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# PYRITE FRAMBOIDS IN PYRITIZED RADIOLARIAN SKELETONS (MID-CRETACEOUS OF THE PIENINY KLIPPEN BELT, WESTERN CARPATHIANS, POLAND)

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**Abstract:** Well preserved pyritized radiolarian skeletons have been found within the grey-green Mid-Cretaceous (Upper Cenomanian) shales in the Pieniny Klippen Belt (Carpathians, Poland). The skeletons contain numerous pyrite framboids in different positions, in channels and inside the abdomen of cryptothoracic forms, but their genetic context is not known. They were formed as a result of the reaction between dissolved iron and sulphide originated from the bacterial sulphate reduction. Two sources of organic matter, "post mortem" *in situ* decaying organic matter of radiolaria and disseminated organic matter from the surrounding sediment could be available for this process. Pyrite found in the radiolarians probably originates from different processes. It is suggested that pyritization of the radiolarian skeletons took place in the water column whereas pyrite framboids in the skeleton's free spaces could have been formed later during the diagenesis of the sediment. However, their simultaneous formation in the water column or in the sediment cannot be excluded.

**Key words:** Radiolaria, pyritization, framboids, Cretaceous, Pieniny Klippen Belt.

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## INTRODUCTION

Pyritized organic remains are common in sedimentary record. Pyrite generally replaces the organic matrix ("soft parts") and carbonate skeletons during all stages of sediment burial history, but may also replace silica skeletons during their journey through the anoxic water column (Bąk & Sawłowicz, 2000). It can adopt various forms, from massive to aggregated, euhedra, and framboids. Excellent descriptions of mechanisms of fossil pyritization were given by Fisher & Hudson (1985), Canfield & Raiswell (1991), Briggs *et al.* (1991), and Raiswell (1997).

Description and genetic interpretation of framboids from fossils is of great value for the depositional and diagenetic history, and also because the origin of pyrite spherules (framboids) is sometimes misinterpreted in some palaeontological studies. Especially in older papers, pyrite framboids were regarded to be microfossils, due to their spherical shape and similarity to bacterial colonies (Love, 1957; LaBerge, 1967). In papers dealing with ore deposits, where pyrite framboids are common, some authors still use the Schneiderhöhn's (1923) term "vererzten Bakterien" (ore-mineralized bacteria). In some cases growth of pyrite, especially transition from framboid (polyframboid) to euhedral crystal, can modify the morphology of different species, e.g., spore exines (Neves & Sullivan, 1964). Martill and Unwin (1997) have shown that the so-called blood corpuscles from blood vessels of an archosaurian limb bone (perhaps also from dinosaur bones) are pyrite framboids. Pyrite framboids are common as infillings of carbonate or silica skeletons of various organisms, but pyrite framboids in pyritized skeletons are much rarer, and to our best knowledge, have not been described yet in detail.

## DESCRIPTION OF THE STUDIED MATERIAL

Pyritized radiolarian skeletons are common in shales of the Pieniny Klippen Belt. The preservation and type of pyritization of these skeletons vary in environmentally different sub-basins of the Pieniny Klippen Belt (Bąk & Sawłowicz, 2000). The studied material includes samples from grey-green shales of the Jaworki Formation (Snežnica Siltstone Member) in the Kietowy stream section of the Branisko Succession (lower to middle bathyal; Bąk, 1995, 1996).

