

OCCURRENCE AND MINERALOGICAL CHARACTERIZATION OF SPECULARITE IN DAMJAN ORE DEPOSIT, NORTH MACEDONIA

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A b s t r a c t: In this paper are presented occurrence and mineralogical characterization of specularite from Damjan ore deposit. The straightforward identification of the studied mineral sample was enabled by SEM-EDS, XRF and XRPD methods. With these methods it can be seen that the phases present are calcite, specularite and quartz. Specularite crystals occur in the surface parts of the deposit. Specularite has a metallic gray to silvery appearance with a glittering luster and reddish streak. It shows a very smooth surface with reflection. The metallic luster is a result of the specific crystal formation of hematite. Manganese and aluminium substitute for Fe^{3+} in the specularite structure. Small amounts of Fe^{2+} and Mn^{2+} substitute for Ca^{2+} in the crystal lattice on calcite. The well-developed morphology of specularite suggests that it crystallized first. The finer-grained calcite likely formed later, filling the interstitial spaces between the earlier-formed specularite.

Key words: specularite; calcite; quartz; Damjan

INTRODUCTION

The Damjan ore deposit is located on the right side of the regional road between Štip and Radoviš, approximately 11 km west of Radovish and 25 km east of Štip. It is located in the central part of the mining region Buchim-Damjan-Borov dol. It is one of the rare skarn deposits that formed at the contact between andesite and Paleogene flysch.

It extends over a length of approximately 800 meters and consists of several ore zones. The ore bodies are lens-shaped and elongated, with variable thicknesses ranging from a few centimeters up to 5 meters.

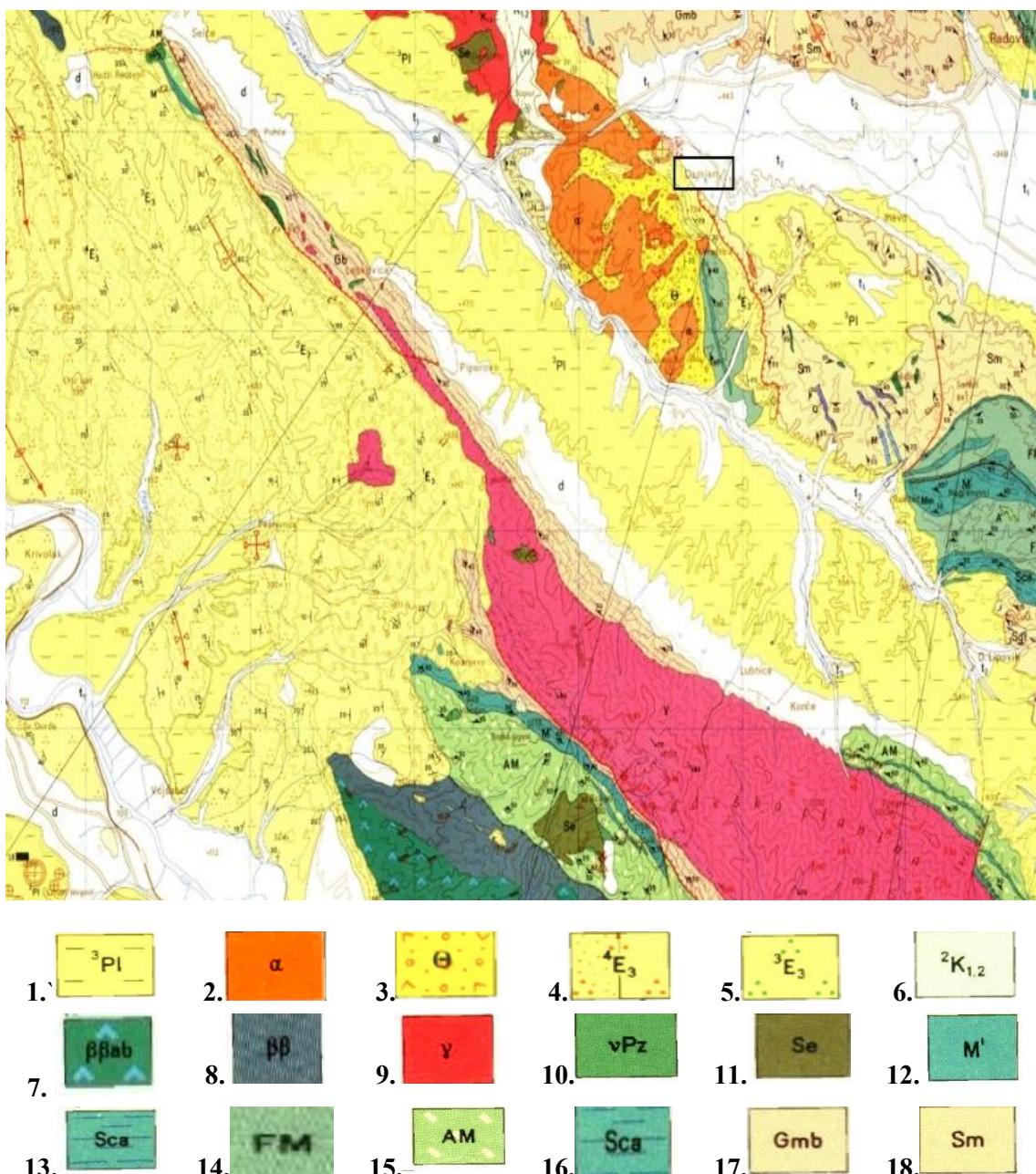
Geological investigations have identified the presence of three main rock types in the area: Paleogene sediments, Tertiary volcanic rocks and skarns.

