

# APPLICATION OF THE WEATHERING PARAMETERS OF BONES TO STRATIGRAPHICAL INTERPRETATION OF THE SEDIMENTS FROM TWO CAVES (DESZCZOWA CAVE AND NIETOPERZOWA CAVE, KRAKÓW–CZĘSTOCHOWA UPLAND, POLAND)

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## Abstract

Analysis of weathering parameters of bones from cave deposits is presented as a useful tool of palaeoenvironmental reconstruction. As an example, we studied profiles of sediments in two Palaeolithic sites: Nietoperzowa Cave and Deszczowa Cave. Our studies included histological and EDS analyses of bone remnants found in these profiles. This method allowed us to reconstruct the changes of palaeotemperature and palaeohumidity, and finally the climatostratigraphy of sediments. The results presented here put a new light onto the stratigraphy of Deszczowa Cave's filling. In particular, besides the Vistulian sediments (MIS 2–5d), we confirmed the presence of layers formed during the Penultimate Glaciation (MIS 6) and Eemian Interglacial (MIS 5e).



**Key words:** cave sediments, Palaeolithic, fossil bones weathering, rubble weathering, palaeoenvironment reconstruction, histological index

## INTRODUCTION

An important element in the studies of Palaeolithic cave sites, is the reconstruction of palaeoenvironment and palaeoclimatic changes at the time of sedimentation of particular strata and cultural layers. Besides the composition of subfossil fauna remnants, the study of weathering degree of the sediment components, including limestone rubble, are the tools for the reconstruction of past environment fluctuations. Such methods have been already used in the investigation of Polish Palaeolithic cave sites, however their results were not always clear and comparable with each other (Madeyska 1981).

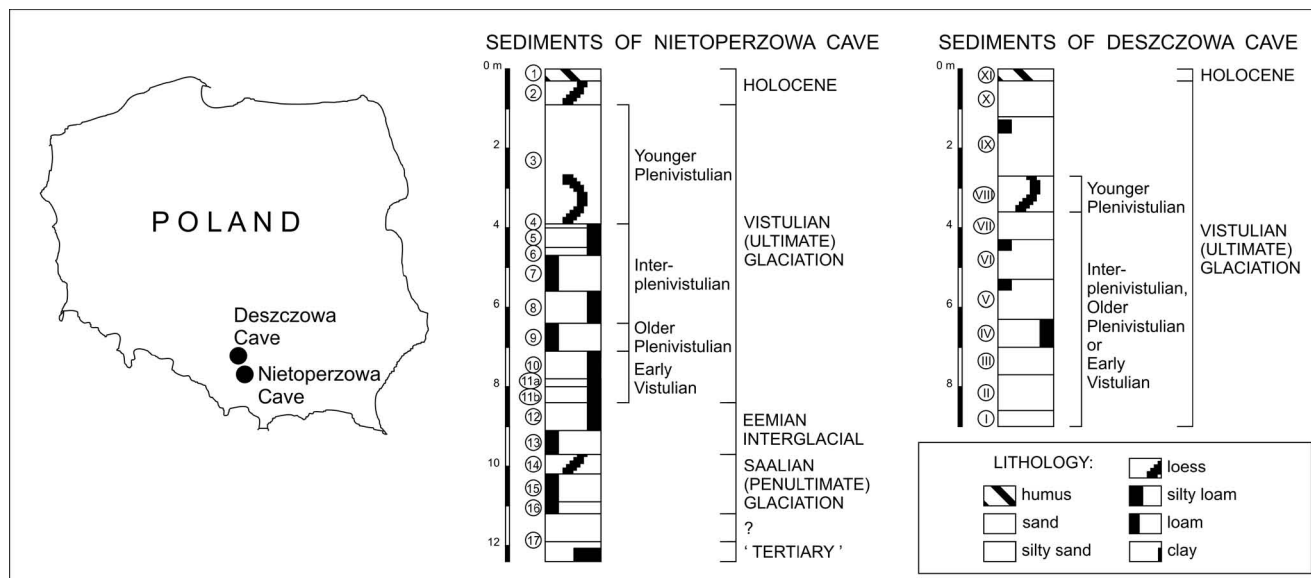
Looking for the supplementary information about the past environment, we propose a new method, *i.e.* analysis of weathering and early diagenesis of bones. The particular aim of this paper is to make an attempt to correlate the sequence of sediments from Deszczowa Cave with the stratotype profile of Nietoperzowa Cave, on the basis of the analysis of bones.

## THE SITES

Two caves were chosen for the study, both situated on Kraków–Częstochowa Upland, so called Polish Jura Chain, in southern Poland (Fig. 1).

Nietoperzowa Cave (NC in the later text) is one of the most important cave sites in Poland with Middle and Upper

Palaeolithic cultural layers and Pleistocene sediments treated as the stratotype profile of Late Penultimate Glaciation (MIS 6), the Eemian (MIS 5e) and Vistulian (MIS 5d-2) (Różycki 1967, Madeyska, 1982, Mojski 1985, Lindner 1992, Wojtal 2007). Stratigraphy was based on geological, palaeontological and archaeological evidence (Chmielewski 1961, 1975, Kowalski 1961, Madeyska-Niklewska 1969, Madeyska 1981, Wysoczański-Minkowicz 1969). It should be mentioned that according to Wysoczański-Minkowicz (1975) the sediments of pre-Vistulian age do not occur in the cave, however it was an isolated opinion. Except of the bottom section of sediments accumulated in flowing water, the whole series originated in dry cave and it contains loams with limestone rubble. Its lithological composition and traces of differentiated degree of weathering indicate on climatic changes during the sedimentation. Bone remnants belong to different species of vertebrates, some of them are characteristic for particular environments. Exploration of the site was terminated in 1969 and that is why only one radiocarbon date of charcoal is available. Basing on these data it was stated that: the oldest part of dry cave sediments – layers 16–14 containing loess material – originated during the end of penultimate glaciation; layers 13–12 with traces of strong chemical weathering represent the Eemian Interglacial (MIS 5e); layers 11–10 – Early Vistulian (MIS 5a–5d); loess layer 9 corresponds to MIS 4; layers 8–4 are dated to Interplenivistulian – MIS 3; loess layer 3 to LGM – MIS 2; and layers 1–2 to the Late Vistulian and the Holocene.



**Fig. 1.** Localization of Deszczowa Cave and Nietoperzowa Cave in Poland, and litho-stratigraphical profiles of the caves' sediments. The shown thicknesses of layers are the maximal known. Numbers in circles are numbers of layers, given in original publications (Madeyska-Niklewska 1969, Cyrek *et al.* 2000). The layers: 1–16 from Nietoperzowa Cave and III–VII from Deszczowa Cave contain a limestone rubble. Stratigraphy of sediments from Nietoperzowa Cave is shown according to Madeyska (1982), and from Deszczowa Cave – according to Cyrek *et al.* (2000); however please note an alternative interpretation of Nadachowski *et al.* (2009), described in the text.

Deszczowa Cave (DC in the later text) is another interesting archaeological site with the sequence of several Middle and Upper Palaeolithic cultural layers. Stratigraphical position of its sediments is based on geological analysis along with U/Th and radiocarbon dates (Cyrek *et al.* 2000, Lorenc 2006, Krajcarz 2010), and the sediments generally correspond to the younger part of the sequence from Nietoperzowa Cave (Fig. 1). However Nadachowski *et al.* (2009) recently presented new data of faunal analysis, mainly of rodent remains, positioning the lowest layers (I–IV) below Early Vistulian or even below Eemian Interglacial (*i.e.* below MIS 5). The overlying layers (V–VII) in their scheme are related to Interplenivistulian (MIS 3), with an erosional hiatus between the layers IV and V.

## MATERIAL AND METHODS

### Material

The materials examined in this paper were samples of bones and limestone rubble from Deszczowa Cave and Nietoperzowa Cave. The samples were collected during archaeological field expeditions – from DC in the years 1989–1997 and from NC in the years 1961–1969.

The samples of the following layers were available to the authors. From Deszczowa Cave: layers III–X for bone analysis (only the bones from these layers were stored) and layers III–VII for limestone rubble morphology (layers with abundant limestone clasts). From Nietoperzowa Cave: layers 3–16 for bone analysis, and for limestone rubble morphology – layers 3–12. Other layers were not examined, because of lack of the rubble or bones.

