ASSOCIATION OF ORGANIC MATTER WITH URANIUM MINERALIZATION IN THE PERMIAN SANDSTONES OF THE WESTERN CARPATHIANS

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Abstract: Uranium mineralization is present in organic rich grey arkosic sandstones in the Permian strata of the Kozie Chrbty Mts. in the Western Carpathians. Dominant ore minerals are uraninite, pyrite, chalcopyrite, tennantite, galena and sphalerite. The organic matter was analysed by microphotometry, Rock-Eval pyrolysis and gas chromatography. The organic matter is of humic type at a stage of thermal maturity corresponding to medium volatile bituminous coal. Uraninite formed from solutions that evidently infiltrated the tissues of terrestrial plants at a very early diagenetic stage. Organic matter acted both as a sorbent and a reducing agent. The bands of uraninite grains in vitrinite are cut by younger quartz-dolomite-sulfide veinlets. The temperature to which the rocks were exposed during deepest burial is estimated at 100 – 150 °C.

Introduction

The Permian red-beds of the Alpine-Carpathian region are frequently accompanied by uranium mineralization. Deposits occur largely in sandstones, showing a genetic relation to the organic matter (Kornichuk & Burtik 1974; Mittempergher 1974; Petrascheck et al. 1977; Vincze & Somogyi 1984; Dolenec et al. 1985; Pagel 1990). A strong affinity of uranium for organic matter has been described from many deposits especially from the Colorado Plateau province, of the U.S.A. (Leventhal 1980; Finch & Davis 1985; Northrop & Goldhaber 1990). Several mechanisms of uranium accumulation controlled by organic substances have been proposed. They depend on the type of organic matter, which may be separated into the following groups:

- synsedimentary humic matter of plant debris,
- allochthonous amorphous humic matter,
- petroleum derived allochthonous bitumens of asphalts.

In the Permian sequence in the region of the Kozie Chrbty Mts., uranium mineralization occurs bound to organic matter. The aim of this paper is to characterize the type and grade of postsedimentary alteration of this organic matter, the relation of the alteration to uranium mineralization and to develop a thermal history of the host rocks.

Methods

Ore minerals were studied by optical microscope, scanning electron microscope (SEM), and analysed by electron microprobe analysis (EMA), energy dispersive X-ray spectroscopy (EDX), and X-ray diffraction (XRD).

Organic petrography was studied by reflected light and vitrinite reflectance in oil was measured at 546 *nm*. Hydrogen and oxygen characteristics were obtained by Rock-Eval pyrolysis (Espitalié et al. 1985). Saturated hydrocarbons of the $CHCl_3$ rock extract were analysed by capillary gas chromatography.

Geology and petrography

The main geologic units of the Kozie Chrbty Mts. region are the Alpine-folded Permian, Triassic and unconformly overlying Paleogene (Fig. 1). The Permian is represented by Malužiná Formation (Vozárová & Vozár 1988) maximum 2.2 *km* thick, which is a clastic sedimentary formation with sporadic chemogenic sedi-
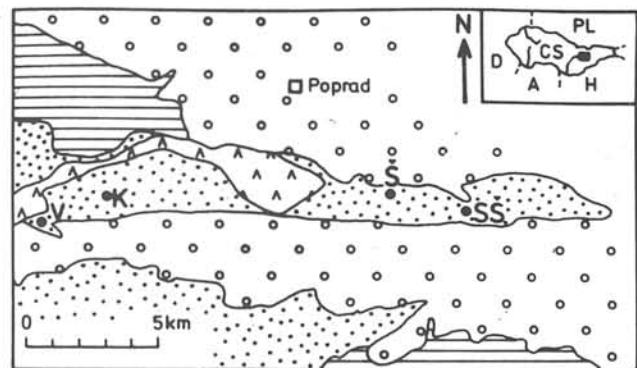
Paleogene: conglomerates, sandstones, claystones

Triassic: dolomites, limestones

Permian: basalts, andesites

Permian: sandstones, conglomerates, shales

● uranium deposits

Fig. 1. Location of the uranium deposits in the Kozie Chrbty Mts. (adapted after Bystrický et al. 1963).
K - Kravany, SŠ - Spišský Štiavnik, Š - Švábovce, V - Vikartovce.

ments. Sandstones are dominant sediments. Andesite-basalt volcanites and accompanying volcanoclastics are frequent. Lithology is characterized by three megacycles that fine upwards. The lower part of each megacycle consists of conglomerates and coarse-grained sandstones. Sandstones and shales dominate in the middle part. Chemogenic sediments such as dolomites, dolomitic limestones and scarce gypsum combine with shales and sandstones to form the upper part of the megacycles. Sedimentation took place in a continental environment with an arid climate, in fluvial, lacustrine and alluvial-lacustrine facies. The presence of carbonates and gypsum indicates their depostition in continental sebkhas or playas (Vozárová & Vozár 1988).

