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**THE ASSOCIATION OF URANIUM, VANADIUM AND ORGANIC MATTER
IN THE COPPER DEPOSITS IN WEISSLIEGEND SANDSTONES
(FORE-SUDETIC MONOCLINE, POLAND)**

Abstract. Two vanadium minerals, montroseite and roscoelite, occur in thucholite- and coffinite-bearing copper ores in the Weissliegend sandstones (Polkowice mine near Lubin) the Fore-Sudetic monocline. These minerals occur with organic matter that is characterized by a high degree of structural ordering and that forms shapeless accumulations metasomatically replacing quartz grains. The organic matter that controls this paragenesis is associated with thucholite. The vanadium and uranium mineralization in these sandstones shows similarities to the sandstone-bearing U-V deposits of the Colorado Plateau (USA).

Key-words: Fore-Sudetic monocline, thucholite, coffinite, montroseite, roscoelite, Colorado Plateau

INTRODUCTION

Investigations on the radioactivity of copper-bearing strata and their mineralization within the Fore-Sudetic monocline started in the 1960s (Grabczak, Niewodniczański 1962; Jarosz, Zaczek 1966). Subsequent studies focussed on thucholite, coffinite and pitchblende (Banaś, Jarosz 1972; Banaś et al. 1978; Piestrzyński 1988, 1989, 1990; Banaś, Salamon 1991). The late Witold Salamon initiated investigations of coffinite occurrences, providing not only a description of this interesting uranium silicate (Salamon, Banaś 2002) but also determining its paragenesis with thucholite and other compounds of vanadium. Despite substantial reserves of vanadium, no vanadium minerals were initially identified. Therefore, the problem of vanadium beneficiation and its recovery from these copper deposits remained unresolved although considerable amounts of vanadium had been recovered from the similar German deposits in Mansfeld; according to Schüller (1959), the annual production of metallic vanadium in Germany was about 40 tonnes at that time.

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In the Fore-Sudetic monocline deposits, the average vanadium content, contained mainly in the shale ore, is 0.11 wt.%, being 83 ppm in the sandstone ore and 32 ppm in the carbonate ore (Kucha et al. 1983). Tokarska (1971) and Szczepkowska-Mamczarczyk (1971) suggested an association of vanadium with organic material. In organic matter from the copper-bearing shales, the vanadium content reaches 0.53% occurring mainly in the form of organic compounds, e.g., vanadyl ethioporphyrins (Kucha et al. 1983). V-porphyrins, together with Ni-porphyrins, were also found in bitumen extracts from the copper-bearing shales (Sawłowicz 1985). Kucha et al. (1983) did not confirm the presence of the vanadium minerals, patronite (VS₂) or quisquite (a V-bearing variety of lignite), as reported by Wojciechowska and Serkies (1969).

Isolated, spherical and discoidal concretion-like accumulations (< 1.5 mm) of thucholite, occur as horizontally-arranged, colony-like forms in the ore zone of the copper deposit (Banaś et al. 1978). More recent observations have shown that the thucholite concretions are present only at the junction between the white-grey sandstones and the organic shales. It is important to stress that vanadium minerals have been found not in the copper-bearing organic shales that are richest in vanadium, but in sandstones containing considerable amounts of organic matter and uranium.

METHODS

Reflected light mineralogical investigations involved the use of Opton and Axiolab (Zeiss, Germany) microscopes with immersion objectives. SEM-EDS analyses were carried out using a Leo 1430 (Germany) electron microscope coupled with an ISIS 300 X-ray energy dispersion (EDX) microprobe (Oxford Instruments Ltd., England). X-ray structural analyses were conducted using a CBS120 transmission diffractometer (INEL, France) equipped with a position-sensitive counter and a quartz monochromator (CoK_α radiation, exposure time 10 hrs, sample size 0.3 mm). In the examination of the morphological features and molecular orientation of the carbonaceous matter, a Philips EM 400 transmission electron microscope (TEM) operating at 100 kV was used, applying various techniques such as bright field (BF), dark field (DF) and selected-area electron diffraction (SAED).

RESULTS

The minerals studied are fine-grained, often sub-microscopic. Thus a number of microscopic determinations could not be made. For this reason, optical observations were combined with chemical analyses under a scanning electron microscope.

Thucholite- and coffinite-bearing uranium accumulations in the sandstones occur in different forms. On the micro scale, both mineral types contain relics of partly corroded quartz grains and are commonly separated by essentially barren zones (Salamon, Banaś 2002). The thucholite assemblages form fine layers with varying orientation; they do not follow the lamination of the enclosing sandstone. Thucholite also occurs in shales and

sandstones as discoidal or oblong accumulations, and as veins in limestones. Coffinite occurs as reaction rims on quartz grains (Fig. 1). This mineral is typically only associated with nickel and cobalt minerals whereas thucholites are accompanied by platinoids, electrum, native copper and simple copper sulphides.

