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SIMS U-Pb Dating of Uraninite from the Guangshigou Uranium Deposit: Constraints on the Paleozoic Pegmatite-Type Uranium Mineralization in North Qinling Orogen, China

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Abstract: Pegmatite-type uranium mineralization occurs in the Shangdan domain of the North Qinling Orogenic Belt, representing a significant uraniferous province. The Guangshigou deposit is the largest U deposit of the district. Within the North Qinling area, a series of Caledonian granitic igneous rocks intruded the Proterozoic metamorphic rocks of the Qinling Group in two magmatic stages: (i) the Early Silurian Huichizi granite that was derived from a low degree of partial melting of thickened lower basaltic crust combined with mantle-derived materials following the subduction of the Shangdan Ocean; and (ii) the Late Silurian–Early Devonian Damaogou granite and associated pegmatites derived from the same source but emplaced in a late tectonic post-collisional extension environment. In the Guangshigou deposit, the U mineralization mainly occurs as uraninite disseminated in U-rich granitic biotite pegmatites, which formed by assimilation-fractional crystallization magmatic processes. Petrographic observations showed evidence for coeval crystallization of uraninite and other rock-forming minerals of the host pegmatite including quartz, feldspar, biotite, zircon, monazite, apatite, and xenotime. In addition, the low U/Th ratios (~19) and Th, REE, and Y enrichments characterized a magmatic origin for uraninite, which was likely derived from fractionated high-K calc-alkaline pegmatitic magma that experienced various degrees of crustal material contamination. In situ U-Pb isotopic dating performed by Secondary-Ion Mass Spectrometry (SIMS) on uraninite from the Guangshigou deposit yielded a crystallization age of 412 ± 3 Ma, which is concomitant (within errors) with the emplacement age of the host pegmatite (415 ± 2 Ma) and constrained the U ore genesis to the Early Devonian, which corresponds to the late Caledonian post-collisional extension in the North Qinling area. Uraninite then experienced various degrees of metamictization and/or post-Caledonian hydrothermal alteration characterized by an alteration rim associated with coffinite, chlorite and limonite. Finally, the characteristics of the pegmatite-related Guangshigou deposit exhibiting Th-rich uraninite which was the product of assimilation-fractional crystallization of pegmatitic magma defined a model significantly different than the one established for the world-class Rössing deposit characterized by Th-poor uraninite hosted in alaskite dykes formed by low degree of partial melting of U-rich metasediments.

Keywords: late Caledonian; uraninite; SIMS; Guangshigou; uranium mineralization; North Qinling

1. Introduction

In intrusive type uranium deposits, the uranium mineralization forms at high temperature in magmatic systems such as pegmatite, alaskite, and granite [1–3], and is typically characterized by magmatic uraninite. Uranium-bearing granitic pegmatites mostly occur in

ancient cratons and orogenic belts, and are commonly associated with granitic plutons [4,5]. Around the world, pegmatite-type uranium deposits have been reported in the Damara Orogen in Namibia [6–9], in the Bohemian Massif [10] and the Sierra Albarrana [11] in the European Variscan Belt, in the Grenville Orogen [12] and Trans-Hudson Orogen [13–15] in Canada, in the Bhilwara district in India [16], and in Brazil [17]. Among the pegmatite-type uranium deposits, the world-class Rössing deposit in Namibia is the largest one with identified in situ resources of ~108,000 tU at an average grade of 0.024% U [3]. At Rössing, the uranium mineralization is closely related to the late-tectonic evolution of the Rössing Dome in the Central Zone of the Pan-African Damara Orogen [18], and is mainly hosted in alaskites (i.e., leucocratic granite/pegmatite dykes) that intruded in metasedimentary rocks of the Khan and Rössing formations [17,19,20].

In China, pegmatite-type uranium deposits are mainly located in the northern Alxa Block of the Qilian Orogenic Belt and the eastern section of the North Qinling Orogenic Belt (NQOB) (Figure 1a,b) [21–25]. The Shangdan triangular domain in the NQOB represents an important U ore field with five pegmatite-hosted U deposits of various sizes such as the Guangshigou deposit, the largest with >3000 t U metal [23], and other more modest deposits including Xiaohuacha, Zhifanggou, Chenjiazhuang, and Shibangou [22] (Figure 1). Caledonian granites and pegmatites, forming a WNW-trending granitoid belt, are present throughout the district. These granitic intrusions were formed in syn- and late-tectonic environments, which recorded the evolution of the orogenic belt from crustal thickening by continental collision to subsequent crustal uplift during the Early Paleozoic [26].

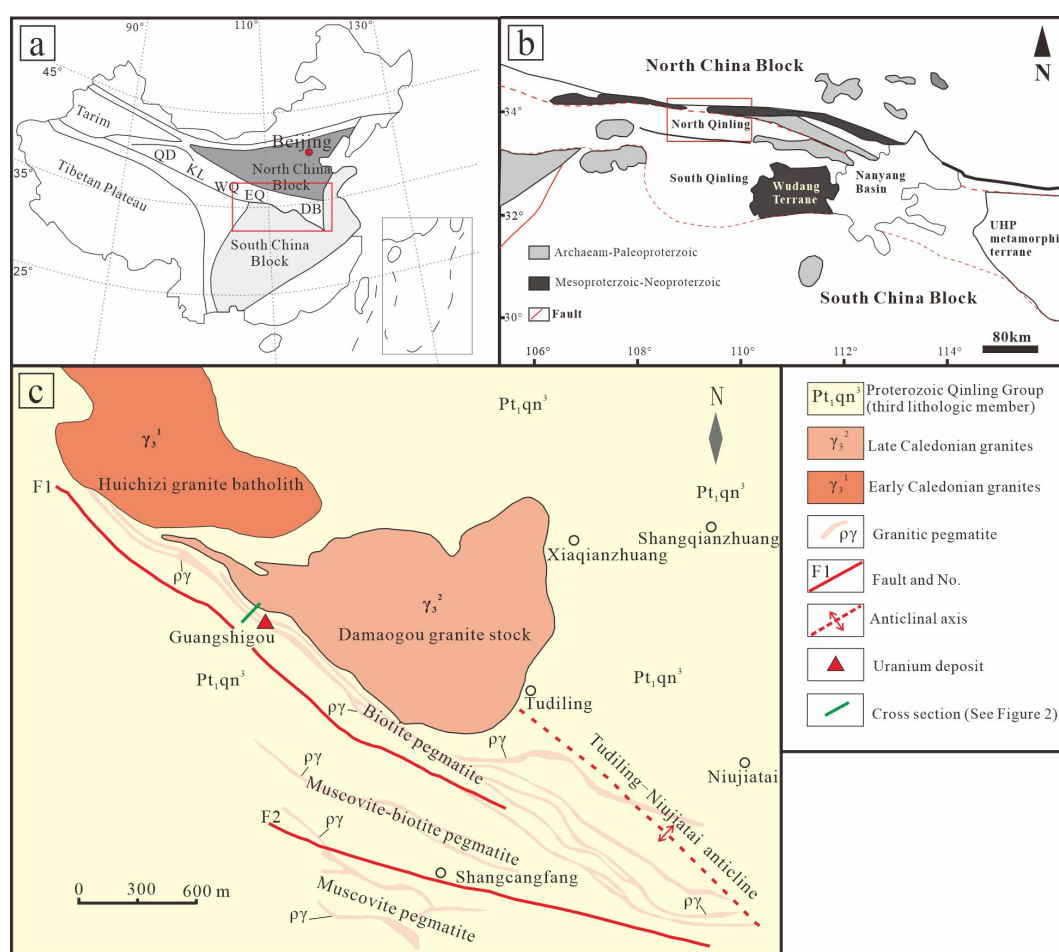


Figure 1. (a) The main geotectonic units of China with the location of the Qinling orogen. (b) Simplified geotectonic map of the Qinling orogen showing the location of the Guangshigou uranium district, modified after [27]. (c) Geological map of the Guangshigou uranium deposit, modified after [28].