The sediment samples of similar weight (*ca.* 10 kg of sediment for each layer) were taken from the same vertical profile. The rubble samples were retrieved from sediment

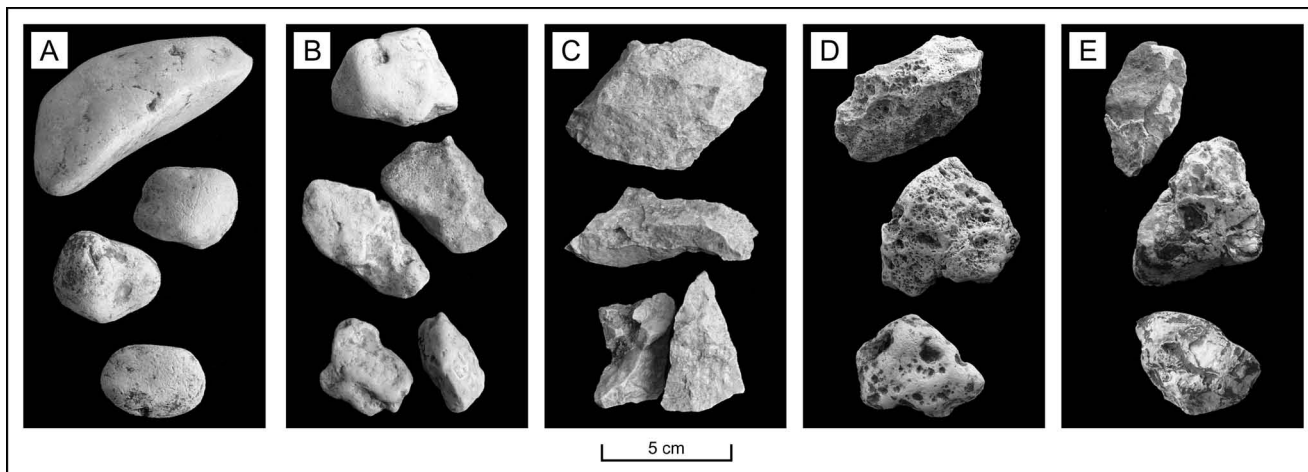
samples by careful sieving and removing the fraction finer than 10 mm. The lithological analysis including morphology of the limestone rubble was conducted in the 1960s for samples from NC, and in the 1990s for DC.

Portions of the removed finer fraction (< 10 mm), containing bone remains, were stored in the Institute of Geological Sciences, Polish Academy of Sciences, dried and packed in moisture-proof plastic bags. Histological analysis of bone remains from both caves was made dozen years later, in the years 2006–2008. Luckily, the well prepared storage in dry, cold and dark conditions had preserved the material against decaying or any contamination.

Bone fragments were picked from the sediment samples, cleaned in distilled water, dried and processed to microscopic sections. Only the fragments meeting the following criteria were chosen for examination: long bones (in anatomical sense); length of fragment about 10 mm; weight about 1 g; preserved bone surface as well as cortical and spongy tissues; cortical tissue about 3–4 mm thick. None of the examined bones was determined to a species level due to a fragmentary preservation, however the former criteria provided that only long bones of big mammals were chosen (the chosen thickness of cortical tissue of long bones indicates mammals bigger than roe deer but smaller than bison). 10 bone fragments from each layer were taken to histological and EDS analysis.

### Morphology of limestone rubble

Analysis of limestone rubble morphology was conducted according to the well known method (Chavaillon-Dutrievoz 1955, Laville 1964), adapted and developed later by Madeyska-Niklewska (1969, 1971). For the analysis, we selected only big rubble samples, containing statistically representative amount of limestone clasts (300–500 clasts each).



**Fig. 2.** Photographs of chosen limestone clasts, exemplifying different clast morphology. A – smooth clasts, without corrosive pits or mineral covers. B – clasts with smooth edges, without corrosive pits or mineral covers. C – sharp-edged clasts, without corrosive pits or mineral covers. D – clasts with corrosive pits. E – clasts with mineral covers. All samples come from Nietoperzowa Cave.

Each rubble sample was sorted by macro- and microscopic features into three kinds (see also Fig. 2): (1) smooth clasts, (2) clasts with smooth edges and (3) sharp-edged clasts. Additionally each group was divided into two parts *i.e.* clasts with and without small corrosive pits. The number of clasts in each group was counted and proportion between the numbers was presented on the diagram.

### Histological analysis of bones

Histological analysis was performed with the method described by Hedges *et al.* (1995). Chosen bone fragments were impregnated with epoxy, glued on a glass slide and polished to achieve transverse sections of cortical tissue. In addition, a small piece of each chosen bone was kept for chemical EDS analysis.

Each specimen (thin section) was classified – according to microscopic features of cortical tissue visible in transparent light – to one of six kinds, named the values of histological index (H.I.). The values from 0 to 5 were recognized as follows: (0) none histological structure apart Haversian canals identifiable, (1) small areas of well preserved structure, or some lamellar structure preserved, (2) lamellar structure well preserved between destructive foci, (3) some areas with well preserved all elements of structure, (4) only minor amounts of destructive foci, and (5) very well preserved structure, indistinguishable from fresh bone (Fig. 3). In addition, the presence of radial cracks around Haversian canals were noticed, according to Pfreundlicher (2004), with division into three groups: (a) numerous cracks, if they run across whole osteons and were visible in almost all osteons, (b) sparse, if they were visible only in peripheral parts of some osteons, and (c) none, if cracks were absent (Fig. 3). It must be remembered that this analysis, likewise the rubble morphology analysis, is always subject to personal view and experience. The preservation of histological structure, firstly determined under transparent light microscope, was also confirmed using electron microscope, according to Bell (1990).

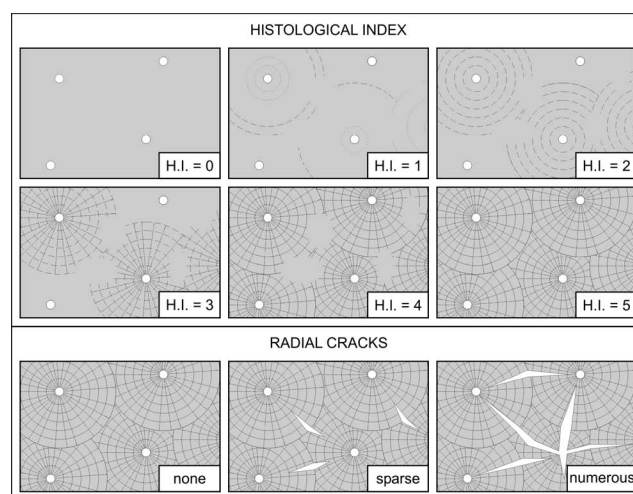
### EDS analysis of carbon content

Fragments of each bone sample were subjected to microprobe chemical analysis (EDS). The samples were washed in 5% HCl for 10 minutes to remove carbonates, and next washed in distilled water and dried. The analysis were conducted using JEOL JSM-6380LA electron microscope with EDS analyzer, in Engineer Geology Lab, Faculty of Geology, Warsaw University. Contents of carbon, calcium and phosphorous were measured, with several repetitions for each fragment.