The carbonaceous matter is one of the basic components of the thucholite-bearing sandstones. The quartz grains of these sandstones (Fig. 2) are heteromorphous and

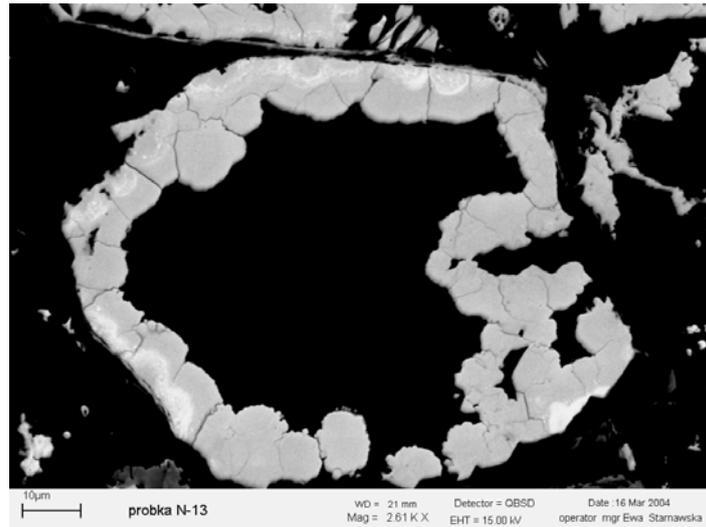


Fig. 1. Coffinite rims (light grey — U-poor; white — U-rich) on a corroded quartz grain (black center). SEM image

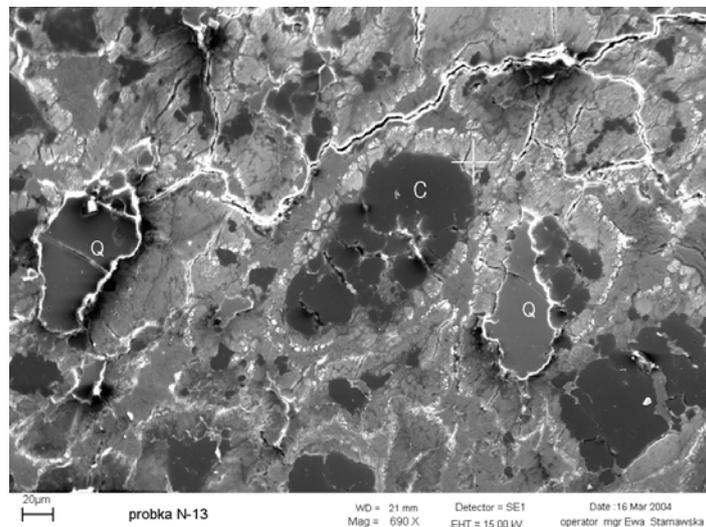


Fig. 2. Accumulations of carbonaceous matter (C) and corroded quartz grains (Q) with uranium oxides (white) and thucholite (grey) rims. Occasionally montroseite occurs with uranium oxides (both phases are indistinguishable in this image). Grey flakes in the thucholite are roscoelite. SEM image

show irregular, corroded borders against the surrounding carbonaceous matter. Veins or lenses of copper sulphides (mainly bornite) occur in the thucholite aggregates. Under a reflected light microscope, the carbonaceous matter is seen to form colonies or irregular accumulations ranging in size from single micrometers to 0.6 mm. Along their margins, grains of uranium oxide accumulate. This oxide is uraninite according to Banaś et al. (1978). The carbonaceous matter is anisotropic and reflectance measurements on a number of particles gave R_o values between 3.6 and 3.8%. These values indicate a high rank of organic matter maturation typical of meta-anthracite (Kwiecińska 1980). In contrast, Kucha (1993) reported that the reflectance of the anisotropic component of the thucholite is close or equal to that of graphite; precise values were not given.

The reflectance data correlates well with the TEM results. The carbonaceous matter consists of small granular particles that are irregular in shape and size. Individual particles are very inhomogeneous with a porous microstructure (Fig. 3). Under an electron beam, they show turbostratic structure in the sense of Bonijoly et al. (1982) or very weak crystalline order (Fig. 4); they can be considered as intermediates between coals and graphite. An electron diffraction pattern typical of graphite was not obtained (Kwiecińska, Petersen 2004) and anisotropic lamellar carbons were not found contrary to a conclusion of Kucha (1993). As the particles are too small to be detected by X-ray diffraction, structural parameters and d_{002} values (mean interplanar distance) are impossible to measure. Therefore, it is difficult to determine the precise structural route by which the carbonaceous material became graphitized.

The discoidal aggregates of thucholite in the sandstone range in size up to 1.5 mm. In reflected light, they resemble those observed by Banaś et al. (1978). The thucholite matter, composed of light and dark varieties with embedded quartz relicts, contains secondary veins and lenses of bornite, chalcopyrite and covellite of a septarian cha-

