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Morphology and Crystallo-Chemical Characteristics of the Fe-, Mn-, Mg-, Ca- Carbonates from Zagrađe near Busovača (Bosnia and Herzegovina)

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Key words: alpine veins, siderite, ankerite, calcite, aragonite, crystal morphology, chemistry, Busovača, Bosnia and Herzegovina.

Abstract

Siderite, ankerite, calcite and aragonite are determined in well known hyalophane contained alpine veins from Zagrađe near Busovača.

Siderite (45.3 % FeO) is the early carbonate in paragenesis. Arrangement of $\{0001\}$, $\{11\bar{2}3\}$, $\{10\bar{1}1\}$ and $\{33\bar{6}2\}$ resulted in two types of habit: isometric with characteristic elements of trigonal symmetry and isometric with elements of pseudomonoclinic symmetry. Two ankerite phases are mainly determined through their Mn content (AI-4.30% MnO, AII-1.94% MnO) and their positions in paragenesis. The unit rhombohedron and twin on basal pinacoid are their representative habits. Saddle-shaped rhombohedrons of calcite and aragonite very rich in crystal faces ({110}, {010}, {011}, {021}, {111}, {121}, {031}, {051}, {012}, {032}, {122} and {362}) have been separated among late carbonates. The crystals of aragonite are strongly elongated in the direction of [001] and flattened on (110) and this face is the common twin plane.

At the beginning initial solution was very rich in iron which rapidly decreased during crystallization. The solution contained more Fe, Mn and Mg at the beginning of crystallization and more Ca during the final stage. Such a complex solution generated the described carbonates according to their variable solubility. Determined elements confirm the existence of members including manganoan ankerites which are more common than generally thought. According to partial substitutions of Mn for Fe in octahedral sites ankerites would be formulated as; $(Ca_{0.94}Mn_{0.06})(Fe_{0.48}\,Mg_{0.45}Mn_{0.16}Sr_{0.02})(CO_3)_2$ and $(Ca_{0.94}Mn_{0.06})\,(Fe_{0.46}Mg_{0.53})(CO_3)_2$.

Ključne riječi: žile alpskog tipa, siderit, ankerit, kalcit, aragonit, kristalna morfologija, kemizam, Busovača, Bosna i Hercegovina.

Sažetak

Iz poznatih žila alpskog tipa sa hijalofanom od Zagrađa kraj Busovače određeni su karbonatni minerali: siderit, ankerit, kalcit i aragonit.

Siderit (45,3% FeO) je najstariji karbonat parageneze. Raspored ploha formi $\{0001\}$, $\{11\bar{2}3\}$, $\{10\bar{1}1\}$ i $\{33\bar{6}2\}$ rezultira dvjema vrstama habitusa: izometričnim s karakterističnim stupnjem simetrije i izometričnim s elementima pseudomonoklinske simetrije. Dvije ankeritne faze se uglavnom razlikuju na osnovu sadržaja Mn (AI-4,30% MnO, AII-1,94% MnO) i pozicijom u paragenezi. Karakteristični habitus im je osnovno romboedrijski a tvore i sraslace po baznom pinakoidu. Sedlasto osnovno romboedrijski kalciti i aragoniti bogati kristalnim plohama formi ({110}, {010}, {011}, {021}, {111}, {121}, {031}, {051}, {012}, {032}, {122} i {362}) izdvojeni su među mlađim karbonatima. Kristali aragonita su izduljeni u smjeru [001] i spljošteni po (110) što je redovno i ravnina sraštanja.

Količina željeza naglo opada tokom kristalizacije u izvornoj otopini. Općenito otopina sadrži više Fe, Mn i Mg početkom kristalizacije a više Ca u finalnom stadiju. Takva kompleksna otopina generira opisane karbonate na temelju razlike topljivosti. Kemizam određivanih karbonata potvrđuje postojanje članova kao što su manganoankeriti u većem opsegu od uvriježenog mišljenja. S obzirom na mogućnost dijelomične supstitucije Fe s Mn u oktaedrijskim pozicijama sastav istraživanih ankerita predočujemo sa: $(Ca_{0,94}Mn_{0,06})(Fe_{0,48}Mg_{0,45}Mn_{0,16}Sr_{0,02})(CO_3)_2$ i $(Ca_{0,94}Mn_{0,06})(Fe_{0,46}Mg_{0,53})(CO_3)_2$.

