In extensional continental settings, crustal-scale normal faults can accommodate deformation and subsidence at their hanging wall via activation and deactivation of subsidiary tectonic structures. Geological data obtained from subsidiary structures are required to infer the position of the tectonic deformation during the spatial-temporal evolution of the growth-fault system, with significant implications for structures belonging to seismogenic settings. Here, we describe a subsidiary tectonic structure (the Amatrice Fault System) accommodating Quaternary extensional deformation in the Amatrice Basin (central Apennines, Italy), which is an intermountain morphostructural depression involved by the 2016–2017 seismic sequence. Structurally, the Amatrice Fault System defines a ∼10 km-long tectonic feature running through the Amatrice Basin, and consists of NNW-SSE-striking and E-W-striking fault segments that interact and link over time. Cross-cutting fault relationships are used to reconstruct a kinematic scenario of fault growth and propagation under an ENE-WSW-directed crustal stretching, consistent with the paleostress regime governing the Quaternary activity of the central Apennines. The analysis of stable carbon and oxygen isotopes on syn-kinematic carbonate mineralizations (calcite veins and calcite fibers on fault surfaces) indicates a meteoric water circulation during the development of the growing fault structure, characterized by variable contributions of organic carbon (soil CO$_2$), and suggesting surface rupture and hydrodynamic interconnection with the vadose zone during faulting. Geochronological U-Th dating on the same mineralizations indicates Middle-Late Pleistocene ages for the main phase of tectonic activity of the Amatrice Fault System, with the younger age being 108 ± 10 ka. To date, we cannot exclude minor activations of the Amatrice Fault System during the Holocene. Our results shed light on the Pleistocene tectonics in the Amatrice Basin, in which the Amatrice Fault System records fault growth, hydrodynamic regime and structural
The Amatrice Fault System

INTRODUCTION

In extensional regime, major faults are promising targets for releasing seismogenic potential during ongoing crustal-scale deformation in active tectonic domains (e.g., Jackson and White, 1989; Lavecchia et al., 1994; Wernicke, 1995; Ofoegbu and Ferrill, 1998). Fault initiation, propagation, and deactivation, as well as the modalities of interaction at fault-segment terminations (e.g., fault linkage, relay ramps, and transverse zones) record the deformation localization during the fault- growth evolution (e.g., Morley et al., 1990; Cartwright et al., 1995; Peacock and Parfitt, 2002; Hus et al., 2006; Fossen and Rotevatn, 2016; Childs et al., 2017; Rotevatn et al., 2019). During crustal stretching, stress concentration and rock-failure recurrence are modulated by overpressure conditions due to fluid circulation within the fault permeability network (e.g., Curewitz and Karson, 1997; Sibson, 2000; Cox et al., 2001; Rowland and Sibson, 2004; Vignaroli et al., 2015; Smeraglia et al., 2016).

While major extensional faults localize the largest amount of displacement and subsidence for basin formation at their hanging wall (e.g., Schlische, 1991; Cowie et al., 2000; Khalil and McClay, 2001; Gawthorpe et al., 2003; Mack et al., 2009; Wilson et al., 2009; Egger et al., 2010; Ford et al., 2017), complex patterns of minor fault systems often accommodate the overall tectonic deformation (e.g., Gibbs, 1984; Destro, 1995; Fossen and Hesthammer, 1997; Cifelli et al., 2007; Balsamo et al., 2008; Faccenna et al., 2011). The structural architecture, spatial distribution, and displacement accumulation of these minor structures are strictly correlated with the growth style and the extent of the master fault, contributing to the whole crustal extension. Moreover, the minor structures directly interact with (and modulate) the processes occurring at the hanging wall of the master fault, such as the accommodation space for basin deposition or the hydrodynamic regime characterizing the fluid flow within the structural permeability network. Therefore, defining the structural architecture of the subsidiary faults, as well as their age of activity and the factors triggering their failure (e.g., fluid circulation), provides useful insights into the localization of the tectonic deformation during the spatial-temporal evolution of the growth-fault system.

The central Apennines (Italy) is an important natural laboratory to test modes of nucleation and growth of active extensional faults, with implication on location and recurrence interval of hazardous earthquakes (e.g., Cello et al., 1997; Cowie and Roberts, 2001; Faure Walker et al., 2010; Wilkinson et al., 2017; Iezzi et al., 2019). In central Apennines, a thickened orogenic nappe pile is reshaped by Quaternary extensional tectonics leading to the formation of crustal-scale faults and intermountain basins (e.g., Malinverno and Ryan, 1986; Cavinato and De Celles, 1999; Mirabella et al., 2018). Active tectonic deformation is mainly localized along a complex seismogenic array of master basin-bounding faults that resulted from the radial propagation of individual fault segments and linkage at their terminations (e.g., Cello et al., 1997; Galadini and Messina, 2001; Galadini and Galli, 2003; Boncio et al., 2004b; Galli et al., 2008; Lavecchia et al., 2016; Pizzi et al., 2017; Falcucci et al., 2018). Within the long seismic history of the central Apennines, three recent and destructive events (the 1997 Colfiorito event, Mw = 6.0; the 2009 L’Aquila event, Mw = 6.1; the 2016–2017 Amatrice seismic crisis, Mw up to 6.5) produced several ruptures at surface along major NW-SE-striking extensional faults (Amato et al., 1998; Cinti et al., 2000; Chiaraluce et al., 2011, 2017). The last 2016–2017 seismic sequence hit the central Apennines with a diachronic activation of km-long fault segments and variable extent of surface ruptures over different regional morpho-structural domains (Carminati et al., 2020). The major amount of coseismic deformation was accommodated over the northwestern part of the area, i.e., along the Vettore Fault and the adjacent syn-tectonics Castelluccio and Norcia basins (e.g., Pizzi et al., 2017; Pucci et al., 2017; Smeraglia et al., 2017; Civico et al., 2018; Perouse et al., 2018; Villani et al., 2018; Galli et al., 2019). Coseismic ruptures also involved the southeastern part of the area (Emergeo Working Group, 2016; Galli et al., 2016; Lavecchia et al., 2016; Pizzi et al., 2017) i.e., along the Gorzano-Laga Fault and the adjacent Amatrice Basin, the latter still representing an under-explored syn-tectonic depression in terms of its structural setting.