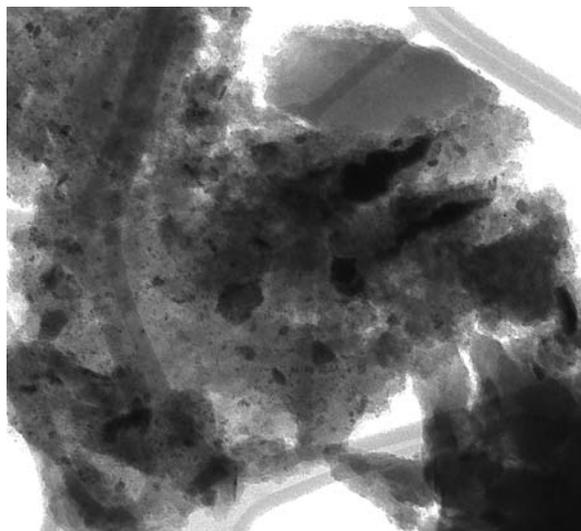


Fig. 3. Carbonaceous matter consisting of small granular particles of different shapes and sizes. Each particle shows a very inhomogeneous and porous microstructure. TEM, magn. 30,000x



Fig. 4. Turbostratic structure of a particle from Figure 3. TEM, electron diffraction pattern

racter. Detailed SEM observations indicate that thucholite rims with leaf-like shapes overgrow the accumulations of carbonaceous matter or quartz grains (Fig. 5). Formed during the process of thucholitization, these rims corrode all of the components in the sandstone ore and, locally, even penetrate quartz grains.

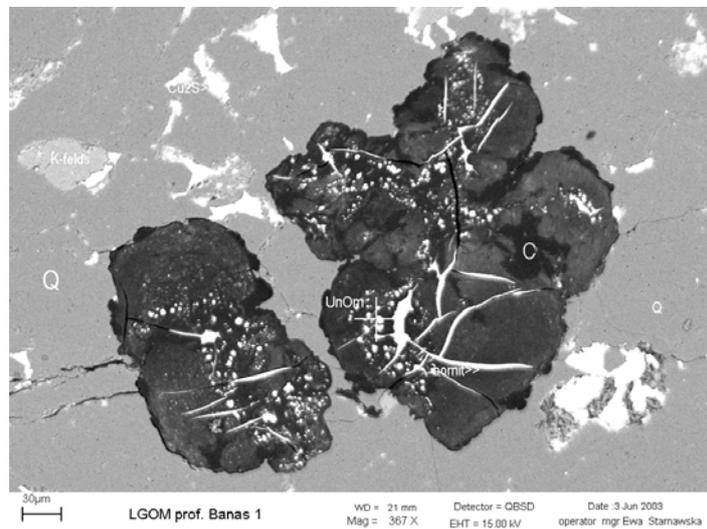


Fig. 5. Sandstone with large accumulations of carbonaceous matter (black) containing bornite (white, stream-like concentrations). Inclusions of uranium oxides (white spots, marked as U_nO_m as the degree of uranium oxidation has not been determined) within thucholite. Grains of chalcocite (white spots) are present among quartz grains. SEM image

The presence of the vanadium minerals, roscoelite and montroseite, was confirmed using reflected light microscopy and SEM and in EDS spectra. Roscoelite $KV_2[AlSi_3O_{10}(OH)_2]$ is the major vanadium-bearing mineral in the sandstone where it occurs as irregular accumulations ranging in size up to 0.8 mm in the porous spaces between grains of quartz, thucholite and rare rutile (Figs. 6, 7). The mineral is yellow-olive in colour with a vitreous-pearly lustre. In reflected light, this vanadium mica appears as accumulations of scaly grains with uneven surfaces and visible cleavage.

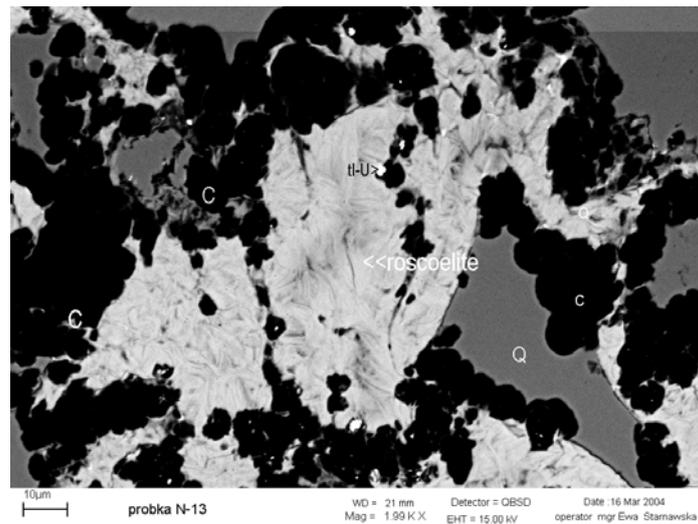


Fig. 6. Carbonaceous matter (C) accumulated around corroded quartz grains (Q). Flake-like, neogenic roscoelite is a component of the sandstone cement. SEM image

