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NEW ISOTOPIC DATA ON KARST DEVELOPMENT IN THE NORTHERN KRAKÓW-WIELUŃ UPLAND (SOUTHERN POLAND)

Helena HERCMAN, Michał GĄSIOROWSKI & Grzegorz SUJKA

Institute of Geological Sciences, Polish Academy of Sciences, Research Centre in Warsaw Twarda St. 51/55, PL-00818, Warszawa, Poland; e-mails: hhercman@twarda.pan.pl; mgasior@twarda.pan.pl

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Abstract: The Kraków-Wieluń Upland is one of the major palaeokarst regions in Poland. However, the stages of karst development in this area are neither well documented nor reconstructed. A series of samples from a new location in the vicinity of Raciszyn was analysed. On the basis of the results of U-series dating, four phases of speleothem deposition were distinguished: (1) older than 600 ka, (2) from more than 600 ka to 290 ka, (3) around 150 ka, and the youngest (4), younger than 3 ka. On the basis of all geochronological data from the region, eight stages of karst development were described. The structure of the oldest speleothems indicates even more stages of deposition and erosion that cannot be recognized using the $^{230}Th/^{234}U$ dating method. These results indicate that the initial creation of empty spaces in the limestone took place in pre-Pleistocene time. After 600 ka ago, climatic conditions were stable for more than 300 ka, allowing the continuous deposition of speleothems. Several episodes of erosion, deposition of clastic sediments and speleothem growth during the Middle and Early Pleistocene were described. This variability of the sedimentation regime clearly reflects climate changes during that period.

Key words: Kraków-Wieluń Upland, caves; speleothems, U-series dating, karst development.

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INTRODUCTION

Uplands are the most important regions of palaeokarst in Poland. In spite of this fact, the history of karst development has not been adequately described, especially using isotopic methods. The record of investigations is especially long in the northern part of the region (Samsonowicz, 1934), but still only a limited number of papers related to the topic exist. However, up to now, several major karstification and depositional stages were identified on the basis of sedimentological and palaeobiological data (Głazek *et al*., 1976; Głazek, 1989). The faunal remains found in karst deposits were the basis for the subdivision of the karst history of the northern part of the Kraków-Wieluń Upland into four series: Miocene, Pliocene/Pleistocene, Pleistocene and Holocene stages. One of the best described karst phenomena in the region is the area near Działoszyn and Raciszyn, where numerous outcrops and caves were intensively investigated by Głazek *et al*. (1976), Głazek and Szynkiewicz (1987) and Szynkiewicz (1993).

In the last two decades of the last century, a series of speleothem $^{230}Th/^{234}U$ dates were estimated at several localities in the Kraków-Wieluń Upland (Głazek, 1986; Duliński, 1988; Duliński and Kuliś, 1990; Pazdur *et al*., 1999; Hercman, 2000). These data indicate the existence of a minimum of five stages of speleothem deposition during the last 200 ka (Hercman, 2000). The highest frequency of speleothem deposition was dated as Eemian and MIS 3 (63–50 ka and 42–35 ka). The age information is still relatively poor and generally accepted and well documented karst development scheme was not formulated so far.

The intensive exploitation of limestone in quarries near Działoszyn (Kraków-Wieluń Upland, Poland) creates a new opportunity to study palaeokarst in this region. In September 2005, during a field trip to the Raciszyn area, a small cave was discovered at the bottom of the lowest exploitation level in the "Trawertyn" Quarry. Using the $^{230}Th/^{234}U$ dating method, it was possible to distinguish several episodes of deposition and erosion.

A series of samples representing different sedimentological stages was analysed to reconstruct their position in time. These data allow the description of karst processes in the region and their correlation with other data from the Kraków-Wieluń Upland and with the major palaeoclimatic episodes of the Middle and Late Pleistocene.

GEOLOGICAL SETTING

The "Trawertyn" Quarry is located at the Kamienna Góra Hill near the Raciszyn (Wieluń Upland, northern part of the Kraków-Wieluń Upland, South Poland; Fig. 1). The hill is composed of late Oxfordian limestone. The quarry has operations at two levels. The higher level consists of

