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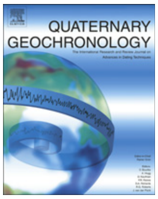

Research Paper

First ^{40}Ar/^{39}Ar age of the Ceprano man (central Italy)

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ABSTRACT

The Ceprano calvarium, found in 1994 in Italy and attributed to *Homo cepranensis*, is one of the most celebrated hominin remains of Europe. It was considered at least 700 ka-old until a recent investigation incorporating magnetostratigraphy and K-Ar ages from the literature assigned to the calvarium an age of ~450 (+50, −100) ka. Here we pin down the age of the Ceprano calvarium to 353 ± 4 ka (±1σ external) by means of new ^{40}Ar/^{39}Ar dating on K-feldspars retrieved from the sediments that hosted the skull. In absence of evidence of reworking, this refined age sinks the conviction that *H. cepranensis* belonged to human evolution at the Brunhes–Matuyama boundary (*c.a.* 781 ka). Our refined age indicates that *H. cepranensis* lived in central Italy probably during the cold period of marine isotope stage (MIS) 10, and that despite his archaic morphology and lack of Neanderthal traits, he was contemporaneous with more advanced species such as *H. heidelbergensis*.

1. Introduction

In 1994, an incomplete hominin calvarium (Ascenzi et al., 1996) attributed to *Homo cepranensis* (Ascenzi et al., 2000) was discovered at Ceprano (Middle Latina Valley, central Italy) (Fig. 1a,b). Since this discovery, the *H. cepranensis* was considered to be at least 700 ka-old or even older (e.g. Ascenzi et al., 1996, 2000; Manzi et al., 2001; Manzi, 2004; Bruner and Manzi, 2007), and was allegedly associated with the Brunhes–Matuyama boundary (Bruner and Manzi, 2007) (*c.a.* 781 ka). Despite the various taxonomic uncertainties, the Ceprano man is morphologically considered, up to the most recent literature, as one of the oldest hominins of Europe (e.g. Manzi et al., 2001; Manzi, 2004; Bruner and Manzi, 2007) and a key specimen to understand the dynamics of hominin evolution and dispersal during the Early–Middle Pleistocene (e.g. Ascenzi et al., 2000; Manzi et al., 2001; Manzi, 2004; Bruner and Manzi, 2007). A recent investigation incorporating magnetostratigraphy from cores taken at the hominin site and old ^{40}K/^{40}Ar ages from the literature from elsewhere in the Ceprano basin showed that the hominin-bearing level lies well within the Brunhes Chron at an interpolated age of ~450 (+50, −100) ka (Muttoni et al., 2009), a conclusion now supported by revised pollen and regional lithostratigraphic analyses (Manzi et al., in press). In order to improve the current age attribution of the Ceprano calvarium by means of direct radiometric age estimates from levels as close as possible to the hominin level, we sampled for ^{40}Ar/^{40}Ar dating the Ceprano 1 core drilled by Muttoni et al. (2009) at the hominin site.

2. Geological setting and material

The Ceprano basin is a tectonic depression within Mesozoic to Miocene limestones (Carrara et al., 1995). It is limited to the southeast by the Roccamonfina volcanic complex (580–150 ka; Rouchon et al., 2008) (Fig. 1a,b) and it was filled by the Liri lacustrine sequence, broadly bracketed by two ^{40}K/^{40}Ar ages of ~354 ± 14 ka[1] and ~583 ± 20 ka reported by Carrara et al. (1995; see also Muttoni et al., 2009) without however providing the details of the analytical procedures followed. Two cores were recently drilled at the Ceprano hominin site and studied for paleomagnetism (Muttoni et al., 2009). We sought for material suitable for radio-isotopic dating throughout the Ceprano 1 core, focusing on the uppermost Unit 1 containing the hominin stratigraphic level. Unit 1 consists in fine- to medium-grained sands

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[1] The uncertainties are quoted at 1σ throughout the text 6.

