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Miha JERŠEK & Breda MIRTIČ:
Corundum from Prilep Dolomitic Marble (Macedonia)

Korundi prilepskega dolomitnega marmorja (Makedonija)

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Corundum from Prilep Dolomitic Marble (Macedonia)

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ABSTRACT – Structure, chemical composition and solid inclusions exert an influence upon the physical properties of minerals. Besides other parameters they also determine the value of the mineral as a gemstone. In this study we investigated corundum from the dolomitic marble of Prilep (Macedonia), which belongs to the highly metamorphic rocks of the Pelagonian Massif. Marbles show a characteristic mineral paragenesis and include dolomite, calcite, corundum, diaspore, and other accessory minerals. The corundum from Prilep marble appears in a wide number of morphological forms, some of which were identified for the first time in this study. Oriented diaspore intergrowth is also seen in corundum crystals. This affects the physical properties of corundum, such as colour, hardness and density. Diaspore intergrowth can be seen as a silvery glittering in the corundum crystals. We introduce the term "diasporescence" for this phenomenon.

Key words: corundum, diaspore, dolomitic marble, diasporescence, morphological forms, Macedonia

IZVLEČEK – KORUNDI PRILEPSKEGA DOLOMITNEGA MARMORJA (MAKEDONIJA) – Na fizikalne lastnosti mineralov vplivajo struktura, kemična sestava in vključki v mineralu. Omenjene lastnosti imajo pomemben vpliv pri določanju kakovosti minerala v draguljarstvu. Preiskovali smo korunde prilepskega dolomitnega marmorja iz Makedonije. Dolomitni marmorji so del visokometamorfnih kamnin Pelagonskega masiva, v katerih najdemo tipično mineralno paragenezo regionalne metamorfoze. To predstavljajo minerali dolomit, kalcit, korund, diaspor in drugi spremljajoči minerali. Kristali korunda v prilepskem dolomitnem marmorju imajo pestre morfološke oblike, med katerimi so nekatere prvič opisane v tem primeru. Določili in preučili smo tudi orientirano preraščanje diaspora in korunda. Le to vpliva na fizikalne lastnosti korunda kot so barva, trdota in gostota. Preraščanje diaspora s korundom se pojavlja kot srebrnkasto svetlikanje v korundu. Pojav smo poimenovali diasporescenca.

Ključne besede: korund, diaspor, dolomitni marmor, diasporescenca, morfološke oblike, Makedonija

Introduction

We first saw corundum crystals from Macedonia more than 10 years ago at the Mineral Exhibition in Tržič, Slovenia. At that time it was very difficult to obtain cut and perfectly polished corundum (ruby) from Macedonia. With the expansion of the exploitation of marble which contains calcite nests of corundum crystals, we had an opportunity to select some interesting Macedonian specimens.

Corundum occurrences in marble are known from the vicinity of Prilep (about 70 km southward of Skopje) in Macedonia (Fig. 1). The Sivec Quarry (Fig. 2) is the most important deposit of dolomite marble and the main source of corundum crystals. However, they can be found also near the quarry and at some other localities (Belovodica and Beloto, both no more than 13 km from Prilep) (Fig.2).

The marble series (thickness = 1500 m) is the uppermost part of highly metamorphic rocks which belong to the central part of the Pelagonian Massif (Fig. 3). The dolomitic marbles are fine-grained and white to grey (calcitic marbles are more coarse grained). The grains are of an isometric shape, with well-defined polysynthetic lamellae. No fossils were found in the metamorphic rocks of the Pelagonian Massif (STOJANOV, 1960; DUMURDJANOV et al., 1990: Fig. 3 and 4). Marbles are characterised by the following minerals: dolomite, calcite, corundum, diaspore, ß-zoisite, rutile, fluorite, achroite, pyrite, muscovite, illite, margarite and chlorite (BARIĆ, 1969; ŽORŽ et al., 1991). The paragenesis mentioned above confirms the regional origin of this dolomitic marble (BARIĆ, 1969).

Corundum, in general, appears in different morphological forms, *i.e.* in combinations of pinacoids, rhombohedrons, bipyramids and prisms (SCHMETZER, 1986). BARIĆ (1963) described tabular and prismatic crystals from Sivec and determined pinacoid c, second order prisms a, rhomboedron r and bipyramidal crystal faces n and w. The morphological characteristics were very similar to the corundum crystals from Campolungo in Switzerland (SCHIFFMANN, 1969). Barić determined oriented diaspore intergrowth with corundum on the crystals from Sivec for the first time (BARIĆ, 1960; 1963; 1969). Diaspore in corundum crystals is oriented in three directions crossing at 60 degrees, resembling a sagenite-like structure (BARIĆ, 1963). One of the finest diaspore crystals is known from Sivec (BARIĆ, 1979).

Natural crystals of corundum always contain inclusions of other minerals, in liquid and/or a gaseous phase. Ions of Si, Fe, Mn, V, Cr, Ti, Ca, Ni, Cd, Zr, Ba and Ga are the most common admixture (HARDER, 1969) and the total amount of inclusions in corundum can exceed 10 percent by weight. This characteristically has an influence on the aesthetic qualities, appearance and colour of corundum crystals (BAUER, 1969 b).

Corundum from Prilep dolomitic marble is rarely of gemstone quality and only useful as cabochons (HENN, 1995). It is mostly light pink and occasionally intensely pink, pinkish-red or blue (BARIĆ, 1977; SCHIFFMANN, 1969). The colour in corundum results from the presence of Cr, V, Fe and Ti ions (Harder, 1969; SCHIFFMANN, 1969; BARIČ, 1969). Corundum also has orange and red fluorescence (Eppler, 1989); the two-colour fluorescence of corundum also shows differently-coloured zones in its structure (SCHIFFMANN, 1969).

Some data on corundum and other minerals from Prilep dolomitic marble have been published by AREM (1987), HENN (1990), JERŠEK (1996) and JERŠEK & MIRTIČ (1997). Within the last few years the foremost goal of our work has been to investigate corundum crystals from the Prilep dolomitic marble. About 100 kilograms of corundum specimens were collected and analysed. They showed different morphological forms, some of them intensely red–coloured, with the silverly–white glittering of diaspore always being present on corundum. We also found good gem quality pieces of corundum. Our colleague, Franc Arbeiter a gem cutter from Mežica, Slovenia, told us how to polish Macedonian rubies. The results were fascinating enough to provide us with a fresh impetus for returning to research on corundum.

