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Mineralogy of the Uranium Deposit of Žirovski Vrh, Slovenia/Yugoslavia
A Reflected Light, SEM, Electron Microprobe and PIXE Investigation

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Abstract

The uranium deposit of Žirovski Vrh is located in the southwestern part of Yugoslavia and is hosted by Permian-Gredenian sandstones. The deposit is interpreted as of epithermal origin and probably formed by transport of uranium in solution and deposition in the sandstone. Xylene structures of primitive plants of younger Paleozoic sediments were encountered.

Recently, microprobe investigations on uranium minerals were performed by Simova et al. (1983, 1984). Based on the UO_2/SiO_2 and $(U + Ca)/Si$ ratios, these authors suggest the presence of five uranium minerals, four of them are thought to be new.

Our studies indicate that the uranium mineralization is not restricted to the uraniferous veins in the sandstones. A cosiderable amount of uranium minerals was discovered in carbonaceous matter and fossil wood. Major components are coffinite and pitchblende. Other REE-rich minerals, presumably carbonates, were also encountered. In contrast to former reports by Simova et al. (1983), our results indicate that coffinite displays a complex chemistry with high concentrations in REE (average 11.4 wt.% total rare earth). Concentrations of REE, Hf, and Pb vary considerably. The totals are close to 100% indicating the reported shortage in the totals to be due to incomplete analysis. Pitchblende in the carbonaceous material contains higher concentrations in REE than in the coexisting coffinite. Due to the high CaO- and P_2O_5-contents, we interpret this result as suggesting the presence of REE-bearing phosphates and carbonates intimately intergrown with pitchblende.

The uranium mineralization is very probably of sedimentary origin or due to low temperature hydrothermal activity. Mobilization of UO_2, PbO, REE_2O_3 took place in a younger episode, thus redistributing these elements among various uranium minerals. The total PbO-content in pitchblende is too high to be attributed to in-situ decay. The measured Pb is probably a mixture of mobilized radiogenic lead, primordial lead, and pristine radiogenic lead. Hence, the total U/Pb ratios cannot be used to determine the age of the mineralization. Both Pb and U were involved in the mobilization and contamination of coffinite and pitchblende and the amount of ^{204}Pb and the $^{206}Pb/^{207}Pb$ ratio in individual minerals are unknown so far. The genetic evolution of the deposit and the temporal determination of the metamorphic episodes can only be obtained by mass spectrometric Pb/U systematics.

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1. Introduction and geologic setting

The uranium deposit of Žirovski Vrh is located west of Ljubljana and belongs to the Škofija Loka-Idrija metallogenic region (fig. 1). The deposit attracted attention after the discovery of the radioactive anomaly and of secondary uranium minerals (Berce et al. 1960; Ivanović 1961; Ramovš 1965; Marković 1961). The uranium mineralization lies in the sedimentary red gradenian layers in a tick Permian succession (Radusinović 1967). According to Ramovš (1965) and Omaljev (1969), the sediments are Permian and Permocarboniferous in age. The sedimentation may have lasted for 20 million years (Omaljev 1969). The clastic sediments in which the uranium mineralization is emplaced are characterized by their red colour due to the presence of hematite. Clay minerals and lime are the major adhesive of the clastic grains.

The geology of the area is typical of the metallogenic province of Northern Italy and West Yugoslavia (Janković 1962, 1963; Janković et al. 1964, 1970, 1971). The intensive volcanic activity in the upper Carboniferous produced the quartz porphyry and is the main source of the clastic sediments (Mittempergher 1972). Žirovski Vrh lies in the youngest upper layers of the sedimentary succession (Omaljev 1969).

The major constituents of the sediments are quartz, fragments of igneous rocks with rare mica and feldspars. Plant residues and coal are ubiquitous.

According to Radusinović (1967), the primary zone of the deposit consists of a complex assemblage of uranium minerals and Pb, Fe, and Cu-sulfides. These assemblages include pitchblende, pyrite, galena, sphalerite, chalcopyrite, tetrahedrite, chalcocite, arsenopyrite, pyrrhotite, marcasite, bornite, and covellite. In addition, he reported the presence of autonite, torbernite, metatorbernite, and other secondary minerals.

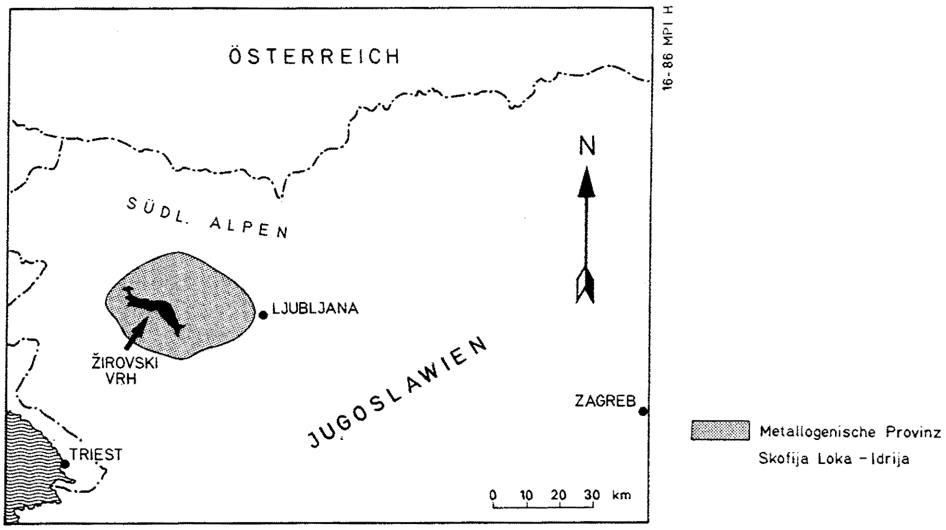

Fig. 1. A geographic map at the three countries corner Yugoslavia-Austria-Italy, depicting the location of the uranium deposit of Žirovski Vrh within the metallogenic province of Skofija-Loka-Idrija.