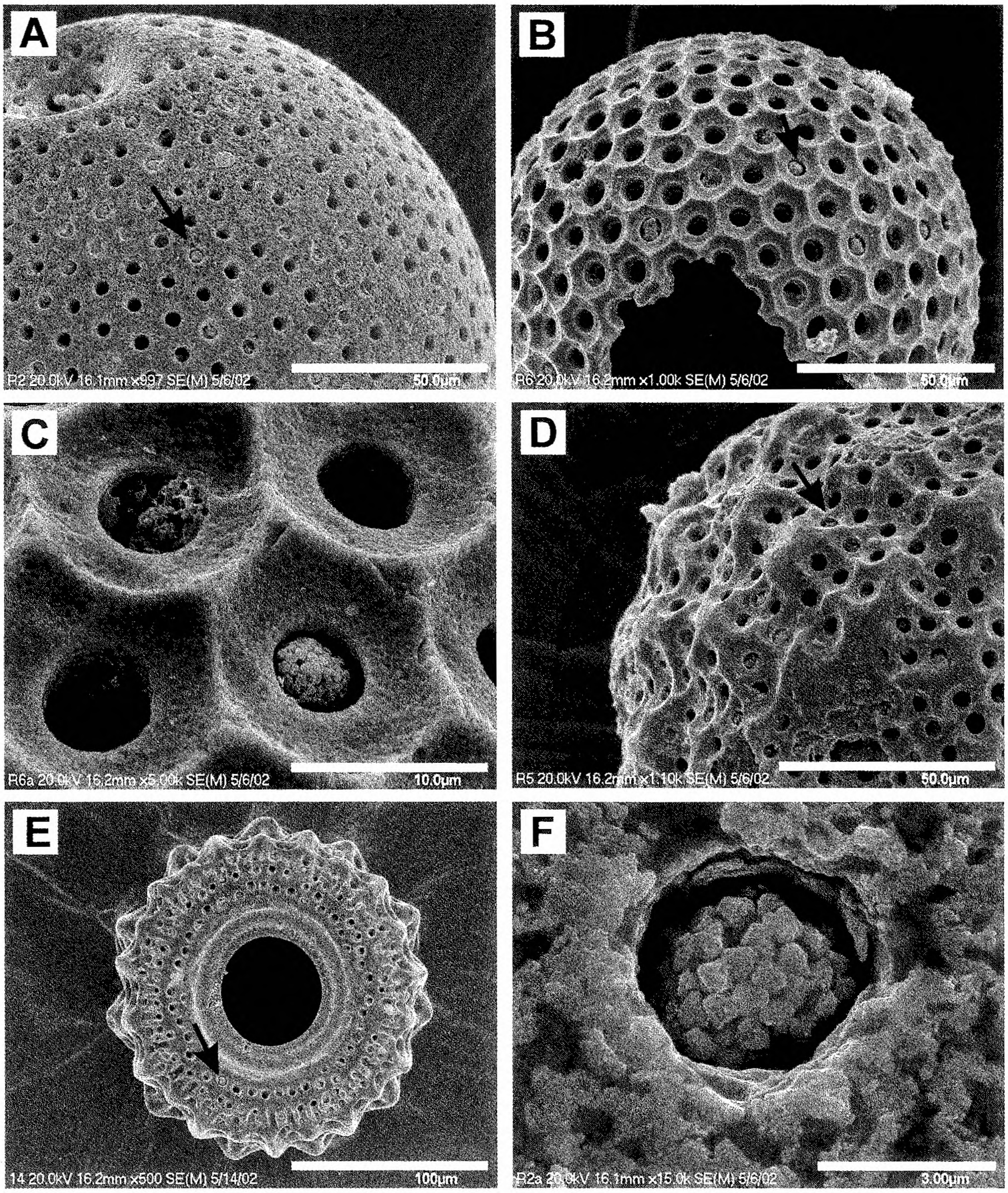

**Fig. 1.** Examples of framboids from pyritized radiolaria. **A** – pyritized radiolarian skeleton (*Holocryptocanium barbui* Dumitrica) with pyrite framboids visible in the uppermost parts of pores (scale bar 50 µm); **B** – pyrite framboids visible in lower parts of pores (arrow, compare with Fig. 1A) of the uncovered by erosion outermost layer of pyritized radiolarian skeleton of *H. barbui* (scale bar 50 µm); **C** – pyrite framboid and irregular cluster of pyrite crystals in pores of pyritized *H. barbui* (magnified fragment of Fig. 1B; scale bar 10 µm); **D** – pyritized radiolarian skeleton (*Holocryptocanium tuberculatum*) with pyrite framboids visible in pores (scale bar 50 µm); **E** – pyrite framboids visible in the last chamber of pyritized *Xitus mclaughilini* Pessagno (scale bar 100 µm); **F** – pyrite framboid with intergrown microcrystals in a pore of pyritized skeleton of *H. barbui* (scale bar 3 µm)

They contain excellent preserved radiolaria. The majority of the radiolarian skeletons are built of pyrite, with only a few siliceous skeletons. The radiolarian assemblage consists predominantly of Nassellaria. The best preserved specimens of pyritized radiolarians were studied using scanning electron microscope (SEM), equipped with EDS.

