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CHRONOLOGICAL CONSTRAINTS ON TSAVORITE MINERALIZATIONS AND RELATED METAMORPHIC EPISODES IN SOUTHEAST KENYA

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ABSTRACT

Tsavorite is exclusively hosted in the Neoproterozoic Metamorphic Mozambique Belt (NMMB). The gemstone mines, widespread between Kalalani (Tanzania) and Mgama Ridge (Kenya), define a continuous corridor over a hundred kilometers in length. The tsavorite is hosted by a metasedimentary sequence defined as the Kurase tsavorite-bearing metasediments (Kurase-TB metasediments) that also hosts rubies. These metasediments underwent amphibolite-facies metamorphism and are surrounded by granulitic gneisses that are also of sedimentary origin (the Kurase high-temperature gneisses). All these rocks lie below the Kasigau Group, a unit dominated by granulite-facies metamagmatic rocks.

To constrain the timing of events that led to this peculiar occurrence of tsavorite, we have performed geochronological analyses of thin sections and of separated grains of zircon, monazite, and rutile using LA-ICP-MS and ID-TIMS, as well as 40Ar/39Ar of muscovite and phlogopite from various lithologies. The results show that the different terranes were metamorphosed synchronously between 620–580 Ma but under different P-T strain conditions. The Kurase-H7 gneisses and the rocks from the Kasigau Group are highly strained and underwent granulite-facies metamorphism with abundant partial melting and emplacement of felsic melts between 620 and 600 Ma. Textural observations also underlined a late regional water flux controlling the occurrence of V-free muscovite and monazite mineralizations at 585 Ma. The latter event can be related to the
activity of the Galana shear zone, in the east. The Kurase-TB metasediments escaped strain and partial melting. They record amphibolite-facies conditions with static heating, since initial sedimentary structures were locally preserved. The age of the tsvavorite mineralization was inferred at 600 Ma from metamorphic zircon rims and monazite from the closest host-rocks, sampled in the mines. Hence, tsvavorite crystallization occurred statically at the end of the metamorphic event, probably when the temperature and the amount of volatiles were at maximum levels.

Conversely, the ruby formed by local metasomatism of felsic dikes and isolated ultramafic bodies. The rubies are older and zircons and monazites from a ruby-bearing felsic dike (plumose) were dated at 615 Ma. Finally, data from rutile and micas indicate a global cooling below 430 °C of the whole region between 510 and 500 Ma.

**Keywords:** tsvavorite, zircon and monazite in situ dating, Ar-Ar dating, Kenya, Tsavo, granulite and amphibolite facies.

**INTRODUCTION**

The Neoproterozoic Metamorphic Mozambique Belt extends from the Arabian-Nubian Shield through East Africa, Madagascar, South India, and Sri Lanka to East Antarctica (Stern 1994, Kriegsman 1995). The NMNB resulted from the closure of the Mozambican Ocean and the accretion of different island arcs and continental blocks from 850 to 480 Ma (Meert 2003, Collins & Pisarevsky 2005, Fritz et al. 2013). Convergence ended with collision and the formation of the Gondwana supercontinent (Burke & Dewey 1972, Shackleton 1996). The orogenic cycle is divided into two distinct events (Meert 2003): (1) between 750 and 620 Ma, the East Africa Orogen (Stern 1994) includes the Arabian-Nubian shield, east Africa, Madagascar, the Seychelles, north-western Pakistan, southern India, and east Antarctica; (2) between 570 and 480 Ma, the Kuunga Orogen (Meert et al. 1995) corresponds to the collision of the previously combined blocks with Antarctica and Australia.

The NMNB, also called the “Gemstone Belt of East Africa” (Malisa & Muhongo 1990) or the “Pan-African Gems and Graphite Belt” (Dissanayake & Chandrajith 1999), contains many gemstone deposits with ruby, sapphire, emerald, garnets (spessartine, tsavorite, hessonite, rhodolite, “color-change” garnet), tourmaline, spinel, tanzanite, and alexandrite (Simonet et al. 2000). Some of these gems are unique and only observed in the NMNB, such as tanzanite and tsvavorite (Feneyrol et al. 2013).

Tsvavorite occurs sporadically in east Antarctica, Pakistan, and Madagascar, but is more abundant in Tanzania and Kenya. The gem-quality green vanadium-rich grossular is only mined in southern Madagascar (Vohibory), south-eastern Tanzania (Ruangwa and Tunduru, Fig. 1a), north-eastern Tanzania (Lela- tema fold belt and Kalalani), and south-eastern Kenya from Mgama Ridge to Ngomet (Fig. 1b). The alignment of mines from Kalalani (Tanzania) towards Mgama Ridge (Kenya) defines a corridor broadly oriented N170° and extending for over a hundred kilometers, forming the largest tsvavorite belt worldwide (Feneyrol et al. 2013). Metallogenetic studies of tsvavorite in the Lelatema fold belt (Tanzania) have provided new models for the genesis of tsvavorite (Olivier 2006, Feneyrol et al. 2010, 2013, 2017, Giuliani et al. 2014, Harris et al. 2014). The age of the tsvavorite mineralization was determined using either the ages of the host rocks (Le Goff et al. 2010, Feneyrol et al. 2011) or Sm/Nd dating of four tsvavorite-bearing samples in quartz veins from Mer- elani, which yielded an isochron age of 606 ± 36 Ma (Feneyrol 2012). Until now, the age of the tsvavorite formation in south-eastern Kenya has remained unknown. The closest radiometric data are for the metamorphic rocks located west of the Galana shear zone, i.e., the Sagala and Taita Hills gneisses (Fig. 2), metamorphosed between 660 and 630 Ma (Hauzenberger et al. 2007, Bauernhofer et al. 2008). The metamorphism was developed during thrusting of nappes in the east–west direction (Fritz et al. 2013).