Mineralization is situated in pelitic-psammitic Kravany beds 175 – 300 m thick (Novotný & Badár 1971). These beds are included in the middle and upper part of the second megacycle by Vozárová and Vozár (1988). Towards the north-east and north, that is in paleocurrent direction, their thickness grows and their carbonate and gypsum content increases.

Ore-bearing rocks are represented by fine-grained arkosic sandstones and arkoses. They are often laminated, showing cross-bedding (Fig. 2). The alternation of layers with variable grain size is made visible by the presence of coaly laminae. Rock texture is psammitic, angular and porous. Sharply angular and partially rounded fragments and abundance of felspars suggest a short transport. Clastic grains are composed mostly of quartz, feldspars (especially plagioclases of albite-oligoclase composition), micas and, from the accessories, zircon and tourmaline. Fragment size varies from 0.05 to 0.25 mm, mostly from 0.1 to 0.2 mm. Granitoids were the main source of clastic material. Rock cement is formed usually by sericite. Quartz, carbonate and chlorite are less frequent. Sericite and chlorite flakes are often accompanied by coaly layers.

Mineralization is present in lenses of grey arkosic sandstones with abundant coal fragments (Fig. 3) in the 25 – 75 m thick ore-bearing horizon of the upper part of the Kravany beds. The ore bodies form irregular lenses of small thickness, usually about 1 m (Veselý & Badár 1984). Lenses of similar sandstones are sterile if surrounded with less permeable pelitic rocks that suggest a postsedimentation supply of ore elements (Novotný & Badár 1971). The Fe^{+3}/Fe^{+2} ratio is 1.3 in mineralized grey sediments, but rise to as high as 2.8 in non-mineralized red sediments.

Organic matter forms accumulations in the form of thin layers and bands in arkosic sandstones. Their thickness ranges from a fraction of a millimetre to a few centimetres. A few larger fragments of coalified tree branches have been found (Fig. 4).

The Permian is overlain by Lower and Middle Triassic sediments represented by sandstones, shales and especially Middle Triassic dolomites and limestones.

Basal transgressive beds of the Paleogene are dominated by breccias, conglomerates and subgraywackes (Marschalko et al. 1966). They contain lignite in the studied area. Basal beds are overlain by claystones passing into flysch with sandstones and siltstones.

The structure of the region is affected by lithofacies development, the Alpine Nappe and younger fault tectonics. Significant morphological features are a result of uplifts and erosion during the Pliocene and Pleistocene (Lukniš 1959).

Fig. 2.   Cross-bedded arkosic sandstone with black interlayers of coaly organic matter (Švábovce).
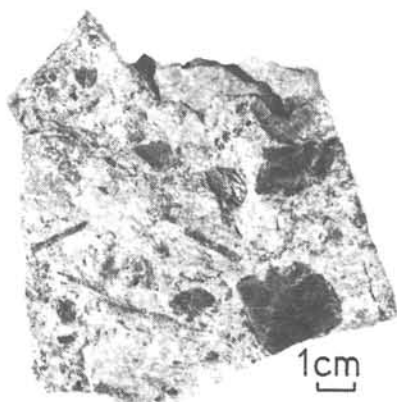

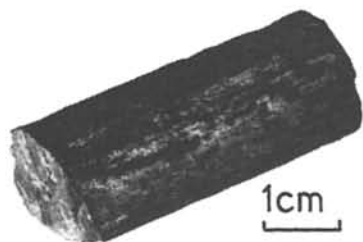**Fig. 3.**   Coaly fragments (black) in arkosic sandstone (Vikartovce).

Fig. 4.   Fragment of coalified and mineralized wooden branch (Kravany).

Ore mineralogy and geochemistry

Uraninite is the main ore mineral. The highest concentrations of ore minerals have been found in the central parts of lenses, particularly in 1 – 3 cm thick coal layers (Rojkovič 1974). Uraninite occurs partly finely dispersed in organic matter and partly in the cement of arkosic sandstones. It forms colloform concentric and botryoidal forms, veinlets and aggregates (Fig. 5). In rich samples it forms a substanctial part of the cement. Cell edges of the analyzed uraninites range from 0.5442 to 0.5454 nm.

Pyrite is a frequent accompanying mineral. It occurs in the following morphological forms: spheroids (size of 5 – 10 μm), framboids (size of 5 – 30 μm), subhedral and euhedral grains and aggregates (Fig. 6). It occurs in the coal, cement of sandstones and in quartz-carbonate veinlets.

