412
GEOME 2
Manuscript received: August 5, 2024
Accepted: October 14, 2024

*Geologica Macedonica*, Vol. **38**, No. 2, pp. 97–103 (2024)
On print ISSN 0352 – 1206
On line ISSN 1857 – 8586
UDC: 552.3:550.93]:552.4.08(497.784/.785)
https://doi.org/10.46763/GEOL24382097b

*Original scientific paper*

# AGE OF THE PEGMATITES IN THE PELAGONIAN METAMORPHIC COMPLEX

**Ivan Boev, Tena Šijakova Ivanova, Sonja Lepitkova**

*Faculty of Natural and Technical Sciences, Goce Delcev University, Stip,*
*Blvd. "Goce Delcev" 89, P. O. Box 201, 2000 Stip, North Macedonia*
ivan.boev@ugd.edu.mk

A b s t r a c t: The Pelagonian metamorphic complex, or tectono-stratigraphic complex, extends approximately 420 km in length and 60 km in width in a NNW-SSE orientation, forming part of the central Hellenides. The occurrence of pegmatites within the Pelagonian metamorphic complex is quite common. They appear within the metamorphic complex of the gneisses and in direct contact with the granitic intrusive bodies. Mainly these pegmatite bodies are made of quartz, feldspars, micas (bioite, muscovite, paragonite, vermuiculite) as well as are the occurrence of rare minerals such as tourmalines, epidotes, apatites, garnets and zircons. The paper presents the new information related to the age of two pegmatitic bodies by the U/Pb dating and the K/Ar dating: Alinci and Čanište. The obtained age is Cretaceous.

**Key words**: Pelagnonian metamorphic complex, pegmatites, age determination

## INTRODUCTION

The Drina-Ivanjica-Pelagonian belt (Pelagonides) lies between the Sava suture, located to the west of the Vardar zone, and the Dinarides ophiolite belt (west Vardar). It is primarily composed of Precambrian and Paleozoic complexes, which are overlain by Triassic and Jurassic carbonates. These formations are thrust and imbricated within a west-verging nappe system situated west of the Vardar s.s. zone. The belt was covered by Upper Cretaceous flysch deposits and subsequently underwent additional west-verging thrusting during the Palaeogene (Zagorchev, 2020).

The Pelagonian metamorphic complex, or tectono-stratigraphic complex, extends approximately 420 km in length and 60 km in width in a NNW-SSE orientation, forming part of the central Hellenides (Figure 1) (Palinkaš et al., 2012). The Pelagonian massif, the largest unit of this belt, spans North Macedonia and Greece (Florina "terrane"). This high-grade metamorphic complex, which reaches amphibolite facies with relict eclogites, is divided into two main parts: (1) a lower complex composed of biotite and two-mica gneisses, amphibolites, hornblende and epidote-hornblende gneisses, leucocratic gneisses, magmatites, and orthogneisses; and (2) an upper complex consisting of gneisses, micaschists, amphibolites, calcareous schists, and massive marbles (Sivec marbles). Rb-Sr age determinations from the northern part of the massif in North Macedonia, along with recent U-Pb data from Greece (Florina terrane), indicate polymetamorphic Neoproterozoic ages for these complexes, with significant dates up to 700 Ma in the metagranites. An important Late Carboniferous metamorphic and igneous event occurred around 300 Ma (Zagorchev, 2020).

The base of the complex consists of a Precambrian crystalline core, primarily composed of ortho- and paragneisses, micaschists, and amphibolites. Granitoid magmatism was emplaced during two main episodes: (1) the Upper Carboniferous and (2) the Late Permian to Early Triassic (Most, 2003). Pelagonian granitoids vary in composition from granite to quartz-diorite, but are predominantly granodioritic (Dumurdžanov, 1985; Most, 2003). The Upper Carboniferous granodiorite (299 ± 1 Ma, U-Pb zircon dating; Most, 2003) experienced compressional deformation, resulting in a greenschist to amphibolite-grade metamorphic overprint. In contrast, the Late Permian to Early Triassic granodiorite (~245 ± 1 Ma, U-Pb zircon dating; Most, 2003) formed as massive intrusive bodies in the eastern Pelagonian zone, including the Selečka Mountain. Investigations of pegmatite ages in Alinci (K/Ar method, 105.2 Ma) suggest significant magmatic activity during the Cretaceous, beginning in the Lower Cretaceous (Boev I. et al., 2021).