Pyrite very perfectly replaces even the finest details of ornamentation in the original silica skeletons of the studied samples (for details see Bąk & Sawłowicz, 2000). At lower magnifications (below 1000x), SEM images reveal very even surfaces of pyritized skeleton elements. However, higher magnifications (5000–10000x) show that these skeletons are built of masses of small irregular grains of pyrite, intergrown or closely packed, sometimes with pores.

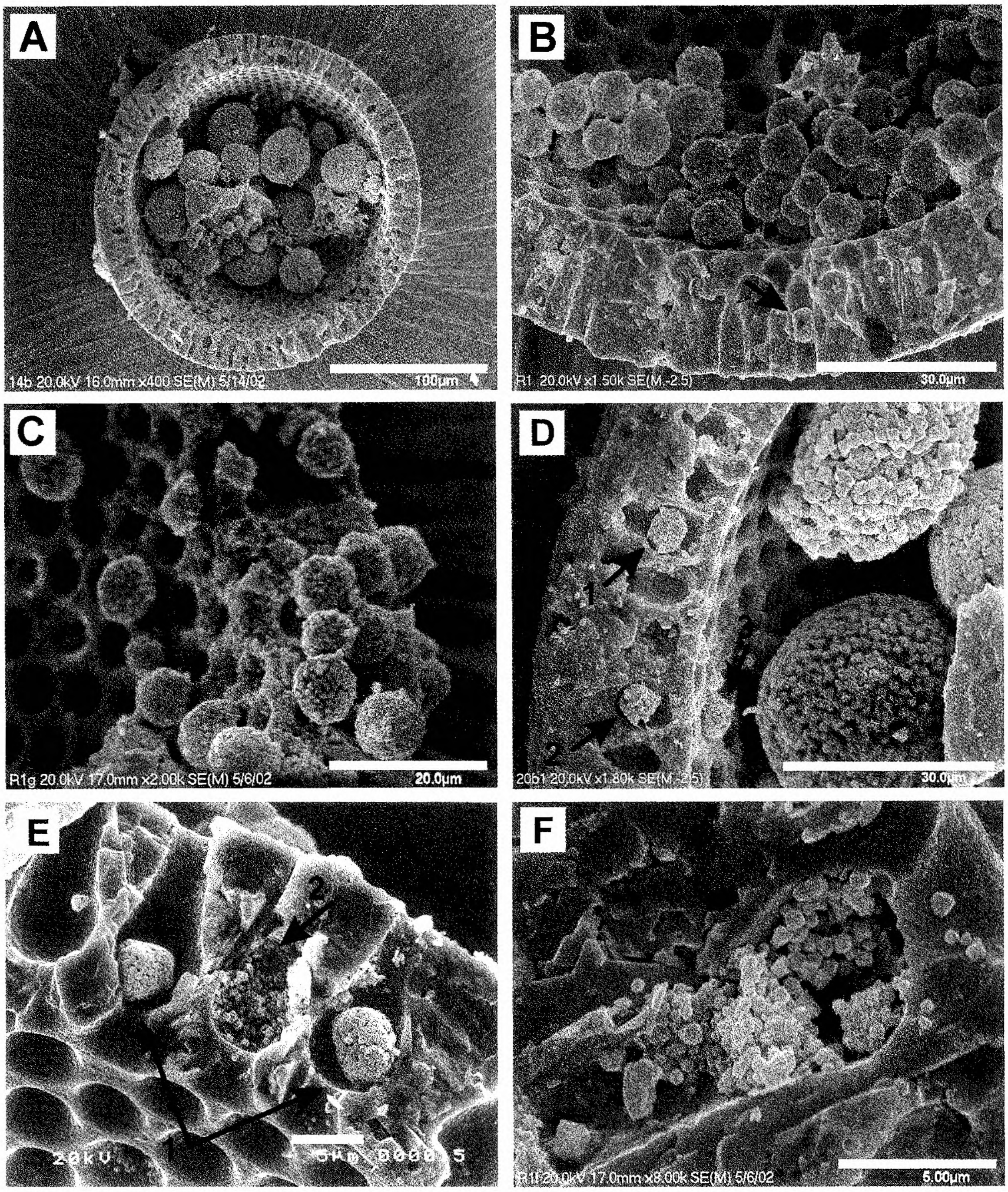

**Fig. 2.** Examples of framboids from pyritized radiolaria. **A** – cross-section of pyritized *H. barbui*: cluster of pyrite framboids of various sizes in the interior of the abdominal chamber (scale bar 100 µm); **B** – cross-section of pyritized *H. barbui*: cluster of pyrite framboids of uniform sizes in the interior of the abdominal chamber (some of the framboids are attached to channels exits; arrowed framboid modified by the shape of channel; scale bar 30 µm); **C** – pyrite framboids attached to inner, outer and broken parts of pyritized *H. barbui* (growth on the broken skeleton in a sediment or artificial destruction during laboratory preparation?; scale bar 20 µm); **D** – cross-section of pyritized *H. barbui*: angular "hipidiomorphic" (1) and irregular in outline (2) pyrite framboids in pores; (3) – pyrite framboid attached to a channel exit (scale bar 30 µm); **E** – crystal-like pyrite framboids in the so-called lamp chimney-like pores of *H. barbui*: (1) – the shapes are probably modified by the shape of pores (framboids removed from their primary position?); (2) – a pore filled by the aggregate of pyrite microcrystals (scale bar 5 µm); **F** – framboidal aggregate (disintegrated framboid or encrustation of channel inner surface?) inside of a channel of *H. barbui* (scale bar 5 µm)

SEM-EDS studies showed that pyrite is actually the only sulphide mineral present, whereby silica or silicates are lacking.

The pyrite framboids are common in the pyritized radiolarian skeletons, usually within confined spaces. They typically occur in two different positions: 1) in channels (pores) (Figs 1A–F), 2) inside the abdomen of cryptothoracic forms, attached to an internal surface, often at the channel exit (Fig. 2A–C).