Kurat et al. (1977) and Simova et al. (1983) conducted mineralogical and microprobe studies on the uranium minerals of Žirovski Vrh. In addition to pitchblende, they reported the presence of "water-bearing coffinite" $USiO_4$ nH_2O and other unidentified U-silicates. The uranium minerals in Žirovski Vrh were also subject of a detailed TEM investigation (Hanzlik 1981). The presence of coffinite was confirmed and, in addition, ekanite, rutherfordite, and uranophane were also found. The latter three minerals suggest the formation of the deposit under oxidizing conditions from underground water.

2. Analytical methods

High quality polished sections were prepared to conduct a combination of studies including reflected light microscopy, scanning electron microscopy (SEM), electron probe microanalysis, and proton probe microanalysis (PIXE). One major intention for the selection of these various methods is to study the fine textures of the mineral assemblages at high resolution in comparison to regular high magnification reflected light microscopy and to secure the quality of the analysis by covering the trace element household by PIXE. This is specifically important to check the amount of H_2O or OH present in the uranium minerals. In many previously published microprobe analyses of uranium minerals, specifically coffinite, the amount of water or OH detected varies considerably. However, the real concentration is unknown since little attempt was made to quantitatively analyze numerous minor elements like the lanthanide group, Zr, Hf, Pb, V, Nb, etc. One major analytical problem is the accurate measurement of lanthanide element concentrations, especially at low concentration levels ($< 0{,}5$ wt.% REE_2O_3). This problem arises from two sources: 1. There are some 19 $L\beta$ and $L\gamma$ X-ray overlaps of elements of lower atomic number over the $L\alpha$'s of elements of higher atomic number. The deconvolution requires accurate experimental determination of the overlapping factors. 2. Since the $L\alpha$, $L\beta$ and $L\gamma$ of REE elements are numerous, it is difficult to measure the background with the required accuracy.

The SEM investigations were conducted using a Cambridge 180 S scanning electron microscope with an attached Si(Li) detector (147 eV resolution). The quantitative analyses of the mineral phases were carried out with a fully automated computer-controlled SEMQ electron microprobe using wavelength dispersive spectrometers. A special analytical procedure and a peak deconvolution programme were used to correct for X-ray overlaps of $L\beta$ and $L\gamma$ lines of lanthanide elements on the $L\alpha$ lines of lanthanides of higher atomic number; this procedure corrected for 19 overlaps in the lanthanide-group elements (El Goresy et al. 1984).

PIXE investigations were performed at the Heidelberg Proton Microprobe attached to the linear Tandem Van de Graaff accelerator of the Max-Planck-Institut für Kernphysik (Bosch et al. 1978). Analyses were carried out on the same grains analyzed previously by SEM and electron microprobe using an excitation energy of 3 MeV protons. The data were collected with a Si(Li) energy dispersive detector. The beam diameter was set at spot size of 1 mm^2, since it was planned to determine the bulk abundance of minor and trace elements in carbonaceous materials.

3. Petrography, assemblages, and mineral chemistry

The uranium mineralization is massively but not exclusively (as believed by Omaljev 1982) concentrated in the grey clastic sediments. Our investigation indicates two distinct types of assemblages in different host materials:

(1) Uranium mineralization in the interstices between the clastic quartz sand grains. All previous reports were confined to this type only (Radusinović 1967; Kurat et al. 1976; Omaljev 1982; Simova et al. 1983).

(2) The investigations of carbonaceous material and graphite-like objects (called anthracite by Omaljev 1982) from Žirovski Vrh revealed a considerable amount of uranium- and REE-rich minerals discovered therein for the first time. Both host materials occur in the same samples and the carbonaceous material or graphite-like objects occur as inclusions in the sandstones. Figure 2 is a low magnification photograph of both lithologies in a part of the sandstone, which is highly enriched in uranium minerals. In each sample studied, the carbonaceous material is of clastic nature indicating that it is not contemporaneous with the uranium mineralization of the first type (fig. 2).

