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Development of the Arabian-Nubian Shield along the Marsa Alam-Idfu transect, Central-Eastern Desert, Egypt: geochemical implementation of zircon U-Pb geochronology

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Abstract

The magmatic complex along the Marsa Alam-Idfu transect, Central-Eastern Desert of Egypt, represents the northern segment of the Arabian–Nubian Shield (ANS), which developed within the framework of the East African Orogen. The basement rocks of the Arabian-Nubian Shield have been developed through three distinct phases of magmatic activity: the island-arc, the syn-orogenic, and the post-orogenic phases. Transitioning of the magmatic phases from the syn-orogenic to the post-orogenic, identifies changing the tectonic regime from a compressional to an extensional setting. The scarcity of comprehensive regional geochronological data that rely on precise isochron methods, such as the zircon U-Pb technique, could limit the comprehensive understanding of this region's geological and tectonic history. That would raise a number of uncertainties ranging from the timing of the different magmatic activities and timing of changes in the tectonic regime to the existence of the pre-Pan-African crust in the CED. Our study provides new insights into the aforementioned uncertainties through zircon U-Pb dating of different rock units along the Marsa Alam-Idfu transect, CED, Egypt. The resulting ages ranged from 729±3 Ma to 570 ± 2 Ma, constraining the temporal evolution of the ANS in the studied region into (1) the island-arc phase, represented by a metamorphic sample with an age of 729 ± 3 Ma. (2) the syn-orogenic phase, represented by calc-alkaline and alkaline granitic samples with ages ranging from 699±4 Ma to 646±2 Ma. These two phases indicate initiation of the compressional subduction regime in the CED since 729±3 Ma and being dominated till 646 ± 2 Ma. (3) the post-orogenic phase, represented by metavolcanics, volcanic rocks, and alkaline plutonic samples with ages ranging from 623 ± 3 Ma to 570 ± 2 Ma. This phase suggests dominance of the compressionalto-extensional tectonic transition setting from 623±3 Ma to 600±1 Ma along with the Dokhan volcanism and activation of post-collision tensional regime activated at 582 ± 3 Ma. Our findings discourage the proposed

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dominance of the island-arc and syn-orogenic phases in the CED and the classical restriction of older magmatic activity to calc-alkaline granitic rocks and younger magmatic activity to alkaline granitic rocks. Additionally, we identified evidence of local magmatic sources by dating five grains with Mesoproterozoic (pre-Arabian–Nubian Shield) xenocrysts with ages ranging from 1549 ± 4 to 1095 ± 25 Ma.

Keywords Magmatic complex, Zircon U-Pb dating, Arabian-Nubian Shield, Eastern Desert, LA-ICP-MS geochronology

Introduction

The Egyptian Central-Eastern Desert (CED) represents a significant portion of the Arabian-Nubian Shield (ANS), where several temporal-spatial geologic and tectonic characteristics remain uncertain [1-4]. The ANS formed during the East African Orogeny (EAO) between ca. 900 Ma and 550 Ma. This occurred through an amalgamation of the juvenile crust by the accretion of microcontinents and island-arcs into the older continental crust of the Archean age (Fig. 1). Termination of such crustal growth was regionally marked by the development of the Dokhan Volcanics, the Hammamat molasse-sediments, and the Younger granites [5, 6].

Crystalline rocks of the CED consist of the metamorphic gneissose granites and the ophiolitic mélange, which are intruded by several granitic units [4, 7]. Basement rocks with ophiolitic and island-arc affinities are considered to be dominant in the CED [7-12]. Ophiolitic rocks are represented by dismembered serpentinite, gabbro, pyroxenite, and volcanic rocks. The island-arc terranes and the consequent granitic intrusions are traditionally divided into the Older (Gray) granitoids (ca. 800–630 Ma), predominantly comprised of rocks with calc-alkaline chemical compositions, and the Younger (Red) granitoids (ca. 630-540 Ma), primarily composed of alkaline rocks [13–17]. However, recent investigations have revealed the contemporary development of granitoids with both calc-alkaline (Gray granites) and alkaline (Red granites) compositions [2, 18–21]. The timing and tectonic setting of the eruption of the Dokhan Volcanics, occurring towards the ANS's final stages, remain a subject of debate, with differing views suggesting their eruption as a response to either the late-stage collision of East and West Gondwana or the transition between convergent and extensional tectonic settings [6, 22-25].

Early geochronological studies on the ANS basement rocks indicated the presence of pre-Neoproterozoic metamorphic suites predating the East African Orogeny [26–29]. However, more recent studies indicate the lack of any large pre-Neoproterozoic basement [18, 19, 23, 30–39]. Additionally, the timing and tectonic setting of the Dokhan Volcanics, occurring towards the ANS's final stages, remain a subject of debate [6, 22–25].

The reported ages of the basement rocks in the ANS exhibit considerable variability, posing challenges to their classification into established categories [2, 9, 18, 19, 23, 30, 35, 40]. Attempts to delineate distinct intrusive

events based on earlier studies face challenges in light of more recent data [22, 30, 34, 41–45]. Additionally, subsequent geological events affected this region with uplift and erosion. These events, including Cambrian thickening-related erosion, Devonian-Carboniferous Variscan tectonic activity, the Cretaceous initiation of the Mid-Atlantic Ridge, and the rifting of the northern Red Sea [46–52], eroded a substantial amount of the basement rocks, and might have removed the majority of the older suites [3].

This study provides new insights into the underlying uncertainties regarding the sequence of the ophiolitic mélange and granitoid emplacement and sorting of the distinct magmatic and volcanic events within the CED. In other words, new constraints on several unresolved critical issues, including the possibility of pre-Neoproterozoic crust existence in the CED, detailed chronological constraints on basement rocks, the tectonic environment of the Dokhan volcanism, and the validity of the classical approach to differentiating magmatic activities based only on a rock's apparent geochemical variations. Our approach to deal with these issues through presenting the precise and well-established zircon U-Pb geochronological results of representative samples from the CED basement that were analyzed using a laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) technique (Table 1).

Geologic setting

The term Pan-African Orogeny was first described by Kennedy (53) to describe a sequence of massive tectonic processes extended across the Gondwana supercontinent. The EAO developed the East Africa and Arabian-Nubian Shield as part of the Pan-African tectonism, through the accretion of continental fragments, and ophiolitic and oceanic island-arcs to the Gondwana margin between 870 Ma and 610 Ma [4, 22, 54]. While the ANS represents the northern part of the EAO that is represented in the Egyptian Eastern Desert by calc-alkaline to tholeiitic igneous rocks, metamorphic rocks including gneiss, schist, metavolcanics, metasediments, and ophiolite complexes [10, 55–60].

The temporal evolution of different magmatic and volcanic activities is essential in the geological and tectonic reconstruction of the ANS development. While ages of the ANS basement rocks indicate being formed during the EAO during the Neoproterozoic *[e.g., 1–9]*, pre-EAO



Fig. 1 (A) The ANS and the East African Orogen in the context of the Gondwana amalgamation (modified after [132, 133]), (B) Location of northern the ANS (represented in C) within the frame of Africa, Arabia, and Eurasia with northern ANS with previous geochronologic studies (modified after [2], . Where, OG is Older Granitoids, YG is Younger Granitoids, PD is Phanerozoic dyke, DV is Dokhan Volcanics, and Oph is ophiolitic sequence. While, G. Samra [2], Feiran area [1, 23, 134], wadi Lithi [135], wadi Kid [136], wadi Nasib and wadi Ghazalla [19], Taba area [20], Gabal Gharib [22], Gabal Dara, Gabal Zeit, Gabal Abu Harba, Gabal Qattar [30, 44], and Fawakhir, W.Ghadir, Gerf Nappe, W. Haimur, and W. Allaqi [35, 40]

