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LA–ICP-MS Zircon U–Pb and Alunite $^{39}Ar/^{40}Ar$ Dating of the Saozhouhe Alunite Deposit: Implications for the Metallogenic Significance of the Xiaotian Basin

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Abstract: As an important part of the northern Huaiyang tectonic belt, the Mesozoic Xiaotian basin hosts a series of gold and alunite deposits. However, the ages of these deposits remain unclear, constraining the further understanding of the genesis of these deposits. In this work, zircon LA–ICP-MS and alunite $^{39}Ar/^{40}Ar$ isotopic dating studies were carried out on the andesitic porphyrite and alunite of the Saozhouhe alunite deposit, respectively. LA–ICP-MS zircon U–Pb dating for the Saozhouhe andesitic porphyrite yields a weighted average age of 122.8 $\pm$ 0.9 Ma (MSWD = 1.8), indicating that these volcanic rocks were formed in the Early Cretaceous period. The alunite 39Ar/40Ar dating yields a plateau age of 121.0 $\pm$ 1.1 Ma (MSWD = 4.4) and an isochron age of 121.2 $\pm$1.9 Ma (MSWD = 8.5), indicating that the alunite was also formed in the Early Cretaceous period. Our finding confirms that the formation of the Saozhouhe alunite deposit is genetically related to the Cretaceous volcanic magmatism, which has great significance in the metallogenetic regularity of and further ore prospecting in the Xiaotian basin.

Keywords: andesitic porphyrite; zircon age; $^{39}Ar/^{40}Ar$ dating; alunite deposit; Xiaotian basin

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1. Introduction

The epithermal deposits were proposed as being related to volcanic and/or subvolcanic activities, and as being hosted in the shallow regions of the crust, including the noble metal (Au and Ag) deposits, base metal (Pb and Zn) deposits, and non-metal (Hg, S, kaolinite, and alunite) deposits. The epithermal deposits are one of the main Au, Ag, Pb, and Zn sources for the global market, and a series of world-class noble metal deposits belong to this type, e.g., the Ladolam Au deposit of Papua New Guinea, the Yanacocha Au–Ag deposit of Peru, and the Veladero Au deposit of Argentina [1–3]. Due to its great production and reserves, the formation of the epithermal deposits is one of the hot spots for geologists since its discovery [4,5]. The epithermal Au deposits, one of the most important types of gold deposits around the world, could be subdivided into two series, i.e., high-S and low-S [4]. The high-S epithermal gold deposits are characterized by containing a series of S-enriched minerals (e.g., alunite and pyrite), which are ideal indicators for recording the ore-forming process of these epithermal gold deposits [5,6]. Recent studies also indicate that the S-enriched minerals could be important prospecting criteria for the porphyry Cu–Au deposits and porphyrite type Fe deposits [5,7,8]. Alunite, one of the common S-enriched minerals in the high-S epithermal gold deposits, could occur as the main mineral of the independent alunite deposit, which is spatially correlated with the epithermal gold deposits. Therefore, the studies on these S-enriched minerals aroused the wide interests of geologists [8].

The approximate EW trending north Huaiyang tectonic belt is a converging zone between the North China Craton and the Yangtze Craton (Figure 1a). As one of the important metallogenic belts of south China, a series of metal (e.g., Mo, Au, Ag, Pb, and Zn) deposits and non-metal deposits (e.g., alunite and barite) are hosted in this region. The Au deposits are mostly classified as epithermal gold deposits, which are related to volcanic hydrothermal events [9–11]. These volcanic rocks, mainly formed during the Early Cretaceous period (137–127 Ma), are intermediate-acid and formed in extensional settings [12–17]. Although abundant studies were carried out on these volcanic rocks and related deposits [9–17], no detailed geochronological data on these deposits were reported so far, constraining the further understanding of the genetic links between the volcanic rocks and these hydrothermal deposits. Therefore, a series of geochronological studies were carried out on the new-found andesitic porphyrite and related alunite deposit in the Xiaotian Basin of the north Huaiyang tectonic belt, including LA–ICP-MS zircon U–Pb dating and alunite $^{39}Ar/^{40}Ar$ dating, aiming to constrain the rock- and ore-forming ages of the Saozhouhe andesitic porphyrite and related alunite deposit. The finding of this study will provide essential guiding significance on the gold and alunite mineralization and further ore prospecting in the Xiaotian Basin.