Figure 1: Geographic map of Macedonia

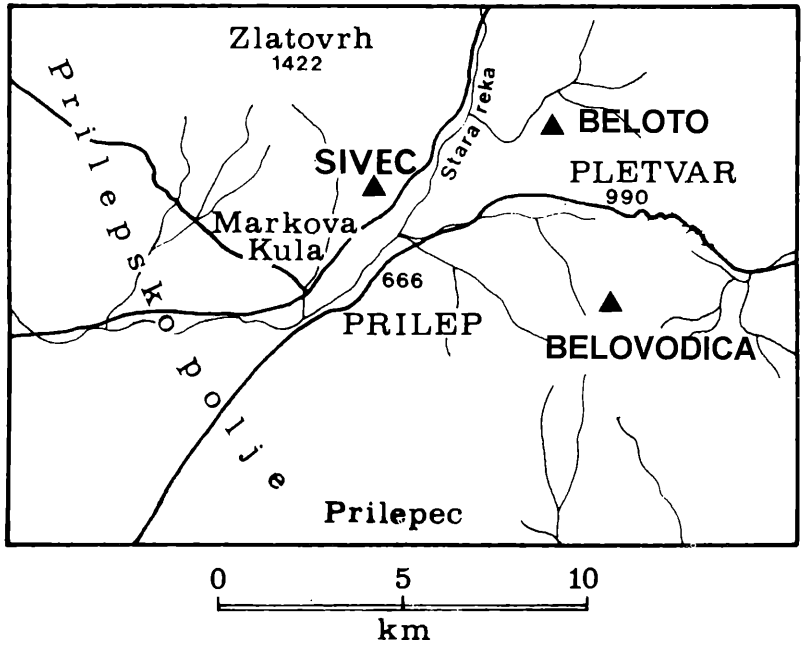


Figure 2: Geographic position of corundum deposits in the vicinity of Prilep (from Žorž *et al.*, 1991)

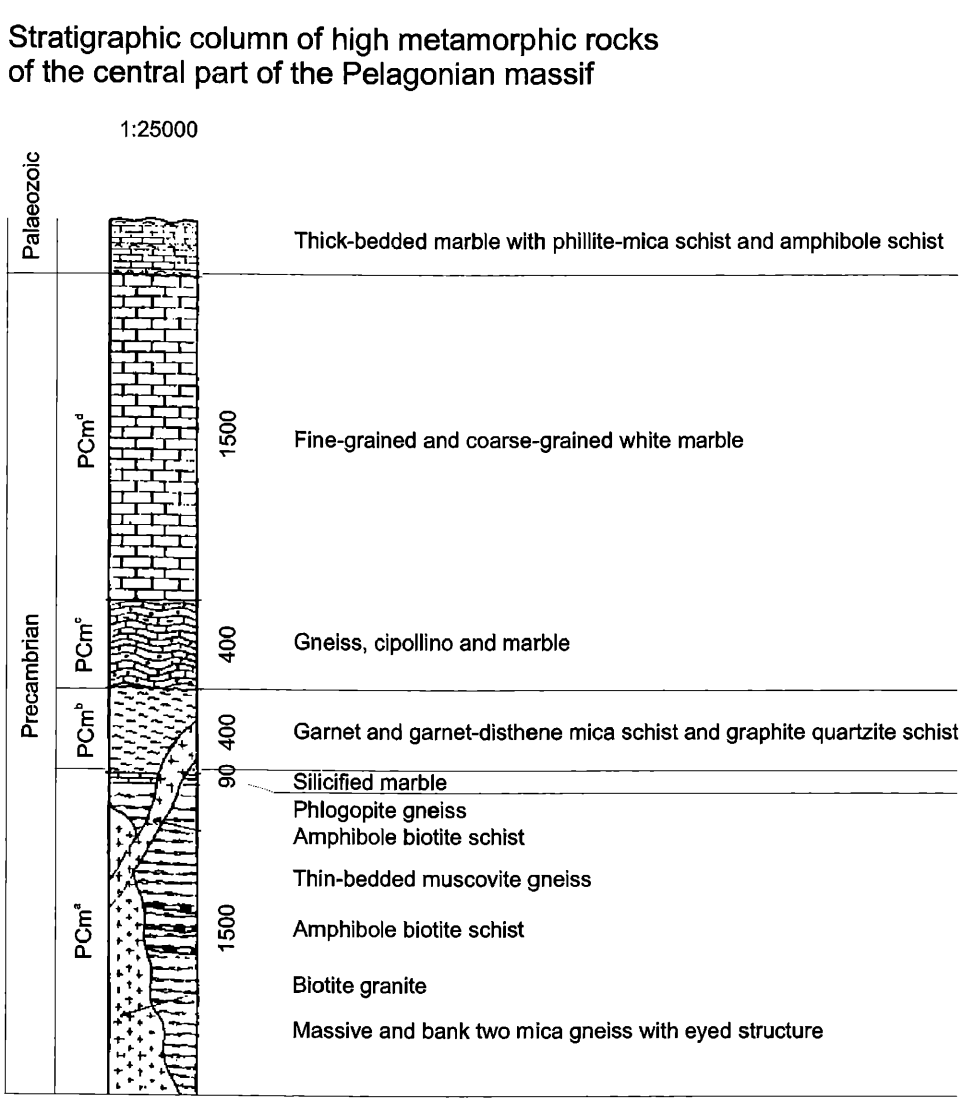
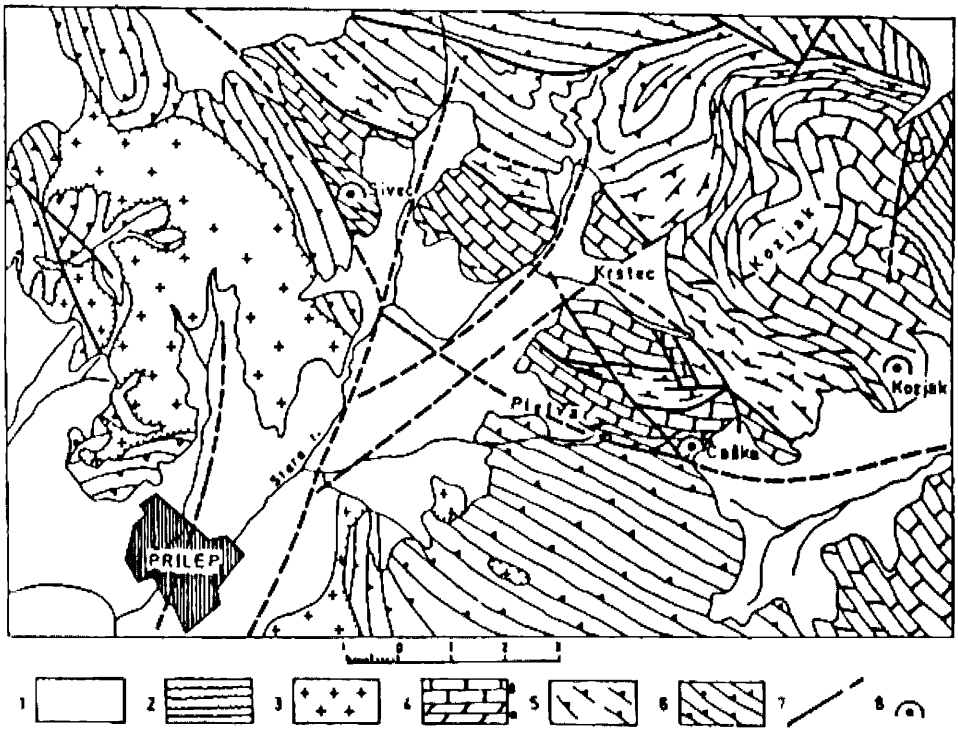


