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New U-Pb and Ar-Ar mineral ages for the Barutin-Buynovo-Elatia-Skaloti-Paranesti batholith (Bulgaria and Greece): Refinement of its debatable age

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Abstract. High precision ID-TIMS U-Pb data on single zircons from hornblende-biotite granodiorite (GDR) in the Greek part of the batholith and isotope Hf-data of the dated zircons are combined in the present study to elucidate the debatable intrusion age of the pluton and to define its magma source. The intrusion age is determined at 55.93±0.28 Ma by two concordant zircons, one grain shows negligible lead loss, and two zircons reveal lead inheritance, pointing to possible Permo-Triassic and Jurassic ages of the inherited cores/grains. The hafnium isotope characteristics of the analyzed zircons (corrected for an age of 56 Ma) are slightly positive (+1 to +3). They argue that the magma was generated either by mixing of initial mantle and crustal magma or by remelting of former subduction/postcollision related materials.

Ar-Ar isotope dating was applied to biotite and potassium feldspar from a hornblende-biotite granodiorite in the Bulgarian part. A plateau age of 49.99±0.88 Ma was obtained on the biotite fraction excluding the older ages of the first and last three steps. This age is confirmed by the isochrone calculations of $^{40}Ar/^{36}Ar$ *vs.* $^{39}Ar/^{36}Ar$ and $^{36}Ar/^{40}Ar$ *vs.* $^{39}Ar/^{40}Ar$ at 50.6±1.2 Ma and 50.6±2.1 Ma, respectively. The data for potassium feldspar are less confident, but generally in the range 42-44 Ma. Both Ar-Ar ages are interpreted as cooling ages, or a slightly reset by metamorphic/tectonic overprint. The new isotope data are in agreement with recent mineralogical, geochemical, oxygen- and strontium isotope data for the BBESP batholith, suggesting Paleocene-Eocene time of intrusion of rock groups, which possibly differ slightly in time and genesis.

Key words: Southern Rhodope Mountains, Petrology, Geochronology, composite plutons

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Триантафилос Солдатос, Антонис Коронеос, Борислав К. Каменов, Ирена Пейчева, Албрехт фон Куадт, Георгиос Христофидес, Ксианг Зенг, Хуанг Санг. Нови U-Pb и Ar-Ar минерални възрасти за батолита Барутин-Буйново-Елатия-Скалоти-Паранещи (България и Гърция): Прецизиране на спорната му възраст

Резюме: Единични циркони от амфибол-биотитов гранодиорит (GDR) от гръцката част на батолита са датирани с високо-прецизния ID-TIMS U-Pb метод. Данните са съчетани с изотопни Hf-данни от същите датирани циркони, за да се изясни спорната възраст на внедряване на плутона и да се определи неговия магматичен източник. Възрастта на внедряване е определена на 55,93±0,28 Ма върху два конкордантни циркона, едното зърно от които показва незначителна загуба на олово, а други две зърна разкриват унаследено олово, което сочи за възможни пермо-триаска и юрска възрасти на унаследените ядра/зърна. Изотопните хафниеви характеристики на анализираните циркони (коригирани за възраст от 56 Ма) са слабо положителни (+1 to +3). Те доказват, че родоначалната магма е генерирана чрез смесване на първична мантийна и корова магми или е получена чрез претопяване на по-стари субдукционни или постколизионини материали.

На биотитови и калиево-фелдшпатови фракции от амфибол-биотитов гранодиорит от българската част на батолита е приложено Ar-Ar изотопно датиране. Получена е плато-възраст от 49,99±0,88 Ма от биотитовата фракция, като са изключени по-старите възрасти на първото и последните три стъпала. Тази възраст е потвърдена чрез изохронни изчисления върху диаграми ^{40}Ar/^{36}Ar *vs.* ^{39}Ar/^{36}Ar и ^{36}Ar/^{40}Ar *vs.* ^{39}Ar/^{40}Ar при 50,6±1.2 Ма и 50,6±2.1 Ма, съответно. Резултатите за калиевия фелдшпат са по-малко достоверни, но най-общо са в диапазона 42-44 Ма. И двете Ar-Ar възрасти са интерпретирани като етапи от охлаждането на плутона или като резултат от слаб ефект на метаморфно/тектонско събитие, оказало влияние върху изотопната система. Новите изотопни резултати са в съгласие с публикувани напоследък минераложки, геохимични, кислородни и стронциеви изотопни данни за батолита BBESP, предлагайки Палеоцен-Еоценска възраст на внедряване на скалните групи, които е възможно да се различават слабо по време на образувани и произход.

Introduction

The composite batholith Barutin-Buynovo-Elatia-Skaloti-Paranesti (BBESP) is exposed in the southernmost part of the Rhodope Mountains in Southern Bulgaria and in Northern Greece (Fig. 1). It covers an area of about 850 km^2 being the biggest magmatic body in the area. The Bulgarian outcropped part of the batholith is around 160 km^2 and it is called Barutin-Buynovo pluton (Kamenov et al. 1974). In Greece the batholith consists of the plutons Elatia (Soldatos 1985), Paranesti (Sklavounos 1981) and Skaloti (Kotopouli & Pe-Piper 1989). Sometimes the name Elatia-Skaloti (Kotopouli 1981; Kotopouli & Pe-Piper 1989) is assigned to the southern Greek parts. Soldatos et al. (2001a b) used the name Elatia-Skaloti-Paranesti for the whole Greek part, which is used also in the present paper. Kamenov et al. (1990) and Jones et al. (1992) unified the Bulgarian and the Greek parts into a large composite batholith – the BBESP.

The batholith is intruded into a complex metamorphic basement consisting of gneisses and marbles with rare amphibolitic intercalations. The plutonic rocks are covered transgressively and discordantly by Low Oligocene molassic sedimentary rocks (Kamenov et al., 1974). The presence of Oligocene rhyolitic rocks in the vicinity of the plutonic intrusion provides an upper limit to the age of the pluton.

The petrographical composition is mainly of biotite (±amphibole) granodiorite, passing locally into quartz-diorite or monzogranite in the Bulgarian part and of granodiorite (GDR), granite (GR) and two-mica granite (TMG) in the Greek part (Fig. 1). The porphyritic type granodiorites which are outcropped in the peripheral parts are considered as a facies subjected to endocontact changes.