The present work describes the tectonic pattern of the Amatrice Basin by introducing the Amatrice Fault System (AFS) as a case of subsidiary tectonic structure (~10-km-long fault structure) accommodating Quaternary extensional deformation at the hanging wall of the Gorzano-Laga Fault. We describe the AFS in terms of its structural architecture, kinematics, and deformation style. We present geochemical ($\delta^{13}$C and $\delta^{18}$O stable isotope) and U-Th dating on selected mineralization (calcite-filled veins and calcite fibers on fault surfaces) related to the AFS. The results are used to reconstruct the spatial-temporal evolution of the AFS in a complex mechanism of growth and link of isolated segments attained in the Early-Late Pliocene time. A kinematic scenario for the AFS, including the hydrodynamic regime feeding fluid flow at the hanging wall of the Gorzano-Laga Fault, is proposed and framed within the Quaternary-to-active tectonics of the central Apennines.
GEOLOGICAL SETTING

The Central Apennines

The Central Apennines of Italy (Figure 1A) are a fold-and-thrust belt resulted from the Meso-Cenozoic tectonic convergence between the European and African plates (e.g., Dewey et al., 1989; Boccaletti et al., 1990). The orogenic construction involved different paleogeographic domains (oceanic-derived units, carbonate-marly siliceous slope and basinal sequences, carbonate platform sequences, and siliciclastic foredeep sequences; Cosentino et al., 2010) in a general eastward migration of thrust fronts toward the foreland (e.g., Patacca et al., 1990; Sani et al., 2004; Barchi et al., 2012). Since the Pliocene, the chain uplift and its progressive emersion toward subaerial conditions corresponded to a switch from syn-orogenic to post-orogenic phase. The post-orogenic regime started affecting the hinterland (Tyrrenian side) domain of the Apennines, generating NW-SE-striking crustal-scale extensional fault systems (e.g., Malinverno and Ryan, 1986; Dewey, 1988; Keller et al., 1994; Doglioni et al., 1996; Faccenna et al., 1997; Jolivet et al., 1998; Cavinato and De Celles, 1999; Patacca et al., 2008; Barchi and Mirabella, 2009) and related morphostructural basins accommodating sedimentation of marine-to-continental deposits (Patacca et al., 1990; Cavinato and De Celles, 1999; Cipollari et al., 1999; Galadini and Messina, 2001; Martini and Sagi, 1993; Bartole, 1995; Doglioni et al., 1998; Mancini and Cavinato, 2005; Brogi et al., 2014; Cosentino et al., 2017). Structurally, these intermountain basins reside at the hanging wall of regional (tens km-long) NW-SE-striking extensional faults. Deformation, displacement, and basin subsidence are accommodated both along the main basin-bounding faults and along secondary (antithetic or synthetic) collateral faults that are distributed throughout the basins (e.g., Calamita and Pizzi, 1994; Cello et al., 1997; Barchi et al., 2000; D’Agostino et al., 2001; Galadini and Galli, 2003; Boncio et al., 2004a; Galli et al., 2005; Blumetti and Guerrieri, 2007; Galli et al., 2016).

The modes of fault growth include interaction of individual fault segments that evolved both along and across strike in response to a general NE-SW stretching direction for the central Apennines belt, confirmed by geological (e.g., Cowie and Roberts, 2001; Roberts and Michetti, 2004; Iezzi et al., 2019) and geophysical (e.g., Pondrelli et al., 1995; Montone et al., 2012) evidences.

The area hit by the 2016–2017 seismic sequence (Figure 1B) is characterized by a complex network of NW-SE-striking (mainly SW-dipping) extensional faults arranged in both en-echelon and collinear geometry with deformation accommodated by relay zones and step over zones at the fault tips (e.g., Boncio et al., 2004b; Pizzi et al., 2017; Galli et al., 2019). The Vettore Fault and the Gorzano-Laga Fault are part of this complex network of segmented structures. The Vettore Fault includes dominant SW-dipping and minor NE-dipping fault segments for an overall length of ~30 km, and it cuts through a Meso-Cenozoic limestone-dolostone succession with a cumulative vertical displacement of 1.2 km (e.g., Galadini and Galli, 2003; Carminati et al., 2020). The Gorzano-Laga Fault, on the other hand, defines a SW-dipping, high angle extensional structure showing up to 2 km of cumulative displacement (e.g., Bachetti et al., 1990; Blumetti et al., 1993; Galadini and Galli, 2003; Boncio et al., 2004b; Bigi et al., 2013). The Gorzano-Laga Fault is ~30 km-long, including its southeasternmost segment in the area of the Campotosto Lake (Figure 1B). No major (i.e., Mw > 4) historical earthquakes were associated to the Vettore Fault before the 2016–2017 sequence (Galli et al., 2019). On the other hand, the 1639 Amatrice earthquake (Mw 6.0) has been attributed to the seismogenic activity of the northwestern segment of the Gorzano-Laga Fault, based on the analysis of the regional seismic records (Boncio et al., 2004a), macroseismic surveys and geological evidences (Galli et al., 2016).

The 2016–2017 seismic sequence had three mainshocks (24 August 2016: Mw 6.0; 26 October 2016: Mw 5.9; 30 October 2016: Mw 6.5) located over the Norcia Basin and the Amatrice Basin, lying at the hanging wall of the Vettore Fault and the Gorzano-Laga Fault, respectively (Figure 1B). On 18 January 2017, four mainshocks (Mw 5.1, 5.5, 5.4, 5.0) attained in the area of the Campotosto Lake, again at the hanging wall of the Gorzano-Laga Fault (e.g., Tinti et al., 2016; Cheloni et al., 2017; Chiaraluce et al., 2017; Carminati et al., 2020). The estimated subsided hanging wall volume is of ~0.12 km³ (Bigami et al., 2019). The coseismic deformation was heterogeneously distributed along both these faults, with estimated fault length activated of ~30 km and maximum offset of ~2 m along the Vettore Fault, and estimated fault length activated < 2 km and maximum offset < 20 cm for the Gorzano-Laga Fault (Emergeo Working Group, 2016; Lavecchia et al., 2016; Pizzi et al., 2017; Pucci et al., 2017; Givico et al., 2018). The modes and styles how the Vettore and the Gorzano-Laga faults evolved over space and time include bilateral ruptures between them, inducing the progressive lengthening of the surface segments through the propagation of the fault tips (Galli et al., 2016; Tinti et al., 2016; Cheloni et al., 2017; Pizzi et al., 2017; Perouse et al., 2018). At depth, both faults are imaged to merge into a unique tectonic structure that links at seismogenic depths of about 7–9 km (Lavecchia et al., 2016; Chiaraluce et al., 2017), where the localization of deformation are likely conditioned by the occurrence of inherited regional structures (e.g., Bigi et al., 2013; Pizzi et al., 2017; Buttinelli et al., 2018; Falcucci et al., 2018; Bonini et al., 2019).