Figure 3: Stratigraphy of the highly metamorphic rocks of the central part of the Pelagonian massif (from STOJANOV, 1960)



Legend:
1. Neogene and Quaternary sediments
2. Precambrian metamorphic complex (phyllite, diabase, amphibole schist and marble)
3. granitic rocks
4. marble series (a-dolomite and calcite dolomite marble, b-calcite marble)
5. mixed series (albite gneiss, mica schist, marble and cipollino)
6. gneiss mica schist series (gneiss, mica schist, quartzite, amphibolite)
7. fault
8. marble quarry

Figure 4: Geological map of the central part of the Sivec-Pletvar-Kozjak area (from DUMURDJANOV *et al.*, 1990)

Material and Methods

About 100 kilograms of corundum crystals from dolomite marble deposits were studied. Specimens were divided according their morphology, structural homogeneity and colour. About 100 suitable corundum crystals were selected for further investigations. Crystals of corundum with dimensions from 0.5 to 12.8 cm along a-axis were investigated. They weighed from 5 to 300 gr.

The morphological forms of corundum crystals were determined by naked eye and by measuring crystal face angles with a reflection goniometer. X-ray diffraction of two-powdered samples was made to determine the crystal structure of uniform and non–uniform samples. Optical microscopic analyses were also performed on the same pieces. Some corundum crystals of gem quality were polished. Chemical composition was determined by non-destructive methods (Auger electron spectroscopy and X-ray fluorescence-XRF). A large specimen of ruby was investigated with Auger electron spectroscopy on four different parts with different colours (from pink, light pink to colourless). The chemical composition of eleven differently–coloured corundum samples was determined by XRF. All the corundum crystals were analysed with long and short wave ultraviolet light (253.7 nm and 365.4 nm). Gemstone quality corundum also was investigated with respect to specific gravity, refractive index and hardness. Specific gravity was measured with Westphal's balance, refractive index with a refractometer (Krüss) and hardness with durimet (a description of the protocol for measuring hardness with durimet was published by Leitz, 1964). Some specimens were cut to cabochons and facet forms.

Results and Discussion

Corundum crystals are covered with a thin layer of dolomite and/or brown–coloured iron hydroxides which does not allow observation of the actual colour and structure of corundum (Figs. 8 and 15). This layer was dissolved in 10% hydrochloric acid. Micas (muscovite, margarite), rutile and calcite crystals are intergrown with corundum crystals. Blue coloured disthen was found in dolomite marble in crystals measuring from 0.03 to 0.12 mm in length.

Morphological forms of corundum crystals from the Prilep marble

Corundum from Prilep marble shows several forms:

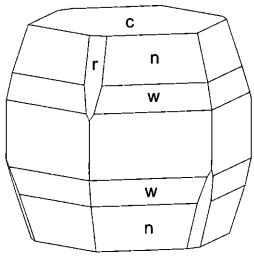
- crystals without developed crystal faces,
- crystals with developed crystal faces,
- twins,
- crystal intergrowth.

Corundum crystals without developed crystal faces

Crystals without developed crystal faces are quite rare. These crystals have the most homogeneous cores. Uniform and non–uniform corundum crystals and crystals with a well-developed cleavage were formed. According to their dimensions and colour such specimens are very suitable for cutting.

Crystals with developed crystal faces

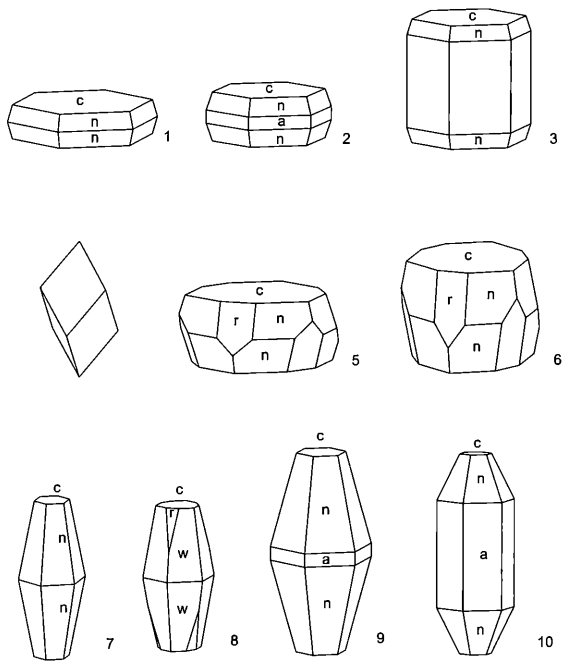
Most of the crystals are not perfect; this means that only one side or part of the crystal is developed. Intergrowth with diaspore is regularly seen in almost all crystal parts (Figs. 10, 13 and 16). Diaspore has a silvery glitter and a fatty lustre. Among the five crystallographic forms (Fig. 5) determined by reflection goniometry, the pinacoid and hexagonal bipyramids are the most common. Faces of hexagonal prism and rhombohedron are rare.



Legend:
pinacoid – **c** {0001}
second order prism – **a** {11$\bar{2}$0}
positive rhombohedron – **r** {10$\bar{1}$1}
hexagonal bipyramids – **n** {22$\bar{4}$3} and
w {11$\bar{2}$1}.

Figure 5: Typical morphology of corundum crystals from the Prilep marble

The most frequent crystallographic form is pinacoid. It was observed in 80% of all of the crystals studied. It is especially well developed in tabular crystals (Fig. 11), which is a typical form of corundum from Sivec (Fig. 6). The rarest of all are rhombohedral crystals (<1%), but prismatic and bipyramidal forms also occur occasionally (Figs. 12 and 13).