1. INTRODUCTION

In the Zagrade area, about 5 km southwest of Busovača (Central Bosnia), a subvertical system of alpine veins cut Palaeozoic schists. Ba-rich veins contain hyalophane and quartz as the main minerals (DIVLJAN, 1954). Rare minerals include: siderite, muscovite, anhydrite, pyrite (BARIĆ, 1955, 1969, 1972/73); anatase, rutile, hematite (ZEBEC, 1980/81; BERMANEC & ZEBEC, 1987); apatite (ZEBEC & ZAGORŠĆAK, 1983) and albite (ZEBEC & BERMANEC, 1985). In this area uncommon metamorphic Cr-Ba-Fe-Cu deposits with two carbonates, siderite and Mn-ankerite, are recorded (JURKOVIĆ et al., 1992).

Recently, prior to 1990, a few hundred samples of carbonates were collected from the old mine outcrop. This material was classified into several according to differences in crystal habit, paragenetic relationships and intensity of alteration. Representatives of these groups have been analysed.

Carbonate minerals are a complex group based on the CO_3 anion as a fundamental chemical unit. Particularly, the rock-forming carbonates belong to the ternary system $CaCO_3$ - $FeCO_3$ - $MgCO_3$ and have caught the attention of many authors from many different points of view. More complex carbonates which belong to the ternary system $CaMg(CO_3)_2$ - $CaFe(CO_3)_2$ - $CaMn(CO_3)_2$ have been the subject of several crystal chemical studies during the last ten years (BERAN & ZEMANN, 1977; EFFENBERGER et al., 1981; REEDER & DALLASE, 1989).

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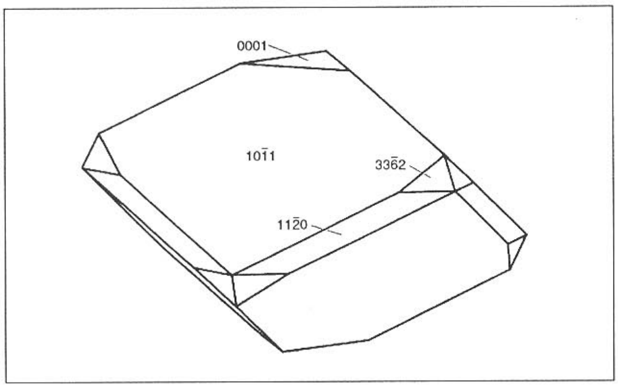

Fig. 1. Siderite, Busovača, isometric habit.

2. EXPERIMENTAL PROCEDURES

Crystal morphology is determined using a Goldscmidt type two-circle reflecting goniometer. The faces were indexed from a gnomonic plot. The parallel perspective projections of measured crystals were constructed from a gnomonic plot, by the method described by TERPSTRA & CODD (1961). The rhombohedron angles of $\{10\bar{1}1\}$ were measured on the cleavege fragments or crystals of rhombohedral carbonates. Such goniometric measurements of rhombohedal angle have been confirmed as a quick, simple and reliable method for distinguishing rhombohedral carbonates. On the same fragments and on the crystal faces (110) of aragonite refractive indices were determined by refractometer (refractometer Eickhorst, Gemmodul M1, width of spectrum band 30 nm, max. 589 nm).

The samples of carbonates have also been analyzed by inductively coupled argon-plasma atomic emission spectrometry (ICP-AES) using an ARL ICP-35000 C spectrometer. The input power was set at 1200 W.

3. RESULTS

3.1. CRYSTAL MORPHOLOGY

3.1.1. Siderite

This is the early carbonate in paragenesis. It is distinguished by an expressive chocolate brown colour and a very evident regular unit rhombohedron habit. Crystals range from a few mm till 2 cm in size. Faces from certain crystals show interference colours, mainly greenish yellow, due to a thin film of oxidation on the surface. Fortunately this effect does not reduce the precision of the measurements.

The faces of the basal pinacoid $\{0001\}$ are very common as well as the dominant unit rhombohedron. The following faces were more rarely determined: $\{33\bar{6}2\}$ and $\{11\bar{2}0\}$. Arrangement of these faces usually results in an isometric crystal habit. Such a habit is represented by Fig. 1. Another characteristic isometric habit has been determined and is represented on Fig. 2. This is the result of the following arrangement of forms: $\{0001\}$, $\{11\bar{2}3\}$, $\{11\bar{2}0\}$ and $\{10\bar{1}1\}$. These two crystal types, besides their isometric habit, show characteristic stages of crystal symmetry.