Most of the deposit area is composed of Paleogene flysch sediments. In the central and southwestern parts, these sediments are intruded by Tertiary magmatic products. Additionally, andesite outcrops from younger volcanic phases cover a significant portion of the region. At the contact

between the volcanic rocks and the flysch sediments, an extensive skarn zone has developed, characterized by significant Fe (iron) mineralization. Parts of the flysch have undergone hydrothermal alteration, particularly sericitization and silicification. A large number of researchers participated in the study of the geological structure and metallogenetic characteristics of the Damjan deposit, including: Dolenc (1959); Ivanov (1966, 1982), Ivanov and Veličković, (1976); Zarić (1977); Serafimovski (1982, 1986, 1993).

Geological map of the Damjan is given on Figure 1.

According to Ivanov (1965, 1982), Petković (1968) and Janković (1967, 1972), the Damjan deposit is classified as a skarn-type deposit. Dolenc (1959) described it as a contact-pneumatolytic hydrothermal metasomatic deposit, while Zarić (1977) considered it a high-temperature, selective metasomatic type.



Legend: 1. sandy series; 2. andesite; 3. piroclastite; 4. yellow sands (upper zone of the flysch); 5. over yellow sandstones; 6. sandstone, marls, clays, limestone; 7. spilites; 8. diabase; 9. granites; 10. gabbro; 11. serpentinites; 12. marble; 13. carbonate shists; 14. carbonate sediments; 15. carbonate schists; 16. amphibole schists with marbles; 17. two-mica gneiss; 18. micaschists

Fig. 1. Geological map of Damjan ore deposit (Basic Geological Map, sheet Kavadarci, R 1:100 000)

The paragenetic relationships within the Damjan deposit are notably complex. The formation of individual minerals and mineral assemblages occurred through a multi-stage genetic process, with mineral components precipitating in three distinct environments: carbonate, silicate, and predominantly mixed silicate–carbonate.

A detailed mineralogical characterization was provided by Ivanov (1965) and Zarić (1977), with

particular emphasis on the role of magnetite in the ore genesis. Based on previous investigations, the ore minerals identified in the Damjan deposit include:

- *iron oxides and related minerals*: magnetite, hematite, specularite;
- *titanium and iron minerals*: ilmenite, rutile;
- *sulfides and other metallic minerals*: galena, pyrite, pyrrhotite, bornite, covellite, gold;

- *secondary and oxidized minerals*: cerussite, malachite, pyrolusite, goethite;
- *other minerals*: jacobsite.

The non-ore minerals include: orthoclase, plagioclase, epidote, chlorite, calcite, apatite, quartz and biotite.

Among the ore minerals, iron oxides are the most abundant, with magnetite and hematite–specularite being the dominant phases. The spatial distribution and abundance of these oxides vary signif-

icantly across different ore bodies. Magnetite content generally increases with depth.

Isolated magnetite concentrations are typically found in zones formerly composed of carbonate rocks, where metasomatic replacement of limestone occurred during the early stages of ore formation. Mixed magnetite–hematite assemblages are primarily found in carbonate–silicate zones, while the former silicate-dominated zones are characterized by specularite, often accompanied by microcrystalline hematite.

ANALYTICAL METHODS

To ensure a reliable characterization of the mineral species in this study, the following analytical methods were used: Fourier Transform Infrared Spectroscopy using the diffuse reflectance method (FTIR/DRS), Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X-ray Spectroscopy (EDS), and X-ray Powder Diffraction (XRPD).

