Miocene age of the Huanan basalt lava flow (NE China) inferred by reset of zircon (U–Th)/He thermochronometer in the underlying sand

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Mafic lavas of Cenozoic age are widely distributed in northeast China and received much attention as an important part of the Circum-Pacific volcanic belt. The age constraints for the volcanic activity were determined mostly by K/Ar and 40Ar/39Ar methods. We present zircon (U–Th)/He ages obtained on the thermally overprinted sands directly underlying a basaltic lava. This thermochronometer is insensitive to weathering and not biased by excess argon, thus it can express accurately the age of thermal effect of the lava flow. As a regional cooling age reference, three granite samples were dated from basement units that have not been thermally influenced by the basalt eruptions. The reference granite samples revealed well-defined Cretaceous (U–Th)/He-ages, while 20 zircon crystals from the sand below the basalt lava revealed a prominent Miocene (U–Th)/He age component of 9.33 ± 0.24 Ma. Raman spectroscopy of these zircon crystals supports their thermally overprinted character. We infer that the sand sample has experienced significant thermal overprint by the overlying basalt lava leading to thermal reset of the majority of the detrital zircon crystals. The obtained age is thus interpreted as the eruption age of the basalt lava. The Huanan basalt flow thus belongs to volcanics of the Laoyeling episode in NE China.

KEYWORDS
(U–Th)/He, basaltic lava, Huanan, Miocene, NE China, Raman
Mafic lavas of Cenozoic age are widely distributed in northeast China. Despite the small size of these occurrences, they represent an important part of the Circum-Pacific volcanic belt (Basu, Wang, Huang, Xie, & Mitsunobu, 1991; Flower, Tamaki, & Hoang, 1998; Zou, Fan, & Yao, 2008; Xu et al., 2015). The age constraints of this volcanic activity were determined mostly by K/Ar and 40Ar/39Ar methods and range from Miocene to Pleistocene except for some Late Cretaceous to Paleogene ages within and east of the Songliao Basin (Figure 1; Fan, Sun, Li, & Wang, 2006; Fan et al., 2011; Fan, Zhao, Li, & Wu, 2012; Liu, 1987; Liu, Chen, Zhong, Lin, & Wang, 2017; Liu et al., 2017; Qiu, Liao, & Liu, 1991; and Zhang, Xu, & Ma, 2006). The digital elevation model is from the U.S. Geological Survey, 2017. The two faults marked with (1) and (2) are the Jiamusi–Yitong and Dunhua–Mishan faults, respectively, and belong to the eastward extension of the Tan–Lu Fault Zone in NE China. Dashed box indicates position of Figure 2. (b) Schematic tectonic map of North Asia (modified after Liu, Chen, et al., 2017; Liu, Li, et al., 2017) [Colour figure can be viewed at wileyonlinelibrary.com]

In the last two decades, new geochronological techniques were introduced for dating young mafic eruptions such as the U–Th disequilibrium method (Zou, Zindler, Xu, & Qi, 2000), indirect dating of volcanics from the surrounding fallout organic material deposits by the 14C method (Xu, Zhang, Qiu, Ge, & Wu, 2012; Yin et al., 2012), fission track dating of volcanic glasses (Renne, 2000), and magnetite or zircon (U–Th)/He (ZH) geo-thermochronology (e.g., Blackburn, Stocchi, & Walker, 2007; Blondes, Reiners, Edwards, & Biscontini, 2007; Cooper, van Soest, & Hodges, 2011; Farley, 2002). The modern 40Ar/39Ar approaches may yield precise ages of young volcanic rocks, but typically the age of young and/or low-K lava samples have high errors due to minor proportions of radiogenic Ar (Blondes et al., 2007; McDougall & Harrison, 1999). The magnetite (U–Th)/He method is
also introduced to date mafic volcanic rocks (Blackburn et al., 2007; Fanale & Kulp, 1962). However, this mineral is not suitable for a wide range of applications due to its disadvantages. For example, (a) the Fe-oxide minerals in mafic volcanic formations have frequently irregular external morphology, thus the ejection (FT) correction is hardly feasible and it would generate significant bias (Hernandez Goldstein, Stockli, Ketcham, & Seman, 2014). (b) The interior of the magnetite grains in lavas are highly heterogeneous, often penetrated by ilmenite and haematite lamellae and they contain apatite inclusions. (c) The U content is usually very low. The studies for example, Fanale and Kulp (1962) and Blackburn et al. (2007) were dealing with pre-Cenozoic ages, with a few ppm or even sub-ppm uranium content. In the case of Miocene–Pliocene lavas the uncertainties would be much more over the expectations for stratigraphical purposes. Furthermore, the Blackburn et al. (2007) study was made on kimberlites, which has atypical actinide contents and distributions.