In this paper we aim to better constrain the timing of the different metamorphic events affecting an area extending from Kalalani (Tanzania) to the Mgama Ridge (Kenya) and especially the age of the meta- morphism leading to tsvavorite formation. We integrated field observations with structural and petrographic studies of two adjacent metamorphic Groups, i.e., the Kasigau and the Kurase (Fig. 2). For the Kurase Group, we made the distinction between tsvavorite-bearing metasediments and high-temperature gneisses. We used in situ U-Th-Pb dating by LA-ICP-MS of zircon, monazite, and rutile, complemented by ID-TIMS dating of dissolved zircon and monazite. We also used 40Ar/39Ar dating of micas. The set of radiometric data allowed us to (1) define the ages of the different metamorphic units, (2) revise the age of tsvavorite formation as well as the formation of a ruby-bearing metapegmatite, and (3) determine the cooling age of the metamorphic pile. At the regional scale, knowledge of the age of gem formation and metamorphosis will result in a better understanding of the thermal evolution of the NMNB.
GEOLOGICAL SETTING

The studied area extends between the east and west Tsavo National parks and includes the town of Voi (Figs. 1b, 2), located on the main road and railway linking Nairobi to Mombasa. It notably consists of the Kasigau and Kurase Groups, which are framed to the east by a 25 km-wide granulitic vertical wrench zone called the Galana shear zone (Fig. 2) dated at 580–550 Ma (Hauzenberger et al. 2007, Bauernhofer et al. 2008). Detailed geological descriptions of the Kasigau and Kurase Groups are available in several reports and associated maps (Sanders 1963, Pohl & Niedermayr 1979, Pohl et al. 1980, Saggerson 1962, Frisch & Pohl 1986), as well as in more recent works (Mercier et al. 1999, Simonet 2000, Hauzenberger et al. 2007, Bauernhofer et al. 2008, Wamunyu et al. 2015). We have summarized and added to them in the following sections.

Geological setting of the Kasigau Group

Lithology. The Kasigau Group is dominated by quartzo-feldspathic ± garnet amphibolitic to biotitic gneisses where the highly aluminous rocks contain sillimanite or kyanite or both. Other rocks are clinopyroxene-bearing amphibolites ± wollastonite-bearing marbles, or calc-silicate layers showing biotite and amphibole replaced by orthopyroxene. Partial melting is intense and the metasedimentary rocks can be assimilated, forming metric dismembered lenses inside the quartzo-feldspathic migmatitic gneisses.

Neoproterozoic metamorphism. Hauzenberger et al. (2004, 2005) estimated metamorphic peak conditions of 820 °C and 11–12 kbar using several geothermobarometers and different lithologies (felsic gneisses and mafic rocks). This metamorphism is Neoproterozoic (665–615 Ma; Hauzenberger et al. 2007). A metamorphic overprint at amphibolite-facies conditions (590–700 °C and 6–10 kbar) is locally indicated by new muscovite growth from sillimanite and actinolite growth around hornblende. These retrograde phases grow both parallel and perpendicular to the schistosity, attesting to their post-tectonic character.

Structure. The metamorphic foliation is flat or shallowly dipping 30° to the east or the north-northeast. Ductile deformation is widespread and indicated by recumbent isoclinal folds and boudinage. Partial melting is important and sometimes leucosomes migrate into dilatant structures between boudins. The Kasigau Group is thrust over the Kurase Group. In the Taita Hills, serpentinized ultramafic rocks localized in the highly strained rocks of the thrust zone have been interpreted as an ophiolitic suture (Fig. 2, Frisch & Pohl 1986).

Geological setting of the Kurase Group

Lithology. Based on petrologic criteria, the Kurase Group has been described as a lithostratigraphic succession (from bottom to top): the Mtongore Formation, the Lualenyi Member, the Mgama-Mindi Formation including the Mtonga-Kore unit, the Mwatate Formation, and the Mugeno Formation (Pohl & Niedermayr 1979, Horkel et al. 1979, Pohl et al. 1980, Saggerson 1962).
Fig. 2. Simplified cross-section and geological map with locations of samples discussed in the text (modified from Pohl & Niedermayr 1979, Horkel et al. 1979, Pohl et al. 1980, Saggerson 1962, Simonet 2000, Hauzenberger et al. 2007, Bauernhofer et al. 2008, Feneyrol et al. 2013).
The Mtongore, Mwatate, and Mugeno Formations, as well as the Mtonga-Kore unit, are called the Kurase high-temperature gneisses (Kurase-H gneisses) in the present study. They are dominated by migmatitic biotitic gneisses. Other lithologies such as marbles and felsic charnockites, are also present. The tsavorite-bearing Lualenyi member and Mgama-Mindi Formation are called the Kurase tsavorite-bearing metasediments (Kurase-TB metasediments) in the present study. The metasediments are mainly muscovite-graphite-bearing gneisses which also contain sillimanite or kyanite or both, intercalated with meter- to several decameter-scale bands of meta-arenites and rudites, quartzites, and marbles (Wamumyu et al. 2015), and a few amphibolites. Tsavorite nodules are observed exclusively in graphitic gneisses and scapolite-diopside-anhydrite-bearing calc-silicate rocks (Suwa et al. 1996, Feneyrol et al. 2013). In a few places, primary sedimentary structures such as cross-stratification and channels are preserved. At the centimeter scale, sedimentary textures are also visible, such as large detritic K-feldspar crystals containing green detritic uvite tourmaline (Martelat et al. 2015). In the Kurase-TB metasediments, migmatization is rare. Intrusive felsic magmatic dikes have been observed. One of these dikes crosscuts isolated ultramafic bodies at the Rockland ruby mine (Simonet 2000).


The Kurase-TB metasediments parageneses reflect lower P and T conditions than the Kurase-HT gneisses (rare migmatization, widespread occurrence of large muscovite crystals, Pohl & Niedermayr 1979). Few quantitative data are available for this unit. Genetic models of tsavorite formation have shown that the transformation of diopside to tsavorite occurred isochemically in a static regime without metasomatism at 7 kbar and 680 °C (Feneyrol et al. 2013). This is in agreement with P-T conditions estimated for the Rockland mine. Thermobarometry applied to the garnet-biotite-sillimanite-graphite-bearing gneisses yields temperature and pressure in the amphibolite facies (650 °C, 7 kbar, Mercier et al. 1999). Locally, parageneses of the ruby-bearing plagioclase (e.g., sapphirine embedded in micas) and the associated metamorphosed ultrabasites are consistent with their formation under granulite-facies conditions (T = 700–750 °C and P = 8–10.5 kbar, Mercier et al. 1999). The ruby formation is associated with local metasomatism, with fluids that transformed the pegmatite in ruby ± apatite ± uvite ± phlogopite-bearing plagioclase, and the adjacent ultramafic rocks in phlogopites. In this case, the metasomatism is controlled by the intrusion of felsic magmatic dikes.