The Amatrice Basin

A composite fault pattern occurs at the surface within the Amatrice Basin, where contractual and extensional faults are exposed. In particular, the fault pattern exposed in the central part of the Amatrice Basin shows complexity mainly due to superimposition of deformation features related either to contractual or extensional tectonic regimes (Koopman, 1983; Centamore et al., 1991; Cacciuni et al., 1995). Structures running close to the Amatrice village has been either interpreted as intraformational thrusts (e.g., Centamore et al., 1991; Cacciuni et al., 1995) or as a west-dipping extensional fault dissecting the stratigraphic sequence of the Laga Formation (Festa, 1999, 2005; Vignaroli et al., 2019), and possibly related to the activity of the Gorzano-Laga Fault.

The Amatrice Basin is filled by up to 60 m-thick of Quaternary continental units, consisting of a multiple stack of alluvial
fan deposits and terraced fluvial deposits of active channel environment (e.g., Centamore et al., 1991; Cacciuni et al., 1995; Mancini et al., 2019; Vignaroli et al., 2019). Below, the Messinian siliciclastic foredeep deposits of the Laga Formation show a maximum thickness of ~1200 m, as inferred by the stratigraphy of the Varoni01 well and interpretation of seismic
lines (Bigi et al., 2013; Porreca et al., 2018; Mancinelli et al., 2019; Figure 1C). The Laga Formation is organized to form an asymmetric, N-S-trending, syncline showing an overturned, west-dipping, western limb at the footwall of intraformational thrust (e.g., Centamore et al., 1991; Cacciuni et al., 1995; Festa, 2005; Mancini et al., 2019; Vignaroli et al., 2019). Below the Laga Formation, the stratigraphic sequence includes Jurassic-to-Miocene pelagic succession (from top to bottom: Marne ad Orbulina Formation, Marne con Cerroforma formations, Scaglia-to-Corniola formations, Calcare Massiccio Formation) and Triassic dolostones-anhydrites (Figure 1C). The Triassic units result to be doubled by the occurrence of SW-dipping regional thrusts that partially involve the Paleozoic basement (Porreca et al., 2018; Mancinelli et al., 2019). The projected hypocenter of the Mw 6.0 seismic event (Chiaraluce et al., 2017) seem to be located in the deepest portion of the thickened dolostones-evaporites sequence (Buttini et al., 2018; Porreca et al., 2018), above the top of the basement placed at a depth of 9–10 km (Mancinelli et al., 2019).

METHODS AND SAMPLES

The geological-structural investigations were carried out along the trace of the AFS, from Cornillo Vecchio, to the north, to Arafranca-Pinaco, to the south (Figure 2A), with the aim to constrain its brittle structural architecture in terms of spatial distribution, geometry, kinematics, and crosscutting relationships of the deformation structures (fault and fracture systems). Collectively, 87 structural data of faults were collected from 11 sites (Table 1 and Figure 3A). Classical field criteria (e.g., Petit, 1987) such as fault offset, growth fibers, and synthetic shears were used to identify the kinematic shear sense of fault segments. The collected structural data are presented in stereographic projections and in statistical analyses of fault populations (Figures 2B, 3B). Statistical analysis of fault populations (Figure 2B) was done by using the Daisy v.4.1 software (Salvini, 2004). An in-house Monte Carlo convergent routine was used to determine the fault kinematic pattern and the orientation of the principal paleostress tensors (e.g., Angelier, 1984, 1990) responsible for generating the fault population under consideration.

The secondary mineralization related to the tectonic features was systematically sampled for both geochemical and geochronological purposes. Collected samples are represented by (i) the calcite fibers growing on fault surfaces, (ii) the calcite-filled veins that are structurally related to the fault segments, and (iii) the country rock of veins (mostly represented by sandstone-dominated facies of the Laga Formation).

Isotopic analyses (δ¹³C and δ¹⁸O) of selected mineralizations were done to characterize the origin and properties of the parental fluids that circulated throughout the active structural features. We performed carbon and oxygen stable isotope analyses on 75 samples (23 fault fibers, 31 calcite veins, and 21 sandstone host rock) from the AFS (Supplementary Table A1).

Oxygen and carbon isotopes are reported with respect to the Vienna Pee Dee Belemnite standard (V-PDB). Analytical details are reported in Appendix A1.
FIGURE 2 | (A) Geological map of the area encompassing both the Amatrice Fault System and the Gorzano-Laga Fault; (B) stereographic projection (Schmidt net, lower hemisphere) and polymodal Gaussian distribution statistics (cumulative fault strike data, fault dip data, and slickenline pitch values) of the measured extensional faults in the study area reported in Figure 3A. The orientation of the principal paleo-stress tensors after a Monte Carlo-convergent method is reported.
alignment of subparallel fault segments (up to 2 km long) with changing geometry along the strike (see below). Cumulative vertical displacement of the NNW-SSE fault system cannot easily be estimated because of the lack of unambiguous stratigraphic markers at both footwall and hanging wall rock panels. The height of the fault-controlled fluvial escarpments merely helps to make an order-of-magnitude estimate (hundreds of meters) for the vertical displacement.

At Cornillo Vecchio (structural measurement sites 1 and 2 in Figure 3A) and at the confluence between the Tronto River and