Chalcopyrite and tennantite are accompanying minerals of uraninite and pyrite in the western part of the Kozie Chrbty Mts. (localities Vikartovce and Kravany; Fig. 1). Sphalerite and espe-

cially galena occur, in contrast to the former, in the eastern part of the region (localities Švábovce and Spišský Štiavnik). These sulfides, as well as pyrite occur in coalified plant tissues often preserving their original structure (Figs. 7, 8). They occur also in the cement of sandstone and quartz-carbonate veinlets. Arsenopyrite, bornite, marcasite and Pb-Mo-S mineral are quite rare. In the supergene zone, autunite, malachite, azurite and goethite were identified.

Quartz and carbonates (dolomite and calcite) form thin veinlets with pyrite, chalcopyrite, galena and sphalerite. Fluid inclusions in the carbonates from Vikartovce and Kravany showed homogenization temperatures ranging from 86 °C to 139 °C with an average value of 103 °C (Fig. 9). Mineralization was dated by $^{206}Pb/^{238}U$ and $^{207}Pb/^{235}U$ method at 263 and 274 Ma (Lepka, pers. comm.).

Elevated uranium values in the arkosic sandstone (average 24 ppm), outside ore lenses, suggests uranium enrichment in the whole ore-bearing horizon. The highest contents of uranium (over 1 wt. %) and accompanying metals were found in the mineralized lenses, associated with coal.

The average total organic carbon (TOC) content in non-mineralized arkosic sandstone is 0.15 wt. %, whereas in mineralized lenses of coaly sandstones it reaches 2.8 wt. % and in coal layers even 30 – 40 wt. %. TOC displays a distinct positive correlation (Fig. 10) with uranium (r = +0.66; Rojkovič 1975). The dominant elements of ore mineralization are U, Cu and Pb with average values of U 0.63, Cu 0.12 and Pb 0.13 wt. % in the

mineralized rocks. The accompanying elements of ore mineralization are Zn, As, Ni, Co, Sb, Mo, Th and Y (Rojkovič 1975).

Type and maturity of the organic matter

Microscopy and organic petrography

The organic matter has a microscopically distinguishable structure of more or less gelified xylitic tissues of higher continental plants forming broken, bent and sometimes even refolded blocks. Cell voids and fissures between the blocks are filled with carbonates, quartz and partly also ore minerals (Figs. 7, 8). Vessels in a preserved branch are filled with carbonates in the centre and with quartz and sulfides in the outer rim.

From the coal-petrographic point of view, the organic matter is composed mostly of inertinite and vitrinite macerals. Inertinite occurs in the form of clearly visible tissues with oval cell voids (fusinite-semifusinite) forming bands and aggregates (Fig. 5) or dispersed as detritus of cell walls (inertodetrinite). Variable reflectance of semifusinite and makrinite (massive structureless inertinite) indicate an uneven fusinitization (partial oxidation) of the original tissue. Desiccation (contraction) fissures are bordered by the secondarily oxidized matter with a lower reflectance. Similar phenomena were described by e.g. Teichmüller (1975), Robert (1988), Speczik & Püttmann (1987).
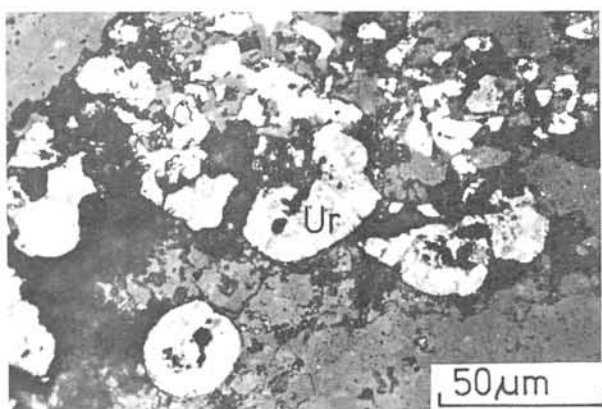

Fig. 5. Colloform concentric uraninite (ur) in the cement of arkosic sandstone (Kravany, reflected light, 1 nicol).
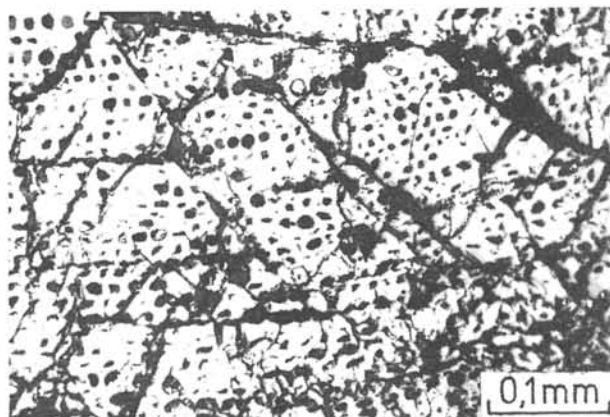

