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3	Neoproterozoic-Cambrian petroleum system evolution of the Micang
4	Shan Uplift, Northern Sichuan Basin, China: Insights from
5	pyrobitumen Re-Os geochronology and apatite fission track analysis
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29 Abstract

The Neoproterozoic strata of the Sichuan Basin is a key target for oil and gas. To evaluate the hydrocarbon evolution and its relationship with tectonic events in the Micang Shan Uplift, northernmost Sichuan Basin, we apply solid bitumen geochemistry (bitumen reflectance and fluorescence) and rhenium-osmium (Re-Os) geochronology.

The geochemistry of the solid bitumen indicates it is highly mature pyrobitumen that 35 formed contemporaneously with dry gas generation during oil thermal cracking. The 36 37 pyrobitumen is enriched in both Re (~106 - 191 ppb) and Os (~3030 - 5670 ppt). The Re-Os isotope data imply an Early Jurassic date for pyrobitumen formation, which 38 coincides well with age estimates from fluid inclusion data and basin modelling. The 39 Re-Os date for pyrobitumen formation coupled with previously presented AFT 40 analysis show that exhumation of the Neoproterozoic strata occurred during the 41 Cretaceous in the Micang Shan Uplift. This extensive uplift led to the erosion of any 42 potential gas reservoirs and surface exposure of bitumen-bearing Neoproterozoic 43 strata. In contrast, the more southern and central portions of the Sichuan Basin have 44 experienced less severe exhumation and as result Neoproterozoic sourced gas systems 45 46 are present. This study shows that through the combined application of Re-Os and AFT methodologies the timing of gas generation and subsequent erosion of any 47

potential gas reservoirs in the Micang Shan Uplift, northern Sichuan Basin can be
quantified. Moreover, the Re-Os and AFT data illustrate the potential to constrain the
timing of gas generation in petroleum systems worldwide.

51 Key words: Solid bitumen, Re-Os geochronology, Petroleum evolution, Gas
52 generation, Sichuan Basin

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54 **1. Introduction**

Hydrocarbon (oil and gas) production and shows sourced from or reservoired in 55 Proterozoic to Cambrian successions occur worldwide, and are an important 56 economic resource (Bhat et al., 2012; Craig et al., 2009; Ghori et al., 2009). For 57 example, the Lena-Tunguska Proterozoic-Early Cambrian (microfossils and K-Ar 58 59 dates on glauconites show the oldest date is 590 - 680 Ma) petroleum province, which includes the Lena Trough, the Kansk Basin, the Tunguska Basin, and the Sukhana 60 Basin, located on the Siberian Craton, northern Russia, has a total estimated resource 61 of 2 Bbbl (billion barrels) of oil and 83 Tcf (trillion cubic feet) of gas (Ghori et al., 62 2009; Meyerhoff, 1982). In addition, three potentially economically viable 63 Proterozoic oil and gas systems have been found in Australia (McArthur, Urapungan 64 and Centralian), which have a combined estimated resource of over 2 Bbbl of oil and 65 10 Tcf of gas (Bradshaw et al., 1994; Jackson et al., 1986; Munson, 2014). In the 66 poorly explored Taoudenni Basin of North Africa, two deep exploration wells 67 (Abolag-1 and Ouasa-1) have intersected a Neoproterozoic reservoir. A short duration 68 open hole test on Abolag-1 well provided an estimated gas resource of 48000 scf 69

(standard cubic feet) per day (Lottaroli et al., 2009). In China, the Sichuan Basin is 70 another potentially prolific Proterozoic-Cambrian petroleum sourced area with an 71 72 estimated resource of 26.2 Bbbl of oil and ~180 Tcf of gas (Fig. 1) (Ghori et al., 2009; Li et al., 2001; Zhang and Zhu, 2006; Zou et al., 2014a). Specific examples in the 73 74 Sichuan Basin include the Weiyuan and Anyue gas fields, which are both sourced and reservoired in Neoproterozoic to Cambrian sedimentary rocks, and contain ~1.4 and 75 ~15.4 Tcf of gas, respectively (Fig. 1) (Korsch et al., 1991; Li et al., 2015; Ma et al., 76 2010). 77

78 It is considered that much of the "easy exploration" around the world has been exhausted, and as a result industry is being forced to focus on more challenging 79 exploration targets, such as Neoproterozoic-Cambrian strata (Bhat et al., 2012; Craig 80 81 et al., 2009; Ghori et al., 2009). However, such systems typically possess significantly more complex geological evolution compared with conventional Phanerozoic 82 petroleum systems, and as a result the exploration risk is greatly increased (Ghori et 83 al., 2009; Katz and Everett, 2016). Further, as a direct outcome of multiple tectonic 84 events, potential reservoirs have experienced high temperatures and pressures as well 85 as uplift and subsidence. These factors, coupled with low quality seismic profiles, 86 resulted in six decades of exploration to find the Anyue giant gas field following the 87 discovery of the Weiyuan gas field in the Sichuan Basin (Wei et al., 2008; Zou et al., 88 2014a). Thus, in order improve exploration 89 to the success rate of Proterozoic-Cambrian petroleum systems, a better understanding of their evolution, 90 including timing of hydrocarbon generation, migration, accumulation and even trap 91

92 destruction, is needed.

Basin modelling, as well as, hydrocarbon fluid inclusion analysis are widely used 93 94 methods to constrain hydrocarbon evolution history (Angevine et al., 1990; Gonzaga et al., 2000; Parnell et al., 1996; Parnell et al., 2000; Schneider, 2003). However, 95 imperfect kinetic models and poorly constrained parameters, such as the 96 paleo-geothermal gradient, pressure and properties of the strata, hamper the accuracy 97 of proposed evolution histories (Braun and Burnham, 1992; Roberts et al., 2004; 98 Tissot et al., 1987). Since the 1980s, isotopic dating methods, for example, authigenic 99 K-Ar illite (Dong et al., 1995; Lee et al., 1985; Meunier et al., 2004; Tohver et al., 100 2008), Ar-Ar K-feldspar (Mark et al., 2010), and Ar-Ar quartz fluid inclusion (Qiu et 101 al., 2011), have all been shown to yield valuable information with regard to the timing 102 103 of hydrocarbon generation and migration. However, authigenic illite K-Ar and Ar-Ar could only yield the maximum timing of hydrocarbon emplacement, given the 104 challenge to isolate ⁴⁰Ar from samples (Dong et al., 1995; Mark et al., 2010). 105 Additionally, Ar-Ar dating of quartz fluid inclusions requires samples to be enriched 106 in gas-liquid inclusions. The latter, combined with the analytical challenge of isolating 107 the Ar from the gas inclusions has limited the available results (Liu et al., 2011; Qiu et 108 al., 2011). Compared to K-Ar and Ar-Ar dating on associated minerals, for example 109 illite, K-feldspar and quartz fluid inclusions, rhenium-osmium (Re-Os) dating directly 110 of the oil or solid bitumen, has shown promise to determine the absolute timing of 111 hydrocarbon (oil and gas) generation (Cumming et al., 2014; Finlay et al., 2011; Ge et 112 al., 2016; Georgiev et al., 2016; Lillis and Selby, 2013; Liu et al., 2017; Selby and 113

114 Creaser, 2005; Selby et al., 2005; Selby et al., 2007).

Solid bitumen can form at different evolutionary stages within a petroleum system 115 (Jacob, 1989; Lewan, 1985; Meyer and De Witt Jr, 1990; Stasiuk, 1997; Wu et al., 116 2000). For example, asphalt and gilsonite are associated with crude oil generation, 117 with impsonite and high maturity pyrobitumen considered to be related to crude oil 118 decomposition or gas formation (Bernard et al., 2012; Lewan, 1997; Stasiuk, 1997; 119 Wu et al., 2000; Xiao et al., 2007; Zhang and Li, 1999). To date, the available Re-Os 120 data are for solid bitumen from the Polaris Mississippi Valley-type Zn-Pb deposit, 121 122 Canada and the Northern Longmen Shan Thrust Belt, Southwest China, gilsonite from the Green River petroleum system in the Uinta Basin, western USA, and pyrobitumen 123 from the Majiang-Wanshan reservoir, South China (Cumming et al., 2014; Ge et al., 124 125 2016; Ge et al., 2017; Selby et al., 2005).