Structure. In the Kurase-TB metasediments, foliation dips east or north-northeast at around 30°. This metamorphic plane is parallel to the original stratification. Sedimentary figures such as cross-stratification and channels can be observed (Martelat et al. 2015). Thus the Kurase-TB metasediments escaped both partial melting and strain. The two contacts between the Kurase-HT gneisses and the Kurase-TB metasediments are not visible in the field due to poor exposure. Nevertheless, the contacts are sharp (a few meters thick) and the fabric is conformable on both sides. These two contacts are considered to be thrust zones (Fig. 2; Pohl & Niedermayr 1979, Pohl et al. 1980).

Analytical Methods

Fourteen localities were selected for geochronology and a set of more than 20 samples were dated: five samples from the Kasigau Group, four from the Kurase-HT gneisses, and 13 samples from the Kurase-TB metasediments. Rocks were characterized by optical microscopy and in situ chemical analyses using a CAMECA SX100 electron microprobe at the Laboratoire Magmas et Volcans (Clermont-Ferrand, France) and at CRPG (Nancy, France) operated at 15 kV and 15 nA for a 1 μm beam size and using TAP and LI F crystals.

In situ U-Th-Pb isotopic data were obtained by laser ablation inductively coupled plasma spectrometry (LA-ICP-MS) at the Laboratoire Magmas et Volcans (Clermont-Ferrand, France). The analyses involved the ablation of minerals with a Resonetics Resolution M-50 instrument incorporating an ultrashort pulse (<4 ns) ATL Atlex Excimer laser system operating at a wavelength of 193 nm (detailed description in Müller et al. 2009). Spot diameters of 15–26 μm and 9–15 μm, repetition rates of 3 and 1 Hz, and laser fluences of 4 and 15 J/cm² were used for the zircon and monazite, respectively (Supplementary Tables 1 and 2, available from the MAC Depository of Unpublished Data, document tsavorite CM55-5_10.3749/canmin.1700019). The ablated material was carried by helium and then mixed with nitrogen and argon before injection into the plasma source of an
Agilent 7500 cs ICP-MS equipped with a dual pumping system to enhance sensitivity (Paquette et al. 2014). The alignment of the instrument and mass calibration were performed before every analytical session using the NIST SRM 612 reference glass by inspecting the $^{238}\text{U}$ signal and by minimizing the ThO+/Th+ ratio (<1%). The analytical method for isotope dating is similar to that developed and described in Paquette & Tiepolo (2007) and Didier et al. (2013) and detailed in Hurai et al. (2010). The signals of the $^{204}(\text{Pb + Hg})$, $^{206}\text{Pb}$, $^{207}\text{Pb}$, $^{208}\text{Pb}$, $^{232}\text{Th}$, and $^{238}\text{U}$ masses were measured. The occurrence of common Pb in the sample was monitored by the evolution of the $^{204}(\text{Pb + Hg})$ signal intensity, but no common Pb correction was applied owing to the large isobaric interference from Hg. The $^{235}\text{U}$ signal was common Pb corrected owing to the large $^{238}\text{U}$ signal and 238U masses were measured. The occurrence of the 204(Pb + Hg), 206Pb, 207Pb, 208Pb, 232Th, and 238U isotopes and isotope ratios were monitored and carefully inspected to verify the presence of perturbations related to inclusions, fractures, mixing of different age domains, or common Pb. Calculated ratios were exported and concordia ages and diagrams were determined by irradiation of KF and CaF$_2$ pure salts with a drilled copper plate for the experiments, (2) a system of lenses for beam focusing, (3) a steel sample chamber, maintained at 10$^{-8}$–10$^{-9}$ bar, with a drilled copper plate for the samples, (4) an inlet line for purification of gases including two Zrn-Al traps, and (5) a multi-collector mass spectrometer (Argus VI from Thermo-Fisher). A custom-made software program controls the laser intensity, the timing of extraction/purification, and the data acquisition. The argon background within the system was evaluated every three sample analyses. The ArArCalc software© v2.5.2 was used for data reduction and plotting. The one-sigma errors reported for the plateau, isochron, and total gas ages include the error on the irradiation factor J. Atmospheric 40Ar was estimated using a value for the initial 40Ar/36Ar of 295.5. Due to the high radiogenic content of the dated micas, the data reported in the 36Ar/40Ar versus 39Ar/40Ar correlation plot do not provide meaningful information on the composition of the initially
trapped argon. A complete set of isotopic results is available in Supplementary Table 4.

**Sample Descriptions**

The dominant petrographic features of the rocks selected for dating are presented in Figure 3 and in the text below. For each of them, the dated phase is provided in brackets as follows: zircon (Zrn), monazite (Mnz), rutile (Rt), phlogopite (Phl), and muscovite (Msc).

For the Kasigau Group, six samples were dated (Fig. 2): Voi17a (Zrn), Voi17b (Zrn), Voi5b (Zrn), Ts2 (Mnz-Msc), and Ts3 (Zrn-Mnz). They all underwent partial melting. Sample Voi17a is a migmatitic gneiss with rare garnet and green amphibole. Sample Voi17b is a leucocratic allochthonous melt with biotite and green amphibole localized in dilatant structures cross-cutting the migmatitic foliation of Voi17a. Voi5b, collected in the Sagala Hills, is an in situ leucosome (quartz-feldspath-garnet-bearing rock, Fig. 3a) segregated in an amphibolitic migmatitic gneiss. Samples Ts2 and Ts3 are from the Maungau-Nyngala Hills. Sample Ts2 (Fig. 3b) is an aluminous layer rich in quartz, biotite, muscovite, and sillimanite. The sillimanite is partly retrogressed, resulting in large flakes of V-free muscovite (Fig. 3b, Table 1). Sample Ts3 is a garnet-biotite-bearing stromatic migmatite.