Scanning electron microscopy SEM-EDS

Analyses and electron micrographs were obtained using a VEGA3 LMU scanning electron microscope, with magnifications up to $2 \times 1,000,000$. Semi-quantitative chemical analyses were performed using appropriate standards, as listed below: O: SiO₂; Na: Albite; Mg: MgO; Al: Al₂O₃; Si: SiO₂; P: GaP; Ca: wollastonite; Ti: Ti; Fe: Fe; Br: KBr.

Prior to analysis, the samples were sputter-coated with a thin layer of gold to ensure surface conductivity for both SEM imaging and EDS analysis.

Infrared spectroscopy (FTIR/DRS)

Infrared analyses were performed using an IR-Prestige-21 spectrophotometer (Shimadzu, Japan). Measurements were carried out by diffuse reflectance spectroscopy (DRS) using the DRS-8000 attachment (Shimadzu, Japan). The powdered sample

was diluted to approximately 5 wt.% with KBr and placed into sample holders (2 mm in diameter and 1 mm in depth).

Spectra were recorded in the range of 400–4000 cm⁻¹, with a resolution of 2 cm⁻¹ and 62 scans. Data processing and spectral manipulation were conducted using IR Solution 1.5 software (Shimadzu Corporation). Second-derivative spectra were also generated to enhance band resolution and to identify overlapping absorption features.

X-ray powder diffraction (XRPD)

The samples were finely ground using an agate mortar and pestle, then manually packed into cylindrical standard sample holders (16 mm diameter \times 2.5 mm height) to achieve a flat surface for analysis. Diffraction patterns were collected using a Shimadzu XRD-6100 diffractometer with Cu K α radiation ($\lambda = 1.54060$ Å), operating at 40 kV and 30 mA. Scanning was performed over the 5–80° 2 θ range, with a step size of 0.02° and a scanning speed of 1.2°/min. The powdered samples were homogenized, and their average bulk composition was determined. The most intense peaks observed in the diffractograms were identified by comparison with reference patterns from the Match 4 software database.

RESULTS AND DISCUSSION

Specularite was first discovered in Europe around the year 1620. It was named by French geologist Pierre Sonnerat for its reflective properties and allusion to the specular ("mirror-like") appearance of this type of hematite. Specularite is a variety of hematite characterized by aggregates of silvery,

metallic, specular ("mirror-like") hematite flakes or tabular, anhedral crystals. It is a member of the hematite-goethite mineral group, which also includes maghemite and akaganeite. Macroscopic characteristics on the specularite from Damjan are given in Figure 2.

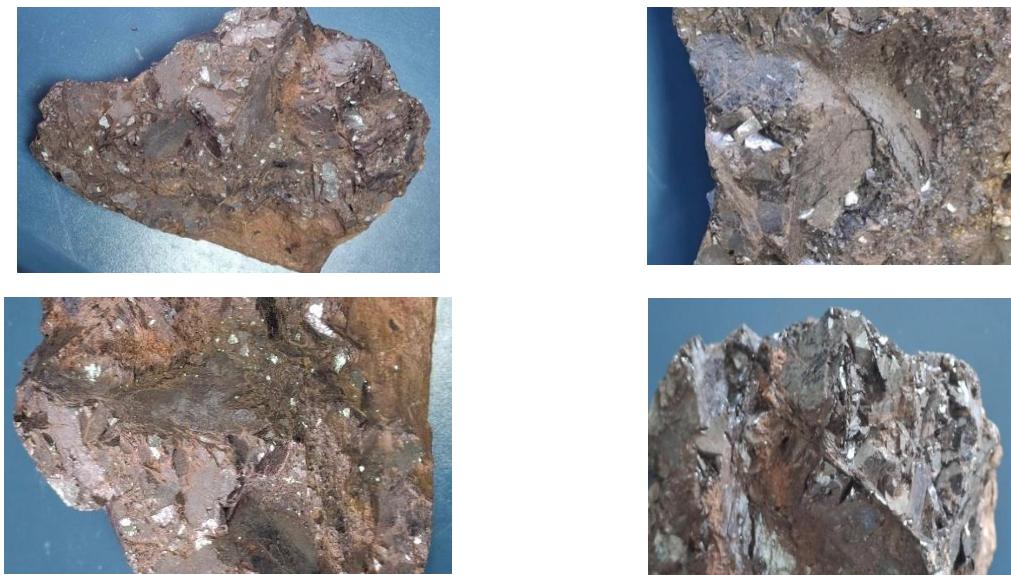


Fig. 2. Microscopic features on specularite from Damjan

Specularite has a metallic gray to silvery appearance with a glittering luster and reddish streak, displaying a very smooth surface with reflection. The metallic luster is a result of the specific crystal formation of hematite. It has a hardness rating of 5 to 6 on the Mohs scale.