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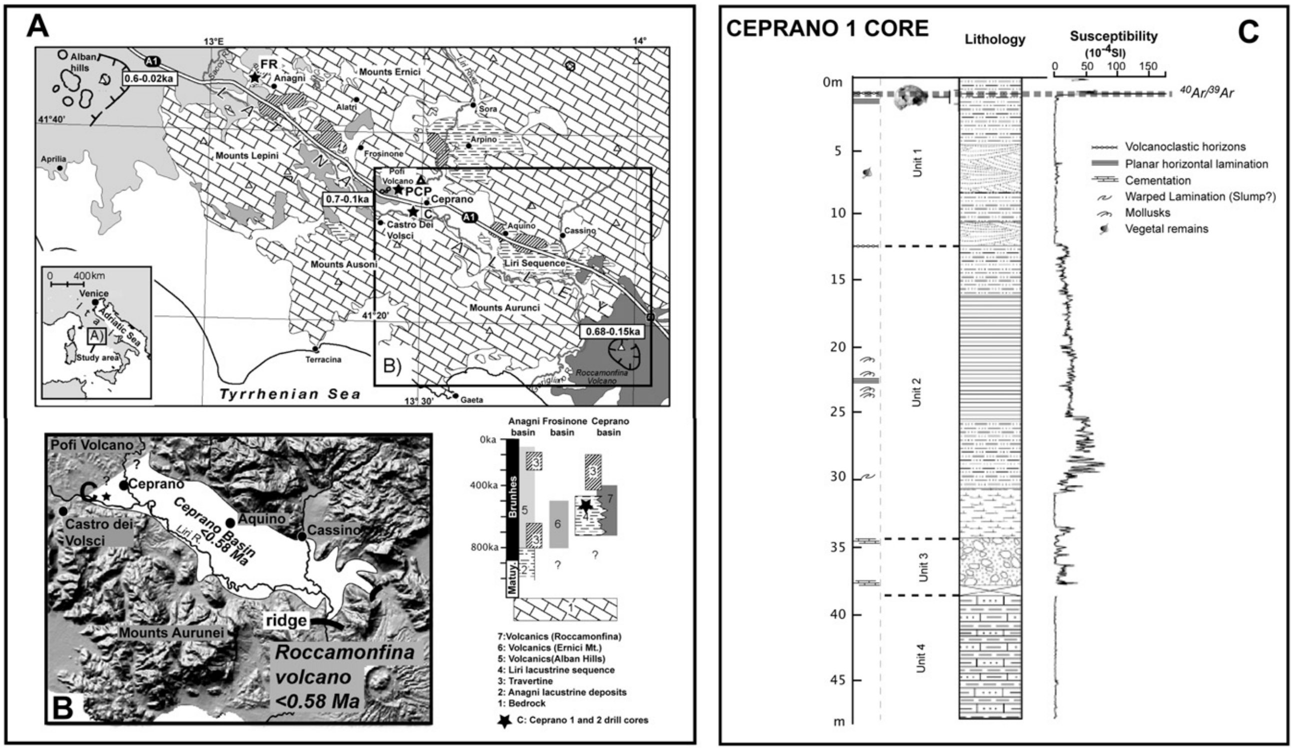

Fig. 1. (A) Simplified geologic map of the Latina Valley (modified from Muttoni et al., 2009) (C = Ceprano; FR = Fontana Ranuccio; PCP = Pofi-Cava Pompi. (B) Digital elevation model of the Ceprano area with indication of basin extension (white shaded area) and stratigraphy (lower right panel). (C) Lithology of the Ceprano 1 core (modified from Muttoni et al., 2009). The locations of the dated horizon (−1.17 to −1.20 m) as well as of the *H. cepranensis remain* are both indicated.

interbedded with thick oxidized silty brown clays of mainly fluvial origin (Fig. 1c). The hominin level falls in the uppermost part of this unit at 1.5 m from core top and 0.3 m below sediments bearing high magnetic susceptibility values (Fig. 1c). Detailed inspection of these high susceptibility sandy layers revealed the presence of reworked volcanic material. In particular, 0.3 m above the hominin level, and 1.0–1.2 m from core top, we found a well-preserved coarse-grained volcanoclastic layer, hereafter referred to as K-layer, which delivered suitable material for radiometric analyses, as illustrated below.