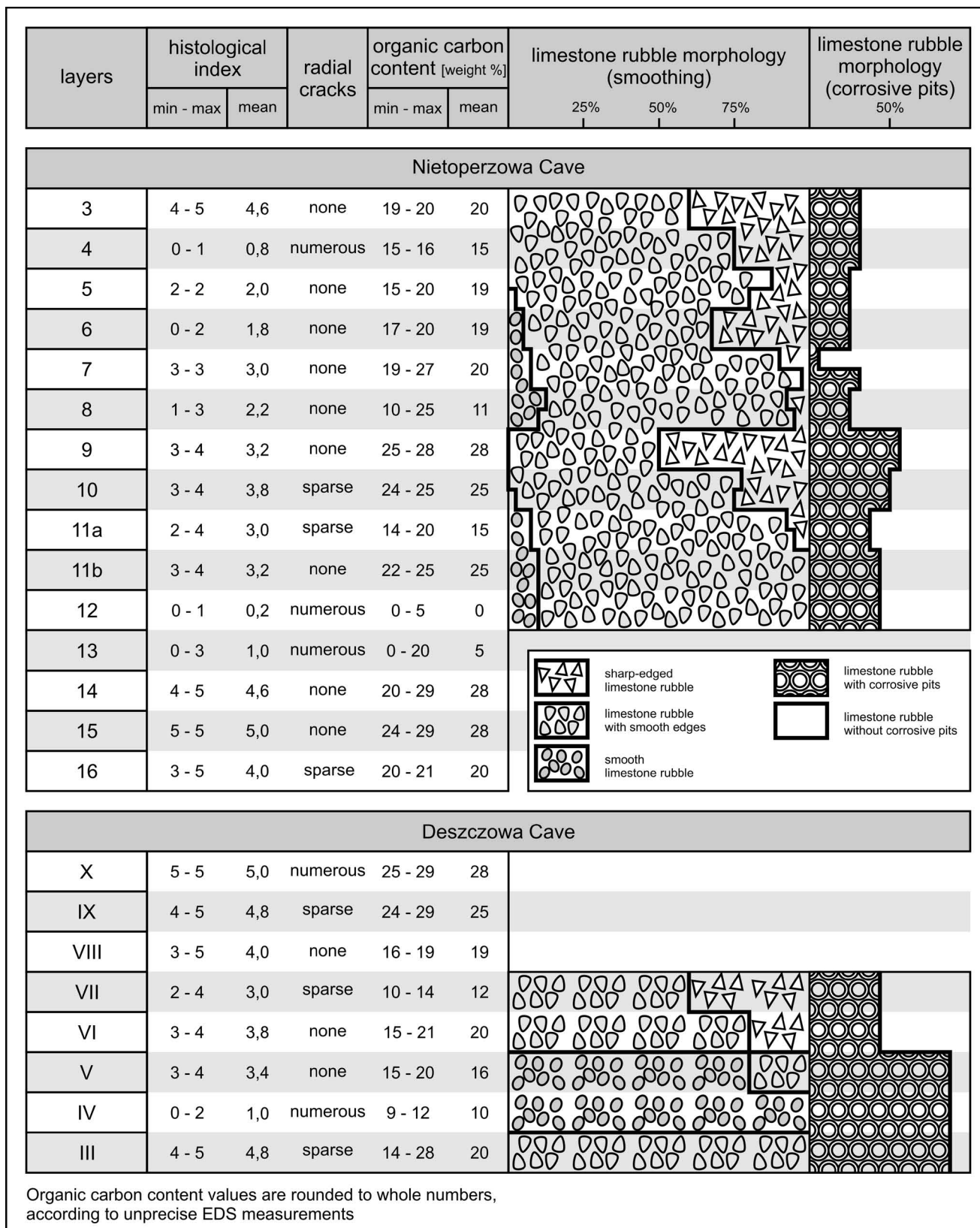
## RESULTS – A RECORD OF WEATHERING

### Limestone rubble morphology

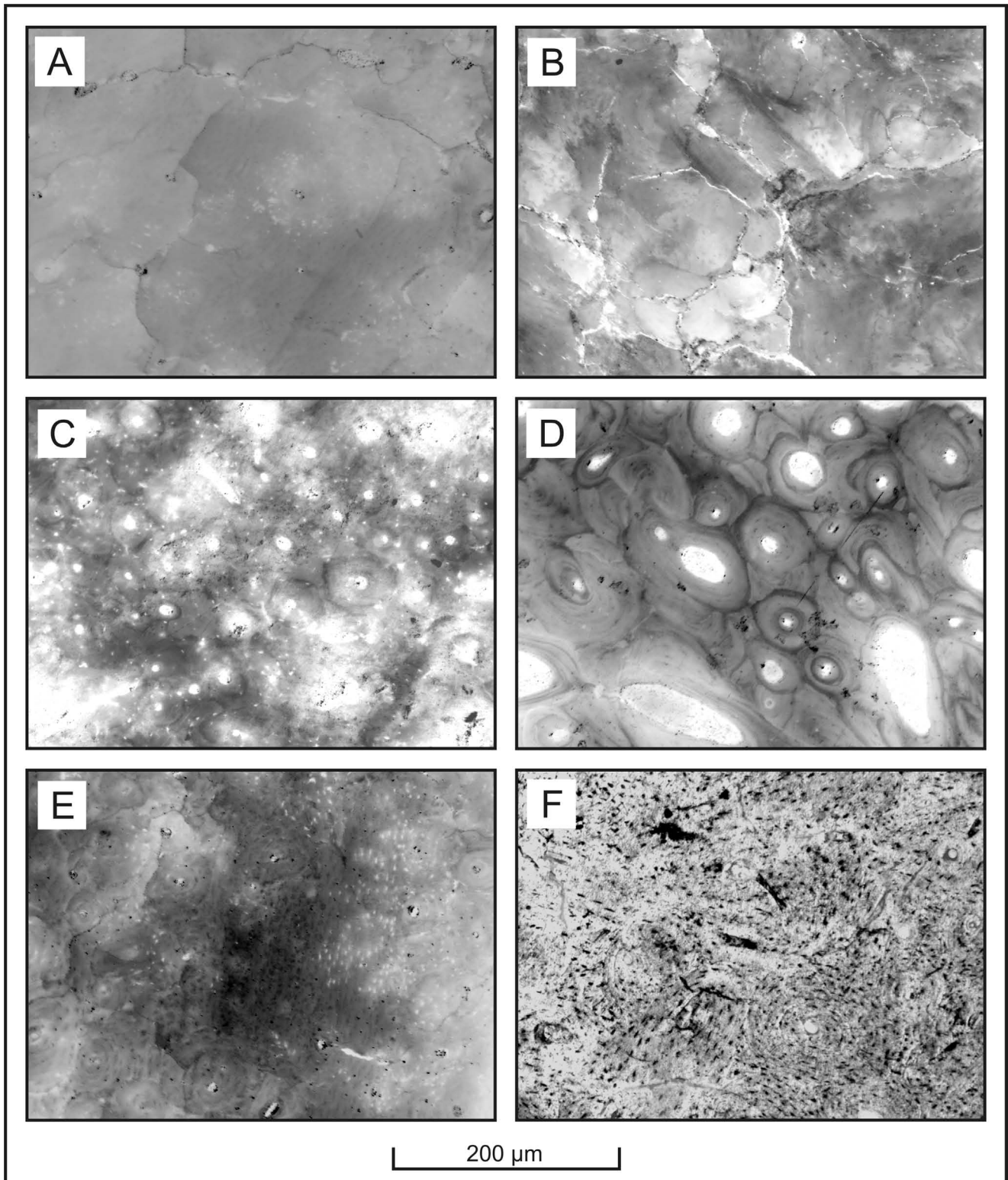
Percentages of three morphological classes of clasts in each layer are presented in Fig. 4. Different proportions of



**Fig. 3.** A schematic key to identification a value of histological index (six upper schemes) and the presence of radial cracks (three lower schemes), after Krajcarz (2010), modified.



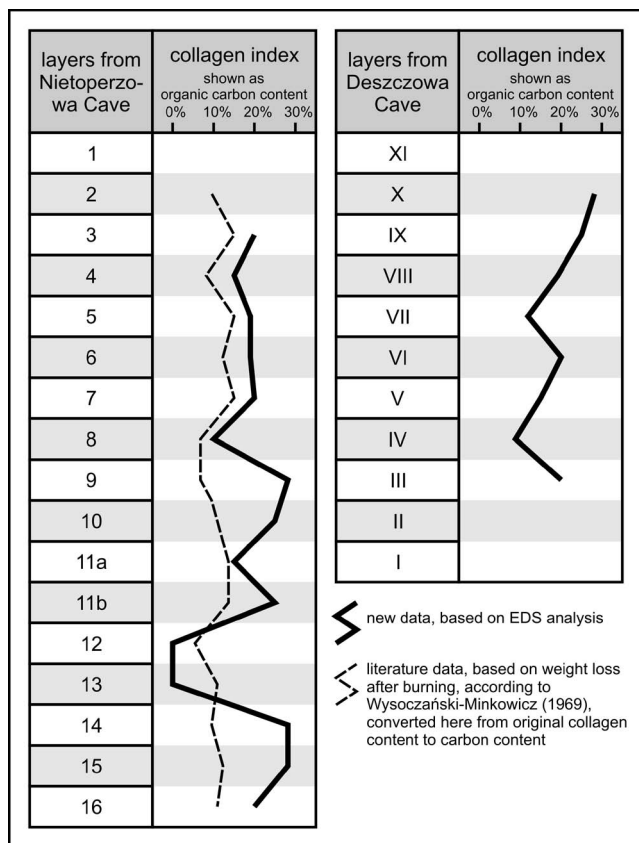
**Fig. 4.** Columns on the left side: Parameters of bone weathering for layers from Nietoperzowa Cave and Deszczowa Cave. Note narrow range of H.I. values for each layer. Diagrams on the right side: Participation of morphological classes of limestone rubble in layers from Nietoperzowa Cave and Deszczowa Cave. In Deszczowa Cave the upper layers (VIII–XI) have a limited abundance of the limestone rubble.



