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Direct shear fabric dating constrains early Oligocene onset of the South Tibetan detachment in the western Nepal Himalaya Soucy La Roche et al.

1 SUPPLEMENTARY METHODS

2 Quartz crystallographic <c>-axis preferred orientations

Quartz crystallographic <c>-axis preferred orientations (CPO; Law, 1990 and 3 references therein) were measured by Camille Partin with a G50 Automated Fabric 4 5 Analyzer at the University of Saskatchewan, Saskatchewan, with a 5 µm 6 resolution on a thin section cut perpendicular to the foliation and parallel to the 7 lineation (Wilson et al., 2007; Peternell et al., 2010; Wilson and Peternell, 2011). This 8 method gives results equivalent to those determined using X-ray (Wilson et al. 2007) 9 and electron back-scattered diffraction (EBSD) (Peternell et al., 2010, 2011). Quartz 10 grains were manually picked with the INVESTIGATOR software (Peternell et al., 11 2010) and a retardation and geometric quality filter (>80%) was applied to all the 12 optical axes plunging less than 70° to the surface of the thin section. CPO fabrics can 13 be used to quantitatively determine the temperature of deformation \pm 50 °C, assuming 14 that strain rate and hydrolytic weakening are not primary controls on the critical 15 resolved shear stress for active slip systems (Kruhl, 1998; Law et al., 2004; Morgan 16 and Law, 2004; Law, 2014). All <c> axis orientation data are presented in equal-area 17 lower-hemisphere stereographic projections oriented such that foliation is represented 18 by a vertical east-west plane and lineation by a horizontal east-west line. 19 Stereographic projections were produced with *Stereonet* (v9) software package of R. 20 Allmendinger.

21 U-Th/Pb Petrochronology

22 Monazite were identified in thin section using a mineral liberation analyzer 650 field 23 emission gun environmental scanning electron microscope (MLA 650 FEG ESEM; 24 Fandrich et al., 2007) at Queen's University, Kingston, Ontario, Canada with the help 25 of Agatha Dobosz. Suitable monazite was selected on the basis of size, lack of cracks 26 and inclusions and relationship to deformation fabrics. 27 Recent studies highlight the need for chemical characterization of monazite to 28 distinguish between different episodes of monazite (re-)crystallization and to avoid 29 sampling multiple domains (Gibson et al., 2004; Kohn et al., 2005; Martin et al., 30 2007; Cottle et al., 2009; Larson et al., 2011). Selected monazite grains were therefore 31 chemically mapped for U, Th, Y, Ca and Si at Queen's University, Kingston, Ontario, 32 Canada, with wavelength dispersive spectrometry on a JEOL JXA-8230 electron 33 microprobe under the supervision of Brian Joy. The electron microprobe experimental 34 conditions were set at an acceleration voltage of 15 kV, beam current of 200 nA, 35 dwell time of 100 ms and step size of $0.5-0.6 \,\mu\text{m}$. X-ray maps were used to identify 36 zonation, detect inclusions and select laser spot locations. 37 Three to sixteen 9.7 µm large spots per grain were analyzed in 7 monazite grains for 38 U-Th/Pb isotopes and trace element concentrations by laser-ablation split stream 39 using dual multi-collector-single-collector inductively-coupled plasma mass 40 spectrometry (LASS-ICPMS) at University of California, Santa Barbara, California, 41 United States of America. Analytical methods are described in Cottle et al. (2012, 42 2013) and Kylander-Clark et al. (2013). Mass bias and Pb/U and Pb/Th downhole 43 fractionation were monitored and corrected for using a primary reference monazite, 44 "44069" (424 Ma, Pb/U isotope dilution-thermal ionization mass spectrometry age 45 (ID-TIMS) (Aleinikoff et al., 2006)). A secondary reference monazite "FC-1" (55.7