Zircon analysis has been proven a versatile tool for examining a wide range of geological processes because zircon crystals have a lot of important features for geochronology and thermochronology including high actinide concentrations, occurrence in variable lithologies and resistance to physical and chemical weathering (Reiners, 2005). Like many other minerals, zircon can also be dated by the (U–Th)/He method to reveal the low temperature (~180–130 °C) thermal history (e.g., Farley, 2002; Reiners, Spell, Nicolasu, & Zanetti, 2004). Comparing to the K/Ar and 40Ar/39Ar methods, zircon (U–Th)/He method has the advantage of performing relatively rapidly on selected zircon crystals without neutron irradiation, and high accuracy on young volcanic rocks (Blondes et al., 2007). Even though mafic to intermediate volcanic rocks rarely contain zircon crystals, the strata below lava flows or the host rocks in contact with basaltic dykes, sills, or necks are often rich in zircon crystals. These zircons may become thermally reset upon significant heating (temperature and time) and the (U–Th)/He age obtained on these crystals then indicates cooling after the heating event. Assuming usual fast cooling of lava flows, this age should reflect the eruption age (Blondes et al., 2007; Cooper et al., 2011).

In this study, we report for first time zircon (U–Th)/He ages from a thermally overprinted basal layer of a lava flow from the Huan region in NE China. Additionally, Raman spectroscopy was used to describe the crystalline state and confirm the thermal reset of the dated zircon crystals.

2 | GEOLOGICAL SETTING

NE China is enclosed by the Siberian Block in the north, the North China Block in the south and the Pacific Plate in the east, tectonically situating in the eastern segment of the world’s largest accretionary orogen, the Central Asian Orogenic Belt (CAOB: Jahn et al., 2000; Sengör et al., 1993; Windley et al., 2007; Figure 1). This area was mainly dominated by the Palaeo-Asian Ocean tectonic domain in the Pre-Mesozoic period, and strongly transformed by the circum-Pacific tectonic domain since the Mesozoic (Liu et al., 2010, 2017). Since the Late Mesozoic a large continental rift system developed in NE China, related to the subduction of the Pacific Plate and back-arc extension of the Japan Sea (Liu, 1988; Xu & Fan, 2015). This rift system includes the Songliao Basin, Jiamusi–Yitong Fault Zone, Dunhua–Mishan Fault Zone, and other adjacent basins (Figure 1). Contemporaneously, about 690 volcanic cones and craters and 50,000 km² of basaltic lavas with small amounts of alkali trachyte were formed in this area. The Cenozoic volcanism is mainly distributed alongside a series of NE to NNE-oriented rift basins and adjacent mountain ranges and on both sides of the Songliao Basin, but major volcanic activity occurred to the east (Liu, 1988; Figure 1). From west to east, the distribution of the volcanic rocks can be divided into several zones, these are the Great Xing’an Range, the Jiamusi–Yitong Fault Zone, the Dunhua–Mishan Fault Zone and the Changbai Mountains. The borehole data from the Songliao Basin reveals over 1 km-thick Palaeogene basalt bodies of tholeiitic composition (Xu et al., 2015). The next volcanic activity peak period appeared in the Neogene and mainly follows the Jiamusi–Yitong Fault Zone and Dunhua–Mishan Fault Zone. The youngest Quaternary volcanic rocks in NE China are distributed around the Songliao Basin with major occurrences in the Great Xing’an Range and even more western areas, to the north of the Songliao Basin, and to the east in the Changbai Mountains, mostly east of Dunhua–Mishan Fault Zone (Bai, Tian, Wu, Xu, & Li, 2005; Bai, Wang, Xu, Liu, & Xu, 2008; Fan & Hooper, 1991; Fan, Liu, Zhang, & Sui, 1998; Fan et al., 1999, 2006, 2007, 2011, 2012; Liu, 1987; Liu et al., 1998; Qiu et al., 1991; Zhang et al., 2000; Zhao et al., 2008; Figure 1). The Cenozoic basalts in NE China are considered products of partial melting of the upper mantle, and mixing of depleted mantle and enriched mantle Type I components (Xu et al., 2015; Zou et al., 2000, Zhou, 2006).

Even though numerous geochronological studies have been published from many occurrences of mafic volcanic formations in NE China, high-precision and weathering-insensitive geochronology such as zircon U–Pb or (U–Th)/He dating has not yet been performed on the young volcanic formations of the Huanan area. Previous studies in this area mainly rely on constraints from lithostratigraphic and palaeontological evidences (HBGMR, 1993).

3 | SAMPLE AND ANALYTICAL METHODS

A sand sample (JB40) was collected in an active basalt quarry close to Qunli village (Figure 2; 46.2983°N, 130.7182°E). A 2–3 m thick horizontal lava flow is exposed along the excavation walls and the contact to the underlying sand is well preserved and accessible. In the surroundings of the quarry, the sand forms only a few metres thick layer; this young, alluvial sediment covers the granitoid basement. The basal layer of the lava is amygdaloid, but the lava shows a low degree of alteration. We collected a loose sand sample from the topmost 3–5 cm, immediately below the base of the basalt lava (Figure 3).
To discriminate the thermal influence imposed by the basalt lava to the underlying granite basement at the sampling position and the untouched area, three granite samples were collected for (U–Th)/He dating from the wider area surrounding the basalt quarry (Figure 2; JB37, 46.4320°N, 131.09425°E; JB39, 46.3189°N, 131.0713°E; JB41, 46.0767°N; 130.6672°E). The zircon grains were separated from the 63–125 μm fraction by shaking table, gravity separation by Na-poly-tungstate, and magnetic separation.