The Pelagonian zone also includes sedimentary sequences of carbonate and clastic rocks deposited during the Triassic and Jurassic. Its geological structure largely reflects polyphase tectono-metamorphic events that occurred during the convergence of the Apulian and European plates from the Upper Jurassic to the Upper Tertiary (Most, 2003).

In this study, we report for the first time the occurrence of spectacular zircon and microcline crystals within the pegmatites near Prilep, which have not been dated until now. We present chemical and geochronological data that demonstrate a Lower Cretaceous age for this magmatic episode.

## *Local geology*

To examine the age of the pegmatites in the Pelagonian metamorphic complex, samples were taken from two localities: Alinci and Čanište. A microcline sample was taken from the Alinci locality to determine the age (for K/Ar geochronology) (Figure 1), and from the Čanište locality zircon was taken (for U/Pb geochronology).

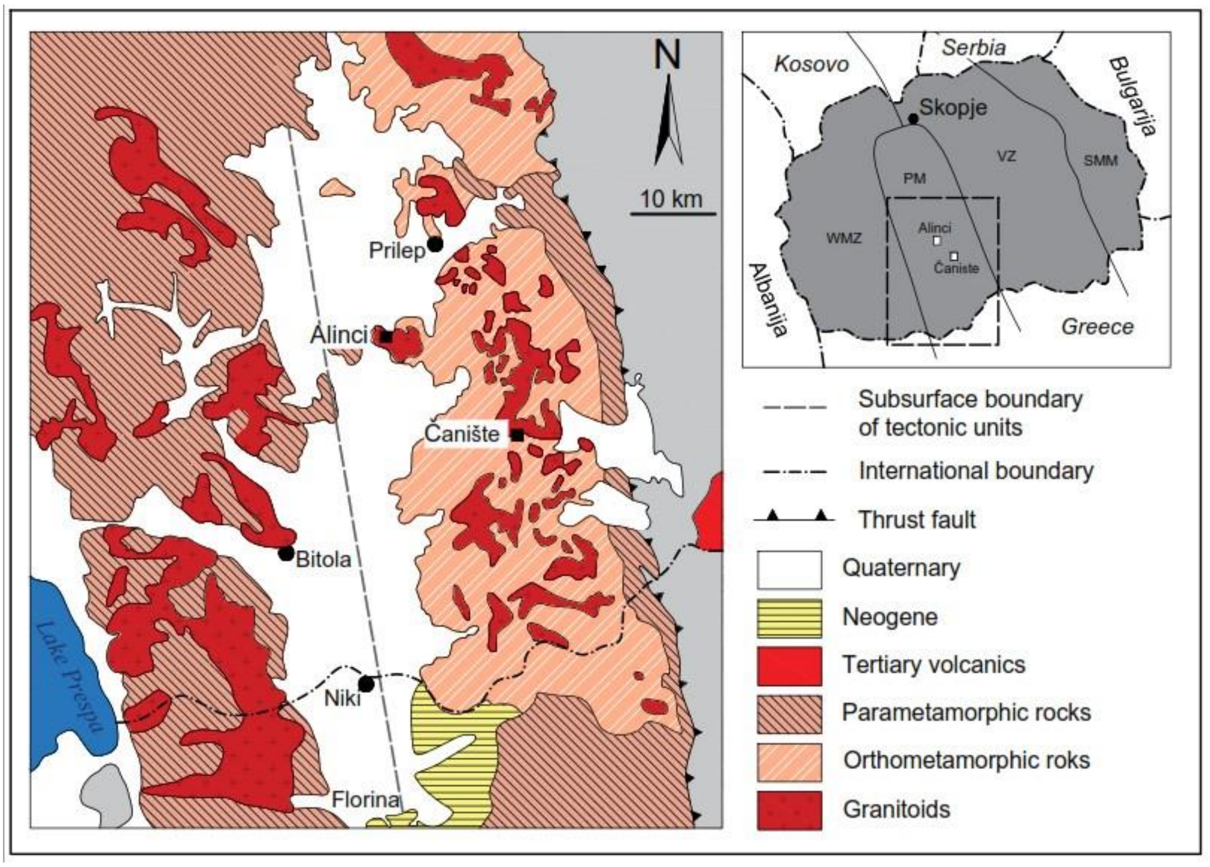



**Fig. 1.** Geological map of the Pelagonian metamorphic complex (by Palinkaš, S. 2012, completed by I. Boev, 2024)

The Alinci locality is situated near the village of Alinci, approximately 3 km from the Prilep–Bitola regional road. It is located within the Pelagonian metamorphic complex, in a metamorphic belt that separates the Prilep and Bitola fields. At this site, metamorphic rocks (gneisses) form three elevations descending from the northern branches of Selečka Mountain into the Pelagonian valley. The locality itself is on a hill known as Crn Kamen (Marić, 1949). The pegmatitic occurrences here are found within a series of alkaline syenites and gneisses. These pegmatites consist of microcline, arfvedsonite, albite, titanite, augite, zircon, and apatite (Barić, 1964).

A distinctive feature of the Alinci deposit is its rare mineral paragenesis, which includes uranium minerals. Of particular interest are the frequent nests, several centimeters in size, filled with needle-like crystals of arfvedsonite. Arfvedsonite, a mineral from the amphibole group, typically occurs as acicular crystals with a greenish, dark, or blue hue and is often found as inclusions in other minerals. Albite is also common, presenting as platy white to completely