Recent studies characterized the Guangshigou deposit as a pegmatite-type uranium deposit, mainly based on geochronological and petrogenetic constraints of the host pegmatites and associated granitic plutons [22,25]. However, the synchronous timing between the host pegmatite and the U mineralization has not been accurately demonstrated to date, which would be necessary to constrain the magmatic origin of uraninite in the Guangshigou deposit and pegmatite-type U ore genesis within the NQOB. Indeed, U-Pb dating on zircon constrained the crystallization age of the ore-bearing biotite granitic pegmatites at 415 ± 2 Ma in the Guangshigou uranium deposit [22], while Isotope Dilution-based Thermal Ionization Mass Spectrometry (ID-TIMS) analyses on uraninite yielded U-Pb ages broadly ranging from 380.4 Ma to 426.7 Ma [23]. To date, these results were considered to represent the emplacement ages of the host biotite granitic pegmatites [22,25], suggest that the Guangshigou uranium deposit is a typical magmatic pegmatite-type uranium deposit [22,25].

The geochronological constraint on the uranium mineralization is a critical point to the understanding of a deposit and to propose a metallogenic model that is integrated to the regional geological evolution. A series of different analytical methods can be used to determine the age of the uranium mineralization. For instance, ID-TIMS method can provide high precision and accuracy U-Pb isotopic ratios of uraninite, but it is unavoidable that the resultant age usually overprints the multistage uranium mineralization due to the presence of U and/or Pb-rich mineral inclusions [29,30]. U-Pb ages of magmatic zircon may constrain the emplacement age of the host pegmatite but cannot be directly considered as the crystallization age of the uranium mineralization. Therefore, in situ U-Pb dating of uraninite would certainly provide a new insight for understanding the genetic history of the Guangshigou U ore deposit. Different analytical techniques may allow in situ age determination on uraninite grains including: (i) Electron Probe Microanalyzer (EPMA), (ii) Secondary-Ion Mass Spectrometry (SIMS), (iii) Sensitive high resolution ion Microprobe (SHRIMP), and (iv) laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS). EPMA allows U-Th-Pb chemical dating based on the assumption that Pb derived solely from the radioactive decay of ^{235}U , ^{238}U , and ^{232}Th [28,31,32], and uraninite did not experience post-crystallization alteration [33] that could be responsible for radiogenic Pb loss. Moreover, even though the average error for U-Th-Pb chemical dating on uraninite has been restrained to ca. 25 Ma by repeated measurements and the counting statistics [34], it still remains relatively high compared with other techniques allowing in situ U-Pb isotopic age determination. For example, LA-ICP-MS technique is a rapid and accurate method for analyzing the U-Pb isotope systematics in uraninite, which usually requires spot size of several tens of microns [29,35]. SIMS can also provide in situ measurement of isotopic ratios but with a spatial resolution on the scale of a few microns [36,37], and many cases of high-precision SIMS dating on uraninite are reported in the literature [36–42], which is why we chose to perform in situ U-Pb isotopic dating by SIMS on uraninite from the Guangshigou deposit. The dating of uraninite from the Guangshigou uranium deposit has previously been performed in several studies using analytical methods such as ID-TIMS, EPMA, and LA-ICP-MS [22,23,28,43], with results broadly ranging from 380 Ma to 430 Ma (Table 1). The present study aims to characterize the chemical and isotopic signatures of the uranium mineralization from the Guangshigou uranium deposit, also providing a new U-Pb isotopic age by SIMS on uraninite. Ultimately, the new geochronological constraint provided by this paper will allow refining the metallogenic model for Paleozoic pegmatite-type uranium mineralization in the NQOB, China.

Table 1. U-Pb age on uraninites in Guangshigou uranium deposit.

No.	Sample	Method	U-Th-Pb Age Ranges on Uraninites	Reference
1	YD-138	ID-TIMS U-Pb Dating	380.40–426.7 Ma, average age of 403.55 Ma	Feng et al. [23]
2	8001G5-6	Chemical U-Th-Pb dating by EPMA	382.0–441.4 Ma, average age of 416.9 ± 10 Ma	Guo et al. [28]
3	8001G5-7	Chemical U-Th-Pb dating by EPMA	366.2–430.0 Ma, average age of 407.6 ± 8 Ma	Guo et al. [28]
4	Group-1	Chemical U-Th-Pb dating by EPMA	330–430 Ma, average 410–420 Ma	Yuan et al. [22]
5	GSG-1	LA-ICP-MS U-Pb dating, $^{206}\text{Pb}/^{238}\text{U}$	401–409 Ma, concordant age 405 ± 3 Ma	Wu et al. [43]
6	GSG-25	LA-ICP-MS U-Pb dating, $^{206}\text{Pb}/^{238}\text{U}$	396–408 Ma, concordant age 403 ± 3 Ma	Wu et al. [43]

2. Geological Setting

The Qinling orogenic belt in central China constitutes a major collisional orogenic belt, which was formed by the convergence and collision between the North China and South China Blocks [44,45]. It extends for ~1500 km from the Qilian-Kunlun Orogen in the west to the Dabie-Sulu Orogen in the East (Figure 1a). The Qinling Orogen is divided by the Shangdan suture zone into the North Qinling Orogen and the South Qinling Orogen including the Qinling micro-continental terrane and the Northern margin of the Yangtze Block (Figure 1b) [22,46]. The Shangdan suture zone (Figure 1b), considered to have formed following the northward subduction of the Shangdan Ocean during the Early Silurian, is characterized by a linear, patchy distribution of ophiolites and arc-related volcanic rocks [47,48].

The NQOB is bounded to the north by the Luonan-Luanchuan fault (Figure 1b) and to the south by the Shangdan suture zone [49,50]. It is characterized by long-term subduction to collision along its southern edge during Early Paleozoic [51–53]. The structure of the NQOB is dominated by thick-skinned crustal deformation and south-verging imbricated thrustfold system [26,54]. It is subdivided from north to south into several lithological units, including the Kuanping Group, Erlangping Group, Qinling Group, Songshugou Complex, and Danfeng Group, which are separated from each other by thrust faults or ductile shear zones [55,56]. The Shangdan triangular domain that represents a productive uraniferous province exhibiting several Caledonian pegmatite-type U deposits [23,24,57] is located in the eastern segment of the NQOB. It is bounded by the Caichuan Fault (Figure 1b) to the north and the Fenshuiling Fault (Figure 1b) to the south.