Location		Elev.	Rock Type	Traditional Suite Classification	Concordant	
Lat.	Long.	m.a.s.l.			Age (Ma)	±2σ
						(Ma)
25.06247	33.78872	410	Gneiss	Metamorphic	729.3	2.7
nic						
25.05656	34.79372	150	Diorite	Older Granite	699.1	4.3
25.06247	33.78872	401	Syenite	Younger Granite	680.4	5.2
25.03839	34.74478	246	Granite	Younger Granite	647.2	3.3
25.0455	34.54231	456	Syenite	Younger Granite	645.9	1.7
nic / Dokhan er	ruptions					
25.02517	34.69506	323	Meta-andesite	Dokhan Volcanics	623.2	2.7
25.07892	33.85236	422	Andesite	Dokhan Volcanics	610.9	2.3
25.05686	34.83792	94	Dacite	Dokhan Volcanics	600.3	1.3
nic / Magmatic	emplacement					
25.04558	34.85236	516	Syenogranite	Younger Granite	581.9	2.5
25.04425	34.56633	475	Syenite	Younger Granite	576.2	1.7
25.0430	34.5570	488	Granite	Younger Granite	569.8	1.9
	Location Lat. 25.06247 25.05656 25.06247 25.03839 25.0455 nic / Dokhan en 25.02517 25.07892 25.05686 nic / Magmatic 25.04558 25.04258 25.0430	Location Lat. Long. 25.06247 33.78872 25.0556 34.79372 25.056247 33.78872 25.056247 33.78872 25.056247 33.78872 25.05247 34.79478 25.03839 34.74478 25.0455 34.54231 25.05217 34.69506 25.0586 34.83722 25.0586 34.83722 25.05458 34.85236 25.04558 34.85236 25.04558 34.56331 25.04558 34.56331	Location Elev. Lat. Long. m.a.s.l. 25.06247 33.78872 410 25.05566 34.79372 150 25.06247 33.78872 401 25.05267 34.79372 150 25.05247 33.78872 401 25.05247 34.74478 246 25.0455 34.54231 456 25.0455 34.69506 323 25.0586 34.83726 242 25.0586 34.83726 94 25.0586 34.85236 516 25.04558 34.85236 516 25.04425 34.56633 475	LocationElev. ma.s.l.Rock Type ma.s.l.Lat.Long.ma.s.l.25.0624733.78872410Gneiss25.0565634.79372150Diorite25.05624733.78872401Syenite25.0563034.79372401Syenite25.0383934.74478246Granite25.045534.54231456Syenite10Jorite123Meta-andesite25.051734.69506233Meta-andesite25.058634.8379294Dacite25.058634.8379294Dacite25.0455834.85236516Syenogranite25.0442534.5633475Syenite	LocationElev.Rock TypeTraditional Suite ClassificationLat.Long.m.a.s.l.25.0624733.78872410GneissMetamorphic25.055634.79372150DioriteOlder Granite25.0624733.78872401SyeniteYounger Granite25.0624733.78872401SyeniteYounger Granite25.0525134.74478246GraniteYounger Granite25.045534.54231456SyeniteYounger Granite25.0558634.83702422AndesiteDokhan Volcanics25.0586634.8370294DaciteDokhan Volcanics25.0455834.85236516SyenograniteYounger Granite25.0455834.85236516SyeniteYounger Granite25.0455834.5633475SyeniteYounger Granite25.0455834.5570488GraniteYounger Granite	LocationElev.Rock TypeTraditional Suite ClassificationConcordantLat.Long.m.a.s.l.MeanorphicAge (Ma)25.0624733.78872410GneissMetamorphic29.325.0556634.79372150DioriteOlder Granite699.125.0624733.78872401SyeniteYounger Granite699.125.0624733.78872401SyeniteYounger Granite647.225.0624734.74478246GraniteYounger Granite645.925.045534.54231456SyeniteYounger Granite645.925.045734.6950652.3Meta-andesiteDokhan Volcanics623.225.058634.8379294DaciteDokhan Volcanics60.325.058634.8379294DaciteDokhan Volcanics60.325.058634.8379294DaciteDokhan Volcanics61.925.0558534.8526516SyenograniteYounger Granite51.925.0442534.5633475SyeniteYounger Granite56.225.043034.5570488GraniteYounger Granite50.8

Table 1 The samples examined for this study

Elev. (m.a.s.l.) means elevations in meters above sea level

were reported only from the metamorphic complexes of Sa'al and Feiran-Solaf with ages of ca. 1030-935 Ma [1, 23]. Meanwhile, the youngest ages, from the Younger granites suite, fall between approximately 630 Ma and 580 Ma [2, 8, 19, 20, 30, 41, 61-64]. Tectonically, the compressional regime of the arc-continent collision was dominated between >700-630 Ma, where the final collisional (late-stage subduction) event occurred ca. 630 Ma [65, 66]. While the period between 630 Ma and 580 Ma is considered as a compressional-to-tensional transition period, and the post-collision tensional regime activated < 610 Ma [65-67]. The Dokhan Volcanics are divided into two sequences separated by conglomerates and/or an unconformity: (1) an older mafic sequence with basaltic, and esitic, and dacitic compositions and ages ranging between 635-620 Ma [68, 69] and (2) a younger felsic sequence with rhyolitic, rhyodacitic, ignimbritic, and vitric pyroclastics compositions and ages ranging between 618-590 Ma [68, 69]. The timing overlap of the Dokhan eruption and the late subduction to tectonic extension allows for arc subduction and/or within-plate models to explain Dokhan evolution [65, 70]. The petrogenetic studies of the Dokhan suggest development in: (1) a subduction setting [69, 71-73], (2) an extensional setting [74, 75], and (3) a transition setting between subduction and extension [63, 68, 76, 77]. While, a setting of a transitional environment became more popular in recent studies [65]. After ANS construction, subsequent geological events, including the Cambrian thickening-related erosion, the Devonian-Carboniferous Variscan tectonic activity, and the Cretaceous initiation of the Mid-Atlantic Ridge, have eroded a substantial amount of the basement rocks, especially the older suites, and further reshaped the outcropped rock units [46-51].

The Egyptian Eastern Desert can be broadly divided into three domains, from south to north: the Southern (SED), the Central (CED), and Northern Eastern Desert (NED) regions [22]. The basement rocks of the CED primarily consist of metamorphic, granitoids, and volcanic sequences, which can be broadly categorized into four litho-tectonic units [35]: (1) domal metamorphic and migmatitic sequence, which represents the lower structure unit; (2) the eugeoclinal thrust sheet [35], which consist of the ophiolitic and island-arc assemblages. These rocks are low-grade metamorphic rocks that tectonostratigraphically overlies the Meatiq dome sequence; (3) various volcanics and metasediments of the Dokhan Volcanics and the Hammamat molasse-type sediments, respectively. These unconformably overly the eugeoclinal units; and (4) late- to post-orogenic granitoids, which intrude all the previous units [78-84]. Crystallization ages of these granitoids are essential in the evolution of the Egyptian CED [35, 85].

The domal metamorphic sequence represents the lithostructural lower unit in the CED [14], represented by a series of double plunging asymmetric antiforms [35]. These antiforms signify the ductile root of a major subhorizontal thrust nappe [42], tectonically associated with the Najd Faults System [35, 86]. These domal structures are rimmed by the ophiolites and island-arc sequences. The CED ophiolitic sequence represents the remnants of oceanic crust from the Mozambique Ocean that thrusted over the ANS juvenile crust during the island arc terranes accretion and collisions during the EAO [14, 54, 87, 88]. These ophiolites can be divided into older and younger metavolcanics that are intruded by subduction-related granitoids [35, 45]. Subsequently, the Dokhan Volcanics erupted between ca. 630 Ma and ca. 590 Ma, exhibiting a range of basic to acidic compositions influenced by the fractional crystallization of basaltic magma and minor crustal contamination [6, 71, 89]. The tectonic setting associated with the Dokhan Volcanics eruptions remains debatable, whether it was activated as a response to the collision of East and West Gondwana or marked the transition between convergent and extensional tectonic settings [6, 22–25]. The granitic intrusions were subdivided based on field observations and bulk chemical compositions into two main groups; (1) an older group (ca. 750-610 Ma) referred to as the "Gray or Calc-alkaline" [90], the Older granites [15], syn- to late-orogenic [2, 14], subduction-related G1 [91], or G α granites [4] and (2) a younger group (ca. 622-543 Ma) referred to as the Red or Alkaline granites [90], Gattarian [17], Younger granites [15], late-orogenic [2, 92], suture-related G2 [91], or $G\beta$ granites [4]. These classifications do not provide key geochemical or geochronological information, and thus, are difficult to use when assessing the tectonic development of this region in a more detailed manner.