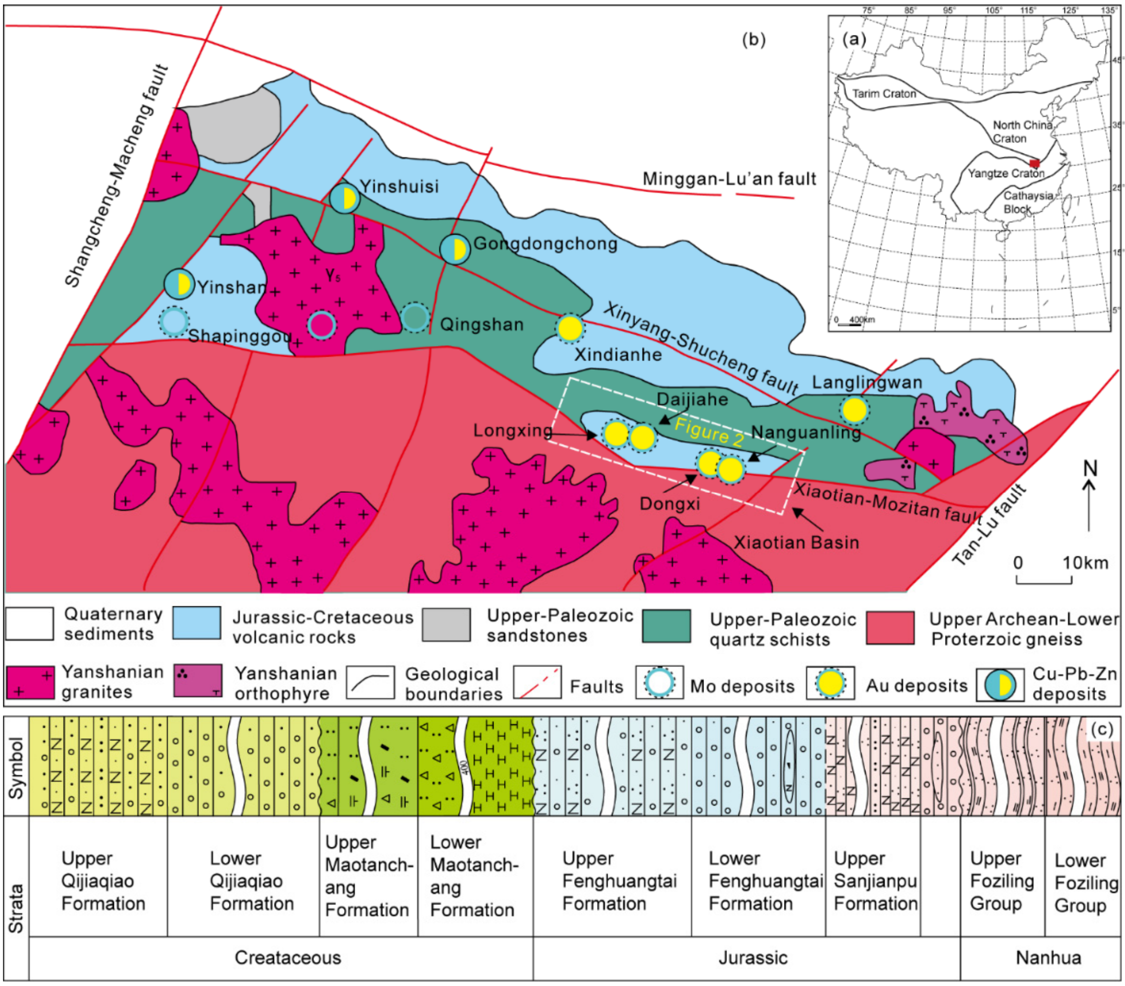

Figure 1. Geological sketch map of China (**a**) and the north Huaiyang Belt (**b**) and the strata sequences of the north Huaiyang Belt (**c**).

2. Geological Background and Ore Deposit Geology

The north Huaiyang Belt, near the Dabie Orogen Belt, was mainly formed in the Caledonian period and is covered by a series of NW-trending Mesozoic volcano-sedimentary basins, i.e., the Jinzhai Basin, Xiaotian Basin, and Huoshan–Shucheng Basin (Figure 1b).

The stratigraphic sequences of the north Huaiyang region are composed of the Upper Archean–Lower Proterozoic metamorphic rocks (basement rocks of the Foziling Group), and the Mesozoic sedimentary rocks (Fenghuangtai and Sanjianpu Formations)/volcanic rocks (Qijiaqiao and Maotanchang Formations). The Cretaceous volcanic rocks mainly unconformably overlie on the basement rocks (Figure 1c). In addition, this region has experienced long terms of tectonic events, resulting in the widespread distributions of the faults and folds. Faults are mainly NW- and NE-trending, however, the NW-trending faults are dominant in this region (Figure 1b). The NW-trending faults are composed of the Xinyang–Shucheng fault, Xiaotian–Mozitan fault, and Minggang–Lu'an fault, which mainly control the distribution of the Cretaceous volcanic basins. The NE-trending faults are mainly composed of the Shangcheng–Macheng fault and giant Tanlu fault. The intrusive rocks can be subdivided into three types, i.e., Neoproterozoic metamorphic gneiss, Mesozoic granitoids, and Mesozoic volcanic rocks. The Mesozoic volcanic rocks are mainly composed of the Cretaceous calc-alkalic volcanic rocks, e.g., andesitic lava, andesite, trachytic volcanic breccia, trachyandesite, and trachyte. The north Huaiyang Belt is also famous for the regional Au–Mo–Cu–Pb–Zn mineralization, hosting the Shapinggou Mo deposit, Yinshan Pb–Zn deposit, Dongxi Au deposit, and so on (Figure 1b). In addition, Au deposits are mainly located in the Xiaotian Basin.