SEM image and EDX spectrum of specularite from Damjan ore deposit are given in Figure 3.

Figure 3a shows a SEM image and EDS spectrum of specularite. Figure 3b displays a well-defined crystalline structures of specularite. The crystals appear, more or less aligned in specific directions, forming a distinct layered pattern. The lamellar or striated texture within the grains is characteristic of specularite, which typically forms platy or tabular crystals. The parallel crystal planes and layering indicate mineral growth under conditions favorable for distinct cleavage – typical of specularite.

Figures 3c and 3d show the presence of calcite. Small amounts of Fe^{2+} and Mn^{2+} substitute for Ca^{2+} in the calcite crystal lattice. However, the Mn and

Fe content is too low to form separate Mn-oxide or Fe-oxide phases.

Figure 3e shows a SEM image and EDS spectrum capturing both specularite and calcite. The spectrum was acquired across the interface or inclusion zone, thereby detecting both minerals in a single analysis. The well-developed morphology of specularite suggests that it crystallized first. The finer-grained calcite likely formed later, filling the interstitial spaces between the earlier-formed Fe-rich crystals. The EDS spectrum displays a mixed signal, typical of contact zones between intergrown specularite and calcite.

Figure 3f indicates that Mn and Al substitute for Fe^{3+} in the structure of specularite. The presence of Si is most likely due to quartz or clay mineral inclusions, while Ca likely corresponds to calcite or an associated alteration mineral.

A SEM image and EDS spectrum of quartz are presented in Figure 3g.

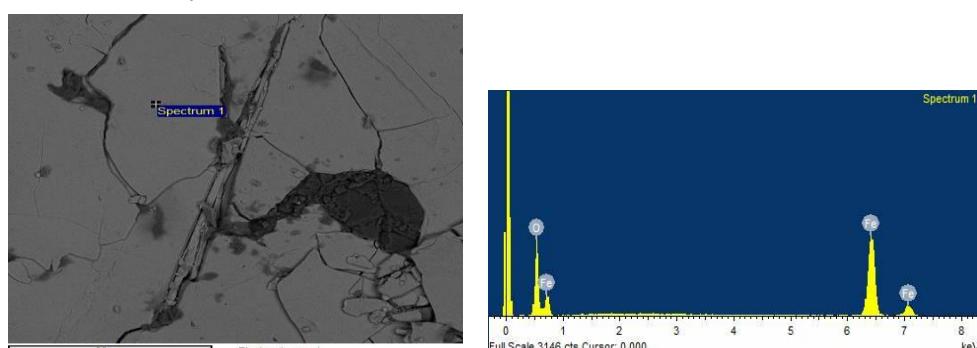


Figure 3a

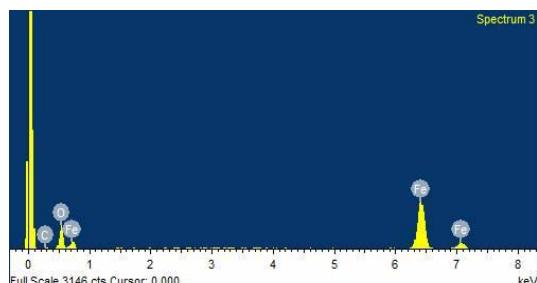


Figure 3b

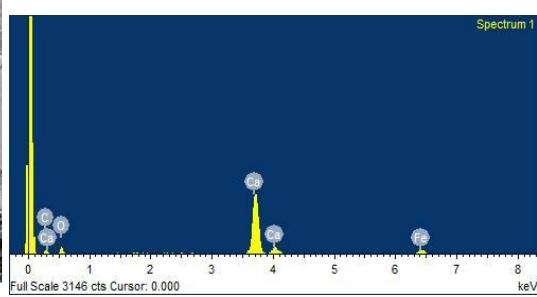


Figure 3c

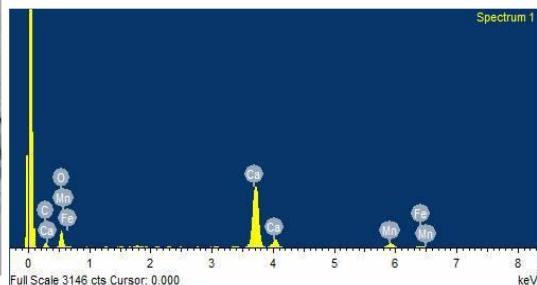
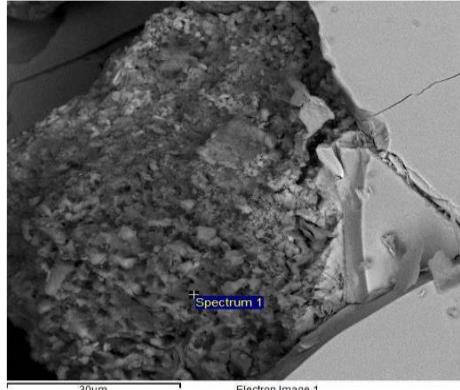