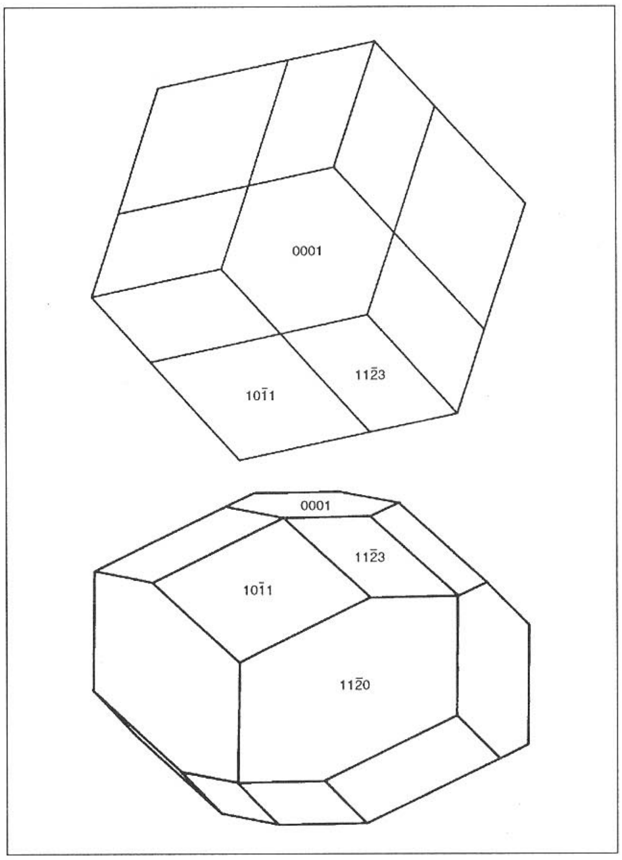

Fig. 2. Siderite, Busovača, isometric habit.

Figure 3 shows a distinctive characteristic crystal of siderite which seemingly assumes a shape of monoclinic symmetry because one pair of centro-symmetric faces of form $\{11\bar{2}3\}$ do not appear. Goniometric measuring over such crystal types have shown the existence of a continuous signal among two faces of the form $\{11\bar{2}3\}$ and intermediate face of unit rhombohedron $\{10\bar{1}1\}$. The same signals are clearly and sharply separated toward the other two faces of the unit rhombohedron.

Only a small number of siderite crystals are completely preserved from the ubiquitous limonisation process. These crystals are pale yellow, almost colourless and transparent. They appear like small druza. It is obviously an earlier deposit than the late rhombohedral ankeritic carbonates.

Occasionally on the siderite crystals, of the unit rhombohedral habit, much steeper saddle-shaped rhombohedral faces incline to the prism position. They are completely limonised which makes any discussion about their composition impossible. However, their forms and orientation have retained a precisely parallel condition.

The crystals of the simple unit rhombohedron habit are mainly up to 1 mm in size, exceptionally up to 3 mm, and are crystallized in the same manner. They are regularly covered with yellowish-ocher coat. These

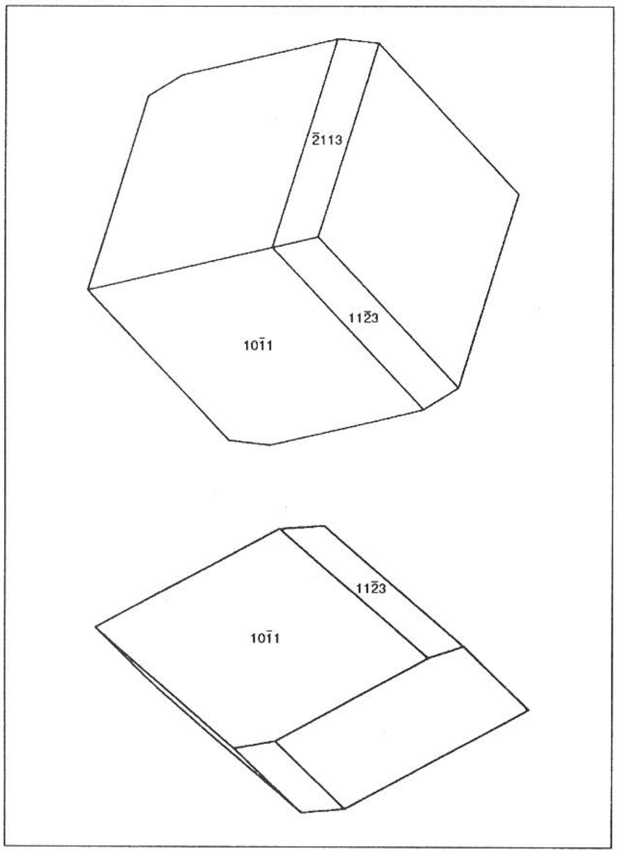

Fig. 3. Siderite, Busovača, with apparent elements of monoclinic symmetry.