The eastern extension of the NQOB that borders the southern margin of the North China Block (Figure 1b) is characterized by a crystalline basement composed of various high-grade metamorphic rocks of medium-low pressure granulite-amphibolite facies belonging to the Lower Proterozoic Qinling Group. These rocks include graphite-bearing and biotite-plagioclase gneisses, graphite-bearing quartz and amphibole-biotite schists, graphitic marble, granulite, and amphibolite [22,24,51], which have been interpreted as a result of multi-stage metamorphism–deformation–migmatization [58]. The U mineralization of the Guangshigou deposit in the Shangdan domain is hosted in Caledonian granitic pegmatite dykes that intruded into these Proterozoic metamorphic rocks [23], especially in biotite-plagioclase gneiss that is the predominant rock type of the Qinling Group and the main wall rock of pegmatite dykes [25]. Based on previous geochronological and structural studies, Caledonian plutons that intruded the Qinling and Danfeng Groups were emplaced in two stages [22], during the Early Silurian and Late Silurian–Early Devonian: (i) Early Silurian granitoids, represented by the Huichizi (434 ± 7 Ma; [52]), Kuanping (452.8 ± 2 Ma; [59]), Shangnan (428 ± 7 Ma; [52]), Huanglongmiao and Zaoyuan (457 Ma and 444 Ma, respectively; [23]) plutons, formed in a collisional and a following uplifting setting [52,59,60]. After emplacement of the Early Silurian plutons, regional deformation was initiated along ductile shearing zones that had little influence on the early regional structures [61,62]; and (ii) Late Silurian–Early Devonian granitoids occur as high-K calc-alkaline granites accompanied by ductile deformation and undeformed pegmatites, which are mainly represented by the Chenjiazhuang granite (415 ± 27 Ma; ref. [63]), Zhifanggou granite and Chenjiazhuang biotite pegmatite (411 Ma and 407 Ma,

respectively; ref. [64,65]. These granitoids formed after the main collisional deformation associated with the NQOB [66–68].

The Guangshigou U deposit is located in the eastern part of the triangular Shangdan domain (Figure 1). U-bearing granitic pegmatites are spatially distributed along the contact zone with biotite gneiss of the Qinling Group in the southern margin of the Huichizi granite batholith and Damaogou granitic stock (Figure 1c), which controls twenty-four orebodies with an average ore grade of 933 ppm U [22]. The granitic pegmatites in the Guangshigou U ore field show three petrographic facies that are concentrically arranged around the Damaogou granite stock and Huichizi granite batholith, varying from (i) an inner zone of biotite granitic pegmatite near the intrusions and associated with the U mineralization [25], through (ii) an intermediate zone of muscovite-biotite granitic pegmatite, and to (iii) an outer zone of muscovite granitic pegmatite which are barren [24,25,69]. The Huichizi granitic batholith consisting of biotite granodiorite and biotite granite, the Damaogou granitic stock and the granitic pegmatites emplaced during the Caledonian magmatic event that affected the NQOB, as indicated by U-Pb isotopic ages on zircon of 422–441 Ma [22,70], 418–420 Ma [22,27], and 412.1–415.6 Ma [22], respectively.

The orebodies, appearing in veined, lenticular, and stratoid shapes are parallel or subparallel to each other and extend over 80–777 m in length, with an average thickness of 2.33 m (maximum thickness of 6.18 m), but display discontinuous mineralization along the pegmatite strike [22] (Figure 2). The U mineralization is mainly hosted in biotite granitic pegmatite which commonly occurs as dykes at the axes of anticlines and/or along fractures cutting Caledonian granites and Proterozoic metasedimentary rocks [24]. The extensive biotite granitic pegmatite dykes vary from 500 to 2000 m in length and 1 to 30 m in width. They usually are parallel to the direction of the regional tectonic fabric ranging from 290° to 320° (Figure 1b). The pegmatite-hosted U mineralization of the Guangshigou deposit is mainly represented by disseminated crystals of uraninite which are preferentially concentrated along the contact boundary between the biotite granitic pegmatite and metasedimentary rocks of the Qinling Group.

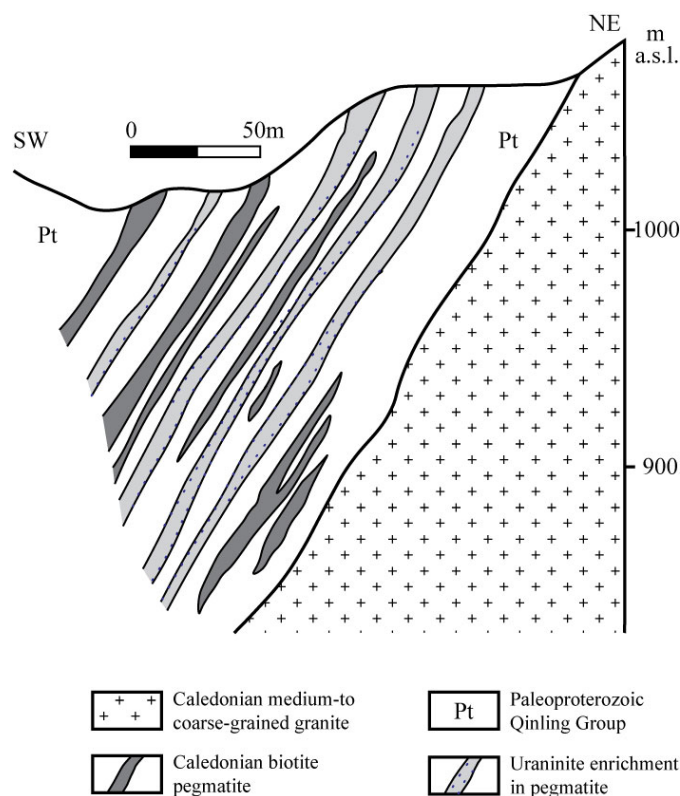


Figure 2. Deposit cross section in Guangshigou U deposit, modified after [24].

3. Sampling and Analytical Methods

Seven representative hand specimens of uranium ore were taken from underground mine tunnels of the Guangshigou deposit, as shown in Figure 1b. The mineralogical and petrographic studies were carried out at the State Key Laboratory of Nuclear Resources and Environment (SKLNRE) of the East China University of Technology (ECUT, China) using optical microscope (transmitted and reflected lights) and an electron probe micro-analyzer (EPMA). Uraninite separation from the Guangshigou deposit was carried out on the samples using conventional density, magnetic techniques and handpicked under a binocular microscope. The selected uraninite grains were cast into an epoxy disc and polished approximately to expose the grain centers on which were performed EPMA and secondary-ion mass spectrometry (SIMS) measurements.