Different morphological forms of pyrite: framboids, ag-

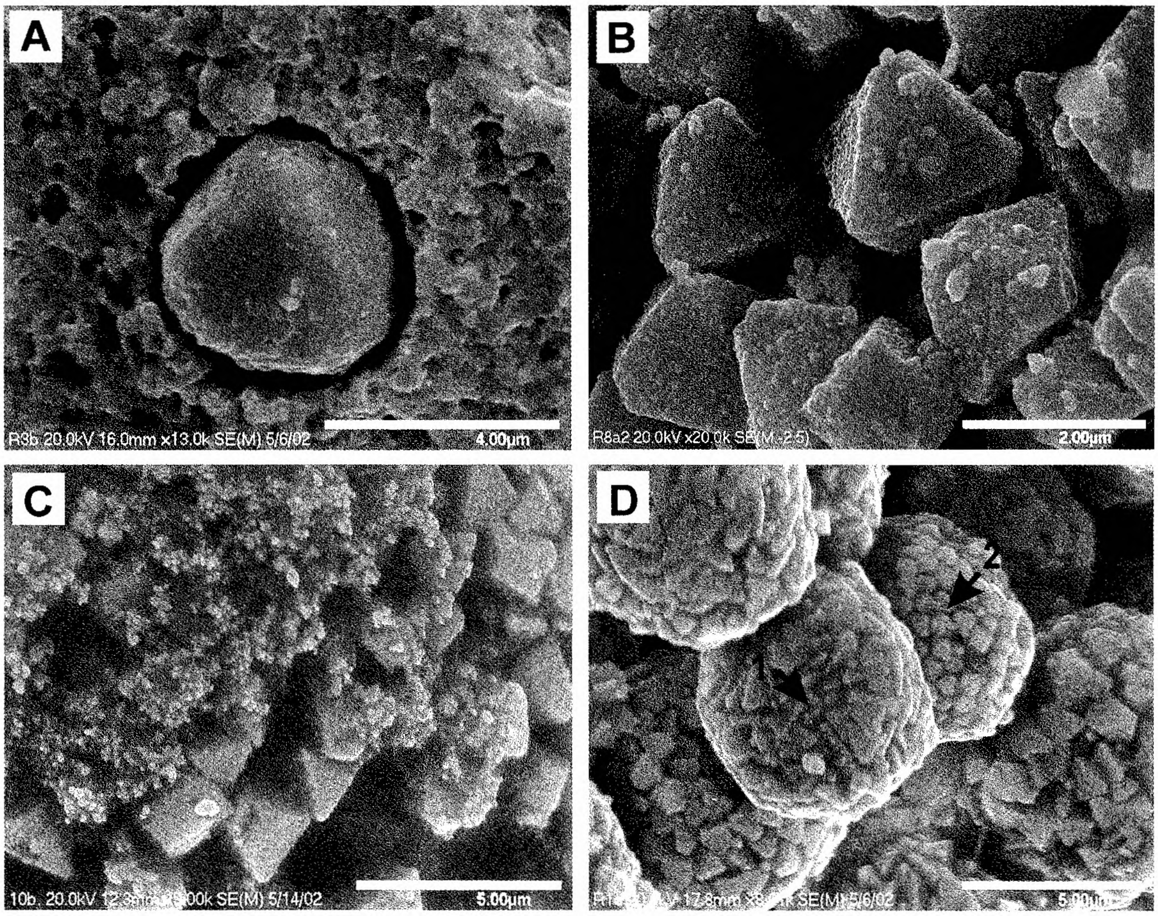

**Fig. 3.** Examples of pyrite from pyritized radiolaria. **A** – pyrite euhedra (possibly recrystallized framboid) in a pore of pyritized *H. barbui* (scale bar 4 µm); **B** – octahedral microcrystals of pyrite framboid from the abdomen of pyritized *H. barbui* (note that microcrystals are built of smaller subunits ranging from 50 to 100 nm – crystal-like microframboid *sensu* Sawłowicz, 1993; scale bar 2 µm); **C** – pyrite octahedral microcrystals (see Fig. 3B) encrustated by minute (200–300 nm) pyrite grains (abdomen of pyritized *H. barbui*; scale bar 5 µm); **D** – pyrite framboids built of closely packed (1) and strongly intergrown microcrystals (2) (pyritized *H. barbui*; scale bar 5 µm)

gregates, and euhedra can be observed in channels (pores). The percentage of pores filled by framboids can be as high as around 40. The size of framboids is rather uniform, averaging at 5 µm. Framboids occur in various positions inside the channels (Fig. 1B, 1C, 2D). They can be seen from both the outer and the inner side, depending on the preservations of layers forming the skeleton. The shape of framboids is not always regular and spherical. Limited space inside the pore might limit the size and modify the shape of the framboids. This is evident in pores of Challengerian structure (lamp chimney – like pores, Fig. 2B, 2E). Closely (Fig. 2E) or loosely (Fig. 2F) packed aggregates of pyrite microcrystals often fill the pores. Some of them may represent disintegrated framboids. Sometimes individual microcrystals of a size similar to those building framboids are scattered on the walls of the pores (Fig. 2F). Pyrite euhedra are rare (Fig. 3A) and could represent a final stage of the framboid growth (see Sawłowicz, 1993).

