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Zircon U-Pb dating method for the solution of geological problems in the menderes massif (western TÜRKIYE): example of the age of granulite-facies metamorphism

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Abstract

The late Neoproterozoic poly-metamorphic basement of the Menderes Massif can be divided into two units (Bozdeğ Unit and Birgi Unit) with distinct metamorphic and magmatic histories. Birgi Unit consists of high-grade paragneiss - schist intercalation and (meta) acidic – basic intrusions. Granulite facies metamorphism has been preserved only in a few localities and is characterized by the orthopyroxe relics. Pelitic granulites and hypersthene orthogneisses (charnockites) form the main lithologies. The zircons in charnockites contain featureless overgrowth and rim textures representing metamorphic growth on magmatic cores and inherited grains. The protolith of the charnockites, yielded a crystallization age of ~ 590 Ma. U/Pb SHRIMP and LA-ICP-MS zircon and monazite ages from granulitic rocks gave an age of ca. ~580 Ma, which is interpreted as the time of granulite-facies metamorphism in the basement of the Menderes Massif. These data indicate that the Menderes Massif experienced acidic magmatic activity and granulite-facies metamorphism in Ediacaran. Furthermore, the basement rocks have been overprinted by a Barrovian-type Alpine metamorphism in Eocene (~ 42 Ma). The granulite-facies metamorphism in the Menderes Massif can be attributed to the orogenic event (600-500 Ma; Malagasy orogeny) causing the final amalgamation processes for the northern part of the Gondwana.

Keywords: *Zircon U-Pb dating, Granulite-Facies Metamorphism, Menderes Massif, Gondwana*

1. Introduction

The Menderes Massif (MM) in western Anatolia substantially shows a complex tectono-stratigraphy as a result of late Alpine compressional tectonics. This crystalline complex is tectonically overlain by the Afyon Zone in the northeast, east, and south, and the eastward continution of the Cycladic Complex in Turkey in the west and northwest. It is covered by Neogene sedimentary/volcanic units (Figure 1a). The Menderes Massif is made up of an Ediacaran – early Cambrian basement and unconformably overlying Paleozoic – early Paleogene cover series (Figure 2). New field evidence and age data indicate that the Precambrian basement of the MM can be divided into two tectonic units; i) Bozdağ Unit (632 – 627 Ma) and ii) Birgi Unit (590-535 Ma). Both units are unconformably overlain by common upper Permian metamorphic cover indicating the tectonic juxtaposition before latest Paleozoic. Bozdağ Unit (older than 630 Ma) is made up of a continuous metasedimentary succession which is dominated by metapelites at the lower levels and passes upwards into marbles with a transition zone composed of black quartzites, phyllite and dolomite intercalation. Additionally, it contains acidic and basic magmatic rocks dated at 632-627 Ma and has subjected to a single-stage Alpine metamorphism (Eocene) under lower greenschist- to upper amphibolite-facies conditions. Tectonometamorphic evolution of the Bozdağ Unit is out of scope of this study. Birgi Unit composes of partially migmatized paragneisses and metapelites forming a regular and continuous sequence. Granitoid and gabbroic rocks have intruded into this metaclastic sequence at various stages of the Malagasy (Pan-African) orogeny. Relict mineral assemblages representing the granulite-, eclogite-, and amphibolite-facies conditions clearly reveal the polyphase metamorphic character of the Birgi Unit (Candan and Dora, 1998; Candan et al. 1995, 2001, 2011, 2016a; Oberhänsli et al., 1997; Koralay, 2015). The intrusion ages of granitoid and gabbroic rocks and the times of the polyphase metamorphisms affecting the basement rocks have been a matter of interest for a long time. Although the geochronological studies in the MM started before the 2000s and have increased until today, when considering the complex tectono-metamorphic history of the MM, more precise dating studies are still needed (Figure 2). It can be seen that U-Pb dating methods from minerals such as zircon and monazite offers the best solution to understanding this complex magmatic – metamorphic evolution. In the following sections, studies on the intrusion ages of the charnockitic granitoids and the ages of the granulite-facies metamorphism affecting the Birgi Unit of basement will be

summarized. This study is mainly based on the paper published by Koralay (2015) and additionally, previously unpublished data is compiled.

2. Methods

Heavy minerals were obtained using routine procedures that involved steel jaw-crusher, steel roller-mill, wilfley table and magnetic separator. Zircon concentrates separated from the non-magnetic fraction using heavy liquids, and single zircons were then handpicked under a binocular microscope at the Department of Geological Engineering, Dokuz Eylül University-Türkiye. Zircon grains studied by cathodoluminescence (CL) were mounted in epoxy resin discs and polished down to expose the grain centers. CL images were obtained on a microprobe CAMECA SX51 at the Institute of Geology and Geophysics, Chinese Academy of Sciences (IGG CAS) and on the Phillips XL40 SEM with attached Gatan CL at the University of Adelaide.