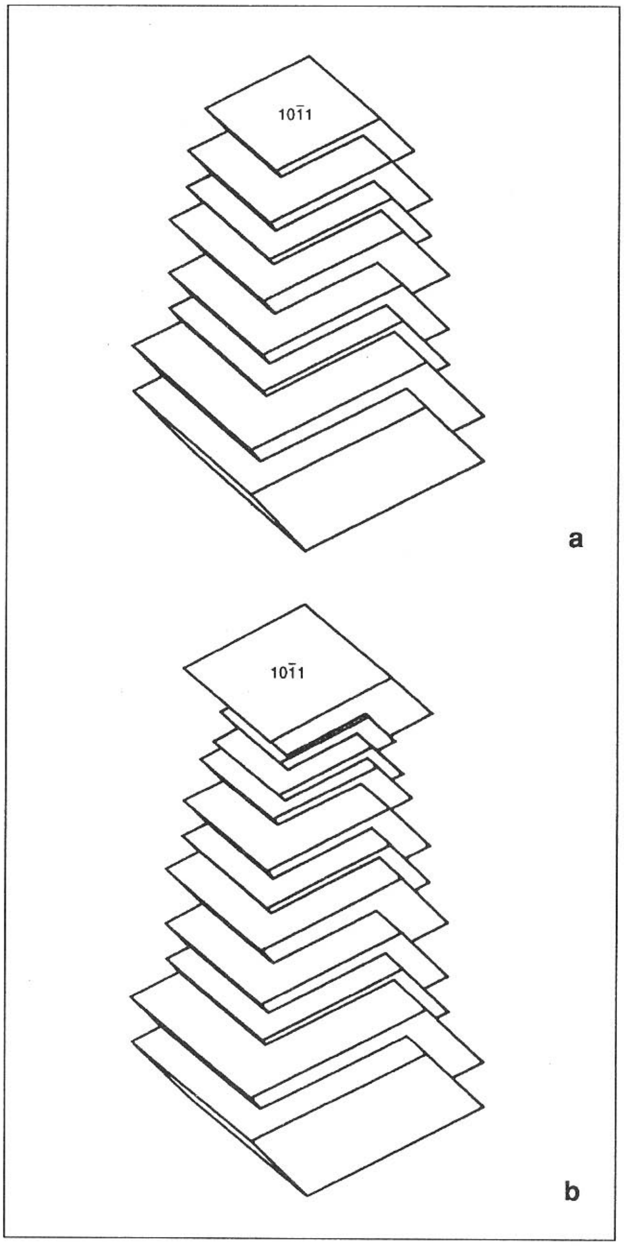

Fig. 4. a) Siderite, Busovača, grown parallel along the [0001] direction. b) Siderite, Busovača, the last rhombohedron seems like a "cap" of the string.

crystals usually grow with [0001] parallel to each other as shown in Fig. 4a. At the same time such formations grew parallel over larger crystal of siderite. These tiny crystals are often fresh and have been determined as siderite by measurements of their rhombohedral angle.

On such a string of parallel grown crystals every successive rhombohedron is somewhat smaller. However, the last crystal of such a string is sometimes bigger and set "apart" from it's predecessors, and looks like a cap over such unity (Fig. 4b). All these crystals exhibit strictly parallel orientation of all three generations. These samples were not analyzed because of the exceptionally small quantity of pure materials.

3.1.2. Ankerite I

The biggest carbonate crystals in this paragenesis are crystals of early ankerite (ankerite I). They are up to 5 cm in size, with the unit rhombohedron as their representative habit. They very often grow over the basal pinacoid (0001) to form twins as shown in Fig. 5. Some crystals are elongated in the direction of one rhombohedral zone, thus deviating from the ideal habit. Figure 6 represents such a type with significant expressive faces of both the unit rhombohedron and the general rhombohedron $\{32\bar{5}1\}$.

All crystals of this generation are regularly, partly or even completely limonised. Limonisation is most obvious on the surface and along the perfect rhombohedral cleavage. Internally some crystals are, however, very fresh and from which the cleaved splinters are completely clear and colourless. Goethite is the result of the limonisation process (as determinated by x-ray powder diffraction analysis).

3.1.3. Ankerite II

Milky white crystals with unit rhombohedral habit, up to 3 mm in size, sometimes grow upon siderite. These crystals are rather brittle and have obvious traces of dissolution, but do not exhibit any trace of limonisation. Ankerite I and ankerite II are very similar if one takes just the Fe, Ca and Mg content into account, but because of the different growth position crystals and their important difference in Mn content it is necessary to distinguish them as two distinct members of this paragenesis.