transparent crystals, with some reaching up to 10 cm in size. Twinned grains, such as polysynthetically twinned albite, or Carlsbad twins, are frequently observed. Arfvedsonite crystals are often found as inclusions within albite. Noteworthy are the well-developed quartz crystals, large titanite crystals (up to 2 cm in size), and the presence of monazite and macedonite (Figure 2).

**Fig. 2.** Microcline+albite+arfvedsonite from the Alinci locality

In addition, the occurrence of rare minerals associated with uranium, such as davidite, is notable (Damjanović, 1961; Žorž et al., 1988/1989, 1999). The Čanište pegmatite is one of numerous pegmatite occurrences within the eastern Pelagonian zone. These pegmatites vary in size, ranging from a few decimeters wide and tens of meters long to larger bodies that span tens of meters in width and hundreds of meters in length. They differ in mineralogical characteristics, internal structures, and degree of fractionation. Among these, pegmatites enriched in uranium and thorium mineralization (such as those at Alinci and Crni Kamen) are particularly interesting (Ivanov et al., 1966; Radusinović & Markov, 1971; Bermanec et al., 1988, 1992).

The Čanište pegmatite, a lens-shaped body up to 10 meters wide, cuts through Precambrian gneisses (Dumurdžanov, 1985; Most, 2003). At the Čanište locality, the pegmatite vein reaches up to 15 meters in width and 50 meters in length. It consists mainly of potassium-sodium feldspar and quartz, with smaller amounts of biotite and epidote in large crystals. Garnet and amazonite are less common, while zircon appears as well, making Čanište the only locality where zircon occurs within the pegmatites (Bermanec et al., 2001).

Zircon in the Čanište pegmatite vein appears in two distinct forms:

i) Zircon found in microdig forms small crystalline aggregates, cream to pale white in color, with significant cracking. These cracks are not filled with other minerals. In addition to zircon, plagioclase, quartz, and fine-grained muscovite are also present (Figure 3).

ii) Zircon which is dark yellow in color and does not show any differences in the microscope compared to the previous variety, this one is also cracked and appears in association with plagioclase, quartz and muscovite (Figure 4).