**Fig. 5.** Microscopic photographs of chosen bone samples from Nietoperzowa Cave. **A** – layer 12, histological index (H.I.) = 0, structure totally destroyed, numerous radial cracks. **B** – layer 4, H.I. = 1, numerous radial cracks. **C** – layer 6, H.I. = 2, no radial cracks. **D** – layer 8, H.I. = 3, no radial cracks. **E** – layer 14, H.I. = 4, sparse radial cracks. **F** – layer 3, H.I. = 5, structure very well preserved, no radial cracks.

these classes in sediments are a well understood record of ancient climatic conditions (Madeyska 1981, 1982, Mirosław-Grabowska 2002). Higher percentage of smooth clasts, along with lower amount of sharp-edged ones, testifies the warm conditions during accumulation. In NC such record is evident in the layers 7–8, and especially in the layer 12. The

highest percentage of sharp-edged rubble is noticed in the layers 3 and 9. The sediments from DC are comparatively poor in limestone rubble. The layer IV is a one with the greatest amount of smooth clasts. The coldest conditions are recorded by high amount of sharp-edged rubble in layer VIII (data not shown in Fig. 4 due to small amount of rubble).



**Fig. 6.** Diagrams of organic carbon content in bones in the profiles of sediments from Nietoperzowa Cave and Deszczowa Cave.

The presence of corrosive pits or hollows on limestone clast's surface is interpreted as an indicator of humid conditions during accumulation, connected with acidification of water seeping into the cave (Madeyska-Niklewska 1969, Madeyska 1981). In NC the sediments mostly enriched in such clasts are found in the layers 4, 6, 8, 10, and especially 11a, 11b and 12. In DC the layers III, IV and V show the same feature. It is assumed that these layers were formed under the most humid conditions.

### Histological index

Wide spectra of H.I. values were observed in samples of bone remains from both caves (see also Krajcarz 2010, where some results for DC were first presented). The chosen photographs are shown in Fig. 5, and full documentation in Fig. 4. In most cases only one or two values are observed in one layer (Fig. 4). It testifies the homogeneity of bone assemblage in each layer, and additionally shows that the bones of the same size and similar anatomical structure are the object of similar weathering changes, despite their systematical affinity.

The values of histological index may easily be interpreted in terms of palaeoclimate (Hedges *et al.* 1995, Hedges, Millard 1996, Smith *et al.* 2007). Lowering of H.I., related to a damage of internal structure, is connected with microbial attack (see Smith *et al.* 2007). Lower values represent thus warm conditions, when microbial activity was more inten-

sive. The highest values, describing well preserved histological structure, represent the most severe conditions during bone accumulation, like in the periods of periglacial or arctic climate, when microbial activity was limited. In NC the highest H.I. values occur in the layers 3, 9–10 and 14–15 (Fig. 4). It allows us to correlate them with some stadials of Pleistocene glaciations. The same is linked to the layers III and IX–X from DC. The distribution of the lowest H.I. values shows which layers were accumulated under the mildest climatic conditions. Among them there are: layer IV from DC and layers 12–13 from NC, and also, but less distinctive, layers 4–5 and 7–8 from NC and VII from DC.

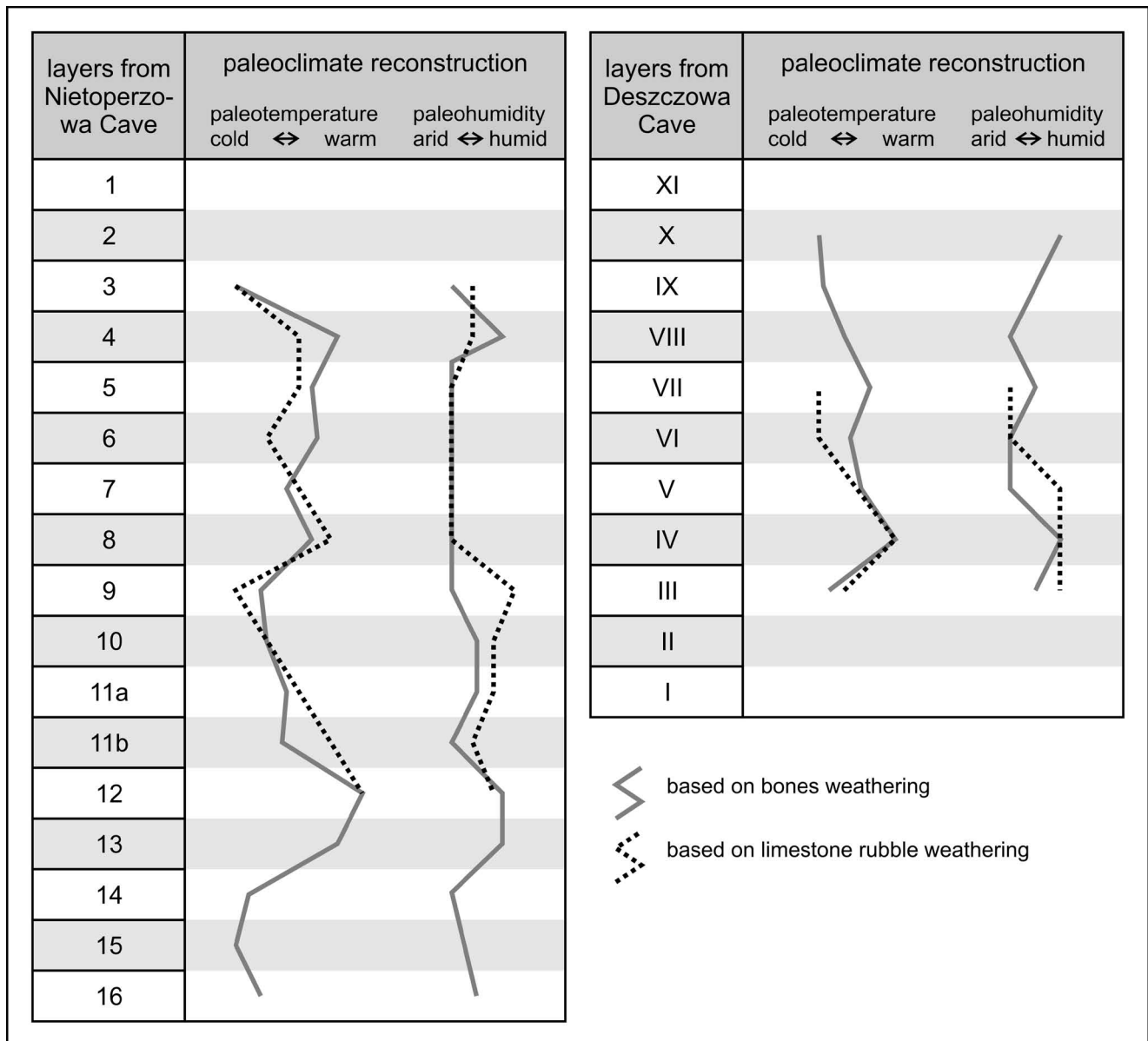
### Radial cracks

The presence of radial cracks in osteons is interpreted as an effect of weathering in water or in humid conditions (Pfretzschner 2004). It is explained by different distribution of stress inside osteon, caused by contraction and expansion. Numerous radial cracks observed in a bone assemblage indicate humid climate during either accumulation, weathering or early diagenesis of bones. The analysis proved that such climatic conditions occurred during sedimentation of the layers 4 and 12–13 from NC and IV and X from DC.