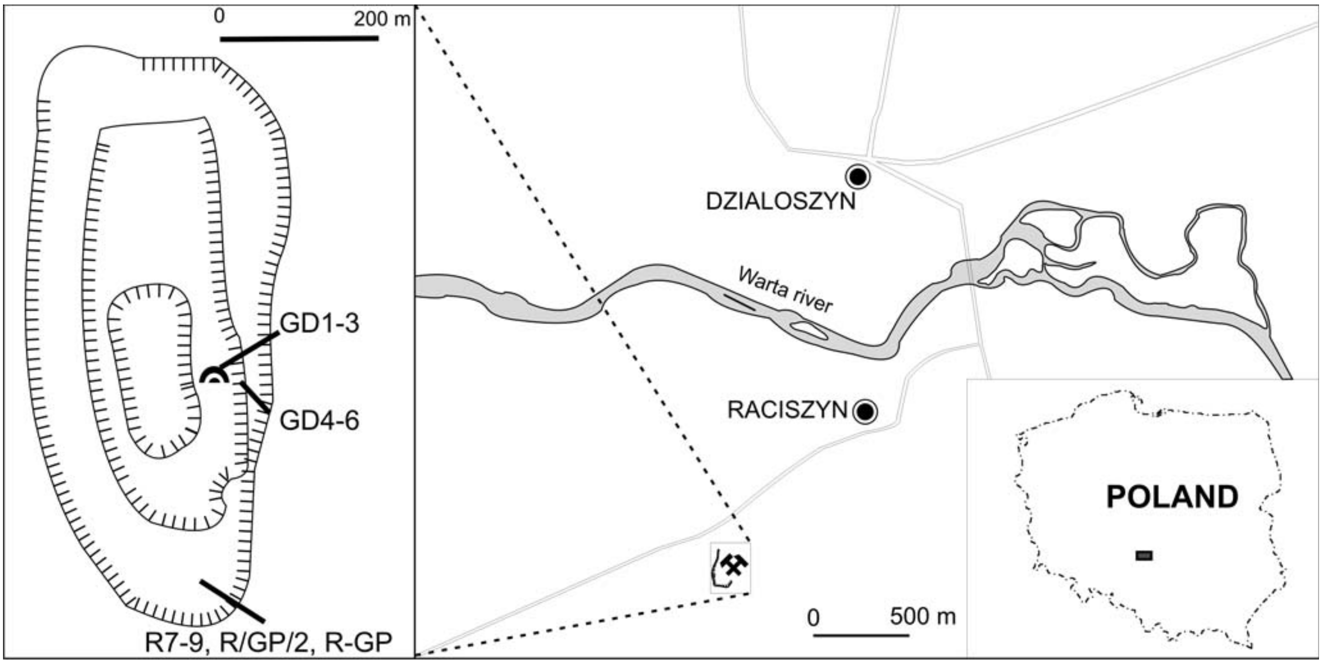

Fig. 1. Region of Działoszyn (quarry marked with an arrow) and cave location within the quarry.

significantly karstified limestone, rich in complex systems of karst structures, such as dozen-metre-high karst chimneys, phreatic channels and large fissures, filled with clays and speleothems. Most of the fissures were entirely filled with calcite; some of them revealed several generations of deposits, separated by apparent erosion surfaces. This indicates more than one stage of flowstone deposition.

The lower level is made up of solid rock, where karst phenomena are not so common and occur predominantly along tectonic or morphological structures. For the most part, they are represented by empty channels cutting the massif in a NE direction. They are oval-shaped conduits, about 20–50 cm in diameter. No speleothems were found in these structures. Large fissures cutting the entire quarry (at least about a hundred metres long) are the second typical forms of karst phenomena occurring at this level. The orientation of these fissures is similar to that of the previously described channels. In one of them, a cave had developed and it was the subject of isotope studies.

The cave was discovered below the bottom of the lower level. Its entrance has the form of a lens, about 40 cm wide and about 120 cm long. From the entrance, the shaft runs vertically down into the massif and ends after about five metres with a horizontal corridor, about seven metres long (Fig. 2). At a depth of approximately four metres, the width of the corridor increases to about one metre. A series of speleothems and clay deposits was found inside this passage. The bottom of the cave was filled with a thick layer of clay. The cave walls were covered with a set of flowstone layers, marking the former extent of the clay deposits. Samples of these flowstones were collected and analysed. Scallops observed on the walls indicate the vigorous inflow of water into the massif. It seems that at the time of formation of the scallops, the fissure was widened and the corridor was created. In its deepest part, the cave reached the level of the groundwater. The morphology of the speleothems indicates that during its history, the cave was filled several times either entirely or to some extent with clay deposits. Several generations of flowstones grew onto those sediments. Several times in their history, speleothems were cut off and the clay deposits were partially washed out. After the last erosion episode, the erosive surfaces were covered with the youngest, transparent calcite.

Above the cave, the fissure was entirely filled with flowstones and clay, which could be seen in the wall of the quarry that intersected with this structure (Fig. 3). The limestone walls are covered with scallops, but directly above them, a massive flowstone was found.

The upper exploitation level of the quarry consists of segments of strongly karstified rock. Under a thin soil layer, an intensively karstified limestone surface exists. The rock is rich in apparent karst structures. The remnants of several large karst chimneys in an eastern wall of the quarry are the most spectacular. In addition, the limestone is cracked with numerous karst forms. Furthermore, the caverns and fissures were filled with the clays and flowstones of at least several generations.

MATERIAL

Eleven speleothem samples were collected: three from the cave (GD1, GD2, and GD3), three from the fissure above the cave (GD4, GD5, and GD6) and five from the upper level of the quarry (R7, R8, R9, R/GP/2, and R-GP). Sample GD1 (Fig. 2) was collected inside the cave and consists of three parts: older, grey, crushed flowstone (subsample GD1/1) covered with clay and glazed with younger, transparent calcite (subsample GD1/2) that forms little stalactites (Fig. 4A). Samples GD2 and GD3 are fragments of