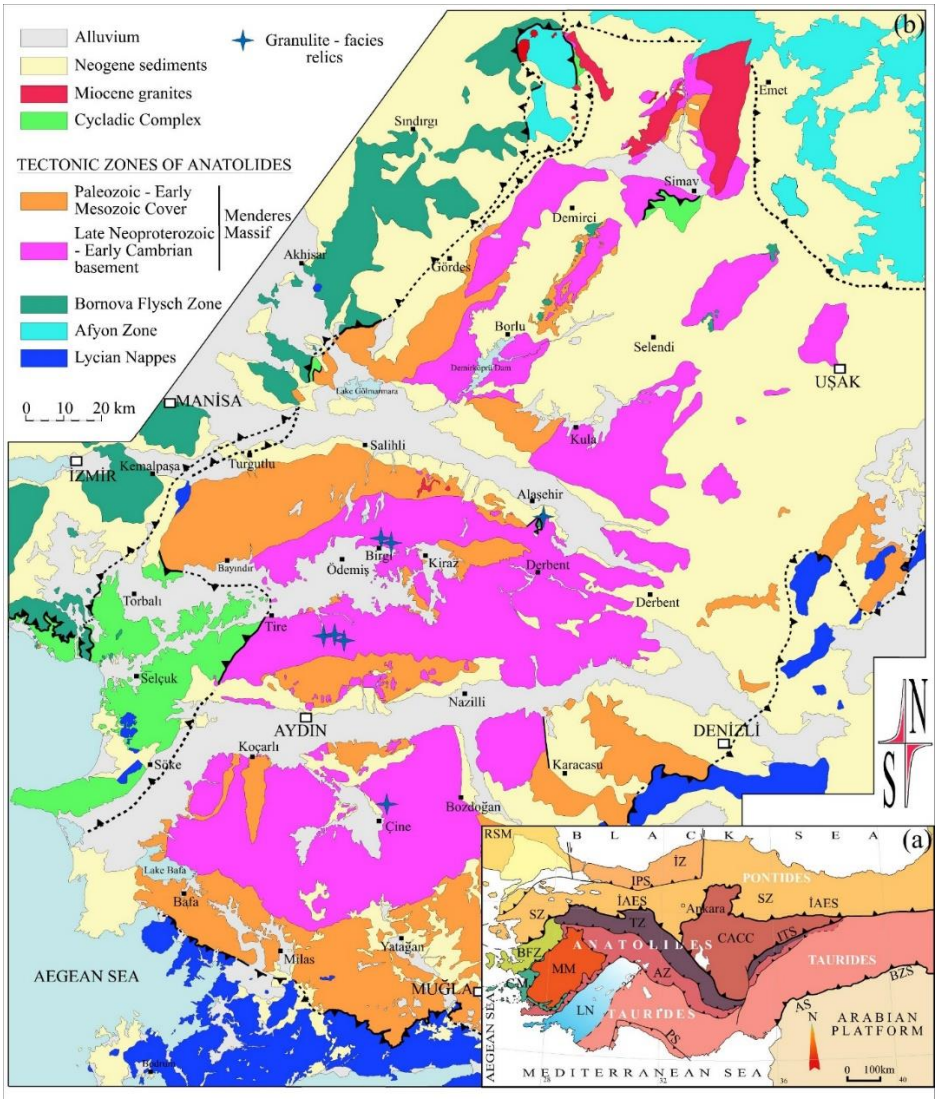


Figure 1. a) Tectonic map of Türkiye showing major continental blocks and tectonic zones (modified from Candan et al. 2016b). b) General distribution of basement and cover series of the Menderes Massif and localities of granulite-facies rocks.

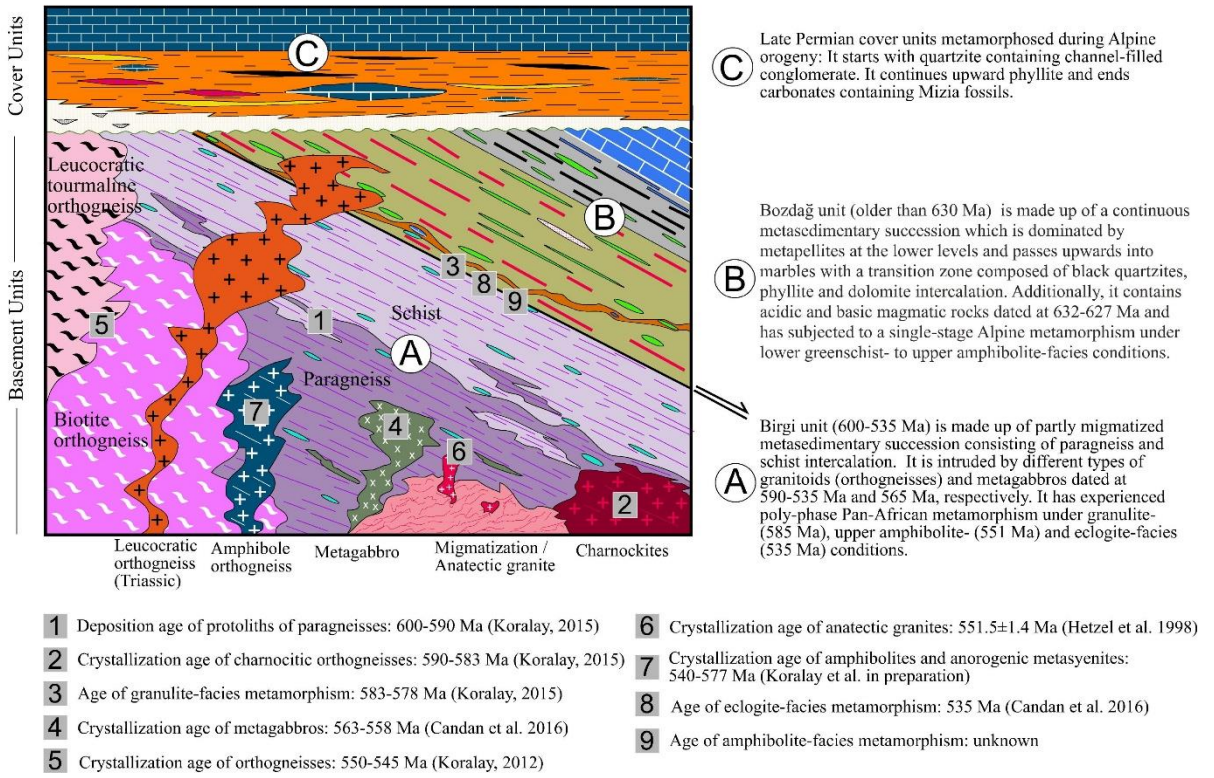


Figure 2. The generalized columnar section of the MM and magmatic and metamorphic events observed in the Birgi Unit of the massif (modified from Koralay et al. 2021).