### Carbon content in bones

Carbon content is treated here as a function of collagen content in bone. However apart organic carbon there may occur mineral carbon in bone, bound in calcite or carbonate apatite (Iacumin *et al.* 1996). The values shown in Fig. 4 are concentrations of organic carbon only, based on EDS analysis. The mineral carbon was removed by washing in acid, and additionally concentrations of organic carbon were calculated from the content of C, Ca and P, with the assumption that the whole calcium is stoichiometrically bound either with phosphate or with carbonate, and that the whole carbon is either stoichiometrically bound with Ca as a carbonate, or it is of organic origin. Because the carbonate carbon may be also included in apatite in an other way than bound with calcium (*i.e.* in the position of OH groups, however in trace amount) the values shown in Fig. 4 should be treated as approximate. The content of organic carbon, treated as content of collagen, is dependent on weathering and connected with the degradation of histological structure (Hedges *et al.* 1995). A diagram of collagen (or organic carbon) content as a function of accumulation time or, simplifying, of sediment's depth is known as collagen index. Collagen index for NC is shown in Fig. 6, in comparison with collagen index published by Wysoczański-Minkowicz (1969), the latter one determined basing on weight loss of bones after burning in 800°C.

Note the occurrence of three distinct peaks in the diagram of Nietoperzowa Cave, in the layers 4, 8 and 12–13, in comparison to two peaks on the Deszczowa Cave diagram, in the layers IV and VII. In Nietoperzowa Cave an additional peak for the layer 11a is also visible on the diagram based on EDS analysis, however it is absent on the literature-derived diagram. That may be due to less precise sampling of Wysoczański-Minkowicz who treated both layers 11a and 11b as one layer 11.



**Fig. 7.** Reconstruction of climatic changes during accumulation of sediments from Nietoperzowa Cave and Deszczowa Cave. The reconstruction is based on limestone rubble morphology (according to Madeyska 1981) and bone weathering parameters (H.I. values as palaeotemperature indicator and the presence of radial cracks as palaeohumidity indicator; new data).

Despite some differences, the general similarity of the two diagrams should be regarded. It indicates the correctness and mutual verifying of both methods.

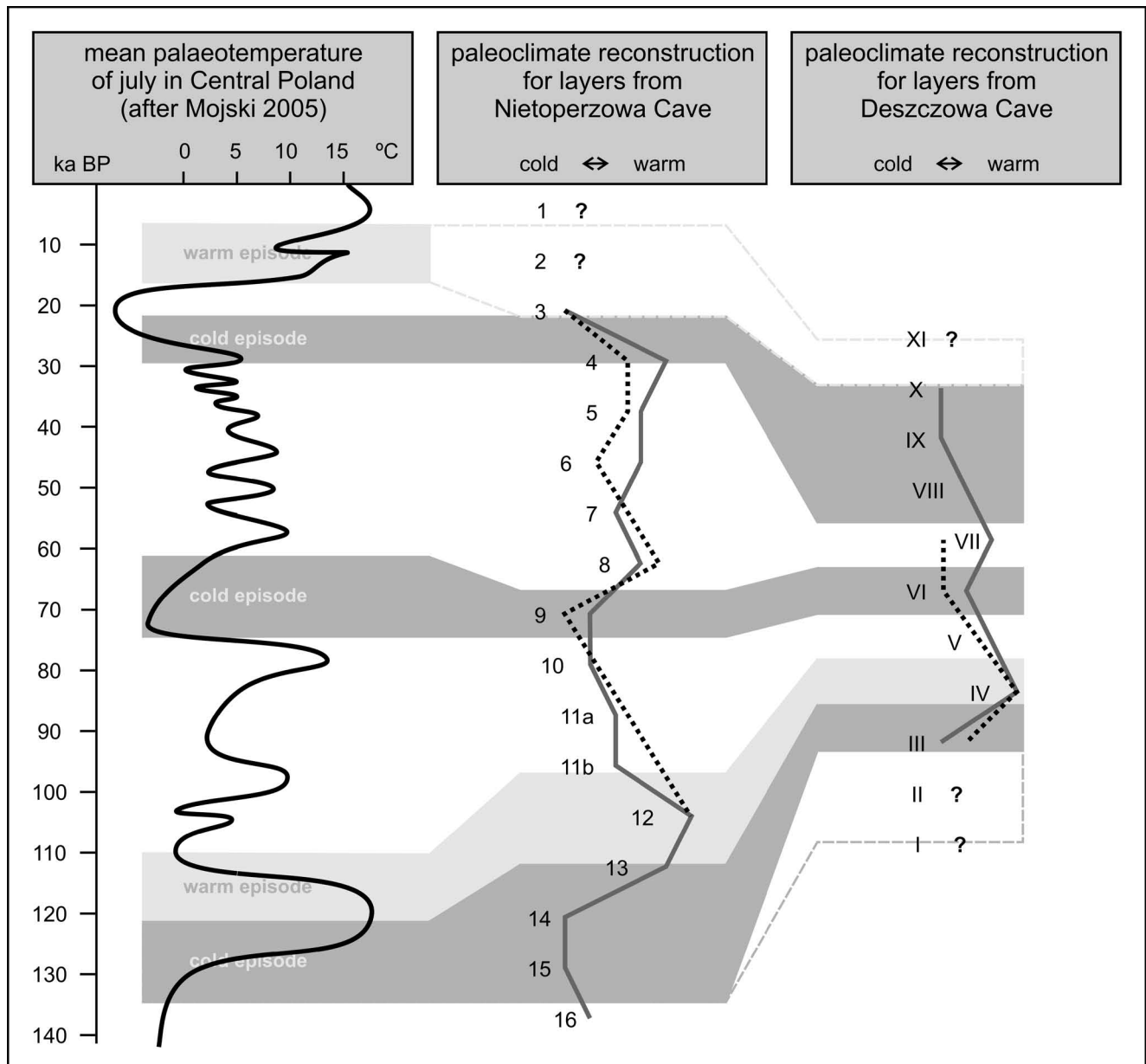
## DISCUSSION

### The weathering and palaeoclimate

Correlation between two cave fillings – on the basis of weathering effects – may be possible only if we assume that the two caves were exposed to weathering factors at the same or similar degree. In the specific case of the two sites analysed here, it should be noticed at first that these caves are not identical either in their geometry or geomorphology, as well as lithology of sediments. Nietoperzowa Cave is much bigger tunnel-like cave, where the samples were taken about 20

meters from the entrance. Deszczowa Cave is rather small and shallow cavern, with a total length of 15 meters. It suggests that the sediments from Deszczowa Cave, situated closer to the outside environment, would be more weathered than the ones from Nietoperzowa Cave. In addition, in Deszczowa Cave there are mainly sandy sediments, more porous and prone to weathering, while in Nietoperzowa Cave there are clayey or silty loams, better preserving rubble or bones. However, the layers with more and less advanced weathering could be indicated in both caves. The sequences of degree of weathering effects in the profiles of sediments may be used as a tool to find a palaeoclimatic correlation – and a stratigraphical correlation as a result.

An attempt to reconstruct palaeoclimatic changes that occurred during sedimentation in both caves is shown in Fig. 7. The changes of temperature are reconstructed on the basis of



**Fig. 8.** Correlation of the most distinctive peaks of the palaeotemperature curves reconstructed for sediments of Nietoperzowa Cave and Deszczowa Cave with the palaeoclimatic curve of the late Quaternary of Poland. The numbers next to the curves denote layers of sediments.

H.I. values, so they are shown in six-level scale. In addition, palaeotemperatures derived from the participation of smooth clasts and sharp-edged clasts are also presented. The changes of humidity were reconstructed based on the presence of radial cracks in bone osteons, and also on the basis of the abundance of limestone clasts with corrosive hollows.

There are two distinct episodes of warm and humid climates recorded in both caves. The older episode represents the warmest and most humid conditions and it is related to the layers 12–13 from NC and the layer IV from DC. The second one represents weaker warming and is recorded in the layers 4 and VII, respectively. From NC there is an evidence for additional weak warm episode in the layer 8, however there is not similar evidence from DC.