Figure 3d

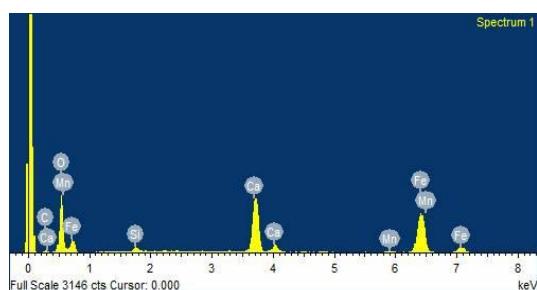
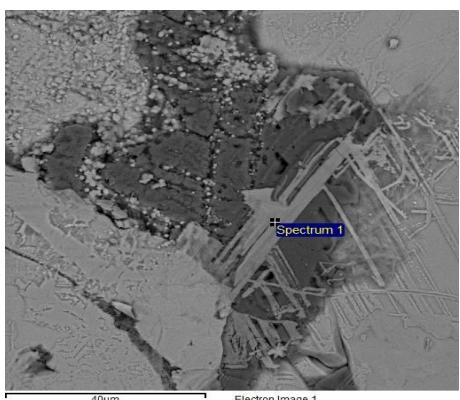


Figure 3e

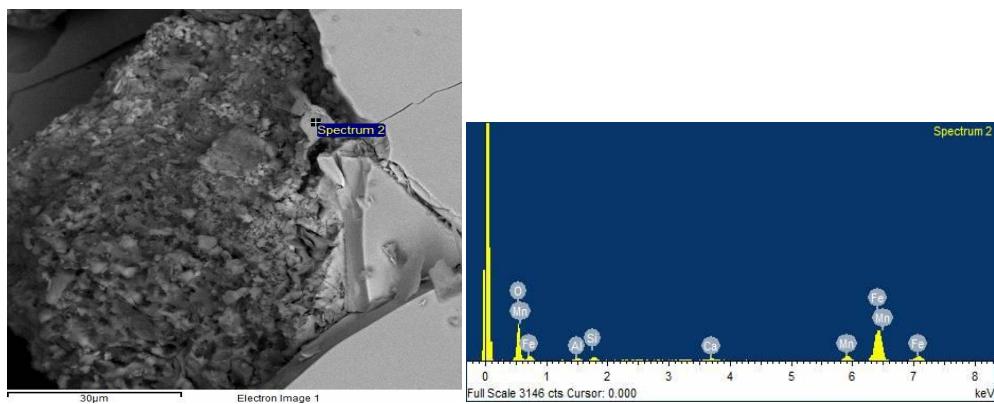


Figure 3f

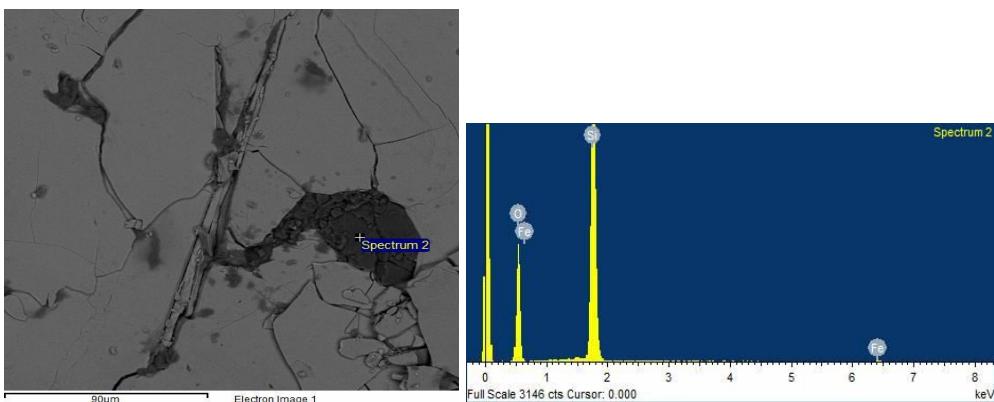


Figure 3g

Fig. 3. SEM image and EDX spectrum of specularite, calcite, and quartz from Damjan ore deposit

The IR spectrum of the analyzed sample (Figure 4, Table 1) reveals a mixture of mineral phases, including calcite, silicate components (phyllosilicates, quartz), and hematite. The dominant phase is calcite, in agreement with the XRD results. Calcite is clearly identified by the diagnostic absorption bands of the CO_3^{2-} ion at 1440 cm^{-1} (ν_3), 877 cm^{-1} (ν_2), and 712 cm^{-1} (ν_4) (Makreski et al., 2004; So et al., 2020). Additional combination bands at 2512 cm^{-1} and 1795 cm^{-1} further confirm its significant presence.