The Xiaotian Basin, located in the northeastern Huaiyang region, has an area of ca. 200 km^2 (Figure 2). This basin hosts abundant Cretaceous volcanic rocks whose distribution are controlled by the Mozitan fault. In addition, these rocks can be subdivided into the Maotanchang and Xiaotian cycles. Rocks of the Maotanchang cycle are composed of early-stage calc-alkalic volcanic rocks (e.g., andesitic agglomerate lava, andesitic agglomerate, andesitic volcanic breccia, and andesite) and late-stage alkaline rocks (e.g., trachytic volcanic breccia, tuff, trachyandesite, trachyte, and quartz trachyandesite). However, rocks of the Xiaotian cycle, mainly explosive facies, and effusive facies, are composed of early-stage caldera assemblies (basalt–andesite–trachyandesite) and late-stage trachytic breccia lava, trachytic tuff, and subvolcanic trachy porphyry. The Xiaotian Basin is also characterized by intense gold mineralization, hosting a series of volcanic–subvolcanic hydrothermal gold deposits, e.g., Dongxi, Nanguanling, and Longxing. Orebodies of these gold deposits are mainly hosted in the volcanic breccia and/or andesitic tuff. Apart from the gold deposits, the Xiaotian Basin also contains a series of non-metal deposits, e.g., the Saozhouhe deposit.

The small-scale Saozhouhe alunite deposit is located in the central part of the Xiaotian Basin (Figure 2). The ore reserve of this deposit is estimated to be 14.1 million tons, with an average ore grade of 37.82%. Stratiform-like alunite ore bodies are hosted in the andesitic volcanic breccia, andesitic tuff, and andesitic porphyrite (Figure 3a). The occurrence of stratiform-like ore bodies is consistent with those surrounding rocks, with a trending angle of 175°–240° and a dip angle of 45°–60°. The NW faults are widely distributed in this deposit, and control the distribution and occurrence of volcanic rocks and ore bodies. Ores, mostly cryptocrystalline massive structures, contain 20–50% alunite, which is mostly hosted in the gaps between the quartz crystals. In addition, because of the impurities, alunite has several colors, e.g., grey, yellow, rosiness, and pompadour. Alteration is mostly unitization, silicification, thematization, and kaolinization. Saozhouhe andesitic porphyrite, an area of 0.1 km^2, occurs as stock, with unconformable contact with the Saozhouhe alunite ore bodies and andesitic tuff of the Cretaceous Maotanchang group.

