NOTES

Further support for a Cretaceous age for the feathered-dinosaur beds of Liaoning,China:New $^{40}Ar/^{39}Ar$ dating of the Yixian and Tuchengzi Formations

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Abstract **We report new $^{40}Ar/^{39}Ar$ dating results obtained from total fusion and incremental-heating analyses of sanidine and biotite from three tuffs found interbedded within the fossil-bearing deposits of Liaoning, northeast China. The first is a new sample of the Bed 6 Sihetun tuff from the Yixian Formation, previously dated by our team as middle Early Cretaceous, and recently considered by Lo et al., partially reset due to metamorphism from a nearby basaltic sill. The second is the Yixian Bed 9 tuff from Hengdaozi considered by Lo et al. to be unaffected by metamorphism and whose age, based on total fusion $^{40}Ar/^{39}Ar$ dating of biotite, argues for a Jurassic age for the Yixian Formation. The third tuff is a previously undated tuff from the upper part of the underlying Tuchengzi Formation. Single crystal total fusion $^{40}Ar/^{39}Ar$ analyses of the Sihetun sanidine showed homogeneous radiogenic Ar, Ca/K ratios, excellent reproducibility and gave a mean age of 125.0 ± 0.18 (1SD) ± 0.04 (SE) Ma. Single sanidine crystal total fusion $^{40}Ar/^{39}Ar$ analyses of the Hengdaozi tuff gave a mean age of 125.0 ± 0.19 (1SD) ± 0.04 (SE) Ma, which is indistinguishable from the Sihetun tuff. The Tuchengzi Formation tuff gave a mean age of 139.4 ± 0.19 (1SD) ± 0.05 (SE) Ma. Detailed laser incremental-heating analyses of biotite from Sihetun, Hengdaozi, and Tuchengzi tuffs show disturbed Ar release patterns and evidence of trapped argon components. We conclude from these analyses that the total fusion dates on biotite by Lo et al. are erroneously old and isotopic dating of both biotite and sanidine from tuffs of the Yixian Formation point to a middle Early Cretaceous age. The upper part of the Tuchengzi Formation can be referred to the Early Cretaceous.**

Keywords: $^{40}Ar/^{39}Ar$ dating, Cretaceous, Yixian Formation, Tuchengzi Formation, western Liaoning.

Excavations in the lake beds of the Yixian Formation of Liaoning, China, continue to unearth a wide variety of spectacular fossils that further help elucidate views on long lived controversies over bird-dinosaur relationships, early diversification of birds and mammals and the evolution of flowering plants[1—4]. Although the "primitive" appearance of some of these fossils have suggested temporal links with the Tithonian (Late Jurassic) faunas of Europe, such as the *Archaeopteryx* bearing Solnhofen limestone of southern Germany, other fossil data indicate a much younger, middle Early Cretaceous age. Support for a Jurassic age of the Yixian fossils comes from K-Ar and Rb-Sr dating of associated volcanics[5,6], whereas; $^{40}Ar/^{39}Ar$ dating of interbedded tephra and overlying lavas lends support to an Early Cretaceous age[7—10]. As a result, two competing camps have emerged; one that views the Yixian fossils as Late Jurassic contemporaries of *Archaeopteryx* and a second that argues that while many of the fossils possess primitive characteristics, the Yixian fossils are of middle Early Cretaceous age, some 20 million years younger than deposits that produced *Archaeopteryx*[7].

1 Background and debate

In 1999, members of our group[7] reported $^{40}Ar/^{39}Ar$ dates on volcanic layers interbedded within the fossil-bearing layers of the Yixian Formation that indicate a Barremian (middle Early Cretaceous) age. The 124.6 Ma dates were based on replicate total fusion $^{40}Ar/^{39}Ar$ analyses of single sanidine crystals and incremental-heating $^{40}Ar/^{39}Ar$ analysis of a multiple crystal sample separated from tuff layers of the Yixian Formation exposed at the sites of Sihetun and Jianshangou. We concluded that our dates are more reliable than prior K-Ar and Rb-Sr dates on biotite and whole rock[5,6] because of the superior argon retentivity of sanidine and the unambiguous relationships of our dated units with fossiliferous horizons of the Yixian Formation[7]. Support for our age for the Yixian Formation comes from $^{40}Ar/^{39}Ar$ dating of overlying lavas and intrusive sills by Smith et al.[8] that yielded dates averaging 121 Ma, and from 125.2 Ma U-Pb dates on zircon reported by Wang et al.[9,10] on the same Bed 6 Sihetun tuff dated by $^{40}Ar/^{39}Ar$ methods reported in Swisher et al. [7]. The underlying basalt also gave $^{40}Ar/^{39}Ar$ dates of 128 Ma[10] which further supports the Early Cretaceous age of the Yixian Formation.