| Site of structural measurement | Latitude  | Longitude | Location | Structures | Sample for isotope data (Supplementary Table A1) | Sample for geochronology (Table 2) |
|-------------------------------|-----------|-----------|-----------|------------|-----------------------------------------------|----------------------------------|
| 1                             | 4722256   | 359112    | North of Cornillo Vecchio | (a) NW-SE-striking, SW-dipping extensional fault; (b) SE-dipping bedding | CV1, CV2, CV3mt |
| 2                             | 4721903   | 359166    | South of Cornillo Vecchio | (a) NW-SE-striking, SW-dipping extensional fault; (b) NE-dipping bedding | |
| 3                             | 4721547   | 359287    | Tronto River-Torrente Castellano confluence | (a) NNW-SSE-striking, WSW-dipping extensional fault; (b) W-dipping bedding | Fin8-1ct, Fin8-2ct, Fin8CT1, Fin8CT2, Fin8CT3, Fin8CT4, Fin8bct, Fin8c1ct, Fin8c2ct, Fin8d, Fin8e1ct, Fin8e2ct, Fin8CT5, Fin8E3, Fin8mt, Fin8bmt, Fin8cmt, Fin8emt, Fin8fmt | Fin8 |
| 4                             | 4721450   | 359504    | Amatrice (Lo Scoglio locality) | (a) NNW-SSE-striking, WSW-dipping extensional fault; (b) E-W-striking veins; (c) E-dipping bedding | Fin2ct, Fin1, FIN1CT, F7mt | Fin1 and Fin2 (on NNW-SSE-striking fault) |
| 5                             | 4721351   | 359544    | Amatrice (Lo Scoglio locality) | (a) NNW-SSE-striking, WSW-dipping extensional fault; (b) E-W-striking veins; (c) E-dipping bedding | Fv3-2, Fv3-3, Fv3-4, Fv3-5, Fv3-1, Fv4a-2, Fv4a-1, Fv4b, Fv4-5, Fv4-3, AMTv1, AMTv2, Fv1, Fv2a, Fv2b, Fv3mt, F3mt, F4mt2, F4mt1, F5mt, AMT | Fv1a, FVEN1, FVEN2, FV4b |
| 6                             | 4720717   | 359553    | Right side of Torrente Castellano | (a) E-W-striking, N-dipping extensional fault; (b) E- and W-dipping bedding | | |
| 7                             | 4720460   | 360181    | South of Amatrice | (a) E-W-striking, S-dipping extensional fault; (b) NW-dipping bedding | | |
| 8                             | 4720366   | 359797    | Left side of Torrente Castellano | (a) NNW-SSE-striking, WSW-dipping extensional fault; (b) ENE-dipping bedding | | |
| 9                             | 4719162   | 359797    | North of Arafranca Pinaco | (a) N-S-striking, W-dipping extensional fault; (b) E-W-striking, subvertical veins; (c) NW-dipping bedding | AR3a, AR3b, AMTCT2, AR3 |
| 10                            | 4718936   | 359870    | Arafranca Pinaco | (a) NW-SE-striking, SW-dipping extensional fault; (b) NWN-SSW-striking, SSW-dipping right strike-slip fault; (c) E-dipping normal bedding | AR2, AR7ct, AR2CT1, AR2CT2, AMTCT, AR2ct3, AR2ct4, AR7ct1, AR7ct2, AR2ct5, AR7mt | AR2 |
| 11                            | 4718682   | 360024    | Arafranca Pinaco | (a) NW-SE-striking, SW-dipping extensional fault; (b) NE-WW-striking veins; (c) NE-dipping bedding | AR1a, AR1b, AR4ct, AR4mt, AR5mt, AR6mt | AR1 |
FIGURE 3 | (A) Structural map of the Amatrice Fault System, from Cornillo Vecchio (to the north) to Arafranca-Pinaco (to the south), with structural measurement sites shown with numbers within circles (see Table 1). Location of samples used for U-Th dating is also reported; (B) stereographic projections (Schmidt net, lower hemisphere) of the collected structural data.
| Sample | Spectrometry | Weight g | Age (kyr ago) | Age corrected (1) | Age corrected (2) |
|--------|--------------|----------|---------------|-------------------|-------------------|
| Vein 1 | Vein 2       | 14.64    | 108 ± 10      | 108 ± 10          | 108 ± 10          |
| Vein 2 | Vein 3       | 23.7 ± 3.7 | 118 ± 6.4    | 118 ± 6.4        | 118 ± 6.4        |
| Vein 3 | Vein 4       | 23.6 ± 1.8 | 119 ± 12     | 119 ± 12         | 119 ± 12         |
| Vein 4 | Vein 5       | 23.4 ± 1.5 | 120 ± 19     | 120 ± 19         | 120 ± 19         |
| Vein 5 | Vein 6       | 24.2 ± 0.6 | 120 ± 19     | 120 ± 19         | 120 ± 19         |
| Vein 6 | Vein 7       | 24.1 ± 0.5 | 120 ± 19     | 120 ± 19         | 120 ± 19         |
| Vein 7 | Vein 8       | 24.0 ± 0.4 | 120 ± 19     | 120 ± 19         | 120 ± 19         |
| Vein 8 | Vein 9       | 24.0 ± 0.3 | 120 ± 19     | 120 ± 19         | 120 ± 19         |
| Vein 9 | Vein 10      | 24.0 ± 0.2 | 120 ± 19     | 120 ± 19         | 120 ± 19         |

### Sample Structure Weights

| Sample | Structure | Weight g |
|--------|-----------|----------|
| Vein 1 | Vein 2    | 14.64    |
| Vein 2 | Vein 3    | 14.64    |
| Vein 3 | Vein 4    | 14.64    |
| Vein 4 | Vein 5    | 14.64    |
| Vein 5 | Vein 6    | 14.64    |
| Vein 6 | Vein 7    | 14.64    |
| Vein 7 | Vein 8    | 14.64    |
| Vein 8 | Vein 9    | 14.64    |
| Vein 9 | Vein 10   | 14.64    |

**Table 2**: U-Th ages of the cable mineralizations (faut fibers and calcite-filled veins) sampled along the Amatrice Fault System.
FIGURE 4 | Northernmost field exposure of the Amatrice Fault System (the approximate location of selected outcrops is shown in the insert). (a) Northern exposure of the NNW-SSE-striking fault surfaces at Cornillo Vecchio village, where the rock panels at the footwall of the fault have been used as foundation by presently damaged masonries; (b) further exposure of the NNW-SSE-striking fault system showing the geometrical cross-cutting relationships between fault surface and bedding. In the inset: details of calcite fibers on the fault surface; (c) exposure of the NNW-SSE-striking fault system at the northern ridge of the Amatrice village, showing sub-parallel, W-dipping fault surfaces; (d,e) details of abrasive striations and calcite fibers on the fault surface documenting major dip-slip movement. Samples used for U-Th dating are represented by black hexagon. LAGa: sandstone-dominated lithofacies of the Laga Formation.

strands. Secondary faults host calcite fibers with pitch values less than 20° (see insert in Figure 6b), which is indicative of strike-slip kinematics.

Kinematic criteria, expressed by stratigraphic offset, synthetic shears, dragging of the bedding at the fault surfaces, and geometrical relationships between the faulting-related foliation and the bedding, indicate normal-sense movement for the NNW-SSE-striking faults.

