy of meltwater flow was also responsible for the removal of the primary (Neogene?) infilling of dolines, to the truncation of karst-related residual clay. Therefore, karstification process in pipes, which caused the development of bottom massive clays at the contact of infilling with carbonate rock, was secondary relative to the process responsible for the development of these forms, and relatively younger from their infillings (*ibid.*).

TL ages of infillings of the pockets vs. sedimentological analysis

Uncritical interpretation of the obtained dating results suggesting the Plenivistulian age of the infillings of pockets is contrary to the current geologic knowledge. These deposits are related to the maximum stadial of the Saalian Glacial (Lindner, *et al.* 1985, 1991; Harasimiuk, *et al.,* in press). In respect of lithofacies they are the same diamictic sands of mass flows as the infillings of pipes and some dolines. However, they bear distinct traces of periglacial transformation (Dobrowolski *et al.,* 1995; Harasimiuk *et al.,* in press) as evidenced by the occurrence of cryogenic structures (involutions). Moreover, micromorphologic image of these deposits shows disintegration of quartz grains without distinct traces of displacement. Grain material disintegrated in such a way is described by van Vliet-Lanoë (1985) as “crushed grains” and unambiguously related to the intensive process of frost weathering in periglacial environment. Periglacial conditions are also evidenced by the incorporation of grain material in ductile chalk, which is commonly found in the deposits deformed cryogenically in permafrost active layer (Dobrowolski, 2006; Jahn, 1951; Maruszczak, 1968; van Vliet-Lanoë, 1988).

Therefore, the obtained TL ages of the pockets’ infillings should be related not to the process of their deposition but to postdepositional destruction. In order to determine the influence of frost weathering on the reduction of TL signal, we decided to perform an experiment. The samples of the pockets’ infillings were subjected to cyclic freezing and thawing, and their thermoluminescence was measured.

Experimental investigations

The influence of freezing on the thermoluminescence of mineral grains in glaciogenic deposits (mostly tills) was the subject of experimental investigations carried out by Bluszcz (1989), Fedorowicz (1994), Prószyńska-Bordas *et al.* (1988). These experiments revealed the reduction of thermoluminescence after successive freeze-thaw cycles but without distinct regularities (*ibid.*).

The authors performed similar experiments for glaciogenic deposits filling the Ch-35 pocket from the Chełm site. Four samples, each of 200 g in weight, were taken at 25 cm intervals (**Fig. 2**). Initial deposit moisture was measured. Each sample was homogenized by stirring into water, and divided into five equal sub-samples. Four of them were subjected to freeze-thaw cycles. Each cycle consisted of one freezing (twenty-four hours at a temperature of –12°C) and one thawing (twenty-four hours at a temperature of about +20°C). Thermoluminescence of each sample was measured five times, i.e. of the untreated sub-sample, and of the four sub-samples after successive five freeze-thaw cycles (**Table 2**).

The results of experiment are as follows:

- The TL intensity of samples increases with depth (except for the lowest Ch-35/4 sample);

Table 2. *Changes of the TL intensity after successive freeze-thaw cycles.*

Sample (initial moisture in %)	TL intensity after each five successive freeze-thaw cycles				
	0	5	10	15	20
Ch-35/1 (2.2%)	650±48	674±53	627±42	582±54	533±46
Ch-35/2 (3.1%)	783±64	769±70	742±61	720±58	688±63
Ch-35/3 (5.6%)	846±75	876±82	893±86	826±68	801±62
Ch-35/4 (7.9%)	654±56	682±63	642±38	596±57	573±43

- The relationship between the TL intensity and the number of freeze-thaw cycles is very similar in most samples. In three cases the TL intensity increased first, and then consistently decreased. Only in the Ch-35/2 sample the TL intensity consistently decreased after each five freeze-thaw cycles;
- Twenty freeze-thaw cycles in all samples resulted in the decrease of the TL intensity from 12 to 18% in comparison to fresh deposits. The TL intensity after 20 cycles was even more than 20% lower in comparison with that after 5 cycles. This is evidence for a considerable influence of freezing on the TL intensity, while the shape of TL glow curves remains unchanged;
- The progressive decrease of the TL signal in the successive measurements of equivalent dose indicates that the examined deposits were probably considerably "rejuvenated" as a result of cyclic freezing and thawing.

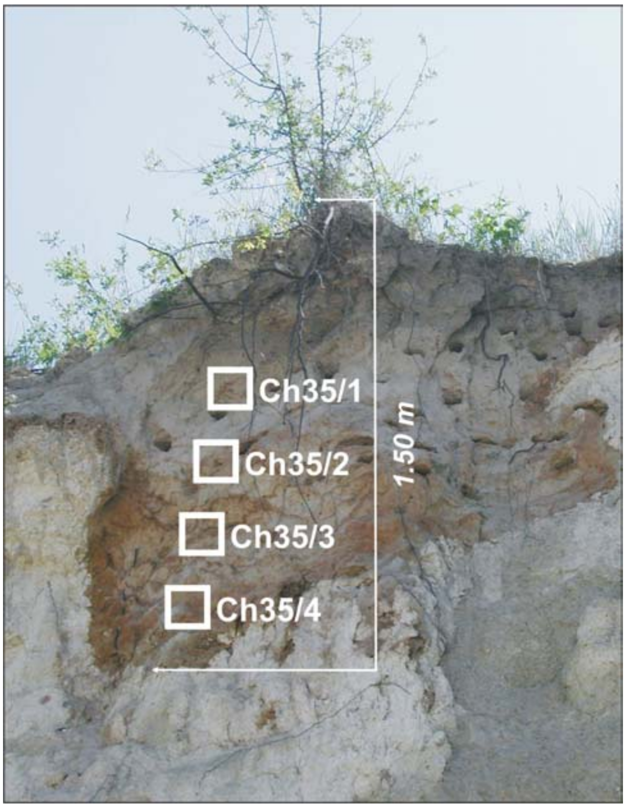

Fig. 2. *Chełm site – pocket Ch-35 – distribution of sampling sites for TL dating.*

5. CONCLUSIONS

Despite apparent inconsistency of the obtained results, we tried to interpret them basing on the current geological knowledge in the context of sedimentology, lithostratigraphy and palaeogeography. Taking into account the complexity of litho- and morphogenetic processes in glacial and periglacial environments, we are of the opinion that a "different from the norm" result of TL dating should be treated as the age of a process deforming a deposit and not the age of a deposit as such. Such approach to the results allows us to make more thorough palaeoenvironmental interpretation.

The gathered data evidence the complexity of morphogenetic processes occurring during freeze-thaw cycles in the top parts of carbonate rocks covered by unconsolidated deposits. Further studies are necessary to verify the results obtained till now.

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