Legend:
c {0001} pinacoid
a {11$\bar{2}$0} second order prism
r {10$\bar{1}$1} positive rhombohedron
n {22$\bar{4}$3} and **w** {11$\bar{2}$1} hexagonal bipyramids

1, 2, 5 and 6 tabular crystals
3 and 10 prismatic crystal
7, 8 and 9 bipyramidal crystals
4 rhombohedral crystal with r faces.

Figure 6: Morphology of corundum crystals from Prilep

Twins

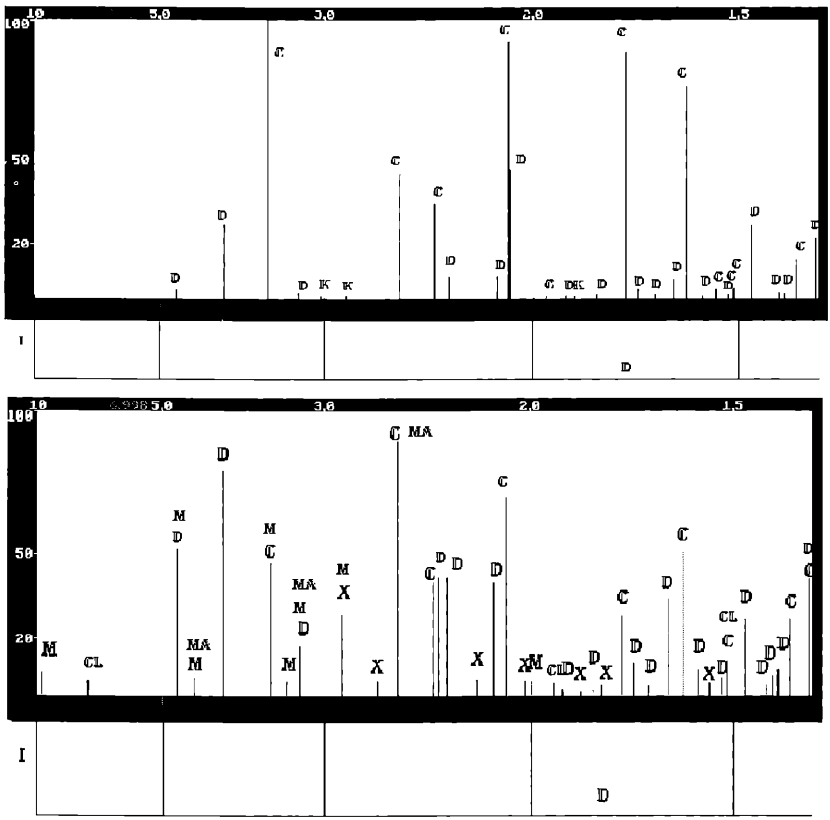
Contact twins on the pinacoid were found among the corundum crystals of Prilep marble. The twins comprise of a, n and c crystallographic forms.

Crystal intergrowths

Intergrown crystals are very common. Their intergrowth can be parallel (Fig. 18) or random (Fig. 19). Parallel-intergrowth crystals are in most cases tabular with developed c and n faces.

Solid inclusions in corundum crystals

Diaspore, calcite, dolomite, muscovite, margarite and chlorite are the most common minerals found among the inclusions in corundum crystals from Prilep. Only diaspore and calcite were determined as the inclusions by the X- ray diffraction of a powdered uniform sample. Besides diaspore some other included minerals were proved in the non–uniform corundum sample (Fig. 7).



Legend:
C – corundum, D – diaspore, K – calcite, X – dolomite, M – muscovite, Cl – chlorite, MA- margarite

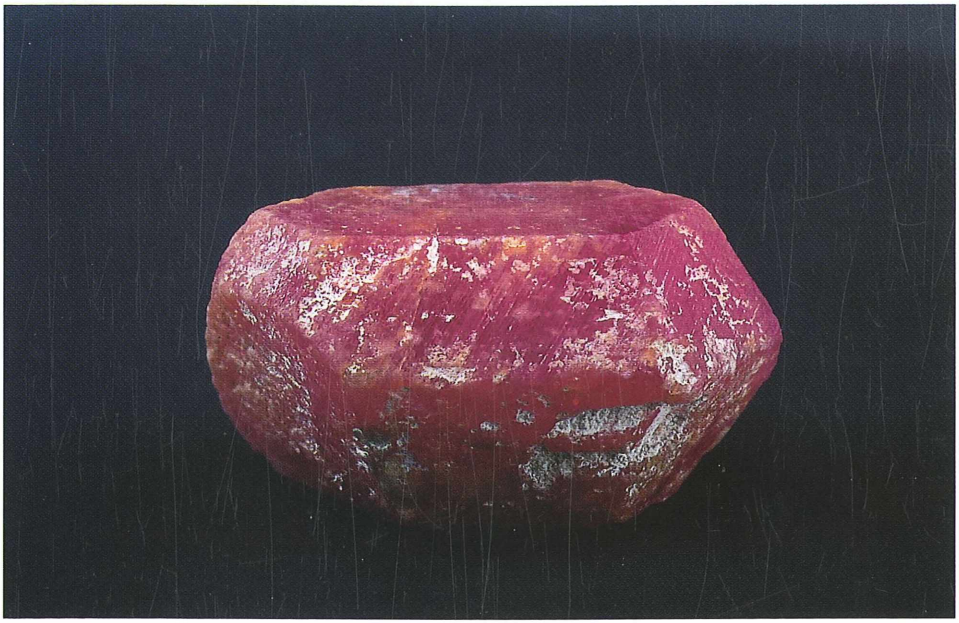
Figure 7: XRD of uniform and nonuniform sample of corundum crystal from Prilep marble

Figure 8: Corundum crystals are often covered with carbonate and iron hydroxide. The left sample is covered with white carbonate, the right with brown iron hydroxide. Dimensions of the right crystal are 2.4 x 1.8 cm.

Figure 9: Characteristically pink and red coloured corundum crystals. Prismatic crystal on the right is blue coloured and is rarely found in Prilep marble. Its height is 3.0 cm.

Figure 10: Intergrown corundum and diaspore crystals. Dimensions of the crystals group are 3.2 x 2.0 cm.

Figure 11: A characteristic tabular corundum crystal of dolomite marble from Prilep. Developed are also the faces of hexagonal bipyramid. The crystal is 1.5 cm high.