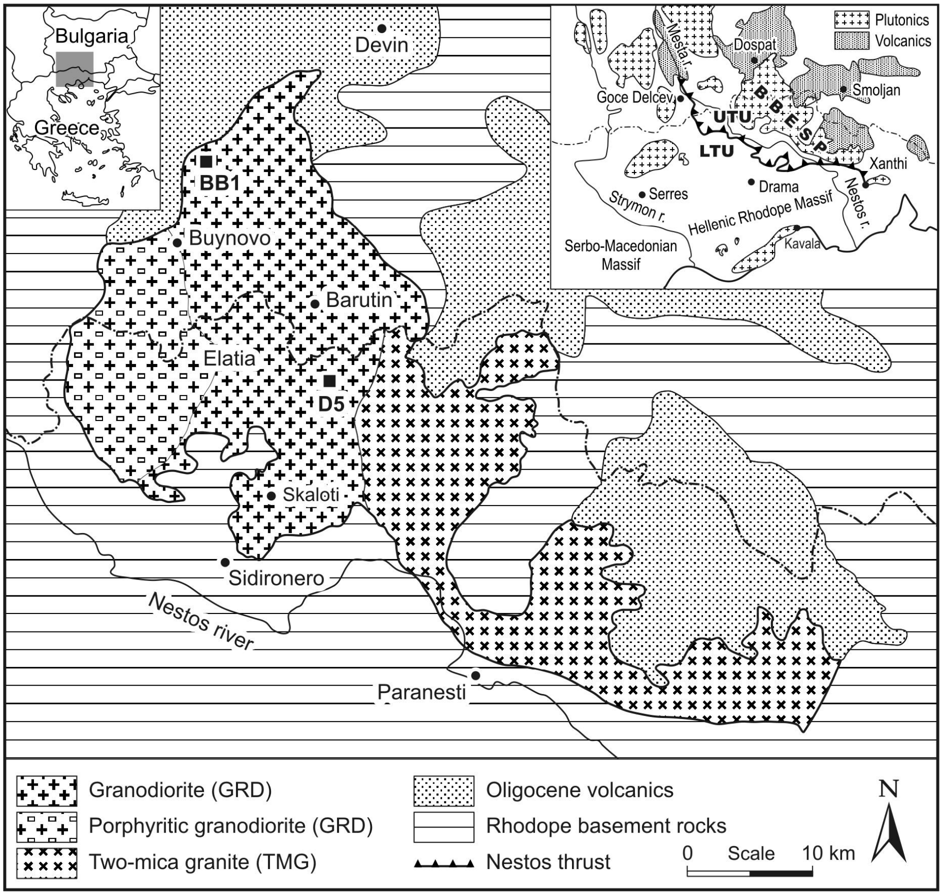

Fig. 1. Simplified geological map of the Barutin-Buynovo-Elatia-Skaloti-Paranesti batholith (BBESP) showing the main rock-types and the location of the analyzed samples. In the inset map the main magmatic bodies and tectonic units of the Rhodope massif in Greece and Bulgaria are shown: (LTU) Lower Tectonic Unit (Pangeo Unit); (UTU) Upper Tectonic Unit (Sidironero Unit)

The age of the batholith is still debatable (Table 1). Biotite K-Ar datings from both the Greek and Bulgarian parts of the batholith gave ages of 29.1±1.2 to 45.0±2.0 Ma (Kamenov et al. 1974; Palshin et al. 1974; Sklavounos 1981). Rb-Sr whole-rock ages are not well defined as 86.7±27.0 Ma (initial $^{87}Sr/^{86}Sr$=0.7060, MSWD=165) and 85±25 Ma (Soldatos 1985; Soldatos & Christofides 1986; Soldatos et al. 2001a). Rb-Sr biotite and muscovite dating obtained an older than 48 Ma age for the granodiorites of the batholith and 47 Ma as a cooling age on the muscovite (Soldatos et al. 2001a), whereas for muscovites of the two-mica granite Soldatos et al. (2001a) calculated isochrones of 43.5±0.9 to 47.8±1.0 Ma. This wide range of age data arises questions on either they are due to the different resistance and closure temperatures of the applied isotope methods or to different intrusion time of the granitoids.

Present study examined new approaches to the age problem. It combines high precision ID-TIMS U-Pb data on single zircons from the

Table 1. *Published geochronological data of the granitoid rocks from the Barutin-Buynovo-Elatia-Skaloti-Paranesti batholith (BBESP)*

Pluton	Method	Mineral or rock	Age (Ma)	$^{87}Sr/^{86}Sr$	Reference
Elatia	Rb-Sr	WR (GRD) errochron	86.7 ±27.0	0.7060	Soldatos (1985)
	Rb-Sr	WR (GRD) errochron	85±25	0.7060	Soldatos et al. (2001a)
	Rb-Sr	Bt (GRD)	34.1±1.0 to 43.0±1.3	0.7061-0.7066	Soldatos et al. (2001a)
	Rb-Sr	Bt (GR)	36.9±1.1 to 42.0±1.2	0.7077-0.7084	Soldatos et al. (2001a)
	U-Pb	**Zrn (GRD)**	**55.93 ± 0.28**		**this study**
Paranesti	K-Ar	Ms	38.3±1.1		Meyer (1968)
	K-Ar	Bt	29.1±1.2 to 38.5±1.5		Sklavounos (1981)
	Rb-Sr	Ms (TMG)	43.5±0.9 to 47.8±1.0	0.7068-0.7075	Soldatos et al. (2001a)
	Rb-Sr	Bt (TMG)	39.4±1.2		Soldatos et al. (2001a)
Barutin-Buynovo	K-Ar	Bt	42.0±2.0		Palshin et al. (1974)
	K-Ar	Bt	45.0±2.0		Kamenov et al. (1974)
	Ar-Ar	**Bt (plateau)** **Bt (isochron)**	**49.99±0.88** **50.6±1.2**		**this study**
	Ar-Ar	**Kf (plateau)** **Kf (isochron)**	**42.15±0.62** **44.9±2.3**		**this study**

WR: whole-rock; Bt: biotite; Ms: Muscovite; Zrn: zircon

granodiorite in the Greek part of BBESP and isotope Hf-study of the dated zircons with the aim to elucidate the intrusion age of the pluton and to define its magma source. We applied also Ar-Ar isotope dating on mineral separates (biotite and potassium feldspar) from a granodiorite in the Bulgarian part as well, as this system is more sensitive to metamorphic and tectonic overprint. The stepwise heating method, which was used in this study, allows calculating a plateau age, discarding altered or inherited domains in the studied minerals. The clarification of the age of the BBESP batholith through refinement of the dating will help not only in petrological studies, but we believe, will be essential for further geodynamic reconstructions and regional correlations.

Geological background

Regional position

The Rhodope massif, which is intruded by the BBESP batholith, lies within the eastern Mediterranean sector of the Alpine-Himalayan orogenic system. To the north it is separated from the Upper Cretaceous Sredna Gora basin by the Alpine Maritsa fault. To the northeast and to the east it is bordered by the Upper Paleogene-Neogene Maritza and Thrace basins. To the west it is separated from the Serbo-Macedonian Massif by the Strymon valley detachment (Dinter & Royden 1993; Lips et al. 2000). The southern parts of the Rhodope massif are hidden in the north Aegean Sea.

The Rhodope massif was understood as an area of large-scale nappes tectonics built up by two large nappe units, designated as Lower Terrain (Pangeon unit: mainly schists and gneisses) and Upper Terrain (metaophiolites mainly, Burg et al. 1996). Thick mylonite zones divided the both terrains. Several intermediate nape units are located also in-between the both terrains. In the Greek literature two litho-tectonic units (Papanikolau & Panagopoulos 1981) are distinguished, the lower tectonic unit (LTU) of lower-grade metamorphism and the upper tectonic unit (UTU) of higher-grade metamorphism (Fig. 1).