The complex tectonic and hydrocarbon evolution of the Sichuan basin has resulted in 126 highly debated timing and source models for many of hydrocarbon systems (e.g., the 127 128 Weiyuan, Ziyang, Anyue and Puguang gas fields (Ma et al., 2007; Wei et al., 2008; Zhu et al., 2006)). Present research based on both structural and sedimentary analysis 129 in the Sichuan basin suggests that the Late Silurian (ca. 420 Ma), Permian (ca. 250 130 Ma) and Early Jurassic (ca.180 Ma) could be key time intervals for both oil and gas 131 generation (Li et al., 2016; Sun, 2008; Wang and Wang, 2011; Wang, 2015). Located 132 in the north of the Sichuan Basin is the Micang Shan Uplift (Liu et al., 2015). 133 Compared with the central or northwest Sichuan Basin, the Micang Shan Uplift has 134 undergone additional tectonism since the Cretaceous (Chang et al., 2010; Sun et al., 135

2011a; Sun et al., 2011b; Yang et al., 2013). Thrust tectonics during the Yanshan (ca.
150 Ma) or Himalayan orogeny (ca. 60Ma) resulted in the uplift of both Cambrian
and Neoproterozoic strata to the surface (Dai et al., 2009).

Here we present solid bitumen Re-Os geochronology of the Micang Shan Uplift, 139 coupled with bitumen reflectance, fluorescence analysis and previously published 140 apatite fission track data (AFT age and thermal history modelling) to determine the 141 Proterozoic-Cambrian hydrocarbon evolution process of the Micang Shan Uplift in 142 the northern sector of the Sichuan Basin. Our data has potential implications for 143 144 understanding the Proterozoic-Cambrian hydrocarbon evolution of other regions in the Sichuan Basin, and illustrates that the combined application of solid bitumen 145 Re-Os geochronology and apatite fission track (AFT) analysis can provide valuable 146 147 chronological data for petroleum evolution. In addition to Micang Shan Uplift, high maturity solid bitumen (pyrobitumen) occurs worldwide, e.g., in the Alberta Basin, 148 Canada (Stasiuk, 1997), the Dahoney Basin, Nigeria (Meyer and De Witt Jr, 1990) 149 and the Basque-Cantabrian Basin, Spain (Agirrezabala et al., 2008), this work are also 150 potentially helpful for the hydrocarbon exploration worldwide. 151

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153 **2. Geological setting**

The Micang Shan Uplift occurs along the northern margin of the Upper Yangtze block
and comprises the northernmost sector of the Sichuan Basin, occupying an area of ~
4500 km² (1737 mi²) (Li et al., 2014). The Micang Shan Uplift is bordered by three
different orogenic belts (Longmen Shan, Hannan Uplift, and the Daba Shan Orogeny),

which lie to the west, northeast and east, respectively (Fig. 2) (Liu et al., 2015; Wang et al., 2014). From north to south, the Micang Shan Uplift is divided into three structural belts: the Basement Thrust Belt, the Submountain Fault-Fold Belt, and the South Foreslope (Huang, 2013). The entire Micang Shan Uplift records at least three orogenic events: 1) the Caledonian (ca. 480 Ma), 2) the Indosinian (ca. 200 Ma), and 3) the Yanshan (ca. 150 Ma) orogenies (Dong et al., 2012; Dong et al., 2011; Sun et al., 2011a; Sun et al., 2011b; Tian et al., 2012; Yang et al., 2013) (Fig. 3).

Structurally the Micang Shan Uplift represents a regional-scale anticline that 165 comprises basement Neoproterozoic metavolcanics and granite, with overlying 166 Neoproterozoic, Paleozoic (Cambrian to Silurian and Permian) and Mesozoic 167 (Triassic and Jurassic) marine carbonate and clastic rocks (Qi et al., 2004; Wang et al., 168 169 2008) (Fig. 3). Solid bitumen is widely distributed within the Micang Shan Uplift in the Neoproterozic Dengying Formation (Dai et al., 2009). The Dengying Formation 170 occurs over an area of 8500 km^2 (3281 mi^2), and has a maximum thickness of ~500 m 171 (1640 ft), with an average thickness of ~100 m (328 ft). The estimated total solid 172 bitumen reserves in the Dengying Formation of the Micang Shan Uplift and adjacent 173 area is about 12.5 billion tons, which is equivalent to ~150 Bbbls of crude oil (Dai et 174 al., 2009). The solid bitumen exists as pyrobitumen as a result of thermal cracking of 175 pre-existing hydrocarbons (this study; see section 4 and 5 for details). Given the total 176 abundance of pyrobitumen in the Micang Shan Uplift, the generated gas volume 177 during pyrobitumen formation is determined to have exceeded 400 Tcf (Liu et al., 178 2015). As a result of tectonic uplift since the Cretaceous, all cap rocks in the Micang 179

Shan Uplift have been exhumed (Chang et al., 2010; Dai et al., 2010). In contrast, cap 180 rocks are well developed in the more southern sectors of the Sichuan Basin. For 181 182 example, the Cambrian and Silurian, and Triassic and Jurassic units are local seals in the Tongjiang and Bazhong regions (Liu et al., 2015). The Early Cambrian 183 Qiongzhusi Formation is considered to be the principal source of the pyrobitumen in 184 the Sichuan Basin, according to current biomarker analysis of the solid bitumen 185 (though high maturity may affect the parameters) and potential source rocks (Liu et al., 186 2015; Zhang, 2013), element geochemistry evaluation of the potential sources (Cao et 187 al., 2014) and the similar carbon isotope values of the Cambrian shale ($\delta^{13}C \sim 27.2 \%$) 188 and pyrobitumen (δ^{13} C ~ 27.5 ‰). 189

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191 **3. Samples and methodology**

The solid bitumen samples analysed in this study are representative of occurrences 192 along the southern margin of the Micang Shan Uplift (Fig. 2). Solid bitumen occurs as 193 accumulations as small as ~ 1 cm (0.39 in) long and $\sim 0.2 - 0.5$ cm (0.08 - 0.20 in) 194 wide, and more commonly as $\sim 3 - 5$ cm (1.18 - 1.97 in) long and $\sim 2 - 3$ cm (0.79 -195 1.18 in) wide (Fig. 4) within pores and fractures that are found widely distributed in 196 the dolomite reservoir of the Dengying Formation. In hand specimens, the solid 197 bitumen possesses a smooth and vitreous surface. Under the microscope, the bitumen 198 is shown to occur intergrown with calcite and quartz, and to be predominantly hosted 199 in hairline fractures or vugs. Eleven samples were collected from seven different 200 locations. From east to west, these locations are the Jiulingzi(JLZ), Nanmushu(NMS), 201

Mayuan(MY), Zhujiaba(ZJB), Kongxigou(KXG), Huitan(HT) and Yangba(YB) areas. The distance between each sample location is ~ 5 to 8 km (3 – 5 mi) (Fig. 2) (see Table 1 for latitude and longitude data). All the samples were collected from surface exposures and from similar stratigraphic levels within the Denying Formation, which dips at ~ 40° towards the southeast.

The solid bitumen fluorescence analyses were conducted at the Wuxi Institute of 207 Petroleum Geology, SINOPEC. The solid bitumen-bearing limestones were first cut 208 and polished into standard thin sections (~ 0.03 - 0.05 mm). The solid bitumen was 209 210 examined using a Nikon ECLIPSE LV100N POL polarizing microscope under transmitted, reflected and fluorescent light at room temperature. The light source was 211 a 100 W mercury lamp, with a digital photomicrography system. Bitumen reflectance 212 213 (BRo, %) of incident light under oil immersion was used to assess the thermal maturity of samples. The reflectance microscope measures the amount of reflected 214 light relative to the incident light and expresses this ratio as a percentage. The BRo 215 value is obtained according to the formula, $BRo = (N - Ne)^2 + K^2 / (N + Ne)^2 + K^2$, 216 where Ne is the refraction index of the oil used, N is the sample refraction, and K is 217 the absorption coefficient (Zhang, 1988). The solid bitumen reflectance analyses were 218 conducted at the Wuxi Institute of Petroleum Geology, SINOPEC, based on the 219 method of (Zhang, 1988). The solid bitumen was first ground to ~ 0.15 mm, then 220 mixed with resin and finally compressed into a cylindrical form. One surface of the 221 cylinder was polished and immersed into oil with a refraction index (Ne) of 1.518. 222 The sample refraction (N) was measured using a monochromatic light with a 223

wavelength of 546 nm and a microscopy spectrophotometer (version: MPV-III,
806(5)). The BRo value is determined through an average of 30 measured points in
each sample.