Fig. 7. Chalcopyrite and tennantite (white) in the voids and fissures of the fusinitic plant tissue (Vikartovce, reflected light, 1 nicol).
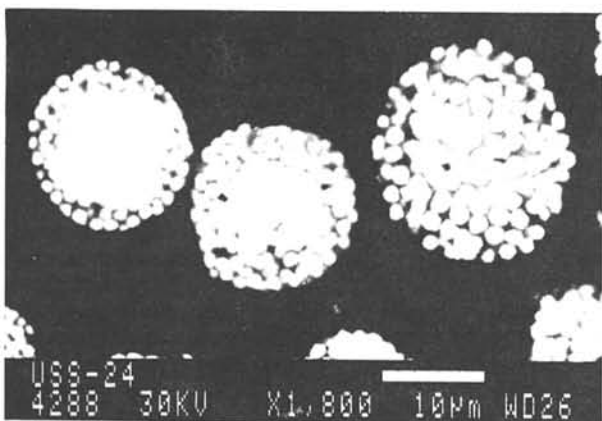

Fig. 6. Framboidal pyrite (white) in the cement of the arkosic sandstone (Spišský Štiavnik, SEM).
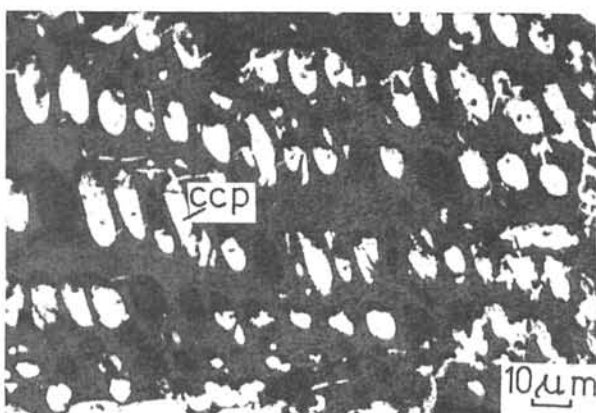

Fig. 8. Chalcopyrite (ccp) in the voids of the fusinitic plant tissue (Vikartovce, reflected light, oil, 1 nicol).

Reflectance of inertinite (R_o) varies from 2.2 to 4.5 % (Fig. 11) and it has a very slight bireflectance (R_{max}-R_{min}) in polarized light. Abundant occurrences of inertinite prove partly a partial oxidation of the organic matter during the peat stage (oxyfusinite) and partly occasional forest fires in the sedimentary and source areas (pyrofusinite).

Vitrinite is represented by structured telinite and homogéneous collinite. Its abundance is similar to inertinite and in the sections of preserved branches it is concentrated mainly in the outer third. Cell voids in telinite are visible only in oil immersion and are 5-10 times smaller than in inertinite. They are compressed or filled with gelified matter or ore minerals. Mean reflectance (R_o) of non-irradiated vitrinite varies from 0.9 to 1.2 % (Fig. 11) and corresponds to a medium volatile bituminous coal rank. Absence of visible macrospores (liptinite group) in the samples also suggests bituminous coal rank, as in this stage liptinite can no longer be distinguished from vitrinite (Teichmüller 1975).

Vitrinite reflectance of Paleogene rocks in the section is 0.45 % and is discussed later in respect to thermal and burial history.
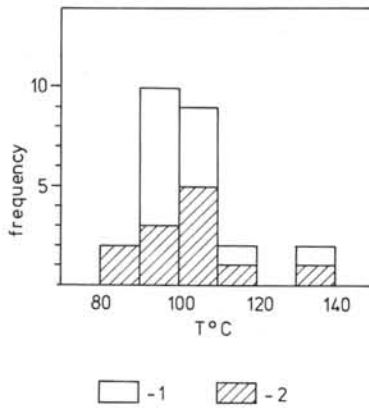

Fig. 9.   Homogenization temperatures of fluid inclusions in carbonates (1 - Kravany, 2- Vikartovce).
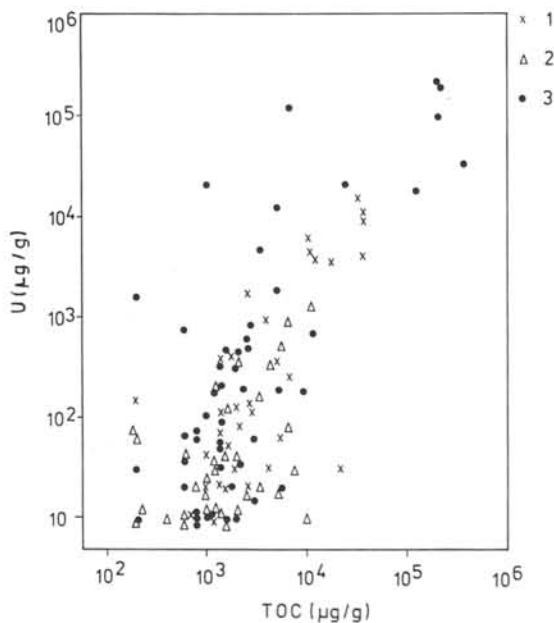