The metamorphic basement of the Rhodope massif is suggested to be a product of contractional and extensional stages (Burg et al. 1996; Ricou et al. 1999; Ivanov et al. 2000). The first stage caused moderate temperature high pressure (MT-HP) to high-temperature moderate pressure (HT-MP) regional metamorphism of the rocks, south-directed thrusting and significant thickening of the crust. Subsequent high-temperature amphibolite facies metamorphism and migmatite and granite formation are related to the extensional collapse of the thickened crust. During this stage migmatites and granites were emplaced within the core of several domes (Byla reka, Kessebir, Central Rhodopean and West Rila-Rhodope) and along a system of detachment faults (Burg et al. 1990; Turpaud 2006). The former two main LTU and UTU of (Papanikolau & Panagopoulos 1981) are refined and explained by extensional tectonics: migmatites and granites in the cores of the domes are referred to the Arda Unit, consisting of mainly Variscan rocks (e.g. Liati & Gebauer 1999; Peytcheva et al. 2004; Turpaud 2006); the overlaying Krumovitsa, Startsevo, Madan and Assenitsa Units (Ivanov et al. 2000), corresponding with the UTU of Papanikolau & Panagopoulos (1981) and Rhodope Terrain of Turpaud (2006) consist mainly of Mesozoic protoliths (Sarov et al. 2003; Ovcharova 2005; von Quadt et al. 2006; Turpaud et al. 2006). The top of this succession is occupied by the Parvenets Unit, recently included into the so called Thracian Unit of Sarov et al. (2006). Upper Cretaceous to Mid-Tertiary plutonic rocks (Jaranov 1960; Meyer 1968, 1969; Kronberg & Raith 1977; Christofides et al. 1998 (and references therein); Peytcheva et al. 1998; Soldatos et al. 2001a, b) are exposed in the domes (LTU) and in the UTU.

During post-collisional extension a set of graben depressions formed. They are filled with sediments of Eocene-Oligocene age and the products of an acid to intermediate volcanism (Eleftheriadis 1995; Eleftheriadis et al. 1989; Harkovska et al. 1998; Yanev & Bardintzeff, 1997; Marchev et al. 1998; Christofides et al. 2004).

The granitoids of the BBESP are intruded into the Mesozoic high-metamorphic rocks of the UTU, corresponding with the Madan Unit (Ivanov et al. 2000), or to the Rhodope Terrain (Turpaud 2006) in Bulgaria. Geophysical studies (Riazkov et al. 1993) reveal that the whole BBESP magmatic complex is a relatively flat batholith elongated south-eastward. It has a tabular shape and is inclined to the northeast and northwest, where its thickest part (4 km) appears (Riazkov et al. 1993). Hosting rocks are amphibolitic and two-mica gneisses, marbles and amphibolites. Contact metamorphism phenomena are of very limited extension.

Petrography

The BBESP batholith consists mainly of granodiorite (GRD), biotite granite (GR) and two-mica granite (TMG). In the Greek part (Elatia-Skaloti) the GRD is a medium- to coarse-grained (hornblende)-biotite grano-diorite, that varies locally from quartz-diorite through quartz-monzodiorite and tonalite to granite (Fig. 1). The outer northern and central-west parts of it become porphyritic with large K-feldspar megacrysts. The biotite GR to leucogranite cut the GRD through the whole outcrop in the form of non-mappable dykes that vary in thickness from several centimetres to several metres. It is a fine- to medium-grained rock. The east-southeastern part of the batholith (Paranesti) is occupied by the TMG, which intrudes the GRD. It resembles the GR the only difference being the presence of white mica. Pegmatitic and aplitic dykes are widespread.

In the Bulgarian part (Barutin-Buynovo) the granodiorite also predominates, passing in some places into quartz-diorite in the neighbourhood of the country marbles. The marginal parts are porphyritic and contain large xenoliths of marbles and gneisses. The granodiorite is medium-grained hornblende-biotite variety and sometimes some monzodiorites and quartz-monzonite varieties may be observed too. Dykes of fine-grained biotite granodiorite-porphyry and biotite granite-porphyry cut the granodiorite. Aplitic and pegmatitic veins occur in all outcrops.

Table 2 shows the average mineralogy and chemistry of BBESP batholith together with the mineralogy and chemistry of the analyzed samples. Details on mineralogy and petrography are given in Kamenov et al. (1974), Soldatos (1985) and Soldatos et al. (2001a, b). What is evident is that the granodiorites in the Greek and in the Bulgarian parts are similar and probably they represent rocks of one and the same magma batch.

Geochemical characteristics and constraint on the geotectonic setting

In the Greek part of the BBESP batholith the silica content ranges in the GDR from 59.8 wt.% to 70.5 wt.%, in the GR from 70.5 wt.% to 73.6 wt.%, and in the TMG from 68.6 wt.% to 75.0 wt.%. In the Bulgarian part the silica content ranges from 61.6 wt.% to 69.71 wt.% for the granodiorites (Table 2). Published major oxides confirm the genetic integrity of the granodiorites from the both parts of the batholith.

BBESP samples show calc-alkaline to high-K calc-alkaline affinities. In terms of the alumina-saturation index, they have meta-luminous to slightly peraluminous character.

Geochemical discrimination diagrams (Soldatos et al. 2001a, b) suggest different tectonic setting for the GDR, GR and TMG. On the R1-R2 diagram (not shown) of Batchelor & Bowden (1985), the GRD samples clearly plot in the pre-plate collision granites field, the TMG samples plot in the syn-collision granites field, while the Gr sample straddle the above two fields (Soldatos et al. 2001a,b). It must be emphasized here that the overlap around field 6 is predictable since all granitoids evolve towards minimum melt compositions. Jones et al. (1992) based on the same diagram suggested a post-collision uplift tectonic setting for the Greek part of the batholith although their data plot also in the pre-plate collision granites and late-orogenic granites fields (G1, corresponding to GDR) and in the late-orogenic granites and syn-collision granites field (G2, corresponding to GR and TMG). For the same part of the batholith, Soldatos et al. (2001a) suggested that the magma genesis is related to a subduction tectonic environment most likely prior to the collision. During a continental crust subduction in Eocene times (Schermer 1990), magmatism with pre-plate collision geochemical features, is generated due to the subduction of the dense oceanic lithosphere preceded the continental slab, giving the BBESP magma (c.f. Christofides et al. 2001).

Previous age data and new sampling

The first geochronological work on the BBESP pluton was done by Meyer (1968) who reported a muscovite K-Ar age of 38.3±1.1 Ma (Table 1) from a pegmatite from Tholos area, (Paranesti). Palshin et al. (1974) reported a biotite K-Ar age of 42±2 Ma from Barutin-Buynovo plutonite (S. Bulgaria). Sklavounos (1981) working on the south-eastern part of the BBESP pluton (Paranesti area) obtained biotite K-Ar ages of 29.1±1.2 to 38.5±1.5 Ma. Kotopouli (1981) suggested a possible intrusion age of Late Cretaceous or Early Tertiary on the ground of geological data in the Skaloti area. Rb-Sr isotopic data on 8 whole-rock samples of GRD from the Elatia plutonite defined an errochron age of 86.7±27.0 Ma (Soldatos 1985; Soldatos & Christofides 1986).