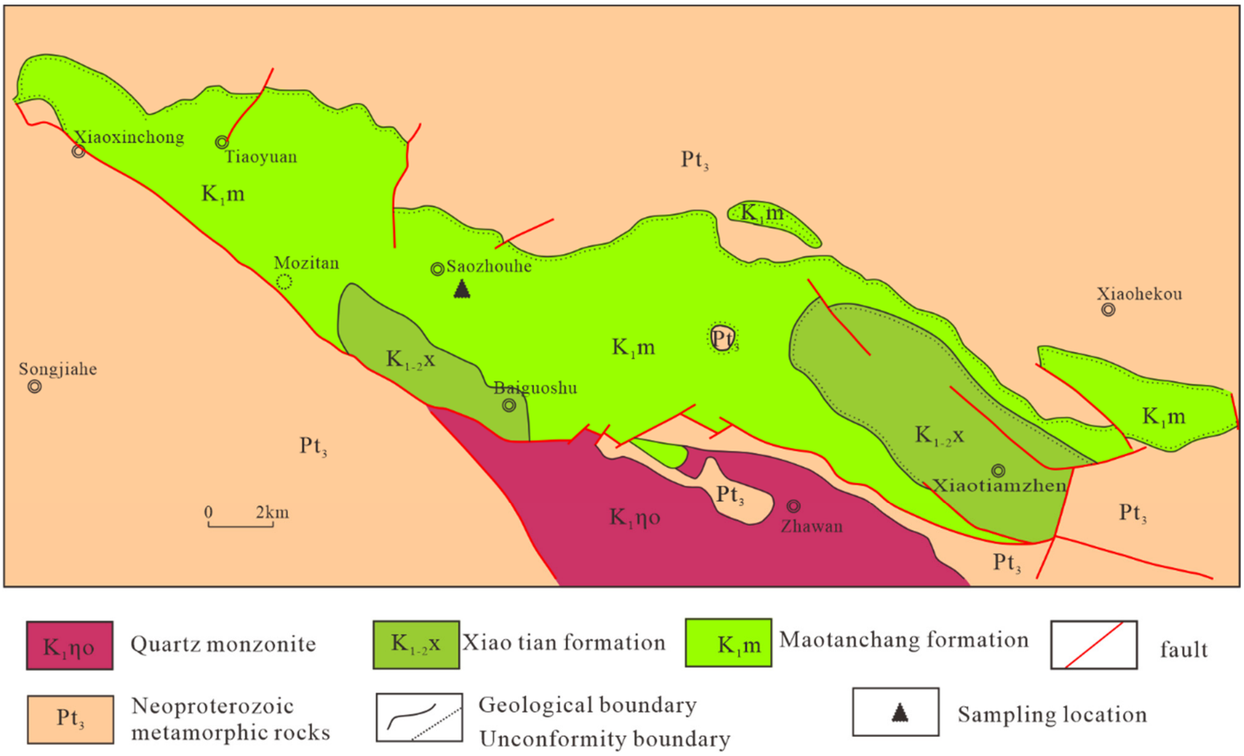

Figure 2. Geological sketch map of the Xiaotian Basin.

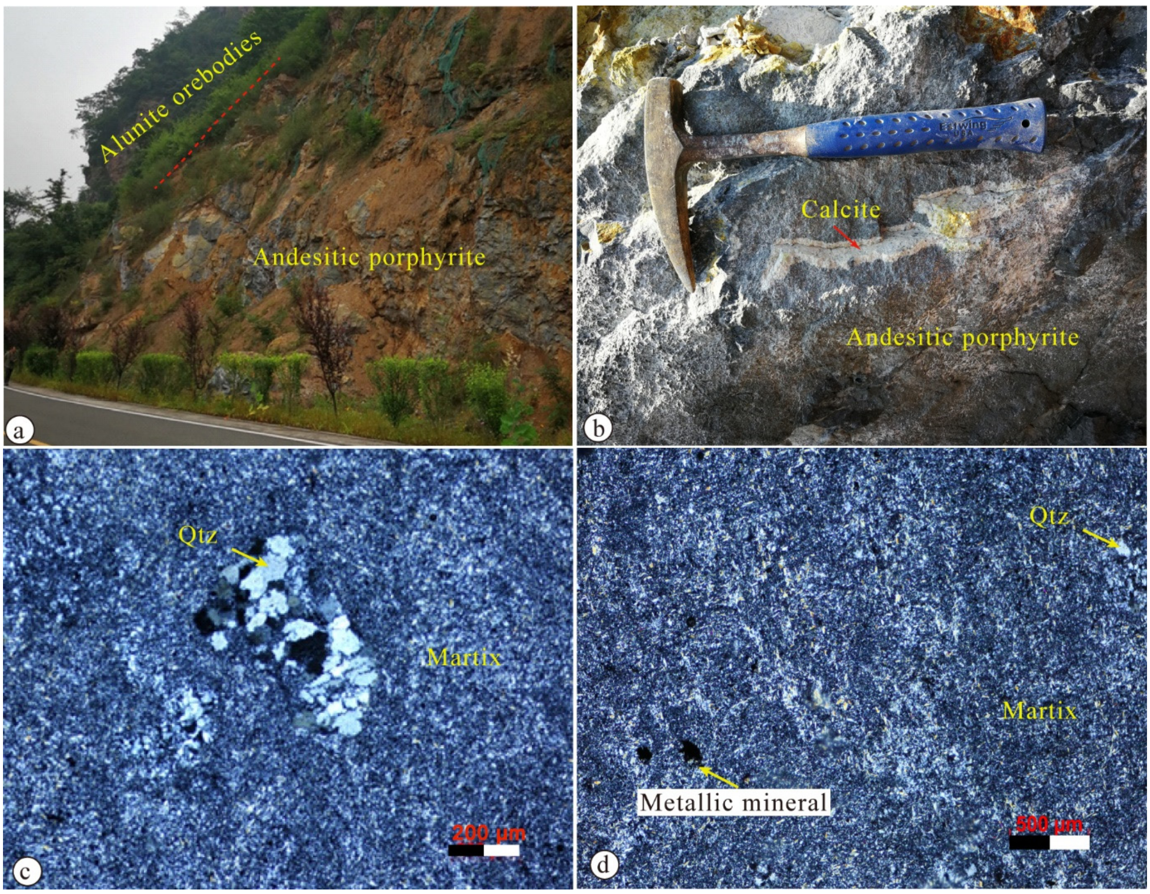

Figure 3. Field (**a**,**b**) and microscope photographs (**c**,**d**) for the Saozhouhe andesitic porphyrite.