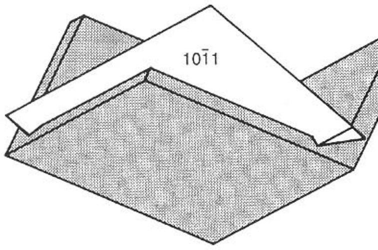

Fig. 5. Ankerite I, Busovača, twin over basal pinacoid (0001).

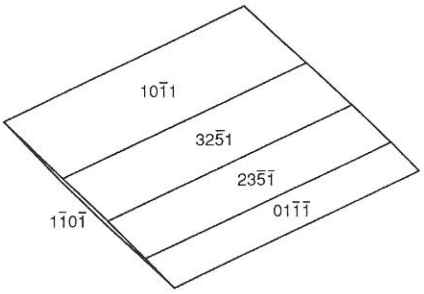

Fig. 6. Ankerite I, Busovača, elongated crystal with expressive faces of one zone.

3.1.4. Calcite

Spherical accumulations with expressive curved rhombohedrons passing into saddle-shaped forms have been separated among the later carbonates. They have been located upon hyalophane, quartz, siderite, ankerite and even sometimes over aragonite. Crystals of flat rhombohedral habit $\{01\bar{1}2\}$ only are indicated. They are sometimes found as overgrowths of early ankerite with the crystal axes oriented in the parallel position. Neither the crystals nor their rhombohedral cleavege fragments are suitable for quantitative goniometric measurements because they are curved.

3.1.5. Aragonite

Aragonite is found as colourless crystals up to 8 mm in size. The crystals are strongly elongated in the direction of [001]. They are usually flattened on (110) and this face is a common twin plane (Figs. 7 and 8). Repetition on (110) in polysynthetic twin lamellae is also common.

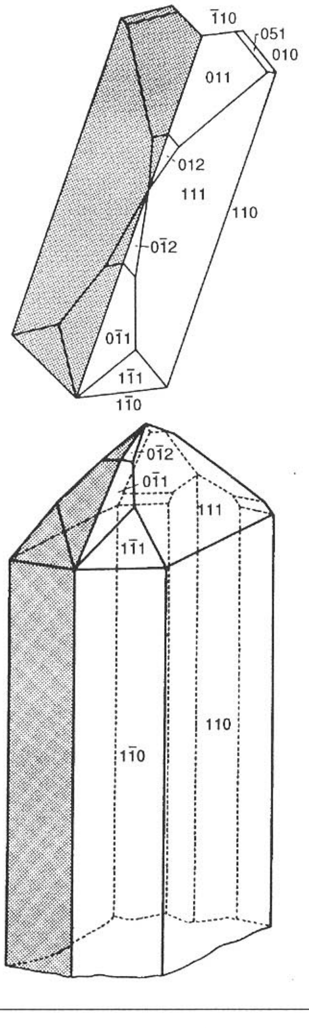

Fig. 7. Aragonite, Busovača, twin on (110).

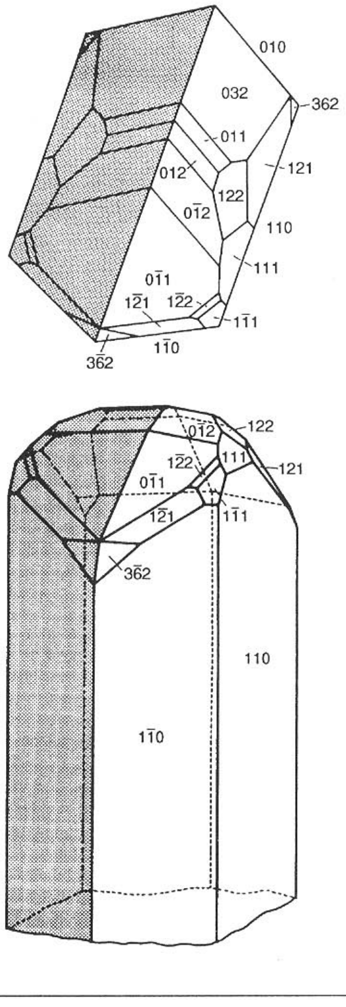

Fig. 8. Aragonite, Busovača, twin on (110).