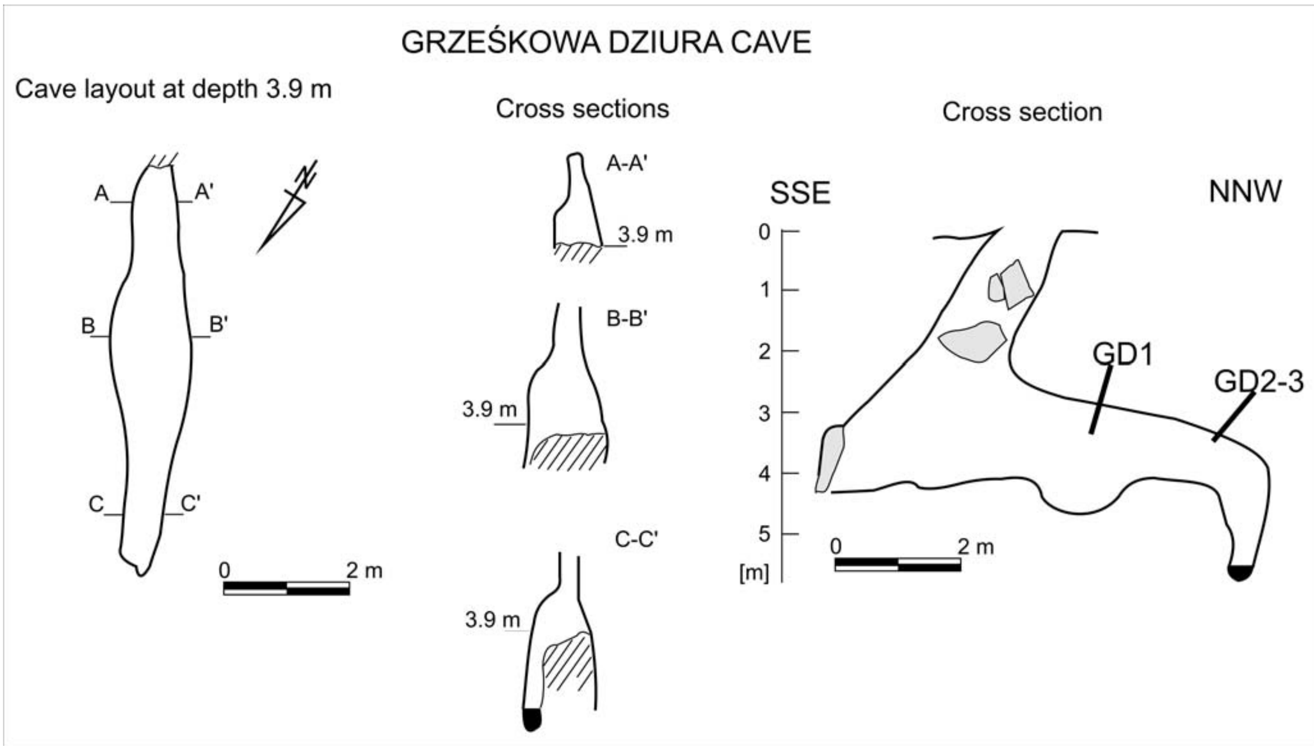

Fig. 2. Grześkowa Dziura Cave and sample locations. **A.** Plan of the cave and vertical cross- sections of the main corridor. **B.** Vertical cross-section of the cave.

yellow-brown, sandy flowstones, highly contaminated with detrital material and partially covered with a thin layer of transparent, white calcite.

The samples of flowstones, collected in the fissure above the cave (Fig. 3), represented the needle-shaped calcite that grew directly on the limestone wall (GD4). Two additional samples were taken from the massive flowstone (GD5 and GD6).

At the upper level of quarry, several generations of calcite flowstone had created a unique opportunity to study the complex depositional history of the speleothems. Several samples were collected from the southern wall of the quarry. An area between two karst forms filled with clastic deposits was chosen (Fig. 5). There, the limestone deposit was karstified and eroded, with some caverns filled with flowstones and clays. Sample R7 consists of two distinct parts: a 1.5-cm-thick flowstone that crystallised on clay deposits cut by an erosion surface and a second generation that crystallised on that surface. The erosion surface was covered with a thin clay film, which indicates a short period of clastic deposition. Samples R8, R9 and R10 had a similar form and were collected within the fissure.

Sample R/GP differs from other samples at the upper level (Fig. 4B). It represents a part of the massive flowstone (about 30 cm thick) that consists of two depositional layers, separated by an erosive surface covered with massive scallops. This sample was found at the base of the rock wall; therefore its exact position in the profile remains unknown. Similar flowstones were found in many places at the bottoms of the walls at the upper level of the quarry.