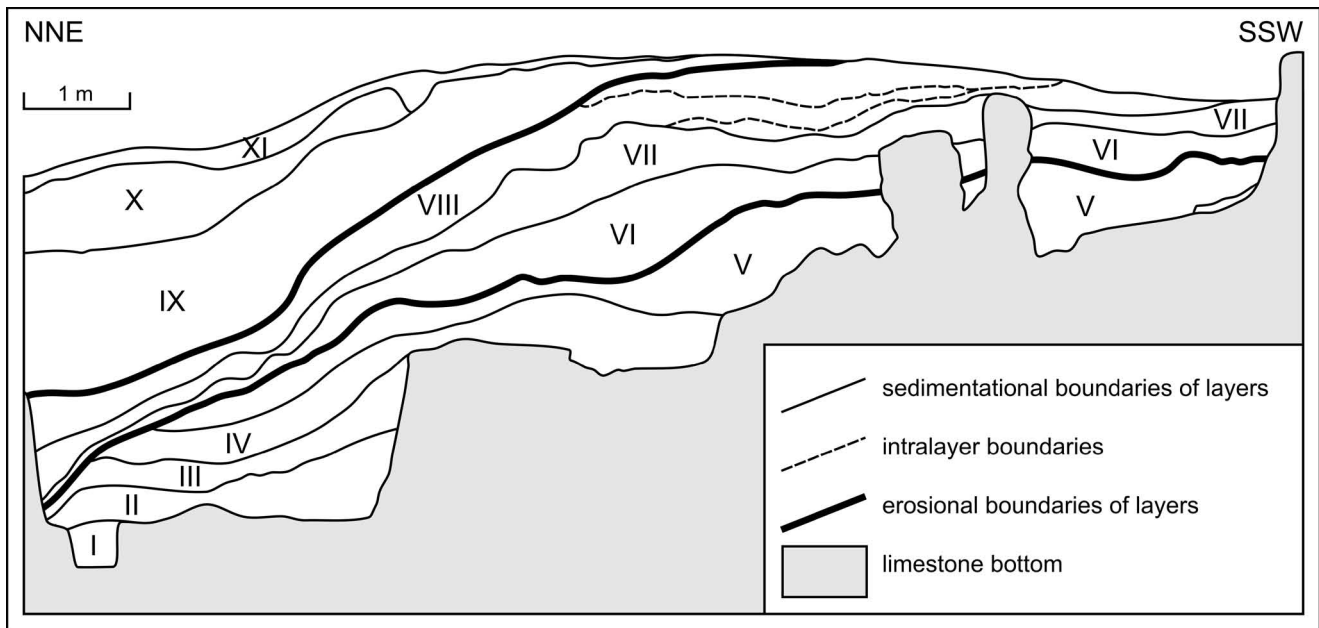
The important argument which helps to build the stratigraphical correlation is the presence of three distinctive

peaks of severe climate, recorded both in NC and DC (Figs 7 and 8). These peaks may be easily correlated with three cold stadials, known in glacial geology as three main last periods of glacier advances (Mojski 2005). They are: Warta Stadial of Saalian (Penultimate) Glaciation, Świecie Stadial (Older Pleniglacial) and Main Stadial (Younger Pleniglacial, LGM) of Vistulian Glaciation.

#### Stratigraphical correlation of the two sites studied

We believe that the two distinct episodes of warm and humid climate, clearly marked on diagrams (see Fig. 7), may be correlated between two caves (Fig. 8). The older episode is marked in Nietoperzowa Cave in the layers 12–13. According to many other known data (Kowalski 1961, Madeyska-Niklewska 1969, Wysoczański-Minkowicz 1969, Chmie-





**Fig. 9.** Cross-section of sediments from Deszczowa Cave (according to Cyrek *et al.* 2000, simplified). Erosional boundaries are traced along geological discordances.

lewski 1975, Madeyska 1981, Wojtal 2007) this peak of weathering intensity should be related to the Eemian (MIS 5e). The interglacial position of these layers is confirmed by the highest values of weathering parameters in the whole profile. The overlying layers – 11b, 11a and 10 – were initially attributed to the Eemian *sensu lato* (Madeyska-Niklewska 1969). That attempt was in accordance with the concept of the stratigraphy of Quaternary in Poland accepted at that time (Różycki 1967). The position of the mentioned layers, including layer 12, according to the faunal composition, was then moved to the Early Vistulian (Madeyska 1981). Our data confirm that the climate during accumulation of these sediments (layers 11–10) was cooler than during Eemian Interglacial (layers 13–12), although the conditions were still quite warm and humid. The Eemian position of layer 12 and post-Eemian position of layers 10–11 are undisputed. In the light of modern stratigraphy of Late Pleistocene these layers should be connected with the warm episodes of Early Vistulian, like Brørup and Odderade interstadials (MIS 5d–5a, see *e.g.* Donner 1996, Allen, Huntley 2000, Caspers, Freund 2001). In Poland in the years 1960s, these interstadials were included in the Eemian (Różycki 1967), so the former stratigraphy was compatible with the one proposed here.

In Deszczowa Cave a similar peak of intensive weathering upon warm and wet climate is readable in the layer IV. Likewise the layers 12 and 13 in NC, this layer is characterized by the most distinctive weathering effects along the whole profile. It is clear that – on the evidence of rubble morphology, bone structure preservation and collagen content in bones – the layer IV from DC and the layers 12–13 from NC may be correlated with each other and located in the stratigraphical position of Eemian Interglacial. The underlying layers: 14–16 from NC and I–III from DC are related to Penultimate Glaciation (MIS 6). Such interpretation confirms the concept of Nadachowski *et al.* (2009), who placed the

lowest layers in DC (layers I–IV) in the pre-Vistulian (before MIS 5).

The sediments of layer V from DC, in the light of our results, had weathered under warm and humid conditions, only slightly more severe than the Eemian. These conditions allow to correlate this layer with the layers 10–11b in NC, and with the Early Vistulian. In the overlying layers VI and VII we can find an indication of increasing weathering, similar to situation observed in the layers 4 to 7 in NC. Such climatic succession allows us to correlate these sediments with climatic cooling in the middle of Interpleniglacial (MIS 3) and the further optimum in the final part of the same interstadial. The layer 3 in NC and the layer VIII in DC show similar degree of weathering, both on the basis of limestone rubble weathering and bones weathering. These layers seem to be accumulated under the most severe climatic conditions since the Saalian Glaciation (since MIS 6). In DC the next two layers – X and XI – represent the later part of the Upper Pleniglacial rather than the Late Glacial (compare with Cyrek *et al.* 2000, Nadachowski *et al.* 2009), due to indications of weathering still under severe climate.

### Discussion of the lithological data

In both caves there are some loess layers (see Fig. 1), *i.e.* layer VIII in DC and layers 14, 9 and 3 in NC. The loess layers may be correlated with main episodes of loess accumulation on the open sites of southern Poland and with the periods of periglacial conditions (Mojski 1985, Lindner 1992, Madeyska 2002, Mirosław-Grabowska 2002). The two best developed loess layers (3 in NC and VIII in DC) should be correlated with each other and with the main period of loess accumulation, *i.e.* the Upper Pleniglacial (Last Glacial Maximum, MIS 2). The severe climatic conditions indicated by loess character stay in accordance with our interpretation of

stratigraphy		MIS	layers from Nietoperzowa Cave			layers from Deszczowa Cave		
			layer number	archaeological cultures	<sup>14</sup> C date	layer number	archaeological cultures	<sup>14</sup> C dates
HOLOCENE		1	1	N		XI		
VISTULIAN (ULTIMATE) GLACIATION	Late Vistulian	2	2			X		19 310 ± 130 BP <sup>N</sup> (22 400 ± 200 BP <sup>L</sup> )
	LGM (Younger Plenivistulian)		3			IX	Em	19 680 ± 140 BP <sup>N</sup> (24 150 ± 200 BP <sup>L</sup> )
	younger warming (Denekamp ? Hengelo ?)	3	4	J		VIII	UP	(16 150 ± 280 BP <sup>C</sup> ) ~19 280 BP <sup>N</sup> 17 480 ± 150 BP <sup>C</sup> 19 530 ± 130 BP <sup>N</sup> 22 530 ± 169 BP <sup>N</sup> ~18 900 BP <sup>N</sup> 20 280 ± 400 BP <sup>N</sup> 24 280 ± 200 BP <sup>N</sup> 18 250 ± 120 BP <sup>L</sup> 20 800 ± 150 BP <sup>N</sup> 28 600 ± 400 BP <sup>N</sup>
			5	J		VII	A	(12 860 ± 110 BP <sup>L</sup> ) 24 620 ± 200 BP <sup>N</sup> (15 850 ± 130 BP <sup>L</sup> ) 26 200 ± 300 BP <sup>N</sup> ~24 470 BP <sup>N</sup> ~31 000 BP <sup>N</sup>
	cooling	3	6	J	38 160 ± 1 250 BP <sup>Ch</sup>	VI	MP	(> 43 000 BP <sup>N</sup> ) 44 000 ± 2 000 BP <sup>N</sup>
			7			V	Mq	(12 050 ± 600 BP <sup>L</sup> )
	older warming (Moershoofd ?)	4	8			IV	MP	(42 800 ± 1 900 BP <sup>L</sup> )
	Older Plenivistulian		9			III		
	Early Vistulian	5a 5b 5c 5d	10		Mq LM	II		
			11a, 11b			I		
12				LM				
13				LM				
EEMIAN INTERGLACIAL		5e	14					
SAALIAN (PENULTIMATE) GLACIATION	6	15						
		16						