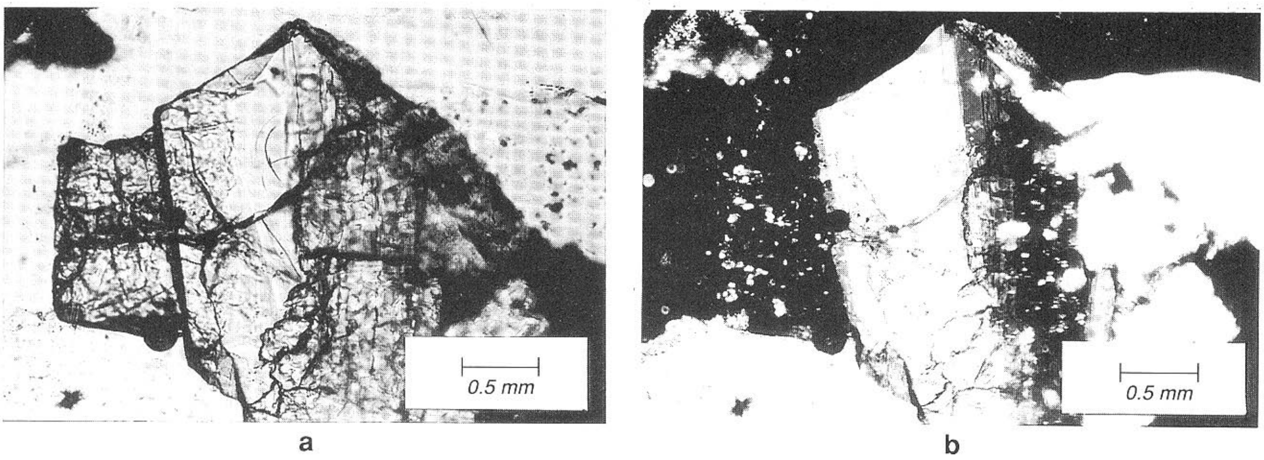

Fig. 9. a) Aragonite, Busovača, section perpendicular to the c axis, N-; b) N+.

Occasionally it is difficult to detect the presence of such twin types, in which case twin sections perpendicular to the *c* axis are very helpful indicators of the existence of twinning (Fig. 9)

Six aragonite crystals were measured. A review of the forms with their measured and calculated angle values is shown in Table 1.

3.2. THE ANGLE OF UNIT RHOMBOHEDRON AND THE REFRACTION INDEX

The angle value of unit rhombohedras were measured over the same rhombohedral cleavege fragment which was used for measurements of refraction indexes by total refractometer. In the case of aragonite, the faces of {110} were used for refraction measurements. The results are compared with the literature date and are shown in Tables 2a and 2b.

3.3. CHEMICAL COMPOSITION

All selected samples were analyzed for iron, calcium, magnesium, manganese, strontium, barium, zinc and lead. Determined elements are shown in the oxide form in Table 3. The amount of CO_2 has not been determined but calculated on the basis of determined cations.

The ternary composition space $CaMg(CO_2)_3$ - $CaFe(CO_2)_3$ - $CaMn(CO_2)_3$ is shown in Fig. 10 together with available carbonate analyses. Our data for rhombohedral carbonates confirm the existence of extensive solid solution between dolomite, ankerite and kutnahorite as well as the existence of the members like manganoan ankerites or manganoan dolomites which are more common than generally thought.

4. DISCUSSION

From the carbonate chemistry (Fig. 11), we can conclude that initial solution was originally very rich in iron (siderite - 45.3% FeO). The quantity of iron decreased during crystallization. The final stage was essentially iron-free (aragonite - 0.03% FeO). Magnesium is constantly present through the siderite and ankerite phase (8.6 - 10.7% MgO) but its value rapidly decreases (2.0% MgO) in the calcite phase and completely disappears during the aragonite phase.

	calculated value		**measured value**		**crystal**					
form	φ	ρ	φ	ρ	**1**	**2**	**3**	**4**	**5**	**6**
110	58°06'	90°00'	57°50' - 58°55'	87°08' - 92°27'	+	+	+	+	+	+
010	0°00'	90°00'	-0°22'	88°10' - 90°11'	+	+	+	+	+	+
011	0°00'	35°46'30"	-0°22' - -0°05'	35°40' - 40°53'	+	+		+	+	
021	0°00'	55°14'30"	0°00'	53°50' - 58°47'	+	+	+	+		
111	58°06'	53°45'	57°50' - 59°01'	53°37' - 53°52'	+	+	+	+	+	+
121	38°46'30"	61°35'30"	38°53'	58°46' - 66°30'	+		+		+	
031	0°00'	65°10'30"	0°00'	65°58'		+				
051	0°00'	74°29'30"	0°21'	73°59'				+		
012	0°00'	19°49'	-0°22'	19°20' - 20°12'				+	+	
032	0°00'	47°14'	0°32'	47°10' - 47°21'				+		
122	38°46'30"	42°45'	39°18'	43°20'				+		
362	38°46'30"	79°46'	39°06'	77°20'				+		