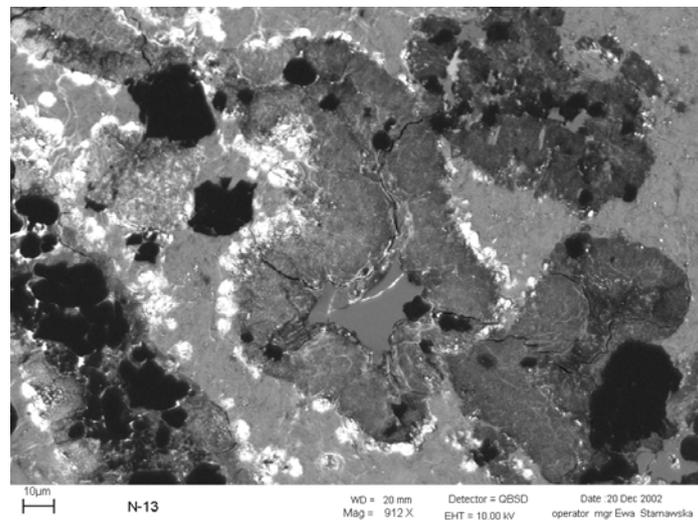


Fig. 7. A sandstone fragment rich in roscoelite cement and carbonaceous matter (black). A corroded quartz grain (center, smooth surface) is rimmed by coffinite that contains inclusions of uranium oxides (white spots) in their outer parts. SEM image

Bireflectance and anisotropy are weak. The mineral shows irregular fractures filled with an unidentifiable submicroscopic substance. As noted above, the presence of roscoelite was confirmed by EDS (Fig. 8).

Montroseite VO(OH) is mostly present within the roscoelite as dispersed, dust-like particles with sizes ranging upwards from <1 μm ; some reach 30 μm (Fig. 2). The mineral was found in small amounts using SEM. As it is difficult to distinguish montroseite in reflected light, the optical properties given below are those of suspected montroseite: colour light grey with a bluish tint, $R < 20\%$, lack of bireflectance, weak anisotropy and suppressed by strong, yellow-brownish internal reflections. Montroseite also occurs within quartz as string-like accumulations. Montroseite appears to be the younger of the two vanadium minerals though the crystallization succession is difficult to establish. The presence of montroseite was confirmed by EDS (Fig. 9).

Within the roscoelite- and montroseite-bearing zones, a mineral with properties similar to those of karelianite (V_2O_3) occurs. The following features characterize this rare mineral: isometric, oval grains with grey-olive colour; reflectance about 18%

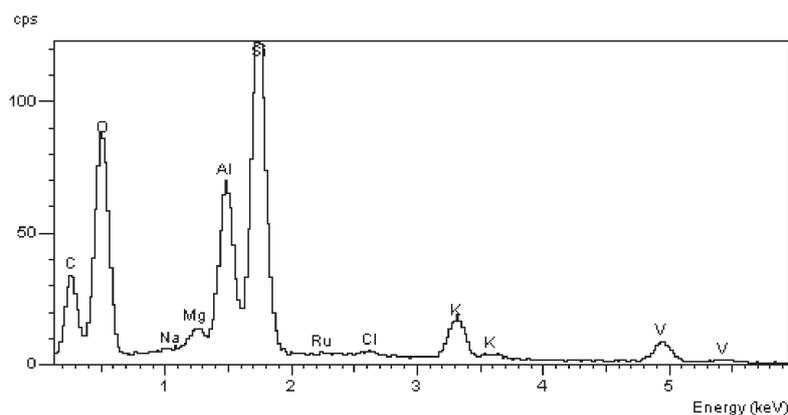


Fig. 8. EDS spectrum of roscoelite

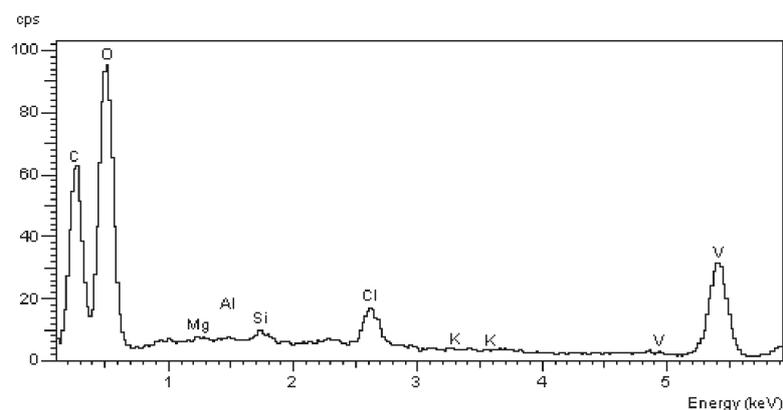


Fig. 9. EDS spectrum of montroseite

(higher than that of roscoelite); weak ΔR , distinct anisotropy; lack of internal reflections and a polishing hardness significantly higher than that of the enclosing ore minerals. The suspected karelianite commonly corrodes thucholite. It also occurs as inclusions in roscoelite (with no signs of corrosion) and is intergrown with chalcopyrite, bornite and covellite.