3. Sampling and Analytical Methods

The andesitic porphyrite and alunite ore samples were collected from the Saozhouhe alunite deposit, and the sampling locations are shown in Figure 2. These andesitic porphyrite samples are mostly grey in color and of porphyritic texture (Figure 3b). Phenocrysts are mainly composed of subhedral columnar quartz with a grain size of 0.25–4 mm (Figure 3c). Matrix is also composed of microcrystalline feldspar, whose grain size is 0.01–0.05 mm (Figure 3c,d). In addition, some metallic minerals were also identified (Figure 3d).

Zircon grains were firstly separated from the andesitic porphyrite by conventional magnetic and heavy liquid techniques before they were hand-picked under a binocular microscope. They were then mounted into epoxy resin blocks and polished to obtain flat surfaces. CL imaging technique and U–Pb dating was carried out using the laser ablation–inductively coupled plasma mass spectrometry (LA–ICP-MS) method at the Chinese Academy of Geological Sciences, Beijing, China. The instrument of a Bruker M90 Quadrupole ICP-MS coupled to a Resolution S-155 193 nm Excimer laser ablation system was used for the analyses. Standard zircon GJ-1 and standard silicate glass (NIST SRM610) were applied to be external standards for dating and trace element analysis. To enhance the transport efficiency of the ablated material, Helium was used as the gas carrier. This carrier gas was mixed with argon inside the ablation cell as a makeup gas before entering the ICP to maintain stable and optimum excitation conditions. Individual analyses are presented with a 1σ error in data tables and Concordia diagrams, and uncertainties in age results are quoted at a 95% level (2σ). Quantitative calibration for zircon U–Pb dating was performed by ICPMSDataCal 10.7 [18]. Weighted mean age calculation and Concordia diagrams were conducted with the help of an ISOPLOT program (version 3.0) [19].

The alunite samples were firstly crushed and then hand-picked under a binocular microscope. After that, these alunite samples were wrapped in aluminum foil and packed in the quartz tube. Several neutron fluence monitors ZBH-25 biotite were also inserted into the quartz tube for the calculation of the fluence gradient. The quartz tube was then vacuum-sealed and irradiated for 15 h in the HFETR facility of the Nuclear Power Institute of China. The analysis of argon isotopes of the irradiated samples was carried out by Argus VI noble gas mass spectrometer at Beijing Research Institute of Uranium Geology, Beijing, China. The sample was step-heated by a double vacuum tantalum furnace. Of all the gas released, the reactive gas was removed by a stainless U-type trap immersed in liquid nitrogen and two Zr–Al getters (one at 450 °C and the other at 25 °C), and only the noble gas was inlet into the noble gas mass spectrometer for argon isotope analysis. The interferences correction values of the reactor derived from irradiated CaF_2 and K_2SO_4 are $(^{36}Ar/^{37}Ar)ca = 3.539 \times 10^{-4}$, $(^{36}Ar/^{37}Ar)ca = 8.735 \times 10^{-4}$, and $(^{40}Ar/^{39}Ar)k = 7.98 \times 10^{-3}$, respectively. J values for each irradiation position were calculated from the $^{40}Ar^{*}/^{39}Ar$ ratios by total fusion of the ZBH-25 biotite, using an assumed age of 132.9 ± 1.3 Ma [20]. The detailed analytical procedures are given in [21]. The ^{40}Ar-^{39}Ar age of the sample was calculated using the ArArCALC 2.40 software that was written by [22].

4. Results

The LA–ICP-MS zircon U–Pb dating result is shown in Table S1 of the Supplementary Materials. These euhedral zircon grains are mostly buff and/or colorless, with a length of 50–100 μm, a width of ca. 50 μm, and aspect ratios of 3:1 to 1:1. In addition, Cl imaging shows that these zircon grains have oscillatory zoned rims, showing a magmatic origin (Figure 4). Th/U ratios range from 1.2 to 2.2, which also indicates that these zircon grains were crystallized from a magmatic source. Eighteen analyses have constant $^{207}Pb/^{235}U$ and $^{206}Pb/^{238}U$ ages, which range from 121.0 Ma to 142.1 Ma and from 120.2 Ma to 125.3 Ma, respectively. In addition, these analyses were plotted on or near the concordant line (Figure 5). In addition, these $^{206}Pb/^{238}U$ ages yield a weighted average $^{206}Pb/^{238}U$ age of 122.8 ± 0.9 Ma (MSWD = 1.8; Figure 5).