3.1 Uranium mineralization in sandstone

In this paragenetic type, the uranium minerals occur as impregnations between the detrital ragged quartz grains (fig. 3, 4, 5). The impregnations contain two types of materials distinctly different in form and reflectance: (a) Compact higher reflecting

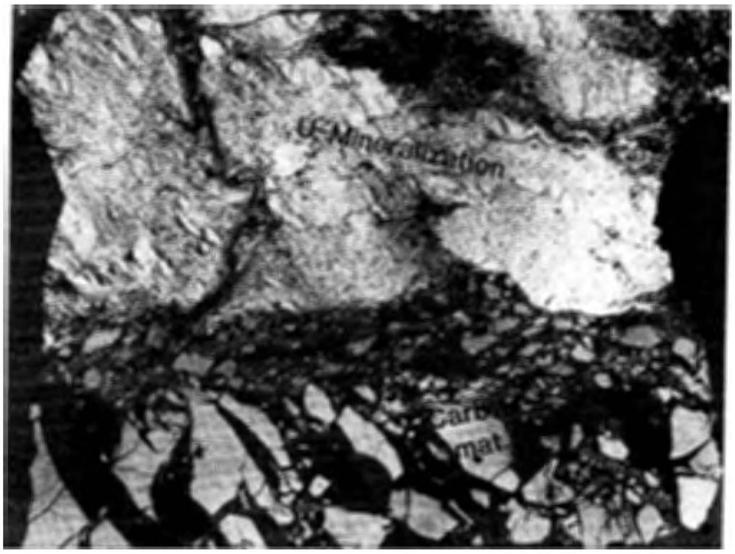

Fig. 2. A low magnification photograph of a polished section showing the uranium impregnations mineralization side to side with the clastic carbonaceous material. Width of photograph: 2.5 cm.

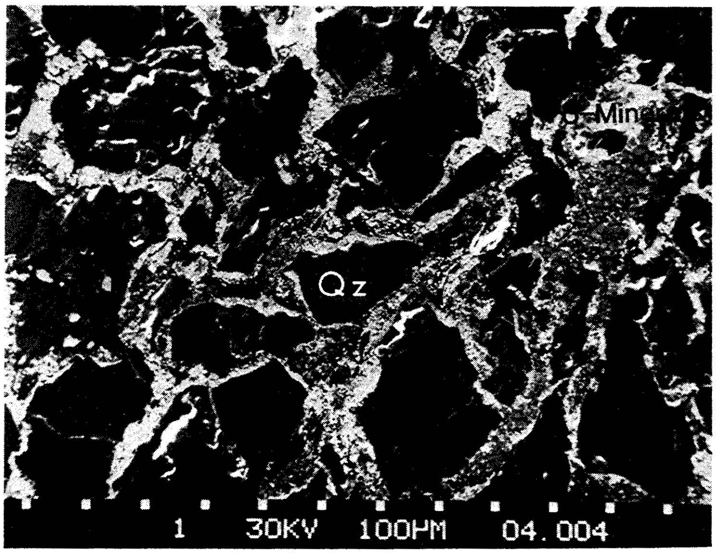

Fig. 3. SEM secondary electron imaging photograph of the impregnation paragenetic type. The uranium mineralization occurs in the interstices of the ragged quartz (Qz) grains.

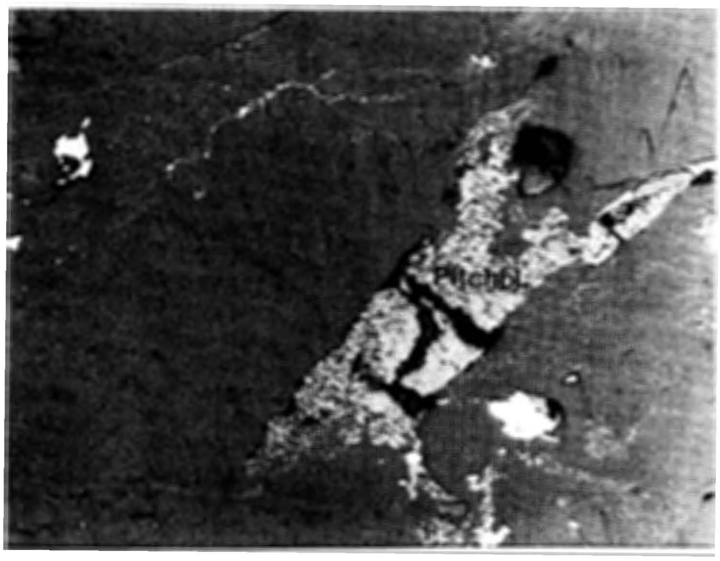

Fig. 4. Details from Fig. 3. Bright compact masses are pitchblende. Darker spherulitic objects consist mainly of coffinite.

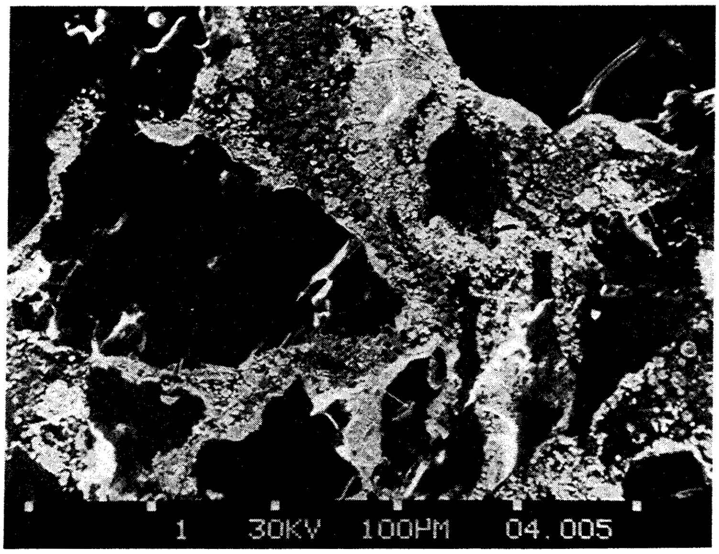

Fig. 5. Reflected light photomicrograph of massive pitchblende in an impregnation vein. Length of photograph 200 microns.