46	Ma Pb/U ID-TIMS age, (Horstwood et al., 2003)) was analyzed concurrently once
47	every eight unknowns to monitor isotopic data accuracy. Trace element concentrations
48	were normalized to an in-house reference "Bananeira" monazite and, based on the
49	long-term reproducibility of secondary reference monazites, are accurate to 3-5%.
50	During the analytical period, repeat analyses of FC-1 gave a weighted mean
51	206 Pb/ 238 U date of 56.4 ± 0.2, mean square weighted deviation (MSWD) = 2.0 (n=71),
52	and a weighted mean 208 Pb/ 232 Th date of 55.8 ± 0.3 Ma, MSWD= 1.9 (n = 60). Data
53	reduction, including corrections for baseline, instrumental drift, mass bias, downhole
54	fractionation, and uncorrected date calculations were carried out using Iolite version
55	2.5. Full details of the data reduction methodology can be found in Paton et al. (2010).
56	Diagrams were constructed with Excel 2013. All uncertainties are quoted at 2σ and
57	include contributions from the external reproducibility of the primary reference
58	material for the 207 Pb/ 206 Pb, 206 Pb/ 238 U and 208 Pb/ 232 Th ratios.
59	Despite much care in selecting analytical spots, analyses overlapping with more than
60	one chemical domain, with inclusions or with matrix material can occur in grains with
61	narrow chemical domains or because of the depth of the ablation pit (3-4 μ m). We
62	identified suspect analyses based on a comparison of ablated monazite grains
63	microphotographs with chemical maps and based on the time-resolved isotopic signal.
64	Such analyses were excluded from geochronological results and interpretations.
65	Mixed domain analyses can nonetheless go undetected if the boundary between the
66	two domains was, at depth, perpendicular to the ablation surface. This would result in
67	ablation of monazite in equal proportions from both domains throughout the ablation
68	pit and would produce a stable, mixed signal of intermediate date and trace element
69	composition.
70	Himalayan monazite grains, due to their young age, have little accumulated

70 Himalayan monazite grains, due to their young age, have little accumulated

71	radiogenic ²⁰⁷ Pb and therefore yield imprecise ²⁰⁷ Pb/ ²³⁵ U dates, which in addition,
72	makes a correction for common Pb less accurate and precise. Furthermore, excess
73	206 Pb from unsupported 230 Th decay (Schärer, 1984) results in 206 Pb/ 238 U dates that are
74	too old relative to the 'true' date. Therefore in this study only 208 Pb/ 232 Th dates,
75	uncorrected for common-lead, are used for interpretations. A common lead correction
76	is not necessary for 208 Pb/ 232 Th dates, given the high thorigenic component to
77	monazite. This is supported by visual inspection of 208 Pb/ 232 Th versus 206 Pb/ 238 U
78	Concordia plots in which analyses with the least 'excess' ²⁰⁶ Pb are essentially
79	concordant.

40 Ar/ 39 Ar step-heating and total fusion analyses

Samples were gently crushed and sieved, and then rinsed several times with water to 81 82 remove fine particles. A small amount of the 250-707 µm fraction of crushed material was placed in a Petri dish with ample amounts of ultra-pure ethyl alcohol. The best 83 84 quality mineral grains or grain aggregates were then picked under a binocular 85 microscope and allowed to dry completely. Individual mineral separates were loaded 86 into 2-3 mm-deep aluminum foil packets which were subsequently stacked vertically 87 into 35-mm long foil tubes and placed into the tubular holes of an aluminum cylinder. 88 Several flux monitor grains of Fish Canyon tuff sanidine (FCT-SAN) (28.201 ± 0.023) 89 1σ Ma; Kuiper et al., 2008) were loaded into each sample packet. J values were 90 interpolated for samples situated between the spaced FCT-SAN monitor grains. GA 91 1550 biotite (99.15 \pm 0.8 1 σ Ma, Spell and McDougall (2003) recalculated to FCT-92 SAN at 28.201 ± 0.023 1 σ Ma, and Min et al. (2000) decay constant) was used as a 93 secondary standard to confirm the accuracy of the interpolations. GSC Irradiations 94 #70 and #71 were each shielded with Cd and irradiated for 160 MWH in medium flux 95 position 8B at the research nuclear reactor of McMaster University (MNR) in