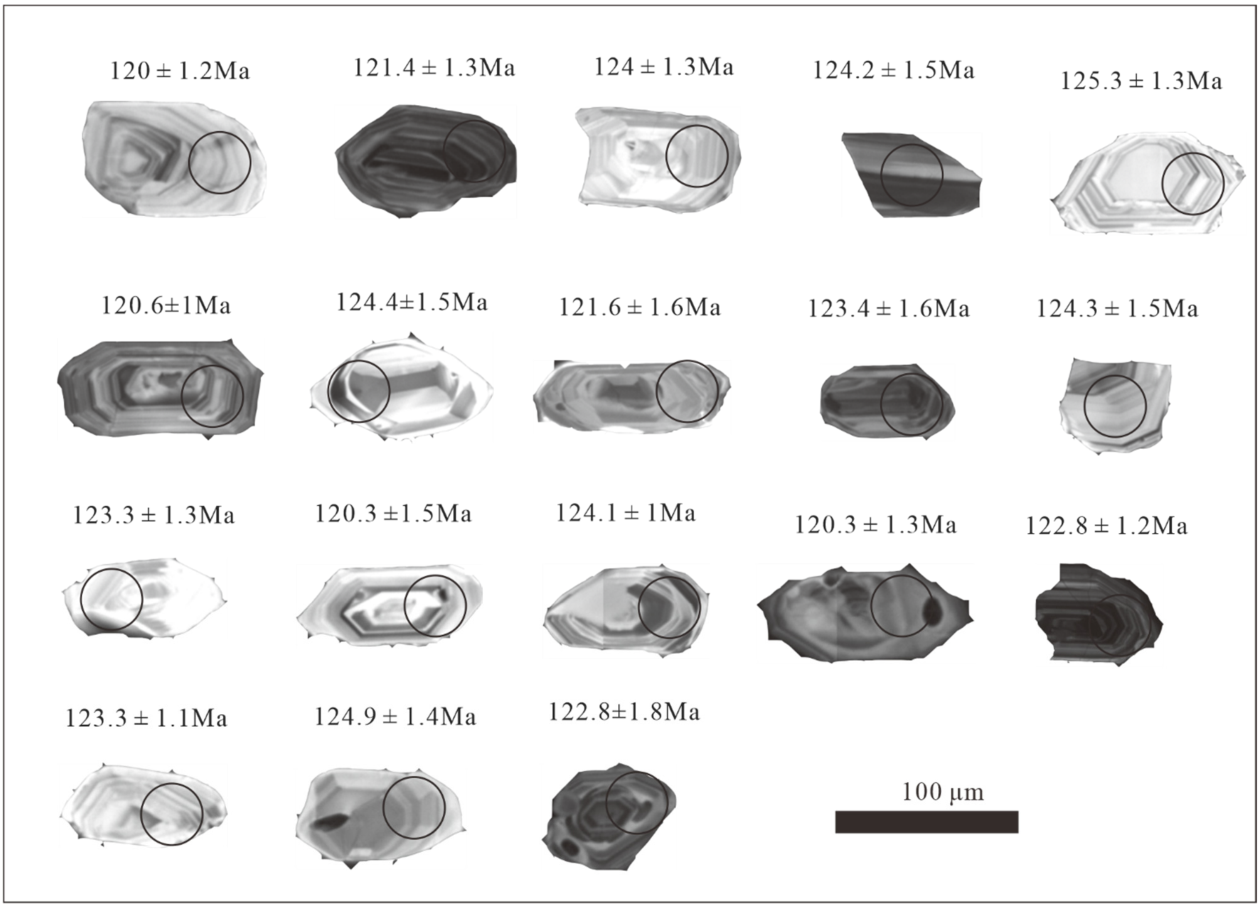

Figure 4. Image of cathode luminescence for determination of zircon.