Four samples were dated from the Kurase-HT gneisses: Voi39 (Zrn), Voi40 (Zrn), Voi2a (Zrn), and Voi3 (Mnz). Sample Voi39 is a segregated leucosome composed of equilibrated grains of quartz and plagioclase, and of large multi-pillimeter-long perthitic K-feldspar crystals (Fig. 3c). Sample Voi40 is a quartzfeldspathic leucosome with oriented magnetite grains (millimetric in length) underlying a disrupted foliation. Samples Voi2a and Voi3 are felsic granulites crystallized micas. They exhibit dehydration melting of biotite resulting in peritectic garnet. In Voi3 small V-free muscovite crystals are found in discrete fractures which post-date the granulitic foliation plane (Fig. 3d, Table 1).

Eleven samples of the Kurase-TB metasediments were collected from mines or quarries: Voi28 (Zrn, Mnz), Voi29 (Zrn, Phl), Voi34–Voi35 (Mnz, Msc), Voi74 (Zrn), Voi79–Voi80 (Zrn, Rt), Voi82 (Msc), Aq1 (Phl), Ts16 (Msc), and Rockland (Mnz, Zrn). They can be separated into four groups: (1) meta-rudites, (2) graphitic gneisses, (3) veins filled with muscovite, and (4) metasomatic desilicated pegmatites at Rockland.

The meta-rudites (Voi28, Voi29, Voi35) are composed of quartz, K-feldspar, plagioclase, graphite, and muscovite (muscovite from Voi28 and Voi29 contains V; Voi29: V2O3 = 0.31 wt.%, Table 1, Fig. 3e). Sample Voi29 also contains F-rich phlogopite (1.45 wt.%, Table 1). Samples Voi28 and Voi29 have been preserved from strain and show sedimentary texture and structure. Sample Voi35 is slightly strained and contains V-free muscovite (Table 1).

The graphitic gneisses (Voi34, Voi74, Voi79, Voi80, Fig. 3f) are aluminous quartz-feldspathic rocks with sillimanite and locally kyanite. This lithology contains the calc-magnesian nodules in which tsavorite is observed (Giuliani et al. 2014). In sample Voi34, both sillimanite and muscovite with high V and Ti contents can be observed (Fig. 3f, V2O3 = 1.75 wt.%, TiO2 = 1.27 wt.%, Table 1). Muscovite seems to develop at the expense of sillimanite, but the reverse situation is also visible.

Sample Voi82 comes from a vein filled with quartz, kyanite, muscovite, and green tourmaline, which cross-cuts the graphitic metasediments (Fig. 3g). The separated muscovite contains V and Ti (V2O3 = 0.46 wt.%, TiO2 = 2.02 wt.%, Table 1). In the Aqua mine (sample Aq1), phlogopite occurs in monomineralic veins cross-cutting the main fabric of a ruby-bearing desilicated felsic dike (Fig. 3h, Table 1). In sample Ts16 the vein is horizontal in a boudin of marble within the graphitic gneiss. Indeed, V-free micas are associated with quartz, zoisite, tourmaline, and graphite (Table 1). These last three occurrences of micas were considered to be representative of the latest crystallized micas.

The desilicated pegmatites (Rockland mine from the Kimbo “pit”) are the unique metasomatic rocks formed close to, or at the contact with, the serpentinitized ultrabasic rocks. They contain ruby as well as green tourmaline.

**U-Th-Pb Geochronological Results**

**Kasigau Group**

For the stromatic migmatite sample Voi17a, 45 spots in the cores of translucent zircons yielded a concordia age of 884.8 ± 2.3 Ma (Fig. 4a; Supplementary Table 1). Nine spots were focused on fine rims which are Th-depleted and characterized by Th/U ratios mostly lower than 0.01. They provided a younger concordia age, 615.6 ± 4.6 Ma, than was provided by the cores.

The Voi17b secant leucosome contains U-rich zircons (600 < U < 2000 ppm) which are darker than the well-shaped crystals from sample Voi17a. When the most metamict grains (i.e., U content up to 3000 ppm) containing common lead are excluded and the best-preserved zircon surfaces are selected, the analytical points define a discordia line with a lower
Fig. 3. Images showing the texture of representative dated samples. In the Kasigau Group: (a) Voi5b (Sagala hills) is an in situ leucosome; (b) Ts2 (Maungau-Nyngala hills) is an aluminous layer (sillimanite is partly retrogressed, giving large muscovite grains that can be oriented at a high angle to the foliation). In the Kurase-H gneisses: (c) Voi39 is a leucosome; (d) Voi3 is a felsic granulite (small muscovite grains are found in discrete fractures). In the Kurase-TB metasediments: (e) Voi29 is a metarudite with V-rich muscovite; (f) Voi34 is a graphitic gneiss (this lithology contains the calc-magnesian nodules in which tsavorite is observed); (g) Voi82 is a vein filled with quartz, kyanite, muscovite, and Cr-rich tourmaline; (h) Aq1 is phlogopite forming mono-mineral veins cross-cutting the main fabric of a ruby-bearing desilicated felsic dike.
intercept at 598.8 ± 3.8 Ma (Fig. 4b; Supplementary Table 1).

Zircon from Voi5b segregated leucosomes is characterized by two different habits: elongated or more rounded grains. Both crystal types record large variations of U content ranging from less than 50 up to 700 ppm. No correlation between shape, U content, and calculated ages was established. An imprecise concordia age of 884 ± 27 Ma can be calculated, the latter being compatible with the older age of Voi17a. The 36 remaining spots, including 13 rims (data in red in Fig. 4c; Supplementary Table 1) yield a lower intercept age at 611.6 ± 3.9 Ma (Fig. 4c).

The aluminous layer (sample Ts2) contains rare elongated subeuhedral monazite grains included in biotite (Fig. 5a). Thirteen in situ analyses obtained from two crystals yield a concordant Th-U-Pb age of 584 ± 6 Ma (Fig. 4d; Supplementary Table 2). The Th content is high at between 3700 and 9000 ppm. The Th/U ratios are low (1.8 < Th < 4.1).