Recently, Lo et al.[11,12] have presented new dating results that challenge our Cretaceous age for the Yixian Formation. Their view comes from recent 147 Ma $^{40}Ar/^{39}Ar$ dates on biotite and whole rock from Yixian tuffs. Jiang et al.[13] utilizing Lo et al.'s dates[11,12] argue that although our team[7] dated volcanic horizons from sites unambiguously associated with the main fossiliferous horizons of the Yixian Formation, our sample localities had been affected by local contact metamorphism. A nearby 121 Ma intrusive basaltic sill was considered to have thermally reset the age of the sanidine from the Sihetun Tuff dated by our team. In contrast, Jiang et al.[13] point out that Lo et al.[11,12] dated samples from Hengdaozi, a site unaffected by the younger intrusive basalts, and therefore, the older 147 Ma dates more accurately reflect the geologic age of the Yixian Formation. Combined with the previously published K-Ar date of (137 ± 7) Ma and Rb-Sr date of (143 ± 4) Ma[5,6] from the Yixian Formation, Jiang et al.[13] conclude that the Yixian fossil-bearing lake beds are Late Jurassic age, contemporaries of the *Archaeopteryx* bearing Solnhofen limestone (note: these workers use an age of around 135 Ma for the Jurassic/ Cretaceous boundary, although most workers now accept an age for the boundary of around 144 Ma[14]).

Lo et al.'s[11,12] dating serves as the basis for Jiang et al.'s arguments against an Early Cretaceous age for the Yixian Formation. The dates are based on laser total fusion ^{40}Ar/^{39}Ar analyses of single crystals of biotite from two samples of a tuff from Bed 9, situated 20—30 m above the main Sihetun fossiliferous layers of the Yixian Formation at Hengdaozi[15,16]. Individual biotite crystal dates from the two samples range from 136 Ma to 155 Ma, with means of (147.1 ± 5.5) Ma and (142.5 ± 3.6) Ma. Plotting the ^{36}Ar/^{40}Ar and ^{39}Ar/^{40}Ar data from these data on isochron plots, Lo et al. derive isochron dates of (147.9 ± 2.1) Ma and (142.2 ± 2.2) Ma[11,12]. ^{40}Ar/^{36}Ar intercept values are within uncertainty of air ratio, which indicated to Lo et al. that the isotopic systems of the biotites were relatively closed, that there was no apparent trapped argon component and no significant alteration. Combining the data for the two samples gave an overall mean age of (145.3 ± 4.4) Ma and a combined isochron date of (147.1 ± 1.8) Ma[11,12].

Lo et al.[11,12] also report dates for groundmass (whole rock) chips from these two samples. Individual total fusion dates range from 116 to 130 Ma, with means of (125.5 ± 4.2) Ma and (120.8 ± 3.1) Ma. An isochron plot of the individual analyses of one of the samples gave an air intercept and date of (127.8 ± 1.9) Ma. Although they point out that this age is comparable to those reported by our group[7] and by Smith et al.[8], the matrix was most likely affected by slight alteration and therefore not geologically meaningful[11,12]. Besides, they also report a whole rock K-Ar date on a fine-grained volcanic ash from Bed 6[15,16] of (147.3 ± 3.3) Ma, but because of its fine-grained character they were unsuccessful in obtaining ^{40}Ar/^{39}Ar dates on this sample[11,12].

At first glance, Lo et al.'s biotite data[11,12] and the arguments raised by Jiang et al.[13] lend credence to the possibility that the Yixian Formation may indeed be of Jurassic age. However, after close examination of Lo et al.'s data and further ^{40}Ar/^{39}Ar analyses of the biotite and sanidine from the Hengdaozi tephra that was analyzed by Lo et al.[11,12] by our group, we disagree with their conclusions.