96	Hamilton, Ontario, Canada. Neutron fluence was c. 0.9 x 10 ¹³ neutrons/cm ² operating
97	at a 2.5 MW power level. Correction factors for typical interference species produced
98	by thermal neutrons during irradiation are included in the footnote of Table DR2.
99	Samples and monitors were loaded into 1.5 mm diameter pits in a copper planchet and
100	placed under vacuum in an all-metal extraction line. Individual aliquots were step
101	wise heated and analyzed, or fused at high power using a Photon Machines Ltd.
102	Fusion 10.6 55W CO ₂ laser coupled to a Nu Instruments Noblesse multicollector
103	mass spectrometer operated at the Geological Survey of Canada, Ottawa, Ontario,
104	Canada. Laser energy was homogenized over a beam radius of 2 mm, gradually
105	increased over 60 s and subsequently held at full power for 30 s. The released gas was
106	then exposed to SAES TM NP-10 (c. 400°C) and HY-STOR® 201 (room temperature)
107	getters in the extraction line for six minutes. Following gettering, the sample gas was
108	expanded into the mass spectrometer. The Nu Noblesse (model 018) is a single-
109	focusing, Nier-source, 75 magnetic sector multicollector noble gas spectrometer
110	equipped with two quadrupole lens arrays. Ar ions were measured with a fixed array
111	of three ETP® discrete dynode ion-counting multipliers. Data collection followed the
112	measurement scheme MC-Y detailed in Kellett and Joyce (2014). Blanks were run
113	every 4 th analysis, in an identical manner to unknowns. Air shots were analyzed every
114	8 th analysis to monitor efficiency and mass fractionation. Full results including blank
115	measurements are included in Table DR2. Sensitivity of the Nu Noblesse at the time
116	of analyses was 7.1-7.5 Amps/mol. Data collection, reduction, error propagation, date
117	calculation and plotting were performed using the software MassSpec (version 7.93)
118	(Deino, 2001), and the 40 K/ 40 Ar decay constant of Min et al. (2000). Weighted mean
119	calculations and plotting were performed with Isoplot (version 4.15) (Ludwig, 2012).
120	The grains were not conducive to progressive step heating, and degassing occurred at

121 low power over few heating steps. The attempt to more evenly degas the grains results 122 in several heating steps with very little gas and large associated uncertainties. Thus, 123 only steps comprising >1% ³⁹Ar released are presented on date spectra and inverse 124 isochron diagrams, and used for date calculations. All uncertainties are quoted at 2σ .

125 INTERPRETATION OF ⁴⁰AR/³⁹AR RESULTS

Eight total fusion dates on single grains range from 17.6 ± 0.2 Ma to 20.1 ± 0.3 Ma

127 (Fig. DR3A). Differences in diffusion radius, resulting in differences in closure

temperatures, cannot account for the 2.5 m.y. date spread. Analyzed muscovite had

radii between 125 and 350 μ m, a difference that may produce a c. 25 °C variation in

130 closure temperature (Harrison et al., 2009), resulting in date variations of at most 1

m.y. with a relatively slow cooling rate of -25 °C/m.y. (e.g. Imayama et al., 2012). For

higher cooling rates (e.g. Sorcar et al., 2014; Wang et al., 2015), date variations due tograin size differences would be even smaller.