Accumulations of uranium oxide with sizes in single nanometers concentrate on the fringes of thucholite and roscoelite (Figs. 10, 11). The presence of uranium oxides was

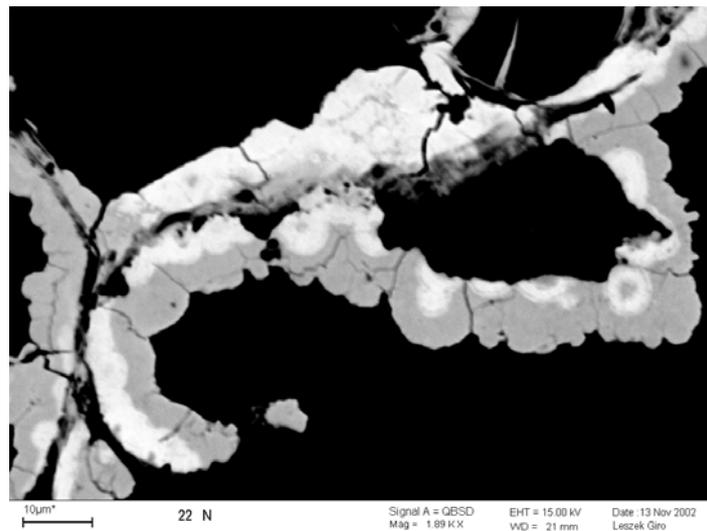


Fig. 10. Spherical concentrations of uranium oxide at the border between thucholite and roscoelite. Thucholite concentrations developed as light grey, suede-like bands. SEM image

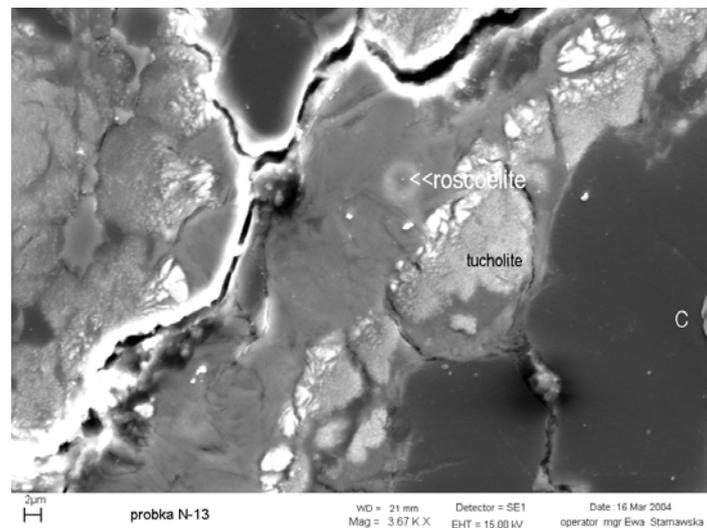


Fig. 11. Spherical concentrations of uranium oxide at the junction between thucholite and roscoelite. SEM image

confirmed by EDS spectra (Fig. 12). The thucholite contains highly dispersed uranium oxides and, possibly, vanadium oxides as well. Due to their very fine grain-size and a contrast approximating that of the uranium minerals, the suspected vanadium oxides could not be positively identified even using XRD or SEM-EDS.

Closer microscope observation reveals the presence of colloform uranium oxides on the margins of thucholite. The oxide grains are scaly, elongated, in some cases spherulitic or oval. The cores of uranium oxide inclusions within thucholite comprise a mineral with optical properties close to those of pitchblende rimmed by an unidentified substance. Radial synergetic (shrinkage) fractures in the pitchblende contain phases