Fig. 10.   Correlation of uranium (U) and total organic carbon (TOC) in rock and coal samples from the studied area: 1 - Švábovce, 2 - Spišský Štiavnik, 3 - Vikartovce.

Rock-Eval pyrolysis

The results of the Rock-Eval pyrolysis show that the organic matter in the studied rocks is poor in hydrogen and oxygen. The hydrogen index (IH) does not exceed 60 mg HC/g C_{org} and the oxygen index (IO) is lower than 30 mg CO_2/g C_{org}. Leventhal et al. (1986) observed a close relationship between the organic CO_2 and the amount of uranium in organic matter from the Grants uranium region. They suppose that the IO increase is a result of organic matter oxidation during uranium reduction. Such correlation was not found in our samples, probably due to CO_2 escape. Maximum Rock-Eval pyrolysis temperatures (T_{max}) vary from 455 to 475 °C. These data are plotted in IH-IO and IH-T_{max} diagrams (Fig. 12), where the maturation paths of main kerogen types (I-sapropel, II-planktonic/liptinite and III-humic) during diagenesis and catagenesis are shown after Espitalié et al. (1985). T_{max} values (455 – 475 °C) of the studied vitrinite-inertinite coal correspond, according to the correlation T_{max}-R_o. (Espitalié et al. 1985; Franců et al. 1989), to the vitrinite reflectance of 0.9-1,5 %, supporting our measurements of R_o in the same samples. Maturity of the uranium - poor organic matter corresponds to the medium volatile bituminous coal rank. The thermal maturity of the overlying Paleogene (T_{max} = 428 – 438 °C, R_0=0.45 %) is distinctly lower than that of the Permian (Fig. 12).

Gas chromatography of saturated hydrocarbons

Saturated hydrocarbons were studied in extracts of samples with low U-content and a sufficient amount of hydro-
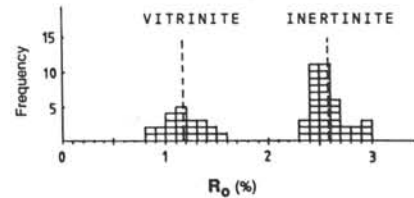

Fig. 11.   Bimodal histogram of reflectance in oil (R_o) of vitrinite (left) and inertinite together with strongly irradiated vitrinite (right) in a characteristic coal sample (Vikartovce).
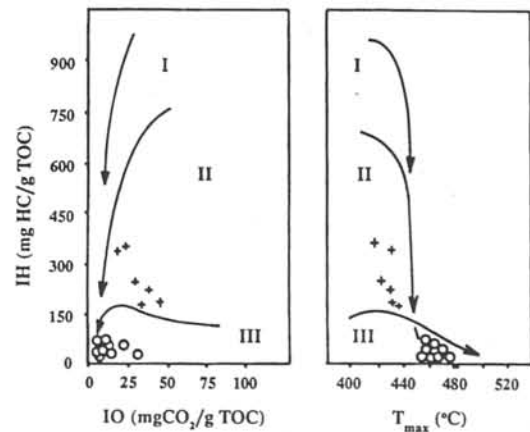

Fig. 12.   Rock-Eval pyrolytic characteristics of the organic matter (samples without strong radioactive alteration).
IH -hydrogen index, IO - oxygen index, T_{max} - maximum temperature of pyrolytic effect indicating degree of diagenesis or degradation of kerogen; I, II and III - kerogen types; crosses - overlying Paleogene, circles - Permian samples.