A sharp absorption band at 3626 cm^{-1} indicates the presence of hydroxyl-bearing silicates. This band, along with those at 1029 cm^{-1} (Si–O stretching) and 908 cm^{-1} (Al–O–H bending), corresponds to hydroxylated aluminosilicates, most likely phyllosilicates (clay minerals) (Madejová et al., 2017; Shontevska et al., 2008). Additional absorption at 799 cm^{-1} , assigned to symmetric Si–O–Si stretching vibrations in quartz (SiO_2), confirms the presence of silicate components.

Broad features between 3185 and 3200 cm^{-1} and a weak band at 1665 cm^{-1} , typically associated

with molecular water, suggest that the clay minerals contain adsorbed and/or interlayer water.

Previous studies indicate that hematite ($\alpha\text{-Fe}_2\text{O}_3$) exhibits two main Fe–O lattice vibration bands, typically observed at 540 – 570 cm^{-1} and 450 – 480 cm^{-1} (Rendon and Serna, 1981; Ruan et al., 2002; Boev et al., 2013; Chukanov, 2014). These bands lie in the low-wavenumber region (600 – 400 cm^{-1}), which overlaps with the Si–O–Si bending modes of silicates, making IR identification of hematite challenging due to spectral overlap.

As shown in Figure 4b, second-derivative processing of this spectral region successfully resolved several overlapping features. Absorptions at 585 cm^{-1} , 562 cm^{-1} , and 536 cm^{-1} are consistent with Fe–O lattice vibrations typical of hematite, while the strong band at 470 – 480 cm^{-1} corresponds to Si–O–Si bending in silicate minerals.

Additionally, weak absorption bands at 2985 cm^{-1} and 2875 cm^{-1} , corresponding to C–H stretching vibrations, indicate the presence of trace amounts of organic matter.

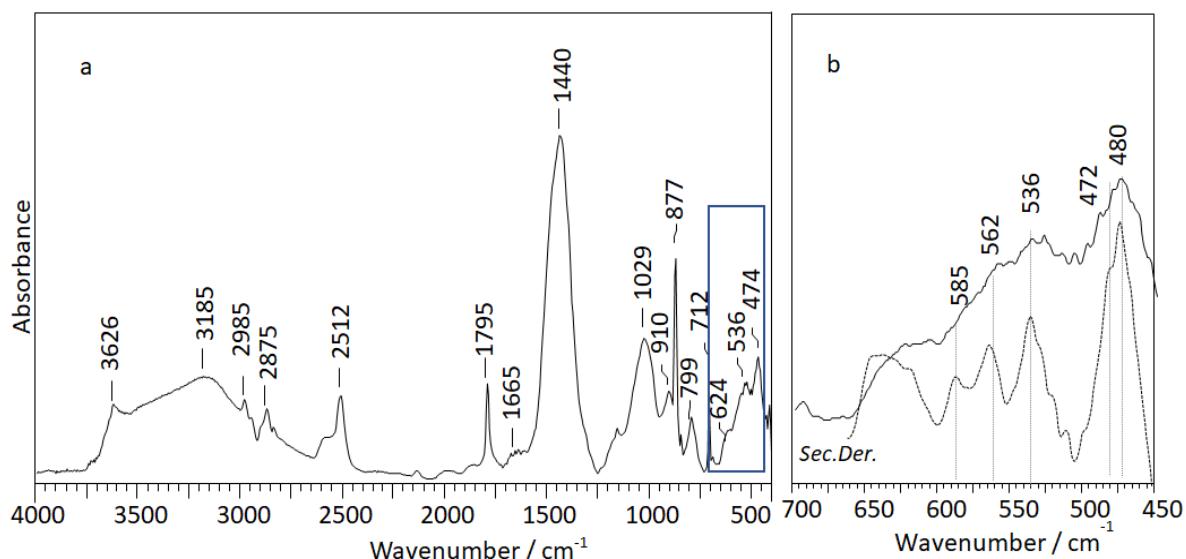


Fig. 4. IR spectra of the analyzed mineral sample. (a) Original IR spectrum; (b) Low-wavenumber region ($600\text{--}400\text{ cm}^{-1}$) with the corresponding second-derivative curve.