We argue that the intrusive sill at Sihetun is of insufficient proximity to our sample sites at Sihetun and Jianshangou to have any major adverse affect on the argon systematics of the dated sanidine. We note from field observation that no physical evidence of metamorphism of either the dated tephra or the adjoining fossiliferous sedimentary beds occurs beyond about 0.5—1 m of the margins of the basalt sill. We also note no apparent disturbance of the argon systematics as observed in the release pattern obtained from our incremental-heating analysis of the sanidine[7].

In contrast, we point out that the single biotite crystal dates reported by Lo et al.[11,12] show much greater spread in age (136—155 Ma) than is expected from a single homogeneous mineral population. This leads us to believe that the Ar systematics of the biotite is disturbed either by alteration or by a trapped Ar component. Given that all of these dates are based on the total fusion analyses, any disturbance of the argon systematics or the determination of trapped argon components cannot be fully examined. Plotting the total fusion dates on a single isochron do show a linear array with an atmospheric intercept, however, we believe this is a result of various crystal to crystal amount of surficial atmospheric contamination released from weak interstitial layers and altered surfaces of the biotite. No incremental heating analyses were made that could address low temperature surficial and higher temperature trapped Ar components. As a result, these total fusion ^{40}Ar/^{39}Ar data result in similar ages as produced previously by total fusion K-Ar techniques.

2 Samples, analyses and results

To directly address these issues, we analyzed sanidine and biotite from three different tuff samples from the Liaoning area. The first, for control purposes, is a new sample (99L-S1) of the Bed 6 Sihetun tuff previously dated by us, and reported by Lo et al.[11,12] to be affected by metamorphism. The second sample (99L-HDZ1), is the Bed 9 tuff from Hengdaozi dated by Lo et al.[11,12] reportedly unaffected by metamorphism. The third tuff (99L-T1) is a previously undated tuff from the underlying upper part of the underlying Tuchengzi Formation.

^{40}Ar/^{39}Ar dating of Liaoning tuffs was accomplished by replicate CO_2 laser, total fusion analyses of single sanidine crystals and incremental-heating of biotite following procedures outlined previously by us in Swisher et al. [7] references therein. All analyses were made using a Mass Analyzer Products 215-50 mass spectrometer at the Berkeley Geochronology Center, in Berkeley California, following procedures outlined by Renne et al.[17] and references therein. The ages were calculated using an age of 28.02 Ma for the co-irradiated Fish Canyon Sanidine monitor mineral[17]. To control and minimize uncertainties due to lateral neutron flux gradients during sample irradiation, each Liaoning sample was arranged in Al sample holders bracketed between multiple samples of Fish Canyon Sanidine. Uncertainties reported are one standard deviation of mean and standard error of the mean and do not take into account uncertainties in the "absolute" age of the monitor mineral.

24 single crystal total fusion $^{40}Ar/^{39}Ar$ analyses of the Sihetun sanidine show homogeneous radiogenic Ar, Ca/K ratios, excellent reproducibility and analytical uncertainty ranging in age from 124.7 to 125.3 (Plate Ⅰ(a)). Our mean age of 125.0 ± 0.18 (1SD) ± 0.04 (SE) Ma is slightly older than the 124.6 Ma we reported[7] previously and attribute this slight age difference to improved standard calibration and control of lateral gradients induced during the two irradiations. We consider this new date a more accurate age for Sihetun and Hengdaozi than previously reported by our group. The sanidine from the Bed 9 Hengdaozi tuff located 20—30 m above the Bed 6 tuff gave indistinguishable results from the Sihetun tuff. 24 single sanidine crystal total fusion $^{40}Ar/^{39}Ar$ analyses of this sanidine gave a mean age of 125.0 ± 0.19 (1SD) ± 0.04 (SE) Ma (Plate Ⅰ).