Table 1. Aragonite, Busovača. Determination of the form as based on axial ratio a : b : c = 0.6224 : 1 : 0.7206 (PALACHE et al., 1951, p 183). The presence of form is indicated with sign +.

		measured angle $\{10\bar{1}1\}$	PALACHE et al. (1951)
siderite			43°23'
	nucleus	43°12'	
	edge	43°24', 43°27'	
ankerite I			43°51'30''
	nucleus	43°51', 43°54'30'',	
	transit	43°53', 43°54'	
	edge	43°54'30'', 43°55'30'', 43°59'	
ankerite II			
	inclusion from hyalophane	43°51'	

Table 2a. Comparison of measured and published unit rhombohedral angles.

	measured	EHLERS (1987)
ankerite I	$\omega =$ /	$\omega = 1.679 - 1.738$
	$\varepsilon = 1.531(1)$	$\varepsilon = 1.500 - 1.542$
aragonite	$\alpha = 1.534(1)$	$\alpha = 1.527 - 1.542$
	$\beta =$ /	$\beta = 1.670 - 1.695$
	$\gamma =$ /	$\gamma = 1.676 - 1.699$

Table 2b. Comparation of measured and published indices of refraction.

Manganese is the most prominent during early crystallization (5.3% MnO). Between siderite and ankerite the Mn content decreased slightly (4.3% MnO). The evident decrease of manganese is shown in late ankerite II (1.9% MnO). Mn like Fe and Mg disappears at the end of the crystallization of the rhombohedral carbonates. The distribution of calcium is inverse. At the beginning of crystallization, in the siderite phase, the value is the lowest (0.69% CaO), increasing through ankerite phase (27.1 CaO), reaching a maximum in the calcite and aragonite phase (54.0 - 55.24% CaO). Strontium is not detected in siderite. The highest value is detected at the beginning of the ankerite phase (0.9% SrO) and after that it successively disappears. The appearance of barium is irregular and low (max. 0.2% BaO - ankerite II).

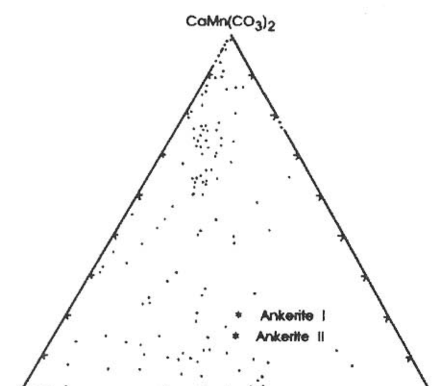

Fig. 10. Carbonate composition (mol %) in the system $CaMg(CO_3)_2$-$CaFe(CO_3)_2$-$CaMn(CO_3)_2$, from "Carbonates: Mineralogy and Chemistry, MSA (1983), with data of Ankerite I - AI and Ankerite II - AII from Busovača.

In earlier literature morphological data of the rhombohedral carbonates, except for calcite, are not well elaborated. There are also less structural data available for ankerite than for calcite or isostructural dolomite. We can compare our results with ankerite from Erzberg, Austria. BERAN & ZEMANN (1977) have analyzed a similar sample ($Ca_{1,00}(Ca_{0,05}Mg_{0,27}Fe_{0,63}Mn_{0,05})(CO_3)_2$) and treated the distribution of the cation structure in a conventional way. There are two distinct cation sites, designated A and B. Both occupy a nearly regular octahedral form in which each corner is an oxygen from a different CO_3 group. The A site is occupied by Ca and the B site by Mg and Fe in the ideally ordered case, where layers of Ca octahedra alternate with layers of (Mg, Fe) octahedra along the c axis. The octahedra are linked by sharing corners simultaneously with octahedra of the opposite kind and with CO_3 groups. There is no edge sharing in the structure.