Isotope Dilution Method: Zircons from metagranodiorite (type-1 charnockite / 05-2) were dated by the conventional U-Pb isotopic dilution method. For the conventional U-Pb isotopic dilution analyses, a standard cleaning procedure was applied in Chinese Academy of Sciences in Beijing-China. The detailed analytical procedures are given in Akal et al. (2012).

Pb-Pb Evaporation Method: Zircons from metagranodiorite (type-1 charnockite / 05-2) were dated by the Pb-Pb evaporation method. Isotope measurements of zircon were performed on a GV IsoProbe-T mass spectrometer by single-zircon evaporation in the Institute of Geology and Geophysics, Chinese Academy of Sciences in Beijing. The detailed analytical procedures are given in Akal et al. (2012).

SHRIMP Method: Zircons from metagranodiorites (type-1 charnockite / 05-2 and type-2 charnockite / 371) were dated by Sensitive High Resolution Ion Microprobe Mass Spectrometer (SHRIMP) at the Beijing SHRIMP Centre, Institute of Geology, Chinese Academy of Geological Sciences. The detailed analytical procedures are given in Koralay (2015).

LA-ICP-MS Method: Zircon dating of metagranodiorites (type-2 charnockite / 371, orthopyroxene metatonalite and type-3 charnockite / 12-28) were performed by Laser Ablation Inductively Coupled Plasma Mass Spectrometer (LA-ICPMS) at University of Science and

Technology of China in Hefei, using an ArF excimer laser system (GeoLas Pro, 193nm wavelength) and a quadruple ICP-MS (PerkinElmer Elan DRCII). The detailed analytical procedures are given in Koralay (2015).

3. Geology and mineralogy of granulite-facies rocks

The generalized lithostratigraphy of the Birgi Unit consists of a thick metaclastic sequence and voluminous syn- to post-metamorphic acidic and basic intrusions (Dora et al. 2001; Candan et al. 2011a, 2011b, 2016a; Koralay et al. 2012). The metaclastic sequence consisting of locally migmatized paragneisses and overlying mica schists forms the oldest unit. Based on the geochronological data, the depositional age of the protoliths of the paragneiss-schist sequence is constrained between 600 Ma and 590 Ma, i.e., Ediacaran (Koralay et al. 2012). These basement rocks showing high-grade metamorphisms are intruded by the protoliths of orthogneisses. Based on the mineralogical compositions and textural properties, different types of orthogneisses have been distinguished: biotite-, leucocratic tourmaline-, and amphibole-orthogneisses (Bozkurt, 2004; Candan et al. 2011b; Koralay et al. 2012). In addition, apart from these orthogneiss types, the existence of orthopyroxene-bearing orthogneisses which is the subject of this study has been documented (Candan 1995; Candan and Dora 1998; Koralay 2015). The U/Pb and Pb/Pb zircon ages of orthogneisses cluster at around ~ 550-537 Ma and are interpreted as intrusion ages of the granitic precursors of the orthogneiss (Hetzel and Reischmann 1996; Loos and Reischmann, 1999; Gessner et al. 2004; Candan et al. 2011a; Koralay et al. 2004, 2011, 2012). They are interpreted as syn- to post-magmatic intrusions, related to the latest Neoproterozoic final amalgamation of the Gondwana (Hetzel and Reischmann 1996; Candan et al. 2011b,c; Koralay et al. 2011, 2012). Additionally, the basement series are intruded by numerous poly-metamorphic basic intrusions occurring as stocks and veins. These well-preserved olivine- and biotite-gabbros were locally converted into eclogitic metagabbros and garnet amphibolites along the contacts or internal shear zones. U-Pb dating of igneous zircons from gabbroic rocks yielded ages of 563-558 Ma, indicating emplacement during the Ediacaran. On the other hand, rims of zircons from eclogitic metagabbro gave 535±3 Ma ages interpreted as the time of eclogite-facies metamorphism (early Cambrian).

The relics of phases indicating a granulite-facies metamorphism are very rare in the MM and are best preserved in Tire and Birgi areas (Figure 1b). The granulite-facies metamorphism is