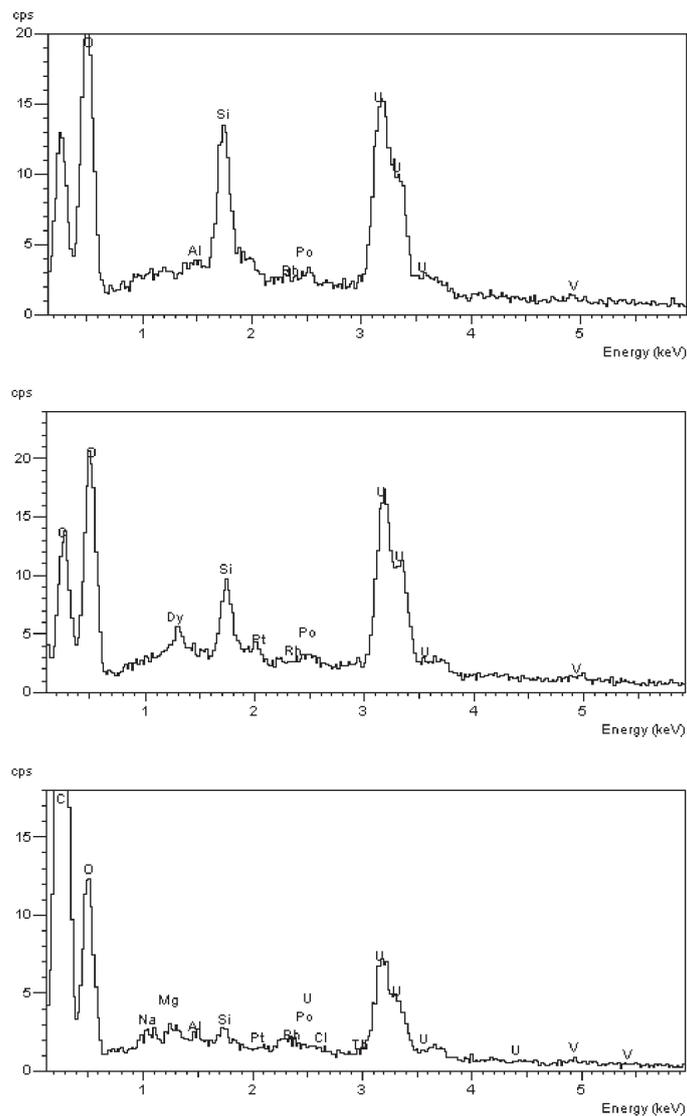


Fig. 12. EDS spectra of uranium oxides

with various UO_2 contents (Fig. 13). Uranium oxide forms of this type occur as colonies of fine grains ranging in size from single micrometers to hundredths of a millimetre. Polygonal and oval grains, previously recognised as uraninite, contain significant thorium (1.25% Th; Banaś et al. 1978) and display colloform textures. Their small grain-size is such that only an immersion objective with magnification 100x allows the identification of their optical features (reflectance about 15%, lack of internal reflections). It is supposed that these grains comprise mainly pitchblende and the products of its decomposition with a distinctly lower reflectance (coffinite). An unidentified, secondary uranium mineral forms anisotropic rims on these. All three minerals are radioactive. The presence of uraninite at the margins of organic particles may indicate



Fig. 13. Colloform variety of UO_2 with the features typical of pitchblende: spherical accumulations, syneretic fractures. Alterations (coffinitization?) start from the grain center and radially spread out. Reflected light, 1 polar, immersion, magn. 300x

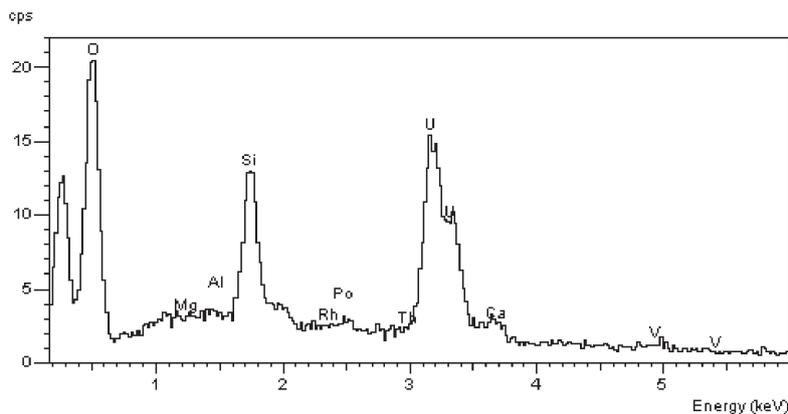


Fig. 14. EDS spectrum of coffinite I (white)

that thucholitization of the carbonaceous matter progressed inwards from the centre of grains.

Samples with coffinite do not contain vanadium minerals. Quartz grains are corroded by coffinite that occurs, as shown on Figure 1, in two zones with different silica contents — white zones of coffinite I and pale grey zones of coffinite II. EDS spectra reveal lower amounts of Si in relation to U in the case of coffinite I (Fig. 14) and higher in the case of coffinite II (Fig. 15). These SEM results confirm earlier microscope

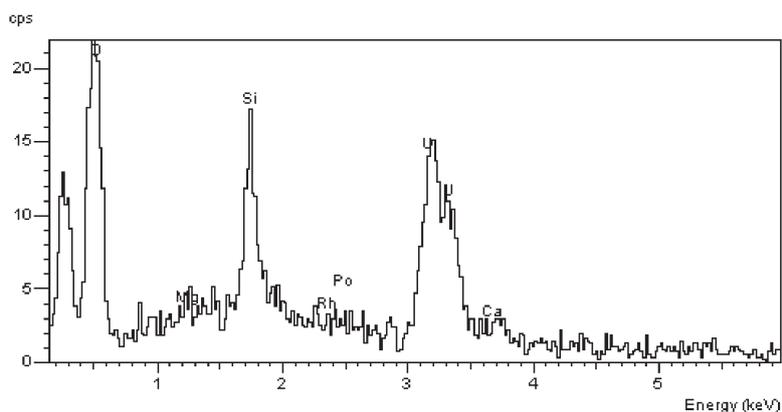


Fig. 15. EDS spectrum of coffinite II (light grey)

TABLE 1

Chemical composition of ore minerals in thucholite (microprobe determinations; wt.%)

	1	2	3	4	5	6	7	8	9	10
S	13.94	15.28	14.58	24.55	25.91	24.54	26.52	25.38	20.65	19.58
Fe	0.15	0.73	0.75	9.90	9.05	10.00	10.07	11.47	0.72	0.96
Ni	0.21	0.41	0.12	—	1.65	0.17	0.33	0.92	0.20	0.94
Co	0.10	0.20	0.24	—	0.10	0.12	0.28	0.87	0.34	0.06
Cu	31.74	25.19	33.20	63.57	61.43	63.53	60.87	63.24	75.25	80.81
Zn	1.27	0.15	0.36	1.64	0.76	0.14	0.59	1.09	0.33	0.93
Ag	52.20	46.69	51.34	0.34	1.05	2.11	0.41	0.52	1.50	0.42
Se	0.65	2.65	—	—	—	0.22	—	—	—	—
As	0.22	7.74	—	—	—	—	—	—	—	—
Te	0.82	1.26	0.60	—	0.05	0.50	0.28	0.52	—	0.98
Total	100	100	100	100	100	100	100	100	100	100

1-3 — stromeyerite; 4-8 — bornite; 9-10 — chalcocite

descriptions of coffinite as zoned, botryoidal, uranium silicate with variable silica contents (Salamon and Banaś 2002).