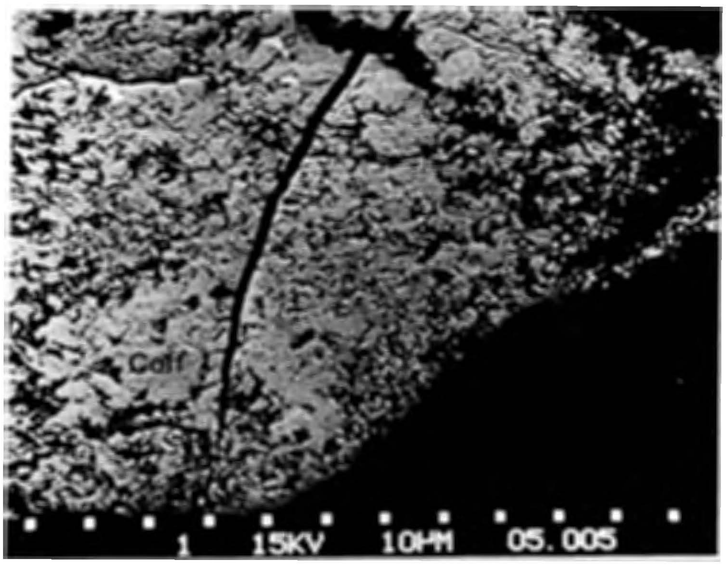

Fig. 6. SEM photograph of a part of vein impregnations consisting mainly of partly compacted coffinite.

material consisting majorily of pitchblende veins usually bordering the quartz grains (fig. 4 and 5), and (b) loosely compacted low reflecting spherules with coffinite as a major component filling the regions between the pitchblende veins (fig. 4 and 5). Secondary uranium minerals also occur within this region. Some of the impregnations may consist exclusively of either pitchblende or coffinite (fig. 6). Both minerals are fine-grained. (< 20 microns). This feature creates difficulties in the microscopic identifications of the individual phases. The problem, however, can be solved by applying BSE or SE imaging techniques under SEM. Using SEM will not only expand the imaging capabilities towards higher magnifications, but also allows fast and accurate identifications based on brightness and qualitative chemical analyses with an attached EDAX system. Both minerals are anhedral. In particular, coffinite rarely displays the typical tetragonal habit. To secure accurate analysis of both minerals, and due to their fine-grained nature, electron microprobe analyses were carried out only in compact areas.

Pitchblende: EDAX analysis indicated that the mineral is not pure UO_2. The spectrum indicated the presence of rare earth elements but no Th. Table 1 depicts the results of quantitative measurements of 32 elements. Of considerable interest are the high concentrations of Pb (6.34% PbO). This amount cannot consist exclusively of radiogenic ^{206}Pb and ^{207}Pb formed by in situ radioactive decay of ^{238}U and ^{235}U. Otherwise, the assumption that all Pb is radiogenic would lead to unreasonably high ages of several billion years. We believe that such high local concentrations of lead are due to its highly mobile nature. Hence, this lead consists not only of pristine radiogenic and ^{204}Pb but also mobilized radiogenic lead. The results also indicate high concentrations in SiO_2, P_2O_5, K_2O, CaO, and V_2O_3. We interpret this as a result of electron beam overlap with minor phosphates and perhaps coffinite intimately intergrown with the pitchblende.

Table 1. Composition of Pitchblende Žirovski Vrh (wt.%).

	%		%	Traces
UO_2	89.34	La_2O_3	0.06	Na
SiO_2	2.14	Ce_2O_3	0.40	Co
MgO	n.d.	Pr_2O_3	n.d.	Ni
Al_2O_3	0.16	Nd_2O_3	0.23	Cu
P_2O_5	0.86	Sm_2O_3	0.09	As
S	0.27	Eu_2O_3	0.16	Se
K_2O	0.55	Gd_2O_3	0.01	Ag
Cao	0.80	Tb_2O_3	0.07	Cs
TiO	0.07	Dy_2O_3	0.34	Th
V_2O_3	0.90	Ho_2O_3	0.18	
Cr_2O_3	n.d.	Er_2O_3	n.d.	Totals = 104.1%
MnO	0.24	Tm_2O_3	0.14	
FeO	0.22	Yb_2O_3	n.d.	
Y_2O_3	0.31	Lu_2O_3	0.06	
ZrO_2	0.04	HfO_2	0.11	
BaO	0.02	PbO	6.34	

The high totals may also be a result of such overlap, since subtracttion of these elements leads to acceptable results.