E-W-Striking Structures
The E-W fault system (structural measurement sites 6 and 7 in Figure 3A and relative stereographic projections in Figure 3B) consists of hundred-meter-long fault segments dipping both to the north and south. The E-W fault structures are less mature with respect to the NNW-SSE-striking faults, showing reduced offset (on the order of tens of meters) and persistence. In map view, these E-W-striking structures are recurrent in the central part of the AFS, where they control escarpment morphologies oriented perpendicular to the Torrente Castellano. Structurally, they define transverse faults that interrupt and dislocate the NNW-SSE-striking faults. At the mesoscale, the E-W-striking faults are half-meter spaced and show rough planar slip surfaces cutting the bedding at high angles (Figure 7a). Well-developed E-W fault surfaces hosting striations and kinematic indicators are rarely exposed. When occurring, slickenlines are provided by abrasive striae showing pitch values generally around 80° and
FIGURE 5 | Central field exposure of the Amatrice Fault System (the approximate location of selected outcrops is shown in the insert). (a) NNW-SSE-striking fault system showing sub-parallel, W-dipping fault surfaces cutting through the sandstone-dominated lithofacies of the Laga Formation (LAGa); (b,c) details of abrasive striations and calcite fibers on the fault surface documenting major dip-slip movement; (d) further exposure of the NNW-SSE-striking fault system at the Amatrice village, showing half-meter spaced fault surfaces inducing drag of the bedding; (e) geometrical relationship between the fault surface and the E-dipping bedding; (f) detail of faulting-related foliation, which is sub-parallel to the fault surface, and the bedding, the latter provided by change in matrix color and grain size; (g) exposure of the stratigraphic boundary between the conglomerates (belonging to the Amatrice-Sommati Unit; UASc) and the underlying LAGa along the fault trace. The stratigraphic boundary does not show evidence for faulting-related displacement. Sample used for U-Th dating is represented by black hexagon. LAGa: sandstone-dominated lithofacies of the Laga Formation; UASc: Amatrice-Sommati Unit, conglomerate facies.
100° (see stereographic projection in Figure 7a). Observed shear criteria, such as synthetic shear fractures and stratigraphic offset, indicate normal-sense movement for the E-W-striking faults.

One of the most important features of the E-W fault system is the occurrence of a systematic fracture network (structural measurement sites 4, 5, 9, and 11 in Figure 3A and relative stereographic projections in Figure 3B) that consists of sub-vertical, planar discontinuities showing pluri-decameter persistence. The fracture spacing systematically decreases from decametric to half-meter upon approaching the main slip surfaces (e.g., at structural measurement sites 4 and 5 in Figure 3A). Locally, E-W-striking fractures are filled by calcite agglomerate and are therefore classified as veins (Figures 7b–e). Veins are up to 2 cm thick and have sharp boundaries with the surrounding sandstones. Texturally, the filling calcite forms either a blocky texture, a coarse-grained agglomerate (up to 3–4 mm in size, see Figure 7f), or a fine-grained (submillimeter) matrix (Figure 7g).

CARBON AND OXYGEN ISOTOPES

All analyzed samples are characterized by negative values of both δ¹³C (between −10.26‰ and −0.29‰) and δ¹⁸O (between −7.28 and −3.09‰) (Supplementary Table A1 and Figure 8). Grouping the data according to sampling location produces no evidence of isotopic differentiation, whereas such evidence does appear upon grouping the data according to the different calcite deposits (host rock, veins, and fault fibers). Figure 8 shows samples using this distinction: four samples of calcite
FIGURE 7 | (a) Exposure (the approximate location of selected outcrops is shown in the insert) of the E-W- striking fault system, south of the Amatrice town, and consisting of half-meter spaced, N-dipping fault surfaces dissecting the LAGa lithofacies. See the stereographic projections for attitude of the measured structural elements; (b) traces (dotted lines) of subvertical veins cross-cutting the LAGa lithofacies at Lo Scoglio locality; (c) close-up on the vein filled by coarse-grained massive calcite; (d,e) exposures of E-W-striking veins filled by fine-grained calcite and cross-cutting the LAGa lithofacies north of Arafranca Pinaco; (f,g) thin sections (crossed polarizers) of the coarse-grained and fine-grained textures, respectively, in calcite veins. Samples used for U-Th dating are represented by black hexagon on outcrop pictures. LAGa: sandstone-dominated lithofacies of the Laga Formation.
fibers on fault surfaces are displayed out of the relevant group because they show clear surficial alteration. Multivariate analysis of variance (MANOVA) for the isotopic variables produces a significant difference (Wilks’ Λ = 0.524, p < 0.001), and the distinct populations of calcite types can be recognized. Samples from the sandstone host rock are characterized by δ13C values between −3.88 and −0.29‰ and δ18O values between −5.38 and −3.79‰. Samples from the calcite-filled veins are characterized by δ13C values between −5.17 and −0.81‰ and δ18O values between −6.59 and −3.89‰. In fact, the carbon isotopic compositions have a more restricted range from −2.58 to −0.81‰ if we do not consider the most negative value. Samples from fault fibers are characterized by δ13C values between −6.32 and −1.55‰ and δ18O values between −7.28 and −4.86‰. The fault fibers showing an altered aspect are characterized by δ13C values between −6.27 and −10.26‰ and δ18O values between −7.08 and −5.87‰.

The isotopic compositions of the carbonate fraction of the host rock are in the range of marine carbonate rocks, despite the average of the oxygen isotopes (−4.4‰, 0.43‰ StdDev) being about two per mill lower than the typical values for most Meso-Cenozoic marine limestones and dolostones in central Italy (Ghisetti et al., 2001). At the thin section scale, carbonate cement as pore-filling is almost absent. The isotopic shift to slightly lower values in δ18O may be due to submillimeter microfractures filled with calcites that, in some cases, may also account for the episodic shift of carbon isotopes toward lower values (down to −3.9‰).

Overall, C- and O-isotope data of newly formed calcite show that the precipitation fluid along the AFS was likely of meteoric origin for both fibers and veins. Carbon isotopes provide evidence of an important contribution of soil CO2 to the carbon species in solution during fault-fiber formation (NNW-SSE-striking structures) with respect to the calcite-filled vein (E-W-striking structures) because the remarkable C-isotope decrease can be ascribed to organic carbon incorporation into the calcite fiber crystal lattice. Oxygen isotopes show that meteoric water may have constituted the dominant oxygen reservoir to which both fault fibers and calcite-filled veins re-equilibrated after dissolution of the host-rock carbonate fraction. There are no evident dissolution structures in the host-rock, but also the development of the fault-vein conduit network in the host-rock seems to be extremely limited. The distribution of isotope data in the δ18O/δ13C diagram seems to indicate that the calcite fibers evolved just from the host-rock isotope composition toward lower values as a function of variable carbon and oxygen molar W/R ratios (Sverjensky, 1981; Rye and Bradbury, 1988).

A different evolution may be envisaged for vein calcites whose carbon isotopes do not evince the presence of an organic component, and oxygen-isotope composition exceeds that of the host rock in some instances. The isotope distribution of vein calcites seems to indicate precipitation from a meteoric-derived groundwater fluid whose provenance may even be external to the host rock; namely, from isotopically unaltered carbonate rocks.