The zircon crystals have variable shapes and colours, but they are mostly pinkish-brown, transparent-translucent and euhedral to slightly rounded. (U–Th)/He analyses were performed at the GÖochron Laboratory of the Geoscience Center, University of Göttingen. Twenty-eight intact zircon crystals were selected by stereo- and petrographic microscopes. The crystals were photographed and their dimensions (length, width, and prismatic length) were used for alpha-ejection correction (Farley, Wolf, & Silver, 1996; Figure 4). The grains were wrapped in...
platinum capsules for helium extraction and heated with an infrared laser. The extracted gas was purified by an SAES Ti-Zr getter at 450°C. The remaining inert gas was measured by a Hidden triple-filter quadrupole mass spectrometer equipped with a positive ion-counting detector. Following degassing, the capsules were retrieved from the gas extraction line the zircon crystals were extracted from the capsules and spiked with calibrated 230Th and 233U solutions in 0.4 ml teflon vials. The crystals were dissolved for 5 days at 220°C in pressurized bombs using a mixture of double distilled 48% HF and 65% HNO3. Each sample batch was prepared with a series of procedural blanks and spiked normals to check the purity and calibration of the reagents and spikes. Spiked solutions were analysed by a Thermo iCAP Q ICP-MS. Procedural U and Th blanks by this method are usually very stable in a measurement session and below 1.5 pg. The ejection correction factors (Ft) were determined for the single crystals by a modified algorithm of Farley et al. (1996) using an in-house spread sheet.

Raman spectroscopy was applied to all zircon samples to identify the thermal influence on the lattice of the zircon crystals as additional information to interpret the (U–Th)/He chronological data. Details of the laboratory procedure can be found in Lünsdorf and Lünsdorf (2016). The IFORS software was used to evaluate the Raman spectra. Fitted peak widths were corrected for the apparatus function after Irmer (1985) and Nasdala et al. (2001).