mainly recognized by rare orthopyroxene relics and pseudomorphic cordierite porphyroblasts (Candan, 1995; Candan and Dora, 1998; Dora et al. 2001; Koralay, 2015). Two main types of granulitic rocks have been distinguished. These are; i) pelitic granulites with orthopyroxene and ii) charnockites which were derived from granitoids (Figure 3). Based on the protolith, textural characteristic and mineralogical composition, three types of charnockites can be distinguished (type-1: massive, medium-grained metagranodiorite, type-2: granodiorite with gneissose texture, and type-3: coarse-grained metatonalite) (Candan and Dora, 1998; Koralay, 2015). The type-1 is sillimanite-rich, medium- to coarse-grained massive rock with orthopyroxene crystals, up to 3 mm in size (Figure 3a). They consists of quartz, plagioclase, orthoclase, orthopyroxene (hypersthene), garnet, sillimanite, and biotite. Whereas, type-2 rocks are characterized by a gneissose texture with bluish orthoclase porphyroclasts, up to 5 cm in diameter, (Figure 3c). They include partly assimilated paragneiss enclaves, up to 100 m, and show a primary intrusive contact relationship with paragneisses. Their mineral assemblage can be given as quartz, plagioclase, orthoclase, orthopyroxene, garnet, and biotite. Metatonalites (type-3) are coarse-grained, dark, and massive rocks with orthopyroxene crystals, up to 1cm (Figure 3d). The granulite-facies assemblage of metatonalite is quartz, plagioclase, orthopyroxene, biotite, rutile, and ilmenite. Moreover, these high-grade rocks are cut by numerous meta-gabbroic stocks and sill-like bodies of *ca*. 565 Ma (Candan et al. 2016a). They have not been affected by granulite facies metamorphism indicating that they postdate the high-T metamorphism.

Pelitic granulites are massive to weakly foliated rocks with fine-grained granoblastic texture (Figure 4). The granulite-facies mineral assemblage is quartz, plagioclase, orthoclase, orthopyroxene, garnet, biotite and rutile. Textural evidence clearly shows the widespread overprinting of granulitic assemblages under upper amphibolite facies conditions. During this overprint, orthopyroxenes are widely consumed by fine-grained biotite. Similarly, biotite porphyroblasts are recrystallized to fine-grained new biotite aggregates or are partially replaced by plagioclase-biotite symplectites. Granulitic garnet occurs as xenoblastic porphyroblasts with quartz inclusions and is essentially almandine-pyrope solid solutions with minor grossular and spessartine components. Furthermore, they are surrounded by fine grained new garnet crystals developed during the retrograde overprint. The same garnet coronas can also be seen around orthopyroxene and biotite. Sillimanite is closely associated with biotite and occurs as fibrolite and small prismatic crystals (Candan, 1995; Koralay, 2015). In the previous studies, based on geological relationships, an age between Cambrian–Ordovician has been assumed for granulite facies metamorphism (Candan, 1995). P-T conditions for this high-T event were estimated at

about 730–760 °C and 6 kbar (Candan and Dora, 1998; Candan et al. 2011). Both type-1 and type-2 charnockites have similar compositions and plot into granodiorite field, on the other hand, type-3 charnockites are mainly tonalitic in composition (Koralay, 2015).

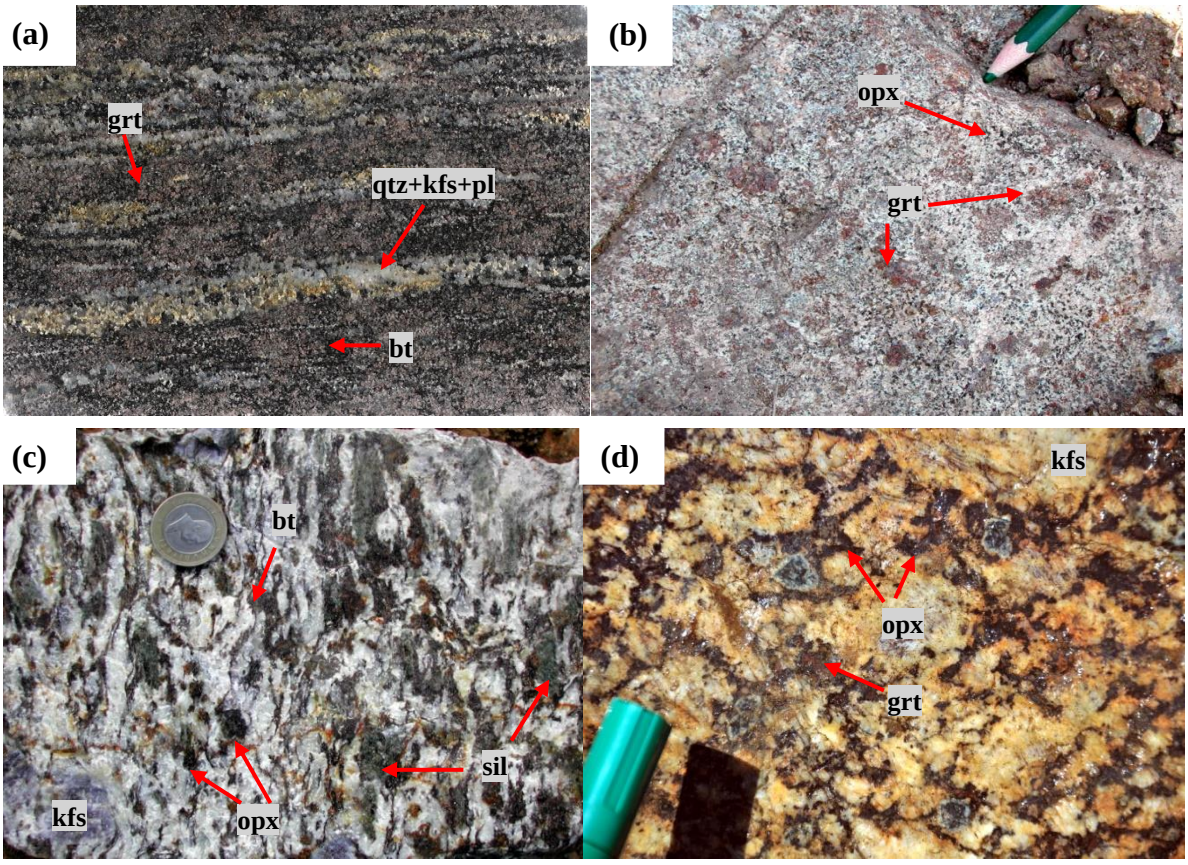


Figure 3. a) Fine-grained pelitic granulite, b) type-1: massive, medium-grained metagranodiorite, c) type-2: granodiorite with gneissose texture, and d) type-3: coarse-grained metatonalite (modified from Koralay, 2015). bt: biotite, grt: garnet, kfs: orthoclase, opx: orthopyroxene, pl: plagioclase, qtz: quartz, sil: sillimanite.