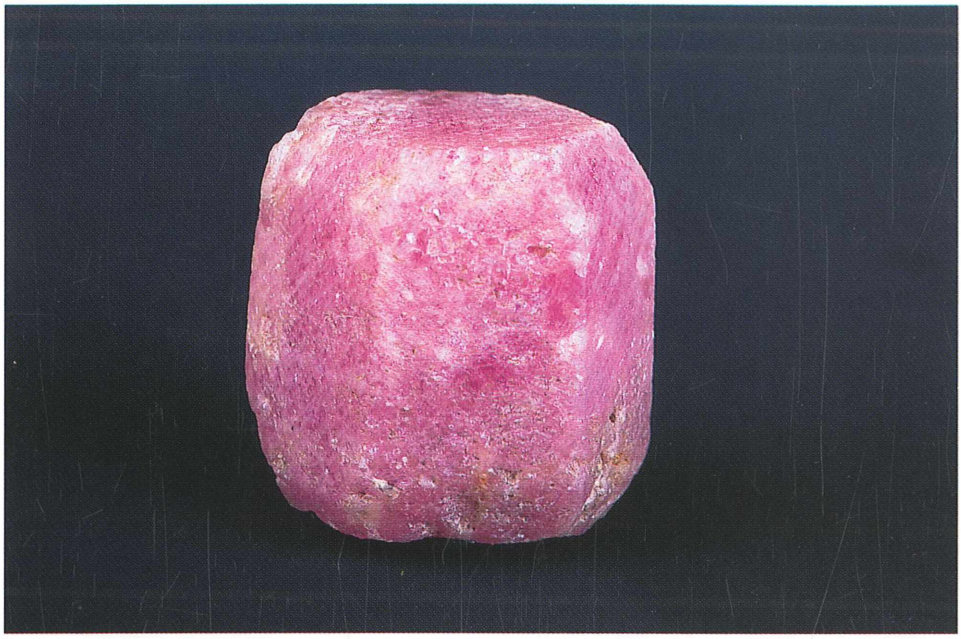
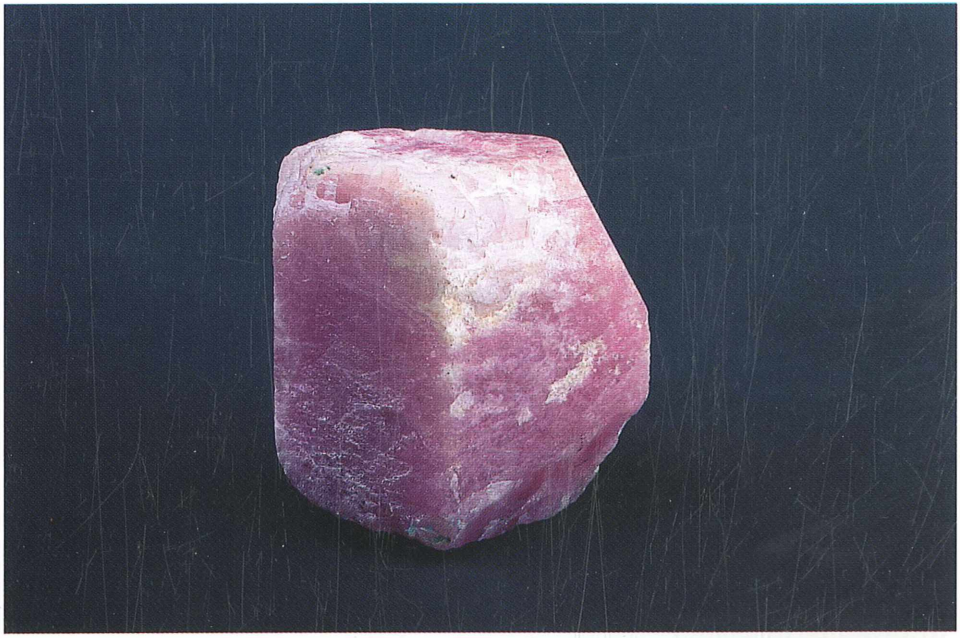


Figure 12: Prismatic corundum crystals from Prilep with developed faces of hexagonal bipyramid. Characteristic, pink coloured, the sample is 2.2 cm high.

Figure 13: Developed prismatic form at one side of the corundum crystal. Transition to bipyramidal morphology is seen on the other side. Oriented intergrowth of white or colourless diaspore is seen. The crystal height is 2.6 cm.



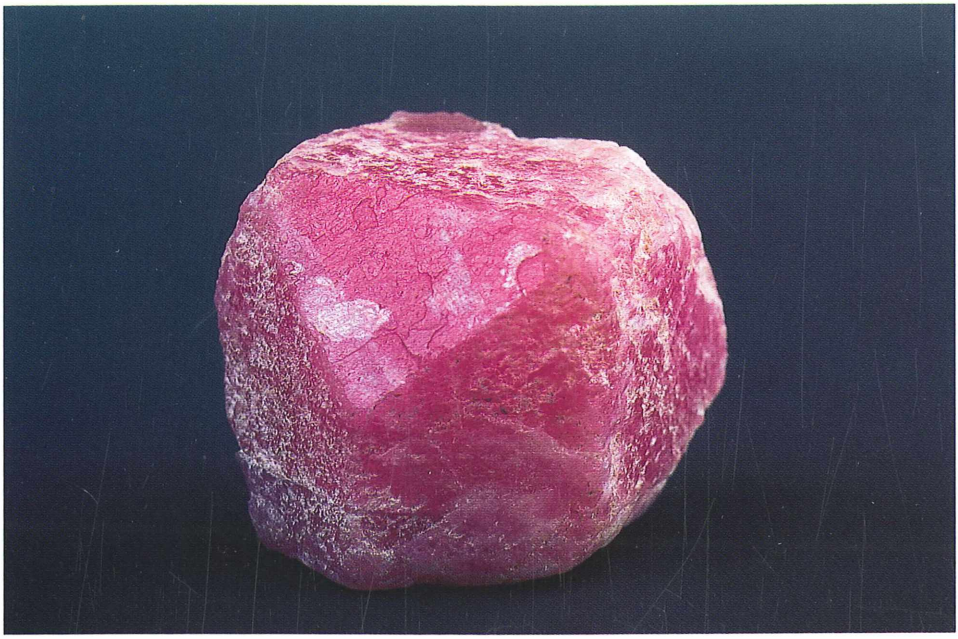
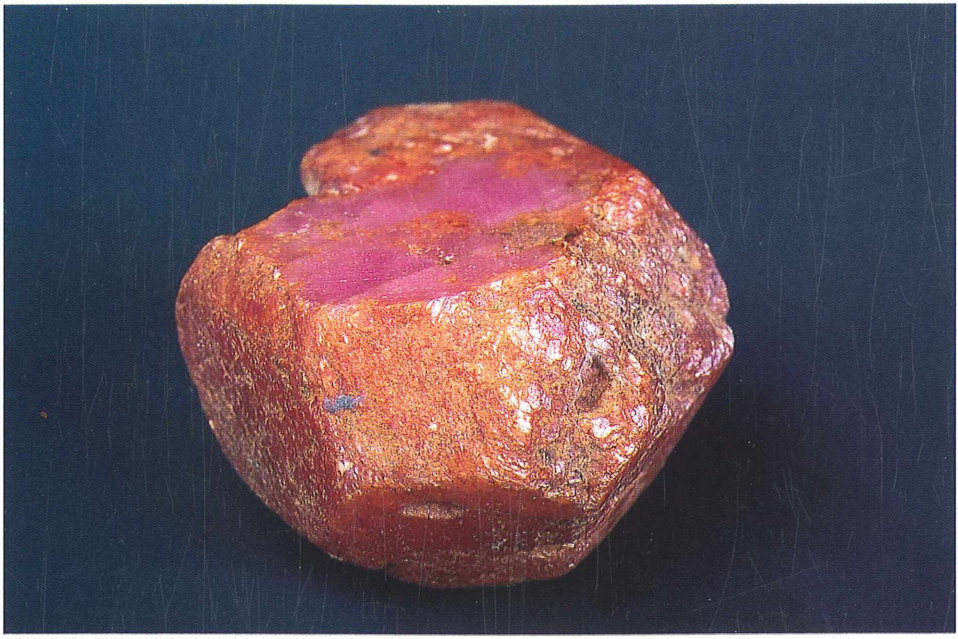