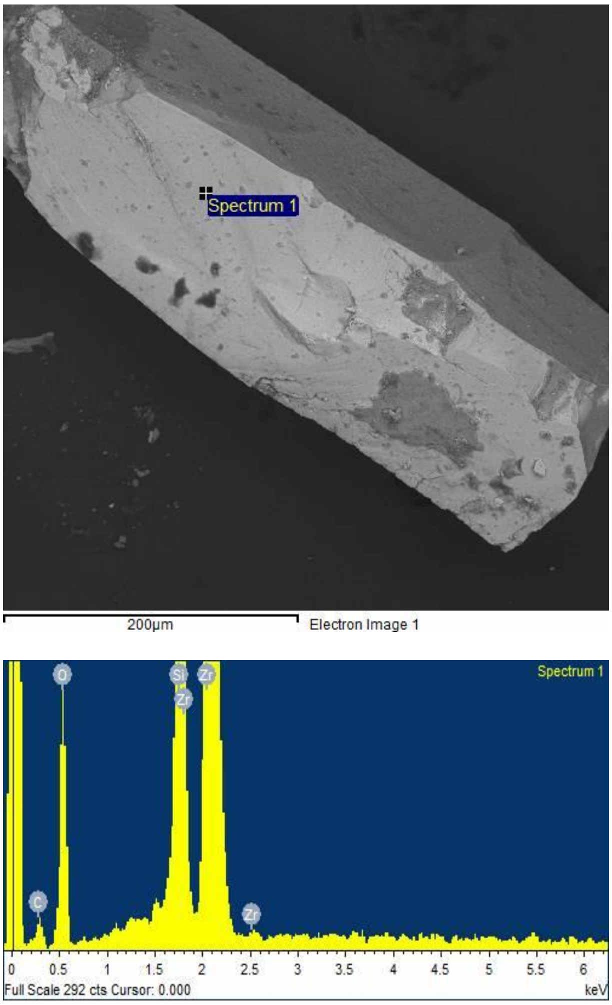



**Fig. 3.** SEM-EDS of cream to pale white zircon from Čanište

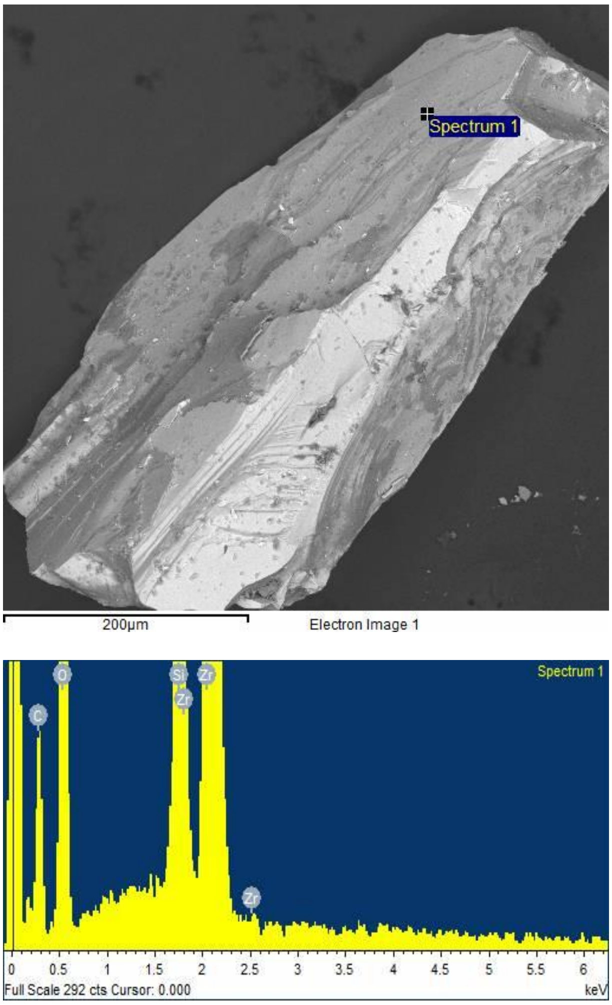



**Fig. 4.** SEM-EDS of dark yellow zircon from Čanište

## METHODOLOGY

Samples were taken on which mineralogical separation of microcline was done and age was determined using the K/Ar method.