4. Geochronology

4.1. Previous Geochronological Studies on Granulite-Facies Rocks

In previous studies, the age of ca. 500 Ma (Cambrian-Ordovician) has been envisaged for granulite-facies metamorphism based on geological relationships (Candan, 1995). There are very limited geochronological age data obtained from the high-T rocks of the basement. Oelsner et al. (1997) has obtained a monazite age of 660 + 61/−63 Ma by the electron microprobe Th-U-Pb method indicating that the age of the granulite-facies metamorphism is at least Precambrian, although the error obtained from monazites by Oelsner et al. (1997) is very high.

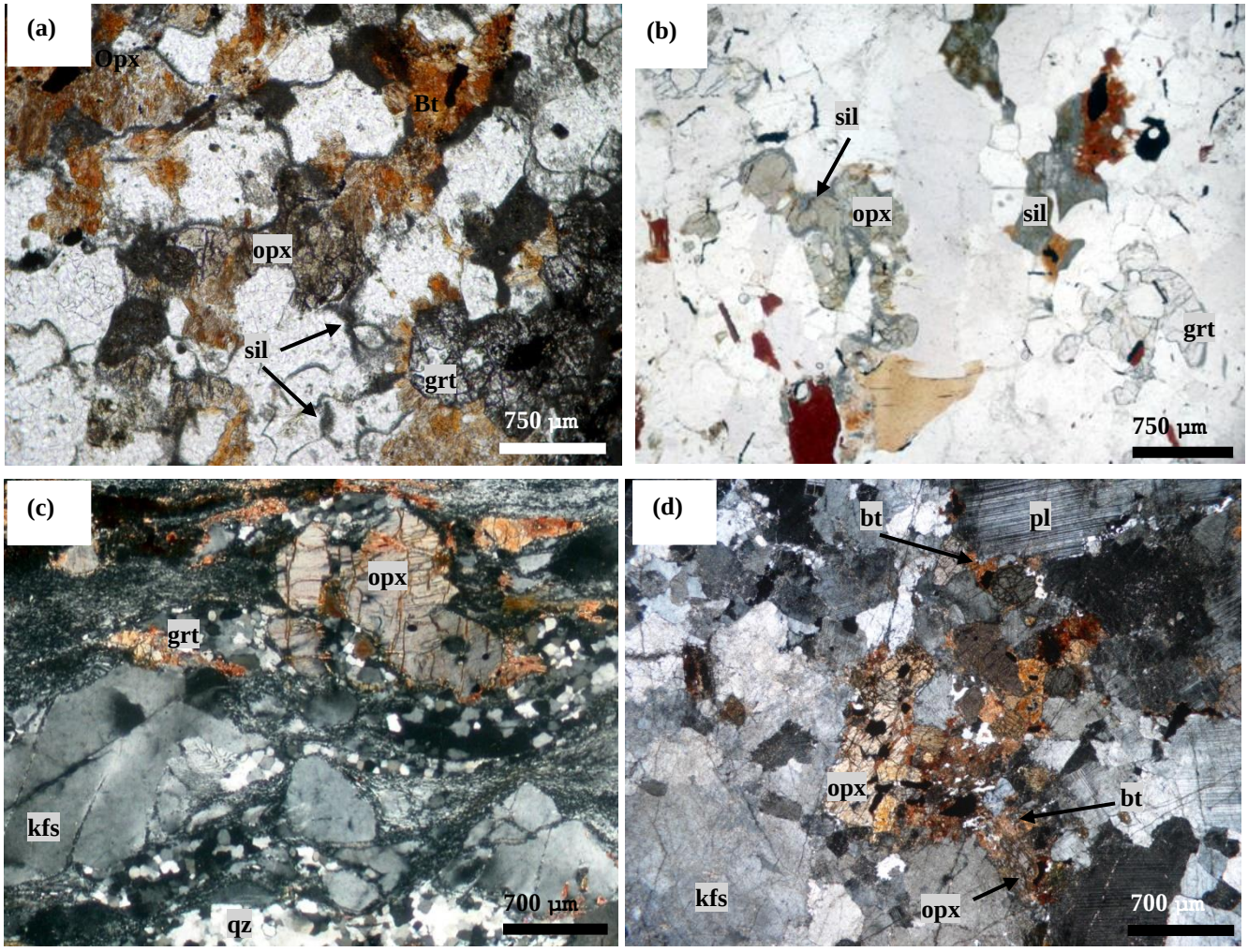


Figure 4. Generalized microscopic views of a) pelitic granulite, b) Type-1 charnockitic metagranodiorite, c) Type-2 charnockitic metagranodiorite, and d) Type-1 charnockitic metatonalite. Mineral abbreviations are the same as in Figure 3 (modified from Koralay, 2015).