carbons detected by pyrolysis. The n-alkane distributions show two hydrocarbon populations (Fig. 13). Dominant group of alkanes around C_{29-31} and very slight predominance of odd over even homologues due to maturity (CPI = 1.0 – 1.2) are characteristic features of the organic matter inherited from higher

and Mesozoic rocks lying between the Permian ore-bearing horizon and the Paleogene lignite. The lignite stage of coalification gives evidence of burial of the basal Paleogene to several hundred meters in the younger Paleogene and subsequent uplift and erosion of the whole complex from the Pliocene to Quaternary.
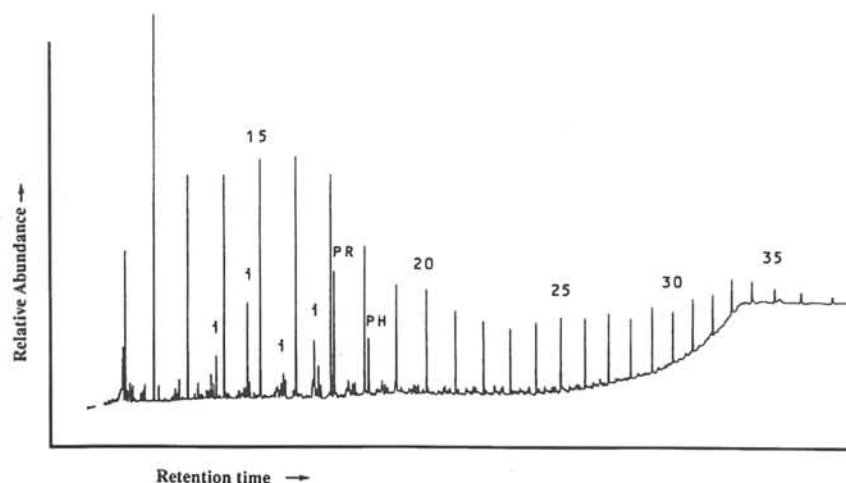

Fig. 13. Gas chromatographic trace of saturated hydrocarbons in chloroform extract of the Permian coaly sandstone (Spišský Štiavnik). The sample is not strongly altered by radiation.

plants(Albrecht et al. 1976; Tissot & Welte 1978).

The n-alkane group around C_{17} is interpreted as a product of catagenesis. An algal origin seems to be of low probability because of the low hydrogen content in organic matter detected by pyrolysis (Fig. 12) and terrestrial depositional environment. CPI values are close to 1 and n-alkanes predominate over isoprenoid hydrocarbons (pristane/n-C_{17} = 0.50 – 0.88; phytane/n-C_{18} = 0.56 – 0.65). Although these results should be treated with caution because of the low hydrocarbon concentration, they are in agreement with vitrinite reflectance of approximately 1.0 % and suggest that the organic matter attained the mature stage and generated hydrocarbons including the group around n-C_{17}.

Maturity and thermal history

The observed catagenetic stage equivalent to that of the Permian coal from non-irradiated layers (R_o=1 %, T_{max}=460 °C) is, under the conditions of the Tertiary basins with rapid accumulation and burial of sediments, reached at the temperature of 140 °C (130 – 150 °C; Tissot & Welte 1978; Franců et al. 1989) corresponding to depth of at least 1.6 – 2.5 km if the geothermal gradient was as high as 50 – 80 °C/km. In the older formations where the rocks had been exposed to a deeper burial for tens of millions of years, this stage may be reached even at lower temperatures (115 °C) and depths. Pelitic sediments of the Kravany beds were exposed the temperature of burial metamorphism up to 150 °C according to illite crystallinity measurements (Šucha 1989).

In the studied case, in addition to a lithospheric heat, radioactivity may have increased the maturity of the organic matter even the data are related to non-mineralized samples. Taking into consideration the complex character of this alteration, it may be concluded that the studied coal in sandstones reached temperatures between 100 and 150 °C either at the end of the Permian or during the Mesozoic.

The difference in maturity between the Permian bituminous coal and Paleogene lignite stages may be explained by different maximum burial depth and temperature due to the total thickness (over 1.5 km according to Novotný & Badár 1971) of the Permian

## Association of mineralization and organic matter

The major part of mineralization is concentrated in local lenticular accumulations of the organic matter in arkosic sandstones. Contact radiographs of the polished sections of rocks document the close relation of the radioactive minerals to the coal fragments and their internal structure (Fig. 14c). The association of the organic matter with uranium differs from that with quartz-carbonate-sulfide mineralization.

Szalay (1964), Breger (1974), Haji-Vasiloiu (1980), Obr & Burdová (1982), Turner-Peterson (1985), Leventhal et al. (1986) described the chemical sorption and reduction of uranyl ion at the oxygenated functional groups of huminite and vitrinite. In the studied samples uraninite is associated with vitrinite where its distribution in polished surface follows the porosity of the original material (Fig. 14c, d). Uraninite impregnation in the porous vitrinite (telinite) takes the form of elongated grains of 5 – 10 μm size. According to EDX analysis, uraninite is accompanied by pyrite, illite and apatite of maximum 2 μm. Mineral impregnation is much poorer in the dense vitrinite (collinite) bands forming grains of 0.5 μm size (Fig. 14e - black area). The distribution of uraninite also follows the original tissues in folded coal fragments (Fig. 14f). It gives evidence of uranium supply in the solutions and its sorption and reduction by the humic detritus in an early stage of postsedimentary evolution prior to the intensive gelification of vitrinite typical of the brown coal stage (Teichmüller 1975). Uraninite was not found in voids of chemically inactive inertinite despite its larger specific surface than that of vitrinite.