### *K-Ar methodology*

Aliquot of the sample was weighted into Al container, loaded into sample system of extraction unit, degassed at ~100°C during 2 days to remove the surface gases. Argon is extracted from the sample in double vacuum furnace at 1700°C. The determination of radiogenic argon content was carried out twice on MI-1201 IG mass-spectrometer by isotope dilution method with $^{38}$Ar as spike, which is introduced to the sample system prior to each extraction.

The extracted gases were cleaned up in two step purification system. Then pure Ar is introduced into custom built magnetic sector mass spectrometer (Reinolds type).

It shall be noted that the test was done twice per sample to ensure the consistency of the result.

Two globally accepted standards (Bern-4M Muscovite and 1/65 "Asia" rhyolite matrix) were measured for $^{38}$Ar spike calibration.

For age calculations the international values of constants were used as follow:

$$\lambda_K = 0.581\times10^{-10}y^{-1},\ \lambda_\beta = 4.962\times10^{-10}y^{-1},$$

$$^{40}K = 0.01167\ (at.\%).$$

Mineralogical separation of zircon was also made from the samples taken from the Čanište locality and the age was determined using the U/Pb method.

### *U-Pb methodology*

U-Pb zircon ages were determined at the London Geochronology Centre at University College, London, using an Agilent 7900 LA-ICP-MS (laser ablation-inductively coupled plasma-mass spectrometry) system, employing a NewWave NWR193 Excimer Laser operated at 10 Hz with a 25 μm spot size and ~2.5 J/cm$^2$ fluence. No cathodo-luminescence imaging was conducted. Two laser spots were placed in each zircon grain to test the consistency of the results. No common Pb correction was applied. The mass spectrometer data were converted to isotopic ratios using GLITTER 4.4.2 software employing Plešovice zircon (Sláma et al., 2008) as a primary age standard and GJ-1 and 91500 as secondary age standards obtaining average ages of 605.0 ± 2.5 ($n$ = 23; MSWD = 0.59, one outlier removed) and 1048 ± 4 ($n$ = 24; MSWD = 0.94), respectively. A NIST SRM612 glass was used as a compositional standard for U and Th concentrations. GLITTER files were post-processed in R using IsoplotR 6.1 (Vermeesch, 2018a, 2018b). Due to the low U-concentrations, relatively young ages, and small spot size, the $^{207}Pb$ signals were too low to permit precise $^{207}Pb/^{235}U$ ages, although they did permit us to confirm the concordance of the U-Pb system. We base our geochronological analysis on a weighted mean of $^{206}Pb/^{238}U$ ages.

## RESULTS

The age of the pegmatite occurrences at the Alinci locality was determined using the K/Ar isotopic method on a sample of the mineral microcline. The mineral microcline is widespread within pegmatite occurrences and occurs in coarser and finer crystals. The performed isotopic tests show that the pegmatite bodies in the Alinci locality have a Cretaceous age (Table 1)

Table below presents the results of the K-Ar geochronology test. The certainty of the ages calculated fall within 2σ error.

T a b l e 1

*K/Ar age determination of microcline from the Alinci pegmatite body*

| Minerals | K, % ± σ | $^{40}$Ar rad, (ng/g) % | $^{40}$Ar air | Age, Ma | 2σ |
|---|---|---|---|---|---|
| Microcline | 12.39±0.13 | 93.1±0.3 | 5.5 | 105.2 | 2.3 |

The age of the Čanište pegmatite body was determined by the U/Pb method based on the mineral zircon. The separation of the mineral zircon showed that there are two varieties of zircon in this locality (W – zircon graine, and Y – zircon graine). (Figure 5).