Thucholite and coffinite accumulations are associated with different ore minerals. Microprobe analyses show that ore minerals, mainly bornite, chalcocite and stromeyerite, occur in the cement of the thucholite-bearing sandstones where they typically corrode barren minerals and organic matter. In contrast, chalcopyrite occurs mainly in thucholite concretion-like bodies. It is worth noting that stromeyerite CuAgS is enriched in As, Se and Te, at the expense of Ag and Cu (Table 1). Reflected light and microprobe studies of samples with coffinite reveal intergrowths of the Ni and Co minerals: gersdorffite and niccolite. The Ni-Co mineralization has been found to be associated only with coffinite. In addition to niccolite and gersdorffite, the analyses indicated the presence of chalcopyrite and various other Ni and Co minerals characterized by different ratios of these two elements (Table 2).

TABLE 2

Chemical composition of Ni-Co ore minerals in paragenesis with coffinite
(microprobe determinations; wt. %)

	1	2	3	4	5	6	7	8	9
Ni	20.07	24.54	23.72	24.49	22.13	28.63	9.93	27.02	23.81
Co	11.34	8.46	7.58	6.99	6.05	0.28	18.73	9.24	10.02
Fe	0.49	0.23	0.38	0.33	0.52	0.03	0.18	0.19	0.31
As	63.38	52.84	58.89	57.41	67.26	70.58	61.89	49.93	52.31
S	5.69	14.38	10.19	10.78	5.08	1.10	8.51	13.63	13.55
Total	100	100	100	100	100	100	100	100	100

1 — rammelsbergite (Rm) > gersdorffite (Gf) > cobaltite (Cb) (sample N 13-18a); 2 — Rm > Gf > Cb (sample N13-13); 3 — Rm > Gf > Cb (sample N 13-14); 4 — Rm > Gf > Cb (sample N 13 p. 14); 5 — Rm > Gf > Cb (sample N13-14a); 6 — Rm (sample N 13-16); 7 — safflorite (Sl) > Cb > Gf (sample N 13 p. 9); 8 — Gf > Cb (sample N 13 p. 13); 9 — Gf > Cb (sample N 13-17)

SOME REMARKS ON THE U-V DEPOSITS OF THE COLORADO PLATEAU

The thucholite-coffinite mineralization of the Weissliegend sandstones in the Fore-Sudetic monocline may bear close comparison with the uranium-vanadium deposits of the Colorado Plateau, USA. The latter, Triassic-Jurassic in age, are situated in the central parts of Colorado, Utah, Arizona and New Mexico (Fischer 1958). The ore-bearing rocks are mainly continental sandstones accompanied by conglomerates, shales, coaly shales, black shales and limestones. Lignites, of historical economic importance, also occur.

The uranium-vanadium ores of the Colorado Plateau can be divided into unoxidized ores and oxidized ores (Weeks 1958). The former ores contain black vanadium oxides with an admixture of Cu, Pb, Fe and Zn sulphides. The latter are rich in secondary uranium-vanadium minerals, e.g., carnotite-tiujamunite, as major components and are conspicuous due to their bright, yellow-orange colours. Typical ores in the Jurassic sandstones have simple mineral compositions: roscoelite intergrown with corroded quartz fills pores in the sandstones and overgrows montroseite developed as isolated, radially arranged crystals (V ores). The beginning of oxidation is marked by replacement of montroseite by paramontroseite. Uranium ores with low vanadium contents in the sandstones and in conglomerates are mineralized mainly with uraninite and coffinite. Characteristic features are numerous organic fragments of, for instance, fossilized tree trunks or pieces of asphaltite replaced by uraninite. Uraninite is commonly associated with pyrite and marcasite, galena, Ni and Co sulphides, clausthalite (PbSe) and eucairite $\text{Cu}_2\text{SeAg}_2\text{Se}$.

The genesis, the form of the ore bodies, the development of mineralization and its alteration in the shallow deposits of Colorado must reflect the primary composition of ores, the intensity and depth of weathering (access of oxygen), the depth and type of overburden and oscillations of the water table. The presence of four-valent vanadium or six-valent uranium indicates intensive oxidation of these ores resulting in the formation of high-valence vanadium minerals (carnotite, tiujamunite). If vanadium occurs only in small amounts or not at all, uranium and copper form a range of other secondary silicates, carbonates, sulphates, phosphates and arsenates (Fischer 1958).