2.1. K-layer mineralogy

The full mineralogical content of the K-layer is provided online (online supplementary table 1). Average composition of clinopyroxene and sanidine (see online supplementary table 2) found in the K-layer were determined using a PGT X-ray energy dispersive spectrometer (EDS) attached to a JEOL 840 scanning electron microscope (SEM) (LSCE, France). Quantitative analyses were obtained by comparing X-ray spectra of individual grains to a series of pure reference spectra. X-ray absorption and fluorescence effects were corrected using the ZAF program supplied by PGT.

The K-layer consists of 94% of loose crystals and 6% of lithic fragments up to 1 cm including whitish-grey pumices and lava fragments. Pumices fragments display the same mineralogy than the loose crystals (see below) including small K feldspars (about 50–100 μm). These crystals are however too small to be individually dated. The loose crystals fraction is dominated by angular to subangular, fresh, light to dark green clinopyroxene (Cpx) (mean composition: $Wo_{52.3}En_{34.1}Fs_{12.9}Ac_{0.7}$), light brown Cpx (mean composition: $Wo_{50.6}En_{43.4}Fs_{5.3}Ac_{0.8}$), transparent K-feldspars (mean composition: $An_{2.3}Ab_{15.4}Or_{82.3}$), milky plagioclase (sericite), brown-amphibole, as well as Fe oxides. Milky quartz and altered phyllosilicates (phologopite and muscovite) also form a minor component and are presumably xenocrysts. K-feldspars are surprisingly fresh, subangular and some are large enough (up to 400–600 μm) to be individually dated. These K-feldspars are homogeneous in composition (Or_{75-87}) but usually zoned with BaO up to 2% in the rim (online supplement table 2). No fresh leucite was detected in the K-layer, albeit this is not surprising as even in-situ tephra deposits found in the nearby Roccamonfina volcanoes yielded mainly altered leucites (Luhr and Giannetti, 1987). Despite the lack of leucite, the mineralogy found in the K-layer is consistent with the mineralogical assemblages of the High-K (HKS) and Shoshonitic (SHO) series described in the nearby Roccamonfina (Luhr and Giannetti, 1987; Giannetti, 2001), Alban Hills (Marra et al., 2009), and Middle Latin Valley (MLV) (Frezzotti et al., 2007; Boari et al., 2009) volcanic fields (Fig. 1a).

2.2. $^{40}Ar/^{39}Ar$ method

About 250 g of the K-layer was processed as follows: 1) The sample was repeatedly washed in deionized water; 2) the 500 μm–1000 μm size fraction, free of clays, was leached in a 100% acetic acid solution and ultrasonic-cleaned for 30 min at 60 °C; 3) after several rinsing with distilled water, the fraction was dried; 4) After drying, the magnetic fraction was eliminated using a REE hand magnet; 5) K-feldspars were separated from the non-magnetic fraction using Sodium Polytungstate heavy liquid (SPT) calibrated in density between $2.57 < d > 2.54$ using deionized water; 6) K-feldspars crystals were then handpicked under a binocular.