Chemical composition and back-scattered electron images of the studied samples were analyzed by a wavelength dispersion spectroscopy (JXA-8100 Electron Probe Microanalyzer, EPMA) at the SKLNRE, ECUT. The analytical conditions were 15 kV accelerating voltage, 20 nA beam current, 5 μm beam diameter, and 20 s counting times per element. For chemical dating, the counting times for U, Th, and Pb were 120 s, 180 s, and 300 s, respectively. Prior to analysis, thin sections were examined under an optical microscope in order to select areas of interest with uraninite grains, and then were carbon-coated to create a conductive surface in a vacuum carbon-coating instrument. Well-characterized natural minerals and synthetic oxide standards were used for calibration. Raw data were reduced with an online correction procedure, including background, dead time and a ZAF calculation. Detection limits of the major and trace elements were <0.1 wt%. The oxygen contents of uraninite were calculated by stoichiometry assuming an ideal composition of UO_2 . A natural uraninite with the UO_2 , ThO_2 , and PbO concentration of 90.20 wt%, 6.20 wt%, 1.75 wt%, respectively, was selected as standard for U-Th-Pb ages calculation [71].

U-Pb isotopic compositions of uraninite were determined using a CAMECA IMS 1280-HR ion microprobe at Centre de Recherches Pétrographiques et Géochimiques (CRPG-CNRS, Nancy, France). The O^- primary ion beam was accelerated at 13 kV, with an intensity ranging between 3.5 and 5 nA. The primary beam was set in Gaussian mode with a raster of 10 μm . The size of the spot on the uranium oxides was ~ 15 μm . Positive secondary ions were extracted with a 10 kV potential, and the spectrometer slits were set for a mass resolving power of ~ 6000 to separate isobaric interferences of rare earth element (REE) dioxides from Pb. The field aperture was set to 2000 μm , and the transfer optic magnification was adjusted to 80. Rectangular lenses were activated in the secondary ion optics to increase the transmission at high mass resolution. The energy window was opened at 30 eV, and centered on the low energy side, 5 eV before the maximum value. An offset of 50 eV was applied during analyses in order to avoid matrix effects due to mixing between uraninite and silicate in natural samples. Ions were measured by peak jumping in monocollection mode using the axial Faraday cup (FC) for ^{238}U and ^{238}UO and the axial electron multiplier (EM) for ^{204}Pb , ^{206}Pb , ^{207}Pb , ^{208}Pb , and ^{248}ThO . Each analysis consisted of 8 successive cycles. Each cycle began with measurement of the mass 203.5 and 203.6 for backgrounds of the FC and the EM respectively, followed by ^{204}Pb , ^{206}Pb , ^{207}Pb , ^{208}Pb , ^{238}U , ^{248}ThO , and ^{238}UO , with acquisition times of 4, 4, 10, 6, 20, 4, 4, 3, and 3 s, respectively (waiting time of 1 s). In order to achieve good reproducibility, automatic beam centering within the field and the contrast apertures and energy and mass calibrations were performed before each measurement, after a 60 s presputtering by rastering the primary beam over a $30\text{ }\mu\text{m} \times 30\text{ }\mu\text{m}$ area to clean the gold coating and avoid contamination. The relative sensitivity factor between Pb and U used for unknown samples was determined from an empirical linear relationship defined between UO^+/U^+ and Pb^+/U^+ from all the measurements performed on the reference uraninite. The standard used was a uraninite sample from Zambia [72], which has a given concordant age at 540 ± 4 Ma [72,73]. Raw age data of the Zambia uraninite standard obtained during the SIMS analytical session are presented in Table 2 and internal precisions were 0.1% and 0.01% for $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{206}\text{Pb}$ ratios, respectively. It is true that because only one reference material with a

U/Pb ratio distinct from the analyzed samples has been used, the correction for mass bias may be slightly erroneous and consequently so might be the estimated age [39]. However, such slight bias does not change the aim of the present study. Initial data treatment, standardization against Zambia uraninite, calculations of U/Pb and Pb/Pb ratios and related ages, and calculation of errors were done using an in-house Excel spreadsheet at the CRPG and previously used for all peer-reviewed publications based on U-Pb SIMS analyses of uranium oxides and zircons carried out at the CRPG for more than 25 years. The error on the calibration curve is reported in the error given for each analysis. Ages and error correlations were calculated using the ISOPLOT flowsheet of [74]. Uncertainties in ages are reported at the 2σ level.

4. Results

4.1. Petrography of the Uranium Mineralization

In the Guangshigou deposit the U mineralization is dominantly hosted in biotite granitic pegmatite. The pegmatites are mainly grey in color and present a medium-grained to pegmatitic texture. Their main mineral assemblage is quartz, biotite, K-feldspar (microcline), and plagioclase (oligoclase to albite). Their accessory mineral assemblage consists of zircon, apatite, monazite, xenotime, and uraninite (Figure 3). The U mineralization in the Guangshigou deposit is mainly characterized by uraninite usually occurring as discrete grains disseminated in pegmatite. Most uraninite grains show euhedral to subhedral crystal shape with a grain size of 0.1 mm to 0.6 mm. They occur (i) as inclusions in major rock-forming minerals such as feldspar, quartz, and biotite (Figure 3a,b); (ii) along cracks within pegmatite or interstitial at the grain boundaries between rock-forming minerals (Figure 3c,d); and (iii) preferentially associated with biotite and zircon clusters (Figure 3a). Monazite is commonly closely associated with uraninite (Figure 3c), and zircon, apatite and xenotime show intergrowth relationships (Figure 3a,b) with some inclusions in uraninite crystals (Figure 3a). Coffinite usually occurs as a secondary overgrowth on uraninite grains (Figure 3d). Some uraninite grains also present a texture of metamictization (Figure 3e,f) characterized by a radiation-induced destruction rim associated with alteration minerals such as chlorite and limonite.

4.2. Composition of Uraninite

Prior to in situ mineral analysis, a series of non-metamict uraninite grains with low fracturing degree were selected for EPMA measurements. All grains show a relatively homogeneous texture with no obvious compositional zoning in the back-scattered electron images (Figure 4). The chemical composition of uraninite from the Guangshigou uranium deposit are listed in Table 3. Uraninite with >1 wt% SiO_2 was considered as altered uraninite.

The EPMA data show that the analytical total of uraninite ranges from 80.05 to 99.98 wt%, indicating a slight to significant deviation from the ideal UO_2 formula most likely because some elements were not analyzed by the EPMA or due to post-crystallization alteration (i.e., oxidation and hydration), or a combination of those. Non-altered uraninite (see Urn-01~32 in Table 3, $n = 32$) is characterized by very high UO_2 (mean = 86.03 wt%, Figure 4a) and relatively high PbO (mean = 5.08 wt%, Figure 4d) and ThO_2 (mean = 4.64 wt%, Figure 4a) contents, and variable low to relatively high concentrations in REE ($0.48 < \sum \text{REE}_2\text{O}_3 < 1.48$ wt%, mean = 0.93 wt%), Fe ($\text{LOD} < \text{FeO} < 2.73$ wt%, mean = 0.30 wt%), and Y ($0.10 < \text{Y}_2\text{O}_3 < 0.35$ wt%, mean = 0.23 wt%, Figure 4b). Relatively homogeneous Ca contents (mean = 0.32 wt% CaO, Figure 4f) suggests that calcium was most likely incorporated during crystallization, and very low Si concentrations (mean = 0.07 wt% SiO_2 , Figure 4e) shows evidence for a low degree of post-crystallization alteration. Altered uraninite (see Urn-33~41 in Table 3, $n = 9$) is characterized by relatively high Si contents ($1.34 < \text{SiO}_2 < 5.70$ wt%, Figure 4e), and tends to show lower UO_2 (mean = 69.75 wt%, Figure 4a) and PbO (down to 2.96 wt%, Figure 4d,e), Y_2O_3 (mean = 0.71 wt%, Figure 4b) and CaO (up to 1.91 wt%, Figure 4f) concentrations relatively to non-altered uraninite.