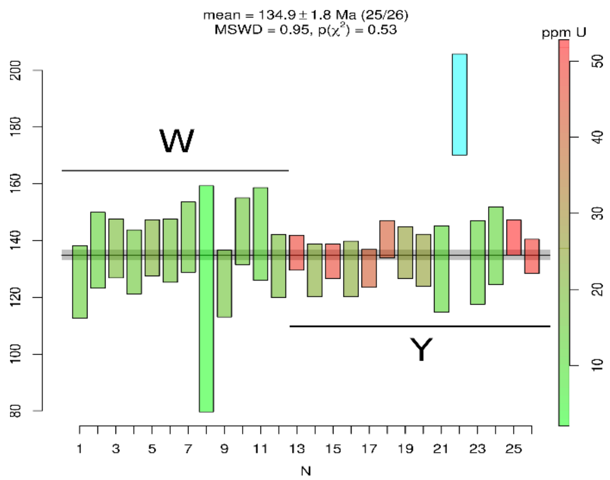



**Fig. 5.** Age of the zircon grain from Čanište pegmatite body

## CONCLUSION

The Pelagonian metamorphic complex has undergone prolonged exposure to polyphase tectonic deformation and metamorphism, resulting in a complex thermal evolution. Based on K/Ar age investigations of biotites (Most, 2001), four distinct tectono-magmatic phases have been identified within the Pelagonian metamorphic complex, which occurred on a pre-Cambrian foundation. The first phase occurred between 447 ± 17 Ma and 267 ± 10 Ma, followed by a second phase of dynamo-thermal metamorphism, accompanied by partial melting, between 148 ± 6 Ma and 114 ± 4 Ma. The third phase took place between 102 ± 4 Ma and 86 ± 34 Ma, while the fourth phase spanned from 64 Ma to 36 Ma.

These data indicate that the Pelagonian metamorphic complex experienced a complex and prolonged thermal evolution lasting approximately half a billion years. The formation of pegmatite bodies at the Alinci locality most probably resulted from partial melting within the gneisses during the Cretaceous period (105.2 ± 2.3 Ma), which corresponds to the third phase of the complex's thermal evolution. Additionally, the formation of pegmatite veins at the Čanište localities occurred during the second phase of thermal evolution, around 134.9 ± 1.8 Ma.

APENDIX: Photografs of zircons grains under the ICP-MS-LA.

***Acknowledgments***

We would like to thank Peter Vermeesch for help with sample preparation and analysis and age determination of zircon grains.

## REFERENCES

Barić, L (1964): *Mineralgänge von Crni Kamen bei dem Dorf Alinci in Macedonien*, Mineralogical Petrographic Museum of Zagreb, 23–30 (1964).

Bermanec, V., Tibljas, D., Gessner, M., Kniewald, G. (1988): Monazite in hydrothermal veins from Alinci, Yugoslavia. *Mineral. Petrol*., **38**, 139–150. doi: 10.1007/BF01164318

Bermanec, V., Tibljas, D., Kniewald, G. (1992): Uranium-rich metamict senaite from Alinci, Macedonia, Yugoslavia. *Eur. J. Mineral*., **4**, 331–335.

Bermanec, V., Palinkaš, L. A., Strmic, S. (2001): Mineralogy of pegmatite with giant epidote crystals, near Čaniše, Macedonia. In: Piestrzynski, A. (ed.): *Mineral Deposits at the Beginning of the 21st Century*, Proceed. 6th Biennial SGA Meeting, 939–942.

Boev, Ivan and Bermanec, Marko (2021): Geology, Petrology and the age of pegmatites in Alinci locality (North Macedonia), *Natural Resources and Technologies*, **15** (2). pp. 33–41. ISSN 1857-6966

Damjanović, A. (1961): DAVIDITE – mineral from Crni Kamen, Macedonia, *Radovi Sektora za istraživanje nukelarnih i drugih sirovina*, Beograd, 1961, pp. 11–15.

Dumurdžanov, N. (1985): Petrogenetic characteristics of the high metamorphic and magmatic rocks of the central and western part of the Selečka Mt. (Pelagonian massif), SR Macedonia, Yugoslavia. *Geol. Maced*., **2**, 173–220.