In order to avoid problems related to alteration and K loss, we choose to pick only transparent K feldspars. Chosen crystals were then slightly leached for 5 min in a 7% Hydro Fluoric solution for further cleaning. Finally, a total of 30 grains were loaded in a single pit on an aluminum disk. K-feldspars were irradiated for 30 min (Irr-15) in the β1 tube of the OSIRIS reactor (CEA Saclay, France). After irradiation, 17 single grains were transferred into individual wells in a stainless steel sample holder and loaded into a differential vacuum Cleartran© window. Each single grain was then totally fused (equivalent to 9% of the laser power) using a focused 25 W CO_2 laser (Synrad©). Gas clean up is achieved by two GP 110© (ZrAl) getters operating at 250° C. Argon isotopes were analyzed using a VG5400 mass spectrometer operated in ion-pulse counting mode with a single muliplier (Balzers© SEV 217 SEN). Calculated sensitivity is about 1.32×10^{-12} mol per Volt measured. Each Ar isotope measurement consists of 20 cycles by peak switching between the different argon isotopes. Neutron fluence (J) was monitored by Alder Creek Sanidine (ACR-2; Nomade et al., 2005) placed in the same pit as the sanidine grains. The J value was determined using three single ACR-2 grains. The corresponding J value (Supplementary Table 3) was calculated using an age of 1.193 Ma for ACR-2 and the total decay constant of Steiger and Jager (1977). J uncertainty corresponds to the standard deviation of the weighted mean of the three ACR-2 single grains and excludes age uncertainty on the standard age. More accurate calibrations of standard monitors that yield ages about 0.7% and 1.0% older than previously proposed have recently been suggested (Kuiper et al., 2008; Renne et al., 2010). However, systematic errors of this magnitude will have a minimal effect on our result. Procedural blanks were measured every three grains. For typical 9 min static blank, typical backgrounds are about $2.0–2.2 \times 10^{-17}$ and 5.0 to 6.0×10^{-19} mol for ^{40}Ar and ^{36}Ar, respectively. The precision and accuracy of the mass discrimination correction was monitored by weekly measurements of air argon of various beam sizes. This monitoring is performed using a dedicated air-calibration system featuring a 6l tank filled with purified atmospheric argon. This tank is connected to the mass spectrometer through the laser extraction system via two pneumatically-actuated air pipettes of approximately 0.1 and 1.0 cc. This system allows for a 1cc (8.0×10^{-3} V on ^{40}Ar) and a 0.1cc (8.5×10^{-4} V on ^{40}Ar) atmospheric aliquots to be delivered into the mass spectrometer and permits a careful monitoring of the mass discrimination over a wide dynamic range with a precision better than 0.2% (±2σ standard deviation for multiples of experiments) for any given bean size measured (see Nomade et al., 2010, for further details). Nucleogenic production ratios used to correct for reactor produced Ar isotopes from K and Ca that are reported in Supplementary Table 3. These ratios we used were obtained by irradiation of pure optical grade CaF_2 and K_2O in three distinct irradiations of in the OSIRIS reactor before and after irradiation 15.

2.3. ^{40}Ar/^{39}Ar result

A total of seventeen K-feldspars single crystals from the K-layer were dated. Full analytical details for individual crystal experiments corrected for backgrounds are given in the online supplementary table 3. Probability density plots and corresponding individual ^{40}Ar/^{39}Ar single crystal ages (2σ) are presented in Fig. 2. Individual total fusion ages are scattered between 338 ± 48 ka and 1002 ± 3 ka ($\pm 1\sigma$) pointing to the presence of several sanidine populations. Despite this apparently scattered ages, the corresponding probability density plot is relatively simple with a dominant mode peaking around 350 ka, which corresponds to the majority of the 17 grains analyzed (Fig. 2). Only three individual crystals display significantly older ages than the dominant mode (Fig. 2). This mode can be fitted by a slightly tailed Gaussian curve centered at 353 ka. Using this homogeneous population of sanidine we calculated a weighted mean age of 353 ± 2 ka ($\pm 1\sigma$ analytical, MSWD = 1.5, prob: 0.1, $n = 14/17$) (Fig. 2). Including all external errors the uncertainty is 4 ka ($\pm 1\sigma$) and will be used hereafter. The data from the prominent mode define an inverse isochron that displays a well spread single linear array with an intercept-calculated age of 351 ± 3 ka ($\pm 1\sigma$ analytical MSWD = 0.3). The atmospheric trapped ^{40}Ar/^{36}Ar ratio of 296 ± 3 ($\pm 1\sigma$) suggest that the age we obtained is not corrupted by excess ^{40}Ar.