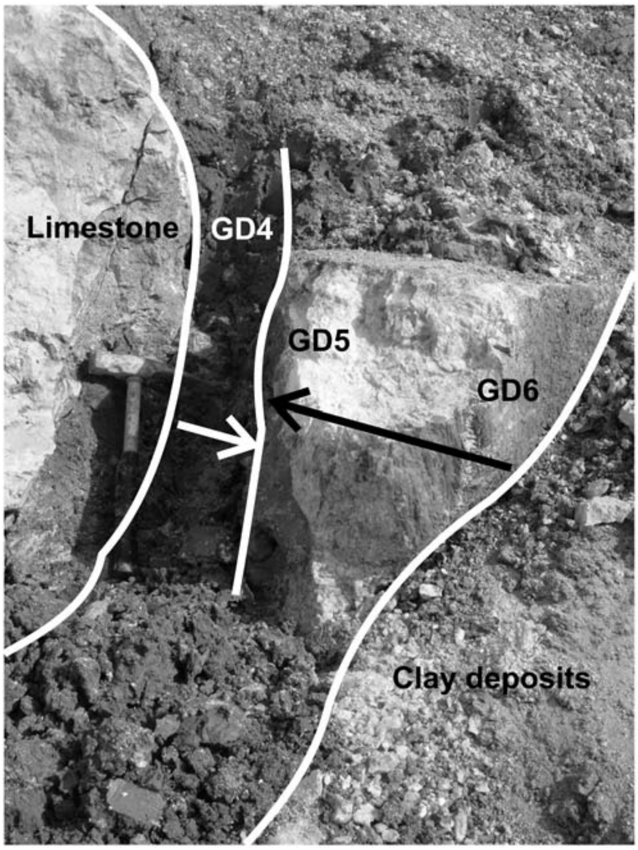

Fig. 3. Scheme of fissure filling above the cave with sample location marked.

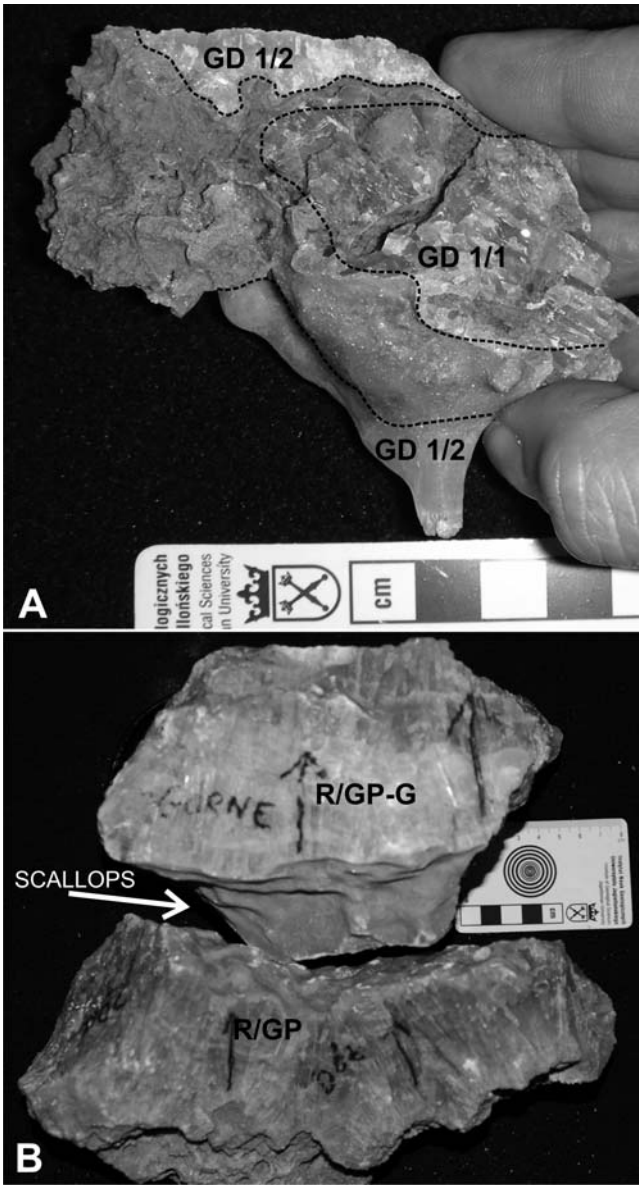

Fig. 4. Appearance of samples GD 1 representing the youngest speleothem' generation (**A**) and sample R/GP collected at the upper level of the quarry (**B**).

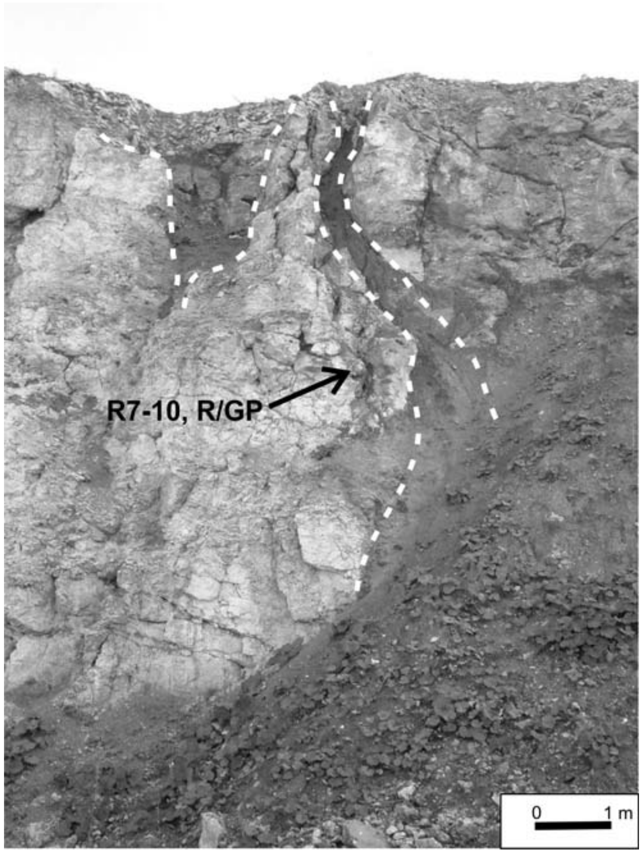

Fig. 5. Upper level of the quarry. Two karst forms (marked with dashed lines) and location where samples were collected (marked with arrow).