The Re and Os isotopic analysis of the solid bitumen samples were analysed at the 227 Laboratory for Source Rock and Sulfide Geochronology and Geochemistry (a 228 member of the Durham Geochemistry Centre) at Durham University following 229 published analytical procedures (e.g. Selby et al., 2005; Selby et al., 2007). 230 Approximately 0.2 - 1.0 g bitumen was first separated from the limestone rocks 231 232 without metal contact and crushed to ~ 1 mm grains using an agate pestle and mortar. Approximately 100 - 200 mg of bitumen were dissolved and equilibrated with a 233 known amount of ¹⁸⁵Re and ¹⁹⁰Os spike solution by inverse *aqua-regia* (3 ml HCl and 234 235 6 ml HNO₃) in a Carius tube for 24 hours at 220°C. Osmium was isolated and further purified from the inverse aqua-regia by CHCl₃ solvent extraction at room temperature 236 and micro-distillation, respectively. The Re was isolated using HCl-HNO₃ based 237 anion chromatography. The purified Re and Os were loaded on Ni and Pt filaments, 238 respectively, and analyzed using Negative Ion Thermal Ionization Mass Spectrometry 239 (N-TIMS). Rhenium was measured using Faraday collectors and Os in peak hopping 240 mode using a secondary electron multiplier (SEM). Measured Re and Os ratios were 241 corrected for oxide contribution and mass fractionation using ${}^{185}\text{Re}/{}^{187}\text{Re} = 0.59738$ 242 (Gramlich et al., 1973) and ${}^{192}\text{Os}/{}^{188}\text{Os} = 3.08261$, spike and blank contributions. All 243 data were blank corrected based on the total procedural blanks values of Re (1.6 \pm 244 0.025 pg) and Os (0.05 \pm 0.004 pg), with an average ¹⁸⁷Os/¹⁸⁸Os ratio of ~ 0.22 \pm 0.06 245

(n = 4). All uncertainties include the propagated uncertainty in the standard, spike 246 calibrations, mass spectrometry measurements, and blanks. The analyses presented in 247 248 this study were conducted prior to using DROsS as the in-house control solution (Nowell et al., 2008) at Durham. The ¹⁸⁷Os/¹⁸⁸Os values of the Os standard solution 249 AB2 during these studies were 0.1611 \pm 0.0066, with the ¹⁸⁵Re/¹⁸⁷Re values of the Re 250 standard solution being 0.5984 ± 0.0002 . These values are in agreement with those 251 previously published for AB2 and Re-std (Cumming et al., 2014; Finlay et al., 2011, 252 2012; Lillis and Selby, 2013; Rooney et al., 2012). The ¹⁸⁵Re/¹⁸⁷Re ratios for samples 253 of this study were corrected for the measured difference of the ¹⁸⁵Re/¹⁸⁷Re value for 254 Restd and the 185 Re/ 187 Re value of 0.59738 \pm 0.00039 (Gramlich et al., 1973). The 255 Re-Os data of this study are regressed using the program Isoplot V. 4.15 (Ludwig, 256 2008) using the ¹⁸⁷Re decay constant of $1.666 \times 10^{-11} a^{-1}$ (Smoliar et al., 1996). The 257 input data contains ${}^{187}\text{Re}/{}^{188}\text{Os}$ and ${}^{187}\text{Os}/{}^{188}\text{Os}$ ratios with their total 2σ uncertainty 258 and associated error correlation, Rho. 259

To further discuss the hydrocarbon and tectonic evolution of the Micang Shan Uplift, 260 we have utilized the previously published Apatite Fission Track (AFT) data (n = 35) 261 obtained from outcrop or borehole samples distributed over a north – south transect 262 across the study area (Lei et al., 2012; Tian et al., 2012; Yang et al., 2013) (Fig. 2; 263 Table. 2). In addition, thermal history modeling results (n = 15) in the study area are 264 also utilized (Tian et al., 2012; Yang et al., 2013). These thermal history models are 265 determined from eight Proterozoic granite, diorite or sandstone samples (MC01, 266 MC02, MC25, NJ1T, NJ2T, NJ3T, NJ5T, NJ6T) located in the Micang Shan Uplift, 267

268	and seven Paleozoic - Mesozoic sandstone samples (MC03, MC05, MC11, NJ12T,
269	NJ15T, NJ17T, HB1-4) that are distributed in the southern area of the Micang Shan
270	Uplift, e.g., the Submountain Fault-fold Belt and the South Foreslope (Fig. 2; Table
271	2).

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273 **4. Results**

The bitumen reflectance (BRo, %), which is used in a similar way as vitrinite 274 reflectance (Ro, %), is an indicator of thermal maturity of bitumen, and has become a 275 276 tool used in basin analysis (Bertrand, 1993; Riediger, 1993). The BRo values become higher with increasing hydrocarbon maturity. According to the linear relationship 277 between BRo and Ro (Jacob, 1989; Landis and Castaño, 1995; Schoenherr et al., 278 279 2007), bitumen at various maturity levels has different BRo values. For example, immature solid bitumen possess BRo values of < 0.25, with mature and over-mature 280 solid bitumen being characterised by BRo values of ~ 1.1 and > 1.7 (Jacob, 1989; 281 Landis and Castaño, 1995; Schoenherr et al., 2007). The BRo values of 3.25 to 4.08 282 (Table 1) for the five solid bitumen samples from the Micang Shan Uplift characterise 283 the solid bitumen as thermally over-mature. 284

Ultraviolet fluorescence of the hydrocarbons (bitumen and oil) is also a useful tool for evaluating hydrocarbon maturity. With increasing maturity, the fluorescence color will gradually change from yellow-green, to brownish and finally to colorless (Chen, 2014; Jacob, 1989; Shi et al., 2015). The microscopic features of the solid bitumen samples (NMS and JLZ) from the Micang Shan Uplift are shown in Figure 5. The reservoir rocks are mainly composed of dolomite, containing pelletoid or granular-mosaic solid bitumen within fissures or between grains, with the solid bitumen possessing no fluorescence, thus characterizing the bitumen as highly mature (Fig. 5). Collectively, the organic petrography implies that the solid bitumen from the Micang Shan Uplift are pyrobitumen which formed through thermal cracking under high temperature environment, $\geq 140^{\circ}$ C (Dieckmann et al., 1998; Pepper and Corvi, 1995; Tsuzuki et al., 1999; Waples, 2000).

The Re and Os abundances of the solid bitumen samples range from ~ 106 to 191 ppb, 297 and ~ 3030 to 5670 ppt, respectively (Table 1). These Re and Os abundances are 298 significantly higher than those of average upper crustal values (Re = 0.198 ppb and 299 Os = 31 ppt) (Esser and Turekian, 1993; Rudnick and Gao, 2003), but similar to 300 301 values previously reported for bitumen and organic-rich sedimentary rocks (Cohen et al., 1999; Ge et al., 2016; Georgiev et al., 2016; Lillis and Selby, 2013; Ravizza and 302 Turekian, 1992; Rooney et al., 2010; Xu et al., 2009a; Xu et al., 2014). The 303 187 Re/ 188 Os and 187 Os/ 188 Os values of the solid bitumen range from ~ 166 to 340, and ~ 304 2.82 to 3.74, respectively (Table 1). All the Re-Os data yield a Model 3 (assumes that 305 the scatter about the best fit line is a combination of the assigned uncertainties and an 306 unknown, but normally distributed variation in the ¹⁸⁷Os/¹⁸⁸Os values (Ludwig, 2008)) 307 date of 239 ± 150 Ma (n = 11, Mean Squared Weighted Deviation (MSWD) = 398) 308 (Fig. 6A), with an initial¹⁸⁷Os/¹⁸⁸Os composition of 2.29 ± 0.64 (Table 1). 309 All the 35 AFT dates range from 123.5 ± 6.0 to 8.8 ± 1.3 Ma. The AFT dates are all 310

311 younger than the intrusion or depositional ages of the sampled rocks, which indicate

that the date represents the timing of thermal resetting. The AFT dates overall show a 312 trend to younger dates from north to south across the Micang Shan Uplift into the 313 314 Sichuan Basin (Fig. 2). The eleven samples in the Micang Shan Uplift possess older dates (~ 64 - 124 Ma) with a mean age of ca. 102 Ma. Eighteen outcrop samples from 315 316 the inner Sichuan Basin possess AFT dates from ca. 60 to 83 Ma, with an average of ca. 70 Ma. The AFT dates from borehole HB1 from north Sichuan Basin (Fig. 2), 317 with the exception of the most-shallow sample, HB1-4, which has a date of ca. 73 Ma, 318 possess considerably younger dates (ca. 9 - 26 Ma). The mean track length of the 319 320 thirty-five samples varies between 10.0 ± 0.45 and 13.23 ± 0.10 µm. Similar to the AFT date distribution, a decrease in track length is observed for samples from north to 321 south (Table 2). The decreasing trend, in both AFT age and track length, from north to 322 323 south implies variable cooling histories across the Sichuan Basin.