The Ts3 migmatitic gneiss contains rare zircon (Fig. 5b, c). Twenty-nine spots from zircon cores yielded a concordia age of 743.8 ± 4.4 Ma (Fig. 4e; Supplementary Table 1). These cores display typically magmatic Th/U ratios (0.2 < Th/U < 0.4–0.5). Thirteen spots on the rims provided a concordia age of 610.5 ± 4.3 Ma (Fig. 4e). These rims are characterized by higher U and lower Th contents than the cores, resulting in low Th/U ratios (Th/U < 0.1). This trend is quite similar to what is observed in sample Voi17a, both rim ages being comparable. The five monazite grains separated from sample Ts3 are a bit cloudy, rounded, and fractured, 100 to 200 µm wide. The Th-U-Pb analyses display two groups of ages (Fig. 4f; Supplementary Table 2). The first group (18 analyses from four grains) yielded a concordant age of 585 ± 4 Ma. The second group (14 analyses from three grains) yielded a concordant age of 516 ±

### Table 1. Representative Muscovite and Phlogopite Analyses

<table>
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<tr>
<th>Mineral</th>
<th>Muscovite</th>
<th>Biotite</th>
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<tbody>
<tr>
<td>Sample</td>
<td>Ts2</td>
<td>Voi29</td>
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<tr>
<td>SiO₂ (wt.%)</td>
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<td>48.34</td>
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<tr>
<td>TiO₂</td>
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<td>Al₂O₃</td>
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<td>FeO</td>
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</tr>
<tr>
<td>MnO</td>
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<td>0.00</td>
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<tr>
<td>K₂O</td>
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<tr>
<td>F</td>
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<td>0.08</td>
</tr>
<tr>
<td>Total</td>
<td>94.93</td>
<td>95.61</td>
</tr>
</tbody>
</table>

Structural formulae calculated on the basis of 11 O pfu

| Si          | 3.091 | 3.16      | 3.055 | 3.119 | 3.122 | 3.214 | 3.058 |
| Al<sup>IV</sup> | 0.909 | 0.84      | 0.945 | 0.881 | 0.878 | 0.786 | 0.942 |
| Al<sup>VI</sup> | 1.702 | 1.72      | 1.624 | 1.646 | 1.868 | 1.718 | 1.874 |
| Ti          | 0.047 | 0.10      | 0.156 | 0.064 | 0.006 | 0.021 | 0.017 |
| Cr          | 0.000 | 0.00      | 0.000 | 0.010 | 0.001 | 0.001 | 0.000 |
| V           | 0.000 | 0.01      | 0.016 | 0.094 | 0.000 | 0.005 | 0.000 |
| Fe          | 0.205 | 0.00      | 0.007 | 0.004 | 0.001 | 0.002 | 0.103 |
| Mn          | 0.001 | 0.00      | 0.000 | 0.000 | 0.002 | 0.003 | 0.000 |
| Mg          | 0.099 | 0.20      | 0.211 | 0.191 | 0.115 | 0.332 | 0.028 |
| Ca          | 0.002 | 0.00      | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| Na          | 0.066 | 0.06      | 0.048 | 0.040 | 0.055 | 0.030 | 0.133 |
| K           | 0.936 | 0.75      | 0.911 | 0.942 | 0.957 | 0.826 | 0.855 |

Structural formulae calculated on the basis of 22 O pfu

| Si          | 3.091 | 3.16      | 3.055 | 3.119 | 3.122 | 3.214 | 3.058 |
| Al<sup>IV</sup> | 0.909 | 0.84      | 0.945 | 0.881 | 0.878 | 0.786 | 0.942 |
| Al<sup>VI</sup> | 1.702 | 1.72      | 1.624 | 1.646 | 1.868 | 1.718 | 1.874 |
| Ti          | 0.047 | 0.10      | 0.156 | 0.064 | 0.006 | 0.021 | 0.017 |
| Cr          | 0.000 | 0.00      | 0.000 | 0.010 | 0.001 | 0.001 | 0.000 |
| V           | 0.000 | 0.01      | 0.016 | 0.094 | 0.000 | 0.005 | 0.000 |
| Fe          | 0.205 | 0.00      | 0.007 | 0.004 | 0.001 | 0.002 | 0.103 |
| Mn          | 0.001 | 0.00      | 0.000 | 0.000 | 0.002 | 0.003 | 0.000 |
| Mg          | 0.099 | 0.20      | 0.211 | 0.191 | 0.115 | 0.332 | 0.028 |
| Ca          | 0.002 | 0.00      | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| Na          | 0.066 | 0.06      | 0.048 | 0.040 | 0.055 | 0.030 | 0.133 |
| K           | 0.936 | 0.75      | 0.911 | 0.942 | 0.957 | 0.826 | 0.855 |

XMg 0.98 0.99
In most of the monazite, the Th content varies between 10,300 and 27,800 ppm and the U content varies between 132 and 1280 ppm (1.4 < Th/U < 149) with no clear correlation with the measured age. The Th/U values for the youngest samples measured are usually ≥24.5 and contain among the lowest values of U (132 < U < 930 ppm) compared to the monazite grains in other units. Some monazite domains which

FIG. 4. Geochronological results obtained from zircon and monazite from the Kasigau Group. Data ellipses for each analysis are defined by standard 2σ errors (Supplementary Table 1).
belong to the “585 Ma” group are enriched in Th (between 45,500 and 59,500 ppm) and, more surprisingly, are strongly enriched in U (up to 31,100 ppm), resulting in low Th/U ratios (1.5 to 10.8).

**Kurase high-temperature gneisses**

The Voi39 pegmatite is characteristic of a differentiated felsic melt. Thirty-five spots on zircons resulted in a single concordia age of 606.5 ± 3.0 Ma (Fig. 6b; Supplementary Table 1), interpreted as the age of crystallization for this pegmatite. Sample Voi40 is a leucosome with a slight fabric containing large (sometimes millimetric in length, Fig. 5d–f) and euhedral zircon crystals, displaying an oscillatory or sector zoning. Twenty-eight spots yielded a concordia age of 616.7 ± 3.7 Ma (Fig. 6a; Supplementary Table 1), which is slightly older than sample Voi39.