METHODS

The speleothem samples were analysed using the ^{230}Th/^{234}U method in the Warsaw Isotopic Laboratory for Dating and Palaeoenvironment Studies of the Institute of Geological Sciences of the Polish Academy of Sciences (Warsaw, Poland). In the first stage of the studies, the alpha spectrometry method was applied. The standard chemical procedure for uranium and thorium separation from carbonates (Ivanovich and Harmon, 1992) was used. Samples were dissolved in 6 M nitric acid, and uranium and thorium were separated by the ion exchange method using a DOWEX 1x8 resin. The efficiency of chemical separation was controlled by the internal standard ^{228}Th-^{232}U. Activity measurements were carried out using the OCTETE PC spectrometer from the EG&G ORTEC company. Spectrum analysis and activity calculations were performed with the "URANOTHOR 2.5" software (Gorka, 2002). Correction for background, counting geometry, chemical yield, and spike decay from the calibration time and the time between chemical separation and counting were applied.

In the final step of studies, the ICP-MS method was used for uranium and thorium isotope analyses. After thermal decomposition of organic matter, a ^{233}U-^{236}U-^{229}Th spike was added to samples before any further chemical treatment. Each calcite sample was dissolved in nitric acid. Uranium and thorium were separated from the carbonate matrix using TRU-resin. The spiked sample was transferred in 1 M HNO_3 to 1 ml TRU-resin column and the carbonate matrix removed by further elution with 1 M HNO_3, followed by 4 M HCl. U and Th were eluted together with a 0.1 M HCl–0.2 M HF mixture. Measurements were performed with a double-focusing sector-field ICP mass analyzer (Element 2, Thermo Finngan MAT, Prague, Czech Republic). The instrument was operated at a low mass resolution ($m/\Delta m \geq 300$). A double pass spray chamber with Teflon nebulizer was used as the sample-introduction system. The measurement results were corrected for counting background and chemical blank. The internal standard sample and blank sample were prepared simultaneously for any series of samples studied and were used for the necessary corrections and quality control.

Monte Carlo simulation (MC), assuming a normal distribution for ^{230}Th/^{234}U and ^{234}U/^{238}U activity ratios (basing on measurements results), was used for estimation of the probability of the estimated age limits. 5000 random selections from activity ratios were used for age calculations and the estimation of age distributions.

RESULTS

The results of the U-series analyses are presented in Table 1. For alpha spectrometry reported errors represent single (1σ) and for mass spectrometry double (2σ) standard deviations. All analysed samples have a low uranium content, which is typical for speleothems from the Kraków-Wieluń Upland (Głazek, 1986; Duliński, 1988; Hercman, 2000). Several samples, despite careful attempts to separate of clean material for analysis, contained too much clastic contamination detected by the presence of ^{232}Th and the results did not allow reliable determination of their age (GD3, R7, R10, R/GP/1 by alpha spectrometry and R/GP-G/2, R9/1, R10 by mass spectrometry). Clean material could not be separated from sample GD2 and therefore it was not analysed.

The low uranium content was responsible for the low accuracy of measurement results. Usually a U content at the level of 0.03 ppm is treated as the minimum level required for the measurement by alpha spectrometry. Most of the samples analysed had a lower U content and the application of the alpha spectrometry technique required a combination of large mass of the sample (10–25 g) and a long measurement time. Application of these requirements allowed the collection of good-quality spectra, containing at least several hundred counts in individual peaks. At the second stage of the studies, the mass spectrometry technique was applied for U and Th isotopes measurements. It allows the use of much smaller samples than in the alpha-spectrometry method; usually 0.5–2 g of calcite is enough to perform isotope analysis. The accuracy of the final age estimation is much better and age errors are ca. one order of magnitude smaller. Consequently, estimation of the equilibrium state between ^{234}U and ^{238}U and the final estimate of possible limits of sample age were more precise.

The age of youngest sample (GD1/2) could be estimated only in the sense of a maximum age limit. The low uranium content in combination with low age of the sample prevented precise determination of the ^{230}Th/^{234}U activity ratio.