The thermal history (time-temperature path) could provide a more detailed 324 understanding of the tectonic evolution of the Micang Shan Uplift and inner Sichuan 325 Basin. Fifteen samples, six of our own remodelled samples (MC01, MC02, MC03, 326 MC05, MC11, MC25) and nine previous published results (Tian et al., 2012) are 327 shown here (Fig. 7). The thermal history are remodelled using the HeFTy software 328 (version 1.8.4) based on the fanning curvilinear annealing model (Ketcham, 2005), 329 c-axis projection (Donelick et al., 1999), and initial mean track length ($L_0 = 16.0 \pm 0.8$ 330 μm) (Shen et al., 2012). For each sample, 100,000 to several million paths were 331 calculated until at least 500 'good' [T-t] paths were obtained (see (Yang et al., 2013) 332 for the detailed modeling methodology). The thermal history modelling results of the 333

fifteen samples can be divided into two groups. Group 1 comprise samples (n = 8)from the Micang Shan Uplift. Although there are some minor differences, cooling between ca. 140 and 100 Ma, with a temperature decrease from ~120 to 60 °C (apatite partial annealing zone, APAZ) is revealed (Fig. 7). Group 2 comprise samples (n = 7)south of the study area and represent a cooling event between ca. 100 and 60 Ma, which implies that cooling to between ~120 and 60 °C in the more southern Sichuan Basin occurred ~40 myrs after that in the more northern Micang Shan Uplift (Fig. 7).

341

342 **5. Discussion**

5.1. Bitumen characteristics in the Micang Shan Uplift

Hydrocarbon evolution is a complex process that begins with diagenesis and the 344 345 formation of the kerogen (Lewan, 1985; Tissot, 1984; Tissot and Welte, 1984). Solid bitumen, as a by-product of organic matter, exists through the entire hydrocarbon 346 evolution process and records events such as hydrocarbon maturity, migration and 347 accumulation pathways and the origin of the hydrocarbons (Wu et al., 2000). 348 Processes, such as biodegradation, oxidation, phase-migration and thermal cracking, 349 can lead to the formation of different types of solid bitumen (asphalt, gilsonite, 350 grahamite, impsonite, anthroxolite, etc) (Jacob, 1989; Meyer and De Witt Jr, 1990; 351 Rogers et al., 1974; Wu et al., 2000). The processes of oxidation, biodegradation, 352 polymerization and devolatilization could result in the removal of the light 353 hydrocarbons and form asphalt and gilsonite (Meyer and De Witt Jr, 1990). 354 Phase-migration during hydrocarbon migration can lead to fluid differentiation 355

through precipitation and de-asphalting (Meyer and De Witt Jr, 1990), resulting in the formation of grahamite, which typically occurs as veins (Stevenson et al., 1990; Zhang and Li, 1999). Thermal cracking, controlled by time and high temperature (Dahl et al., 1999; Vandenbroucke et al., 1999), of all hydrocarbons (e.g., oil, asphalt, gilsonite and grahamite) can result in the generation of dry gas and high maturity pyrobitumen (impsonite, antraxolite) (Meyer and De Witt Jr, 1990; Rogers et al., 1974).

High maturity pyrobitumen formed by thermal cracking is insoluble in most organic 363 364 solvents and possesses different physical and chemical features compared with solid bitumen (Jacob, 1989; Mancuso et al., 1989; Rogers et al., 1974; Wu et al., 2000; 365 Zhang and Li, 1999). For example, the solid bitumen types asphalt and gilsonite are 366 367 characterized by low bitumen reflectance (BRo < 1.0%), high H/C atomic ratio (> 0.8), low Tmax (~ 450 °C), and a yellow-green fluorescence colour. In contrast, the 368 high maturity pyrobitumen types impsonite and anthroxolite possess high bitumen 369 reflectance (BRo > 2.0 %), low H/C atomic ratio (< 0.6), high Tmax (~ 500 °C), dark 370 or no fluorescence, and contain high adamantine (3 - + 4 - methyl diamantane)371 concentrations (Fang et al., 2014; Hwang et al., 1998; Jacob, 1989; Shi et al., 2015; 372 Wang et al., 2013; Yang et al., 2014). 373

All solid bitumen samples collected from the Micang Shan Uplift in this study have similar features (high bitumen reflectance (BRo = 3.25 - 4.08), no fluorescence and are insoluble in chloroform). Previous research in the Micang Shan Uplift area found that the bitumen was also characterized by high Tmax values (~ 540 °C) (Huang,

2010). Collectively, the BRo values, fluorescence, Tmax, and lack of solubility in 378 organic solvents characterise the solid bitumen in the Micang Shan Uplift as highly 379 380 mature pyrobitumen. Although no gas reservoirs are known in the Micang Shan Uplift, the Neoproterozoic Weivuan gas field in the Sichuan basin (Fig. 1), ~ 200 km (124 mi) 381 south of the Micang Shan Uplift, possesses methane (CH₄) which comprises 85 % -382 97 % of the total gas composition, with lesser amounts of nitrogen (N₂) (< 8%), 383 carbon dioxide (CO₂) (< 5%) and ethane (C₂H₆)(< 1%) occupying the remaining 384 components (Wei et al., 2008). 385 The molecular and isotope data (C₁-C₃ composition and $\delta^{13}C_1$ - $\delta^{13}C_3$ value) are 386 useful tools for identifying the gas formation mechanism (Behar et al., 1992; 387 Prinzhofer and Huc, 1995). During thermal cracking, the $ln(C_2/C_3)$ value will increase, 388 but the $\ln(C_1/C_2)$ and the $\delta^{13}C_2 - \delta^{13}C_3$ values will remain the same for the generated 389 gas (Prinzhofer and Huc, 1995). The gas from both the Ziyang and Weiyuan gas fields 390 in the Western Sichuan Basin display $\ln(C_1/C_2)$ values within a narrow range (~5.5 -391 7.0), but the $\ln(C_2/C_3)$ ratio spans a much wider range (~1.0 - 6.5) (Liu et al., 2009). 392 These data indicate that the gas was formed by thermal cracking of a previously 393 formed reservoired oil. This also suggests that oils reservoired in the rest of the 394 Sichuan Basin could have also experienced thermal cracking to form highly mature 395 pyrobitumen and the accumulations of dry gas (CH₄) where trapped. 396

397

5.2. Timing of oil thermal cracking in the Micang Shan Uplift

399 Source rock hydrous pyrolysis experiments show that, initially, bitumen quantities

increase as the amount of kerogen decreases, but as a result of increasing temperature, 400 bitumen abundance decreases as it is cracked into liquid oil (Behar et al., 1991; 401 Lewan, 1985). At higher temperatures (~ 360 °C), hydrous pyrolysis experiments 402 show that the abundance of both bitumen and oil decrease in response to gas 403 generation, as well as mass increasing in the source rock, which indicates the 404 generation of high maturity pyrobitumen. The hydrous pyrolysis experiments indicate 405 that the high maturity pyrobitumen and gas form contemporaneously during the last 406 stages of hydrocarbon evolution. Further, recent studies on the thermal cracking of oil 407 408 (Hill et al., 2003) and hydrocarbon composition numerical modelling (evolution of C_{14+} to C_1 compounds with time) in the Fahud Salt Basin, North Oman (Huc et al., 409 2000), found although pyrobitumen could form at the beginning of oil formation, the 410 411 majority of the pyrobitumen formed together with the dry gas during the oil cracking event under high temperatures condition. 412

The Sichuan Basin contains more than 50 gas fields, which include the Ziyang, 413 414 Weiyuan, Anyue and Puguang giant gas fields (Fig. 1) (Luo et al., 2013; Wei et al., 2008; Zou et al., 2014a). To date, there is still no agreement on the timing of the gas 415 generation. Basin modelling of the Nanjiang area in the northern Sichuan Basin 416 suggests that oil generation may have begun during the Late Cambrian to Ordovician 417 (Wang and Wang, 2011). Following the exposure of the strata between the Devonian 418 and Carboniferous, the entire Late Cambrian to Ordovician units of the Sichuan basin 419 underwent rapid burial to ~7000 m (22,965 ft) between the Late Permian and Late 420 Cretaceous (Fig. 8) (Liu et al., 2010; Ma et al., 2008; Wang and Wang, 2011). 421

422	Fluid inclusions can represent micron scale samples of the fluids (oil, gas and water)
423	that flowed through and interacted with the host rocks during the evolution of a
424	hydrocarbon system (Cooley et al., 2011; Haszeldine et al., 1984). Physical and
425	chemical (pressure, temperature, fluid composition) conditions recorded by fluid
426	inclusions within petroliferous basins have been widely applied to constrain both oil
427	and gas migration and accumulation (Aplin et al., 1999; Bodnar, 1990; Bourdet et al.,
428	2010; Cao et al., 2006; Oxtoby et al., 1995; Ping et al., 2017; Pironon, 2004;
429	Teinturier et al., 2002). Fluid inclusion analysis in the Weiyuan Gas field, western
430	Sichuan Basin, show that the homogenization temperatures of the aqueous fluid
431	inclusions that are coeval with the gas-bearing fluid inclusions range from 108 -
432	212°C, with an average of 158°C (Tang et al., 2004). Furthermore, the fluid inclusion
433	data from the Neoproterozic Dengying Formation reservoir in the Micang Shan Uplift
434	possess homogenization temperatures that fall between 105 and 245 °C, with 87% of
435	the values being higher than 140 °C and average temperature at 180 °C (Fig. 8) (Wang,
436	2015). As to the few low temperature (< 120 °C) data, it may relate to early
437	hydrocarbon migration during the Silurian (Zou et al., 2014b) or the post-entrapment
438	modification (Okubo, 2005). Applying the generally high homogenization
439	temperatures to basin burial models within the northern Sichuan Basin (Liu et al.,
440	2015; Sun, 2008; Yuan et al., 2012), a gas migration event is suggested to have
441	occurred between the Triassic and Jurassic.