Table 1

Summary of the main IR absorption bands and their vibrational assignments for the analyzed mineral sample. Band assignments were made according to reference IR data for the respective mineral phases*

IR / cm^{-1}	Description of vibration mode	Tentative assignment
3626	$\nu(\text{OH})$ structural hydroxyl	Phyllosilicate (clay, mica)
~3200	$\nu(\text{OH})$ H_2O stretching	Interlayer/adsorbed water
2985–2875	$\nu(\text{CH})$ stretching	Organic matter (trace)
2512	$\nu_1+\nu_3$ (CO_3^{2-}) combination	Calcite (CaCO_3)
1795	$\nu_1+\nu_4$ (CO_3^{2-}) combination	Calcite (CaCO_3)
1665	$\delta(\text{H-O-H})$ bending	Molecular/interlayer water
1440	$\nu_3(\text{CO}_3^{2-})$ asymmetric stretch	Calcite (CaCO_3)
1029	$\nu_{\text{as}}(\text{Si-O-Si})$ asymmetric stretch	Phyllosilicate (clay)
908	$\delta(\text{Al-O-H})$ bending	Phyllosilicate (clay)
877	$\nu_2(\text{CO}_3^{2-})$ out-of-plane bending	Calcite (CaCO_3)
799	$\nu_{\text{s}}(\text{Si-O-Si})$ symmetric stretch	Quartz (SiO_2)
712	$\nu_4(\text{CO}_3^{2-})$ in-plane bending	Calcite (CaCO_3)
536, 562	$\nu(\text{Fe-O})$ lattice vibration	Hematite (Fe_2O_3)
472, 489	$\nu(\text{Fe-O}) / \delta(\text{Si-O-Si/Al})$ bending	Hematite/Silicate

*Chukanov, 2014; Madejová, 2017; Makreski, 2004; So, 2020; Rendon, 1981; Ruan, 2002; Shontevska, 2008

XRPD patterns of the examined specularite from Damjan are given in Figure 5.

From the XRPD diagram, it can be seen that the present phases are calcite, specularite, and quartz.

There is no fundamental difference in the X-ray diffraction (XRD) patterns of hematite and specularite because both minerals have the same crystal structure and chemical composition. Hematite and

specularite are both Fe_2O_3 with a trigonal crystal structure (rhombohedral, R-3c).

Since specularite is simply a highly crystalline variety of hematite, its diffraction peaks align exactly with standard hematite XRD patterns. Being highly crystalline, specularite may show sharper, more intense peaks due to its larger, well-ordered grains. In contrast, more massive, earthy, or fine-grained hematite often shows broader peaks due to smaller crystal sizes.

Specularite typically forms in oxidizing environments, especially during the alteration of iron-rich minerals or precipitation from hydrothermal fluids. Specularite and calcite minerals can form together under the following conditions:

- A hydrothermal system rich in both iron and calcium;
- Suitable pH and redox conditions that allow both iron oxidation (forming hematite) and carbonate stability (allowing calcite formation);
- Availability of space in veins or cavities for crystallization.

Specularite in the Damjan deposit occurs in multiple generations. The first generation, characterized as high-temperature specularite, appears as well-developed crystals. The second generation of specularite typically occurs in the interstices of compact magnetite and usually forms in paragenesis with second-generation magnetite (Serafimovski, 1993).

The presence of manganese (Mn) has been detected in specularite. Manganese could be present

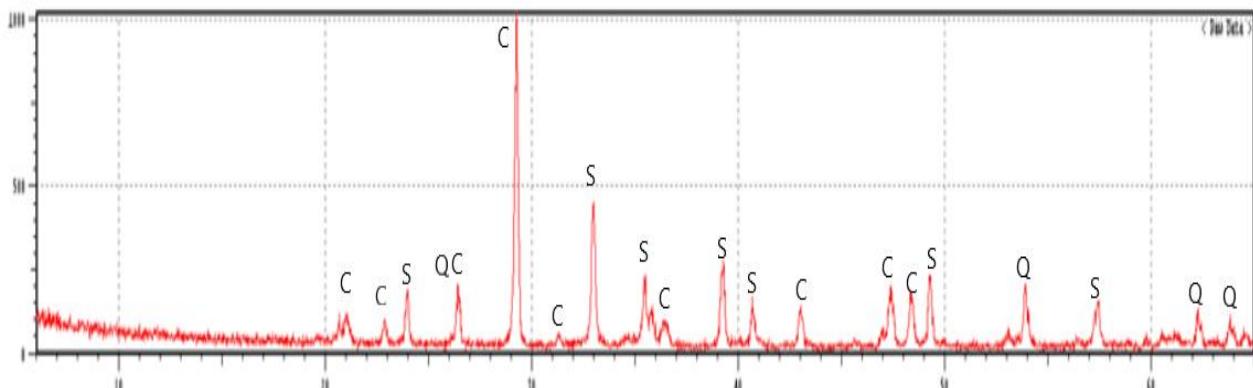
as an impurity within the specimen. Mn oxides often form alongside iron oxides in nature and can sometimes substitute for iron in the crystal lattice. The presence of Mn might also indicate that the specularite formed in an environment containing manganese-rich minerals, leading to mixed oxide phases.