DISCUSSION

Dating and age limit of the oldest samples

The oldest speleothems (GD 4, R 9 and R/GP/2) were older than 350 ka and exceeded the range limit of the ^{230}Th/U dating method using the alpha spectrometry method (Table 1). The low accuracy of the uranium activity ratios ^{234}U/^{238}U make it difficult to reach a decision on whether both isotopes were in secular equilibrium and as to the age limit for these samples. The initial ^{234}U/^{238}U activity ratios for the younger samples is between 1.09 and 1.50. Samples R 8 and R-GP-G had initial ^{234}U/^{238}U activity ratios 1.4±0.6 and 1.5±0.5, respectively. Typical values of initial ^{234}U/^{238}U activity ratios for the Kraków-Wieluń Upland speleothems are between 1.1 and 2 (Głazek, 1986; Duliński, 1988; Duliński and Kuliś, 1990). A reasonable estimate of the initial ^{234}U/^{238}U activity ratio for studied speleothems was a maximum at level 2. Considering the low accuracy of the results for activity measurement, the value of the ^{234}U/^{238}U activity ratio cannot be recognized from the secular equilibrium after about 0.5–1.0 Ma. It means that the minimum age of samples R 4, R9 and R/GP/2 may be estimated as 500 ka. For better estimates of the age limit, several samples were dated using mass spectrometry. The errors of estimated activities ratios are much smaller (Table 1). On the basis of MC simulation of the probabilities for age limits, the age of the GD 4 flowstone can be estimated for the period from 400 till more than 600 ka. The start of deposition of the samples studied from the upper level (R9 and R/GP) can be estimated as more than 600 ka. The oldest samples dated by the mass spectrometry technique revealed ^{234}U/^{238}U activity ratios different than 1, indicating a lack of radioactive equilibrium and indicating an age of less than 1,200 ka.

Phases of karst development and their palaeoenvironmental implications

Despite the low accuracy, the results obtained provided useful information on the karstification process at the Kamienna Góra Hill during the period from more than 600 ka until the Holocene (Fig. 6). The karstification processes began much earlier than 0.6–1 Ma and led to the formation of fissures, caves and probably karst chimneys, followed by the deposition of clay. The oldest speleothems were deposited on red-brownish clay sediments. Differences in calcite structure and colour rather indicate several phases of deposition and erosion older than 600 ka. However, it is difficult to specify the number of phases of speleothem deposition. The sample representing the oldest phase of speleothems deposition (R9) consisted of two layers (lower R9/2 and upper R9/1 and R9), separated by an erosion surface with scallops. Samples R7, R8 and R10 look similar. The lower layer of R9 was older than 600 ka. A similar age was obtained for sample R/GP, which consisted of numerous scallops. The erosion event (>600 ka) documented with scallops in samples R9 and R/GP was probably short and rapid and no clay sediments were deposited. The upper layer of sample R9 was younger than 600 ka and consisted of calcite similar to sample R 8, which also was collected from the same karst form at the upper level. This younger phase of speleothem deposition also was documented for sample R-GP-G and samples GD 6, GD5 and GD4, collected from the fissure above the cave. This package of thick flowstone was deposited during a relatively long time period (>600–290 ka), indicating stable conditions throughout the whole of this period. The younger generations of speleothem were found only inside the cave. They were represented by clear, transparent calcite stalactites and thin flowstones covered the wall of the cave and fragments of older speleothems and deposits. The pieces of older, crushed flowstone are 146 ka old. Its present position on the wall of the cave indicated that about 150 ka ago the cave was filled with clay to about