442 All the Re-Os data of the pyrobitumen from the Micang Shan Uplift area yield a date

443 of 239 ± 150 Ma (n = 11, initial ¹⁸⁷Os/¹⁸⁸Os (Osi) = 2.29 ± 0.64 , MSWD = 398). The

444 MWSD value obtained from the best-fit of all the Re-Os data suggests that the sample 445 set have not fully met the criteria to obtain a precise isochron. For example, the entire 446 sample set does not represent contemporaneous formation, or possess identical initial 447 isotope compositions (Osi), or that the Re-Os systematics have been disturbed (Cohen

448 et al., 1999; Kendall et al., 2009; Selby et al., 2007).

The percent deviation from the best-fit line of all the Re-Os data is illustrated in 449 Figure 6B, which shows that samples HTB01, HTB02, JLZ4, YB01, KXG5 all show 450 a large deviation (with the exception of sample KXG5 ($\sim 2.2\%$), > 4.4 %) from the 451 452 best-fit line. Calculating the Osi value for each sample at 239 Ma (Osi₂₃₉), which is based on the Re-Os date of all eleven samples, shows that six samples (My601, 453 My603, NMS955, NMS1030, NMS1068, ZJB01) possess very similar Osi₂₃₉ values 454 (2.25 - 2.30), with the remaining five samples (KXG05, HTB01, HTB02, YB01, 455 JLZ04) possessing either slightly less or more radiogenic Osi values (1.96 - 2.15, n =456 2; 2.36 - 2.52, n = 3, respectively) (Table 1). The differences in the Osi values may 457 458 relate to samples bearing different initial Os isotope compositions or samples that represent different generation ages, or have experienced disturbance to the Re-Os 459 isotope system. We discuss these possibilities below. 460

461 As stated above, the Re-Os data for the six samples with similar Osi values (My601,

462 My603, NMS955, NMS1030, NMS1068, ZJB01, which are the samples that show the 463 least deviation of the best-fit line of all the data, Fig. 6B) yield a Model 1 (which 464 considers that only the assigned uncertainties produce the scatter about the best-fit 465 line (Ludwig, 2008)) Re-Os date of 184 ± 23 Ma (Osi = 2.50 ± 0.09 , MSWD = 1.0) 466 (Fig. 6C).

The remaining five samples plot in ¹⁸⁷Re/¹⁸⁸Os-¹⁸⁷Os/¹⁸⁸Os space either above or 467 468 below the ca. 184 Ma isochron (Fig. 6C). Of these samples, sample KXG05 possesses an Osi₂₃₉ value of 2.36, which falls between the Osi₂₃₉ values for HTB01 and HTB02 469 (2.49 - 2.52), and JLZ04 and YB01 (1.96 - 2.15), and will therefore not fall along any 470 best-fit line (Fig. 6C). Although the isochron dates determined from two samples are 471 not considered a robust reflection of the true geologic age (Ludwig, 2008), we note 472 that the Re-Os data for samples HTB01 and HTB02, and JLZ04 and YB01, yield 473 474 Re-Os dates that are within uncertainty of the dates determined from the 6 samples $(205 \pm 32 \text{ Ma}, \text{Osi} = 2.68 \pm 0.16 \text{ and } 173 \pm 12 \text{ Ma}, \text{Osi} = 2.34 \pm 0.06$, respectively 475 (Fig. 6C)). 476

477 In the Micang Shan Uplift, the pyrobitumen may suffer from biodegradation, water washing during the migration and uplift process since formation. Previous work 478 (Lillis and Selby, 2013; Selby and Creaser, 2005) found that the these effects do not 479 appreciably disturb the Re-Os system. However, studies have found that the Re-Os 480 system of the highly mature hydrocarbons, for example oil from Bighorn Basin, USA 481 and bitumen from North Hebei Depression, China, may exhibit evidence of 482 disturbance (Li et al., 2017; Lillis and Selby, 2013). Moreover, Re-Os isotope dating 483 of pyrobitumen, which formed contemporaneously with dry gas, together with clastic 484 AFT dating in the Majiang-Wanshan reservoir, South China show that the Re-Os date 485 486 coincides with the timing related to gas generation and that the hydrocarbon thermal cracking event at temperatures of $>140^{\circ}$ C could reset the previous oil or asphaltene 487

488 Re-Os system (Ge et al., 2016).

In this study, Latest Triassic to the Middle Jurassic dates (ca. 205 – 173 Ma) derived 489 490 from the Re-Os pyrobitumen data, especially the ~184 Ma date determined from six samples, from the Micang Shan Uplift agree well with previous basin modelling 491 (Yuan et al., 2012) and the general temporal understanding of oil and gas 492 accumulation events in the northern Sichuan Basin (Liu et al., 2015). This age 493 agreement indicates that the Late Triassic to Jurassic is the key period for thermally 494 cracking of oil in Neoproterozoic reservoirs that resulted in gas generation and high 495 496 maturity pyrobitumen (Fig. 8). Thus, although we cannot rule out disturbance to the Re-Os systematics of the pyrobitumen, the variation in the Osi values shown by the 497 sample set most likely represents variations in the Os isotope composition of the 498 499 petroleum that thermally cracked to produce pyrobitumen during the Late Triassic to the Middle Jurassic, and or the protracted interval over which pyrobitumen and gas 500 generation occurred in the Michan Shan Uplift. 501

502

503 **5.3. Petroleum system evolution of northern Sichuan Basin**

Since the Late Triassic collision between the north China block and Yangtze block, which lead to the formation of the Qinling Orogeny (Yin and Nie, 1993), the Micang Shan Uplift was affected by a continuous compressional tectonic regime, with the most severe event occurring during the Cretaceous when the Yangtze block collided with Qinling Orogeny (south margin of the north China block) (Sun et al., 2011a; Sun et al., 2011b). During this tectonism the E-W trending fold and thrust structures between the Micang Shan Uplift and the inner Sichuan Basin were developed (Dong
and Santosh, 2016; Xu et al., 2009b).

512 The Group 1 AFT samples from the Micang Shan Uplift have older AFT ages (ca. 64 - 124 Ma) with the thermal history modeling showing that the samples entered the 513 apatite partial annealing zone (APAZ) between ca. 140 and 100 Ma, and cooled 514 through the APAZ before ca. 90 Ma. In contrast, Group 2 samples, which are 515 generally located at the transition zone between the Micang Shan Uplift and the inner 516 Sichuan Basin, possess slightly younger ages (ca. 60 - 83 Ma) and thermal history 517 518 modeling shows that the samples in this area pass in to the APAZ from ca. 100 to 60 Ma. The ~ 40 myr difference between both the AFT ages and the modeled cooling 519 and uplift models coincide well with the N-S propagation of the tectonic front (Hu et 520 521 al., 2012; Xu et al., 2009b). The AFT dates (ca. 60 to 124 Ma) and thermal history models indicate a prolonged and continuous cooling and uplift during the Cretaceous 522 in the study area. Given the present-day thermal gradient (18 - 21°C/km (Hu et al., 523 2000; Lu et al., 2005)) and the near surface temperature (~ 20°C), more than 5000 m 524 (16,404 ft) of strata is estimated to have been eroded as a result of the tectonic uplift 525 during the Cretaceous. However, the much younger AFT dates (ca. 9 - 26 Ma) from 526 the borehole HB1 (2594 - 4485 m (8510 - 14,714 ft) deep) in the Sichuan Basin 527 (Table 2) indicate that the Cretaceous tectonic event did not affect the sediments 528 within the inner Sichuan Basin, with the Jurassic and Triassic units currently at a 529 depth of more than 2500 m (8202 ft). Moreover, many gas fields that are reservoired 530 in the Neoproterozoic Dengying (Wei et al., 2008), Permian Changxing, and Triassic 531

Feixianguan formations (Ma et al., 2008) have been discovered within the central or 532 southern Sichuan Basin (Fig. 1), which further prove that the tectonic event did not 533 severely affect the inner Sichuan Basin. The presence of large quantities of 534 pyrobitumen in the exposed and near-surface Neoproterozoic Dengying Formation 535 and the absence of the Permian Changxing and Triassic Feixianguan formations in the 536 northern Sichuan Basin (e.g., around the Micang Shan Uplift) suggest that ~ 5000 m 537 (16,404 ft) of exhumation has occurred. Further evidence for uplift and erosion of the 538 Proterozoic, Paleozoic and Mesozoic units of the Micang Shan Uplift is supported by 539 540 a north-south paleocurrent direction (He et al., 1997; Meng et al., 2005), sandstone composition analysis (Dickinson et al., 1983; Liu et al., 2006), and detrital zircon 541 U-Pb dates of the early Paleogene strata in the western Sichuan Basin (ca. 1800 – 130 542 543 Ma) (Jiang et al., 2013).