The structural fabric of the CED is concluded in three major deformational events [93]. These are represented by (1) Early NW-SE shortening (D1) associated with the compressional (subduction) tectonic regime associated with the accretion of island arcs and obduction of ophiolites over old continent. D1 produced NNW-directed thrusts and ENE–WSW oriented folds in the CED. These thrusting events were suggested to remain active in CED until ca. 650 Ma at Sibai dome [81, 94, 95], and until ca. 630 Ma at Meatiq and Sibai domes [35]. (2) the D2 structures were developed by changing the tectonic regime from compressional arc-accretion setting to the sinistral transpressional setting at ca. 650 Ma, which was marked by an oblique collision between the Arabian-Nubian Shield and the Nile Craton. This produced NW-trending upright folds, NE-dipping and SW-dipping thrusts, and discreet NW-SE tending shear zones in the CED [93]. (3) the D3 structures were developed during the tectonic transition regime between the compressional and extensional settings between ca. 620 Ma and ca. 580 Ma [93, 96]. The D3 was associated with the exhumation of the CED domal structures, intrusion of gabbroic and granitoid rocks, and development of major NW-trending sinistral shear zones and strike-slip faults related to the Najd fault system [81, 86, 97]. While Andresen et al. (2010) interpreted the D3 deformational event to have resulted from an extensional fault breakaway system. The D3 deformational event was responsible for forming the eugeoclinal rocks, two tectono-metamorphic events, an intervening episode of exhumation and erosion, and emplacement of post-orogenic granites after 630 Ma.

The scarcity and imprecise of many of the available regional geochronological data obscures our knowledge of the ANS geological history. Earlier studies suggested pre-Neoproterozoic basement rocks existence in the Egyptian ANS, but recent geochronological investigations present no conclusive evidence supporting this notion [2, 18, 19, 23, 30–38]. Accordingly, the current consensus is that the basement rocks in the Egyptian ANS are largely Neoproterozoic, with little evidence supporting an older origin.

While data scarcity is an issue, ongoing improvements in geochronological techniques are helping to refine the geological history of the region. Therefore, the temporal and spatial evolution of the different ANS rock units in the CED remains inadequately outlined, and efforts to categorize distinct events require further investigation. Consequently, we conduct the current study, which dates the basement rocks of Marsa Alam-Idfu transect, that signifies the north extent of the western ANS exposures in the Egyptian CED, characterized by the ophiolitic, volcanic, and granitic exposures (Fig. 2).

Methods

Zircon crystals were concentrated using conventional mineral separation techniques such as rock crushing, sieving, Frantz magnetic separator, and heavy liquids. Approximately 100-50 zircon crystals were mounted in a Teflon. Mounted crystals were polished to expose their surfaces. Then, the polished zircon grains were etched in in a NaOH-KOH eutectic melt at 220±5 °C for 60-210 min [2, 34]. The isotopic ratios of U/Pb and Th/Pb were determined using the LA-ICP-MS unit at Kanazawa University, Japan. The LA-ICP-MS instrumental conditions and specifications are presented in Tamura et al. [98] and the applied analytical parameters, including fluence, repetition rate, laser wavelength, spot size, laser fluence, and repetition rate, are summarized in Table 2. Challenges such as the mass bias of the instrument, during-ablation dissociation of U-Th/Pb, and elemental fractionation induced by the laser are common in age dating using the LA-ICP-MS technique. To mitigate these challenges, several approaches were adopted: (1) employing a 213 nm laser wavelength [99, 100]; (2) utilizing a mixture of post-ablation helium and argon gas to carry to the mass spectrometer; (3) limiting the ablation time to ca. 30 s to prevent excessive heating [98, 100-103; and (4) conducting further measurements of external reference materials such as AS3, GJ1, Fish Canyon, and Plěsovice zircon standards to correct any lasting laser-induced fractionation and minimize instrument mass bias [101, 102].

To obtain a more accurate quantitative analysis of geological samples, the calibration approach should combine the external calibration method with an internal standard



Fig. 2 Geologic map for examined samples along the Marsa Alam-Idfu transect area where samples and their zircon U-Pb ages are represented, and the mapped area eliminated by a red box on the map of Egypt

ICP-MS	
Model	Agilent 7850
Forward power	1200 W
Plasma gas flow	15 L min ⁻¹
Carrier gas flow	1.10 L min ⁻¹ (Ar), 0.3 L min ⁻¹ (He)
Interface	Ni sampler/Ni skimmer
Laser	
Model	UP-213 (New Wave Research)
Wavelength	213 nm (Nd-YAG)
Spot size	25 μm
Repetition rate	5 Hz
Energy density	7 J cm ⁻² (Attenuater: 50–60%)
Warming up	10 s

normalization [104]. The external calibration method is based on the sensitivity obtained by analysis of reference materials containing analyzed elements of known concentrations. The NIST glasses SRM 610 are frequently used as primary calibration standards because they contain many trace elements in high and homogenously distributed concentrations [105]. However, the NIST glasses have the disadvantage of having a completely different matrix from those natural minerals [106-109]. Therefore, the calibration method against multiple external standards with natural composition is preferable [106, 109, 110]. However, they may have some compositional heterogeneities and do not match the range of concentrations expected in the sample for all analyzed elements [105, 110]. Therefore, we have used an integration of analyzing both NIST glasses SRM 610 and multiple natural external standards during our analyses. While, the internal standard calibration method is used to correct for the elemental fractionation caused by sensitivity drift, matrix effect, and the difference in ablation yield between samples and reference materials [111–114]. Therefore, a calibration approach of combining the external and internal standard calibrations was used routinely during this study to obtain accurate and precise trace element contents by LA-ICP-MS [112, 113]. During this study, zircon reference materials with established chronological origins were regularly analyzed and compared to published data to monitor the precision of our measurements. Sample analyses were frequently sandwiched by the GJ-1 zircon standards analyses with similar analytical conditions. Additionally, isotopic ratios were regularly examined by analyzing different zircon standards with established ages. The produced ages of the zircon standards AS-3, GJ-1, Plěsovice, and Fish Canyon tuff were 1099±2, 612±2, 341±2, and 28.8±0.3 Ma, respectively. These ages overlap with the reference values, which were 1099 Ma [115], 609 Ma [102], 337.1 Ma [116], and 28.4 Ma [117] for AS-3, GJ-1, Plěsovice, and Fish Canyon tuff, respectively. The 238U signal intensities were adjusted using the standard material SRM 610, where the 238U concentration was 456 ppm [118, 119], while, the signal intensity of 29Si was monitored as an internal standard to track the chemical composition of zircon crystals [112, 118].

For each analysis, time-resolved signals were carefully studied to ensure stable flat signal intervals (free from inclusions, core-rim features, and zones with high common Pb or evidence of fractionation). The isotopic ratios after background correction were considered to calculate the average isotopic intensities. Zircon ages can be corrected for common Pb contamination using different methods [120]. The most common approach involves measuring ²⁰⁴Pb to subtract the common Pb component from the radiogenic Pb isotopes. Still, due to the very low 204 counts (Table 3) (²⁰⁴Pb and ²⁰⁴Hg) and isobaric interference from ²⁰⁴Hg, it was not possible to measure the ²⁰⁴Pb counts with sufficient precision. All age results presented in this work are, therefore, not commonly Pb corrected. The concordant ages and 2σ error, as represented in the text, Fig. 2, and the Concordia diagrams, were calculated using the IsoplotR code [121].

Results

We acquired U-Pb age data from 114 zircon grains belonging to 11 basement rocks, with 10–12 grains from each collected sample. Grains with inclusions or cracks and those exhibiting detectable levels of common 204 Pb were excluded. Cathodoluminescence (CL) imaging unveiled diverse internal structures. However, the focus during the analyses was typically directed towards the core to determine the crystallization age (Fig. 3). The percentage of discordance was computed by comparing the 206 Pb/ 238 U and 207 Pb/ 235 U ages, and grains exceeding 10% discordance were omitted from the calculations. The reported ages and $\pm 2\sigma$ error ranges were calculated using IsopltR [121].