Ivanov, T., Bogoevski, K., Radović, N., Ivanova, V. (1966): Guidebook for Fieldtrips in SR Macedonia, Yugoslavia. *6th Meeting of Geologists of Yugoslavia*, Ohrid, 17 p.

Marić, L. (1948): Metamorfne kamnine Bakarnega Gumna in Vesleca južno in jugozahodno od Prilepa, Razprave. Pazred za prirodoslovne in medicinske vede Slovenske akad. zn. in um. 4, Ljubljana, pp. 229–246.

Most, T. (2003): *Geodynamic evolution of the eastern Pelagonian zone in northwestern Greece and the Republic of Macedonia*. Unpubl. PhD Thesis, University of Tuebingen, Tuebingen, 195 pp.

Palinkaš, S., Bermanec, V., Palinkaš, L., Boev, B., Gault,R., Procháska, W., Bakker, R. (2012): The evolution of the Čanište epidote-bearing pegmatite, Republic of Macedonia: evidence from mineralogical and geochemical features. *Geologia Croatica* **65**, 3, 423–434.

Radusinović, D., Markov, C. (1971): Macedonite – lead titanate: a new mineral. *Am. Min*., **56**, 387–394.

Sláma, J., Košler, J. et al. (2008): Plešovice zircon – A new natural reference material for U–Pb and Hf isotopic microanalysis, *Chemical Geology*, Volume **249**, Issues 1–2, pp. 1–30.

Žorž, M., Vidrih, R., Mikuž, V., Kobler, G., Mušić, B., (1988/1989): Minerals from Alinci in Macedonia, *Proteus*, **51**, 326–330 (in Slovenian).

Žorž, M., Jeršek, M., Mladenovski, G. (1999): Some mineral locations in Macedonia and their paragenesis, In: *Hidden Treasures of Macedonia, Scopolia* **41** (Supplement 2), 9–69.

Vermeesch, P. (2018a): Dissimilarity measures in detrital geochronology. *Earth – Science Reviews*, **178**, 310–321. https://doi.org/10.1016/j.earscirev.2017.11.027

Vermeesch, P. (2018b): Statistical models for point-counting data. *Earth and Planetary Science Letters*, **501**, 1–7. https://doi.org/10.1016/j.epsl.2018.08.019

Zagorchev, I. (2020): Geology of the Balkan Peninsula, Bulgarian Academy of Sciences, Sofia, Bulgaria, *Encyclopedia of Geology*, Volume 2E. https://doi.org/10.1016/b978-0-08-102908-4.00056-4