Integrating the pyrobitumen Re-Os data, AFT results (age and thermal history 544 modeling) as well as previous basin models from the Micang Shan Uplift, permits the 545 546 hydrocarbon evolution in the Micang Shan Uplift area to be quantitatively described as: (1) prior to the Caledonian Orogeny, Cambrian shales were buried to ~ 2500 m 547 (8202 ft) depth throughout the whole Sichuan Basin and began to generate oil. 548 However, subsequent uplift and exhumation during the Devonian to Carboniferous 549 (Caledonian Orogeny) halted oil generation (Fig. 8a); (2) the Late Paleozoic-Early 550 Mesozoic witnessed rapid sedimentation of the Permian to Triassic strata which 551 resulted in the burial of the Neoproterozoic strata to ~ 7000 m (22,965 ft). Thermal 552 cracking of the hydrocarbons under a high temperature environment (≥ 140 °C) as a 553

result of burial the generation of pyrobitumen and dry gas occurred during the Late 554 Triassic to Middle Jurassic (Fig. 8b); and (3) since the Cretaceous, related to the N-S 555 556 compression between the north China block and Yangtze block, the Micang Shan Uplift experienced rapid uplift and exhumation, bringing the pyrobitumen-bearing 557 Neoproterozoic strata to the surface and also resulted in the loss of the any 558 gas-bearing reservoirs. In contrast, in the central and southern sectors of the Sichuan 559 Basin, Paleozoic and Mesozoic gas reservoirs have been preserved due to limited 560 uplift during the Late Cretaceous (Fig. 8c). 561

562

563 **6. Conclusions and Implications**

High maturity pyrobitumen Re-Os analysis, as well as previous basin modelling of the 564 565 North Sichuan Basin, indicate that hydrocarbon thermal cracking (dry gas generation from oil cracking) in the northern Sichuan Basin occurred during the Early Jurassic 566 (ca. 184 Ma), as a result of a high temperature environment due to rapid 567 sedimentation and burial during the Late Permian to Jurassic in the Sichuan Basin. 568 The AFT data (age and thermal history) indicate that the prolonged Cretaceous 569 Yanshan Orogeny resulted in significant uplift and exhumation (~ 5000 m (16404 ft)) 570 and caused the loss of Paleozoic and Mesozoic gas reservoirs in the Micang Shan 571 Uplift, with only pyrobitumen remaining in the Neoproterozoic strata. 572

573 Previous work has shown that combined AFT and solid bitumen Re-Os 574 geochronology on the Majiang-Wanshan reservoir, South China, has the potential to 575 constrain the timing of oil cracking (pyrobitumen generation), and by inference, the

timing of gas generation (Ge et al., 2016). Although the AFT and Re-Os dates were 576 found to be similar in that work, the slightly older Re-Os date (ca. 80 Ma) compared 577 578 to the AFT date (ca. 70 Ma) suggest that the temperature condition of resetting of the Re-Os systematics in hydrocarbons, specifically high maturity pyrobitumen, may be 579 higher than that of the AFT closure temperature (110 \pm 10 °C) (Ge et al., 2016). 580 Moreover, the Re-Os age derived from the pyrobitumen of the Micang Shan Uplift in 581 this study is significantly older than the AFT dates, which further supports that the 582 closure temperature of the Re-Os systematics in pyrobitumen is higher than that of the 583 584 AFT closure temperature.

Deep burial and complex tectonic events have greatly affected the hydrocarbon systems in the Sichuan Basin, and even the whole of the south China block, resulting in gas being the main hydrocarbon resource (Zhao et al., 2004). The Re-Os data from this study imply that the gas in the Sichuan Basin formed during the Latest Triassic to the Middle Jurassic by thermal cracking. As such, the areas that have been relatively tectonically stable since the Mesozoic, e.g., in the ancient uplift or slope in the central and eastern Sichuan Basin (Zou et al., 2014a), may be prospective gas targets.

In addition to South China, pyrobitumen occurs worldwide, e.g., in the Alberta Basin, Canada (Stasiuk, 1997), the Dahoney Basin, Nigeria (Meyer and De Witt Jr, 1990) and the Basque-Cantabrian Basin, Spain (Agirrezabala et al., 2008). Thus, Re-Os pyrobitumen chronology (coupled with AFT dating) could be employed to yield quantitative timing of gas generation in other basins worldwide, and may enhance our understanding of the evolution of hydrocarbon systems and help guide gas 598 exploration.

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- 1205

1206 Figure Captions

- 1207 Fig. 1. Simplified map of the Sichuan Basin showing the distribution of oil and gas
- fields (Substantially modified from Li et al., 2015; Li et al., 2001; Ma et al., 2010).
- 1209 Fig. 2. Simplified geological map of the Micang Shan Uplift, northern Sichuan Basin.
- 1210 (A) Regional map of the Micang Shan Uplift and its adjacent area, Longmen Shan
- 1211 Orogeny, Hannan Uplift and Daba Orogen in the west, northeast, east, respectively. (B)

Simplified geologic and structural map of Micang Shan Uplift, northern Sichuan
Basin (Substantially modified from Wang et al., 2014), showing the location of both
bitumen and the AFT samples.

Fig. 3. Combined stratigraphic sequences, petroleum system and tectonic events in the
northern Sichuan Basin (Substantially modified from Huang, 2013; Huang, 2010;
Wang, 2015).

1218 Fig. 4. Examples of pyrobitumen outcropping in the Jiulingzi (JLZ), Nanmushu1219 (NMS), Kongxigou (KXG) and Yangba (YB) areas.

1221 1070 area; (C) and (D) from the Nanmushu 1030 area; and (E) and (F) from the 1222 Jiulingzi area.

Fig. 5. Petrography of the pyrobitumen of this study: (A) and (B) from the Nanmushu

1223 Fig.6. A. Re-Os isochron plot showing all the data in the Micang Shan Uplift; B. Plot

1224 of percent deviation from the 239 Ma best-fit line; C. Re-Os isotope data of bitumen

1225 from Mayuan, Nanmushu and Zhujiaba area with Osi₂₃₉ from 2.25 - 2.30. Data-point

1226 ellipses shown with 2-sigma (2 σ) absolute uncertainty (sigma (σ) is the standard

deviation of the age). MSWD is short for Mean Square Weight Deviation. Data labels

are sample numbers listed in Table 1.

1220

1229 Fig. 7. Collected apatite fission track (AFT) thermal history modeling results from the

1230 Micang Shan Uplift and inner Sichuan Basin (Substantially modified from Tian et al.,

1231 2012; Yang et al., 2013). D: determinted apatite fission track age; M: modeled apatite

1232 fission track age; GOF: goodness of fit; N: number of measured fission track; Lm:

1233 mean length of the measured fission track length; SD: standard deviation.

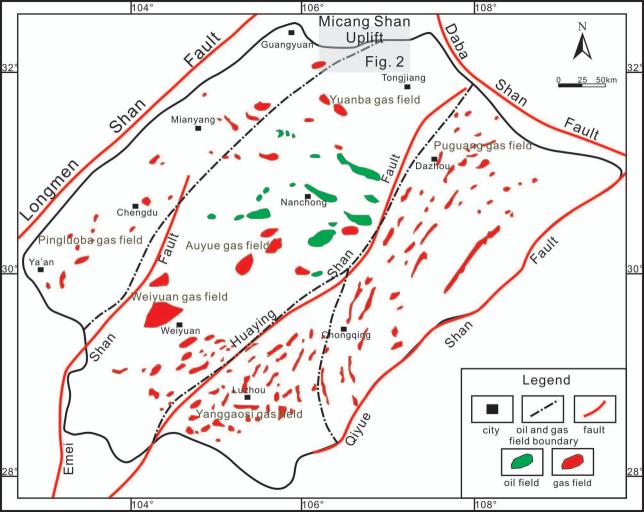
Fig. 8. A. Basin modelling and fluid inclusion results for the Micang Shan Uplift, showing the key time for gas generation. B. Cartoon model showing the hydrocarbon evolution of the Micang Shan Uplift, north Sichuan Basin: (a) original hydrocarbon reservoir formation process prior to the Silurian; (b) pyrobitumen and dry gas formation by thermal cracking during the Jurassic; and (c) reservoir alteration and erosion during the Cretaceous Yanshan orogeny.