134 The date spectra of four different step heated grains provide insights on the possible 135 causes for older and younger dates. Two aliquots (3409-05 and 3477-07) yield older 136 dates for initial heating steps compared to subsequent steps, suggesting the presence of extraneous ⁴⁰Ar (Fig. DR3C and E; McDougall and Harrison, 1999). The gas 137 138 released during these initial steps has a different chemical composition, evidenced by 139 elevated Ca/K and Cl/K (Fig. DR3C and E). This suggest interaction with a fluid that 140 contained dissolved Ca, Cl and Ar, which may have been incorporated in the 141 muscovite as fluid inclusions or could be responsible for back-diffusion of argon into the crystalline structure (Kelley, 2002). The 309 ± 5^{40} Ar/³⁶Ar intercept in the inverse 142 143 isochron of aliquot 3477-07 is slightly above the atmospheric composition ratio of 144 298.56 (Lee et al. 2006; Mark et al. 2011) and also suggests the presence of a small excess ⁴⁰Ar component in this aliquot. The inverse isochron date of 18.0 ± 0.2 Ma for 145

146	this aliquot accounts for excess ⁴⁰ Ar and is equivalent, within uncertainty, with the
147	step heat plateau date (18.1 \pm 0.2) that does not account for the excess ⁴⁰ Ar
148	component. The statistically insignificant variation between these two dates suggests
149	that the effect of any excess ⁴⁰ Ar component is within the uncertainty of the step heat
150	dates. Nonetheless, the presence of excess ⁴⁰ Ar likely accounts for older dates in total
151	fusion experiments and some initial steps of the step heating experiments.
152	A third aliquot (3409-04) yields two young initial steps, indicative of ⁴⁰ Ar loss (Fig.
153	DR3B; McDougall and Harrison, 1999). This is further supported by the 261 ± 9
154	40 Ar/ 36 Ar intercept in the inverse isochron for this aliquot (Fig. DR3F), below the
155	atmospheric composition ratio of 298.56 (Lee et al. 2006; Mark et al. 2011). Partial
156	recrystallization of muscovite during deformation below its isotopic closure
157	temperature could cause ⁴⁰ Ar loss. In sample JD-46B, grain boundary migration is the
158	dominant quartz recrystallization mechanism, indicating temperature of deformation
159	above 500 °C. Moreover, a CPO fabric from an adjacent quartzite sample indicates a
160	temperature of deformation of $600 \pm 50^{\circ}$ C. Muscovite occurs in S-, C- and C'-planes,
161	suggesting pre- to syn-kinematic growth. Muscovite is straight and display uniform
162	extinction, although very few grains are bent around garnet porphyroblasts and
163	display undulose extinction. No low-temperature deformation textures such as quartz
164	bulging or subgrain rotation, brittle fractures or kinked mica flakes were observed.
165	These textures all suggest that deformation occurred at a temperature well above the
166	isotopic closure of muscovite (465-480 °C for a 350 μm radius, 50-100 °C/Ma
167	cooling rate at 500 MPa, Harrison et al. 2009), and that the rocks did not experience
168	post-cooling deformation that could have induced ⁴⁰ Ar loss.
169	A second possibility for post-cooling ⁴⁰ Ar loss is the interaction with a fluid that could
170	leach Ar out of the muscovite crystal. Ca/K and Cl/K for the two first young steps of

171 aliquot 3405-04 are not significantly higher or lower compared to that of subsequent 172 heating steps of the aliquot, implying that fluid did not alter the Ca and Cl content of 173 the grain (Fig. DR3B). Epidote, an unusual mineral for metapelite of typical 174 composition, was observed locally on the outcrop, but no other clear evidence for 175 fluid interaction, such as chloritization or veining, was observed at the outcrop and thin section scales. Even if the cause of the ⁴⁰Ar loss remains speculative, minor post-176 cooling ⁴⁰Ar loss can nonetheless account for the few young dates in the total fusion 177 178 experiments.