Figure 14: Prismatic form of corundum crystal with developed rhombohedrons faces. Lamellar intergrowth of diaspore and corundum crystals is seen in directions parallel to the rhombohedron faces. The sample is 1.5 cm high.

Figure 15: Rhombohedral corundum crystals are most rarely found in the Prilep marble. Rhombohedral corundum crystal is covered with iron hydroxide. Diaspore glittering can be seen on the rhombohedron faces. The sample is 2.2 cm high.



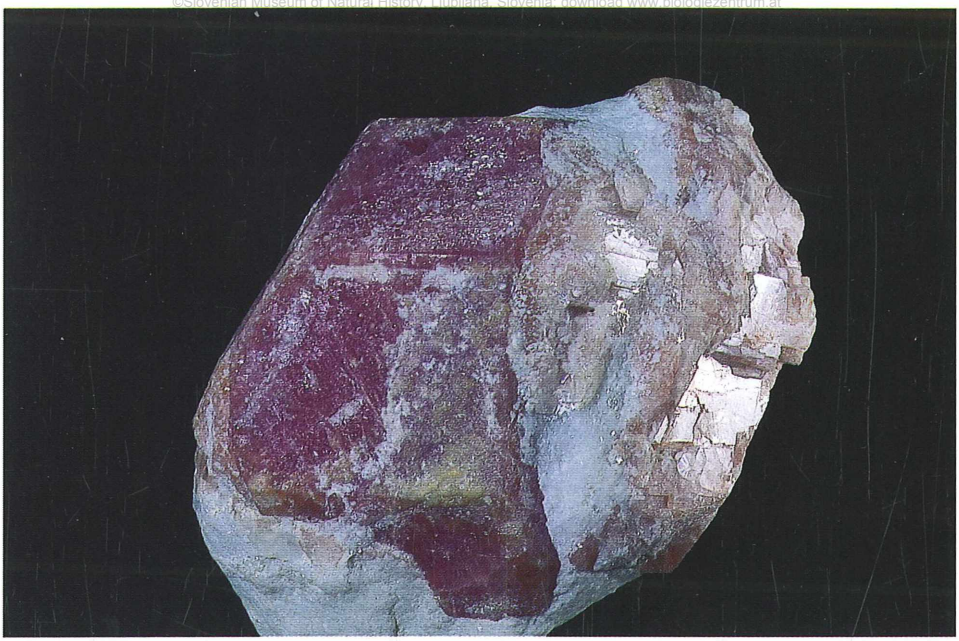


Figure 16: Right part of the corundum crystal is covered with translucent diaspore crystals up to two millimetres in diameter. The corundum crystal is 6.0 cm high.

Figure 17: One of the biggest corundum crystals from dolomite marble. The pinacoid diameter is 6 cm. Pseudohexagonal structure caused by oriented intergrowth of diaspore is seen on the left side.

Figure 19: Group of prismatic corundum crystals is 4.0 x 7.0 cm wide.

Figure 18: Intergrown crystals of intense pink coloured corundum. The crystal group is 3.5 cm high.

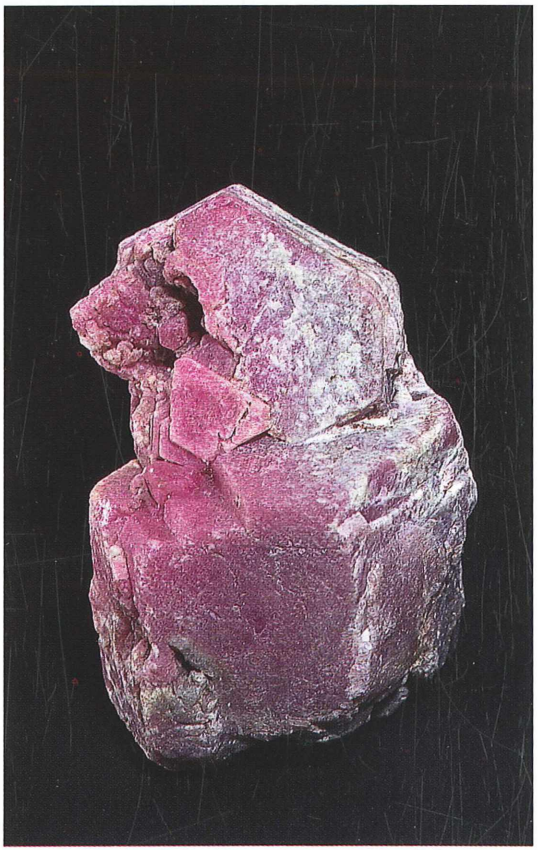


Figure 20: Diaspore crystals are very rarely seen in the dolomite marble. Two yellow–coloured diaspore crystals are seen in the figure. The left crystal is 0.5 cm high.