The tuff from the upper part of the underlying Tuchengzi Formation gave a mean $^{40}Ar/^{39}Ar$ age of 139.4 ± 0.19 (1SD) ± 0.05 (SE) Ma, based on 19 single sanidine crystal analyses (Plate Ⅰ(a)). This represents the first isotopic dating for the Tuchengzi Formation and although considered previously middle Jurassic in age, at least the upper part of the formation can be referred to the Early Cretaceous. The new date leads further credence to our Cretaceous age for the overlying Yixian Formation and limits the previously recognized hiatus between the two formations to less than 15 million years. As a result, the stratigraphic location of the continental Jurassic-Cretaceous boundary in northeastern China must be sought for within the lower part of the Tuchengzi Formation or within underlying formations.

Our new analyses indicate that all three tuffs give dates consistent with a Cretaceous age for the Yixian Formation, as well as for the underlying upper Tuchengzi Formation. The excellent agreement between the sample from Sihetun and that from Hengdaozi supports our view that the Sihetun sanidine was not affected by contact metamorphism event. The congruence between our new age for the Sihtuen tuff of 125.0 ± 0.18 (1SD) ± 0.04 (SE) based on $^{40}Ar/^{39}Ar$ dating of sanidine and the U-Pb zircon dates reported by Wang et al. [9] of (125.2 ± 0.9) Ma argues that given the vastly different isotope system behavior and different closure temperature of the zircon and sanidine, the Sihetun tuff was unaffected by metamorphism.

3 Comparison and discussion

What, then is the explanation for the erroneously old total fusion biotite and whole rock dates? To examine this issue more closely, we performed detailed laser incremental-heating analyses of biotite from Sihetun, Hengdaozi, and Tuchengzi tuffs (Plate Ⅰ(b)). In all three cases, the incremental-heating analyses show disturbed Ar release patterns. Of importance, both the Sihetun and Tuchengzi biotites give integrated ages of 152 Ma and the Hengdaozi biotite 135 Ma. These ages are comparable to the reported total fusion data[11,12] and lead to an explanation of the old dates. Plotting the $^{36}Ar/^{40}Ar$ against the $^{39}Ar/^{40}Ar$ ratios obtained for the individual temperature increments on an isochron (Plate Ⅱ), the Sihetun and Hengdaozi biotites both indicate excess-trapped Ar components[18]. For the Sihetun biotite, the lower temperature steps give an isochron age of 154 Ma with a $^{40}Ar/^{36}Ar$ intercept of 332; however, the higher temperature steps give an age of 127 Ma with an intercept of 940 (Plate Ⅱ(a)). Similarly the lower temperature steps for Hengdaozi give an isochron age of 151 Ma with $^{40}Ar/^{36}Ar$ intercept of 279 and the higher temperature more radiogenic steps give an age of 127 Ma with a $^{40}Ar/^{36}Ar$ intercept of 910 (Plate Ⅱ(b)). Whereas the Sihetun biotite shows a sharp intersection of the two slopes on the isochron plot, the Hengdaozi biotite shows a broader mixing interval with an estimated age of about 134 Ma (Plate Ⅱ).

Contrary to the conclusion drawn by Jiang et al.[13], we note that our detailed incremental-heating analyses of the biotite show evidence of trapped argon components and conclude that the total fusion dates of Lo et al.[11,12] are erroneously old. All isochrons gave intercept values greater than the expected air ratio of 295.5, thus making all total fusion dates too old. Wang et al. [9] also discovered in their XRD analysis that the biotite was almost entirely weathered into the vermiculite, the K element largely ran off during the weathering state, which led to an open K-Ar system, therefore the Ar-Ar age of the biotite has little geological meaning.

Our new data help explain the prior K-Ar dates on biotite as well as the intermediate but younger age of the whole rock chips, in the latter case, the total Ar being dominated by the contribution from sanidine and glass rather than biotite. We conclude that the K-Ar and total fusion $^{40}Ar/^{39}Ar$ dating techniques employed to date the biotite and whole rock by Lo et al.[11,12] and other workers[5,6] were insufficient to characterize the disturbed argon systematics of the Liaoning biotites.

As a result, we disagree with the conclusions reached by Jiang et al.[13] and conclude that all isotopic dating of tuffs from the Yixian Formation point to a middle Early Cretaceous age. Reasons for the anatomical similarities of certain Yixian taxa with Late Jurassic taxa elsewhere will have to focus on biological and ecological explanations.

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