Coffinite: The composition of coffinite is displayed in Table 2. A striking feature are the high sums (> 102 %) and high concentration in lanthanides (12.25 wt.% REE). The analyses also indicate high concentrations in Hf (1.85 wt.% HfO_2). Likewise pitchblende, other oxides like Al_2O_3, P_2O_5, K_2O, and V_2O_3 were detected. However, we believe also that in this case it is due to contamination from phosphates and silicates intimately intergrown with coffinite. A very important result, however, is that even after subtraction of these elements from the totals, the sum is still close to 100 % (97.87 %). Taking into account the analytical uncertainties, we belive that this result has a severe consequence to the interpretations of electron microprobe analyses of coffinite. In all previous analyses of coffinite published in the literature, any considerable shortage of the totals was explained as due to the presence of OH substituting for other cations in the coffinite structure. However, the major part of these analyses either did not include the REE or only some of these elements were analyzed. If in our case we would ignore the REE elements, Zr, and Hf, we would have a shortage of 14.1 %, which would have been attributed to OH in the coffinite structure. Our results indicate that the amount of OH (if present) is much smaller than anticipated. Ignoring the REE, Zr, and Hf, would certainly lead to erroneous results and misleading interpretations.

Table 2. Composition of Coffinite Žirovski Vrh (wt.%).

	%		%	Traces
UO_2	64.45	La_2O_3	0.30	Na
SiO_2	16.41	Ce_2O_3	1.08	Co
MgO	0.16	Pr_2O_3	0.30	Ni
Al_2O_3	1.31	Nd_2O_3	1.54	Cu
P_2O_5	1.13	Sm_2O_3	0.62	As
S	0.17	Eu_2O_3	0.51	Se
K_2O	0.57	Gd_2O_3	0.66	Ag
Cao	0.27	Tb_2O_3	0.78	Cs
TiO	0.38	Dy_2O_3	0.70	Th
V_2O_3	0.80	Ho_2O_3	1.05	
Cr_2O_3	0.20	Er_2O_3	0.89.	Totals = 102.5 %
MnO	0.23	Tm_2O_3	0.82	
FeO	0.49	Yb_2O_3	1,00	
Y_2O_3	0.84	Lu_2O_3	1,16	
ZrO_2	0.20	HfO_2	1.85	
BaO	0.35	PbO	1.27	

A striking feature is the high preference of Hf, Zr, and REE to coffinite in comparison to pitchblende which is probably structure-controlled.

3.2 Carbonaceous material

In reflected light, no visible inclusions of uraniferous minerals were observed in the carbonaceous material or graphite-like objects. However, carbonaceous materials are potential hosts for uranium, REE, Zr, V, etc. Consequently, they were subject to a detailed SEM study. An X-ray spectrum of an area of 2 mm^2 collected at 30 keV excitation voltage indicated without doubt that the material is highly enriched in U and other elements (fig. 7). In addition to the high concentrations in U, considerable amounts of Al, Si, S, Ca, Cs, REE, As, Y, etc. were recorded. This illustrates the complexity of the assemblages enriched in these elements.

Under the SEM the carbonaceous material and graphite-like objects were found to be loaded with minute inclusions (< 10 microns, fig. 8). Surprisingly, these inclusions are dominantly oriented along one direction, specifically in the graphite-like objects (fig. 8). At higher magnification, it became evident that the inclusions are majorily of prismatic nature and hence it is expected that pitchblende is not the major component of these inclusions (figs. 9 and 10). Due to the high penetration depth of electrons in carbon, it was possible to image some of the inclusions below the polished surface of the graphite-like material to obtain information on the morphology of the inclusions in the third dimension (fig. 10).

Electron microprobe analyses of various inclusions indicate the quite complex nature of the assemblages in comparison to those found in the sandstone impregnations. In addition to pitchblende and coffinite, various phases with high concentrations in REE were found. Some of these phases were found to be selectively enriched in some lanthanide elements; e.g.: 15.1 wt.% Nd_2O_3, 1.96 wt.% SmO_2, 2.86 wt.% Gd, and 14.4

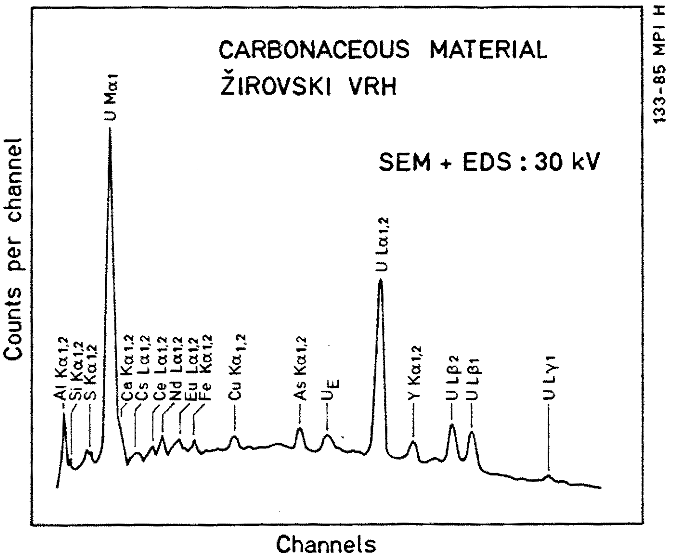

Fig. 7. EDAX spectrum of carbonaceous material depicting the K lines of Al, Si, S, Ca, Fe, Cu, and As, the L lines of REE, and M lines of Cs and U.