Two large monazite grains, one in the quartzofeldspathic matrix, one in a garnet (~150 μm in diameter, Fig. 5g), were analyzed *in situ* in the stromatic migmatite Voi3. Thirty-two analyses yielded a concordant Th-U-Pb age of 578 ± 3 Ma (Fig. 6c; Supplementary Table 2). The Th and U contents vary from 8400 to 43,500 ppm and from 640 to 2540 ppm, respectively, with no correlation with the measured age. The Th/U ratios are between 4 and 60. The felsic granulite sample Voi2a contains rounded zircon with irregular zoning characteristic of metamorphic crystallization. Data plot around 610 Ma (Fig. 6d; Supplementary Table 1), nevertheless, two time-populations may be distinguished within analytical uncertainties. Thirty-six spots from crystal cores define a concordia age of 619.0 ± 2.9 Ma, and 10 spots mainly located in the U-poor rims yield a concordia age of 599.1 ± 7.4 Ma.
Kurase tsavorite-bearing metasediment

In the slightly strained meta-rudite sample Voi35, two types of monazite grains have been found: large grains showing complex chemical zoning (up to 300 μm in length, Fig. 5h) and smaller grains around 70 μm in diameter. Two groups of ages are obtained; they do not correlate with the grain size (Fig. 7b; Supplementary Table 2). The older group yields a concordant Th-U-Pb age of $615 \pm 2$ Ma ($n = 27$) and is characterized by a strong enrichment in U (up to 45,870 ppm) and very low Th/U ratios (between 0.5 and 3). The younger group consists of five $^{208}\text{Pb}/^{232}\text{Th}$ ages scattered between $507 \pm 13$ and $553 \pm 14$ Ma. The U contents in these monazite domains are lower than in the older monazites ($1700 < \text{U} < 7700$ ppm) and the Th/U ratios are between 5 and 20.

The meta-rudite samples Voi28 and Voi29 contain rare yellow zircon crystals which are cloudy with numerous cracks. They are often highly U-enriched (up to 6000 ppm U), and consequently mostly metamict, and often contain significant amounts of common Pb. Only the analyses of the best-preserved crystals were selected and they define two similar concordia ages of $603.4 \pm 4.5$ Ma and $606.7 \pm 5.8$ Ma for samples Voi28 and 29, respectively (Fig. 7c, d; Supplementary Table 1). These are maximum ages because a weak contribution of common Pb remains possible. Four monazite grains separated from sample Voi28 were analyzed. Thirty-two analyses yield a concordant Th-U-Pb age of $596 \pm 2$ Ma (Fig. 7e; Supplementary Table 2). In this sample the Th and U contents vary from 19,800 to 53,200 ppm and from 1660 to 8040 ppm, respectively. The Th/U ratios are between 4 and 14.

In the graphitic gneisses, zircon crystals are rare compared to the other rocks. In the three samples, the oldest measured ages scatter between 650 and 850 Ma (Fig. 7f, g; Supplementary Table 1). Ten spots from

**Fig. 6.** Geochronological results obtained from zircons and monazites for samples (A) Voi40, (B) Voi39, (C) Voi3, and (D) Voi2a (Kurase-HT gneisses). Data ellipses for each analysis are defined by standard $2\sigma$ errors (Supplementary Tables 1 and 2).
graphitic gneiss Voi74 yielded a concordia age of $596.4 \pm 3.9$ Ma (Fig. 7f), which is in agreement with individual spot analyses obtained from samples Voi79–Voi80 (Fig. 7g). All these analyses plotting at 600 Ma correspond to rims displaying high U (>800 ppm) content and a low Th/U ratio (<0.05, Fig. 5i,j). Finally, in samples Voi74 and Voi79, a few spots yielded unusually young ages of around 450 Ma (Fig.
7f, g). Like zircon, monazite is very rare in the graphitic gneiss. Only sample Voi34 provided a few small elongated or rounded monazite grains (three, 20–150 μm in length, Fig. 5k). The Th-U-Pb data are scattered between 737 and 547 Ma (Fig. 7a). Considering that the older ages (between 747 and 660 Ma) can be attributed to inheritance, a concordant U-Pb age was calculated at 595 ± 6 Ma (n = 17), in agreement with the weighted average 208Pb/232Th age of 597 ± 11 Ma (MSWD = 5.2).

The desilicated pegmatites from the Kimbo pit in the Rockland mine provided U-rich zircon (3700 ppm). Five zircon crystals were analyzed by ID-TIMS, and they show upper intercepts of 612.4 ± 0.5 Ma (Fig. 8a; Supplementary Table 3). The 208Pb/206Pb ratio can be considered as a proxy of the Th/U ratio and provides very low values, below 0.01. Finally, samples from the Kimbo pit provided three euhedral abraded monazite crystals. Analyses performed with an ID-TIMS indicate upper intercepts at 609.0 ± 3.3 Ma (Fig. 8b; Supplementary Table 3).

**U-Pb rutile geochronology**

Rutile grains from two graphitic gneisses (Voi79 and Voi80) were dated. Nineteen spots were analyzed in each sample and yield a concordia age of 515.2 ± 4.1 Ma and a lower intercept age of 515.2 ± 6.2 Ma for Voi79 and Voi80, respectively (Fig. 9a, b; Supplementary Table 1).
ARGON GEOCHRONOLOGICAL RESULTS