179 Besides older and younger dates explained by minor excess argon and argon loss,

180 respectively, the sample is dominated by an Ar isotopic composition equating to dates

between 19 and 18 Ma, as evidenced by the weighted mean date from the total fusion

data (18.7 \pm 0.7 Ma), plateau dates of 18.8 \pm 0.3 Ma and 18.1 \pm 0.2 Ma (aliquot 3409-

183 05 and 3477-07), and two large individual degassing steps at 18.5 ± 0.4 and 18.9 ± 0.1

184 Ma in aliquot 3477-02, which comprise 25 and 59%, respectively, of the ³⁹Ar released

in that grain (Fig. DR3A and C-E). Because aliquot 3409-05 yields a plateau

186 comprising the greatest percentage of released ³⁹Ar (95.5%; Fig. DR3C), and because

187 its inverse isochron date (18.8 \pm 0.3 Ma, ⁴⁰Ar/³⁶Ar intercept of 299 \pm 5; Fig. DR3G) is

188 indistinguishable from the plateau date, we interpret this aliquot as the most

189 representative cooling age for the sample.

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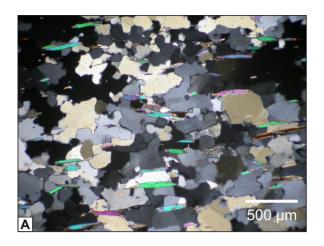
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321 SUPPLEMENTARY TABLES

- 322 Data Table DR1. Monazite U-Th/Pb geochronology
- **323** Data Table DR2. Muscovite 40 Ar/ 39 Ar thermochronology



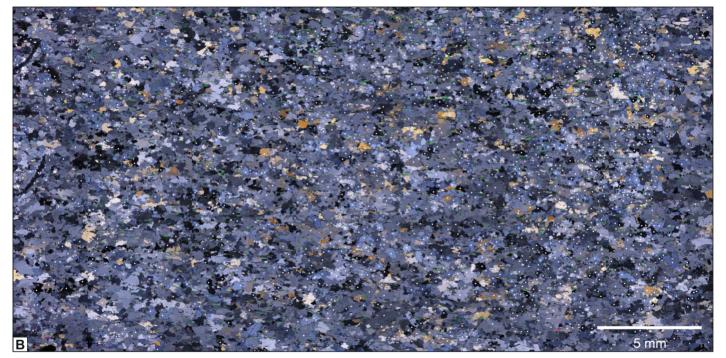
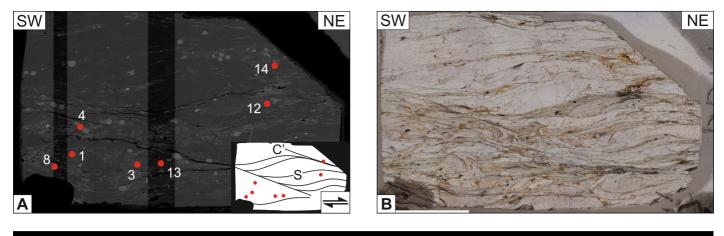
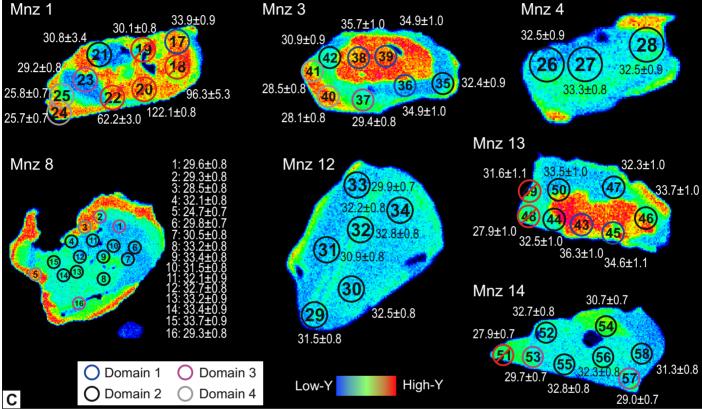


Fig. DR1. A. Cross-polarized light photomicrograph of grain boundary migration microstructure in quartzite JD-46A. B. Lambda-0 photomicrograph of quartzite JD-46A with the location of each quartz crystallographic <c>-axis orientations indicated by a white dot.