4.2. Geochronological Methods Applied to Granulite-Facies Rocks

To better approximate the age of granulite-facies metamorphism in the MM, several different methods were applied. First, the U-Pb isotope dilution method was tried on different zircon populations. Four different zircon populations yielded a lower intercept age of 612±12 Ma in the concordia diagram (Figure 5). This age has been interpreted as the age of granulite-facies metamorphism. Later, the single zircon evaporation method had been widely used for dating of high-T and ultra-high-T rocks such as granulites and charnockites for many years before more modern methods such as SHRIMP and LA-ICP-MS emerged (Kröner et al. 1997, 2015; Sommer and Kröner, 2015). The reason why this method has been preferred for many years is that the round, multi-faceted grains represent typical granulite-facies zircons (Figure 6, Wu and Zheng, 2004). Based on this general view, four round and multi-faceted zircon grains from the

study area were selected and their ages were determined by ^{207}Pb-^{206}Pb single-zircon evaporation method. One of these grains (grain 1) yielded an age of 590±7 Ma at five different temperature steps (Table 1). The age of 590±7 Ma can be interpreted as the age of granulite-facies metamorphism of this sample.

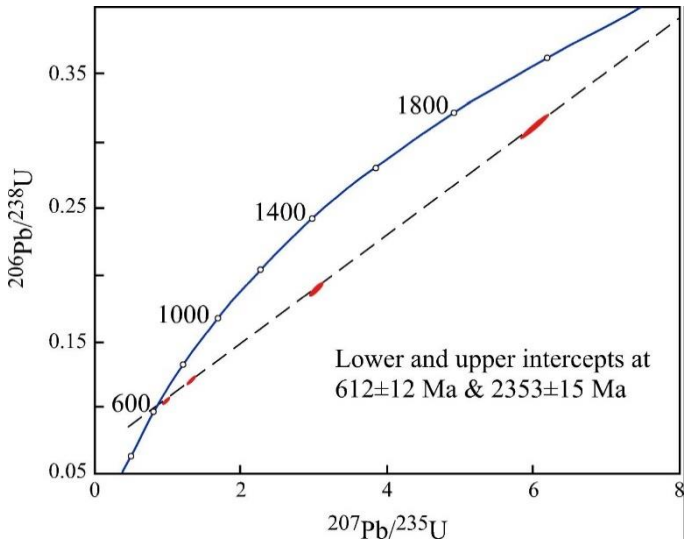


Figure 5. Concordia diagram showing isotope dilution analyses of four different zircon populations. Ellipses shown represent 95% confidence limits.

Table 1. Metamorphic zircon $^{207}Pb/^{206}Pb$ ratios obtained by the evaporation method and corresponding ages. mf: multi-faceted, e: ellipsoidal, y: yellowish, cl: clear, tr: transparent, l: large, m: medium.

Sample number	**Zircon definition**	**Grain**	**Number of steps**	**$^{207}Pb/^{206}Pb$ ratios (weighted)**	**weighted $^{207}Pb/^{206}Pb$ plateau age**
05-02	mf, e, y, cl, tr, l	1	5	0.059594±0.000019	589.9±6.9
	mf, e, y, cl, tr, l	2	3	0.063421±0.000046	722.4±15.0
	mf, e, y, cl, tr, m	3	5	0.064230±0.000025	749.2±8.2
	mf, e, y, cl, tr, m	4	2	0.219674±0.000076	2978.3±5.6

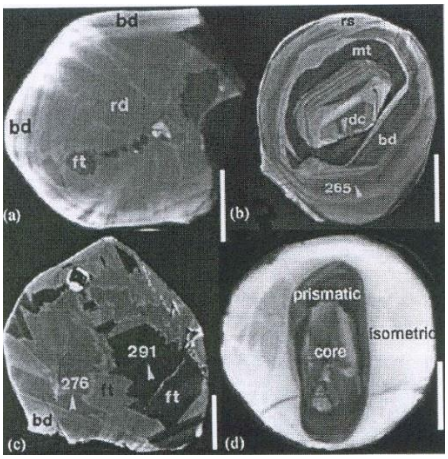


Figure 6. Typical granulite-facies metamorphic zircon CL images, a) Sector zoning, b) planar zoning, c) fir-tree zoning, d) no or weakly zoning. All images are taken from Vavra et al. (1996, 1999).

CL studies on these zircons in the following years revealed that they might have an extremely complex internal structure (Figure 7). The textural characteristics of these zircon grains can be given as; i) many of the grains contain irregular, bright luminescent featureless rims representing metamorphic growth around inherited cores, ii) Cores of the zircons have angular and fractured shapes with no zoning, flow zoning, oscillatory zoning, and sector zoning. Some of the cores have euhedral and multifaceted shapes reflecting typical metamorphic growth and display sector zoning and fir-tree zoning, iii) The majority of zircons show oscillatory and planar-zoned overgrowths representing crystallization from anatectic melts. Textural relationships clearly reveal that granulite-facies unzoned zircon overgrowth should have occurred prior to planar-zoned overgrowth.