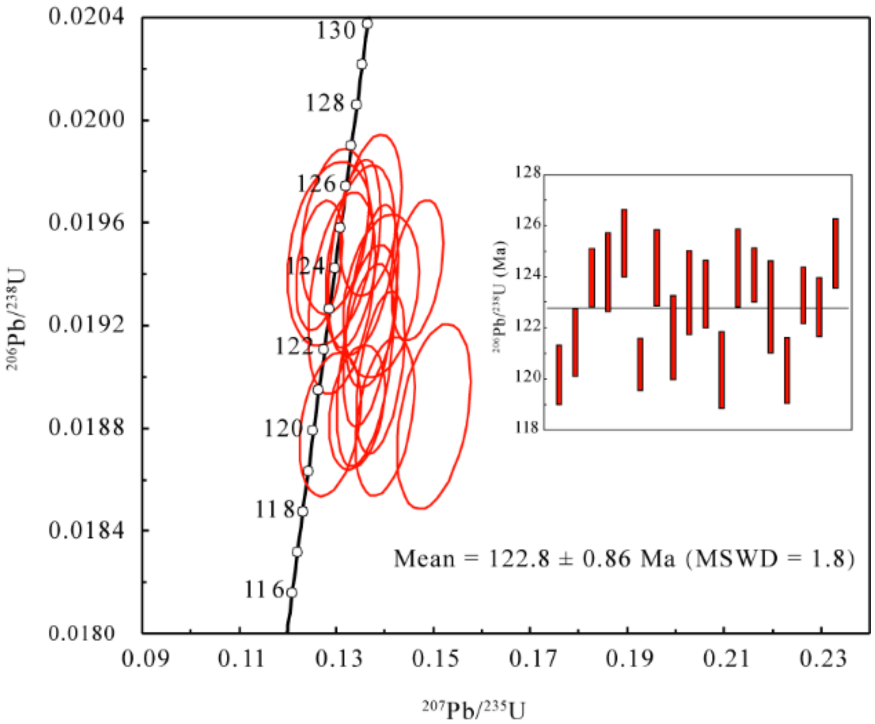

Figure 5. LA–ICP-MS zircon U–Pb age for the Saozhouhe andesitic porphyrite.

The $^{39}Ar/^{40}Ar$ isotopic data for the alunite sample of the Saozhouhe deposit are represented in Table S2 of Supplementary Materials. Four heating stages were carried out in this stage, which were 650 °C, 700 °C, 750 °C, and 900 °C. Since the ^{39}Ar of the alunite sample is released at 650–750 °C, the stages from 650–750 °C obtain the flat age spectrum, yielding a plateau age of 121.0 ± 1.1 Ma (MSWD = 4.4; Figure 6a) with 89.5% release of ^{39}Ar. In addition, the plateau age is consistent with the isochron age of 121.2 ± 1.9 Ma (MSWD = 8.5; Figure 6b), confirming that this age is reliable.

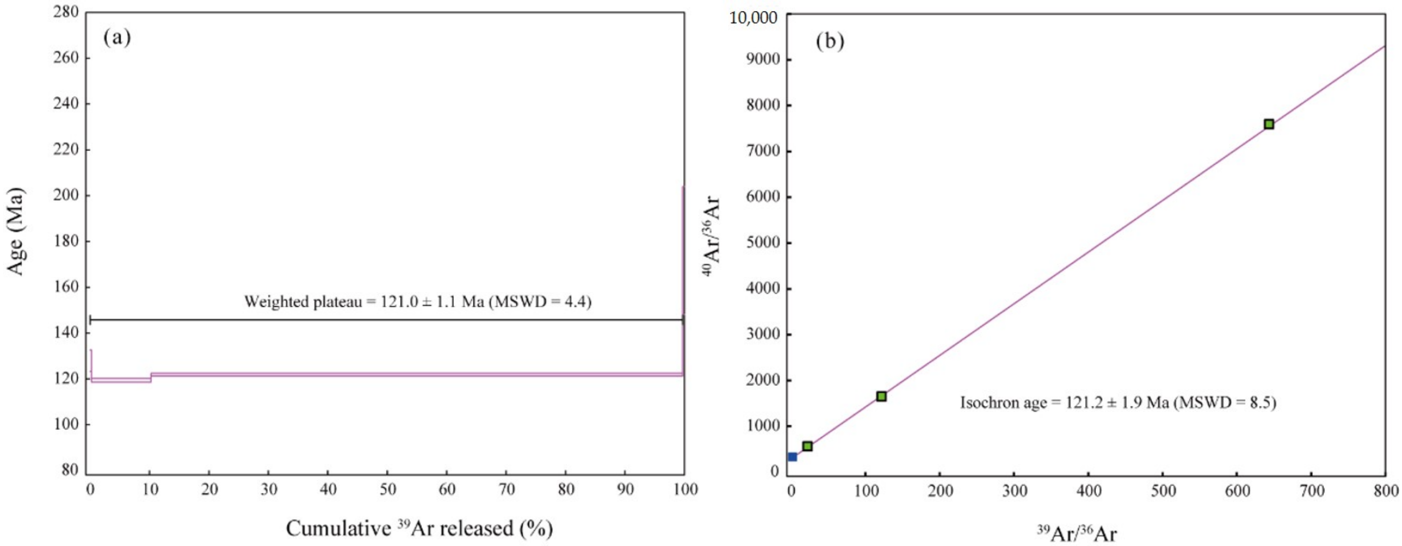

Figure 6. $^{39}Ar/^{40}Ar$ age spectra (**a**) and isochron (**b**) for alunite sample from the Saozhouhe deposit.