Soldatos et al. (2001b) presented new Rb-Sr isotopic data on micas and discussed thoroughly the age of the Greek part of the pluton. They found that the Rb-Sr biotite ages range between 34.1±1.0 to 43.0±1.3 Ma in GRD and between 36.9±1.1 to 42.0±1.2 Ma in GR. In TMG the biotite age is 39.4±1.2 Ma, while the muscovite ages range between 43.5±0.9 to 47.8±1.0 Ma. Based on the above results they concluded that the age of the TMG should be at least or not too much older than 48

Ma. This age is considered as the younger limit for the GRD, which must be older than 48 Ma, given the field relations between the two rock types. Hence, the age of the Greek part of the batholith was considered at least 50 Ma.

The samples for present study were taken from the GDR in both, the Bulgarian and Greek part of BBESP. The D5 sample (Soldatos et al., 2001a, b) represents a hornblende-biotite granodiorite (GRD, Fig. 1). It is a coarse-grained, grey to dark grey rock with 3.8 vol.% hornblende and 14.4 vol.% biotite (Table 2). Sample BB1 for the Ar-Ar dating was taken from Barutin-Buynovo pluton south of and not very far from the town Dospat (Fig. 1). Its petrographical composition is characterized as an even-grained and coarse-grained, grey in colour biotite granodiorite (Table 2). Sericite and chlorite in insignificant quantities are observed in the sample as secondary minerals.

Analytical techniques

U-Pb isotope analyses of zircons

High-precision "conventional" U-Pb zircon analyses were carried out on single zircon grains at the Institute of Isotope Geochemistry and Mineral Resources, ETH-Zurich, using an ion counter system (Finnigan MAT 262 thermal ionisation mass-spectrometer). Selected zircons were air-abraded to remove marginal zones with lead loss, rinsed several times with distilled water and acetone in an ultrasonic bath and washed in warm 4N nitric acid. All single grain zircon samples were spiked with a ^{235}U-^{205}Pb mixed tracer. Total blanks were less than 0.002 ng for Pb and U. For further details see von Quadt et al. (2002). The PBDAT and ISOPLOT softwares of Ludwig (1988, 2001) were used for calculating the uncertainties and correlations of U/Pb ratios. The calculation of the U/Pb ratios include uncertainties of the spike calibration, Pb blank measurements, common Pb correction, U and Pb fractionation and U decay constant. All uncertainties are included in the error propagation for each individual analysis. The decay constants of Steiger & Jäger (1977) were used for age calculations, and corrections for common Pb were made using the Stacey & Kramers (1975) values. The reported "Concordia age" in the figures as well as in the paper is referred to the calculation software ISOPLOT of Ludwig (2001); the $^{206}Pb/^{238}U$ mean age is based on the average of the selected analyses and the number of analyses is taken into account for the error calculation.

Hf isotope analyses of zircons

Hf isotope ratios in zircons were measured on a Nu Instruments multiple collector inductively coupled plasma mass spectrometer (MC-ICP-MS; David et al. 2001) at the Institute of Isotope Geochemistry and Mineral Resources, ETH-Zurich. High amounts of Zr in the samples did not create a significant matrix effect, and this was tested by repeated analyses of Zr-doped standard solutions. Measured $^{176}Hf/^{177}Hf$ was corrected for instrumental mass fractionation using a $^{179}Hf/^{177}Hf = 0.7325$ (exponential law for mass bias correction). Repeated analysis of the JMC-475 standard solution during the measurement session yielded a $^{176}Hf/^{177}Hf$ ratio of 0.282141 ± 14 (2 σ). For the calculation of the ε_{Hf} values the following present-day ratios of the Chondritic Uniform Reservoir (CHUR) were used: $(^{176}Hf/^{177}Hf)_{CH}$=0.282769 (Nowell et al. 1998) and $(^{176}Lu/^{177}Hf)_{CH}$= 0.0334. For age correction of the Hf isotope ratios at 52-56 Ma, a $^{176}Lu/^{177}Hf$ ratio of 0.0050 was used for all zircons.

Ar-Ar on mineral separates (biotite and K-feldspar)

Analyses of three samples for $^{40}Ar/^{39}Ar$ step-heating age were carried out in the Institute of Geology and Geophysics, Chinese Academy of Science, following the procedure described by Hu et al. (1985). The samples were ground into 60-80 mesh and packaged in an aluminium foil together with flux monitors. The flux monitors include ZBJ hornblende (132.8±3.1 Ma), ZBH-25 biotite (132.7±2.8 Ma), and international

Table 2. *Modal composition (vol.%) and chemical composition (major elements, wt.% and trace elements, ppm) of the granitoid rocks from the BBESP batholith. Data from Kamenov et al. (1974), Sklavounos (1981), Soldatos (1985), Kamenov et al. (1990), Soldatos et al. (2001a,b)*