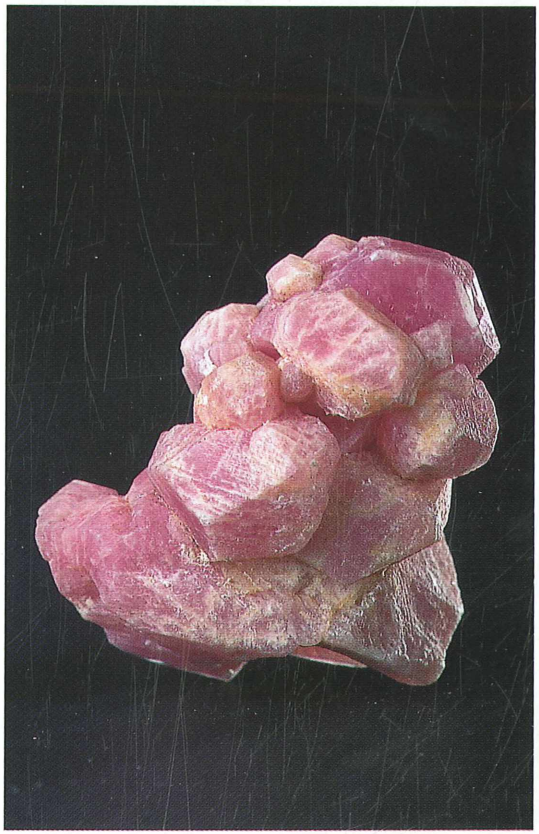


Figure 21: Intergrown corundum crystals are 4.0 cm high.

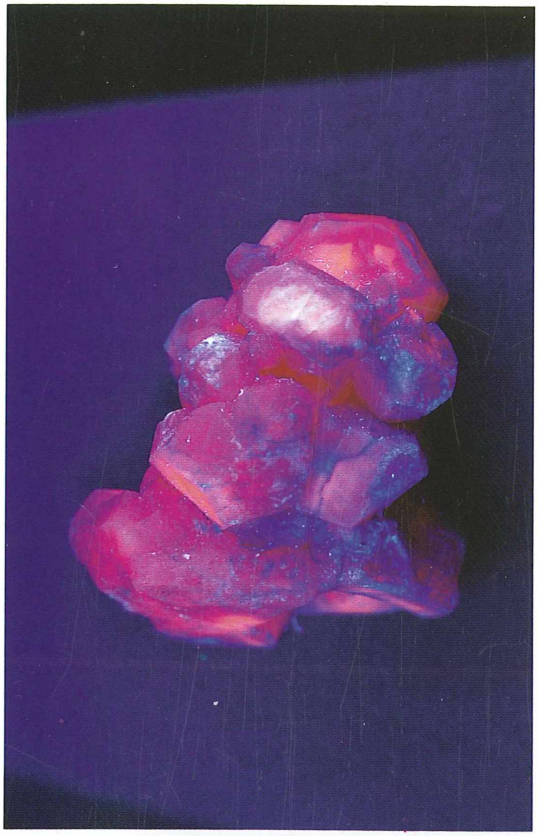


Figure 22: The same specimen as in Fig. 21 showing red and orange fluorescence colours under UV light.

Figure 23: Fluorescence in Prilep corundum unveils a sixfold symmetry of corundum. Crystal peaks with orange fluorescence colour are often detected. The outer part of the crystal is expressed in red coloured fluorescence. Dimensions of the crystal group are 3.5 x 3.0 cm.

Figure 24: Prilep corundum crystals are rarely of gem quality. They are used mostly for cabochon cutting. Three cabochon and one facet cut from corundum are seen. The largest sample has 17.35 carats.

Figure 25: Corundum of gem quality with vivid fluorescence colours. The same samples as in Figure 24 with intense red and orange fluorescence.

Figure 26: Lamellar intergrowth of diaspore detected as glittering lamellas – diasporescence in a Prilep ruby. The phenomenon is determined on cabochon cut parallel to rhombohedron faces. The ruby has 11.12 carat

The photographs show corundum crystals from the M. Jeršek and M. Kardelj collection. Jewellery design by N. Šturm. Photography by C. MIinar.

Pronounced oriented intergrowth of diaspore and corundum crystals crossing at 60° could be seen on the pinacoid faces (Fig. 17), bipyramids, hexagonal prisms (Fig. 13) and rhombohedron faces (Fig. 14). A pseudohexagonal structure of diaspore on the pinacoid of corundum crystal is frequently observed. Oriented intergrowth of diaspore and corundum causes a change in the corundum's physical properties. Because of the common twin lamellae and intergrowth of diaspore with corundum, the crystals do not show periodic darkening during movement under a polariscope.

Silvery glittering is seen depending on the orientation of the cut in the ruby, due to the parallel lamellae of the diaspore within. The silvery colour of reflected light can be seen in cabochon cut from homogeneous coloured ruby (Fig. 26). This glitter is due to the light reflected from the diaspore lamellae. Reflected light moves over the surface of ruby, depending on the angle of incident light and on the orientation of the diaspore lamellae. This phenomenon is unique in the world of gemstones and we have named it "diasporescence"

Diaspore intergrows in two ways: (1) by oriented intergrowth of corundum and diaspore – this can be observed on pinacoids, hexagonal prisms and also on hexagonal bipyramids, and (2) by lamellar intergrowth of diaspore – this can be seen on rhombohedron faces of corundum.

Hardness

The hardness of corundum from Prilep varies due to diaspore inclusions, and it generally is lower than corundum without diaspore intergrowth. On average, this means that the hardness is lower by about 10%.

Specific gravity

The specific gravity of the investigated Prilep corundum specimens is between 3.55 and 3.93.

Table 1. Measured specific gravity of different corundum crystals from Prilep

Sample designation	Specific gravity
1	**3.55**
2	**3.70**
3	**3.75**
4	**3.89**
5	**3.90**
6	**3.92**
7	**3.93**

Refractive index

Refractive indices No and Ne were measured on gemstone quality corundums: average values were 1.760 for No and 1.768 for Ne. Birefringence was 0.008.

Colour

The most common colour of corundum from the Prilep marble was light pink or pink violet, less common were orange, red and blue–coloured crystals (Fig. 9). If light pink is proposed as the basic colour of the corundum from Prilep, then varieties from light pink to intense pink, violet red and red and finally through to orange can be detected. Prilep corundum can also be grey, blue and (rarely) green coloured, and sometime white to colourless. However, intensely red–coloured corundum is rarely found in Prilep. Colour is a function of the chemical composition of impurities (Tab. 2 and 3).