Individual framboids and their clusters attached to the inner surface have been found only inside the abdominal chambers of *H. barbui*. However, it cannot be excluded that they also occur in other species skeletons, because only skeletons of *H. barbui* have been found broken and available for the observations of their interior. The outline of the framboids in clusters is usually very regular. The size of the framboids is usually uniform (~15 µm; Fig. 2B) but occasionally varies from 5 to 40 µm (Fig. 2A). The size ratio of microcrystals forming the framboids to the whole framboid can be differentiated (Fig. 2D, 3D). The morphology of microcrystals forming framboids varies from idiomorphic to xenomorphic. They are often overgrown and/or welded at one point (Fig. 3B, 3D). It should be emphasized that microcrystals are sometimes made of smaller subunits (Fig. 3B) or encrustated by minute pyrite grains (Fig. 3C).

## DISCUSSION

Different styles of fossilisation, i.e. pyritization of silicieous skeletons and formation of pyrite framboids in free spaces (pores and abdomens) in fossil skeletons, distinguished in the studied samples, suggest a differentiation of the pyritization processes.

Pyrite may be formed via at least three pathways, including: (1) the reaction of precursor sulphides with polysulphides, (2) the progressive solid-state oxidation of precursor iron sulphides, and (3) the oxidation of iron sulphides by hydrogen sulphide, with different rate of the formation for each process and for a greigite intermediary (see a review in Rickard *et al.*, 1995). In low temperatures pyrite growth is usually preceded by the formation of unstable iron monosulphides (Berner, 1980, 1984; Sweeney & Kaplan, 1973).