Various hypotheses for the origin of the Colorado Plateau deposits have been proposed. According to Fisher (1958), for example, late-epigenetic or hydrothermal processes were of critical importance. Marmont (1987, *vide* Craig and Vaughan 1994) hypothesised that metal- and sulphate ions, leached at low temperatures from granites, arkoses and volcanic ashes by underground waters, were re-precipitated in a reducing environment as ore minerals. The oxidizing solutions transported the soluble uranyl ion $[\text{UO}_2]^{6+}$ in carbonate complexes whereas the uranium ion $[\text{UO}_4]^{4+}$ was stabilized under reducing conditions as uraninite (UO_2). Reducing bacteria in the solutions resulted in the formation of copper sulphides. Vanadium, possibly transported as $[\text{VO}_4]^{3+}$, mineralized some parts of the deposits. The great distance of the Colorado deposits from any known magmatic source tells against granite related hydrothermal activity. Another hypothesis reveals that meteoric waters, mixing with low-temperature hydrothermal solutions, may have been involved.

CONCLUSIONS

The highest concentrations of vanadium in the sedimentary rocks of the Fore-Sudetic monocline (the Lubin area) were noted in shales rich in organic matter (0.5 wt.% V in organic extracts). Even with the considerable tonnage of ore involved — several million tons (Kucha et al. 1983), no minerals of vanadium had been previously reported and research had centred on vanadate porphyrins as the main carrier of vanadium.

Kucha et al. (1983), in a detailed overview of the vanadium distribution in the separate mining fields of the area, reported the highest vanadium contents in samples from the Rudna mine. They also determined that the sandstones underlying the shales do not contain more than 0.01wt.% V overall and their uppermost part with the carbonate cement even less. The present authors have identified vanadium minerals in the Polkowice mine just in these sandstones, poor in vanadium (according to Kucha et al. 1983). In such circumstances, the amount of vanadium would not be a single *sine qua non* condition for the precipitation of vanadium compounds. The fact that the vanadium minerals, roscoelite, montroseite and, tentatively, karelianite occur in the paragenesis with coffinite and U-bearing thucholite may suggest that it was not the amount but the form of vanadium, i.e., the presence of trivalent vanadium, that controlled the mineralization. It is probable that this ion could exist in the uppermost part of the Rotliegendes sandstone — rich in compounds of uranium-bearing hydrocarbons and carbonaceous matter. Their presence resulted in the precipitation of V and U minerals (roscoelite, montroseite, coffinite) associated with thucholites, chalcopyrite, Ni-Co sulphides and arsenides. This mineral composition is similar to that in the Colorado Plateau sandstones.

It is necessary to ask why carnotite ores are absent in the Fore-Sudetic monocline deposits. In the mineralization of deeper levels of the deposits, vanadium participated, as the trivalent element, in the crystallization of roscoelite and montroseite. Precipitation of carnotite requires pentavalent vanadium, typical of a shallower oxidation zone. Such a zone did not exist in the Fore-Sudetic deposits.

According to the Fischer (1958) classification, the mineralization in the sandstones of the Fore-Sudetic monocline belongs to the U-V group. It is concluded here that circulation of underground waters enriched in metal ions leached from carbonaceous Zechstein shales and Lower Rotliegendes sandstones, led to metal precipitation under reducing conditions in the uppermost part of these sandstones — the Weissliegendes sandstones. In this environment, the ratio V_2O_5/U_3O_8 was approximately 1:1.

The uranium-vanadium mineralization in the deposits of the Fore-Sudetic monocline currently has no economic importance. New data reported here extend our knowledge of this interesting occurrence and its similarity to the well-known deposits of the Colorado Plateau suggests genetic comparison. The significant vanadium occurrences in the Fore-Sudetic monocline merit further investigation.

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**WSPÓŁWYSTĘPOWANIE URANU, WANADU I MATERII ORGANICZNEJ
W PIASKOWCACH BIAŁEGO SPĄGOWCA ZE ZŁÓŻ MIEDZI
NA MONOKLINIE PRZEDSUDECKIEJ**

Streszczenie

Wanad jest w złożach lubińskich jednym z najliczniej występujących pierwiastków towarzyszących (około 11 milionów ton). Nie jest jednakże odzyskiwany na skalę przemysłową. Pierwiastek ten został dość dobrze rozpoznany m.in. przez Wojciechowską i Serkiesa (1969) oraz Kuchę i in. (1983). Nie zidentyfikowano natomiast minerałów wanadu. Autorzy niniejszej pracy stwierdzili w polu górniczym Polkowice obecność paragenezy mineralnej uran-wanad. Ustalono, że pewne fragmenty łupkowej rudy thucholitowo-coffinitowej zawierają zespół minerałów wanadowych: roscoelit ($KV_2[OH]_2AlSiO_{10}$), montroseit ($VO[OH]$) i prawdopodobnie karelianit (V_2O_3). Obecność tych kruszców potwierdzona została za pomocą analiz w mikroobszarze (SEM-EDS). Na bazie studium porównawczego ze złożami uranowo-wanadowymi z Plateau Colorado przeprowadzono dyskusję genetyczną nad pochodzeniem mineralizacji w Polkowicach. Zebrane materiały złożowe pozwoliły przy okazji ustalić nowe dane mineralogiczne thucholitów oraz coffinitu. Trzeba podkreślić, że dotychczasowe znalezisko mineralizacji U-V nie przedstawia wartości praktycznej, zwraca jednak uwagę przy dalszych geologicznych pracach poszukiwawczych na obecność tych cennych metali.