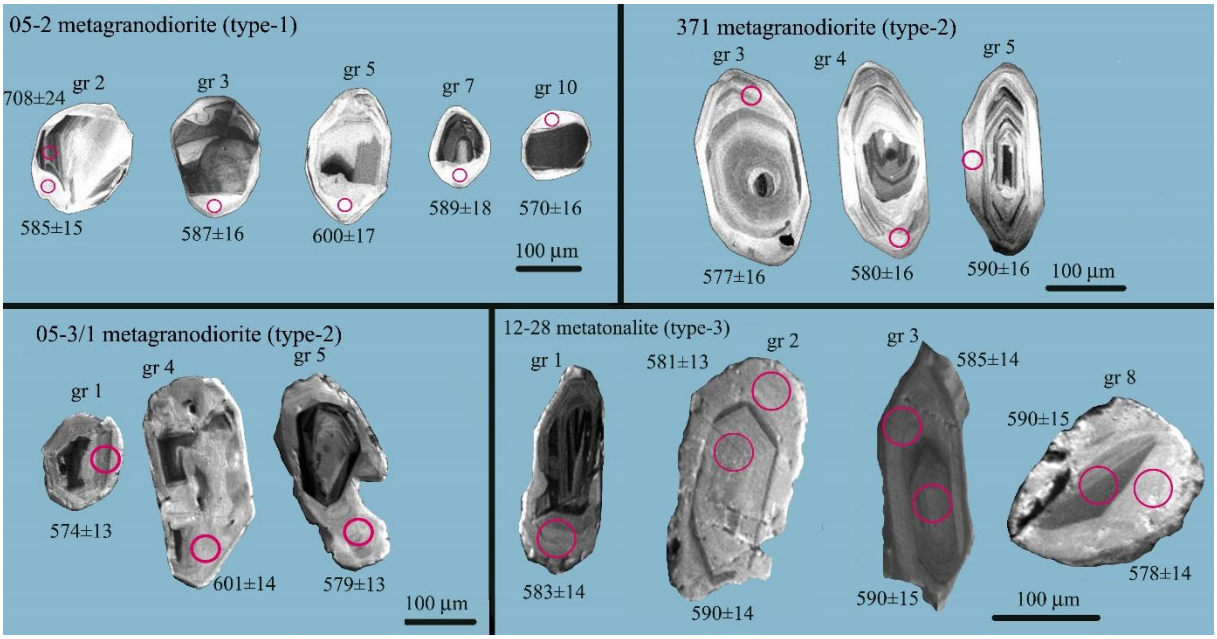


Figure 7. Cathodoluminescence (CL) images of the representative internal textures of typical zircon populations for metagranodiorite (type-1; 05-2), metagranodiorite (type-2; 371 and 05-3/1) and metatonalite (type-3; 12-28) (modified from Koralay, 2015).

Since a reliable age could not be obtained by the isotope dilution and Pb-Pb evaporation method, samples were dated using SHRIMP and LA-ICP-MS, which are the more advanced and reliable methods. Four samples, three charnockitic metagranodiorite (type-1 and type-2) and one charnockitic metatonalite (type-3), have been selected for age determinations. In order to find out the primary crystallization ages of the protoliths of the samples, core parts of the zircons showing typical oscillatory zoning were dated. Moreover, metamorphic overgrowths with Th/U ratios between 0.04 and 0.14, which are typical for metamorphic zircons (Wu and Zheng, 2004), were targeted. Magmatic domains yielded a $^{206}Pb/^{238}U$ weighted mean age of 591±6 Ma interpreted as the crystallization age of igneous precursor for type-3 charnockite (metatonalite) (Figure 8). The metamorphic rims yielded ages of 580±6 Ma, 582±9 Ma, 589±14 Ma, and 580±5 Ma from the samples 05-02, 371, 05-3/1, and 12-28, respectively (Figure 5; Koralay, 2015; see supplementary Table B). Moreover, one monazite grain yielded a weighted mean age of 578±3 Ma, which was dated by LA-ICP-MS from metagranodiorite (type-1, sample 05-2; Koralay, 2015; see supplementary Table D). This age is consistent with the U-Pb SHRIMP and LA-ICP-MS ages obtained from featureless rims of charnockitic samples. As a result, the age of ~ 580 Ma has been interpreted as the age of granulite-facies metamorphism affected the basement (Birgi Unit) of the Menderes Massif. Koralay (2015) reports biotite and whole-rock Rb-Sr isochron age of 42±1 Ma interpreted as the cooling age of the Alpine overprint on the basement.

5. Interpretation of age data and discussion

As known from previous studies, prior to the Eocene Alpine metamorphism, the basement of the Menderes Massif (Birgi Unit) has undergone a complex metamorphic history during the latest Neoproterozoic, which is ascribed to the assembly of Gondwana (Candan et al., 2011a and references therein). The metatonalite (type-3) sample from the Birgi region yields an age of 591±6 Ma (Figure 8). Based on well-defined oscillatory zoning and high Th/U ratios of zircons, this age is interpreted as the protolith age of the metatonalite. However, the metamorphic zircons from other samples yielded ~ 580 Ma, which was interpreted as the age of granulite-facies metamorphism.

As it is emphasized in many studies, two tectonothermal events, ~650-620 Ma East African orogeny and ~600-500 Ma Malagasy-Kuunga orogeny, play a crucial role in the final configuration of the Neoproterozoic to Early Palaeozoic supercontinent Gondwana (Koralay, 2015 and references therein). Both orogenic events are characterized by the existence of widespread granulite-facies metamorphic rocks. In the Mozambique Belt of the East African Orogen, granulite-facies rocks have been documented in many places and their ages are between 652 Ma and 618 Ma. However, the Late Neoproterozoic to Cambrian granulite-facies metamorphism of the second major orogenic episode (Malagasy orogeny), which is located eastern part of the East African orogeny has been reported in many places and their ages range from 600 Ma to 520 Ma (Koralay, 2015 and references therein). The age of granulite-facies metamorphism (~580 Ma) in the Menderes Massif rules out a possible genetic relationship to the East African orogeny. However, there is a close relationship between the age of the granulite-facies metamorphism in the Menderes Massif and the high T metamorphism that developed as a result of the Malagasy orogeny (600-500 Ma). Considering the inferred latest Neoproterozoic - early Cambrian paleogeographic setting of the Anatolide-Tauride Block, the granulite-facies metamorphism in the Menderes Massif can be attributed to the northward continuity of Malagasy Orogen (Koralay, 2015 and references therein).