2.4. A refined age model of the Ceprano 1 core

Based on our new ^{40}Ar/^{40}Ar age of 353 ± 4 ka for the K-layer, we propose a refined age model for the Ceprano 1 core (Fig. 3). Sediments of Unit 1 hosting the K-layer were most probably emplaced during the glacial marine isotope stage (MIS) 10 (Lisiecki and Raymo, 2005) (Fig. 3). As we could not detect any obvious unconformity between Units 1 and 2, contrary to Manzi et al. (in press), the underlying Unit 2 bearing interglacial pollens (Muttoni et al., 2009; Manzi et al., in press) was most probably deposited during MIS 11, and the underlying Unit 3 during MIS 12 (Fig. 3). This new age model, which was actually contemplated by Muttoni et al. (2009) albeit as a second best choice, implies sediment accumulation rates for Unit 2 of ~40 cm/ka (Fig. 3). This refined age model has implications for the origin of the dated volcanoclastic material as well as for the age of the Ceprano calvarium, as discussed hereafter.

3. Discussion

Our new ^{40}Ar/^{39}Ar age of 353 ± 4 ka for the K-layer can be used to infer the origin of the dated volcanoclastic material. Several volcanic sequences from the Ceprano general area agree in age with the K layer. These are the Brown Leucitic Tuff (BLT) from Roccamonfina (c.a. 355 to 340 ka; Rouchon et al., 2008), the Villa Senni unit from the Alban Hills (365–351 ka; Marra et al., 2009), and the HKS-SHO series from the MLV volcanic field (*c.a.* 413 to 252 ka; Boari et al., 2009). The relatively high Potassium content (*c.a.* 12–13 wt%) of the K-feldspars (Table 3) retrieved from the K-layer seems to exclude the BLT (*c.a.K* = 11.4 to 8.3 wt%; Luhr and Giannetti, 1987; Rouchon et al., 2008) as the source of the volcanoclastic material of the K-layer. Moreover, the K-layer grain size (phenocrystals up to 2 cm) and subangular shape both suggest a proximal source, arguably located closer to Ceprano than to the Roccamonfina or Alban Hills (Fig. 1a). We currently favor the Pofi

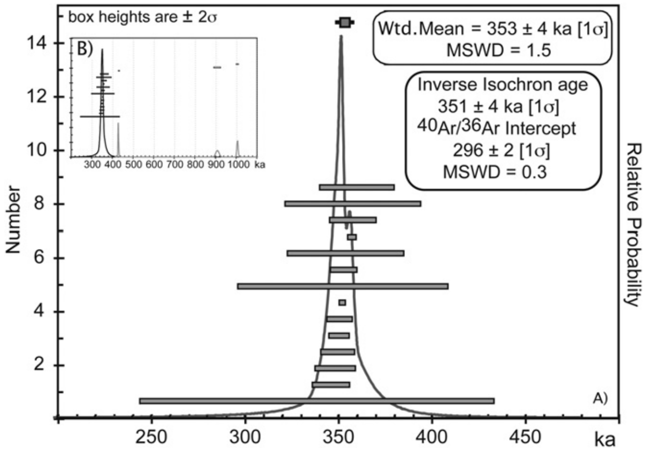

Fig. 2. (A) ^{40}Ar/^{39}Ar age distribution ($N = 14$) of the K-feldspar crystals from the dominant mode corresponding to a slightly tailed Gaussian centered at 353 ± 4 ka ($\pm 1\sigma$ external). (B) Individual ^{40}Ar/^{39}Ar age distribution of all measured K-feldspar crystals ($N = 17$) and corresponding probability density plot.