Table 2. SIMS data uraninite grains from Guangshigou uraninite deposit.

Sample	Measurement	Measured Ratios ^a								Calibrated Ratios ^b					U-Pb and Pb-Pb Ages ^c (Ma)					
		²⁰⁷ Pb/ ²⁰⁶ Pb	±	²⁰⁴ Pb/ ²⁰⁶ Pb (×10 ⁻⁷)	± (×10 ⁻⁷)	²⁰⁶ Pb/ ²³⁸ U	±	²³⁸ UO/ ²³⁸ U	±	²⁰⁶ Pb/ ²³⁸ U	±	²⁰⁷ Pb/ ²³⁵ U	±	Correl. Err.	²⁰⁶ Pb/ ²³⁸ U	±	²⁰⁷ Pb/ ²³⁵ U	±	²⁰⁷ Pb/ ²⁰⁶ Pb	±
Standard	Zambia1	0.05850	0.00020	2	4	0.181	0.000	3.26	0.01	0.0892	0.0081	0.720	0.009	0.890	551	4	550	3	-	-
	Zambia2	0.05840	0.00010	6	4	0.161	0.001	3.04	0.01	0.0883	0.0112	0.710	0.011	0.982	545	6	544	4	-	-
	Zambia3	0.05834	0.00012	0	0	0.149	0.001	2.91	0.01	0.0869	0.0118	0.699	0.012	0.984	537	6	538	5	-	-
	Zambia4	0.05831	0.00004	0	0	0.150	0.001	2.93	0.01	0.0874	0.0126	0.699	0.013	0.998	537	6	538	5	-	-
	Zambia5	0.05861	0.00012	0	0	0.166	0.000	3.11	0.01	0.0901	0.0080	0.707	0.008	0.966	540	4	542	3	-	-
	Zambia6	0.05833	0.00008	0	0	0.129	0.000	2.60	0.01	0.0915	0.0087	0.724	0.009	0.985	556	5	553	3	-	-
	Zambia7	0.05843	0.00009	0	0	0.126	0.001	2.54	0.01	0.0890	0.0101	0.737	0.010	0.987	564	5	560	4	-	-
	Zambia8	0.05845	0.00006	2	1	0.446	0.003	6.56	0.02	0.0901	0.0092	0.718	0.009	0.993	550	5	549	3	-	-
	Zambia9	0.05813	0.00011	5	3	0.122	0.001	2.52	0.01	0.0820	0.0161	0.722	0.016	0.992	556	8	552	6	-	-
	Zambia10	0.05825	0.00011	0	0	0.320	0.003	5.33	0.01	0.0869	0.0119	0.659	0.012	0.986	508	6	514	4	-	-
	Zambia11	0.05799	0.00006	0	0	0.125	0.000	2.62	0.01	0.0854	0.0097	0.695	0.010	0.993	537	5	535	4	-	-
	Zambia12	0.05806	0.00015	0	0	0.137	0.000	2.80	0.01	0.0871	0.0062	0.684	0.007	0.918	528	3	529	2	-	-
	Zambia13	0.05793	0.00010	4	2	0.121	0.001	2.56	0.01	0.0864	0.0102	0.696	0.010	0.984	538	5	536	4	-	-
	Zambia14	0.05793	0.00010	0	0	0.135	0.001	2.75	0.01	0.0884	0.0101	0.691	0.010	0.985	534	5	533	3	-	-
	Zambia15	0.05819	0.00009	6	2	0.488	0.003	7.14	0.03	0.0851	0.0090	0.709	0.009	0.983	546	4	544	4	-	-
Grain A	A-1	0.05511	0.00010	95	23	0.085	0.001	2.40	0.02	0.0681	0.0105	0.516	0.011	0.982	425	4	423	4	412	4
	A-2	0.05561	0.00006	107	24	0.091	0.000	2.51	0.01	0.0678	0.0079	0.518	0.008	0.985	423	3	424	3	432	3
	A-3	0.05517	0.00006	125	21	0.088	0.001	2.47	0.02	0.0667	0.0096	0.505	0.010	0.991	416	4	415	3	413	2
	A-4	0.05550	0.00009	120	23	0.126	0.001	3.20	0.01	0.0641	0.0097	0.489	0.010	0.983	401	4	404	3	427	4
	A-5	0.05556	0.00013	102	5	0.300	0.001	6.16	0.03	0.0645	0.0067	0.493	0.007	0.939	403	3	407	2	430	5
	A-6	0.05468	0.00046	130	18	0.187	0.001	4.54	0.02	0.0587	0.0087	0.441	0.012	0.711	368	3	371	4	393	19
	A-7	0.05563	0.00032	86	22	0.097	0.001	2.76	0.03	0.0617	0.0114	0.472	0.013	0.890	386	4	392	4	434	13
	A-8	0.05544	0.00049	91	12	0.109	0.001	3.09	0.03	0.0582	0.0140	0.443	0.017	0.843	364	5	373	5	426	20
	A-9	0.05565	0.00024	76	18	0.086	0.001	2.44	0.02	0.0670	0.0135	0.513	0.014	0.948	418	5	420	5	435	10
Grain B	B-1	0.05597	0.00072	44	12	0.075	0.001	2.84	0.02	0.0457	0.0193	0.352	0.023	0.831	288	5	306	6	450	28
	B-2	0.05515	0.00058	59	12	0.119	0.001	3.83	0.00	0.0467	0.0129	0.355	0.017	0.770	294	4	308	4	416	24
	B-3	0.05520	0.00009	122	27	0.095	0.000	3.12	0.02	0.0499	0.0079	0.379	0.008	0.972	314	2	326	2	414	4
	B-4	0.05526	0.00019	77	22	0.050	0.000	2.05	0.01	0.0537	0.0081	0.408	0.009	0.915	337	3	348	3	419	8
	B-5	0.05550	0.00007	94	14	0.195	0.005	4.90	0.04	0.0554	0.0239	0.423	0.024	0.998	348	8	358	7	428	3
	B-6	0.05565	0.00025	68	14	0.070	0.001	2.31	0.01	0.0599	0.0195	0.459	0.020	0.972	375	7	383	6	436	10
	B-7	0.05540	0.00019	86	21	0.094	0.002	2.92	0.02	0.0546	0.0212	0.416	0.022	0.985	343	7	353	6	425	8
	B-8	0.05537	0.00014	72	16	0.073	0.001	2.42	0.01	0.0574	0.0169	0.437	0.017	0.988	360	6	368	5	424	6
Grain C	C-1	0.05480	0.00018	88	18	0.079	0.000	2.33	0.01	0.0662	0.0082	0.499	0.009	0.921	413	3	411	3	400	8
	C-2	0.05515	0.00015	124	18	0.267	0.004	5.77	0.05	0.0620	0.0178	0.470	0.018	0.987	388	7	391	6	412	6
	C-3	0.05515	0.00013	76	19	0.082	0.001	2.41	0.01	0.0654	0.0094	0.496	0.010	0.963	408	4	409	3	415	6
	C-4	0.05451	0.00019	145	33	0.081	0.001	2.44	0.01	0.0629	0.0117	0.471	0.012	0.953	393	4	392	4	385	8
	C-5	0.05414	0.00049	76	6	0.243	0.003	5.61	0.04	0.0586	0.0143	0.436	0.017	0.844	367	5	368	5	374	20
	C-6	0.05492	0.00070	136	7	0.122	0.002	3.29	0.02	0.0592	0.0163	0.447	0.021	0.786	371	6	375	6	402	28
	C-7	0.05483	0.00015	109	22	0.072	0.001	2.32	0.01	0.0614	0.0112	0.463	0.012	0.965	384	4	386	4	400	6
	C-8	0.05428	0.00075	51	9	0.217	0.004	5.04	0.13	0.0595	0.0189	0.445	0.023	0.806	373	7	374	7	381	31
	C-9	0.05462	0.00040	71	16	0.081	0.001	2.43	0.02	0.0635	0.0124	0.477	0.014	0.858	397	5	396	5	394	16
	C-10	0.05448	0.00042	87	21	0.087	0.001	2.51	0.01	0.0648	0.0144	0.485	0.016	0.878	404	6	402	5	387	17