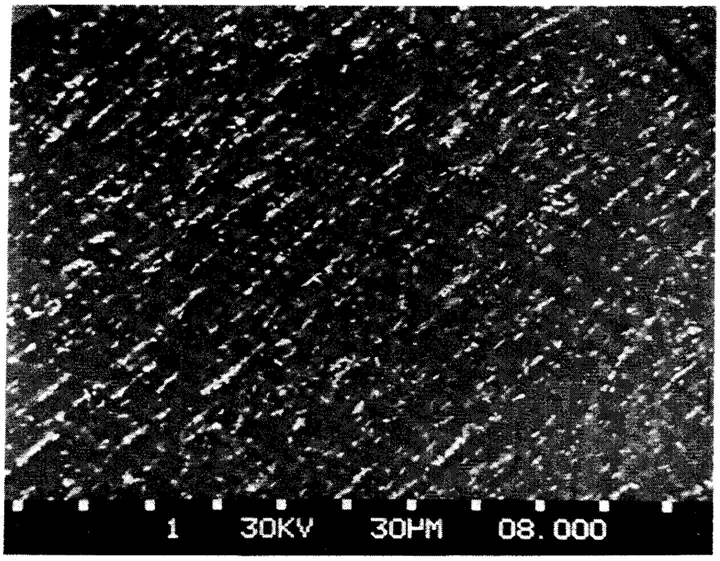

Fig. 8. SEM photograph of carbonaceous materials displaying well ordered U- and REE-rich minerals.

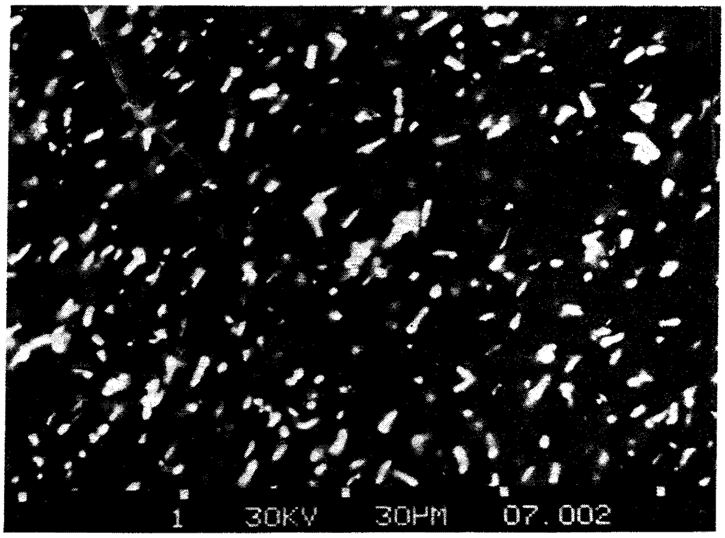

Fig. 9. Detail from Fig. 8 showing the prismatic nature of the mineral inclusions in carbonaceous materials.

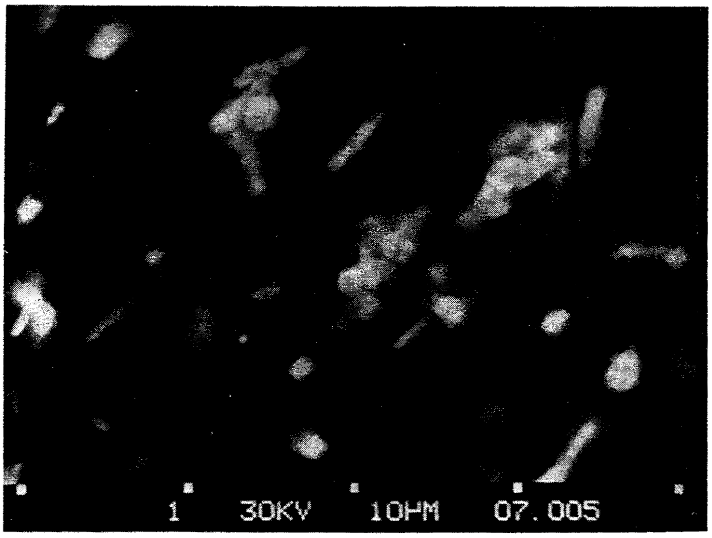

Fig. 10. High magnification SEM photograph of mineral inclusions in carbonaceous materials. The prismatic nature of the inclusions is evident.

wt.% UO_2. The totals were always below 100% (between 40—80%). We believe that the major part of these REE-rich minerals consists of REE-rich carbonates.

Pitchblende: Table 3 depicts the composition of pitchblende in the carbonaceous material. Chemical composition of pitchblende in carbonaceous material is distinct from its counterpart in the impregnations: a) it contains lower concentrations in UO_2 and PbO; b) it is considerably enriched in lanthanide elements. This raises the question of the genetic link between the U-rich minerals in the carbonaceous material and their counterparts in the impregnations.

Coffinite: Coffinite in the carbonaceous material is also enriched in lanthanide elements, Zr and Hf. Likewise in the impregnations, coffinite here shows higher Hf than Zr concentrations. However, the total REE is lower than in coffinite in the impregnations. In contrast, the PbO-content is higher than in coffinite in the impregnations. Compared to the behaviour in the impregnations, this clearly indicates a different partitioning behaviour of these elements in the carbonaceous materials (table 4).