Dates according to:  
<sup>Ch</sup> - Chmielewski (1975)  
<sup>C</sup> - Cyrek et al. (2000)  
<sup>L</sup> - Lorenc (2006)  
<sup>N</sup> - Nadachowski et al. (2009)  
 (in parenthesis) - dates treated in original publication as questionable

Archaeological cultures (after Chmielewski 1975 and Cyrek et al. 2000):  
 A - Aurignacian Mq - Micoquo-Prondnikian  
 Em - Epimagdalenian MP - unidentified Middle Palaeolithic  
 J - Jerzmanowician N - unidentified Neolithic  
 LM - Levallois Mousterian UP - unidentified Upper Palaeolithic

**Fig. 10.** Stratigraphical correlation between layers from Nietoperzowa Cave and Deszczowa Cave, proposed by the authors. All known radiocarbon dates for both sites and archaeological cultures are shown to support the correlation. Layers 1, 2 from Nietoperzowa Cave and layers I, II and XI from Deszczowa Cave were not examined in this work and their stratigraphical position presented here is only hypothetical.

cold palaeoclimate for the layers 14, 9 and 3 in NC and layer VIII in DC.

Nadachowski *et al.* (2009) proposed a wide hiatus between the layers IV and V in DC. Our analysis of cross-section of sediments in Deszczowa Cave shows an erosional episode, but in the upper position, with erosional surface at the bottom of the layer VI (Fig. 9). This erosional horizon fits well to our stratigraphical interpretation. The absence of any signs of climatic collapse over the Eemian layer in the lower part of the DC profile suggests that the sediments of Lower Pleniglacial (or Lower Plenivistulian, MIS 4) have been eroded. Probably the hiatus between the layers V and VI corresponds to very long period, encompassing late Early Vistulian, whole Lower Pleniglacial and Early Interpleniglacial.

### Discussion of the palaeontological data

The biostratigraphy of sediments from NC, presented by Kowalski (1961) and based on the palaeoecological analysis of rodent remains, is fully compatible with our stratigraphical interpretation.

Nadachowski *et al.* (2009) located the lowest layers from DC (layers I–IV) in position of the so-called Warta Glaciation (MIS 6) on the basis of palaeontological data. The

primitive rodents morphotypes (*Dicrostonyx cf. simplicior*, *Microtus oeconomus malei*) along with presence of small fox (*Vulpes cf. praeglacialis*) (Cyrek *et al.* 2000, Nadachowski *et al.* 2009) indicated the pre-Vistulian age of these sediments. A small wolverine from layer III (*Gulo gulo*, M.T. Krajcarz unpublished data), typical for the Saalian sediments, also confirmed our interpretation.

However, our new data stay in contradiction to the proposed by Nadachowski *et al.* (2009) position of layer IV. In their opinion that layer should be connected with late Saalian (Warta, MIS 6). Our data clearly suggest the interglacial age of that layer. Stratigraphical interpretation of Nadachowski *et al.* is based mainly on the participation of rodents species with different ecological requirements. However some non-quantitative data shown by them and also by Cyrek *et al.* (2000) may support our suggestion. These data include the presence of forest or thermophilic species in the sediments of layer IV, like: *Ursus arctos*, *Lutra lutra*, *Glis glis* or *Tetrao tetrix*.

### Discussion of the archaeological data

The Micoquian flint knives of Bockstein type were found between stone artifacts assemblage of layer V in DC

(Cyrek *et al.* 2000). Such artifacts are connected in Europe with either late Eemian or the phases of Early Vistulian (Derek 1981, Madeyska 2002), which allows to accept our interpretation of Early Vistulian age of layer V. In addition, the presence of Micoquian assemblage in layer V from DC supports the correlation of the mentioned layer with the layer 10 in NC, where the Micoquian artifacts were also found (Chmielewski 1975).

The assemblages of Jerzmanowician culture were found in layers 4–6 in NC and a single assemblage of Aurignacian culture in layer VII in DC. Both cultures indicate the early phase of Upper Palaeolithic (so they are connected with MIS 3) and allow us to correlate these layers with each other.

### Discussion of the geochronometrical data

The only radiocarbon date known from NC ( $38\,160 \pm 1\,250$  BP, charcoal, layer 6, Chmielewski 1975) stays in accordance with our stratigraphical scheme. The layer VI in DC, correlated by the authors with layer 6 and several older layers from NC, has two radiocarbon dates, both over 40 000 BP (Nadachowski *et al.* 2009). These dates are close to the limit of the method, however they allow us to accept the correlation. The layer VII in DC, correlated as directly younger than the layer 6 in NC, has many radiocarbon dates (see Fig. 10). The oldest of them (about 31 000 BP, Nadachowski *et al.* 2009) fits well to the correlation.

There are only two dates from the lower layers (Lorenc 2006; see also Fig. 10). One of them (about 12 000 BP) may not be accepted as it does not fit to the whole sequences of dates. It may be a consequence of an incorrect sampling (K. Cyrek, pers. comm.). The other date is acceptable, however is close to the method limit, so we suppose that the true age of this sediment might be much older than the date suggests.

The dates from the upper layers of DC (see Fig. 10), all between 15 000 BP and 30 000 BP, support our stratigraphical scheme.

There are several U/Th dates of speleothems known from DC (Cyrek *et al.* 2000). Some of them indicate the Middle Pleistocene age and are much older than the sediments, and the youngest ones correspond to the Eemian. The dated speleothems were found at the cave bottom, directly under the sediments of layer IV (K. Cyrek, pers. comm.). Data presented by Krajcarz (2010) suggest, that formation of speleothems was intensive during accumulation of layer IV. This allows us to connect the dates of the youngest speleothems with layer IV and to accept the Eemian age of that layer.

### Discussion of the geochemical data

Distribution of Mn precipitations and calcite cement indicates that the layer IV in DC was formed under the warmest and most humid climatic condition (Krajcarz 2010). The content of organic carbon, phosphates and  $\text{Fe}_2\text{O}_3$  in the sediment of layer IV, the highest over the whole profile, also confirms the warmest climate (Cyrek *et al.* 2000). It allows us to accept our interpretation of interglacial age of layer IV. The sediment of layer 12 in NC is characterized by the highest content of organic matter and Fe in the whole profile of NC (Madeyska-Niklowska 1969). This fact allows us to treat that

layer as interglacial sediment and to correlate it with layer IV in DC.

Krajcarz (2010) connected the second phase of calcite formation, however weaker than the first one, with layer VII in DC. It indicates warm conditions during sedimentation of that layer and allows us to correlate it with an interstadial. The presence of initial calcite covers in layers IX and X in DC (Krajcarz 2010) may probably be connected with later karst processes during Holocene. However Holocene layers were not examined in this study.