Table 2. Cont.

Sample	Measurement	Measured Ratios ^a								Calibrated Ratios ^b					U-Pb and Pb-Pb Ages ^c (Ma)					
		²⁰⁷ Pb/ ²⁰⁶ Pb	±	²⁰⁴ Pb/ ²⁰⁶ Pb (×10 ^{−7})	± (×10 ^{−7})	²⁰⁶ Pb/ ²³⁸ U	±	²³⁸ UO/ ²³⁸ U	±	²⁰⁶ Pb/ ²³⁸ U	±	²⁰⁷ Pb/ ²³⁵ U	±	Correl. Err.	²⁰⁶ Pb/ ²³⁸ U	±	²⁰⁷ Pb/ ²³⁵ U	±	²⁰⁷ Pb/ ²⁰⁶ Pb	±
Grain D	D-1	0.05583	0.00092	153	20	0.066	0.001	2.26	0.03	0.0588	0.0203	0.451	0.026	0.771	368	7	378	8	438	37
	D-2	0.05464	0.00065	158	19	0.062	0.001	2.06	0.02	0.0661	0.0111	0.496	0.016	0.675	413	4	409	5	389	27
	D-3	0.05571	0.00104	172	25	0.088	0.001	2.60	0.03	0.0617	0.0154	0.472	0.024	0.629	386	6	392	8	432	41
	D-4	0.05460	0.00053	160	23	0.067	0.001	2.15	0.02	0.0651	0.0107	0.488	0.015	0.731	406	4	403	5	387	22
	D-5	0.05537	0.00117	178	32	0.070	0.001	2.20	0.02	0.0653	0.0132	0.496	0.025	0.524	408	5	409	8	418	47

^a Raw ratios; ^b Ratios corrected common lead calibrated against the uraninite standard (Zambia, dated at 540 Ma); ^c ages calculated from the calibrated Pb/U and Pb/Pb ratios.

Table 3. Representative Electron Probe Microanalyzer (EPMA) analyses (wt%) of uraninite in Guangshigou uranium deposit.