Quartz-carbonate-sulfide mineralization, on the other hand, often fills fusinite (inertinite) cell voids, as well as secondary fissures cutting across the original plant structure or the broken coal fragments (Figs. 7, 8, 14f). It is evidently younger than the sorption of uranium by the organic matter.

Radiation damage of the organic matter

Reaction rims or "halos" in the organic matter frequently occur at the contact with the radioactive minerals (uraninite, coffinite,
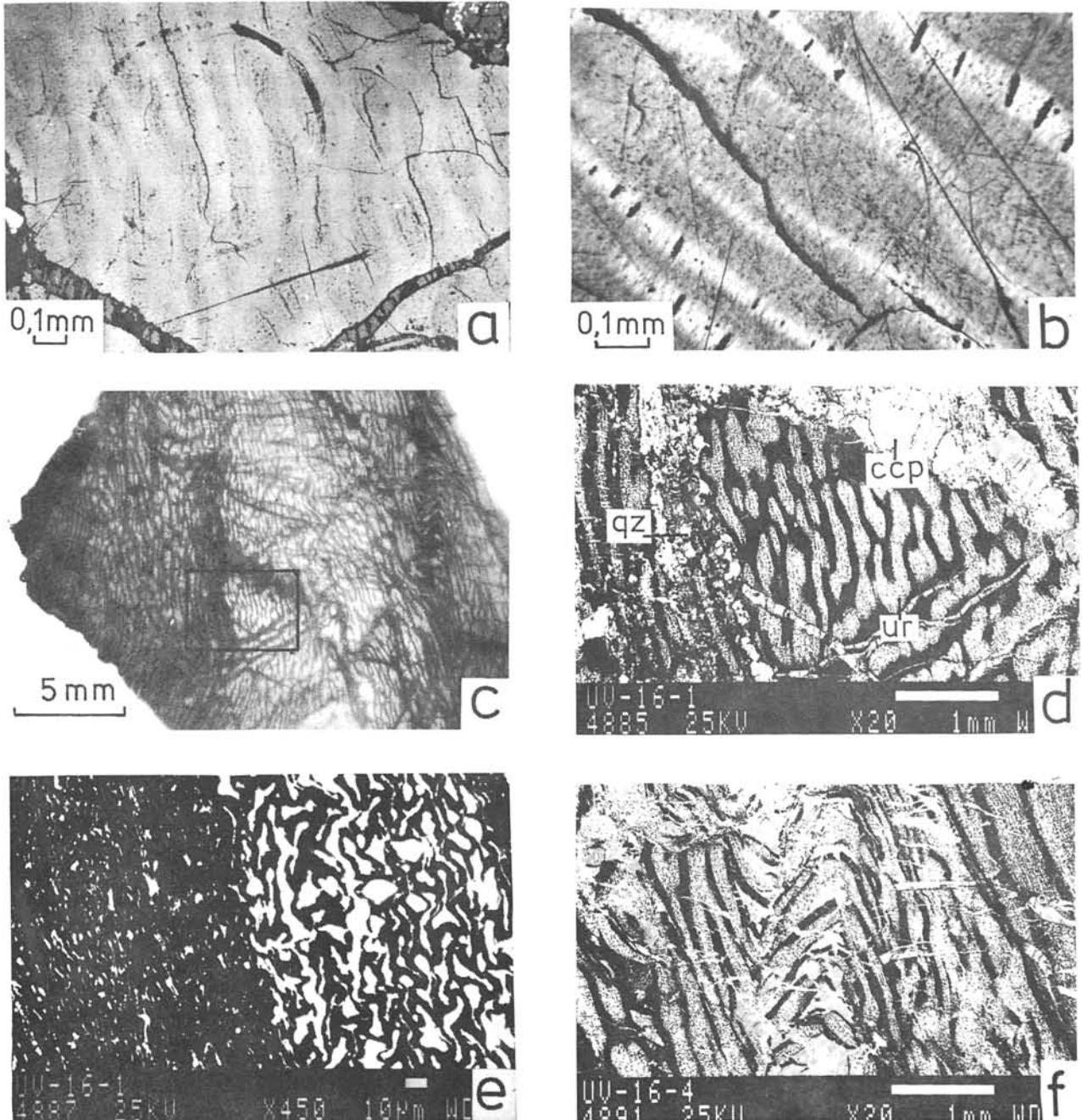
Fig. 14.   Coal macerals and uraninite distribution in a sample from Vikartovce:
a - collinite (homogeneous vitrinite) forms lighter bands along darker telinite impregnated by mineral grains (reflected light, partially crossed nicols);
b - contraction fissures in collinite (reflected polarized light); c - autoradiograph of mineralized coal fragment with visible woody structure; d - SEM photomicrograph of the same polished surface area as in Fig. 14a and 14c frame (back-scattered image), telinite bodies are bright due to concentration of uraninite (ur), younger veinlets are filled with quartz (qz) and chalcopyrite (ccp); e - detail of Fig. 14d with uraninite (bright) concentrated in coarser grains in telinite (right) and finely dispersed in dense collinite (left); f - SEM photomicrograph (back-scattered image) of folded telinite bands with uraninite which are cut by younger quartz-carbonate-sulfide veinlets.