In order to get information on the abundance of minor and trace elements in the mineral assemblages of the carbonaceous material, PIXE (Proton-Induced X-ray Emission) analyses were carried out in several areas of a polished section. The collected spectrum is shown in fig. 11. In addition to the U-, M-, L-lines, X-ray lines of Al, Si, P, S, Ag, Ca, Ba, V, REE, Pb, Se, Y, and Zr were detected. In agreement with previous reports on Žirovski Vrh, no Th spectra were observed. This figure also demonstrates the superiority of PIXE over electron microprobe techniques for detection of heavy elements with low concentrations (see also fig. 7).

Table 3. Composition of Pitchblende in Carbonaceous Material Žirovski Vrh (wt.%).

	%		%	Traces
UO_2	68.39	La_2O_3	0.52	Na
SiO_2	4.52	Ce_2O_3	0.76	Co
MgO	0.24	Pr_2O_3	n.d.	Ni
Al_2O_3	0.47	Nd_2O_3	0.71	Cu
P_2O_5	1.31	Sm_2O_3	0.76	As
S	0.38	Eu_2O_3	0.61	Se
K_2O	0.59	Gd_2O_3	0.51	Ag
Cao	0.70	Tb_2O_3	0.90	Cs
TiO	0.36	Dy_2O_3	0.80	Th
V_2O_3	0.78	Ho_2O_3	0.89	
Cr_2O_3	0.18	Er_2O_3	0.81	Totals = 96.5%
MnO	0.29	Tm_2O_3	1.13	
FeO	0.45	Yb_2O_3	0.78	
Y_2O_3	0.51	Lu_2O_3	1.05	
ZrO_2	0.31	HfO_2	1.68	
BaO	0.32	PbO	4.83	

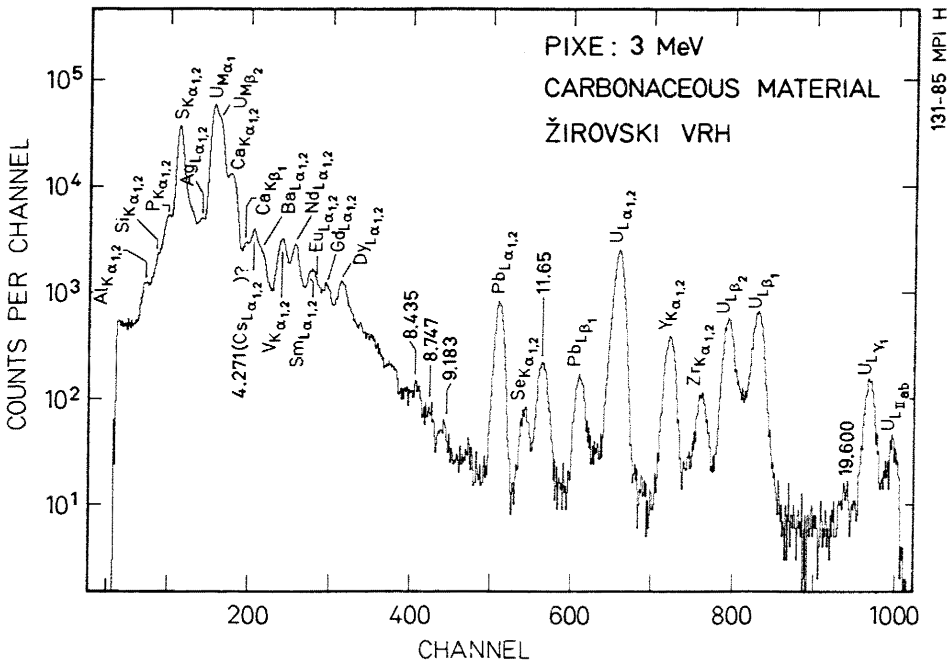

Fig. 11. A PIXE X-ray spectrum of carbonaceous materials demonstrating the enrichment of carbonaceous materials in U, Pb, Zr, Y, REE, V, Al, Si, P, Ca, and Ba.

The origin of the carbonaceous material could be discerned by examination at high resolution with the SEM. Plant residues with well preserved cellular structure are quite abundant. The material now consists of pitchblende or pitchblende and coffinite (figs. 12 and 13).

Table 4. Composition of Coffinite in Carbonaceous Material Žirovski Vrh (wt.%).

	%		%	Traces
UO_2	62.23	La_2O_3	0.36	Na
SiO_2	12.95	Ce_2O_3	0.91	Co
MgO	0.25	Pr_2O_3	n.d.	Ni
Al_2O_3	6.19	Nd_2O_3	1.19	Cu
P_2O_5	2.99	Sm_2O_3	0.64	As
S	0.79	Eu_2O_3	0.08	Se
K_2O	0.65	Gd_2O_3	0.34	Ag
Cao	1.15	Tb_2O_3	0.68	Cs
TiO	0.46	Dy_2O_3	0.69	Th
V_2O_3	0.71	Ho_2O_3	0.73	
Cr_2O_3	0.11	Er_2O_3	0.23.	Totals = 101.7%
MnO	0.16	Tm_2O_3	0.55	
FeO	0.38	Yb_2O_3	0.48	
Y_2O_3	1.59	Lu_2O_3	0.77	
ZrO_2	0.19	HfO_2	0.94	
BaO	0.34	PbO	1.97	