Phlogopite and muscovite grains selected for dating are mainly representative of the Kurase-TB metasediments, but one sample comes from the Kasigau Group (Fig. 2). Results from single grains of muscovite and phlogopite (Table 1) are discussed together, as they are considered, for this specific chemistry, to record the same Ar-cooling temperature (around 430 °C, Harrison et al. 2009). Taken together, all phyllosilicates display high precision plateau ages for at least 65% of the 39Ar released (Fig. 10; Supplementary Table 4). For two samples (Ts16 and Voi35), age determinations of two muscovite grains are relatively consistent with the flat portion of the spectra, yielding plateau ages between 509.4 ± 1.8 Ma and 504.4 ± 1.8 Ma for Ts16 (Fig. 10c) and between 498.7 ± 1.7 Ma and 504.4 ± 1.8 Ma for Voi35, which indicates a homogeneous isotopic composition of these micas for argon. Considering all the results, the obtained ages vary from 518.3 ± 1.5 to 495.0 ± 1.5 Ma. The ages from phlogopite (sample Voi29 and Aq1) are similar to the ones measured in muscovite. Micas from the rock matrix show lobed grain limits (Voi29, 499.0 ± 1.4 Ma) and display ages similar to the late crystallized ones with nicely euhedral shapes taken from dilatant structures (Aq1, 508.1 ± 1.5 Ma). Samples Voi34 and Voi82 correspond to the graphitic gneiss hosting the productive nodule and the V-rich muscovite (Table 1). They display ages consistent with the other rocks. The ages are slightly higher in sample Voi82, where the grains are large (centimetric) with gem-quality kyanite (518.3 ± 1.5 Ma, Figs. 3g, 10f). The sample from
Kasigau (Ts2) contains muscovite yielding the youngest age (Fig. 10a). The first third of the age spectrum measured provides scattered ages, with a saddle-shaped pattern. However, a plateau age of 495.0 ± 1.5 Ma can be calculated for 60% of the 39Ar released. Thus we consider that the results are quite homogeneous and that all samples recorded a common history of cooling below 430 °C which occurred between 510 and 500 Ma. This is compatible with U-Pb results obtained for rutile from samples Voi79 and 80. The slight spread of ages from 495.0 to 518.3 Ma can be explained by different crystal sizes (sample Voi82) acting on argon retentivity and by weak argon loss associated with weathering (Ts2).

**Linking Late Neoproterozoic Events to Metamorphism**

Ages measured from zircon and monazite display a statistically dominant group (Fig. 11) corresponding to late Neoproterozoic ages ranging from 620 to 585 Ma. We discuss below their relationship with regional tectonometamorphic events undergone by the Kasigau Group, the Kurase-HT gneisses, and the Kurase-TB metasediments.

**Kasigau Group**

In the Kasigau Group, zircon from high-grade migmatites shows metamorphic rims dated at 615.6 ± 4.6 Ma (Voi17a) and 610.5 ± 4.3 Ma (Ts3). These are synchronous with the crystallization ages of the segregated leucosomes (611.6 ± 3.8 Ma, Voi5b). This suggests that this age corresponds to the peak conditions of the metamorphic event in the granulite facies (820 °C, 11–12 kbar, Hauzenberger et al. 2004). These results are in agreement with previous studies (665–615 Ma, Hauzenberger et al. 2007). The crystallization age of a felsic dike (Voi17b), cross-cutting the high-grade migmatite (Voi17a), is younger (598.8 ± 3.8 Ma). In Ts2, clear retrogression in the amphibolite facies is shown by the crystallization of large millimetric muscovite grains from sillimanite cutting across the foliation planes. The associated monazite crystals record a single age of 584 ± 5 Ma that is interpreted to be the age of the retrogression in the amphibolite facies associated with water influx. This is also supported by monazites in sample Ts3 that provided very similar results (18 spots displaying ages of 585 ± 4 Ma). Sample Ts3 also contains fractured and U-poor monazites which give younger ages of 516 Ma ± 5 Ma (14 spots) and are not related to any mineral overprint.

**Kurase high-temperature gneisses**

To constrain the granulite facies event in the Kurase-HT gneisses, we selected rocks with clear evidence of fluid-absent biotite melting (Voi2a, Voi3) in addition to pegmatite rocks and segregated leucosomes close to the felsic charnockites (Voi39, Voi40). Zircon dating provided a crystallization age of pegmatite sample Voi39 of 606.5 ± 3.0 Ma and of the leucosome in sample Voi40 of 616.7 ± 3.7 Ma. Metamorphic zircons from the felsic granulite Voi2a display two age populations: one at 619.0 ± 2.9 Ma (36 spots in zircon cores) and the other at 599.1 ± 7.4 Ma (10 spots in rim). This suggests that the granulite-
facies event (820 °C 11–12 kbar, Hauzenberger et al. 2004) occurred at the same time as continuous pulses of magma between 620 and 600 Ma. These ages are slightly younger than those obtained for the same lithology northward (Figs. 2, 11; sample K37, 660 to 630 Ma, Hauzenberger et al. 2007). Monazite grains from the similar felsic granulite Voi3 recorded different ages, even when located inside the garnet produced by dehydration melting of biotite. The 32 analyses yield a concordant Th-U-Pb age of 578 ± 3 Ma, compatible with the occurrence of small muscovite grains in late fractures. This age is similar to the one associated with clear retrogression in the amphibolite facies conditions in the Kasigau Group (monazite from Ts2 and Ts3). As monazite crystallization is highly sensitive to fluid circulation, those ages may be related to volatile migration along garnet fractures during retrogression.

Kurase tsavorite-bearing metasediments

Samples Voi34 and 35 come from the northern part of Mgama Ridge. The slightly strained meta-rudite sample (Voi35) bears large U-rich monazites that provided one group of ages at 615 ± 2 Ma (n = 27). As the monazites are found in the same layers as the centimetric muscovites, it suggests that the 615 Ma age reflects amphibolite-facies conditions. Four analyses yield a concordant Th-U-Pb age of 578 ± 3 Ma, compatible with the occurrence of small muscovite grains in late fractures. This age is similar to the one associated with clear retrogression in the amphibolite facies conditions in the Kasigau Group (monazite from Ts2 and Ts3). As monazite crystallization is highly sensitive to fluid circulation, those ages may be related to volatile migration along garnet fractures during retrogression.

Ages of the ruby-bearing plumasites (Rockland mine) are interesting in two ways: first, they are associated with a rare example of metasomatism associated with gem formation, and second, the desilication occurred under local granulite-facies conditions (T = 700–750 °C and P = 8–10.5 kbar, Mercier et al. 1999). In this case, separated zircon and monazite crystals display similar results with ages of 612.4 ± 0.5 and 609.0 ± 3.3 Ma, respectively, attesting to their synchronous crystallization.