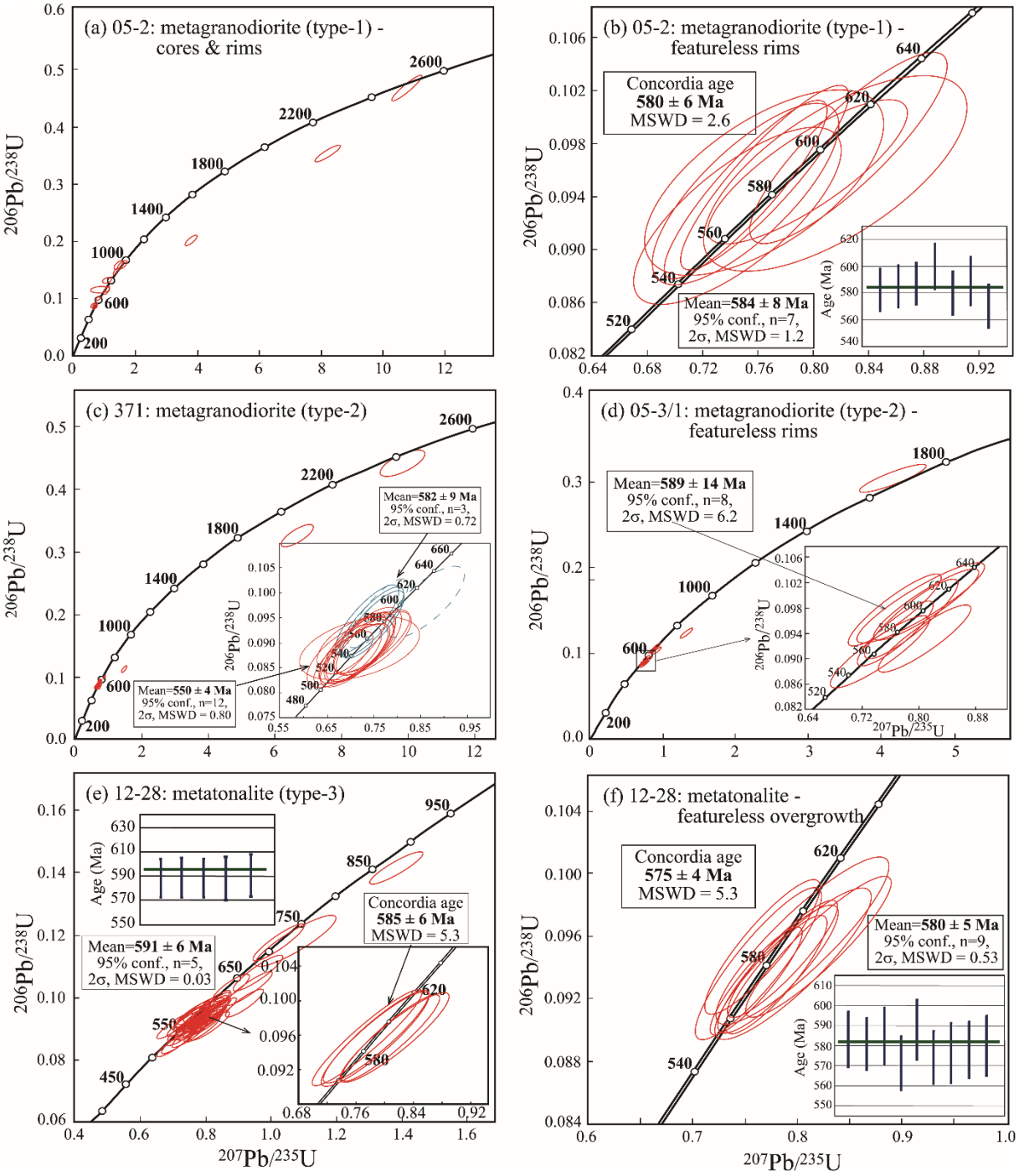


Figure 8. Zircon U-Pb concordia diagrams of metagranodiorite (type-1; 05-2), metagranodiorite sample (type-2; 371 and 05-3/1) and metatonalite sample (type-3; 12-28). All images are taken from Koralay (2015).

6. Conclusions

Mineralogical and textural evidence indicates the presence of former high-T metamorphism under granulite-facies conditions in the poly-metamorphic basement (Birgi Unit) of the Menderes Massif. Granulite-facies rocks in the Menderes Massif include orthopyroxene bearing pelitic granulites and charnockitic orthogneiss intrusions. U/Pb SHRIMP and LA-ICP-

MS zircon and monazite ages from granulitic rocks yield an age of ca. ~580 Ma, which can be interpreted as the time of granulite-facies metamorphism in the basement of the Menderes Massif. Moreover, the crystallization age of the protolith of the metatonalite, which experienced the granulite-facies metamorphism, has been determined as 591±6 Ma. Granulitic rocks were overprinted by Alpine (Eocene; 42 Ma) metamorphism. Based on the inferred latest Neoproterozoic - early Cambrian paleogeographic position of the Anatolide-Tauride Block plaeced to the north of present-day Arabia, the granulite-facies metamorphism in the Menderes Massif can be attributed to the orogenic event (600-500 Ma; Malagasy orogeny) causing the final amalgamation processes for the northern part of the Gondwana.

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