Table 1

U-series dating results

Sample	Lab. No.	Method	U cont. [ppm]	$^{234}U/^{238}U$	$^{230}Th/^{234}U$	$^{230}Th/^{232}Th$	Age [ka]	MC simulation of age limit [age (probability)]
GD 1/1	W 1749	[2]AS	0.035 ± 0.004	1.06 ± 0.17	0.75 ± 0.13	43 ± 40	146^{+54}_{-39}	
GD 1/2	W 1750	AS	0.085 ± 0.006	1.55 ± 0.12	0.03 ± 0.01	20 ± 53	< 3	
GD 3	W 1751	AS	0.219 ± 0.007	1.313 ± 0.016	0.29 ± 0.01	2.6 ± 0.3	[1]DC	
GD 3	95	[3]MS	0.265 ± 0.003	1.324 ± 0.008	0.123 ± 0.006	0.91 ± 0.04	DC	
GD 4	W 1539	AS	0.092 ± 0.006	0.93 ± 0.07	1.20 ± 0.10	48 ± 18	Out of range	> 350 ka (0.99)
GD 4/1	96	MS	0.1259 ± 0.0001	1.011 ± 0.004	1.03 ± 0.01	24.4 ± 0.2	Out of range	> 400 (1) > 500 ka (0.88) > 600 ka (0.86)
GD 4/2	97	MS	0.0777 ± 0.0001	1.009 ± 0.005	1.00 ± 0.02	32.8 ± 0.4	Out of range	> 400 ka (0.84) > 500 ka (0.60) > 600 ka (0.52)
GD 5	W 1540	AS	0.060 ± 0.004	1.08 ± 0.08	0.90 ± 0.06	171 ± 223	233^{+51}_{-37}	
GD 5	98	MS	0.0552 ± 0.0001	1.012 ± 0.005	0.93 ± 0.02	>1000	290^{+18}_{-16}	
GD 6	99	MS	0.0682 ± 0.0002	1.004 ± 0.004	1.02 ± 0.02	69.5 ± 0.8	Out of range	> 400 ka (0.98) > 500 ka (0.91) > 600 ka (0.87)
R 7/1	W 1879	AS	0.020 ± 0.003	1.28 ± 0.22	0.88 ± 0.14	18 ± 10	DC	
R 7/2	W 1772	AS	0.091 ± 0.006	1.03 ± 0.09	1.04 ± 0.08	7 ± 1	DC	
R 8	W 1881	AS	0.022 ± 0.003	1.15 ± 0.17	1.01 ± 0.14	28 ± 16	$370^{+\infty}_{-148}$	> 350 (0.56)
R 8	102	MS	0.0322 ± 0.0001	0.998 ± 0.005	0.91 ± 0.02	31.4 ± 0.6	260^{+20}_{-18}	
R 9	W 1880	AS	0.025 ± 0.004	1.07 ± 0.22	1.05 ± 0.21	25 ± 19	Out of range	> 350 (0.63)
R 9/1	104	MS	0.0270 ± 0.0001	0.994 ± 0.005	1.05 ± 0.02	6.6 ± 0.1	DC	
R 9/2	105	MS	0.0373 ± 0.0001	0.985 ± 0.005	1.02 ± 0.02	20.2 ± 0.2	Out of range	> 400 ka (0.99) > 500 ka (0.96) > 600 ka (0.93)
R 10	W 1795	AS	0.016 ± 0.002	0.97 ± 0.13	0.92 ± 0.13	14 ± 7	DC	
R 10	103	MS	0.0235 ± 0.0001	0.979 ± 0.006	1.03 ± 0.02	12.3 ± 0.2	DC	
R/GP/1	W 1796	AS	0.015 ± 0.002	1.08 ± 0.16	0.95 ± 0.14	16 ± 7	DC	
R/GP/1	83	MS	0.0346 ± 0.0001	0.991 ± 0.008	1.05 ± 0.03	30.7 ± 0.6	Out of range	> 400 ka (0.99) > 500 ka (0.98) > 600 ka (0.97)
R/GP/2	W 1797	AS	0.013 ± 0.002	1.03 ± 0.13	0.98 ± 0.12	32 ± 24	Out of range	> 350 (0.52)
R/GP/2	84	MS	0.0229 ± 0.0001	0.96 ± 0.02			Lost Th	
R/GP-G	W 1883	AS	0.022 ± 0.003	1.22 ± 0.16	0.99 ± 0.12	120 ± 111	297^{+285}_{-88}	
R/GP-G/1	100	MS	0.0300 ± 0.0001	0.965 ± 0.006	1.07 ± 0.02	49.5 ± 0.9	Out of range	> 400 (1) > 500 (1) > 600 (0.99)
R/GP-G/2	101	MS	0.0247 ± 0.0001	0.980 ± 0.006	1.07 ± 0.02	11.6 ± 0.2	DC	

[1]DC – detrital contamination. Measured activity of ^{232}Th indicates contamination by no-radiogenic thorium, so U-Th dating method cannot be applied to acquire credible dates.
[2]AS – alpha spectrometry
[3]MS – mass spectrometry with ICP-MS