Unlike hematite ore, specularite ore has not yet been efficiently utilized in the iron-making industry. Magnetization roasting technology is an effective method for processing specularite ore (Xinran Zhu et al. 2023).

In today's strongly promoted sustainable development strategy, steel enterprises are increasingly trying to incorporate specularite into the blast furnace production process (Naito et al., 2014; Chun and Zhu, 2016; Sheng-li et al., 2018; Guo et al., 2020; Kumar et al., 2022; Zhu et al., 2023):

Specularite has been used effectively and successfully for decades in anti-corrosive paint, serving the following purposes:

- *Barrier effect*: The highly laminar structure lengthens the diffusion paths for water and oxygen through the film until they reach the substrate. The plates are oriented and overlapping, hindering access of water, oxygen, electrolytes, and pollutants.
- *Shield effect (UV protection)*: The iron micaeous sheets align parallel to the film surface, providing excellent waterproofing and protection against ultraviolet radiation, unlike aluminum pigments which absorb much of the infrared radiation (Restrepo et al., 2009).



CONCLUSION

After summarizing the data collected in this research, we can confirm that the studied mineral sample is specularite. Specularite from Damjan deposite has a gray-black color, metallic luster, and a reddish streak. The crystals are more or less aligned in certain directions. Mangan and aluminium substitute for Fe^{3+} in the specularite structure. Silicon is present as quartz or clay inclusions. Small amounts of Fe^{2+} and Mn^{2+} have replaced Ca^{2+} in the crystal lattice of calcite. The Mn and Fe contents in calcite are too low to form individual Mn-oxide or Fe-oxide minerals.

Specularite is formed in skarn deposits when iron-rich fluids interact with the host rock. In some cases, the iron may originate from a magmatic intrusion containing high levels of iron. These iron-rich fluids oxidize upon contact with oxygen, forming iron-rich hematite or specularite. The crystals often form as elongated or prismatic shapes, which is typical of the specular variety of hematite. The well-developed morphology of specularite suggests that it crystallized first.

The finer-grained calcite likely formed later, filling the interstitial spaces between the earlier-formed specularite.

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Резиме

МИНЕРАЛОШКА КАРАКТЕРИЗАЦИЈА НА ХЕМАТИТ-СПЕКУЛАРИТ ОД ДАМЈАН, СЕВЕРНА МАКЕДОНИЈА

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Клучни зборови: спекуларит; калцит; кварц; Дамјан

Во овој труд е претставена минералошката карактеризација на спекуларит од наоѓалиштето Дамјан. Спекуларитот од рудното наоѓалиште Дамјан има сивоцрна боја, метален сјај и црвеникава боја на огреб. Кристалите се повеќе или помалку порамнети во одредени насоки, покажувајќи слоевитост. Мanganот и алуминиумот го заменуваат Fe^{3+} во структурата на спекуларит. Силициумот е присутен како кварцна или глинена инклузија. Мали количини на Mn^{2+} и Fe^{2+} го заменуваат Ca^{2+} во кристалната решетка на калцитот. Содржината на мangan и железо во калцитот е премногу ниска за формирање на одделни минерали на Mn-оксид и Fe-оксид.

Спекуларитот се формира во скарнови кога течностите богати со железо доаѓат во контакт со матичната карпа. Во некои случаи железото може да потекнува и од магматската интрузија, која содржи високи нивоа на железо. Овие течности богати со железо оксидираат при контакт со кислород, што доведува до формирање на железни оксиди како што е хематит-спекуларит. Добро развиената морфологија на спекуларитот сугерира дека тој кристализирал прв. Пофинозрнистот калцит веројатно се формирал малку подоцна исполнувајќи ги меѓупросторите на претходно создадениот спекуларит.