4 | RESULTS

4.1 | Zircon (U–Th)/He ages

Twenty-eight euhedral or slightly rounded zircon crystals were dated (Figure 4 and Table 1). The crystal sizes with c-axis parallel and perpendicular dimensions range from 120 to 319 μm and 55 to 98 μm,
| Sample | He Vol. [ncc] | 1s [%] | Mass [ng] | 1s [%] | Conc. [ppm] | 238U- Mass [ng] | 1s [%] | Conc. [ppm] | 232Th- Mass [ng] | 1s [%] | Conc. [ppm] | Th/U ratio | Sm Mass [ng] | 1s [%] | Conc. [ppm] | eU [ppm] | Sphere radius [μm] | Ejection correct. He-age [Ma] | Uncorr. He-age [Ma] | Ft-Corr. He-age [Ma] | 2s He-age [Ma] | Sample unweighted avr. ± 1 SE |
|--------|---------------|--------|-----------|--------|-------------|----------------|--------|-------------|----------------|--------|-------------|------------|-------------|--------|-------------|--------|-------------------|-----------------|----------------------|---------------------|----------------------|
| JB40 z1 | 1.436         | 1.1    | 1.081     | 1.8    | 447.7       | 0.232          | 2.4    | 96          | 0.21           | 0.016  | 3.1         | 7          | 470.3       | 53     | 0.769       | 10.5   | 13.6             | 1.1              |                      |
| JB40 z2 | 1.756         | 1.1    | 1.713     | 1.8    | 417.8       | 0.425          | 2.4    | 104         | 0.25           | 0.012  | 3.1         | 3          | 442.1       | 59     | 0.791       | 8.0    | 10.1             | 0.7              |                      |
| JB40 z3 | 1.418         | 1.1    | 1.752     | 1.8    | 937.1       | 0.371          | 2.4    | 199         | 0.21           | 0.019  | 3.1         | 10         | 983.7       | 36     | 0.672       | 6.4    | 9.5              | 1.0              |                      |
| JB40 z5 | 0.734         | 1.1    | 0.922     | 1.8    | 831.4       | 0.387          | 2.4    | 349         | 0.42           | 0.012  | 3.1         | 11         | 913.4       | 37     | 0.679       | 6.0    | 8.8              | 0.9              |                      |
| JB40 z6 | 0.179         | 1.6    | 0.181     | 2.2    | 186.2       | 0.092          | 2.5    | 95          | 0.51           | 0.007  | 3.1         | 8          | 208.4       | 34     | 0.652       | 7.3    | 11.2             | 1.3              |                      |
| JB40 z7 | 5.636         | 1.0    | 2.107     | 1.8    | 614         | 0.641          | 2.4    | 187         | 0.30           | 0.015  | 5.5         | 4          | 658.3       | 42     | 0.711       | 20.6   | 29.0             | 2.8              |                      |
| JB40 z8 | 2.100         | 1.1    | 2.627     | 1.8    | 1861        | 0.136          | 2.4    | 96          | 0.05           | 0.005  | 5.5         | 4          | 1883.3      | 46     | 0.735       | 6.5    | 8.9              | 0.8              |                      |
| JB40 z9 | 3.226         | 1.0    | 3.896     | 1.8    | 836         | 1.051          | 2.4    | 226         | 0.27           | 0.022  | 5.5         | 5          | 889.5       | 51     | 0.758       | 6.4    | 8.5              | 0.7              |                      |
| JB40 z10| 2.605         | 1.0    | 2.176     | 1.8    | 762         | 0.456          | 2.4    | 160         | 0.21           | 0.008  | 5.5         | 3          | 799.5       | 41     | 0.706       | 9.4    | 13.4             | 1.3              |                      |
| JB40 z11| 2.736         | 1.1    | 1.897     | 1.8    | 480         | 0.488          | 2.4    | 123         | 0.26           | 0.023  | 5.5         | 6          | 508.8       | 44     | 0.723       | 11.3   | 15.6             | 1.4              |                      |
| JB40 z12| 0.831         | 1.2    | 1.081     | 1.8    | 277         | 0.248          | 2.4    | 64          | 0.23           | 0.017  | 5.5         | 4          | 291.7       | 44     | 0.727       | 6.0    | 8.3              | 0.8              |                      |
| JB40 z13| 0.765         | 1.2    | 1.441     | 1.8    | 618         | 0.325          | 2.4    | 140         | 0.23           | 0.056  | 5.5         | 24         | 650.9       | 45     | 0.734       | 4.2    | 5.7              | 0.5              |                      |
| JB40 z14| 9.985         | 1.0    | 3.738     | 1.8    | 1,200       | 0.298          | 2.4    | 96          | 0.08           | 0.007  | 5.5         | 2          | 1,222.4     | 41     | 0.711       | 21.7   | 30.5             | 2.9              |                      |
| JB40 z15| 0.719         | 1.2    | 1.032     | 1.8    | 750         | 0.156          | 2.4    | 114         | 0.15           | 0.005  | 5.5         | 3          | 776.4       | 36     | 0.671       | 5.6    | 8.3              | 0.9              |                      |
| JB40 z16| 1.539         | 1.1    | 1.185     | 1.8    | 361         | 0.290          | 2.4    | 88          | 0.25           | 0.017  | 5.5         | 5          | 382.0       | 42     | 0.714       | 10.2   | 14.2             | 1.3              |                      |
| JB40 z17| 0.775         | 1.2    | 0.901     | 1.8    | 380         | 0.238          | 2.4    | 100         | 0.26           | 0.011  | 5.5         | 4          | 403.4       | 41     | 0.707       | 6.7    | 9.5              | 0.9              |                      |
| JB40 z18| 2.153         | 1.0    | 2.447     | 1.8    | 505         | 0.653          | 2.4    | 135         | 0.27           | 0.042  | 5.5         | 9          | 536.4       | 52     | 0.764       | 6.9    | 9.0              | 0.7              |                      |
| Sample  | Vol. [ncc] | 1s [%] | 238U- Mass [ng] | 1s [%] | Conc. [ppm] | 232Th- Mass [ng] | 1s [%] | Conc. [ppm] | Th/U ratio | Sm Mass [ng] | 1s [%] | Conc. [ppm] | eU [ppm] | Sphere radius [um] | Ejection correct. (Ft) | Uncorr. He-age [Ma] | Ft-Corr. He-age [Ma] | 2s [Ma] | He-age 2s [Ma] | Sample unweighted aver. ± 1 SE |
|---------|------------|--------|----------------|--------|-------------|----------------|--------|-------------|-----------|--------------|--------|-------------|----------|----------------|----------------------|-------------------|-------------------|---------|-------------|-----------------|
| JB40    | 3.319      | 1.1    | 3.335          | 1.8    | 958         | 0.660          | 2.4    | 190         | 0.20      | 0.010        | 5.5    | 3           | 1003.0   | 48              | 0.745                | 7.9               | 10.6              | 0.9     |             |                 |
| JB40    | 1.227      | 1.1    | 0.529          | 1.8    | 146         | 0.236          | 2.4    | 65          | 0.45      | 0.021        | 5.5    | 6           | 160.9    | 52              | 0.763                | 17.3              | 22.7              | 1.8     |             |                 |
| JB40    | 0.633      | 1.3    | 0.650          | 1.8    | 129         | 0.338          | 2.4    | 67          | 0.52      | 0.032        | 5.5    | 6           | 144.6    | 47              | 0.741                | 7.2               | 9.7               | 0.8     |             |                 |
| JB37    | 7.550      | 1.2    | 0.857          | 1.8    | 261         | 0.329          | 2.4    | 100         | 0.38      | 0.017        | 6.5    | 5           | 284.7    | 45              | 0.732                | 66.5              | 90.9              | 8.2     |             |                 |
| JB37    | 11.127     | 1.2    | 1.178          | 1.8    | 194         | 0.317          | 2.4    | 52          | 0.27      | 0.025        | 6.5    | 4           | 205.9    | 49              | 0.751                | 73.1              | 97.3              | 8.3     | 94.1        | 3.2             |
| JB39    | 88.920     | 1.2    | 7.681          | 1.8    | 597         | 0.931          | 2.4    | 72          | 0.12      | 0.070        | 6.5    | 5           | 614.1    | 77              | 0.838                | 92.5              | 110.3             | 7.0     |             |                 |
| JB39    | 99.685     | 1.2    | 11.233         | 1.8    | 1,331       | 0.919          | 2.4    | 109         | 0.08      | 0.084        | 6.5    | 10          | 1356.3   | 64              | 0.807                | 71.7              | 88.8              | 6.4     |             |                 |
| JB39    | 61.323     | 1.2    | 6.356          | 1.8    | 638         | 1.252          | 2.4    | 126         | 0.20      | 0.052        | 6.5    | 5           | 667.2    | 68              | 0.818                | 75.9              | 92.8              | 6.3     |             |                 |
| JB39    | 26.639     | 1.2    | 2.790          | 1.8    | 793         | 0.563          | 2.4    | 160         | 0.20      | 0.034        | 6.5    | 10          | 830.7    | 50              | 0.755                | 75.0              | 99.4              | 8.4     | 97.8        | 4.7             |
| JB41    | 20.940     | 1.2    | 1.756          | 1.8    | 385         | 0.495          | 2.4    | 109         | 0.28      | 0.099        | 6.5    | 22          | 410.4    | 45              | 0.732                | 91.9              | 125.5             | 11.3    |             |                 |
| JB41    | 33.814     | 1.2    | 2.419          | 1.8    | 507         | 0.450          | 2.4    | 94          | 0.19      | 0.070        | 6.5    | 15          | 529.3    | 49              | 0.753                | 109.9             | 146.0             | 12.4    | 135.7       | 10.2            |
The measured zircon crystals reveal radii ranging from 34 to 59 μm and the effective uranium concentration (eU, where eU is calculated as \([\text{U ppm}] + 0.235 \times \text{[Th ppm]}\); Gordon Gastil et al., 1967) covers a wide range from 145 to 1883 ppm. The Ft-corrected zircon ZHe ages of the dated crystals from the JB40 sand sample range from 5.7 to 30.5 Ma (Figures 4 and 5). Except for the youngest single zircon He age of 5.7 ± 0.5 Ma and three older He-ages >20 Ma, the ages reveal a tight distribution between 8.3 and 15.6 Ma. The Ft-corrected ZHe ages of the three granites samples from the region also reveal tight clustering with unweighted ZHe mean ages of 94.1 ± 3.2, 97.8 ± 4.7, and 135.7 ± 10.2 Ma for samples JB37, JB39, and JB41, respectively (Table 1). ZHe ages show no correlation with eU concentrations (Figure 6) implying that the effect of radiation damage density on the measured apparent (U-Th)/He ages is negligible (e.g., Cook, Royden, Burchfiel, Lee, & Tan, 2013; Flowers, Ketcham, Shuster, & Farley, 2009; Reiners, 2005; Shuster, Flowers, & Farley, 2006).