**U-Th GEOCHRONOLOGY**

Three samples of calcite-filled vein from the E-W-striking fault collected near the Amatrice village were dated by α spectrometry (Fven1A; Figure 7d) and by the MC-ICP-MS method (Fven1 and Fven2; Figures 7c,d). Sample Fven1A is characterized by a moderate detrital contamination (230Th/232-Th activity ratio = 24.2 ± 3.6) due to a moderate acid leaching (see Appendix A2), whereas samples Fven1 and Fven2 are affected by a severe contamination due to dissolution of a non-carbonatic component attributable to the strong acid attack. Both samples are characterized by lower 230Th/232-Th activity ratio (5.82 ± 0.15 and 1.80 ± 0.06, respectively). By assuming a detrital component at secular equilibrium, with the crustal 232Th/238U value of 3.8, we obtained a corrected age of 118.6 ± 6.4 ka for sample Fven1A and a corrected age of 251 ± 47 ka for sample Fven2, whereas age older than the range of the method (≥800 ka) is obtained for sample Fven1. Eventually, considering these three Fven samples as coeval, if we plot the 230Th/232Th and 234U/232Th activity ratios against their respective 238U/232Th activity ratios (method L/L of Schwarcz and Latham, 1989), we obtain a corrected age of 108 ± 10 ka.

The other samples dated through the MC-ICP-MS method give older ages. In particular, the oldest age obtained is from the calcite fibers from NNW-SSE-striking fault surfaces (355 ± 57 ka; sample Fin8; Figure 4b). A similar age (348 ± 46 ka) is obtained from a calcite-filled vein from the E-W-striking fault collected near the village of Arafanca-Pinaco (sample AR1; Figure 7e). Moreover, if we assume a crustal detritic component in radioactive equilibrium, three samples belonging to both types of calcite mineralization are older than the range of the method (≥800 ka). However, the present δ234U values that are significantly greater than zero (at a significance level > 6σ) constrain
their mineralization ages to less than about 1250 ka, which is the time required to obtain secular equilibrium in the $^{234}$U/$^{238}$U activity ratio.

**DISCUSSION**

**Tectonic Synthesis**

Our multidisciplinary dataset is used to address the spatial-temporal evolution of the AFS within the tectono-stratigraphic setting of the Amatrice Basin.

(1) The internal structural architecture of the AFS includes kilometer-long extensional fault segments oriented NNW-SSE and accompanied by subsidiary extensional fault segments oriented E-W. Structural complexities reside in the style and modalities of link and interaction at the fault terminations, including fault overlaps linked by relay zones, collateral faults, and fault segmentation by transverse structures. The recognized fault array for the AFS can be framed within a kinematic scenario of fault growth and propagation under an ENE-WSW-directed crustal stretching (Figure 9A). The AFS developed over time by mechanisms that are commonly related to fault population, including radial propagation of isolated fault segments, fault overlapping, and fault linkage (Morley et al., 1990; Cartwright et al., 1995; Peacock and Parfitt, 2002; Hus et al., 2006; Fossen and Rotevatn, 2016; Figure 9B). In this scenario, the E-W-striking system represents the last structural step of the AFS, suggesting conditions of local stress perturbation that induced a change in deformation style (from longitudinal to transverse faults) during the final stage of the AFS growth. This kinematic scenario offers a comparison term for the growth of the Quaternary-to-active faults of the central Apennines (see below).

(2) The analysis of the isotopic data suggests a meteoric water ingress within tectonic discontinuities to crystallize calcite as fibers on fault surfaces. According to the negative values of C-isotopic data, a significant contribution of organic carbon (soil CO$_2$) was incorporated into the calcite fibers on fault surfaces, suggesting an open circulation connected to the vadose zone. Conversely, the dominant carbon reservoir for calcite in veins is represented by the host rocks or an adjacent carbonate rock, whose dissolution provided the carbonate for calcite-vein precipitation. The variability presented by the oxygen isotopes in the veins results from the variable oxygen molar W/R ratios experienced by each carbon species in solution during its path to the deposition sites. The calcite closest to the host-rock values likely precipitated near the place where dissolution occurred. In any case, carbon from the soil zone did not enter the vein system, as if the vein openings were sealed against the vertical infiltration that characterizes the circulation through fault surfaces. Therefore, we propose that the change in deformation style (from longitudinal to transverse faults) corresponds to a change in the fluid pattern. First, a hydraulic system open to vadose-water influx percolated along the NNW-SSE-striking system. Next, a hydraulic system of fluid circulation, closed to vertical influx, precipitated calcite within the E-W-striking system after dissolution from the surrounding matrix or from adjacent carbonate rocks (Figure 9B). These propositions require the assumption of nearly constant temperature (close to the surface temperature) during calcite deposition, which seems to be a reasonable assumption if we consider the recent development of the fault system and the remarkable contribution of soil CO$_2$ as a possible source of organic carbon for the fibers on the fault surfaces.

(3) The U-Th ages allow dating of the syn-tectonic mineralization along the AFS and this represents the first geochronological record provided for the Amatrice Basin. Apart from the samples that are older than the range of the U-Th method (>800 ka), both fault fibers and calcite-filled veins gave ages spanning from ∼350 to 108 ka. These ages constrain the main phases of the tectonic activity of the AFS to a period spanning from the late Early Pleistocene to the early Late Pleistocene. Therefore, we infer the AFS accumulated deformation and most of its displacement during the Pleistocene, dissecting the stratigraphic sequence of the Laga Formation. The cross-cutting relationships between the dated structures and the overlying continental deposits show that NNW-SSE-striking faults do not cross through the conglomerates and cobbles (Figure 5g). Similar observations along other sections of the AFS (Mancini et al., 2019; Vignaroli et al., 2019) suggest that the deposition of the continental deposits filling in the Amatrice Basin post-dated the structures we dated at the early Late Pleistocene. Anyway, we cannot exclude the possibility that the activation of younger structures within the AFS (i.e., the veins dated to the Late Pleistocene) may have involved the overlying sediments by producing not appreciable deformation at the mesoscale. In addition, the diachronic deposition of the continental deposits cannot be excluded over the Amatrice Basin, being influenced by the hydrodynamics of local alluvial drainages (e.g., Cacciuni et al., 1995; Mancini et al., 2019). Finally, we aware that our ages do not preclude the fact the AFS (or its segments) reactivated during the Holocene, in consequence to the seismogenic activity of the adjacent Gorzano-Laga Fault (e.g., Galli et al., 2016). Further geochronological constraints and/or geomorphic analyses could be addressed to elucidate possible minor activations of the AFS connected to the seismic activity in the Amatrice Basin.