Modal composition	Elatia-Paranesti							Barutin-Buynovo		
	GDR (33*)	(min-max)	Gr (6)	(min-max)	TMG (21)	(min-max)	sample D5	BB (19) GDR	(min-max)	sample BB1
Quartz	23.0	(11.6-39.4)	28.7	(24.0-31.7)	31.5	(26.2-37.0)	18.4	20.4	(15-37)	19.4
K-feldspar	12.4	(0.0-35.9)	36.4	(14.0-66.0)	27.1	(6.0-45.0	12.0	14.5	(5-23)	15.2
Plagioclase	48.3	(27.4-66.6)	26.3	(6.7-42.7)	34.5	(21.3-52.5)	49.6	52.0	(40-62)	50.3
Biotite	11.2	(2.0-22.0)	5.9	(3.0-13.6)	4.3	(0.0-9.0)	14.4	8.5	(9-16)	9.7
Hornblende	0.4	(0.0-3.8)					3.8	4.0	(0-5)	3.1
Muscovite	0.0	(0.0-1.2)			2.2	(0.0-5.3)				
Accessories	4.6	(0.0-8.0)	2.6	(0.0-2.2)	1.4	(0.1-6.7)	1.8	0.6	(0-4)	2.3
Chemical composition	GDR (33)	(min-max)	Gr (6)	(min-max)	TMG (25)	(min-max)	sample D5	BB (29) GDR	(min-max)	sample BB1
SiO_2	65.54	(59.84-70.50)	71.79	(70.50-73.58)	72.24	(68.64-75.04)	61.32	67.93	(61.57-70.33)	64.95
TiO_2	0.56	(0.36-0.77)	0.28	((0.16-0.45)	0.19	(0.01-0.36)	0.68	0.41	(0.04-0.90)	0.46
Al_2O_3	16.62	(14.98-18.50)	14.88	(14.06-15.47)	14.24	(12.99-15.36)	17.57	16.90	(1.85-21.14)	16.66
Fe_2O_3	1.12	(0.39-2.00)	0.51	(0.16-0.83)	0.27	(0.02-0.64)	1.96	1.14	(0.00-2.15)	0.99
FeO	2.14	(1.36-3.06)	1.04	(0.50-1.95)	1.02	(0.23-1.84)	2.45	1.71	((0.18-4.30)	2.33
MnO	0.08	(0.05-0.15)	0.05	(0.04-0.08)	0.03	(0.01-0.07)	0.09	0.09	((0.00-0.18)	0.13
MgO	1.56	(0.93-2.41)	0.58	(0.42-0.79)	0.31	(0.10-0.68)	1.99	1.25	(0.10-3.05)	3.05
CaO	3.85	(2.24-5.28)	1.70	(1.13-2.26)	1.46	(0.67-2.63)	5.17	2.51	(2.26-4.77)	3.72
Na_2O	4.07	(3.16-4.67)	3.36	(2.81-3.75)	3.87	(2.96-4.62)	4.20	3.80	(1.80-4.95)	3.69
K_2O	2.79	(1.80-4.84)	4.65	(3.69-5.73)	4.35	(2.91-6.96)	2.23	3.23	(1.70-3.98)	3.06
P_2O_5	0.30	(0.13-0.44)	0.09	(0.05-0.15)	0.07	(0.00-0.32)	0.41	0.13	(0.00-0.34)	0.12
LOI	1.26	(0.71-2.11)	1.09	(0.77-1.40)	1.04	(0.18-2.32)	1.83	0.89	(0.07-1.41)	0.59
Trace elements										
Rb	118	(83-178)	181	(144-275)	113	(46-210)	100	No trace elements are available for BB		
Sr	587	(376-835)	239	(125-344)	377	(105-1010)	835			
Ba	670	(235-1186)	650	(191-1162)	1013	(286-1602)	659			
Zn	62	(37-112)	38	(18-54)	37	(13-65)	65			
Pb	16	(9-32)	33	(17-45)	70	(27-118)	9			
Cu	4	(0-10)	4	(0-6)	7	(0-18)	2			
Ni	10	(3-21)	3	(1-5)	16	(0-27)	18			
Cr	30	(24-37)	22	(20-25)	11	(7-21)	36			
Nb	18	(11-25)	6	(0-11)	8	(3-10)	11			
Zr	235	(139-344)	165	(93-258)	144	(101-172)	296			
Y	16	(8-28)	7	(3-12)	13	(5-25)	11			
Ce	76	(24-108)	76	(21-226)	55	(24-82)	100			
Nd	32	(3-49)	25	(9-66)	20	(11-31)	43			
V	53	(26-93)	19	(9-36)	14	(9-18)	84			
La	45	(15-75)	33	(7-90)	34	(23-50)	75			
Sc	10	(6-21)	6	(3-8)	7	(5-10)	11			
U	4	(1-8)	5	(1-11)	8	(3-13)	1			
Th	15	(4-29)	23	(6-41)	17	(10-27)	18			

* average of (n) samples

standard sample BSP-1 hornblende (2,060±17.5 Ma). Both samples and flux monitors were evacuated and sealed hermetically in quartz. K_2SO_4 and CaF_2 were added in each package to monitor the interfering reactions $^{40}K(n,\alpha)$ ^{40}Ar, $^{41}K(n,\alpha)$ ^{40}Ar, $^{40}Ca(n,\alpha)$ ^{37}Ar, $^{40}Ca(n,\alpha)$ ^{36}Ar and $^{42}Ca(n,\alpha)$ ^{39}Ar. Then, the samples were put in the H8 channel of a 49-2 Reactor for 3,092 min irradiating with instantaneous fast neutron flux ($3.88*10^{14}$n/cm^{2}sec), i.e. the total amount of the integrated fast neutron flux is $1.2*10^{18}$ n/cm^2. The cadmium foil with 0.5mm in thickness was used as a shield to prevent the interference of slow neutrons. During irradiation, the sample box was circulated by cooling water with temperature 42°C at outlet and was rotated with 2 - 8 r.p.m. in order to eliminate the transverse gradient of neutron flux.

After cooling to the safe dosage, the irradiated samples were put in an Ar-extraction system to carry out step heating analysis. A high frequency oven was used to heat samples with each step of 20 min. The extracted argon was purified by CuO and sponge titanium and then introduced directly to RAG-10 mass spectrometer for argon isotope analysis, operating in the stable mode. Apparent ages were corrected for mass discrimination, memory effect, interference of K and Ca to Ar isotope, and ^{37}Ar radioactive decay. The measured correction factors for the interference of K and Ca to Ar isotope are $(^{40}Ar/^{39}Ar)_K=3.05*10^{-2}$, $(^{36}Ar/^{37}Ar)_{Ca}=2.64*10^{-4}$ and $(^{39}Ar/^{37}Ar)_{Ca}=6.87*10^{-4}$ (Hu et al. 1985). A half life of 35.1 days was adopted to correct the radioactive decay of ^{37}Ar. Uncertainty is quoted at one sigma and does not include the calculated J-factor error. Isochron regressions used the method described by York (1969).

Isotope data

U-Pb zircon dating and Hf-isotope tracing

Five zircons from GRD (sample D-5) were dated using conventional ID-TIMS technique. The analytical results are shown in Table 3 and Fig. 2. Three of the zircons (D5-3 to D5-5) are concordant at about 55-56 Ma, whereas two of them (D5-3 and D5-4) are with overlapping 2-sigma error ellipses at 55.93 ± 0.28 Ma (Fig. 2). This age is interpreted as an intrusion age of the GRD since only long prismatic to needle-like zircons were chosen, which usually are newly saturated magmatic, lacking any lead inheritance (similar to the zircons in Fig. 3a and c).

The rest two zircon grains (D5-1 and D5-2) reveal lead inheritance, pointing to possible Permo-Triassic and Jurassic ages of the inherited cores/grains (about 240 Ma for D5-1- upper intercept age of the discordia on Fig. 2, and about 130 Ma for D5-2 – apparent $^{206}Pb/^{238}U$ age, Table 3). Magmatic protoliths with similar ages and subduction/post-collisional geochemical characteristics are known from the northern parts of the Central and Western Rhodope (von Quadt & Peytcheva 2005; Cherneva et al. 2006; von Quadt et al. 2006), for the Startsevo Unit in Central Rhodope and from Northern Greece (Ovtcharova 2005) and for the Northern Greece (Turpaud 2006). Inherited cores are observed on the cathode-luminescent images of prismatic zircons from sample D5 (Fig. 3b).

The hafnium isotope characteristics of the analyzed zircon (corrected for an age of 56 Ma) are shown in Table 4. The epsilon hafnium (ε-Hf) values are slightly positive - generally in the range +1 to +3. They argue for mixed crust-mantle source of the magma. The magma was generated either by mixing of initial mantle and crustal magma or (more probably) by remelting of former subduction/postcollision related materials. The second scenario could explain the similar ε-Hf values of all zircons either with or without lead inheritance.