Different genetic origins have been proposed for framboids, ranging from a purely inorganic origin, based on laboratory synthesis (e.g., Farrand, 1970; Křibek, 1975; Graham & Ohmoto, 1994), through indirect biogenic formation (e.g., Kalliokoski & Cathles, 1969; Lougheed & Mancuso, 1973), to a direct biogenic origin (e.g., Love, 1957; Locquin & Weber, 1978). Recent reviews by Wilkin and Barnes (1997a), and Sawłowicz (2000) suggest that framboids may form by aggregation, possibly enhanced by magnetic properties of the monosulphide precursor. Biogenic processes are not necessary for the framboid formation but can be helpful, especially in the production of sulphide. Common occurrence of framboids in sediments rich in organic matter results mainly from the availability of sulphide from the bacterial sulphate reduction (BSR) which, in turn, depends on the presence of metabolizable organic matter. The organic matter may also be helpful in stabilization of gels and colloids, which are important for the framboid formation (Sawłowicz, 2000).

The radiolarian skeletons represent a special environment for pyrite framboid formation with their open space and decaying organic matter. Formation of pyrite depends on the availability of iron and sulphur. Iron hydroxides and oxides supply most of the reactive iron used in the Fe-sulphide formation (Boesen & Postma, 1988; Canfield, 1989). In sedimentary environments, the major source of sulphur incorporated into iron sulphides is $H_2S$ or $HS^-$, resulting from the BSR. There are many pieces of evidence that precipitation of pyrite framboids in organic-rich environments is biologically induced. A role of sulphate reducing bacteria seems to be especially important although still not clear. Kohn *et al.* (1998) observed strong fractionation of sulphur isotopes in foraminifera chambers and suggested that bacteria were intimately involved in the production of pyrite. On the other hand, Benning *et al.* (1999) found through experimental studies that sulphate-reducing bacteria do not precipitate iron sulphides directly, and that their role is probably limited to the production of bisulphide ions. Bacterial cell walls can also serve as preferred nucleation sites for metal sulphides and make adsorbed metal ions more reactive (see review in Fortin *et al.*, 1997). Bacterial sulphate reduction depends on the availability of sulphates and organic matter. Sulphates are abundant in the marine environment and their reduction to sulphide occurs mainly at the site of the decomposing organic carbon either in anoxic marine sediments (Berner, 1980) or in the water column (e.g., Black Sea – Muramoto *et al.*, 1991). Two sources of organic matter could be active during the formation of framboids in radiolarian skeletons. One is the “post mortem”, *in situ* decaying easily metabolizable organic matter of the living organism, including symbiotic bacteria. It produces an anaerobic microenvironment and local nutrients for a growth of bacteria (e.g., *Desulphovibrio desulphuricans*), which reduce sulphates from the surrounding seawater to sulphide (Berner, 1984, Kohn *et al.*, 1998). It should be stressed out that in that case the hosting environment need not be anoxic. The other or additional source of organic matter is the surrounding sediment. In that case formation of the framboids in skeleton requires $H_2S$ produced in the sediment outside of the skeleton and its transfer into it. These processes may not always be genetically related to pyritization of skeletons, if fossils serve only as available spaces in the sediment.

The time of the pyrite framboid formation and its relationship to the pyritization of the skeletons is difficult to estimate. Pyrite framboids typically form in an euxinic water column and/or during early diagenesis in a sediment (Lyons, 1997; Wilkin & Barnes, 1997a, b; Sawłowicz, 2000). Bąk and Sawłowicz (2000) assumed that pyritization of skeletons responsible for the excellent preservation of the radiolarians described here took place in the anoxic water column. It cannot be excluded that framboids were formed within the skeletons, especially in their pores, during their travel to the sediment and simultaneously with the pyritization of the skeletons. However, in our opinion their formation during diagenesis of the sediment is much more probable. The occurence of framboids in pores or attached to the internal surface of pyritized radiolarian skeletons suggests that the framboid formation took place after the pyritization of skeletons. One can expect that their simultaneous formation would lead to an intergrowing what is not observed. Earlier formation of framboids in siliceous radiolarian skeletons is hardly possible, because the subsequent process of skeleton pyritization would cause the infilling of framboid interstices, overgrowths on framboid or framboid growth to euhedra (see Sawłowicz, 1993). This was not observed in the studied samples. It is not clear if framboids from the pores and those from the abdomens of the radiolarian skeletons formed in the same process. A major peak of framboid formation is typical for the early stages of diagenesis when the bacterial sulphate reduction is most active. Late diagenetic formation of pyrite framboids is rare, depending on the rejuvenation of BSR processes under specific conditions.