No.	UO ₂	ThO ₂	PbO	SiO ₂	CaO	FeO	La ₂ O ₃	Y ₂ O ₃	Ce ₂ O ₃	Pr ₂ O ₃	Nd ₂ O ₃	Eu ₂ O ₃	Gd ₂ O ₃	Tb ₂ O ₃	Dy ₂ O ₃	Ho ₂ O ₃	Er ₂ O ₃	Tm ₂ O ₃	Lu ₂ O ₃	Yb ₂ O ₃	Total	REE	U/Th	U + Th	U-Th-Pb Ages
Urn-01 *	85.53	4.58	5.17	0.02	0.30	0.27	<0.01	0.19	0.20	<0.01	<0.01	<0.01	0.11	<0.01	0.18	0.11	0.01	<0.01	<0.01	0.11	96.77	0.70	18.3	90.11	430.4
Urn-02	85.73	4.54	5.22	0.05	0.31	0.68	<0.01	0.26	0.15	<0.01	0.04	<0.01	0.17	0.12	0.25	0.14	0.03	<0.01	<0.01	0.03	97.72	0.94	18.5	90.27	433.5
Urn-03	84.93	5.03	4.87	0.18	0.22	0.58	<0.01	0.23	0.15	<0.01	0.07	<0.01	0.02	<0.01	0.25	0.05	0.02	<0.01	<0.01	<0.01	96.60	0.56	16.5	89.96	408.5
Urn-04	85.63	4.91	5.21	0.03	0.54	0.39	<0.01	0.20	0.13	<0.01	0.05	<0.01	0.33	0.12	0.43	<0.01	0.05	0.01	0.13	<0.01	98.16	1.24	17.1	90.54	432.7
Urn-05 *	84.91	4.74	5.09	0.04	0.30	0.27	0.04	0.35	0.07	0.04	<0.01	0.01	<0.01	<0.01	0.06	0.01	0.01	<0.01	0.03	0.20	96.17	0.48	17.5	89.65	426.8
Urn-06	86.18	4.65	5.18	0.07	0.32	0.48	<0.01	0.25	0.06	<0.01	0.05	<0.01	0.12	0.02	0.35	0.09	<0.01	<0.01	0.24	0.12	98.18	1.06	18.1	90.83	428.1
Urn-07	86.78	4.50	5.18	<0.02	0.36	0.69	<0.01	0.25	0.04	0.07	<0.01	0.02	0.19	<0.01	0.09	0.06	0.29	<0.01	<0.01	0.14	98.66	0.91	18.9	91.28	425.5
Urn-08 *	87.43	4.45	5.28	0.02	0.26	0.94	<0.01	0.26	<0.01	0.03	<0.01	0.05	0.05	<0.01	0.28	<0.01	0.01	0.13	0.09	0.18	99.46	0.83	19.2	91.87	430.4
Urn-09	87.51	4.49	5.14	0.03	0.29	0.90	<0.01	0.26	0.07	<0.01	<0.01	<0.01	0.19	<0.01	0.28	0.05	0.03	<0.01	0.12	0.13	99.49	0.86	19.1	92.00	419.0
Urn-10	87.62	4.56	5.30	0.05	0.87	0.57	<0.01	0.27	0.02	<0.01	<0.01	<0.01	0.24	<0.01	0.22	<0.01	<0.01	0.03	0.16	0.07	99.98	0.75	18.8	92.18	430.9
Urn-11	87.04	4.50	4.85	0.08	0.24	0.34	<0.01	0.23	0.18	0.09	<0.01	<0.01	0.21	<0.01	0.26	0.07	0.04	<0.01	0.21	0.19	98.53	1.26	18.9	91.54	398.3
Urn-12 *	87.15	4.52	5.05	0.06	0.21	0.41	<0.01	0.24	0.19	<0.01	<0.01	<0.01	0.16	<0.01	0.42	0.11	0.29	<0.01	0.19	0.13	99.13	1.48	18.9	91.67	413.6
Urn-13	87.64	4.46	5.36	0.03	0.45	0.45	<0.01	0.24	0.16	0.01	<0.01	<0.01	0.08	<0.01	0.39	0.17	<0.01	0.08	<0.01	0.05	99.57	0.95	19.2	92.10	435.7
Urn-14 *	86.66	4.55	5.03	0.10	0.53	0.66	<0.01	0.24	0.04	0.10	<0.01	<0.01	0.13	<0.01	0.43	0.16	0.21	0.10	0.14	0.12	99.20	1.43	18.6	91.20	414.1
Urn-15	87.09	4.64	5.16	0.07	0.34	0.35	<0.01	0.18	0.17	<0.01	0.11	0.03	0.09	0.15	0.16	0.21	0.04	0.15	<0.01	0.20	99.14	1.30	18.4	91.74	422.3
Urn-16	86.37	4.65	5.09	0.03	0.26	0.49	0.04	0.24	0.09	0.09	<0.01	<0.01	0.07	<0.01	0.16	0.17	<0.01	0.01	<0.01	0.19	97.95	0.82	18.2	91.03	420.0
Urn-17 *	85.73	4.63	5.37	0.08	0.31	0.18	<0.01	0.18	0.13	0.05	<0.01	<0.01	0.10	<0.01	0.18	0.03	<0.01	<0.01	0.01	0.19	97.17	0.69	18.1	90.37	445.3
Urn-18	85.07	4.67	4.83	0.08	0.63	0.11	0.01	0.21	0.08	<0.01	0.16	<0.01	0.27	<0.01	0.32	0.05	<0.01	<0.01	<0.01	0.04	96.52	0.92	17.8	89.75	405.2
Urn-19	85.58	4.60	4.89	0.09	0.58	0.15	0.10	0.18	0.29	0.02	<0.01	<0.01	0.12	<0.01	0.26	0.02	0.07	<0.01	0.16	0.15	97.26	1.19	18.2	90.18	407.8
Urn-20	84.73	4.73	5.00	0.06	0.17	0.07	<0.01	0.22	0.04	<0.01	0.03	0.06	0.09	<0.01	0.16	0.02	<0.01	0.16	0.14	<0.01	95.68	0.70	17.5	89.46	420.3
Urn-21	85.38	4.64	4.85	0.16	0.48	0.09	<0.01	0.18	0.02	0.07	0.08	0.08	0.09	<0.01	0.21	0.16	0.07	0.21	0.13	0.11	97.01	1.23	18.0	90.02	405.4
Urn-22 *	86.07	4.77	5.01	0.08	0.31	0.13	<0.01	0.18	0.09	0.01	<0.01	<0.01	0.24	<0.01	0.15	0.14	0.14	0.24	0.02	0.03	97.61	1.07	17.6	90.84	414.9
Urn-23	85.53	4.90	5.08	0.05	0.68	0.07	<0.01	0.26	0.13	0.08	0.09	0.02	0.13	<0.01	0.23	0.07	0.14	0.02	0.06	0.14	97.68	1.11	17.1	90.43	422.8
Urn-24 *	85.17	4.68	4.87	0.08	0.32	0.04	<0.01	0.26	0.18	0.12	<0.01	<0.01	0.04	<0.01	0.32	0.06	0.06	<0.01	<0.01	<0.01	96.37	0.95	17.8	89.86	407.9
Urn-25	85.56	4.61	4.87	0.13	0.23	0.13	0.07	0.26	0.13	0.19	<0.01	<0.01	0.07	0.07	0.33	0.09	<0.01	0.11	<0.01	0.21	97.06	1.28	18.1	90.17	406.3
Urn-26	86.45	4.70	4.89	0.09	0.26	0.12	<0.01	0.23	0.08	0.05	0.01	0.05	0.14	<0.01	0.35	0.07	<0.01	0.06	<0.01	<0.01	97.55	0.81	18.0	91.15	403.8
Urn-27	86.19	4.57	5.13	0.06	0.31	0.13	<0.01	0.22	<0.01	0.02	0.01	<0.01	0.15	<0.01	0.24	0.06	<0.01	<0.01	<0.01	0.14	97.23	0.62	18.4	90.76	424.2
Urn-28 *	85.58	4.59	5.08	0.10	0.33	0.16	0.03	0.22	0.11	<0.01	<0.01	<0.01	0.11	<0.01	0.10	<0.01	<0.01	0.12	<0.01	0.31	96.84	0.78	18.2	90.16	423.0
Urn-29	86.00	4.75	5.41	0.05	0.34	0.11	<0.01	0.25	0.03	0.10	0.01	0.02	0.16	<0.01	0.18	0.23	0.10	0.04	0.21	<0.01	97.99	1.09	17.7	90.75	447.0
Urn-30 *	86.09	4.70	4.91	0.05	0.53	0.15	<0.01	0.20	0.14	<0.01	0.04	<0.01	0.30	<0.01	0.25	<0.01	<0.01	0.05	0.21	0.38	98.00	1.37	17.9	90.79	407.0
Urn-31 *	86.93	5.18	4.87	0.24	0.47	1.02	<0.01	0.23	0.14	0.06	<0.01	<0.01	0.03	0.15	0.35	<0.01	<0.01	0.16	<0.01	<0.01	99.83	0.89	16.4	92.11	399.5

Table 3. Cont.