Ar-Ar dating

The $^{40}Ar/^{39}Ar$ incremental heating results carried out on sample BB1 are presented with their age spectra and $^{40}Ar/^{36}Ar$ *vs.* $^{39}Ar/^{36}Ar$ and $^{36}Ar/^{40}Ar$ *vs.* $^{39}Ar/^{40}Ar$ isochrons (Fig. 4 and 5). For the analyses monomineral fractions

Table 3. *U-Pb isotope data on zircons from sample E-1 (Elatia pluton, Greek part of BBESP batholith)*

Anal. No.	Size fraction μm	weight in mg	Description	U ppm	Pb ppm	$^{206}Pb/^{204}Pb$	$^{206}Pb/^{238}U$	2σ error %	$^{207}Pb/^{235}U$	2σ error %	$^{207}Pb/^{206}Pb$	2σ error %	$^{206}Pb/^{238}U$ apparent ages	$^{207}Pb/^{235}U$ apparent ages	$^{207}Pb/^{206}Pb$ apparent ages	Rho
E-1/1	75-125	0.0068	long prism colourless transp	471.3	5.768	300.90	0.010488	0.47	0.060139	1.25	0.047811	1.10	67.26	67.88	89.9	0.47
E-1/2	75-125	0.0077	long prism colourless transp	100.6	4.734	83.15	0.020511	0.52	0.134839	3.94	0.047680	3.71	130.90	128.50	84.4	0.48
E-1/3	75-125	0.0146	long prism light beige transp	285.9	2.887	766.30	0.008711	0.54	0.056323	1.10	0.046896	0.88	55.90	55.64	43.9	0.57
E-1/4	75-125	0.0125	long prism light beige transp	228.2	2.380	1250.00	0.008725	1.11	0.056438	1.31	0.046916	0.69	56.00	55.75	44.9	0.85
E-1/5	75-125	0.0147	long prism light beige transp	406.2	3.888	1584.00	0.008523	0.65	0.055226	1.29	0.046994	1.10	54.71	54.58	48.9	0.52

transp – transparent; prism- prismatic; Rho - correlation coefficient $^{206}Pb/^{238}U$ – $^{207}Pb/^{235}U$

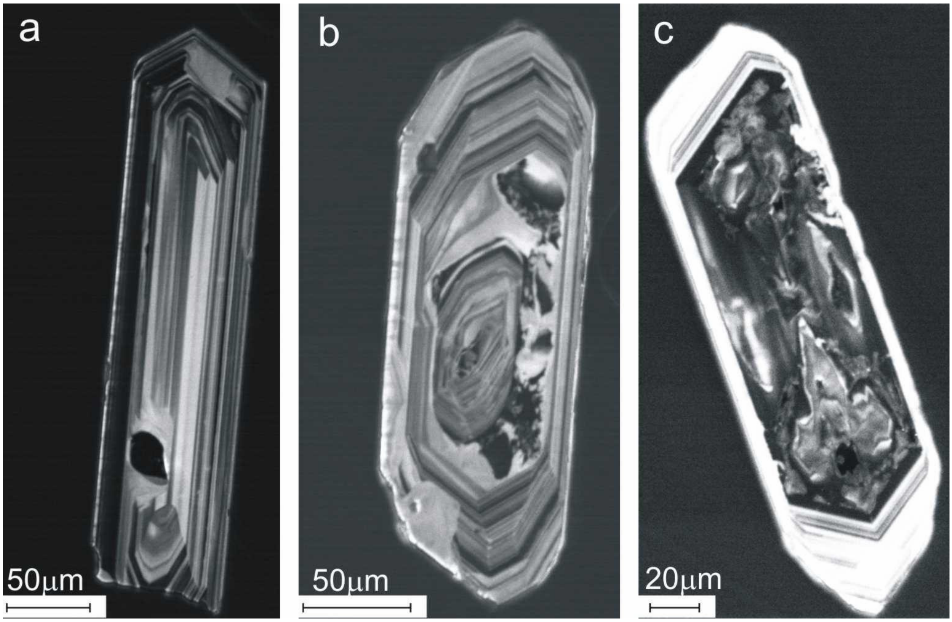

Fig. 2. Cathode-luminescent images of zircons from sample D5 (Elatia pluton, Greek part of the BBESP batholith). The long-prismatic grains (*a* and *c*) reveal magmatic oscillatory zoning, and the prismatic grain (b) has inherited core with magmatic rim

of biotite (BB1-B) and K-feldspar (BB1-F) are used. A plateau age of 49.99±0.88 Ma was obtained on the biotite fraction (Table 1) excluding the older ages of the first and last three steps (Fig. 4). The released ^{39}Ar is insignificantly high compared to the totally released. This age is confirmed by the isochron calculations of ^{40}Ar/^{36}Ar *vs.* ^{39}Ar/^{36}Ar and ^{36}Ar/^{40}Ar *vs.* ^{39}Ar/^{40}Ar. The yielded ages are 50.6±1.2 Ma and 50.6±2.1 Ma respectively. The MSWD values are acceptable (4.5 and 9.7, whereas 1 and less is statistically the best) and the age around 50 Ma should be well constrained.

The Ar-Ar results on the K-feldspar separate (sample BB1-F) are demonstrated on Fig. 5. The plateau age of 42.15±0.62 Ma is younger than that of the biotite, but it is the supported by the ^{40}Ar/^{36}Ar *vs.* ^{39}Ar/^{36}Ar and ^{36}Ar/^{40}Ar *vs.* ^{39}Ar/^{40}Ar errochrones, which are through the points makes them more confident - 44.9±3.3 Ma and 44.9±2.3 Ma (Table 1). The MSWD values of the isochrons (errorchrons?) are rather high - 43 and 85 and we use the dating on the K-feldspar fraction only to confirm once more the Eocene cooling age of the rock.

Discussion

The BBESP batholith is a composite magmatic body consisting mainly of granodiorites and granites (GRD, GR and TMG). Present high-precision ID-TIMS U-Pb data on single zircons from granodiorite (GDR) in the Greek part of Barutin-Buynovo-Elatia-Skaloti-Paranesti (BBESP) define an intrusion age of 55.93 ± 0.28 and magma generation either by mixing of initial mantle and crustal magma or by remelting of former subduction/postcollision related materials.