4.2 | Raman spectra of the zircon crystals

Raman spectra of the zircon crystals

ZHe ages are determined by the retentivity of He in zircon crystals, which is influenced by the alpha-damage inflicted in its crystalline lattice due to self-irradiation (e.g., Guenthner, Reiners, Ketcham, Nasdala, & Giester, 2013). Raman spectroscopy offers the opportunity to quantify the degree of metamictization in zircon crystals (Nasdala, Irmer, & Wolf, 1995) selected for (U-Th)/He analysis. The accumulated alpha-damage is estimated from the position and the width of the ν3(SiO4) Raman band, the stretching vibration of the SiO4 tetrahedra about 1,000 cm\(^{-1}\) (Dawson, Hargreave, & Wilkinson, 1971). In our case, the four samples reveal distinct, narrow internal, and external vibrational modes in the spectral range from 972.1 to 1,010.5 cm\(^{-1}\). All of the analysed zircon crystals have tightly distributed full width at half-maximum (FWHM) values ranging from 3.4 to 9.0 cm\(^{-1}\), with averages of 5.1 cm\(^{-1}\) (JB40), 5.5 cm\(^{-1}\) (JB37), 5.2 cm\(^{-1}\) (JB39), and 6.8 cm\(^{-1}\) (JB41), respectively (Table 2).