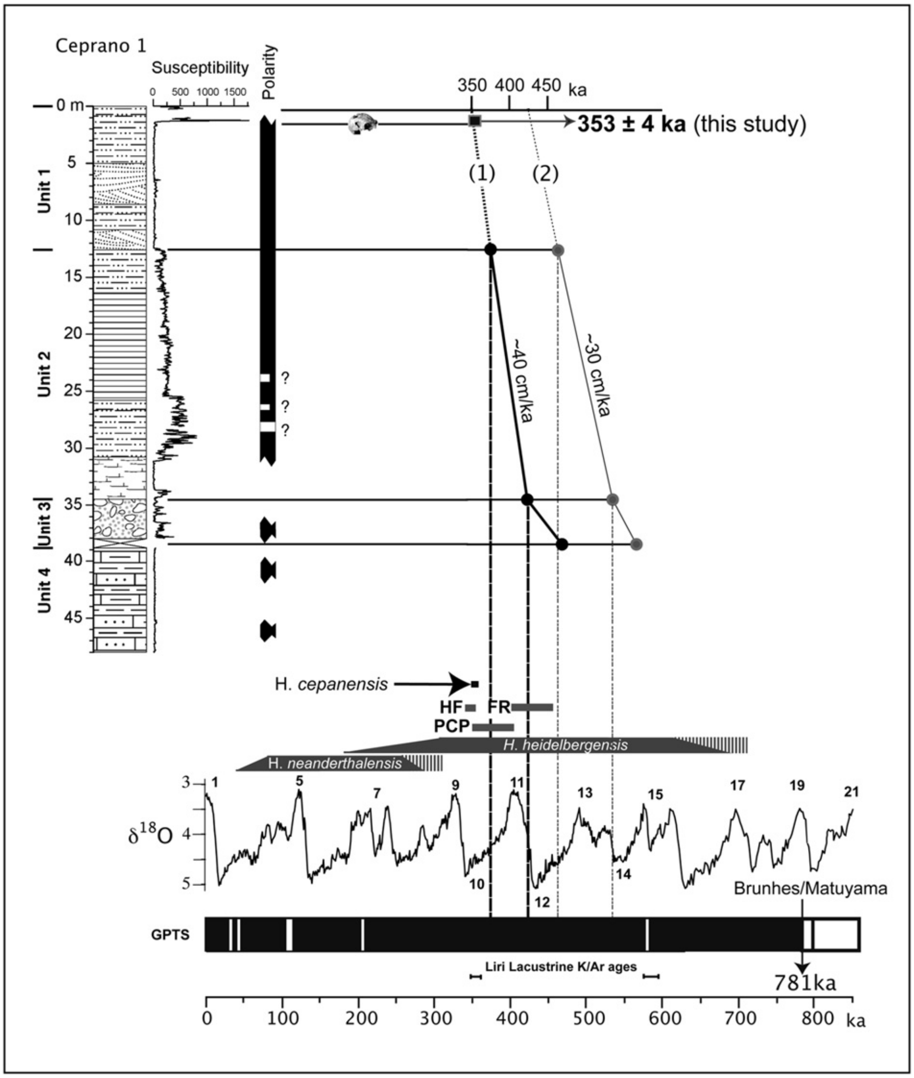

Fig. 3. Age model of sedimentation for the Ceprano 1 core developed by taking into account our new ^{40}Ar/^{39}Ar age of 353 ± 4 ka (curve (1)) compared with the model developed by Muttoni et al. (2009) (curve (2)). The Middle Pleistocene marine oxygen isotope record (LR04 stack, Lisiecki and Raymo, 2005). In our refined age model (1), lacustrine sedimentation of unit 2 corresponds to MIS 11 and alluvial fan deposition of unit 3 to MIS 12. Unit 1 was probably deposited during glacial MIS 10 stage. The age estimates of the various archeological sites discussed in the text are indicated (HF = hominin footpints; FR = Fontana Ranuccio; PCP = Pofi-Cava Pompi). The age windows of *H. neanderthalensis* and *H. heidelbergensis* are also reported for comparison with the revised age of *H. cepranensis* of 353 ka.

polygenic volcano of the MLV volcanic field as the source of the K-layer. The Pofi volcano, located only a few kilometers north of Ceprano (Fig. 1a), experienced important volcanic activity from 410 to 345 ka (Boari et al., 2009), and its products display clinopyroxene compositions (Frezzotti et al., 2007) similar to the K layer (online supplementary table 2).