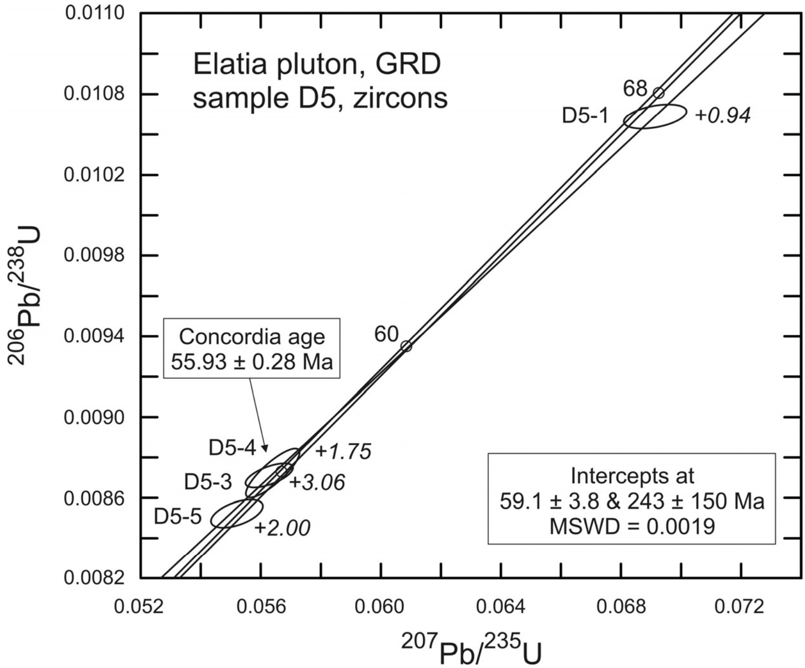

Fig. 3. Concordia diagram for zircons of sample D5 (GRD, Elatia pluton, Greek part of the BBESP batholith). The concordia age is calculated using zircons D5-3 and D5-4 (see Table 3). The epsilon Hf isotope values (corrected for an age of 56 Ma) are written in italics next to the corresponding zircon point

Ar-Ar isotope plateau age of biotite from the granodiorite in the Bulgarian part of BBESP is well constrained at 49.99±0.88 Ma. The data for the potassium feldspar are less confident, but generally in the range 42-44 Ma. These younger Ar-Ar ages we interpret as cooling ages, but, as they differ from 6 to 12-14 Ma from the intrusion age. One can explain this gap with two possible scenarios: (i) overprinting metamorphism and deformations or (ii) formation of BBESP according to the model of the metamorphic domes as a "layered tilted" magmatic body (more probable).

Soldatos et al. (2001a, b) carried out extensive field, petrological and geochemical studies, combined with strontium and oxygen isotope data to suggest that GRD, GR and TMG in the Greek part of the pluton comprise three distinct rock groups that do not have a common magma source and evolution. They also concluded that the south-westward decrease of the Rb-Sr biotite ages could be attributed to a thermal/tectonic event, very probably to the thrust between the Upper and Lower tectonic unit (Fig. 1, inset) of the Rhodope, located to the southwest of the batholith. The discrepancy in the Ar-Ar biotite and K-feldspar from the GDR (from 50 to 42, this study) are supported by the former difference within the Rb-Sr ages of muscovite (44-48 Ma) and biotite (34-43 Ma), the wide range of biotite ages, even the difference in K-Ar ages (29-42 Ma; Palshin et al. 1974; Sklavounos 1981; Soldatos et al. 2001b). The spreading of muscovite and biotite ages in the TMG and GRD, respectively suggests high temperature of the geological processes – they had exceeded the blocking temperature of

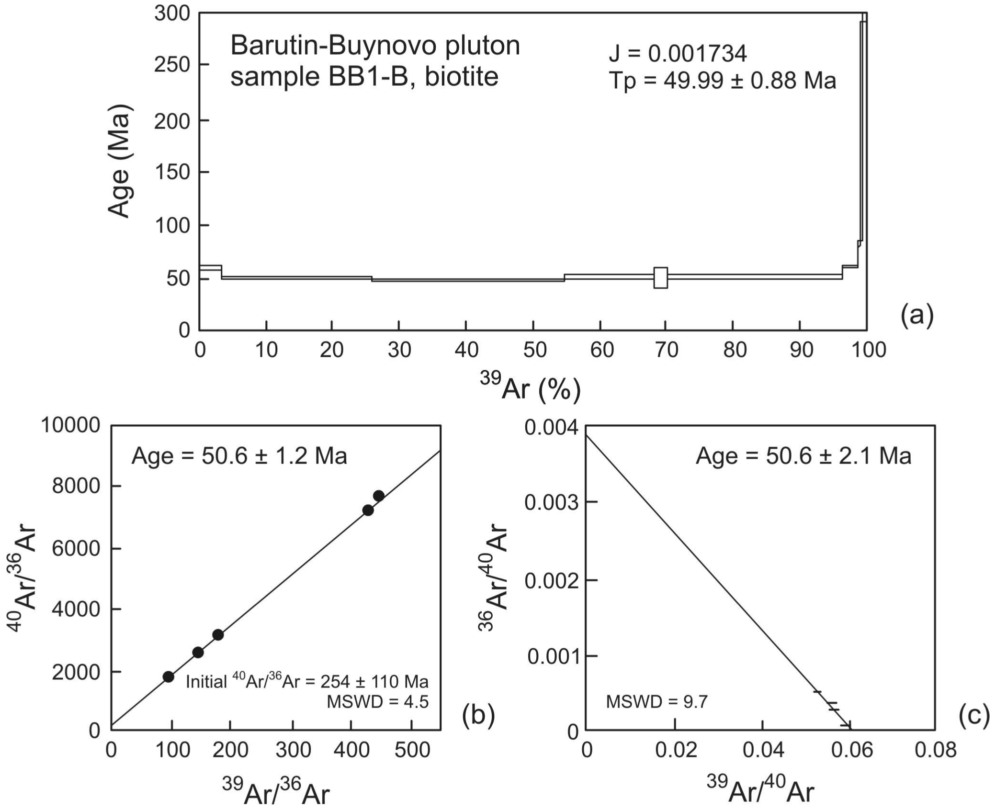

Fig. 4. $^{40}Ar/^{39}Ar$ age spectra (a) and $^{40}Ar/^{36}Ar$ - $^{39}Ar/^{36}Ar$ (b) and $^{36}Ar/^{40}Ar$ - $^{39}Ar/^{40}Ar$ (c) isochrons of biotite sample BB1-B (granodiorite, Barutin-Buynovo pluton, Bulgarian part of the BBESP batholith)

biotite (300±50 ºC) and probably had reached up the blocking temperature of muscovite (550±50 ºC), or just parts of the magmatic muscovite were reset by the overprinting event. If we suppose an overprinting metamorphism at certain temperature conditions then it should have left some traces in the petrology of the rocks, which is not the case.

Alternatively, a "layered tilted" magmatic body, related to postcollisional collapse, extension and doming (e.g. Turpaud 2006; the core-complex model, proposed for the Central Rhodopean Dome by Ivanov et al. 2000 and Sarov & Gerdjikov 2001) could interpret the general rejuvenation of the mica ages from NE to SW, to the centre of the Dome (Pangeon). In the periphery (in NE direction) the tilted blocks and set of graben depressions are formed, filled with sediments and volcanics of Eocene-Oligocene age. By "tilting", one can explain the younger ages in the SW sector of the batholith (34 Ma) as it cooled some 9 Ma after the NE sector (43 Ma). In this way biotite ages define the post intrusive regional cooling history.