5 | DISCUSSION

5.1 | Identification of the principal age component of the single-crystal ZHe data

Visualizing and interpreting the ages obtained on detrital zircon crystals forms a key part to unravel the corresponding geological questions in detrital zircon geochronological and thermochronological studies. The probability density plot (PDP) and the kernel density estimate (KDE) are the most used methods for visualizing detrital age distributions (Devroye, 1987; Hurford, Fitch, & Clarke, 1984; Silverman, 1986; Vermeesch, 2012; von Eynatten & Dunkl, 2012). However, it has been pointed out that the PDP lacks any theoretical basis as a probability density estimator, although it may serve as a data visualization tool (Galbraith, 1998, 2010; Vermeesch, 2012).

The ZHe age distribution is visualized as KDE plot by the DensityPlotter v8.4 software (Figure 7; Vermeesch, 2012). The KDE age spectrum shows a typical left-hand asymmetry and the mean of the dominating (about 75%) youngest age component is 9.33 ± 0.24 Ma (Figure 7). To further corroborate the result, we also use the SIMPLEX method (Cserepes, 1989) to perform a best-fit model to identify the age components by the Popshare software (Dunkl & Székely, 2002). This approach results in a similar best-fit model age at 9.2 ± 0.8 Ma.

5.2 | Zircon reset analysis

In the study area, most of the basalt lava overlies the basement dominated by granitoid rocks. In our study site, the lava covers alluvial
| Sample       | Aliquot  | Scale intensity | Shape | Area      | HWHM [cm\(^{-1}\)] | Centre [cm\(^{-1}\)] | Scaled_intensity | Shape | Area      | HWHM [cm\(^{-1}\)] | Corr FWHM [cm\(^{-1}\)] | Centre [cm\(^{-1}\)] |
|--------------|----------|-----------------|-------|-----------|---------------------|-----------------------|--------------------|-------|-----------|---------------------|--------------------------|-------------------|
| JB40         | JB_Points00 | 13.426          | 0.930 | 636.008   | 3.076               | 976.246               | 91.795             | 0.780 | 4,179.228 | 3.102               | 4.9                      | 1,009.286         |
|              | JB_Points01 | 12.933          | 0.984 | 674.038   | 3.334               | 976.246               | 84.662             | 0.775 | 4,141.691 | 3.342               | 5.5                      | 1,009.482         |
|              | JB_Points02 | 12.898          | 0.979 | 738.396   | 3.676               | 976.246               | 90.315             | 0.842 | 4,543.066 | 3.363               | 5.5                      | 1,009.286         |
|              | JB_Points03 | 12.050          | 0.687 | 459.716   | 2.677               | 977.028               | 85.441             | 0.516 | 3,145.437 | 2.744               | 3.9                      | 1,010.459         |
|              | JB_Points04 | 8.149           | 0.987 | 312.851   | 2.439               | 977.419               | 45.620             | 0.513 | 1,615.646 | 2.641               | 3.6                      | 1,010.459         |
|              | JB_Points05 | 14.672          | 0.983 | 626.901   | 2.723               | 976.833               | 90.120             | 0.769 | 4,189.730 | 3.180               | 5.1                      | 1,010.264         |
|              | JB_Points12 | 11.592          | 0.986 | 608.307   | 3.356               | 975.660               | 76.930             | 0.820 | 3,961.219 | 3.468               | 5.8                      | 1,008.895         |
|              | JB_Points13 | 13.924          | 0.988 | 789.011   | 3.629               | 975.269               | 89.816             | 0.857 | 4,969.772 | 3.687               | 6.3                      | 1,011.133         |
|              | JB_Points14 | 10.022          | 0.916 | 641.540   | 4.207               | 975.464               | 67.544             | 0.988 | 4,322.868 | 4.105               | 7.3                      | 1,008.309         |
|              | JB_Points15 | 14.272          | 0.866 | 558.459   | 2.586               | 976.246               | 91.525             | 0.643 | 3,845.737 | 2.997               | 4.6                      | 1,009.482         |
|              | JB_Points16 | 7.732           | 0.982 | 364.740   | 3.013               | 976.051               | 48.804             | 0.358 | 1900.080  | 3.082               | 4.8                      | 1,009.873         |
|              | JB_Points17 | 13.463          | 0.717 | 526.283   | 2.716               | 976.051               | 93.207             | 0.671 | 3,828.205 | 2.899               | 4.4                      | 1,009.286         |