Sample MI01 (sample 1 on Fig. 2) is represented by grains that displayed transparency to yellow coloring, were characterized by subhedral faces, and had an average length/width ratio of 2:1. Inclusions were present in approximately 50% of grains, while cracks were observed in approximately 50% of them. The Th/U ratios ranged from 1.1 to 0.17, averaging around 0.46 (Table 3). Grain D7 exhibited discordance exceeding 10% and was therefore disregarded during the age calculations and interpretations. The nine remaining grains displayed concordant ages and formed a single population with a concordant age of 600 ± 1 Ma (Fig. 4). This age was counted as the age of formation of the MI01 dacitic sample (Table 1; Fig. 2).

Sample MI02 (sample 2 on Fig. 2) is represented by grains displaying yellow to brown coloring, were characterized by prismatic shapes with subhedral faces, and had an average length/width ratio of 3:1. Inclusions were present in approximately 50% of the grains, while voluminous cracks were observed in approximately 55% of them. The Th/U ratios ranged from 0.7 to 0.28, averaging around 0.44 (Table 3). Grain F6 exhibited discordance exceeding 10% and was therefore disregarded during the age calculations and interpretations. The nine remaining grains displayed concordant ages, with grain E2 yielding a pre-Pan-African concordant age of 1549 ± 4 Ma and grain E8 yielding an

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older concordant age of 723 ± 10 Ma (Fig. 5). The seven remaining grains formed a single population with a concordant age of 699 ± 4 Ma (Fig. 6). This age was counted as the age of formation of the MI02 diorititc sample (Table 1; Fig. 2).

Sample MI03 (sample 3 on Fig. 2) is represented by grains that displayed transparency to yellow coloring, were characterized by equidimensional shapes with euhedral faces, and had an average length/width ratio of 3:1. Inclusions were present in the majority of the grains, while voluminous cracks were observed in approximately 60% of them. The Th/U ratios ranged from 0.48 to 0.16, averaging around 0.31 (Table 3). Grain F5 exhibited discordance exceeding 10% and was therefore disregarded during the age calculations and interpretations. The 11 remaining grains displayed concordant ages and formed a single population with a concordant age of 647 ± 3 Ma (Fig. 6). This age was counted as the age of formation of the MI03 granitic sample (Table 1; Fig. 7).

Sample MI04 (sample 4 on Fig. 2) is represented by grains that displayed transparency to yellow coloring, were characterized by prismatic shapes with subhedral faces, and had an average length/width ratio of 2:1. Inclusions were present in many grains, while voluminous cracks were observed in approximately 50% of them. The Th/U ratios ranged from 1.36 to 0.06, averaging around 0.59 (Table 3). Grains D7 and E7 exhibited discordance exceeding 10% and were therefore disregarded during the age calculations and interpretations. The eight remaining grains displayed concordant ages, with grain D3 yielding a younger concordant age of 594 ± 5 Ma (Fig. 5). The seven remaining grains formed a single population with a concordant age of 623 ± 3 Ma (Fig. 5). This age was counted as the age of formation of the MI04 metavolcanic sample (Table 1; Fig. 2).

Sample MI05 (sample 5 on Fig. 2) is represented by grains displaying yellow to brown coloring, were characterized by prismatic shapes with euhedral faces, and had an average length/width ratio of 3:1. Inclusions and voluminous cracks were observed in approximately 65% of the grains. The Th/U ratios ranged from 0.57 to 0.3, averaging around 0.46 (Table 3). All the ten dated zircon grains displayed concordant ages and formed a single population with a concordant age of 570 ± 2 Ma (Fig. 8), which was counted as the age of formation of the MI05 granitic sample (Table 1; Fig. 2).

Sample MI06 (sample 6 on Fig. 2) is represented by grains that displayed transparency to yellow coloring, were characterized by prismatic shapes with euhedral faces, and had an average length/width ratio of 4:1. Inclusions and

						5	22				- (AA								
5	204pb	²³⁸ U	Th/U	t ratios a ± 2σ	²⁰⁶ Pb/	rs +2σ	²⁰⁷ Pb/	+2a	²⁰⁸ Pb/	±2σ	Age (Ma ²⁰⁶ pb/	i) and 20 ±2σ	²⁰⁷ Pb/	+ 2σ	²⁰⁷ Pb/	+2σ	Conc.	+ 2σ	%aliscordance
	2	•			²³⁸ U) 	²³⁵ U) 	²³² Th) 	²³⁸ U		²³⁵ U		²⁰⁶ Pb			1	
MI01																			
A1	0.006	227	0.640	0.025	0.09783	0.00309	0.81081	0.00280	0.03041	0.00074	602	4	603	m	607	4	603	m	0.2
A3	-0.007	278	0.503	0.021	0.09729	0.00340	0.80593	0.00307	0.03047	0.00073	599	4	600	4	606	5	600	m	0.3
B2	0.001	923	0.170	0.011	0.09795	0.00624	0.81186	0.00566	0.03020	0.00077	602	7	604	9	607	œ	604	9	0.2
B5	0.006	528	0.357	0.019	0.09827	0.00473	0.82311	0.00435	0.03070	0.00086	604	9	610	5	629	9	610	5	0.9
C6	0.003	168	1.098	0.041	0.09714	0.00264	0.79830	0.00236	0.03054	0.00084	598	m	596	c	588	4	596	Ś	-0.3
D1	0.005	279	0.545	0.023	0.09715	0.00340	0.80813	0.00309	0.03037	0.00076	598	4	601	4	615	Ŝ	601	4	0.6
*D7	-0.003	839	0.409	0.028	0.05470	0.00326	0.73216	0.00476	0.02446	0.00092	343	4	558	9	1568	5	555	9	38.5
E2	-0.002	713	0.252	0.015	0.09896	0.00554	0.79516	0.00484	0.03053	0.00083	608	7	594	9	539	8	594	9	-2.4
F4	0.001	856	0.241	0.016	0.09759	0.00598	0.80313	0.00537	0.03039	0.00089	600	7	599	9	591	8	599	9	-0.3
B	-0.005	624	0.315	0.018	0.09791	0.00512	0.80794	0.00462	0.03053	0.00087	602	9	601	5	597	7	601	5	-0.1
MI02																			
A1	0.010	170	0.294	0.009	0.11239	0.00309	0.96840	0.00302	0.03459	0.00050	687	18	688	16	069	19	688	15	0.1
A2	-0.035	223	0.278	0.009	0.11107	0.00350	0.97397	0.00348	0.03457	0.00055	679	20	691	18	727	22	691	17	1.7
C	-0.003	104	0.497	0.012	0.11175	0.00240	0.98043	0.00239	0.03546	0.00052	683	14	694	12	728	15	694	12	1.6
C5	0.037	286	0.517	0.022	0.11400	0.00407	0.98750	0.00401	0.03528	0.00087	696	24	697	20	702	25	697	20	0.2
D3	0.002	75	0.403	0.008	0.11638	0.00214	1.02708	0.00216	0.03654	0.00041	710	12	717	11	741	13	718	[]	1.1
E1	0.004	138	0.696	0.021	0.10784	0.00266	0.92140	0.00255	0.03394	0.00068	660	15	663	13	672	17	663	13	0.4
**E2	0.003	337	0.303	0.013	0.26464	0.01092	3.58033	0.02394	0.08142	0.00171	1514	56	1545	53	1588	37	1549	4	2.0
E8	-0.005	65	0.445	0.009	0.11538	0.00197	1.03792	0.00204	0.03601	0.00040	704	11	723	10	781	12	723	10	2.6
*F6	0.003	26	0.119	0.001	0.15182	0.00165	1.16698	0.00148	0.06947	0.00025	911	6	785	7	441	6	785	7	-16.0
F9	0.004	56	0.539	0.010	0.11384	0.00180	0.98310	0.00176	0.03504	0.00039	695	10	695	6	695	11	695	6	0.0
MI03																			
A1	0.013	562	0.336	0.018	0.10675	0.00532	0.91959	0.00514	0.03435	0.00096	654	16	662	14	069	17	662	13	1.3
A2	0.140	482	0.168	0.008	0.10648	0.00492	0.89714	0.00462	0.03497	0.00064	652	14	650	12	642	16	650	12	-0.3
A8	0.023	258	0.383	0.014	0.10573	0.00357	0.86081	0.00321	0.03471	0.00070	648	10	631	6	568	12	630	6	-2.7
B1	0.064	693	0.334	0.020	0.10357	0.00573	0.87447	0.00537	0.03230	0.00100	635	17	638	15	647	19	638	14	0.4
B8	0.032	729	0.410	0.026	0.10991	0.00625	0.97051	0.00626	0.03540	0.00125	672	18	689	16	742	20	689	16	2.4
Ü	0.388	757	0.280	0.017	0.10418	0.00603	0.92454	0.00601	0.03477	0.00103	639	18	665	16	753	19	665	16	3.9
D6	0.085	389	0.273	0.012	0.10206	0.00423	0.84976	0.00388	0.03255	0.00068	626	12	625	11	617	14	625	11	-0.3
E2	0.296	890	0.158	0.010	0.10964	0.00689	0.96367	0.00686	0.03444	0.00083	671	20	685	18	732	22	685	17	2.1
*F5	0.014	1027	0.089	0.006	0.12214	0.00829	0.67413	0.00476	0.04262	0.00083	743	24	523	14	0	26	520	14	-42.0
F9	0.002	541	0.260	0.014	0.10304	0.00503	0.89289	0.00487	0.03450	0.00083	632	15	648	13	702	16	648	13	2.4
6	0.001	114	0.482	0.013	0.10439	0.00234	0.91455	0.00230	0.03323	0.00050	640	7	659	9	725	8	659	9	2.9
G5	0.002	331	0.281	0.012	0.10169	0.00389	0.84070	0.00354	0.03256	0.00064	624	11	620	10	601	13	619	10	-0.8
MI04																			
A1	0.021	121	0.546	0.015	0.10011	0.00231	0.83616	0.00212	0.03177	0.00052	615	7	617	9	623	∞	617	9	0.3