To summarize, our results document that tectonic deformation and surficial fluid circulation were active in the Amatrice Basin during the Pleistocene. The hydrodynamic interconnection with the vadose zone suggests the AFS acted as a growing fault structure that produced ruptures at the surface. Meteoric waters infiltrated downward into the tectonic cracks and were channelized through structural conduits within the rock volume, compatible with a scenario of tectonically
FIGURE 9 | (A) Reconstructed fault array for the AFS in the area between Cornillo Vecchio and Arafranca Pinaco. While NW-SE-striking isolated faults prevail at the central portion of the AFS, its northernmost and southernmost tips are characterized by overlapping geometries and relay zones. Black arrows represent the general stretching direction; (B) three-stage cartoon for the structural evolution of the AFS according to fault cross-cutting relationships and absolute ages provided by U-Th dating. This kinematic scenario includes the pattern of fluid circulation during faulting, as likely deduced from the isotopic analysis (see text for details); (C) possible hydrological scenario for the study area during the Pleistocene fault activity. Schematic C-O diagrams in gray boxes illustrate the isotopic signature and evolutive trend of the circulating fluid.
maintained vertical permeability (e.g., Sibson, 2000; Cox et al., 2001; Rowland and Sibson, 2004). Similar scenarios of fluid-assisted tectonic deformation in the central Apennines have been documented in faulted carbonate rocks (e.g., Maiorani et al., 1992; Conti et al., 2001; Ghisetti et al., 2001; Agosta et al., 2008; Smeraglia et al., 2016, 2018), suggesting the interplay between transient permeability at the fault damage zones and mixing of fluids from different reservoirs (meteoric, groundwater, subcrustal). Notably, the AFS provide a case history of fluid-rock interaction in siliciclastic units affecting by extensional faulting. Fluids permeating the AFS preserve the geochemical signature of cold water circulation at shallow depths, with the dominant meteoric water that progressively changes to fluids in isotopic equilibrium with the surrounding siliciclastic host rock. Overall, two options are here proposed to model the fluid-rock interaction along the AFS in the Early-Late Pleistocene. The first model invokes a dominant infiltration of meteoric fluids (the open hydraulic system) and progressive (over time) mixing with groundwater that has exchanged isotopically with carbonates, which plays a dominant role in the development of the veins (Figure 9C). In this scenario, the isotopic transition correlates with the fluid flow that moved throughout the host rock adjacent to the fault zones (e.g., Pili et al., 2002). This scenario is in line with the general considerations regarding the calcite deposits during fluid flow in diverse fault systems from the central Apennines (Ghisetti et al., 2001; Agosta et al., 2008). Given the surficial conditions under which the AFS developed, the lithological control on the fluid composition could be more important than the advective transport of meteoric waters in the final stage of the Pleistocene fault activity. Possibly, NNW-SSE-striking faults of the AFS led to a structural permeability to azimuthal infiltration of meteoric water (soil CO₂ contributes to C species in solution) greater than that produced by the E-W-striking structures. The second model invokes a discontinuous fluid history involving a two-step development of newly formed calcites: initially, an open system meteoric circulation developed, mainly driven by a vertical infiltration from the vadose soil zone through the fault surfaces; this was a localized flow along the extensional fault segments oriented NNW-SSE. The subsequent formation of the E-W subsidiary fault segments promoted a horizontal fluid flow, likely originating from a lateral water reservoir; it was closed to the former meteoric circulation and, consequently, to contributions of carbonatic matter from the soil zone.

Implication for the Quaternary-to-Active Extensional Faulting in Central Apennines

The reconstructed structural pattern of the AFS can be framed within the post-orogenic regional extensional tectonic regime described for the axial domain of the central Apennines, where the main NW-SE-striking extensional faults (i.e., oriented parallel to the belt axis) accommodated a crustal-scale extensional regime and the onset of continental sedimentation in intermountain basins (e.g., Malinverno and Ryan, 1986; Cavinato and De Celles, 1999; Cavinato et al., 2002; Giaccio et al., 2012; Mancini et al., 2012; Pucci et al., 2014; Cosentino et al., 2017). In this scenario, the AFS defines a minor tectonic structure resembling (in terms of fault architecture, geometries, and kinematics) the adjacent Gorzano-Laga Fault, which, in turn, represents the basin-bounding master fault that localized up to 2 km of displacement during its activity (e.g., Bachetti et al., 1990; Boncio et al., 2004b). Overall, the evolution of both the AFS and the Gorzano-Laga Fault is compatible with the general NE-SW stretching direction for the central Apennines belt (e.g., Montone et al., 2012). This is inferred from the orientation of the σ₃ (minimum compression) obtained by fault-slip analysis for the AFS (Figure 2B) compared with the direction of the active stress tensor (N74°-trending σ₃ axis) computed for the Gorzano-Laga Fault by inversion of the focal mechanisms (Bonicio et al., 2004b). Therefore, by linking the Pleistocene tectonic activity of the AFS and the still-ongoing activity of the Gorzano-Laga Fault, it is proposed a self-similar paleostress regime for the area of the Amatrice Basin during the Quaternary.

The structural evolution we illustrated for the AFS reinforces the general scenario of fault growth proposed for the Quaternary-to-active faults of the central Apennines, in which faults nucleated as isolated segments able to modulate their along-strike throws and displacements during propagation and interaction at the fault tips (e.g., Cello et al., 1997; Cowie and Roberts, 2001; Roberts and Michetti, 2004; Faure Walker et al., 2010; Lavecchia et al., 2016; Pizzi et al., 2017; Civico et al., 2018; Iezzi et al., 2018, 2019; Villani et al., 2018; Galli et al., 2019). In particular, the study of partial and total coseismic ruptures occurring along master extensional faults suggests a propagating fault model (Rotevatn et al., 2019 and references therein) characterized by variable displacement accrual during fault lengthening. The structural evolution we proposed for the AFS (Figures 9A,B) can add some insights on the tectonic activity at the hanging wall of the master extensional faults. The AFS fault architecture suggests a two-stage mechanism of fault growth. A first stage of dominant lengthening and displacement accumulation during formation of longitudinal (NNW-SSE-striking) faults is followed by a second stage of limited longitudinal fault propagation, fault overlapping and local development of transverse (E-W-striking) structures. The switch from longitudinal to transversal fault development, as constrained by both structural field observation and U-Th ages of tectonic-related mineralizations, corroborates the occurrence of complex patterns of minor fault systems accommodating the overall tectonic deformation. Similar patterns of longitudinal and transverse faults accommodating different stretching rates within the extending crust have been already documented elsewhere in central Apennines, in which transverse structures (showing strike-slip to extensional kinematics) seem to develop at the end-of-life lengthening of the longitudinal (NW-SE-striking) faults (Faccenna et al., 1994; Aiello et al., 2000; Acocella and Funicelli, 2006; Liotta et al., 2015; Bucci et al., 2016; Vignaroli et al., 2016).