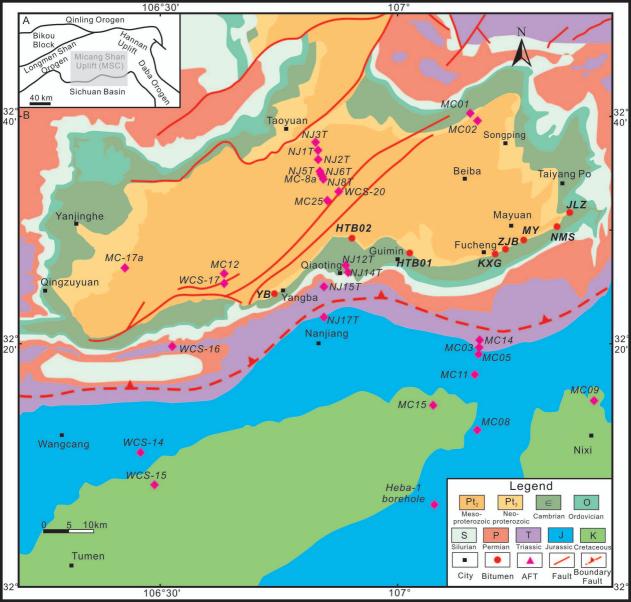
1240

1241 Table 1. Re-Os isotopic and BRo data of the solid bitumen from the Micang Shan

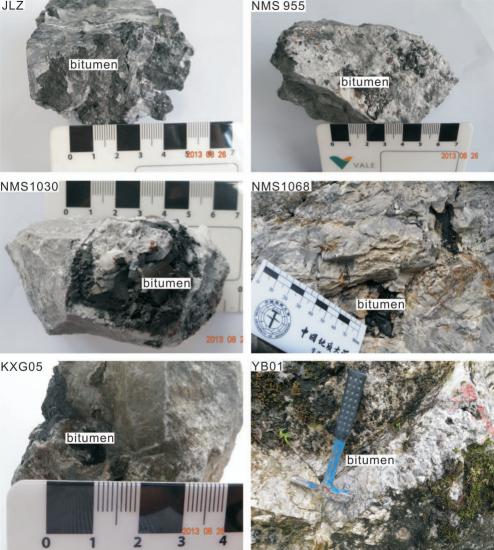
1242 Uplift, North Sichuan Basin

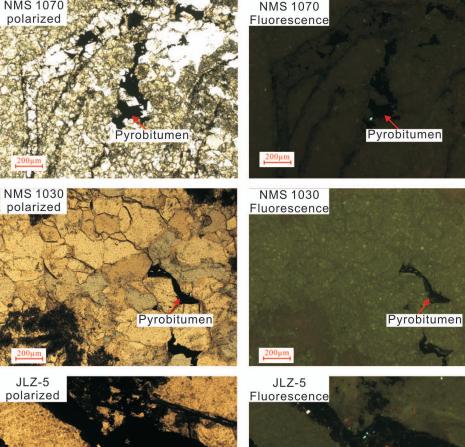
Table 2. Apatite fission track data from the Micang Shan Uplift and inner SichuanBasin



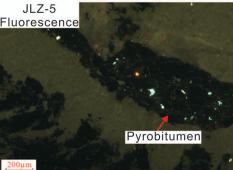


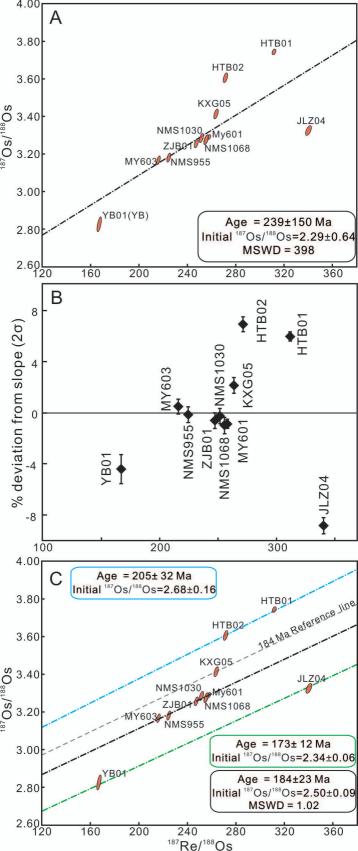
Perio	d	Formation	Lithology	Thickness (m)	Petroleum System	Tectonic Event			
Cretaceous	145	Jiange (K₁j)	• • <td>0-2000</td> <td></td> <td></td>	0-2000					
	145	Penglai (J ₃ p)	• • • • • • • • • • • • • • • • • • •	650-1400		Manakan			
		Suining (J_2s)		340-500	Reservoir	Yanshan event			
Jurassic		Shaximiao (J_2s)		600-2800	Reservon	ovent			
	201	Ziliujing (J_1z)		200-900					
	201	Xujiahe (T ₃ x)	·····	250-3000	Reservoir				
Talanaia					Source	Indosinian			
Triassic		Jialingjiang (T ₁ j)	Si IIII Si IIII Si I	900-1700		event			
	252	Feixianguan(T₁f)			Reservoir				
	2.52	Changxing (P ₂ c)		000 500					
		Longtan (P ₂ I)		200-500	Source				
Permian		Maokou (P₁m)			Reservoir	Hercynian			
		Qixia (P₁q)		200-500	Reservoir	event			
	299	Liangshan (P₁I)			Source				
Carboniferous	359	$Huashiban(C_2h)$		0-500					
		Hajiadian (S₂h)							
Silurian		Xiaoheba (S ₁ x)		0-1500					
	444	Longmaxi (S₁I)			Source				
Ordovician		Baota (O ₂ b)		0-600					
	485	Meitan (O₁m)		0 000					
		Douposi (∈₂d)							
		Shimendong (∈₁s)				Caledonian event			
Cambrian		Yanwang (∈₁y)		0-2500		eveni			
		Xiannvdong (∈₁x)							
	541	Qiongzhusi (∈₁q)			Source				
Ediacaran		$Dengying(Z_2d)$		200-1100	Reservoir				
	635	Guanyinya(Z₂g)							
Pre- Ediacaran		Liangjiang(Pt)				Jinning event			