zircon, etc.). Such rims were observed mostly in the homogeneous amorphous organic matter (kerogen) of resin origin (Obr & Burdová 1982) or in the petrified bitumens (Landais & Connan 1980; Curiale et al. 1983; Leventhal et al. 1987; Robert 1988). They are less frequently observed in humic coals. A visible spatial bond between the radiation damage and its source was described only in gelified vitrinite (Breger 1974; Müller et al. 1983). These

phenomena are absent in fusinite due to its primary low reactivity and overall high reflectance.

Under the influence of α-particles, radiation damage is manifested as a local coalification, mainly by increased reflectance. In more detailed studies some authors (e.g. Leventhal et al. 1987) describe a dark rim closely adjacent to a radioactive mineral in a bitumen. This reflectance decrease is attributed to oxidation of

the organic matter and a partial decomposition of its molecular structure by intensive α-radiation.

In the studied samples, such dark rims of $1 - 10 \mu m$ thickness passisng to the external lighter rims are observed in some cases around the uraninite grains. More porous telinite densely impregnated by uraninite has roughly by $0.2 - 0.5 \%$ lower reflectance than more homogeneous adjacent bands of collinite with higher reflectance and bireflectance, and contraction fissures (Fig. 14b). Beside a variable reflectance caused by uneven fusinitization in the sedimentary stage, grey bands along the fissures and rims of the organic fragments were observed. Their origin is considered as secondary due to oxidation of organic matter during uranium reduction as described earlier and experimentally verified by Leventhal et al. (1986), Speczik &Püttmann (1987), and Meunier et al. (1990).

Conclusions

From microscopic, Rock-Eval pyrolysis and gas-chromatographic study, uranium mineralization of the Kozie Chrbty Mts. is shown to be bound ro organic matter of the humic type. The source material was represented by remnants of terrestrial higher plants which did not undergo a long transport. This is supported by the preservation of fragments of branches. During sedimentation and shortly afterwards they were exposed to a restricted oxidation. Organic material was not intensively microbially reworked.

Clastics of granitoids are the presumed uranium source. Uranium in solution was adsorbed and reduced from the filtering ground waters by the organic matter in peat or lignite stages, i.e. before an intensive gelification of the humic matter. This conclusion is supported by the distribution of uraninite in the coal matter, where uraninite outlines the original porosity of the plant tissues. Primary adsorbtion and reduction of uranium took place probably near the surface, where the hydrodynamic conditions, permeability and bedding of arkosic sandstones and local accumulations of the organic matter were the controlling factors.

Uranium was probably first bound in an organo-uranium compound (based on Meunier's et al. 1990) and reduced by the vitrinite-type organic matter. Formation of uraninite is supposed to occur later during diagenesis and catagenesis.

After a partial lithification, the coal was fractured, sometimes also folded by tectonic strain during the Early Alpine orogeny. The fissures were secondarily filled with quartz-carbonate-sulfide mineralization often cutting older uranium mineralization. Low-temperature hydrothermal solutions caused a partial uranium remobilization and formation of younger uraninite-sulfide mineralization in sandstone cement.

The present stage or catagenetic alteration of the studied Permian coal not affected by radioactivity corresponds to medium volatile bituminous coal. Most of the described coals associated with uranium accumulations are in the lignite, i.e. lower stage (e.g. Breger 1974; Landais & Connan 1980; Obr & Burdová 1982; Müller et al. 1983; Leventhal et al. 1986; Ilger et al. 1987). Our results show that a major uranium removal from its organic sorbent need not take place during further coalification.

The observed catagenic stage of the Permian coal would require burial to a depth of at least $2 km$ and exposure to temperature between $100 - 150 °C$, most probably at the end of the Paleozoic or early Mezosoic era. New uplift and asssociated erosion in the Pliocene and the Pleistocene brought up the sediments of the Permian, Mesozoic and Paleogene to shallower depths.

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