Our refined age model of the Ceprano 1 core further challenges the conventional wisdom that *H. cepranensis* is close in age to the Brunhes–Matuyama boundary (*c.a.* 781 ka) as reported up to the most recent literature (Bruner and Manzi, 2007). This ancient age was mainly supported by the archaic morphological characteristics of *H. cepranensis*, comparable with much older hominins such as *H. ergaster/erectus* (Bruner and Manzi, 2007). Because of the highly fossilized nature of the skull, "incompatible" with the type of hosting sediment, Ascenzi et al. (1996) proposed that the Ceprano calvarium was reworked from much older sediments. This hypothesis has been later questioned based on the absence of stringent and documented evidence of reworking (Muttoni et al., 2009; Manzi et al., in press). As we found three xenocrysts in the K-layer ranging from 1.0 Ma to 420 ka in age, we should conclude that older reworked crystals are present in the dated horizon. However, we believe that the presence of only three reworked grains out of seventeen grains analyzed does not constitute a solid argument for substantial reworking in the K-layer. Older grains predating eruptive activity are common in volcanoclastic horizons and primary tephra deposits as a consequence of complex eruption dynamics (e.g. Walter, 1994; Deino et al., 1998). Moreover, we could find no sedimentological evidence to justify reworking of an object the size of the Ceprano calvarium (1050–1200 cm^3; Bruner and Manzi, 2007), whereby the observed faint planar lamination and the very fine-grained lithology of the hominin level suggest deposition in low-energy environments.

In the absence of evidence of reworking, the simplest hypothesis is to consider the ^{40}Ar/^{39}Ar age of the K-layer (353 ± 4 ka) as the closest direct age of *H. Cepranensis*. This age is statistically younger than the K/Ar age of 385 ± 20 ka from the upper Liri lacustrine sequence reported (without analytical details) by Carrara et al. (1995; see also Muttoni et al., 2009) and considered by Manzi et al. (in press) as a minimum age for the calvarium deposition.

4. Conclusion

Our new $^{40}Ar/^{39}Ar$ age (353 ± 4 ka) on the K-layer above the Ceprano hominin level in the Ceprano 1 core place *H. cepranensis* in close temporal contiguity with the human footprints suite recovered in a Pleistocene volcanic ash of the Roccamonfina volcano and dated with $^{40}Ar/^{39}Ar$ on leucite to 345 ± 6 ka (Scaillet et al., 2008). This age is also ~50 to 100 ka younger than the age of the Acheulean-bearing site of Fontana Ranuccio, dated with K/Ar to ~460–400 ka (Segre Naldini et al., 2009), and is relatively close to the suggested age of 350–400 ka for the Cava Pompi hominin site, which yielded Acheulean tools and mammal remains (including humans') from levels beneath a sequence of reworked volcanic sand belonging to the Pofi polygenic volcano and very similar to the K-layer dated in this study (Biddittu and Segre, 1978). These numerical ages altogether suggest human occupation in central Italy during both glacial and interglacial stages of the Middle Pleistocene from MIS 12 (Fontana Ranuccio) into the ensuing MIS 11–10 (Ceprano, Roccamonfina footprints, Cava Pompi). Additional known hominin fossils of Europe and Africa broadly dated to the late Middle Pleistocene could be broadly contemporaneous with *H. cepranensis*. These are the Arago 21 cranium from France (Yokoyama et al., 1991), which incidentally shares several common features with *H. cepranensis* (Guipert and Malfart, 2004), the crania found at Sima de Los Huesos in Spain (Bischoff et al., 2003), and the Steinheim skull from Germany (Adam, 1985).

More accurate numerical age data are required to unravel the history of hominin presence in Italy and Europe during the Middle Pleistocene, but one element appears now relatively well established, namely that *H. cepranensis* can no longer be considered a representative of the earliest peopling of Europe, which according to a recent analysis occurred probably around MIS 22 at ~870 ka (Muttoni et al., 2010). Rather, *H. cepranensis*, in spite of his archaic morphology and lack of Neanderthal traits, could be contemporaneous with *H. heidelbergensis* (Fig. 3), considered to be the ancestor of *H. neanderthalensis*.

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Appendix. Supplementary material

Supplementary data related to this article can be found online at doi:10.1016/j.quageo.2011.03.008.

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