CONCLUSION

The Amatrice Fault System (AFS) provides a promising target to capture snapshots of the Pleistocene, fluid-assisted, tectonic
deformation in the Amatrice Basin, which is a seismically active domain of the central Apennines. The AFS can be considered a minor structure that accommodated tectonic deformation at the hanging wall of a master extensional fault, the Gorzano-Laga Fault.

Through new U-Th ages and a reconstructed scenario of fault growth and structurally controlled fluid circulation, we propose the AFS nucleated and evolved through radial propagation of isolated longitudinal faults followed by linkage at the fault terminations (including the activation of transverse faults) in a period spanning from the late Early Pleistocene to the early Late Pleistocene, although minor reactivations during the Holocene cannot be excluded a priori. The fault pattern created a transient structural permeability, possibly developed in coseismic conditions, to the azimuthal infiltration of meteoric waters that progressively equilibrated with the geochemical imprint of the siliciclastic host rock. We conclude that the AFS is an interesting topic to be studied for a better understanding of the processes occurring at the hanging wall of a master fault, such as the spatial-temporal fault growth and the hydrodynamic regime characterizing the fluid flow within the structural permeability network. The study of minor tectonic structures and their evolution may represent an interesting perspective in elucidating the relation between the localized deformation along the master faults and the long-term tectonic structures lying at their hanging wall. This approach is expected to be relevant for the assessment of the style and the modality of crustal-scale deformation accommodated in extensional settings that release tectomogenic potential.

**DATA AVAILABILITY STATEMENT**

All datasets generated for this study are included in the article/Supplementary Material.

**AUTHOR CONTRIBUTIONS**

GV planned the structural work and the samples collection. MM, FB, MC, and MB participated at the fieldwork. GV wrote the manuscript, with significant contributions by all the authors. GV drew all the figures with contributions by all the authors. MB and FG performed stable isotope analyses. MV performed geochronological analyses through α spectrometry. T-LY and C-CS performed geochronological analyses through multi-collector inductively coupled mass spectrometer. All the authors contributed to the data processing, result discussion, and final interpretation, reviewed the manuscript, and approved its submission to Frontiers in Earth Sciences – Structural Geology and Tectonics.

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**SUPPLEMENTARY MATERIAL**

The Supplementary Material for this article can be found online at: https://www.frontiersin.org/articles/10.3389/feart.2020.00130/full#supplementary-material

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**Conflict of Interest:** The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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APPENDIX A

(A1) C- and O-Isotope Determination
The samples were analyzed for oxygen and carbon stable isotopes; this analysis was performed in the Mass Spectrometry Lab of Istituto di Geologia Ambientale e Geoingegneria of the Italian Research National Council (Rome, Italy), by means of the usual acid digestion technique at 72°C using a Thermo Gasbench II on line with a Delta + mass spectrometer. The oxygen and carbon isotopic compositions were expressed in the usual delta notation ($\delta^{18}$O and $\delta^{13}$C), which represents the relative deviation in parts per thousand of the heavy isotope/light isotope ratio of the samples from that of an international reference standard (V-PDB). Approximately 0.15 mg of powder were weighted in duplicate and measured along with 0.15 mg of three standards to normalize the raw $\delta^{18}$O and $\delta^{13}$C values to the V-PDB scale: MC-200, CaCO3 (Merck CCM) and Solnhofen limestone (SLNF) (calibrated against references NBS18 and NBS19). The data obtained were normalized by a linear calibration equation derived from a plot of accepted versus measured values for the three aforementioned internal standards.

(A2) U-Th Dating
As reported in Table 2, we performed U-Th dating analyses at two laboratories, (1) through $\alpha$ spectrometry performed at the Laboratorio di Geochimica Ambientale of the Istituto di Geologia Ambientale e Geoingegneria, CNR, and (2) through a Thermo Electron Neptune MC-ICP-MS (Shen et al., 2012) hosted at the HISPEC, National Taiwan University. For $\alpha$-spectrometry dating, after crushing the sample Fven1, we selected 15 g of the calcite-filled vein and after treatment for 1 day with H$_2$O$_2$ they were dissolved with an excess of 1N HNO$_3$. After dissolution we added to the acid nitric solution a known amount of $^{232}$U spike in secular equilibrium with $^{228}$Th and 3 drops of FeCl$_3$. After 1 day the pH of solution was adjusted to pH = 3 by adding drops of concentrated NH$_4$OH and the Fe(OH)$_3$ precipitate, containing U and Th, was separated from solution. The precipitate was redissolved by HCl and warmed up to dryness. Then the sample was dissolved in 10 N HCl and uranium was separated from thorium by passing the acid solution through a 10 N HCl-conditioned anionic resin (Fluka Dowex 1X-8) column. Then filtrate (containing Th) and leachate by 0.1 N HCl (containing U) were dried and redissolved with 7 N HNO$_3$. Two 7 N HNO$_3$-conditioned anionic resin columns were used to purify U (subsolution 1) and Th (subsolution 2). Finally, U and Th were separately extracted at different pH with TTA and the organic layer of each subsolution was separated and deposed in warming conditions over stainless disks up to 500°C and counted by alpha spectrometry.

For MC-ICP-MS dating, we covered about 0.05 g of each sample with H$_2$O and dissolved it gradually with double distilled 14 N HNO$_3$. After dissolution, we added a $^{229}$Th–$^{233}$U–$^{236}$U spike (Shen et al., 2003) to the sample, followed by 10 drops of HClO$_4$ to clear the organic matter. We followed the chemical procedure described in Shen et al. (2003) for the separation of uranium and thorium. Age correction was calculated using an estimated atomic $^{230}$Th/$^{232}$Th ratio of $4 \pm 2 \times 10^{-6}$. The value is the one typical for a material at secular equilibrium with the crustal $^{232}$Th/$^{238}$U value of 3.8 and an arbitrarily assumed 50% error. Half-lives of U-Th nuclides used are given in Cheng et al. (2013).

Age calculation was earned by using ISOPLOT, a plotting and regression program for radiogenic-isotope data (Ludwig, 2003).