Table	(conu			.	,														
פֿ	Con.	000	Isotopi	c ratios à	and 20 erro	ors	LOC		000		Age (Ma	a) and 20	Jerrors		FOC				%discordance
	qd+oz	N 7	Th/U	±2σ	²³⁸ U	±2σ	²³⁵ U	±2σ	²³² Th	±2σ	²³⁸ U	±2σ	²³⁵ U	±2σ	²⁰⁶ Pb/	±2σ	Conc.	± 2σ	
B5	0.004	449	0.916	0.054	0.10086	0.00448	0.87056	0.00430	0.03289	0.00136	619	13	636	12	694	15	636	12	2.6
C6	-0.007	90	1.362	0.133	0.10221	0.00203	0.83360	0.00182	0.03067	0.00108	627	9	616	۰2	572	7	616	5	-1.9
68	-0.00	133	0.697	0.021	0.10049	0.00243	0.84845	0.00227	0.03155	0.00062	617	7	624	9	646	œ	624	9	1.0
**D3	0.002	101	0.604	0.015	0.09400	0.00198	0.79424	0.00182	0.03039	0.00048	579	9	594	Ŀ.	648	7	594	5	2.4
*D7	-0.012	69	1.479	0.039	0.38159	0.00744	3.46731	0.01035	0.05319	0.00110	2084	17	1520	12	802	1	1511	12	-37.1
E2	-0.008	321	0.057	0.002	0.10049	0.00378	0.84982	0.00353	0.03258	0.00028	617	[625	10	650	13	625	10	1.2
*E7	0.036	134	0.679	0.020	0.18855	0.00475	3.07829	0.01223	0.03360	0.00065	1114	13	1427	15	1932	10	1432	15	22.0
F3	0.061	663	0.305	0.018	0.10170	0.00550	0.86772	0.00520	0.03325	0.00096	624	16	634	14	669	18	634	14	1.6
F7	0.011	175	0.200	0.006	0.10096	0.00280	0.87750	0.00271	0.03231	0.00039	620	00	640	7	709	6	640	7	3.1
MI05																			
A1	-0.006	140	0.372	0.010	0.09361	0.00232	0.85452	0.00234	0.03242	0.00048	577	7	627	9	812	∞	627	9	8.0
A8	-0.003	918	0.562	0.043	0.09134	0.00578	0.77329	0.00531	0.02959	0.00136	563	17	582	15	652	20	582	15	3.1
B3	-0.003	158	0.545	0.017	0.08686	0.00228	0.72652	0.00205	0.03004	0.00057	537	7	555	9	626	∞	555	9	3.2
B9	0.005	98	0.295	0.007	0.09033	0.00187	0.75206	0.00168	0.03059	0.00033	557	9	569	5	616	7	569	Ŝ	2.1
C6	-0.099	182	0.437	0.014	0.08882	0.00250	0.75226	0.00229	0.03064	0.00055	549	7	570	7	653	6	570	7	3.7
C7	0.003	113	0.482	0.012	0.08946	0.00198	0.73852	0.00176	0.02874	0.00043	552	9	562	5	598	7	562	Ŝ	1.6
*D2	-0.003	119	0.482	0.013	0.08946	0.00204	0.75368	0.00186	0.02890	0.00044	552	9	570	5	642	7	570	2	3.2
E3	0.002	248	0.353	0.013	0.09452	0.00312	0.76270	0.00272	0.02923	0.00055	582	6	576	8	548	=	576	8	-1.2
F8	0.008	102	0.568	0.014	0.08942	0.00189	0.76056	0.00174	0.02920	0.00045	552	9	574	5	662	7	574	ۍ	3.9
G4	0.003	95	0.535	0.013	0.09056	0.00185	0.77007	0.00170	0.02976	0.00043	559	5	580	Ŋ	662	9	580	Ŋ	3.6
MI06																			
A1	0.005	43	0.265	0.004	0.09175	0.00126	0.77560	0.00115	0.02822	0.00019	566	4	583	m	649	4	583	ŝ	2.9
A2	0.003	96	0.444	0.010	0.09274	0.00190	0.74702	0.00165	0.02933	0.00039	572	9	566	5	545	7	566	5	-0.9
B5	0.010	345	0.053	0.002	0.09152	0.00355	0.75973	0.00319	0.02940	0.00025	565	10	574	6	610	12	574	6	1.6
B7	0.013	380	0.053	0.002	0.09292	0.00378	0.76981	0.00340	0.02977	0.00027	573	1	580	10	606	13	580	10	1.2
D3	0.004	188	0.804	0.030	0.09690	0.00279	0.79243	0.00248	0.03189	0.00080	596	8	593	7	578	10	593	7	-0.6
D6	0.004	74	0.583	0.013	0.09302	0.00168	0.75203	0.00146	0.02968	0.00040	573	5	569	4	553	9	569	4	-0.7
E2	0.001	104	0.342	0.008	0.08850	0.00189	0.76602	0.00177	0.03054	0.00037	547	9	577	5	700	7	577	5	5.3
*F1	-0.002	37	0.756	0.012	0.09758	0.00125	1.35476	0.00216	0.03677	0.00040	600	4	870	IJ.	1636	4	867	5	31.0
F8	0.008	110	0.424	0.011	0.09843	0.00216	0.80267	0.00192	0.03085	0.00043	605	9	598	5	572	7	598	5	-1.1
99	-0.002	72	0.428	0.009	0.09131	0.00162	0.73793	0.00141	0.02905	0.00033	563	5	561	4	552	9	561	4	-0.4
MI07																			
A1	-0.010	47	0.509	0.009	0.10609	0.00153	0.89773	0.00145	0.03370	0.00034	650	4	651	4	651	Ŝ	651	4	0.1
B2	0.006	103	0.100	0.002	0.10876	0.00232	0.88602	0.00210	0.03488	0.00023	666	7	644	9	569	7	644	9	-3.3
B5	-0.004	77	0.530	0.011	0.10873	0.00201	0.86699	0.00177	0.03357	0.00044	665	9	634	5	522	7	634	5	-5.0
*C8	0.003	70	0.634	0.014	0.11195	0.00197	1.66490	0.00387	0.04440	0.00060	684	9	995	7	1763	Ŝ	994	7	31.3
D9	-0.003	31	1.677	0:030	0.11029	0.00129	0.95842	0.00126	0.03444	0.00050	674	4	682	m	708	4	682	ŝ	1.2