DISCUSSION AND CONCLUSIONS

Tonien inheritance

Ages older than 700 Ma are recorded in some zircon cores and rarely in monazites (Voi34) from the Kasigau Group and the Kurase-TB metasediments and not from the Kurase-H7 gneisses. These ages are in agreement with those obtained from neighboring rocks (Hauzenberger et al. 2007). In this study, all the rocks belong to the Eastern Granulite Belt and display inherited ages older than 700 Ma, usually interpreted as emplacement ages of the magmatic rocks (850–950 Ma) with no record of Archean or Paleoproterozoic events (Hauzenberger et al. 2007, Fritz et al. 2009, 2013, Tenczer et al. 2013).

Late Neoproterozoic metamorphic events

In the regional frame of the East African Orogen, granulite-facies metamorphism was associated with hot nappes stacking in the interval 640–620 Ma (Tenczer et al. 2013). The younger ages reported in these studies are in agreement with the 620–615 Ma ages obtained in the present study. The absence of ages between 630 and 645 Ma in our samples may illustrate the regional east–west diachronism of the granulite-facies metamorphism in the belt (Tenczer et al. 2013). It could also be the result of heterogeneous finite strain as demonstrated to the west, in the Lossogonoï district, where newly formed metamorphic zircons in a vertical shear zone formed under amphibolite-facies conditions at 610 Ma and postdate the Pan-African metamorphic peak at 640–620 Ma (Le Goff et al. 2010).

Monazites from the migmatites in the Kasigau Group and Kurase-H7 gneisses yield concordant U-Th-Pb ages of about 580 Ma (samples Ts2, Ts3, Voi3). These ages only recorded in monazites are comparable with the one obtained eastward in the Galana vertical shear zone where they are associated with granulite-facies metamorphism (Hauzenberger et al. 2007) or magmatic dike emplacement to the north (Simonet et al. 2004). This 580 Ma event is linked to the final amalgamation of micro-continents forming Gondwana (Kuunga orogeny, Meert 2003). Thus we consider that...
shearing along the large Galana Shear zone and the emplacement of magmatic dikes are associated with local to more pervasive fluid mobility in neighboring terrains, controlling monazite crystallization at 585 Ma.

Synchronous metamorphism of Kasigau and Kurase Groups

As shown by Hauzenberger et al. (2004, 2007), our results show that the Kasigau and Kurase-HT gneisses (Taita Hills) synchronously underwent metamorphism at the same peak conditions with abundant partial melting. In the Kurase Group, the Kurase-TB metasediments escaped partial melting and were preserved from deformation. Moreover, sedimentary structures were preserved. These rocks were metamorphosed at lower grade than the Kurase-HT gneisses, under amphibolite-facies conditions, and in a very static regime. Ages close to 615 Ma are only recorded in sample Voi35, which is strained, and at Rockland, where local metasomatism and higher metamorphism occurred (granulite-facies conditions, Mercier et al. 1999). Our results show that these three units, Kasigau Group, Kurase-HT gneisses, and Kurase-TB metasediments, were metamorphosed at the same time (620 to 600 Ma), but under different P-T and strain conditions.

Timing of tsavorite crystallization

No U-rich minerals have been found in the tsavorite nodules. In our area, the samples found closest to tsavorite nodules of gem quality are the Voi34, Voi74, Voi79, and Voi80 samples. These samples contain rare monazite and zircon that crystallized synchronously at around 600 Ma. This is the youngest part of the amphibolite-facies metamorphism recorded in the Kurase-TB metasediments from 615 to 600 Ma. Tsavorite formed in nodules, the largest 10 cm in diameter, that were statically heated during prograde metamorphism up to 7 kbar and 680 °C (Feneyrol et al. 2012, 2017; Giuliani et al. 2014). Thus tsavorite grew during isochronal metamorphism by the transfer of elements over more than 10 cm. Taking into account the size of the nodules and the lack of strain, we suggest that the tsavorite formation is related to a slow mechanism that may have been completed at the end of the prograde metamorphism at around 600 Ma when the temperature and the amount of volatiles were at their highest.

Paleozoic event

In the Kurase Group, ages close to 516 Ma are recorded only in separated, U-depleted monazite crystals (Ts3). Other isolated spots give young dates of 530 ± 6 Ma (n = 5, Voi35). These dates may be related to more isolated events due to local tectonic structures. Such ages have been associated with the latest events affecting Gondwana, e.g., in zircon-bearing calcite veins (Paquette et al. 1994). These ages at around 516 Ma are close to the 40Ar/39Ar ages of 510–500 Ma and to the rutile ages of 520–495 Ma. In the Aqua mine two rutiles included in corundum (ruby and pink sapphire) display compatible U-Pb ages of 533 ± 11 and 526 ± 13 Ma (in situ ICP-MS dating, Sorokina et al. 2017). 40Ar/39Ar ages have been obtained from muscovite and biotite from the same area (Bauernhofer et al. 2009). Their interpretation is not straightforward due to inherited argon in some samples. Nevertheless, an unfoliated pegmatite from the Taita Hills yielded an age of 505 Ma and two well-foliated mafic migmatites from the Galana shear zone display plateau ages of 500 and 490 Ma. Moreover, in this part of the Mozambican belt, Feneyrol (2012) reported similar 40Ar/39Ar results of 516–492 Ma for a marble and two graphitic gneisses from Meralani, Lemshucu, and Namalulu. Möller et al. (2000) dated several samples and especially rutile crystals from two metapelites from the Kurase Group (samples A108 and A144) displaying 206Pb/238U ages of around 529–510 Ma, consistent with our U-Pb ages in rutile. Element-diffusion estimates from garnet-biotite profiles in samples from the Kurase Group (Taita Hills) suggest a slow cooling rate of about 3–5 °C/my (Hauzenberger et al. 2005). Data converge to a cooling below 430 °C at a regional scale at 510–500 Ma. This is compatible with a slow cooling rate (T < 5 °C/my), operating over a large area, indicative of large-scale geological processes, such as isostatic readjustment, instead of fast tectonic processes.

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