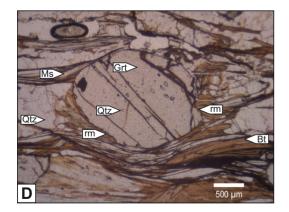


Fig. DR2. A: Back-scattered electron image of thin section JD-46B with location of analyzed monazite grains. Foliation (S) and top-to-the-NE shear bands (C') are outlined in the inset. B: Thin section photograph in plane polarized light of thin section JD-46B. C: Maps of relative Y concentration of all analyzed monazite grains with date spots in Ma. Numbers inside each circle refer to analysis number. Each spot is 9.7 µm large and color coded according to domain attribution. Note that trace element contents used for analysis classification were acquired with LASS-ICPMS. Hence, the relative Y concentration map may be misleading if a significant portion of the ablated material belonged to a domain underlying the surficial domain observed in the map. These analyses were used for interpretation only if a significant portion of the isotopic

signal was stable enough to calculate a reliable date and trace element composition. Red date spots were discarded for age interpretations because they overlap with matrix material, inclusions, or more than one chemical domain. Note that Group 4 analyses were obtained in the two shortening quadrants of only one monazite grain (#8) because the low aspect ratio (c. 1:1) of that specific grain would allow infinite rotation under general shear strain instead of resting in the flow plane (Ghosh and Ramberg, 1976). D: Plane-polarized light photomicrograph of garnet wrapped by the foliation and containing randomly oriented quartz inclusions, attesting for pre-kinematic growth. Resorbed margins are likely the consequence of decompression. Bt—biotite; Grt—garnet; Ms—muscovite; Qtz—quartz; rm—resorbed margins.

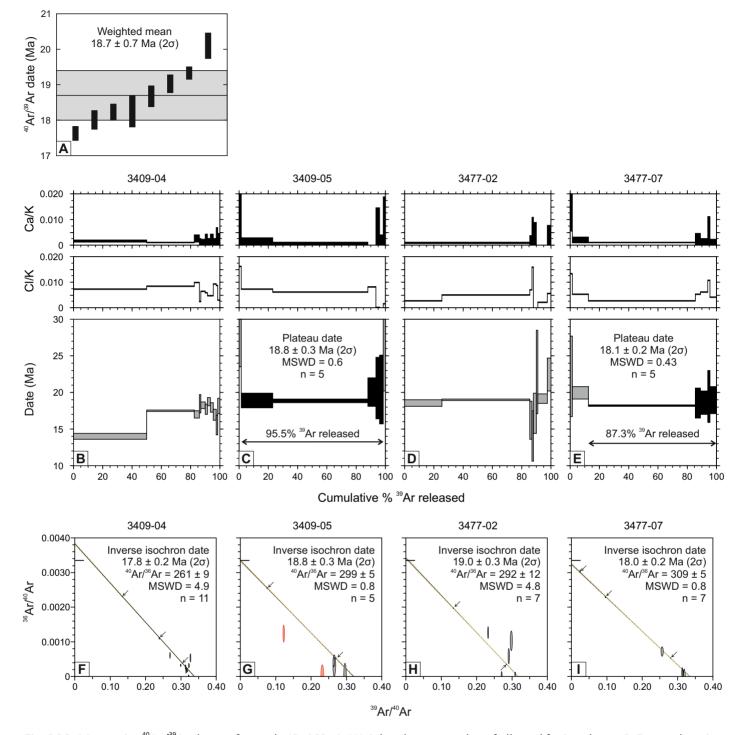


Fig. DR3. Muscovite ⁴⁰Ar/³⁹Ar dates of sample JD-46B. A: Weighted average plot of all total fusion dates. B-E: step-heating date spectra with Ca/K and Cl/K of each individual steps. F-I: Inverse isochron plots. Data ellipses in red on the inverse isochron plots were not included in the regressions and date calculations because they do not appear to comprise the same population and each yield less than 2% of the released ³⁹Ar. Arrows indicate small data ellipses. All uncertainties are 2σ except the error envelope on the inverse isochron regression lines (1 σ). MSWD—mean squared weighted deviation; n—number of analyses in date calculations.