



Supplementary material: Amphibolite facies metamorphic event within the Upper Sebtides tectonic units (Internal Rif, Morocco): a record of a hyperextended margin at the border of the western Tethys

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⁴⁰Ar/³⁹Ar dating—analytical procedures

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The samples BB16-12B, BB16-43 and BB16-14 were crushed and 200–315 µm size fraction was cleaned in ultrasonic bath. Muscovite and biotite were carefully handpicked under a binocular microscope to select only grains without evidence of alteration or inclusions. Selected grains were packaged in aluminum foils and were irradiated for 97 h in the McMaster Nuclear Reactor together with Fish Canyon sanidine grains as flux monitor (28.030 ± 0.056 Ma, Jourdan and Renne, 2007). The argon isotopic interferences on K and Ca were determined by the irradiation

of KF and CaF₂ pure salts from which the following correction factors were obtained: $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 2.97 \times 10^{-2} \pm 10^{-3}$ at 1σ, $(^{38}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 1.24 \times 10^{-2} \pm 5 \times 10^{-4}$ at 1σ, $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 7.27 \times 10^{-4} \pm 4 \times 10^{-5}$ at 1σ, and $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 2.82 \times 10^{-4} \pm 3 \times 10^{-5}$ at 1σ. ⁴⁰Ar/³⁹Ar step heating analyses were performed at Geoazur Nice (France) using a continuous 100 W PhotonMachine CO₂ (IR) laser used at 5–15% during 30 s. Argon isotopes were measured in static mode using an ARGUS VI mass spectrometer from Thermo-Fischer. Measurements were carried out in multi-collection mode using four Faraday cups equipped with 10¹² ohm (masses 40, 39, 38 and 37) and one low-background compact discrete dynode ion counter to measure mass 36. Collector gain calibration is performed by the computer-controlled application of predetermined voltages to each collec-

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tor. Mass discrimination for the mass spectrometer was monitored by regularly analyzing air pipette volumes. The raw data were processed using the ArArCALC software [Koppers, 2002], and ages were calculated using the decay constants given by Steiger and Jäger [1977]. Blanks were monitored after every three sample analyses. All parameters and relative abundance values are provided in Supplementary Materials 2–6 and have been corrected for blanks, mass discrimination, and radioactive decay. Atmospheric ^{40}Ar was estimated using a value of the initial $^{40}\text{Ar}/^{36}\text{Ar}$ of 298.56 [Lee *et al.*, 2006]. Our criteria for the determination of a plateau are as follows: a plateau must include at least 70% of ^{39}Ar released, over a minimum of three consecutive steps agreeing at 95% confidence level. Plateau ages are given at the 2σ error level, and the plateau age uncertainties include analytical and J -value errors. All the errors on the inverse isochron, total fusion ages, and initial $^{40}\text{Ar}/^{36}\text{Ar}$ ratios are quoted at the 2σ error.

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Samples BB16-46 were crushed and sieved and the selected grain size for the crystals was in the order of 100–200 μm , then they were cleaned and dried. Muscovites were finally selected under a binocular microscope. Samples were packed in aluminum foil for irradiation in the core of the Triga Mark II nuclear reactor of Pavia (Italy) with several aliquots of the Taylor Creek sanidine standard (28.34 ± 0.08 Ma, Renne *et al.*, 1998) as flux monitor. Argon isotopic interferences on K and Ca were determined by irradiation of KF and CaF₂ pure salts from which the following correction factors were obtained: $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 0.00969 \pm 0.00038$, $(^{38}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 0.01297 \pm 0.00045$, $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.0007474 \pm 0.000021$ and $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.000288 \pm 0.000016$. Argon analyses were performed at Géosciences Montpellier Laboratory (France). The gas extraction and purification line consists of (a) an IR-CO₂ laser of 100 kHz used at 3–15% power to heat samples during 60 s, (b) a lenses system for beam focusing, (c) a steel chamber, maintained at 10^{−8}–10^{−9} bar, with a copper holder in which 2 mm-diameter blind holes were milled, (d) two Zr–Al getters for purification of gases. Two different mass spectrometers

were used: a MAP 215-50 noble gas mass spectrometer and a multi-collector mass spectrometer (Argus VI from Thermo-Fisher). Aliquots of 40 to 50 grains of biotite and white micas were distributed as micropopulation five to ten grain deep in one and two holes of the copper holder, respectively, and were step heated. Blank analyses were performed every three sample analyses. Raw data of each step and blank were processed and ages were calculated using the ArArCALC-software [Koppers, 2002]. The criteria for defining plateau ages are: (1) plateau steps should contain at least 70% of released ^{39}Ar , (2) there should be at least three successive steps in the plateau and (3) the integrated age of the plateau should agree with each apparent age of the plateau within a 2σ confidence interval. All the subsequent quoted uncertainties are at the 2σ level including the error on the irradiation factor parameter J . With the MAP spectrometer the atmospheric contribution was difficult to determine precisely because of a high ^{36}Ar background on blanks. Thus, errors on individual ages are large (up to 5%) but we choose to present these results because they are complementary to those obtained on the Argus. Raw data can be downloaded from Supplementary Material 7.

References

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