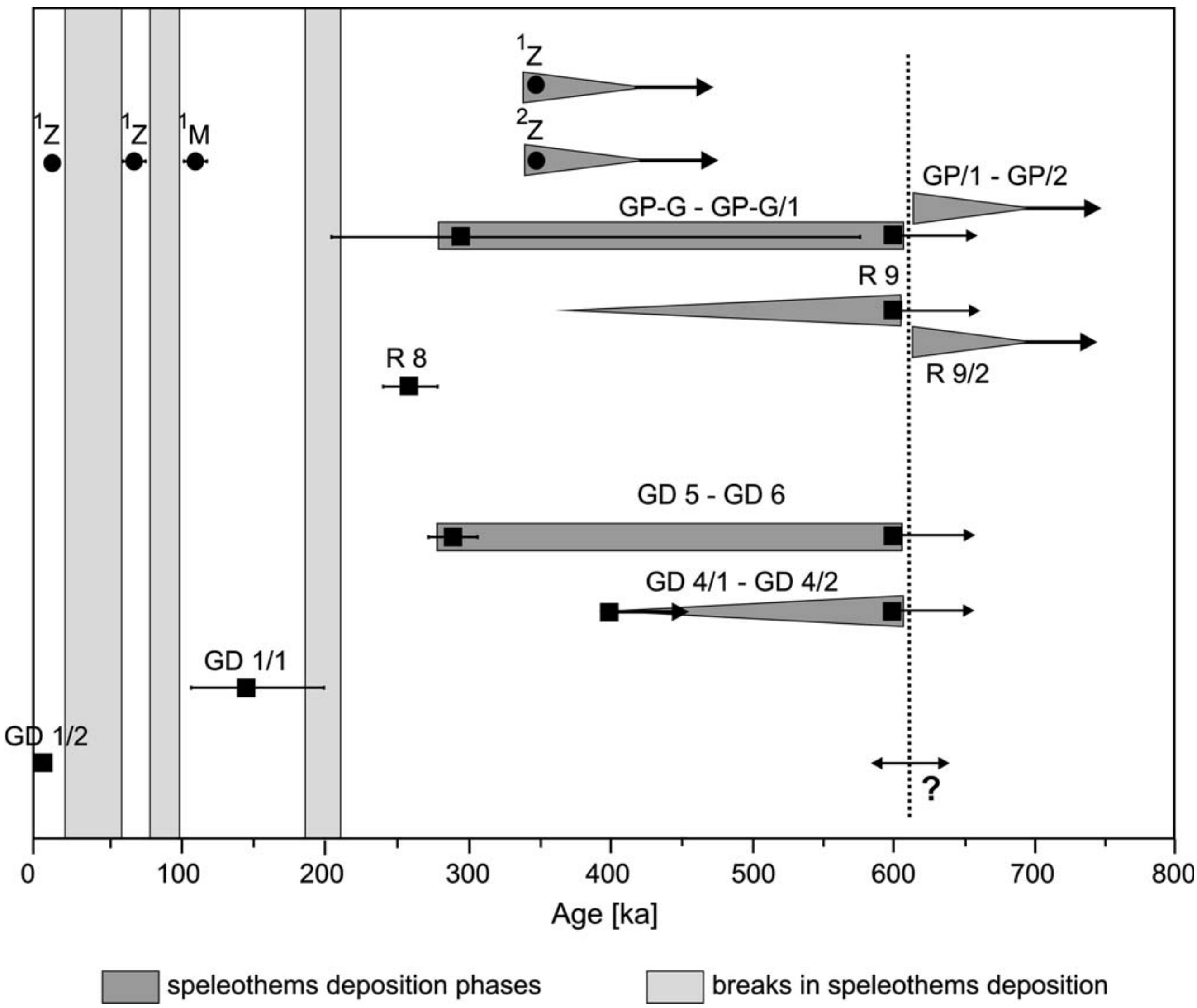

Fig. 6. The scheme of phases of speleothem deposition in the Działoszyn region. Squares – results of this study; circles – earlier published data: [1]Głazek (1986); [2]Duliński (1988).

1.2 m above the present floor. After that, the older flowstones and the clay deposit were cut off and washed to the present sediment level. The youngest, transparent calcite fixed older clasts to the wall and formed small stalactites of Holocene age (GD1/2).

Despite the long history of karst studies in the Działoszyn area, it is still not possible to formulate a time scheme of karst development. On the basis of earlier studies (Głazek *et al*., 1976, 1977; Głazek and Szynkiewicz, 1987; Głazek, 1989; Szynkiewicz, 1993), the first stage was characterized by the creation of empty spaces in the Oxfordian limestone. A comparison of the chemical and palaeozoological characteristics of the karst deposits at the Raciszyn Quarry and sites located in its vicinity indicate a pre-Pleistocene age for the forms occurring in Raciszyn. The deposits and faunal remains found within the karst forms indicate two possible periods of significant creation of the karstic forms: (1) Palaeogene, after the Laramian uplift of the Silesian Monocline, or (2) Late Miocene, as a result of the uplift of the meta-Carpathian range. Redeposition processes constitute the next step in the formation of the karst structures. The youngest sediment series is represented by brown clay marl, deposited before the Pleistocene glaciations. Part of the karst forms are filled by Pleistocene deposits (Głazek *et al*., 1976; Głazek *et al*., 1977; Głazek and Szynkiewicz, 1987; Głazek, 1989; Szynkiewicz, 1993).

After that, the oldest generation of speleothems was deposited (Fig. 6). The oldest speleothems collected in the Raciszyn Quarry crystallised on the brown or red clays. This stage was documented only in a few samples and it ended before 600 ka with an erosion event indicating intense water inflow. It is documented by massive scallops at the surface of the lower flowstone R/GP with a lack of clay deposition. This may document the termination of MIS 16 and the input of a high volume of water, originating from the melting ice sheet of Günz (San) glaciation or a short humid event during MIS 15 (Lang and Wolff, 2011). The erosion event was probably rapid, but short in time, and followed by a long period (MIS 15–MIS 9) of stable conditions, documented by the deposition of thick flowstones (R/GP-G, GD4<GD5,

GD6). It indicates that the strong glacial conditions of MIS 12 did not impact on speleothem growth. After a period of stable conditions, more diverse conditions were recognized during MIS 6. At the beginning of that period, there was an erosion event, which resulted in cave formation, followed by the deposition of clastic sediments. During the younger part of MIS 6 or at the beginning of MIS 5e (Eemian interglacial), speleothems were deposited again. The erosion process caused wash-out cave sediments, dated as referable to the Vistulian glaciation. During the youngest phase, the deposition of transparent flowstones and stalactites took place inside the cave.

CONCLUSIONS

The stages of karst development described in the study area are generally correlated with major climatic events in the Pleistocene. Confirmation of speleothem growth during long periods between >600 ka and ~300 ka, including the strong glacial of MIS 12, is the most outstanding feature of the work presented. The age record obtained creates a unique opportunity to further study depositional processes using more precise dating tools and stable isotope analysis. The reconstruction of glacial-interglacial cycles, the characteristics of glacial terminations and the internal structure of the interglacial period would further an understanding of the influences of glacial climatic conditions on speleothem deposition. The studies conducted at Raciszyn provided new data to refine the state of knowledge on karst development in this region. However, it should be noted that geochronological information is insufficient to build a clear scheme of karst development. It is to be hoped that the continuation of the work at Raciszyn will gradually provide new data and information about karst development in this region. The low concentrations of uranium in the speleothems of the Kraków–Wieluń Upland require the use of the mass spectrometry technique for determination of thorium and uranium isotopes.

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