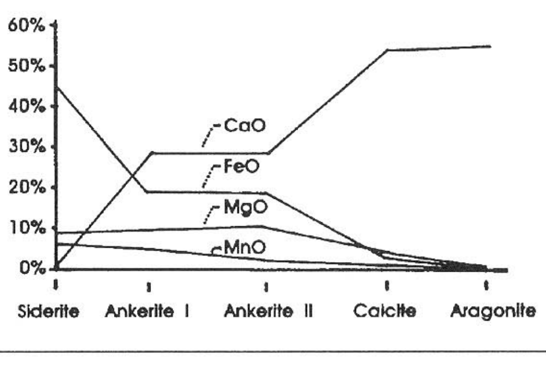

Fig. 11. Distribution of Fe, Ca Mg and Mn through the main carbonate phase.

	siderite	ankerite I	ankerit II	calcite	aragonite
Zn	190	35	1000	<	115
Pb	95	<	700	<	<
CaO	0.690	27.120	26.360	54.000	55.240
MgO	8.610	9.320	10.710	2.040	0.004
FeO	45.300	17.710	16.620	0.820	0.030
MnO	5.330	4.300	1.940	0.040	0.001
SrO	0.000	0.920	0.220	0.090	0.400
BaO	0.016	0.006	0.210	0.000	0.004
CO_2	41.003	45.369	45.171	43.545	
Tot.	100.949	104.745	99.919	102.161	99.223
CaO	0.684	25.892	26.381	52.858	55.672
MgO	8.529	8.898	10.719	1.997	0.004
FeO	44.874	16.908	16.633	0.803	0.030
MnO	5.280	4.105	1.942	0.039	0.001
SrO	0.000	0.878	0.220	0.088	0.403
BaO	0.016	0.006	0.210	0.000	0.004
CO_2	40.618	43.313	43.895	44.215	43.886
Tot.	100.000	100.000	100.000	100.000	100.000
Ca	0.013	0.938	0.943	0.938	0.991
Mg^{2+}	0.229	0.449	0.533	0.049	0.000
Fe^{2+}	0.676	0.478	0.464	0.011	0.001
Mn^{2+}	0.081	0.118	0.055	0.001	0.000
Sr^{2+}	0.000	0.017	0.004	0.001	0.004
Ba^{2+}	0.000	0.000	0.000	0.000	0.000
CO_3^{2-}	1.000	2.000	2.000	1.000	1.000

siderite $(Fe_{0.68}Mg_{0.23}Mn_{0.08}Ca_{0.01})CO_3$
ankerite I $Ca_{0.94}(Fe_{0.48}Mg_{0.45}Mn_{0.12}Sr_{0.02})(CO_3)_2$
ankerite II $Ca_{0.94}(F_{e0.46}Mg_{0.53}Mn_{0.06})(CO_3)_2$
calcite $(Ca_{0.94}Mg_{0.05}Fe_{0.01})CO_3$
aragonite $(Ca_{0.99}Fe\text{-}Sr_{0.01})CO_3$

Table 3. Chemical analyses of carbonates. The elements have been determined by ICP AES and are shown as oxides. The amount of CO_2 has been calculated on the basis of determined cations. Zn and Pb (in ppm) are are not given in structural formulae.

In the ankerites there are 3 major cation species that are distributed over two distinct sites. In the structural refinement of ankerite from Austria, BERAN & ZEMANN (1977) assumed that the larger A site was completely filled by Ca, leaving Mg and Fe on the smaller B site. This choice of cation distribution reflects the opinion that the larger relative size of Ca dictates its strong preference for the larger A site.

Recently, considerable attention has been focused on substitution at the A site. Refined occupancies (REEDER & DOLLASE, 1989) show that the A site is usually 94% to 99% filled with Ca. EPR studies have shown that most specimens contain some Mn on the A site. By combining Fe and Mn if is suggested that the preference for the B site is stronger for Fe than for Mn. In this case the rest of the A site will usually be filled by Mn. According to such a point of view our ankerites would be expressed in this manner;

ankerite I

$$(Ca_{0.94}Mn_{0.06})(Fe_{0.48}Mg_{0.45}\,Mn_{0.16}Sr_{0.02})\,(CO_3)_2$$

ankerite II

$$(Ca_{0.94}Mn_{0.06})(F_{e0.46}Mg_{0.53})(CO_3)_2$$

5. CONCLUSIONS

The main conclusions from the present study are:

1. Five carbonate phases with obvious morphological differences are separated and analysed. Two ankerite phases are determined through chemical differences and their paragenetic position.
2. Calcium and iron show an inverse distribution of their concentration in the crystallising solution. This complex solution probably generated the sequence of described carbonates in accordance with their variable solubilities. The initial solution was initially richer in Fe, Mn and Mg, with more Ca during the final stage of crystallisation. Sr is the most abundant minor constituent becoming less important toward the end of crystallisation. Ba did not influence crystallization of the carbonates in the veins in Busovača.
3. The present results are compatible with new crystallo-chemical interpretations of anhydrous rhombohedral carbonates.
4. The goniometric measurements of rhombohedral angle have confirmed as a quick, simple and reliable method for distinguishing siderite from ankerite.

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