(Continues)
| Sample   | Aliquot | ScaleIntensity | Shape | Area  | HWHM [cm$^{-1}$] | Centre [cm$^{-1}$] | Scaled_intensity | Shape | Area  | HWHM [cm$^{-1}$] | Corr FWHM [cm$^{-1}$] | Centre [cm$^{-1}$] |
|----------|----------|----------------|-------|-------|------------------|------------------|------------------|-------|-------|------------------|----------------------|------------------|
| JB_Points52 |          | 13.332          | 0.653 | 526.979 | 2.809            | 975.269          | 94.097           | 0.676 | 4,112.200 | 3.083           | 4.8                  | 1.008.700        |
| JB_Points53 |          | 13.421          | 0.681 | 532.055 | 2.790            | 975.464          | 92.573           | 0.688 | 4,023.187 | 3.053           | 4.8                  | 1.008.954        |
| JB_Points54 |          | 12.294          | 0.627 | 480.804 | 2.804            | 975.660          | 92.312           | 0.663 | 3,591.145 | 2.752           | 4.0                  | 1.008.895        |
| JB_Points55 |          | 13.523          | 0.700 | 552.100 | 2.855            | 975.269          | 90.668           | 0.731 | 4,065.666 | 3.105           | 4.9                  | 1.008.504        |
| JB_Points56 |          | 13.462          | 0.783 | 555.673 | 2.807            | 975.464          | 92.266           | 0.724 | 3,984.224 | 2.995           | 4.6                  | 1.008.504        |
| JB_Points57 |          | 13.177          | 0.860 | 640.517 | 3.232            | 975.073          | 96.727           | 0.798 | 4,915.740 | 3.448           | 5.7                  | 1.008.113        |
| JB_Points58 |          | 13.353          | 0.796 | 558.328 | 2.831            | 975.464          | 92.076           | 0.726 | 4,141.994 | 3.120           | 4.9                  | 1.008.504        |
| JB_Points59 |          | 13.659          | 0.817 | 547.807 | 2.695            | 975.073          | 95.655           | 0.698 | 4,089.496 | 2.992           | 4.6                  | 1.008.309        |
| JB_Points60 |          | 12.599          | 0.819 | 521.256 | 2.780            | 975.269          | 92.372           | 0.689 | 3,779.379 | 2.870           | 4.3                  | 1.008.309        |
| JB_Points61 |          | 12.713          | 0.987 | 574.117 | 2.878            | 975.269          | 90.996           | 0.553 | 3,440.688 | 2.782           | 4.0                  | 1.008.700        |
| JB_Points62 |          | 13.088          | 0.631 | 487.739 | 2.666            | 975.660          | 92.683           | 0.651 | 3,721.196 | 2.854           | 4.2                  | 1.008.895        |
| JB_Points63 |          | 13.491          | 0.745 | 566.837 | 2.895            | 975.073          | 94.514           | 0.680 | 4,270.497 | 3.185           | 5.1                  | 1.008.309        |
| JB_Points64 |          | 13.320          | 0.725 | 491.280 | 2.554            | 975.660          | 92.385           | 0.656 | 3,723.374 | 2.859           | 4.3                  | 1.009.091        |
| JB_Points65 |          | 12.760          | 0.620 | 438.320 | 2.465            | 975.660          | 93.861           | 0.561 | 3,274.705 | 2.556           | 3.4                  | 1.008.895        |
| JB_Points66 |          | 40.289          | 0.982 | 2071.293 | 3.291           | 974.291          | 68.831           | 0.901 | 3,608.680 | 3.467           | 5.8                  | 1.007.136        |
| JB_Points72 |          | 69.601          | 0.958 | 3654.670 | 3.389           | 974.096          | 68.852           | 0.966 | 3,820.453 | 3.571           | 6.0                  | 1.006.940        |
| JB_Points78 |          | 12.763          | 0.986 | 758.537 | 3.813            | 973.314          | 82.491           | 0.967 | 4,764.442 | 3.719           | 6.4                  | 1.005.767        |
| JB_Points80 |          | 12.970          | 0.956 | 674.335 | 3.357            | 973.900          | 86.656           | 0.899 | 4,557.209 | 3.453           | 5.8                  | 1.006.745        |
| JB_Points84 |          | 51.412          | 0.971 | 3678.692 | 4.640           | 972.532          | 54.838           | 0.986 | 3,892.660 | 4.570           | 8.3                  | 1.005.181        |
| JB_Points85 |          | 58.107          | 0.966 | 3584.590 | 3.988           | 973.118          | 57.305           | 0.977 | 3,788.342 | 4.259           | 7.6                  | 1.005.572        |
| JB_Points86 |          | 53.225          | 0.986 | 3197.582 | 3.856           | 973.118          | 51.276           | 0.974 | 3,400.902 | 4.277           | 7.7                  | 1.005.767        |
| JB_Points87 |          | 12.765          | 0.822 | 686.892 | 3.632            | 972.923          | 91.042           | 0.907 | 5,260.163 | 3.792           | 6.6                  | 1.005.572        |
| JB_Points88 |          | 11.937          | 0.858 | 776.612 | 4.360            | 972.336          | 87.021           | 0.940 | 5,882.466 | 4.409           | 7.9                  | 1.005.181        |
| JB_Points89 |          | 11.818          | 0.777 | 693.871 | 4.030            | 972.727          | 90.310           | 0.906 | 5,953.288 | 4.343           | 7.8                  | 1.005.181        |
| JB_Points91 |          | 10.716          | 0.987 | 805.760 | 4.681            | 972.141          | 71.087           | 0.988 | 5,414.262 | 4.911           | 9.0                  | 1.004.399        |
| JB_Points92 |          | 13.393          | 0.988 | 789.764 | 3.780            | 973.705          | 87.881           | 0.843 | 5,001.379 | 3.812           | 6.6                  | 1.006.158        |