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## Streszczenie

# FRAMBOIDY PIRYTOWE W SPIRYTYZOWANYCH SZKIELETACH RADIOLARII (KREDA ŚRODKOWA, PIENIŃSKI PAS SKAŁKOWY, KARPATY ZACHODNIE, POLSKA)

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Doskonale zachowane, spirytyzowane szkielety radiolarii zostały znalezione w szaro-zielonych środkowokredowych łupkach pienińskiego pasa skałkowego (górny cenoman) (PPS-Karpaty Zachodnie, Polska). Szkielety mieszczą w sobie także liczne framboidy pirytowe. Piryt budujący szkielety oraz framboidy powstał w wyniku odmiennych procesów. Pirytyzacja szkieletów radiolarii prawdopodobnie miała miejsce w kolumnie wody, podczas gdy framboidy pirytowe wypełniające wolne przestrzenie szkieletów formowały się później, podczas diagenezy osadu.

Framboidy pirytowe występują w szkieletach w dwóch różnych położeniach: w kanałach (Fig. 1A–F) i wewnątrz komory brzusznej form kryptotoraksalnych, przyczepione do wewnętrznej powierzchni, często u ujścia kanału (Fig. 2A–C); lecz ich kontekst genetyczny nie jest znany. Wielkość framboidów jest podobna, średnio około 5 μm. Framboidy wewnątrz kanałów występują w różnych położeniach (Fig. 1B, 1C, 2D). Można je obserwować zarówno od strony zewnętrznej jak i wewnętrznej, zależnie od stopnia zachowania warstw budujących szkielet. Kształt framboidów nie zawsze jest kulisty. Ograniczona przestrzeń wewnątrz porów mogła wpływać na wielkość i modyfikować kształt framboidów, co jest szczególnie widoczne w porach o kształcie "lampy naftowej" (Fig. 2B, 2E). Pory często są wypełnione przez ciasno (Fig. 2E) lub luźno (Fig. 2F) ułożone agregaty mikrokryształów pirytu. Niektóre z nich mogą reprezentować zdezintegrowane framboidy. Niekiedy pojedyncze mikrokryształy są przyczepione do ścianek porów (Fig. 2F). Duże idiomorficzne kryszttały są rzadkie (Fig. 3A) i mogą reprezentować końcowy etap wzrostu framboidów (Sawłowicz, 1993).

Pojedyncze framboidy i ich skupienia przyczepione do wewnętrznej powierzchni szkieletu zostały znalezione tylko wewnątrz brzusznej komory *Holocryptocanium barbui*. Należy wspomnieć, że szkielety jedynie tego gatunku znaleziono pokruszone, co pozwoliło na obserwacje ich wnętrza. Zarys framboidów w skupieniach jest sferyczny, a ich wielkość jest zwykle podobna (około 15 µm) (Fig. 2B), niekiedy wahając się od 5 do 40 µm (Fig. 2A). Stosunek wielkości mikrokryształów tworzących framboidy do całego framboidu może być rozmaity (Fig. 2D, 3D). Morfologia mikrokryształów budujących framboidy waha się od idiomorficznych po ksenomorficzne. Często są one zrośnięte lub poprzerastane (Fig. 3B, 3D). Należy podkreślić, że mikrokryształy są czasami złożone z mniejszych cząstek (Fig. 3B, 3C).

Framboidy pirytowe utworzyły się w wyniku reakcji pomiędzy rozpuszczonym żelazem i siarką powstałą w wyniku bakteryjnej redukcji siarczanów. Substancja organiczna niezbędna dla tego ostatniego procesu pochodziła z dwóch różnych źródeł. Jedno z nich to pośmiertny rozkład "*in situ*" materii organicznej żywego organizmu, zaś drugi to materia organiczna rozproszona w otaczającym osadzie.