	lsotopic ratios and 20 errors
3 (continued)	Con.
Table	פֿ

%discordance			-1.8	-4.7	-0.5	2.2	-2.9		-2.1	1.8	0.1	2.9	-1.1	-0.6	2.4	0.6	0.1	0.4		1.8	1.3	0.4	3.9	2.7	0.0	1.8	1.2	11.9	2.4		-2.1	-5.3	0.0	-0.8	-1.2	6.0-	149	
	±2σ		9	Ŝ	4	Ŝ	5		00	7	12	6	2	12	9	11	10	7		7	10	12	7	4	9	9	10	ø	7		9	10	00	9	7	18	10	
	Conc.		749	632	649	654	652		582	586	587	762	549	591	621	600	594	585		613	601	602	639	610	616	609	608	674	617		713	1145	726	760	724	729	754	
	±2σ		7	7	5	9	9		20	16	14	13	15	25	14	17	17	15		6	14	16	6	S	7	00	13	6	6		7	10	6	7	00	22	10)
	²⁰⁷ Pb/	²⁰⁶ Pb	708	527	636	703	586		590	594	594	772	556	598	629	607	602	592		652	629	609	728	671	635	648	634	952	670		666	1032	747	742	697	708	1103)
	±2σ		9	5	4	5	5		œ	7	12	6	5	12	9	11	10	7		7	10	12	7	4	9	9	10	Ø	7		9	10	œ	9	7	19	10	
errors	²⁰⁷ Pb/	²³⁵ U	749	632	649	654	652		582	586	587	762	549	591	621	600	594	585		613	601	602	639	610	616	609	608	674	617		713	1146	726	760	724	730	753)
and 20	±2σ		7	9	4	9	5		10	7	14	10	9	13	7	12	12	∞		00	12	14	œ	5	9	7	1	00	8		9	12	6	7	∞	21	10	
Age (Ma)	/qd ₉₀₃	³⁸ U	763	561	553	540	571		595	576	586	740	555	594	506	596	594	583		502	593	669	514	593	510	598	501	594	502		728	1207	720	266	732	736	41	
			054 7	019 6	035 6	032 (021 6		071 5	061	059 5	055 7	090	091	050 6	065 5	067	059		055 6	019	122	127 0	038 5	056 6	035 5	070 6	055 5	064 (048	027	067 7	051 7	051 7	192	106	
	±20		00.00	00.00	00.00	0.00	3 0.00		0.00	00:0	0:00	0.00	00.00	0.00	00.0	00:00	0.00	00:00 t		00.00	00.00	00.00	3 0.00	00.00	00.00	0.00	00.00	00.00	00.00		0.00	00.00	00.00	00.00	0.00	0.00	0.00	
	²⁰⁸ Pb/	²³² Th	0.03959	0.03455	0.03500	0.03418	0.03458		0.02876	0.03105	0.02958	0.03906	0.02795	0.03095	0.0316	0.03057	0.03022	0.03034		0.03143	0.03120	0.0305	0.03233	0.03199	0.03148	0.03126	0.03098	0.02899	0.0319(0.03623	0.06337	0.0383(0.03919	0.03802	0.03736	0.04492	
	±2σ		0.00237	0.00181	0.00137	0.00184	0.00169		0.00291	0.00231	0.00422	0.00356	0.00162	0.00406	0.00221	0.00384	0.00354	0.00233		0.00245	0.00368	0.00435	0.00254	0.00145	0.00204	0.00217	0.00343	0.00319	0.00256		0.00220	0.00632	0:00309	0.00266	0.00262	0.00748	0.00422	
	- 'dd	5U	09193 (36268 (39492 (90441 (90087 (77469 (78132 (78224 (11844 (71685 (78904 (34337 (30542 (79528 (77860 (32906 (30757 (30835 (37648 (32322 (33396 (32142 (32054 (94217 (33555 (01841 (09130 (04499 (11339 (03970 (05150 () 06660	
	20	23	35 1.(0.5 0.8	46 0.8	89 0.9	85 0.9		35 0.7	54 0.7	172 0.7	31 1.	90 05	157 0.7	34 0.8	123 0.8	94 0.7	10 <u>0.</u>		63 0.8	02 0.8	80 0.8	e1 0.8	54 0.8	21 0.8	34 0.8	373 0.8	88 0.9	273 0.8		26 1.0	56 2.0	304 1.(58 1.	964 1.0	'47 1.(342 1.(
rors	±2σ		0.00	0.002	0.001	0.001	0.00		0.00	0.002	0.00	00.00	0.001	00.00	0.002	0.00	0.00	0.002		0.00	0.00	0.00	0.002	0.001	0.002	0.002	0.00	0.002	0.002		0.002	0.00	0.00	0.002	0.002	0.007	00.00	
nd 2o er	²⁰⁶ Pb/	²³⁸ U	0.12569	0.10798	0.10653	0.10435	0.10973		0.09665	0.09339	0.09521	0.12172	0.08991	0.09658	0.09856	0.09690	0.09647	0.09459		0.09793	0.09643	0.09743	0.09992	0.09641	0.09932	0.09722	0.09776	0.09648	0.09789		0.11952	0.20585	0.11808	0.12612	0.12027	0.12100	0.10453	
ratios a	±2σ		0.013	0.002	0.009	0.006	0.002		0.022	0.018	0.009	0.008	0.032	0.022	0.013	0.012	0.014	0.018		0.014	0.001	0.043	0.111	0.011	0.019	0.006	0.016	0.015	0.018		0.012	0.001	0.015	0.010	0.010	0.058	0.021	
Isotopic	Th/U		0.583	0.086	0.565	0.290	0.137		0.545	0.546	0.170	0.286	1.098	0.409	0.482	0.252	0.315	0.536		0.444	0.022	0.707	1.409	0.585	0.679	0.224	0.378	0.437	0.546		0.539	0.043	0.497	0.445	0.403	0.752	0.549	
	²³⁸ U		78	81	43	75	25		274	169	562	165	102	511	128	434	380	174		164	396	553	155	58	113	132	331	204	177		80	102	148	93	107	850	242	
Con.	⁰⁴ Pb		0.045	.005	.003	0.004).004		.005	0.002	0.020	006	0.001	0.003	000.0	0.001	000	0.012		0.001	0.003	002	001	0.011	0.001	0.003	00.007	0.007	0.002		0.003	001	0.006	001	0.008	0.004	013	
			1	U	U	J	0	~	U	1	1	0	1	J	0	1	J	'	6	I	I	0	0	1	1	U	U	1	0	0	0	J	1	U	1	1	0	

<u>ب</u>	Con.		Isotopi	c ratios	and 20 erro)rs					Age (Ma	ו) and 2נ	J errors						%discordance
	²⁰⁴ Pb	²³⁸ U	Th/U	±2σ	²⁰⁶ Pb/ ²³⁸ U	±2σ	²⁰⁷ Pb/ ²³⁵ U	±2σ	²⁰⁸ Pb/ ²³² Th	±2σ	²⁰⁶ Pb/ ²³⁸ U	±2σ	²⁰⁷ Pb/ ²³⁵ U	±2σ	²⁰⁷ Pb/ ²⁰⁶ Pb	±2σ	Conc.	±2σ	
E5	0.009	173	0.511	0.017	0.19212	0.00551	2.08976	0.00822	0.05954	0.00115	1133	15	1145	14	1168	12	1146	13	1.1
E8	-0.003	181	0.545	0.018	0.19613	0.00576	2.13186	0.00863	0.05985	0.00122	1154	16	1159	14	1167	12	1159	14	0.4
F4	0.005	407	0.517	0.026	0.11798	0.00503	1.04142	0.00511	0.03649	0.00108	719	15	725	13	741	15	725	13	0.8
MI11																			
A1	0.004	158	0.697	0.023	0.10827	0.00287	0.94532	0.00283	0.03596	0.00077	663	17	676	15	718	18	676	15	1.9
A3	0.002	145	0.441	0.013	0.10855	0.00275	0.97064	0.00280	0.03499	0.00057	664	16	689	14	769	17	689	14	3.6
A8	0.004	215	0.674	0.026	0.11104	0.00343	0.93762	0.00326	0.03489	0.00085	679	20	672	17	647	22	672	17	-1.0
B6	-0.008	291	1.128	0.056	0.11234	0.00404	0.97233	0.00397	0.03489	0.00129	686	23	069	20	669	25	069	20	0.6
C	0.008	196	0.437	0.015	0.17551	0.00533	1.93377	0.00790	0.05648	0.00107	1042	29	1093	27	1194	24	1095	25	4.7
C6	0.000	193	0.608	0.021	0.10948	0.00320	0.96500	0.00320	0.03514	0.00077	670	19	686	17	738	20	686	16	2.3
08	0.003	209	0.804	0.031	0.10666	0.00324	0.87826	0.00296	0.03446	0.00091	653	19	640	16	592	21	640	16	-2.0
E6	0.008	180	0.295	600.0	0.10982	0.00310	0.97020	0.00311	0.03565	0.00053	672	18	689	16	743	19	689	16	2.5
F2	-0.002	206	0.405	0.014	0.11433	0.00347	0.97192	0.00334	0.03707	0.00069	698	20	689	17	661	21	689	17	-1.3
F6	0.001	130	0.351	600.0	0.14072	0.00343	0.98630	0.00270	0.03648	0.00050	849	19	697	14	232	20	695	14	-21.8
Gr=G ±2σei	ains, A1 gr. ror for ²³⁸ U	ain symbc I was calcı	ol, *D9 refe ulated fron	rs to grair ר the stan	ns with %Disc dard deviation	cordance ^{>} 10 on, ±2σ erro	which were r was calcula	excluded frc ted for both	im age calcu the isotopic	Lations, **A9	refers to gra	ains with rdance is	ages differ: between ²⁽	s from the ³⁶ Pb/ ²³⁸ U	s sample ac	ae of pop ²³⁵ U ages	ulation, Co	onc.= con	centration by µg/g,