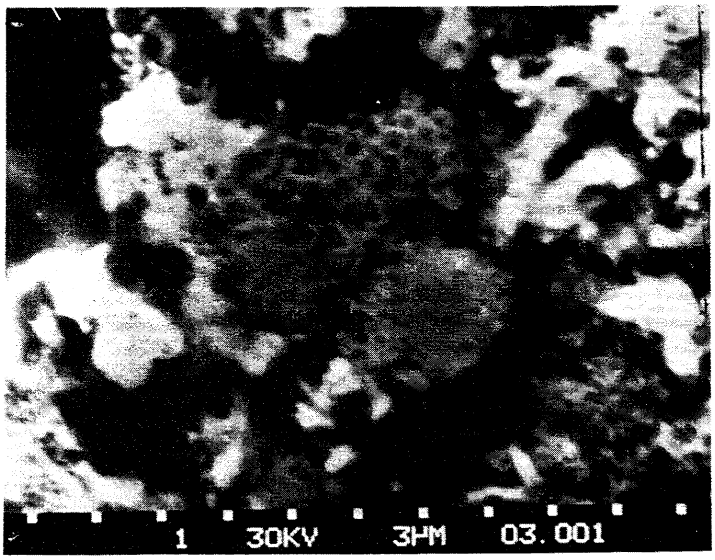

Fig. 12. SEM photomicrograph of plant residues in carbonaceous materials.

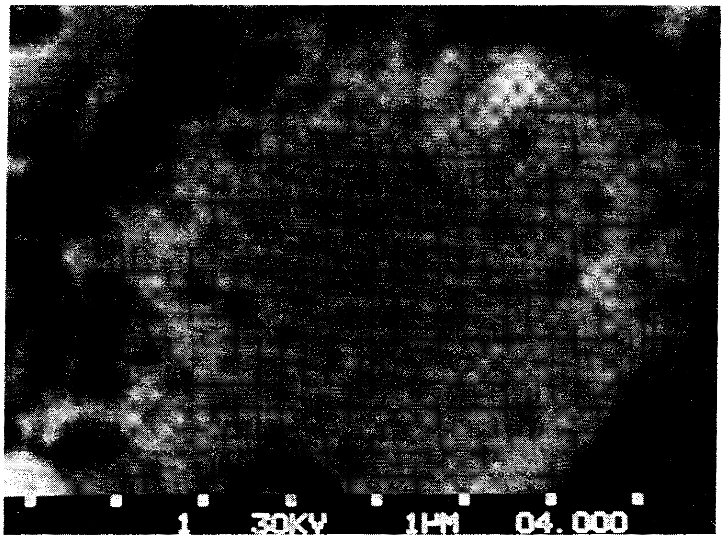

Fig. 13. Detail from Fig. 12 depicting the plant cellular structure now consisting of pitchblende.

4. Discussion

The uranium-rich mineralization in Žirovski Vrh is much more complex than anticipated in all previously published reports on this deposit. Uranium-rich minerals are present both as impregnations and inclusions in pristine carbonaceous materials. This raises the question of the genetic relationships between U and the rare earth elements. Compared to the ore in the impregnations, the carbonaceous materials and the graphite-like objects are enriched in REE. Also in the carbonaceous materials pitchblende shows higher concentrations of lanthanides than in the impregnations. If the emplacement of U, REE, Zr, and Hf were contemporaneous, then why do REE, Zr, and Hf show higher preference to pitchblende and coffinite in the carbonaceous materials? It is noteworthy that our investigations indicate the presence of REE-rich carbonates in carbonaceous materials. These minerals could have acted as traps for U introduced by solutions. Pitchblende and coffinite precipitated in the impregnations should then contain lower concentrations of these elements.

More difficult to explain is the relatively high concentrations of REE in coffinite compared to pitchblende in the impregnations, on the one hand, and the higher REE abundance of pitchblende compared to coffinite in the carbonaceous material, on the other. Crystal chemical effects cannot explain this contrasting behaviour. Nor could this feature be explained as a result of formation at different temperatures since both parageneses occur together. It is more plausible to attribute this feature to a high concentration of these elements in the carbonaceous materials prior to the U-ore emplacement. We suspect, however, that the high REE-content of pitchblende in the carbonaceous material is perhaps the result of the presence of submicroscopic clastic aggregates of REE-rich inclusions in pitchblende. The same may also hold for SiO_2, P_2O_5, K_2O, and CaO detected in both coffinite and pitchblende due to the presence of phosphates.

If all detectable minor elements in coffinite are measured with the electron microprobe, then the deviation of the totals from 100% is quite small. For this reason, we believe that the amount of H_2O or OH in the coffinite structure is very small. Previous reports on high OH-concentrations in coffinite are definitely in error due to lack of complete analysis.

5. Conclusions

Several conclusions can be drawn from the present investigations:

(1) The uranium mineralization in Žirovski Vrh is probably of sedimentary origin, perhaps cogenetic with the sandstone.

(2) Mobilization of UO_2 and PbO took place in a younger episode thus redistributing these elements among various uranium minerals.

(3) Carbonaceous materials enriched in REE-minerals acted as traps for U both during the primary mineralization and the late mobilization episodes.

(4) The total U/Pb ratios measured with the electron microprobe cannot be used to determine the age of the mineralizations since a) both elements are involved in the mobilization and b) the amount of ^{204}Pb and the $^{206}Pb/^{207}Pb$ is unknown so far.

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