5. Discussion

The north Huaiyang Belt experienced intense volcanism during the Mesozoic period, since this period is mostly regarded as the transition from the Tethys domain to the Pacific domain [16,23]. The continuous subducted event of the Pacific plate results in the intense lithosphere extension and asthenosphere upwelling of south China and encourages partial melting of the lower crust and generation of the related magmatism [24,25]. Then, the following large-scale delamination of the lower crust of south China causes not only the formation of widespread Early Cretaceous volcanic basins in the north Huaiyang region and other regions of the middle–lower Yangtze River region, but also a series of gold and alunite deposits in the north Huaiyang region [9–17]. Previous studies indicate that three volcanic eruption cycles from early stage to late stage occurred in the north Huaiyang region, i.e., Maotanchang, Xiaotian, and Xianghongdian cycles. Furthermore, the volcanic rocks of these cycles were formed by the same magma chamber [26–28]. In addition, a series of evidence shows that the volcanic rocks from these cycles are products of continuous volcanism [29]. Recent high-precision zircon U–Pb dating data of these volcanic rocks in this region show that the intense volcanic eruption likely occurred within the range of 136–126 Ma [15,17–33]. The similar ages of these rocks also support the viewpoint that rocks of the Maotanchang, Xiaotian, and Xianghongdian cycles might be formed at the same time. In this study, a new zircon age of the andesitic porphyrite (122.8 ± 0.9 Ma) is reported, which is much younger than previously reported data. It is suggested that it is likely the product of the final stage of the Cretaceous volcanism in the Xiaotian Basin.

Previous studies mainly focused on the rock-forming ages of these volcanic rocks, with very limited works conducted on the gold- and alunite-deposits in the volcanic basins of the north Huaiyang region, meaning that the genesis of the gold- and alunite-deposits in these basins remains unclear. Based on the detailed geological investigation, we found that the andesitic porphyrite coexists with the Saozhouhe alunite deposit, showing a genetic relationship between the andesitic porphyrite and the Saozhouhe alunite deposit. In this work, we reported the alunite $^{39}Ar/^{40}Ar$ isotopic age of 121.0 ± 1.1 Ma, which is the first detailed ore-forming age in the Xiaotian Basin. This $^{39}Ar/^{40}Ar$ isotopic age is consistent with the zircon U–Pb age of the andesitic porphyrite in this study, further confirming that the formation of the Saozhouhe alunite deposit is genetically related to the surrounding andesitic porphyrite.

The Mesozoic period is an important period for south China since it was a conversion period from the Tethyan tectonic domain to the Pacific tectonic domain [16,23]. The continuous subduction of the Pacific plate resulted in the intense lithosphere extension and asthenosphere upwelling, which, in turn, caused intense partial melting and delamination of the thickened lower crust [24,25]. In response to these tectonic events, a series of Cretaceous volcanic basins was formed in the north Huaiyang belt, with the involvement of intense volcanic activities, intermediate-acid magmatic activities, and metallogenic activities [9,13,34]. Besides the epithermal Au and alunite deposits, the north Huaiyang region also hosts several small to super-large Cu–Mo–Pb–Zn deposits, e.g., the super-large Shapinggou Mo deposit, Qingshan Mo deposit, and Yinshan Pb–Zn deposit (Figure 1). In addition, recent studies also indicate that the north Huaiyang region shows great prospecting potential for Cu–Au deposits [10,35]. Together with the previously reported data, the age of the andesitic porphyrite indicates that the final volcanic activities in the Xiaotian Basin occurred at ca. 120 Ma. Meanwhile, the formation of the Saozhouhe alunite deposit responded to final volcanic activities in the Xiaotian Basin. In addition, similar to the Saozhouhe alunite deposit, other gold deposits in the Xiaotian Basin, e.g., Longxing, Daijiahe, and Lianhuadi, are also spatially related to the andesitic porphyrite, which might also indicate a genetic relationship between the formation of these gold deposits and the andesitic porphyrite in the Xiaotian Basin. This finding highlights that the gold and alunite deposits in the Xiaotian Basin are closely related to the Cretaceous volcanic activities.

6. Conclusions

The zircon age for the andesitic porphyrite of the Xiaotian Cretaceous Basin is 122.8 ± 0.9 Ma, which is consistent with the alunite $^{39}Ar/^{40}Ar$ isotopic age of 121.0 ± 1.1 Ma. Our finding confirms a genetic relationship between the Saozhouhe alunite deposit and andesitic porphyrite in the Xiaotian basin, and provides great significance for metallogenetic regularity and further ore prospecting in the Xiaotian basin.

Supplementary Materials: The following are available online at https://www.mdpi.com/article/10.3390/min12091122/s1, Table S1: U–Pb isotopic ages of andesitic porphyrite from the Saozhouhe Xiaotian Basin, Table S2: $^{40}Ar/^{39}Ar$ laser stepwise heating analytical data for the alunite sample from the Saozhouhe deposit.

Author Contributions: Q.W. and J.C. conceived and designed the experiments; C.D., K.S., and L.W. took part in the field campaigns; Y.L. and J.D. took part in the discussion; K.S. and L.W. analyzed the data; Q.W. and J.C. wrote the paper. All authors have read and agreed to the published version of the manuscript.

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