voluminous cracks were observed in approximately 70% of the grains. The Th/U ratios ranged from 0.8 to 0.05, averaging around 0.38 (Table 3). Grain F1 exhibited discordance exceeding 10% and was therefore disregarded during the age calculations and interpretations. The nine remaining grains displayed concordant ages and formed a single population with a concordant age of 576 ± 2 Ma (Fig. 8), which was counted as the age of formation of the MI06 syenitic sample (Table 1; Fig. 2).

Sample MI07 (sample 7 on Fig. 2) is represented by grains that displayed transparency to yellow coloring, were characterized by prismatic shapes with euhedral faces, and had an average length/width ratio of 3:1. Inclusions and voluminous cracks were observed in approximately 75% of the grains. The Th/U ratios ranged from 1.68 to 0.09, averaging around 0.5 (Table 3). Grain C8 exhibited discordance exceeding 10% and was therefore disregarded during the age calculations and interpretations. The nine remaining grains displayed concordant ages, with grain E3 yielding an older concordant age of 749 ± 6 Ma (Fig. 5). The eight remaining grains formed a single population with a concordant age of 646 ± 2 Ma (Fig. 6), which was counted as the age of formation of the MI07 syenitic sample (Table 1; Fig. 2).

Sample MI08 (sample 8 on Fig. 2) is represented by grains displaying yellow to brown coloring, were characterized by prismatic shapes with euhedral faces, and had an average length/width ratio of 3:1. Inclusions and voluminous cracks were observed in approximately 70% of the grains. The Th/U ratios ranged from 1.1 to 0.17, averaging around 0.46 (Table 3). All analysed zircon grains displayed concordant ages, with grains B4 and C7 yielding older concordant ages of 762±9 Ma and 621±6 Ma, respectively (Fig. 5; Table 3). While grain B6 shows a younger concordant age of 549 ± 5 Ma (Fig. 5; Table 3). The seven remaining zircon grains formed a single population with a concordant age of 582 ± 3 Ma (Fig. 8), counted as the age of formation of the MI08 synogranitic sample (Table 1; Fig. 2).

Sample MI09 (sample 9 on Fig. 2) is represented by grains displaying transparent to yellow coloring, were characterized by prismatic shapes with subhedral faces, and had an average length/width ratio of 2:1. Inclusions and voluminous cracks were observed in approximately 55% of the grains. The Th/U ratios ranged from 1.41 to 0.02, averaging around 0.56 (Table 3). Grain F2 exhibited discordance exceeding 10% and was therefore disregarded during the age calculations and interpretations. The nine remaining grains displayed concordant ages and formed a single population with a concordant age of 611±2 Ma

Table 3 (continued)



Fig. 3 Cathodoluminescence (CL) images for some of the analyzed zircons, representing location and ages of analyzed spots

(Fig. 5), which was counted as the age of formation of the MI09 and esitic sample (Table 1; Fig. 2).

Sample MI10 (sample 10 on Fig. 2) is represented by grains displaying brown coloring, were characterized by prismatic shapes with euhedral faces, and had an average length/width ratio of 3:1. Inclusions and voluminous cracks were observed in approximately 75% of the grains. The Th/U ratios ranged from 1.13 to 0.04, averaging around 0.55 (Table 3). Grain C5 exhibited discordance exceeding 10% and was therefore disregarded during the age calculations and interpretations. The 11 remaining grains displayed concordant ages, with grains A3, E5, and E8 yielding older concordant ages of 1145 ± 10 Ma,

1146 \pm 13 Ma, and 1159 \pm 14 Ma (Fig. 5), respectively (Table 3). The eight remaining zircon grains formed a single population with a concordant age of 729 \pm 3 Ma (Fig. 6), which was counted as the age of formation of the MI10 gneissic sample (Table 1; Fig. 2).

Sample MI11 (sample 11 on Fig. 2) is represented by grains that displayed transparent to yellow coloring, were characterized by prismatic shapes with euhedral faces, and had an average length/width ratio of 4:1. Inclusions and voluminous cracks were observed in approximately 65% of the grains. The Th/U ratios ranged from 1.13 to 0.3, averaging around 0.61 (Table 3). Grain F6 exhibited discordance exceeding 10% and was therefore disregarded



Fig. 4 Concordia diagram for all zircon grains with discordance percent ≤ 10% for Dokhan Volcanics samples, plotted using IsopItR [121]

during the age calculations and interpretations. The nine remaining grains displayed concordant ages and formed a single population with a concordant age of 680 ± 5 Ma (Fig. 6), which was counted as the age of formation of the MI11 syenitic sample (Table 1; Fig. 2).

Discussion

The Th/U ratios across the concordant grains varied between 1.68 and 0.02, averaging ca. 0.48 (Table 3). These ratios aligned with an igneous source [122, 123] for all zircons except for six grains, which exhibited < 0.1 values. These grains were E2 from sample MI04, B5 and B7 from sample MI06, E6 from sample MI07, B6 from sample MI09, and A3 from sample MI10 (Table 3). These were magmatic samples except for samples MI04 and MI10, which were metamorphic rocks (Table 1). However, all six grains were within the age population of their sample (Table 3). The Th/U ratio is usually used as an indicator of the relative depletion or enrichment of U when this ratio is disturbed [124]. However, this might not be the case here as all the aforementioned Th/U ratios are for concordant grains, where any relative depletion or enrichment of the U concentrations would have caused considerable discordance (Table 3).

The reported ages during this study are not corrected for common-lead (²⁰⁴Pb) as their concentrations show low values and are frequently lower than our analytical detection limit (negative values). This would reduce the common-lead potential induced errors and consequently strengthen the reliability of the resulted ages. Additionally, the ²⁰⁸Pb/²³²Th ages, which are substantially vulnerable to contaminations of common Pb [125], are concordant with the ²⁰⁶Pb/²³⁸U ages. These indicate the lack of any effect of common-lead on the produced ages. Any grains displaying discordance>10% were omitted from their sample's crystallization age calculations and the data assessments (Table 3).

The five concordant zircon grains with pre-Pan-African ages range between 1549±4 Ma and 1095±25 Ma (Table 4; Fig. 5). Although recent geochronological research on the ANS crystalline rocks suggests an absence of any pre-Pan-African units [2, 35, 40, 42, 63, 126, 127], zircon grains with comparable ages have been frequently reported and interpreted as xenocrystic grains [2, 19, 25, 30, 34, 87], suggesting reworked older crust, contaminations of the country rocks, or from a detrital source [128]. These inherited grains are reported from metamorphic and relatively older samples (i.e., MI02, MI10, and MI11). This might support that rock suits with pre-Pan-African ages previously existed and eroded in the studied region [3].

Samples MI02, MI07, and MI08 yielded inherited grains E8 (723 \pm 10 Ma), E3 (749 \pm 6 Ma), B4 (762 \pm 9 Ma), and C7 (621 \pm 6 Ma), respectively (Table 4). These grains



Fig. 5 Single zircon crystals U-Pb ages for samples with pre-Pan-African, Inherited, or younger ages

show older ages than the sample population, consistent with the reported reworked Neoproterozoic crust in the ANS [2, 8, 19, 30, 44]. Grain B6 (549 ± 5 Ma) yielded a younger age than the sample MI08 population, suggesting that it was affected by consequent magmatic events.