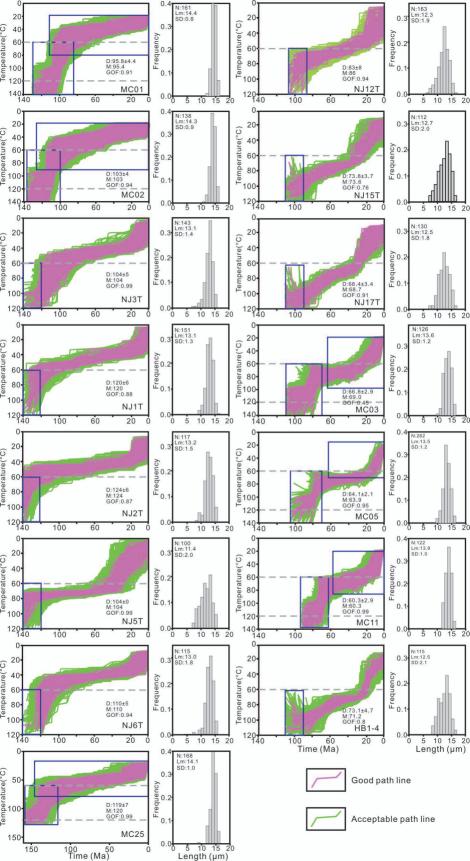


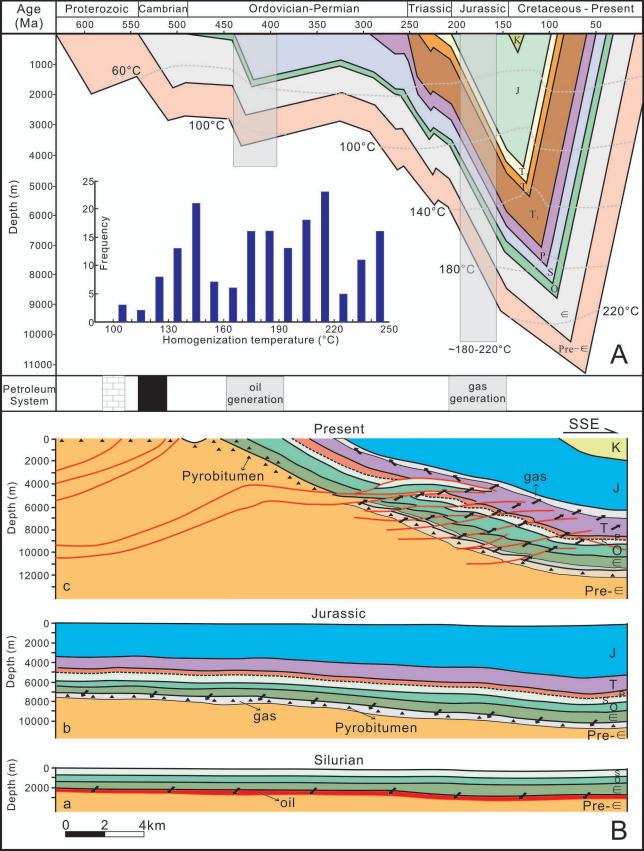


Pyrobitumen









Sample name	Latitude	Longitude	Strata	BRo (%)	Re (ppb)	±	Os (ppt)	±	¹⁸⁷ Re/ ¹⁸⁸ Os	±	¹⁸⁷ Os/ ¹⁸⁸ Os	±	rho	Osi (239Ma)
HTB01	32°28'58"	107°05'39"	Dengying formation	/	179.5	0.6	4087.2	18.6	311.6	1.3	3.746	0.012	0.419	2.502
HTB02	32°30'02"	106°52'42"	Dengying formation	/	177.4	0.6	4579.2	28.3	271.4	1.5	3.608	0.021	0.554	2.525
MY601	32°31'32"	107°19'08"	Dengying formation	/	191.4	0.7	5053.0	21.7	257.8	1.0	3.291	0.010	0.391	2.263
MY603	32°31'28"	107°18'54"	Dengying formation	/	154.8	0.5	4823.7	28.7	216.0	1.2	3.168	0.019	0.552	2.306
NMS955	32°31'28"	107°18'56"	Dengying formation	3.97	143.5	0.5	4307.0	26.5	224.4	1.3	3.182	0.020	0.591	2.286
NMS1030	32°31'48"	107°19'38"	Dengying formation	3.94	184.9	0.6	5002.3	30.6	251.5	1.4	3.286	0.020	0.580	2.283
NMS1068	32°31'41"	107°19'42"	Dengying formation	3.25	113.8	0.4	3030.4	19.1	255.4	1.5	3.277	0.021	0.625	2.258
ZJB01	32°29'52"	107°09'54''	Dengying formation	/	153.8	0.5	4220.9	25.3	247.2	1.3	3.258	0.019	0.555	2.271
JLZ04	32°32'11"	107°19'48"	Dengying formation	4.08	188.1	0.7	3777.1	23.8	340.2	2.0	3.326	0.021	0.606	1.969
YB01	32°28'19"	106°47'10"	Dengying formation	/	145.2	0.5	5669.5	47.4	166.8	1.5	2.826	0.032	0.653	2.160
KXG05	32°29'39"	107°09'35"	Dengying formation	3.97	106.2	0.6	4812.9	30.4	263.6	1.5	3.415	0.021	0.585	2.363

Table 1.Re-Os isotopic data on the bitumen from the Micang Shan Uplift, North Sichuan Basin

Footnotes: BRo = Bitumen reflectance; Re (ppb) = rhenium (part per billion); Os (ppt) = Osmium (parts per trillion); 187 Re/ 188 Os = 187 rhenium/ 188 Osmium; 187 Os/ 188 Os = 187 Osmium; / 188 Osmium; rho = the associated error correlation of the 187 Re/ 188 Os and 187 Os/ 188 Os; Osi(239Ma) = initial 187 Os/ 188 Os ratio at 239 Ma.

AFT					Elevation	AFT age		Length		
sample	Latitude	Longitude	Lithology	Strata	(m)(f t)	(Ma)	±	(μm)	±	Ref
Uplift										
MC01	32°41'54"	107°07'27''	Granite	Proterozoic	1106(3628)	95.8	4.4	13.00	0.10	Yang et al., 2013
MC02	32°41'17"	107°08'24''	Granite	Proterozoic	1073(3520	103.0	3.7	12.90	0.10	Yang et al., 2013
NJ3T	32°37'12"	106°49'30"	Diorite	Proterozoic	1650(5413)	103.0	5.1	13.14	0.12	Tian et al., 2011
NJ1T	32°36'58"	106°49'41"	Diorite	Proterozoic	1554(5098)	119.8	5.9	13.13	0.10	Tian et al., 2011
NJ2T	32°36'36"	106°49'48"	Diorite	Proterozoic	1588(5210)	123.5	6	13.23	0.10	Tian et al., 2011
NJ5T	32°35'42"	106°50'28"	Diorite	Proterozoic	1268(4160)	103.7	4.2	11.40	0.25	Tian et al., 2011
NJ6T	32°35'28"	106°50'28"	Sandstone	Proterozoic	1286(4219)	110.1	5.3	12.95	0.12	Tian et al., 2011
MC-8a	32°35'22"	106°50'40"	Diorite	Proterozoic	1269(4163)	99.0	/	/		Sun, 2011
NJ8T	32°34'26"	106°50'42"	Siltstone	Pre-cambrian	1259(4130)	93.9	4.1	/	/	Tian et al., 2011
WCS-20	32°33'09"	106°52'11"	Granite	Proterozoic	703(2306)	63.7	6.4	12.10	0.20	Lei et al., 2012
MC25	32°32'45"	106°51'53"	Granite	Proterozoic	1317(4321)	110.7	6.8	12.80	0.10	Yang et al., 2013
Basin										
NJ12T	32°28'26"	106°52'44"	Sandstone	Ordovician	636(2087)	82.9	7.8	12.25	0.17	Tian et al., 2011
MC-17a	32°28'19"	106°26'44"	Diorite	Proterozoic	625(2050)	81.0	/	/	/	Sun, 2011
NJ14T	32°27'18"	106°53'20"	Sandstone	Silurian	575(1886)	60.8	5.7	/	/	Tian et al., 2011
MC12	32°26'50"	106°38'37''	Diorite	Proterozoic	978(3209)	68.9	3.5	/	/	Yang et al., 2013
WCS-17	32°26'02"	106°38'38"	Conglomerate	Cambrian	1139(3736)	80.4	10.6	12.10	0.20	Lei et al., 2012
NJ15T	32°25'30"	106°51'50"	Sandstone	Triassic	506(1660)	73.8	3.7	12.66	0.20	Tian et al., 2011
NJ17T	32°22'19"	106°51'11"	Sandstone	Jurassic	557(1827)	68.4	3.4	12.49	0.16	Tian et al., 2011
MC14	32°21'39"	107°10'17''	Sandstone	Jurassic	500(1640)	62.4	5.2	/	/	Yang et al., 2013
MC03	32°21'07"	107°10'06''	Sandstone	Triassic	515(1690)	66.8	2.9	11.80	0.20	Yang et al., 2013
MC04	32°20'51"	107°10'37"	Sandstone	Jurassic	550(1804)	78.2	5.8	/	/	Yang et al., 2013
MC05	32°19'31"	107°10'44''	Sandstone	Jurassic	505(1657)	64.1	2.1	11.50	0.10	Yang et al., 2013
WCS-16	32°19'17"	106°32'13"	Conglomerate	Cambrian	541(1775)	69.8	6.2	11.40	0.30	Lei et al., 2012
MC11	32°17'56"	107°09'32"	Sandstone	Triassic	497(1631)	61.6	3	12.20	0.20	Yang et al., 2013
MC09	32°15'26"	107°26'05"	Sandstone	Cretaceous	448(1470)	60.3	2.3	/	/	Yang et al., 2013
MC15	32°14'52"	106°58'30"	Sandstone	Cretaceous	461(1512)	66.8	2.5	12.20	0.20	Yang et al., 2013
MC08	32°13'06"	107°10'41"	Sandstone	Jurassic	481(1578)	70.2	4.5	/	/	Yang et al., 2013
WCS-14	32°11'34"	106°27'50''	sandstone	Jurassic	462(1516)	70.7	4.7	10.60	0.30	Lei et al., 2012
WCS-15	32°08'18"	106°29'19"	sandstone	Cretaceous	419(1375)	77.4	6.1	10.90	0.30	Lei et al., 2012
Borehole										
HB1-4	32°05'49"	107°06'07''	Sandstone	Jurassic	-68(-223)	73.1	4.7	12.48	0.20	Tian et al., 2011
HB1-5	32°05'49"	107°06'07''	Sandstone	Jurassic	-2594(-8510)	25.6	2.2	11.02	0.40	Tian et al., 2011
HB1-6	32°05'49"	107°06'07''	Sandstone	Jurassic	-2972(-9751)	16.1	1.5	10.62	0.34	Tian et al., 2011
HB1-8	32°05'49"	107°06'07''	Siltstone	Triassic	-3398(-11148)	17.3	3	10.00	0.45	Tian et al., 2011
HB1-9	32°05'49"	107°06'07''	Sandstone	Triassic	-3496(-11470)	14.0	2.5	11.15	0.41	Tian et al., 2011
HB1-1	32°05'49"	107°06'07''	Sandstone	Triassic	-4485(-14715)	8.8	1.3	10.22	0.48	Tian et al., 2011

Table. 2 Apatite fission track data from the Micang Shan Uplift inner Sichuan Basin