## CONCLUSIONS

Analysis of weathering parameters (or early diagenesis) of bones from cave sediments is a useful method in research of cave sites. It gives highly comparable results with lithological characteristics of cave sediments and with the fauna composition. This method allowed us to reconstruct the changes of temperature and humidity, and put a new light onto stratigraphy of sediments from Nietoperzowa Cave and especially Deszczowa Cave:

1. layer IV from Deszczowa Cave and layers 12–13 from Nietoperzowa Cave were formed upon the warmest and the most humid climatic conditions over the whole sequences of sediments from these sites (besides the Holocene, not studied here), and these layers should be correlated with Eemian Interglacial (MIS 5e);

2. the former stratigraphy of sediments from Nietoperzowa Cave, published in some earlier papers (summarized by Madeyska 1981) is confirmed; some doubts concerning the Eemian age of layer 12 are dispelled;

3. the original stratigraphical position of sediments from Deszczowa Cave (presented by Cyrek *et al.* 2000) should be changed as mentioned by Nadachowski *et al.* (2009), however the stratigraphy presented in the latter work seems only partly correct;

4. layer III from Deszczowa Cave corresponds to the Saalian Glaciation.

One should expect that the new tool presented here, would be used in future verification of the stratigraphy of other important Palaeolithic cave sites of southern Poland.

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## REFERENCES

- Allen J. R. M., Huntley B. 2000. Weichselian palynological records from southern Europe: correlation and chronology. *Quaternary International* 73/74, 111–125.
- Bell L. S. 1990. Palaeopathology and diagenesis; an SEM evaluation of structural changes using backscattered electron imaging. *Journal of Archaeological Science* 17, 85–102.
- Caspers G., Freund H. 2001. Vegetation and climate in the Early- and Pleni-Weichselian in northern central Europe. *Journal of Quaternary Science* 16, 31–48.
- Chavaillon-Dutrievoz N. 1955. État de surface des cailloutis et des

- vestiges osseux dans les couches archéologiques d'Arcy-sur-Cure. *Bulletin du Société Préhistorique Française* 52, 345–363 (in French).
- Chmielewski W. 1961. Civilisation de Jerzmanowice. IHKM PAN. Wrocław–Warszawa–Kraków, 1–92 (in French).
- Chmielewski W. 1975. Middle and Upper Palaeolithic (original: Paleolit środkowy i górny). In Chmielewski W., Hensel W. (eds), *Prehistory of Poland I: Palaeolithic and Mesolithic* (original: Prahistoria Ziemi Polskich I: Paleolit i mezolit), 9–158. Zakład Narodowy im. Ossolińskich Wydawnictwo PAN, Wrocław–Warszawa–Kraków–Gdańsk (in Polish).
- Cyrek K., Nadachowski A., Madeyska T., Bocheński Z., Tomek T., Wojtal P., Miękina B., Lipecki G., Garapich A., Rzebiń-Kowalska B., Stworzewicz E., Wolsan M., Godawa J., Kościów R., Fostowicz-Frelik L., Szyndlar Z. 2000. Excavation in the Deszczowa Cave (Kroczyckie Rocks, Częstochowa Upland, Central Poland). *Folia Quaternaria* 71, 5–84.
- Derek A.R. 1981. The Lower and Middle Palaeolithic periods in Britain. Routledge and Kegan Paul, London.
- Donner J. 1996. The Early and Middle Weichselian interstadials in the central area of the Scandinavian glaciations. *Quaternary Science Reviews* 15, 471–479.
- Hedges E.M., Millard A.R., Pike A.W. 1995. Measurements and Relationships of Diagenetic Alteration of Bone from Three Archaeological Sites. *Journal of Archaeological Science* 22, 201–209.
- Hedges R.E.M., Millard A.R. 1996. A diffusion-adsorption model of uranium uptake by archaeological bone. *Geochimica et Cosmochimica Acta* 60, 2139–2152.
- Iacumin P., Bocherens H., Mariotti A., Longinelli A. 1996. Oxygen isotope analyses of co-existing carbonate in biogenic apatite: a way to monitor diagenetic bone phosphate? *Earth and Planetary Science Letters* 142, 1–6.
- Kowalski K. 1961. Pleistocene rodents from Nietoperzowa Cave in Poland. *Folia Quaternaria* 5, 1–19 (in Polish with English summary).
- Krajcarz M. 2010. Geochemical record of diagenesis of bone remains from Deszczowa Cave (Częstochowa Upland). *Przeгляд Geologiczny* 58, 163–172 (in Polish with English summary).
- Laville H. 1964. Recherches sédimentologique sur la paléoclimatologie du Würmien récent en Périgord. *Anthropologie* 68, 1–48 (in French).
- Lindner L. (ed.) 1992. Quaternary – sediments, methods of researches, stratigraphy (original: Czwartorzęd – osady, metody badań, stratygrafia). Wydawnictwo PAE, Warszawa (in Polish).
- Lorenc M. 2006. Radiocarbon dating of some Late Pleistocene faunal assemblages in caves in Poland. *Acta Zoologica Cracoviensia* 49A, 41–61.
- Madeyska T. 1981. Le milieu naturel de l'homme du Paléolithique moyen et supérieur en Pologne à la lumière des recherches géologiques. *Studia Geologica Polonica* 69, 7–125 (in Polish with French summary).
- Madeyska T. 1982. The Stratigraphy of Palaeolithic sites of the Cracow Upland. *Acta Geologica Polonica* 32, 227–242.
- Madeyska T. 2002. Evidence of climatic variations in loess and cave Palaeolithic sites of southern Poland and western Ukraine. *Quaternary International* 91, 65–73.
- Madeyska-Niklowska T. 1969. Upper Pleistocene deposits in caves of the Cracow Upland. *Acta Geologica Polonica* 19, 341–390 (in Polish with English summary).
- Madeyska-Niklowska T. 1971. Methods used in researches on the upper pleistocene sediments of the Cracow Upland caves. *Światowit* 32, 5–25 (in Polish with English summary).
- Mirosław-Grabowska J. 2002. Geological value of Biśnik Cave sediments (Cracow–Częstochowa Upland). *Acta Geologica Polonica* 52, 97–110.
- Mojski J.E. 1985. Riverine, lacustrine and cave sediments and paleosols of non-coastal part of Polish Lowland and Southern-Polish Uplands (original: Osady rzeczne, jeziorne, gleby kopalne i utwory jaskiniowe na pozostałej części Nizy Polskiego i wyżyn środkowopolskich). In Mojski J.E. (ed), *Geology of Poland, I Stratigraphy, 3b Cenozoic, Quaternary* (original: Geologia Polski, I Stratygrafia, 3b Kenozoik, Czwartorzęd), 1–244. Wydawnictwo Geologiczne, Warszawa (in Polish).
- Mojski J.E. 2005. Polish Lands in the Quaternary. The outline of morphogenesis (original: Ziemia polskie w czwartorzędzie. Zarys morfogenezy). Państwowy Instytut Geologiczny, Warszawa (in Polish).
- Nadachowski A., Żarski M., Urbanowski M., Wojtal P., Miękina B., Lipecki G., Ochman K., Krawczyk M., Jakubowski G., Tomek T. 2009. Late Pleistocene Environment of the Częstochowa Upland (Poland) Reconstructed on the Basis of Faunistic Evidence from Archaeological Cave Sites. Institute of Systematics and Evolution of Animals, Polish Academy of Sciences, Kraków.
- Pfretzschner H.-U. 2004. Fossilization of Haversian bone in aquatic environments. *Comptes Rendus Palevol* 3, 605–616.
- Różycki S.Z. 1967. Pleistocene of Middle Poland (original: Plejstocen Polski Środkowej). PWN, Warszawa (in Polish).
- Smith C.I., Nielsen-Marsh C.M., Jans M.M. E., Collins M.J. 2007. Bone diagenesis in the European Holocene I: patterns and mechanisms. *Journal of Archaeological Science* 34, 1485–1493.
- Wojtal P. 2007. Zooarchaeological studies of the Late Pleistocene sites in Poland. Institute of Systematics and Evolution of Animals, Polish Academy of Sciences, Kraków.
- Wysoczański-Minkowicz T. 1969. An attempt at relative age determination of fossil bones by fluorine-chlorine-apatite method. *Studia Geologica Polonica* 28, 7–79.
- Wysoczański-Minkowicz T. 1975. Chronology of Late Pleistocene (original: Chronologia późnego plejstocenu). Sesja Naukowo-Sprawozdawcza Pracowni Geologii Czwartorzędu ZNG PAN – streszczenie referatów, Warszawa, 94–111 (in Polish).