## Р е з и м е

## СТАРОСТ НА ПЕГМАТИТИТЕ ВО ПЕЛАГОНСКИОТ МЕТАМОРФЕН КОМПЛЕКС

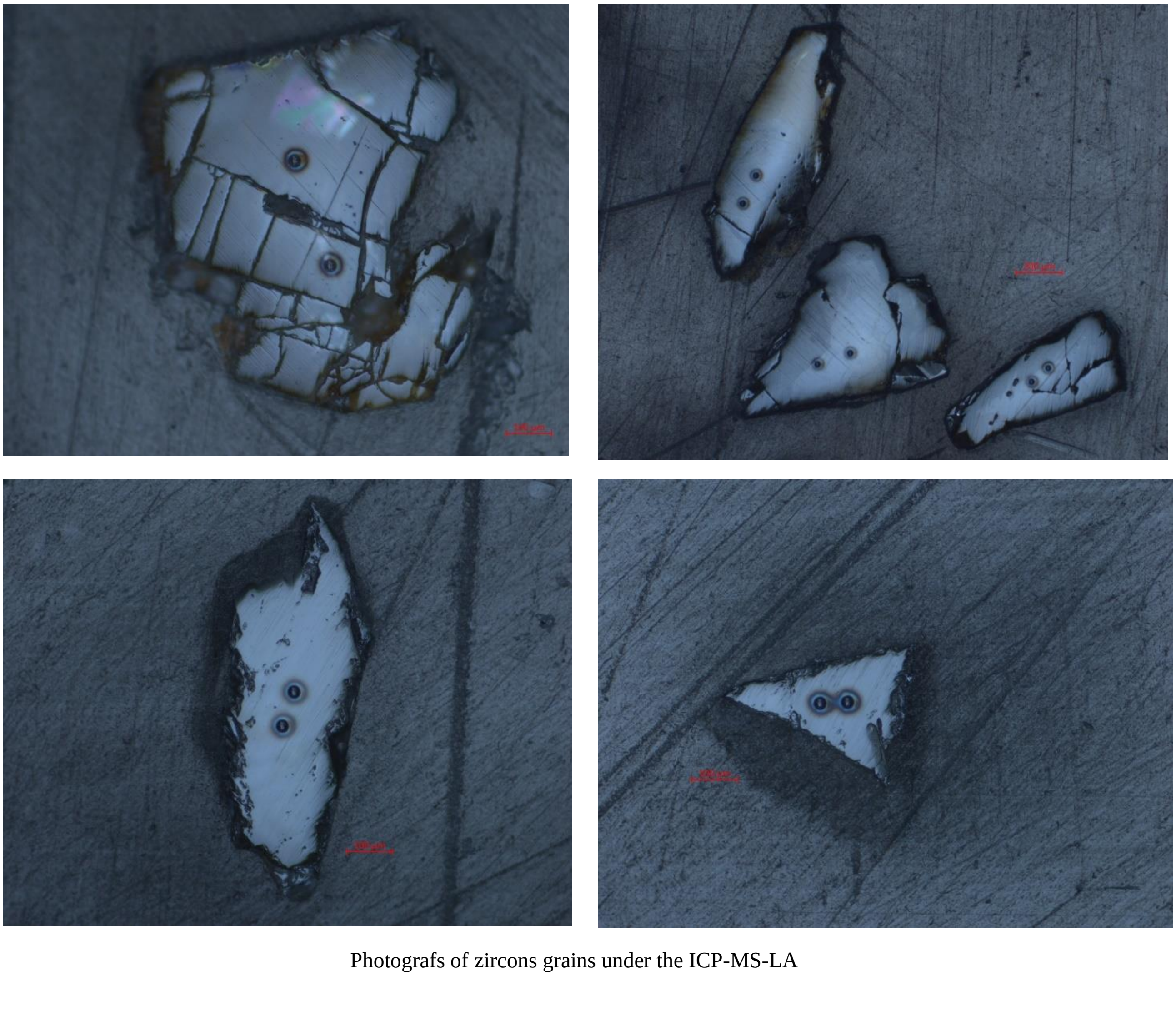
**Иван Боев, Тена Шијакова Иванова, Соња Лепиткова**

*Факултет за природни и технички науки, Универзитет “Гоце Делчев”, Штип,*
*бул. „Гоце Делчев“ 89, п.фах 201, 2000 Штип, Северна Македонија*
ivan.boev@ugd.edu.mk

**Клучни зборови:** пелагнонски метаморфен комплекс; пегматити; староснa определба

Пелагонискиот метаморфен комплекс, или тектоностратиграфски комплекс, се протега приближно 420 km во должина и 60 km во ширина во ориентација ССЗ-ЈЈИ, формирајќи дел од централните Хелениди. Појавата на пегматити во рамките на пелагонискиот метаморфен комплекс е доста честа појава. Тие се појавуваат во метаморфниот комплекс на гнајсевите и во директен контакт со гранитните интрузивни тела. Овие пегматитни тела се направени главно од кварц, фелдспат, мика (биотит, мусковит, парагонит, вермикулит), а има појава и на ретки минерали како што се турмалини, епидоти, апатити, гранати и циркони. Во трудот се претставени новите информации поврзани со староста на две пегматитни тела од Алинци и Чаниште со датирање U/Pb и K/Ar. Добиената старост е креда.

## APENDIX



Photografs of zircons grains under the ICP-MS-LA