The new U-Pb zircon data of the present study, being in accordance with the results of Soldatos et al. (2001b), establish an undisputable Late Paleocene magmatic age of 56 Ma for the GRD. This Eocene age is supported by the new 50 Ma Ar-Ar biotite cooling age. Consequently, the plutonic

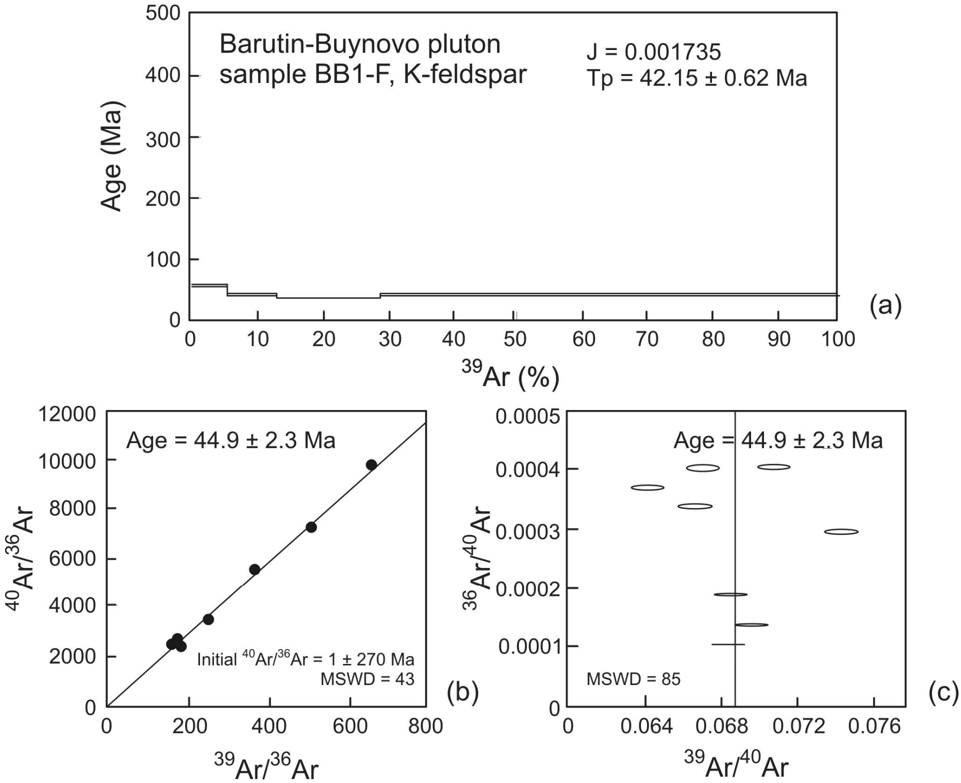

Fig. 5. $^{40}Ar/^{39}Ar$ age spectra (a) and $^{40}Ar/^{36}Ar$ - $^{39}Ar/^{36}Ar$ (b) and $^{36}Ar/^{40}Ar$ - $^{39}Ar/^{40}Ar$ (c) errorchrons of K-feldspar sample BB1-F (granodiorite, Barutin-Buynovo pluton, Bulgarian part of the BBESP batholith)

magmatism in the UTU of the Rhodope massif is considerably older (Eocene or older) than that in the Lower Unit (Pangeon)/Arda Unit. Similar ages are published recently in Bulgarian part of the West Rhodopes - Smylyan pluton (43 Ma, Ovtcharova et al. 2003, U-Pb on Zr) and some of the small granite bodies along the Yugovo River (43 Ma, Ovtcharova et al. 2003), pegmatites around the village Dolen (Arnaudov et al. 1969, 1974 – 52-49 Ma, U-Pb on Zr), Pripek granite in Startsevo allochthon (Ovtcharova et al. 2003 – 53 Ma, U-Pb on zircons and monazites). Eocene ages are obtained also for the Sithonia complex in the UTU (Christofides et al. 2001).

The Hf-zircon and the Sr-WR isotope data suggest mixed crust-mantle source of the GDR magma. Traditionally they are related to a subduction tectonic environment, most likely prior to the collision. Peraluminous or metaluminous melts with such isotope peculiarities could be also produced trough melting of amphibolitic or basaltic rocks under pressure of 8-16 kbar and temperature of 850-1100 ºC. This scenario is supported by the Hf isotopic composition of the analyzed zircons of sample D5 (Table 4). The AFC modeling (Soldatos et al. 2001a) employed to test this hypothesis used the trace element abundances of the less evolved GRD rocks as the initial composition, which was modified through fractionation and assimilation of different proportion of the basement metamorphic rocks. Potential source rocks for GR are gneisses and rocks of tonalitic composition. Melts produced by dehydration melting of tonalitic rocks under

Table 4. *Hf-isotope data of zircons from sample D5 (Elatia pluton, Greek [art of BBESP batholith)*

Lab. N	Age (Ma)	$^{176}Hf/^{177}Hf$	2 σ error	Epsilon Hf today	Epsilon Hf T 56	Epsilon Hf T core
E-1/1	240 (?)	0.282769	0.000001	-0.11	0.94	4.37 (?)
E-1/2	150 (?)	0.282807	0.000001	1.24	2.28	4.03 (?)
E-1/3	56	0.282829	0.000006	2.02	3.06	
E-1/4	56	0.282792	0.000005	0.71	1.75	
E-1/5	56	0.282799	0.000007	0.95	2.00	

*Abbreviations: s – small; *During analysis the $^{176}Hf/^{177}Hf$ ratio (JMC 475 standard) of 0.282141 ± 14 (2 sigma) is measured using the $^{179}Hf/^{177}Hf$ = 0.7325 ratio for normalization; **For the calculation of the εHf values the present-day ratios $(^{176}Hf/^{177}Hf)_{CH}$= 0.282769 and $(^{176}Lu/^{177}Hf)_{CH}$= 0.0334 are used, and a $^{176}Lu/^{177}Hf$ ratio of 005*

temperature of 825-850 ºC and pressure of 5-8 kbar are similar in composition to the GR rocks.

With the new precise ages of the BBESP we could point out that Middle and Late Alpine granitoid plutonism in Rhodope area may be distinguished into the following chronological three groups: (1) Upper Cretaceous plutons (fragments of the Rila-West Rhodope batholith and possibly Dautovo-Kresna pluton; (2) Paleocene - Eocene plutons (BBESP batholith, Smylyan pluton, Yugovo River granites, some of the pegmatites around the village Dolen, Pripek granite, Sythonia pluton etc.); (3) Oligocene plutons (Central Pirin pluton, Teshevo pluton, Osogovo pluton, Xanthi pluton etc.). Mixed geotectonic setting is suggested for most of these plutons – between island-arc and collision-related when only geochemistry is applied. More accurately conformity of all these three groups with the geodynamic environment requires much detailed work in commitment with modern studies in the fields of structural geology, geochemistry, petrology and geophysics, but the obtaining of precise age data, like the presented for BBESP undeniably will facilitate such task.

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