Abbreviation: FWHM, full width at half-maximum. HWHM, half width at half maximum.
sand. For the proper evaluation of the potential thermal overprint, we should first review the cooling age pattern of the basement that experienced no thermal overprint by young basalt eruptions. Zircon U-Pb studies indicate that the emplacement ages of the granitoid rocks in Huanan and its adjacent areas are Pre-Mesozoic, mostly Early to Middle Permian (Bi et al., 2014; Dong et al., 2017; Yang, Ge, Zhao, Yu, & Zhang, 2015) (Figure 2). Low-temperature thermochronology performed on basement samples far from basalt occurrences yield Early Cretaceous to early Late Cretaceous ZHe ages (136–94 Ma; Figures 2 and 4; Table 1). These ages are considerably older than the ZHe age of sand sample from below the basalt lava. The zircons in the loose sand layer overlying the granitoid basement thus do not carry the regional cooling age signature, instead, their ZHe ages are mostly determined by the thermal effect of the basalt lava.

Zircon He diffusion experiments on pristine crystals reveal that the closure temperature of the ZHe thermochronometer is around 160–200 °C in case of duration of the thermal overprint in the range of millions of years (Reiners et al., 2004). Even though the eruption temperature of the overlying basalt lava could be variable, the temperature of basaltic lavas is mostly above 950 °C (Francis, 1993). Blondes et al. (2007) presented calculations on the necessary time and temperature relations for reset of the ZHe thermochronometer in case of very short, shock-like thermal events like contact with lava. The laboratory derived He-in-zircon diffusion experiments indicated that partly or complete He loss in xenolithic zircon crystals should happen in magmatic entrainment or contact time of less than 1 hr (Blondes et al., 2007). The sample JB40 experienced proper temperature–time integral for complete reset as it situated close enough to the basalt lava and the heat of the basalt lava could lead to the full removal of the pre-eruption accumulated radiogenic helium from the majority of the zircon grains.

The Raman spectra of well-ordered zircon crystals show distinct, narrow vibrational modes in the spectral range from 200 to 1,010 cm⁻¹. With increasing radiation damage, all of the main Raman bands of the zircon crystals decrease in intensity and become increasingly broader (Nasdala et al., 2001). The FWHM [the full width at half-maximum of the ν3(SiO4) vibration (FWHM)] for the sand sample JB40 and the three granitoid samples [Colour figure can be viewed at wileyonlinelibrary.com]
Ulonska, Schleicher, Pidgeon, and van Bronswijk (2001) and Nasdala, Irmer, and Jonckheere (2002) have found some miscorrelation between the Raman bandwidths and positions. These annealed zircon crystals mostly plot above the peak position-peak width trend established for zircons derived from unheated or slowly cooled geological settings (Nasdala et al., 2001, 2002). In our case, the Raman parameters obtained on sample JB40 plot somewhat off the trend constrained by the three granite samples reflecting the regional cooling history (Figure 8). This property of the lattice of the zircons from the sand sample below the lava flow supports their shock-like thermal reset.

In summary, we can conclude that the detrital zircon crystals have been heated and their ZHe clock became fully reset at the contact with the basalt. The ZHe age of 9.33 ± 0.24 Ma of the sand sample is thus interpreted to represent the eruption age of the overlying basalt lava.

### 5.3 Relation to other Miocene basalt lava occurrences

Liu distinguished 10 Cenozoic volcanic episodes in NE China, which are listed in Figure 9. According to the measured age, the Huanan basalt lava in this study belongs to the Laoyeling volcanic episode (βN̄31, 11–7 Ma), which is characterized by alkali olivine basalt, basanite, and basalt with ultramafic xenoliths. The magma of this volcanic episode mainly originated from partial melting of the upper mantle caused by extension of the East Asian continent, driven by the slab...
rollback of the Pacific plate’s westward subduction (Xu et al., 2012; Xu & Fan, 2015).

6 | CONCLUSIONS

1 (U-Th)/He dating of detrital zircon grains from a sand layer directly below a basalt lava flow in the Huanan region reveals a dominant age component of 9.33 ± 0.24 Ma. This implies, together with the Raman data that the reset of the 2He thermochronometer was caused by the thermal effect of the basalt lava, which erupted at this time.

2 The result also implies that the basalt in the Huanan area belongs to the Laoyeling volcanic episode.

3 As a well-developed weathering insensitive geochronometer, the zircon (U-Th)/He method provides a fast and high accuracy dating tool for young, mafic volcanic rocks.

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