Table 2. Amounts of aluminium and oxygen in four different coloured parts on polished corundum surfaces, in atomic % (investigated by Auger electron spectroscopy)

colour	measurement	X_{Al} [at.%]	X_O [at.%]	ϖ_{Al} [mas.%]	ϖ_O [mas.%]
violet	1	37.66	62.63	50.38	49.62
violet	2	36.59	63.41	49.24	50.76
light violet	3	36.74	63.26	49.40	50.60
light violet	4	35.08	64.92	47.60	52.40
	average	**36.52**	**63.55**	**49.15**	**50.85**

Legend:

X_{Al} [at.%].....atomic per cent of aluminium in compound
X_O [at.%].....atomic per cent of oxygen in compound
v_{Al} [mas.%].....mass share of aluminium in compound
v_O [mas.%].....mass share of oxygen in compound

The amount of aluminium decreases from 37.66 at.% in the violet sample to 35.08 at.% in the light violet sample. Calculation of the molar composition of the crystal parts investigated shows that in the violet coloured sample corundum and diaspore can be found in equal amounts, however, only diaspore can be detected in the lighter part. The chemical composition of ten different coloured corundum samples were determined by non-destructive XRF (the results are shown in table 3).

Table 3. Amount of trace elements in corundum from Prilep, in weight % × 10^{-3}

Sample designation	Cr	V	Fe	Ti	Ga	Ba	K	Zn	Pb	Ge	Cu
15	1.00	3.80	x	2.80	2,11	4.77	61.00	x	x	x	x
20	74.80	3.05	15,70	n.d.	2,95	x	x	2.67	0.13		9.10
14	29.00	4.91	15,80	n.d.	3,70	x	x	3.03	1.43	0.17	9.70
18	51.40	n.d.	25,30	x	4,89	482.00	125.00	4.59	3.30	x	14.50
16	22.70	n.d.	13,00	1.26	2,01	x	x	2.71	1 10	x	8.00
17	35.840	2.28	14,30	1.68	3,44	x	x	2.89	0.85	x	8.40
19	50.10	5.82	11,60	10.50	3,01	47.30	43.90	1.89	0.67	x	5.80
22	66.00	1.97	11,80	3.91	2,41	9.63	n.d.	2.00	0.69	0.20	5.80
21	267.00	n.d.	17,90	0.69	x	n.d.	8.17	3.48	1.41	0.30	11.00
23	x	2.52	x	2.90	6,25	4.29	48.60	x	x	x	x
24	7.90	2.11	x	1.32	2,69	5.38	24.70	x	x	x	x

Legend:
n.d. = not detectable
x = not present

Chromium content is highest in intense violet red coloured samples (20, 22), attaining up to 75.10^{-3} wt.%. The lowest amounts were found in pale to light pink coloured corundum crystals (sample 23). The intensity of colour (pink, red or violet) corresponds to the amount of chromium (samples 14, 16 and 17). Iron concentration is below the limits of detection in samples 23, 15 and 24 with a low amount of chromium. Other samples with chromium contain higher amounts of iron (between 11.10^{-3} and 25.10^{-3} wt.%).

The amount of vanadium varies by up to 6.10^{-3} wt.%. Vanadium influences the intensity of violet colour. The average amount of titanium does not exceed the value of 4.10^{-3} wt.%. However, it is characteristically high in sample 19, which is intensely blue–coloured.

Traces of gallium are characteristic of Prilep corundum. Other trace elements are Zr, Sr, Ni and U.

Fluorescence of corundum crystals from Prilep

The Prilep corundum crystals investigated emit three different wavelengths of fluorescence spectrum after the incidence of longwave and shortwave ultraviolet light, *i.e.* yellow, orange and red. We found several crystals of corundum which showed two-colour fluorescence (Fig. 21 and 22). Distribution of this two-colour fluorescence could prove the six–fold symmetry of the different coloured zones (Fig. 23). The yellow fluorescence is always present at the top or in the centre of crystals but is observed very rarely.

The other mineral which has an intense red fluorescence is probably spinel. Green mica (margarite) emits white to light green fluorescence colours and is intergrown with corundum.

Conclusion

Many different mineral inclusions were observed in corundum crystals from the dolomitic marble from Prilep. Intergrowth of diaspore with corundum affects the physical properties of corundum, which is important in the classification of the gemstone. Prilep corundum has a lower hardness than that from other deposits. Chromophore elements included in corundum exert an influence upon colour. The most frequent colour is pink, but intense pink, violet pink and red violet are seen occasionally. Blue and green corundum are very rare.

Corundum from Prilep is transparent and could be used as a gemstone, cabochon being the most common cut form. Exceptionally, some corundum specimens can be totally transparent and can be cut to facet forms. The cleavage of corundum parallel to the rhombohedron faces results from the lamellar intergrowth of diaspore and corundum.

Transparent and coloured types of corundum show a silvery glittering because of the lamellar intergrowth of diaspore under rhombohedron faces. Transparent and clear–coloured rubies are cut perpendicular to the rhombohedron faces to emphasise this phenomenon. The uniform silvery–white reflection of diaspore, which travels in dependence upon incident light and diaspore orientation, can be seen in reflected light. This phenomenon, that we name "diasporescence", is unique in the world of gemstones. If transparent corundum of gemstone quality is cut to cabochon forms, then the diasporescence is stressed. A silvery reflection also can be seen in facet forms.

Corundum from Prilep appears in a wide number of morphological forms, as shown by the investigation of more than 100 kilos of corundum crystals. The indices of crystal faces were determined by reflection goniometer. The crystallographic forms of corundum crystal from Prilep dolomitic marble described by Barić (1969; 1977) were seen in this study as well. Barić (1969), Schiffmann (1974) and Žorž (1991) determined tabular and prismatic forms. The bipyramidal and rhombohedral forms described by Schmetzer (1986) as being characteristic of corundum were also observed. The contact twins on pinacoid are described for the first time from Prilep corundum crystals. Intergrown crystals, with a large number of crystallographic forms, are relatively common.

Diaspore intergrowth with corundum influences the crystal physical properties such as hardness, density and colour. Specific gravity is low in comparison to Schiffman's measurements (1974); the intergrowth of diaspore results in a specific gravity ranging from 3.3 – 3.5. Higher amounts of diaspore cause a lower specific gravity and lower intensity of pink colour.

The corundum from Prilep dolomitic marble shows red, orange and yellow fluorescence. Yellow fluorescence is described here for the first time. Comparison of chemical composition and fluorescence colour shows that an increased amount of chromium causes the red fluorescence. Blue–coloured corundum does not show fluorescence. All fluorescence colours are more intense at longwave than shortwave UV light. Many of the investigated crystals show a two coloured fluorescence, which proves the six–fold symmetry of the differently coloured zones, as previously observed by Schiffman (1974).

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