The produced zircon U-Pb ages for all samples have Pan-African ages that range between 729 ± 3 Ma and 570 ± 2 Ma (Table 1). The metamorphic sample MI10 yielded the oldest crystallization age of 729 ± 3 Ma, belonging to the second ophiolitic age maxima [60]. This age probably dates to an early stage of the island-arc synorogenic phase when the micro-continents convergence into the Archean plates (Fig. 1) during the EAO development in the studied region [2, 35]. Comparable ages have been reported from different parts of the ANS [2, 14, 22, 60, 87, 88, 129, 130]. Andresen et al. [35] reported a comparable age of 736 ± 1 Ma for the sample from the eugeoclinal thrust sheet, which was interpreted as either the age of the oceanic crust or the volcanic arc [35, 131]. While samples MI02, MI11, MI03, and MI07 yielded ages of 699 ± 4 Ma, 680 ± 5 Ma, 647 ± 3 Ma, and 646 ± 2 Ma, respectively. These samples represent the syn-orogenic stage of the ANS development when a compressional tectonic regime (Convergent) was dominated [2, 22]. These samples are characterized by changing their chemical composition chronologically from the calc-alkaline (diorite) to alkaline (syenite) affinities. This magmatic differentiation from calc-alkaline to alkaline affinities was previously noticed [*e.g.*, 1–3].

The volcanic samples MI04, MI09, and MI01 yielded crystallization ages of 623 ± 3 Ma, 611 ± 2 Ma, and 600 ± 1 Ma, respectively (Table 1). These samples yielded comparable ages to those previously reported from the Dokhan Volcanics [6, 71, 89], marking the initiation of the transition period from compressional-to-extensional tectonic settings [6, 22–25]. All the dated Dokhan



Fig. 6 Concordia diagram for all zircon grains with discordance percent < 10% for island-arc and syn-orogenic samples, plotted using IsopItR [121]

samples should belong to the older mafic Dokhan sequence, which previously dated between 635–620 Ma [68, 69]. Our results suggest extending this phase of volcanism to ca. 600 Ma in the studied region.

The remaining samples, MI08, MI06, and MI05, yielded crystallization ages of 582 ± 3 Ma, 576 ± 2 Ma, and 570 ± 2 Ma, respectively (Table 1). These samples represent magmatic emplacements during the post-orogenic stage of the EAO development in the ANS, where the tensional tectonic regime was dominated [2, 35]. Comparable ages have been reported from different parts of the ANS [2, 14, 22, 60, 87, 88, 129, 130].

Interpretation

The produced zircon U-Pb crystallization ages represent constructing the studied region in the CED through several magmatic pulses: (1) island-arc phase; represented by sample MI10 with an age of 729 ± 3 Ma. (2) syn-orogenic phase; represented by samples MI02 and MI11, MI03, and MI07 with ages of 699±4 Ma, 680 ± 5 Ma, 647 ± 3 Ma, and 646 ± 2 Ma, respectively. These two phases represent the domination of the compressional regime of the arc-continent collision between 729 ± 3 Ma and 646 ± 2 Ma in the region of study. Furthermore, all the dated grains with pre-Pan-African (Paleo- to Meso-Proterozoic) ages belong to these two phases, indicating a probable reworked older crust or contaminations from a detrital source. (3) post-orogenic phase; represented by all other samples. The transition between the compressional synorogenic and the extensional post-orogenic tectonic regimes was marked by the eruption of the older mafic Dokhan sequence which extended in the studied region from 623 ± 3 Ma (sample MI04) to 600 ± 1 Ma (sample



Fig. 7 Single zircon crystals U-Pb ages for samples within each sample populations

MI01). Afterwards, the last phase of the post-orogenic plutonism activated from 582 ± 3 Ma (sample MI08) to 570 ± 2 Ma (sample MI05). This suggests activation of the extensional tectonic regime in the studied region at 582 ± 3 Ma, while this phase might have extended to 549 ± 5 Ma as indicated by the grain B6 in sample MI08. The reworked nature of the Neoproterozoic crust in the ANS has been emphasized by the dating of some inherited grains with Pan-African ages (Table 4).

Additionally, rock suits classification based on their apparent chemical composition into Calc-alkaline "Grey" and Alkaline "Red" granitoids does not accurately capture the geological dynamics of the studied region, resulting in misleading interpretations of the chronological sequence and changes in the tectonic setting [2, 18–20]. Instead, the magma has differentiated from calc-alkaline to alkaline affinities over time [2, 19, 30].





Fig. 8 Concordia diagram for all zircon grains with discordance percent < 10% for post-orogenic samples, plotted using lsopltR [121]

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Sample Code	ت	Concent	rations	Age (Ma)	and 2o erro	ırs						%discordance	Sample	
		²⁰⁴ Pb	²³⁸ U	²⁰⁶ Pb/ ²³⁸ U	±2σ	²⁰⁷ Pb/ ²³⁵ U	±2σ	²⁰⁷ Pb/ ²⁰⁶ Pb	±2σ	Conc.	±2σ	I	Conc. (Ma)	±2σ
Island-arc														
MI10	A3	0.001	102	1207	12	1146	10	1032	10	1145	10	-5.3	729	ĸ
	ES	600.0	173	1133	15	1145	14	1168	12	1146	13	1.1		
	E8	-0.003	181	1154	16	1159	14	1167	12	1159	14	0.4		
Syn-orogenic														
M102	E2	0.003	337	1514	56	1545	53	1588	37	1549	4	2.0	669	4
	E8	-0.005	65	704	11	723	10	781	12	723	10	2.6		
MI11	C	0.008	196	1042	29	1093	27	1194	24	1095	25	4.7	680	2
MI07	ß	-0.045	78	763	7	749	9	708	7	749	9	-1.8	646	2
Post-orogenic / I	Dokhan e	ruptions												
MI04	D3	0.002	101	579	9	594	5	648	7	594	S	2.4	623	2
Post-orogenic / I	Magmatic	: emplacem	ents											
M108	B4	0.006	165	740	10	762	6	772	13	762	6	2.9	582	m
	C	0.000	128	606	7	621	9	629	14	621	9	2.4		
	B6	-0.001	102	555	9	549	5	556	15	549	5	-1.1		
More details are pr	ovided in th	he caption of	Table 3.											



Fig. 9 Distribution chart for the analyzed single zircon U-Pb concordant ages from all samples

Conclusion

- The studied region in the CED was constructed through the island-arc, the syn-orogenic, and the post-orogenic magmatic phases (Fig. 9).
- The compressional regime of the arc-continent collision (the island-arc and syn-orogenic phases) was dominated between 729 ± 3 Ma and 646 ± 2 Ma in the CED.
- The Dokhan volcanism erupted during the tectonic transition setting between the subduction and the extension regimes from 623 ± 3 Ma to 600 ± 1 Ma.
- The post-collision tensional regime activated at 582 ± 3 Ma and may have extended to 549 ± 5 Ma.
- The reworked nature of the ANS plutonism has been emphasized by dating pre-Pan-African xenocrystic grains (Fig. 9).
- Classification of the ANS granitoids based on their chemical composition results in misleading interpretations of the chronological sequence and changes in the tectonic setting.

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Author contributions

Conceptualization, S.M., R.H., and N.H.; methodology, S.M., A.T., and N.H.; validation, S.M., R.H., K.A., A.A.S., M.S.F., and N.H.; formal analysis, A.T., M.A.G., K.A., and S.M.; investigation, S.M., K.A., R.H., M.S.F., A.A.S., and N.H.; resources, N.H., M.S.F., and K.A.; data curation, S.M., A.A.S., and M.A.G.; writing original draft preparation, S.M., R.H.; writing—review and editing, N.H., K.A., M.S.F., A.A.S., and M.A.G.; Visualization, S.M., K.A., M.S.F., and M.A.G.; project administration, R.H., S.M., A.T., K.A., and M.S.F.; funding acquisition, N.H., K.A., and M.S.F. All authors have read and agreed to the published version of the manuscript.

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Data availability

No datasets were generated or analysed during the current study.

Declarations

Competing interests

The authors declare no competing interests.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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