No.	UO ₂	ThO ₂	PbO	SiO ₂	CaO	FeO	La ₂ O ₃	Y ₂ O ₃	Ce ₂ O ₃	Pr ₂ O ₃	Nd ₂ O ₃	Eu ₂ O ₃	Gd ₂ O ₃	Tb ₂ O ₃	Dy ₂ O ₃	Ho ₂ O ₃	Er ₂ O ₃	Tm ₂ O ₃	Lu ₂ O ₃	Yb ₂ O ₃	Total	REE	U/Th	U + Th	U-Th-Pb Ages
Urn-32 *	82.47	4.69	4.75	0.34	0.64	2.73	<0.01	0.10	0.12	0.02	0.01	0.05	0.09	<0.01	0.23	<0.01	0.01	0.01	0.11	0.13	96.50	0.77	17.2	87.15	410.6
Urn-33	73.65	3.40	3.90	2.94	1.21	<0.02	0.10	0.95	0.27	0.18	0.27	0.01	0.22	<0.01	0.26	0.08	0.09	<0.01	0.02	0.23	87.78	1.73	21.2	77.05	379.9
Urn-34	73.28	2.81	4.04	2.09	0.95	<0.02	0.03	1.06	0.25	0.07	0.15	<0.01	0.22	<0.01	0.54	0.06	0.41	<0.01	<0.01	<0.01	85.97	1.72	25.5	76.09	395.8
Urn-35	68.25	4.30	3.29	4.62	1.68	0.04	0.07	0.44	0.14	<0.01	<0.01	<0.01	0.01	<0.01	0.10	<0.01	<0.01	0.14	<0.01	0.05	83.13	0.52	15.5	72.55	345.2
Urn-36	70.49	3.38	4.08	4.69	1.71	<0.02	<0.01	0.52	0.13	<0.01	0.04	0.10	0.07	<0.01	0.25	0.03	0.14	0.06	0.10	<0.01	85.79	0.90	20.4	73.87	413.6
Urn-37	71.00	3.16	4.34	5.23	0.57	0.04	<0.01	0.75	<0.01	0.18	0.10	<0.01	0.10	<0.01	0.37	0.11	0.18	<0.01	0.03	<0.01	86.16	1.06	22.0	74.16	436.2
Urn-38	63.92	4.37	2.96	5.70	1.91	<0.02	<0.01	0.36	0.18	0.03	<0.01	<0.01	0.04	<0.01	0.13	0.01	0.30	0.07	0.01	0.06	80.05	0.82	14.3	68.29	331.5
Urn-39	69.60	3.30	4.40	2.54	0.88	0.08	<0.01	0.67	0.15	<0.01	<0.01	<0.01	0.22	<0.01	0.29	<0.01	0.10	0.02	0.06	0.15	82.46	1.00	20.6	72.89	450.1
Urn-40	68.76	2.92	4.34	2.17	0.92	<0.02	<0.01	0.83	0.16	0.06	<0.01	<0.01	0.21	0.08	0.39	0.09	0.25	0.03	0.10	0.12	81.44	1.51	23.0	71.68	450.0
Urn-41	68.88	3.34	4.56	1.34	0.48	0.08	<0.01	0.87	0.23	0.04	<0.01	<0.01	0.20	<0.01	0.22	0.06	0.08	0.07	<0.01	0.01	80.46	0.92	20.2	72.23	470.2

Note: Urn01 to 32 are non-altered uraninite; Urn33 to 41 are altered uraninite with SiO₂ contents > 1.00 wt%; U + Th represent UO₂ + ThO₂; <x.xx where x.xx represents the detection limit of the analysis.

* indicates the data points have been marked the location in the Figure 5 (grain A: Urn-1 (A-1), Urn-5 (A-5), Urn-8 (A-8); grain B: Urn-12 (B-2), Urn-14 (B-4), Urn-17 (B-7); grain C: Urn-22 (C-2), Urn-24 (C-4), Urn-28(C-8) and grain D: Urn-30 (D-3), Urn-31 (D-4), Urn-32(D-5)).

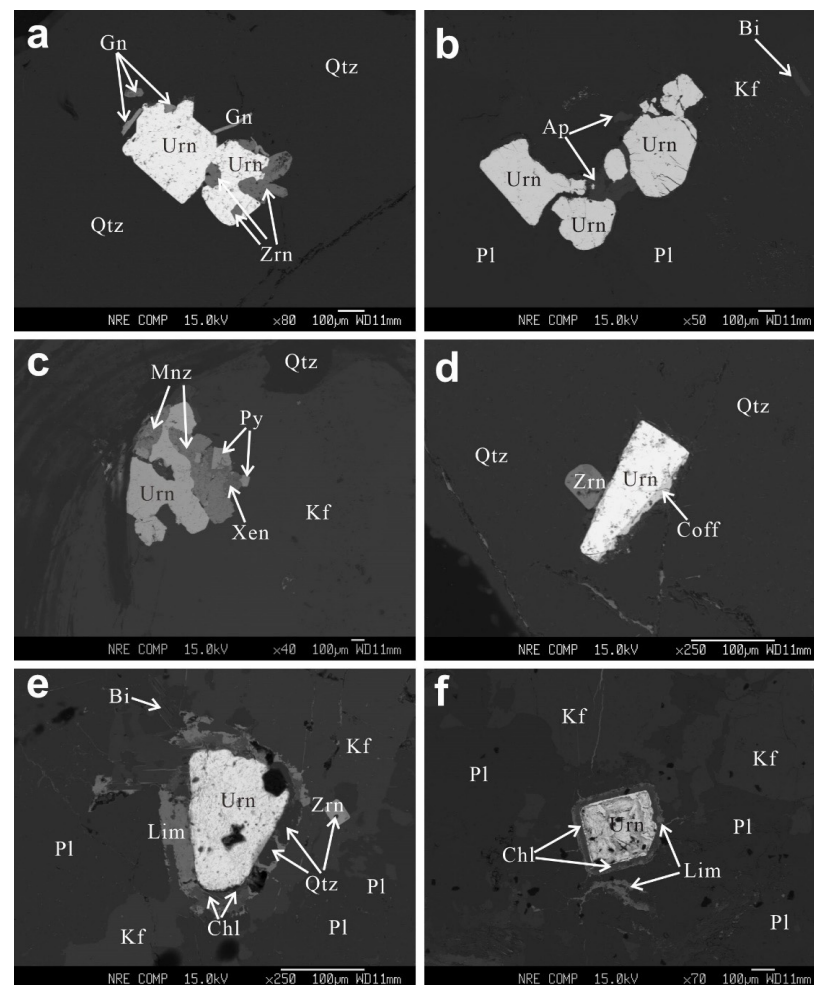


Figure 3. Back-scattered electron images of uraninite occurrence in the Guangshigou uranium deposit. (a) uraninite associated with galena and zircon in quartz. (b) micro-crack bearing uraninite associated with apatite between K-feldspar and plagioclase. (c) uraninite associated with monazite, xenotime and pyrite. (d) uraninite showing an alteration rim of coffinite, accompanied with zircon. (e,f) metamict uraninite showing a destruction rim associated with chlorite and limonite. Lim—Limonite, Urn—Uraninite, Zrn—Zircon, Mnz—Monazite, Qtz—Quartz, Kf—K feldspar, Pl—Plagioclase, Chl—Chlorite, Py—Pyrite, Gn—Galena, Coff—Coffinite, Ap—Apatite, Bi—Biotite, Xen—Xenotime.

4.3. In Situ U-Pb Isotopic Dating on Uraninite

Non-metamict and non-altered uraninite from the Guangshigou deposit was selected for in situ U-Pb isotopic dating in order to constrain the timing of the Paleozoic pegmatite-related U event in the NQOB. Based on back-scattered electron images and preliminary analyses obtained by EPMA, the most suitable areas of four large uraninite grains were selected for U-Pb in situ dating by SIMS (Figure 5). Special caution was taken in selecting zones devoid of galena inclusions, having relatively homogeneous U and Pb contents, and low Si contents, and thus showing the less developed post-crystallization alteration. The 32 analyses performed on the uraninite grains display very low common lead contents with $^{204}\text{Pb}/^{206}\text{Pb}$ values ranging from 4×10^{-6} to 2×10^{-5} (Table 2); hence, no common lead correction was applied to the data. Measured U-Pb isotopic ratios are listed in Table 2. They were plotted in a concordia diagram presented in Figure 6a. The 32 measured isotopic ratios show 17 concordant dates and 15 weakly discordant plots which is indicative of more or less significant radiogenic Pb loss due to post-crystallization alteration. They define a discordia line with a concordia upper intercept giving an age of crystallization at 412 ± 3 Ma (Figure 6a) for uraninite in the Guangshigou deposit.

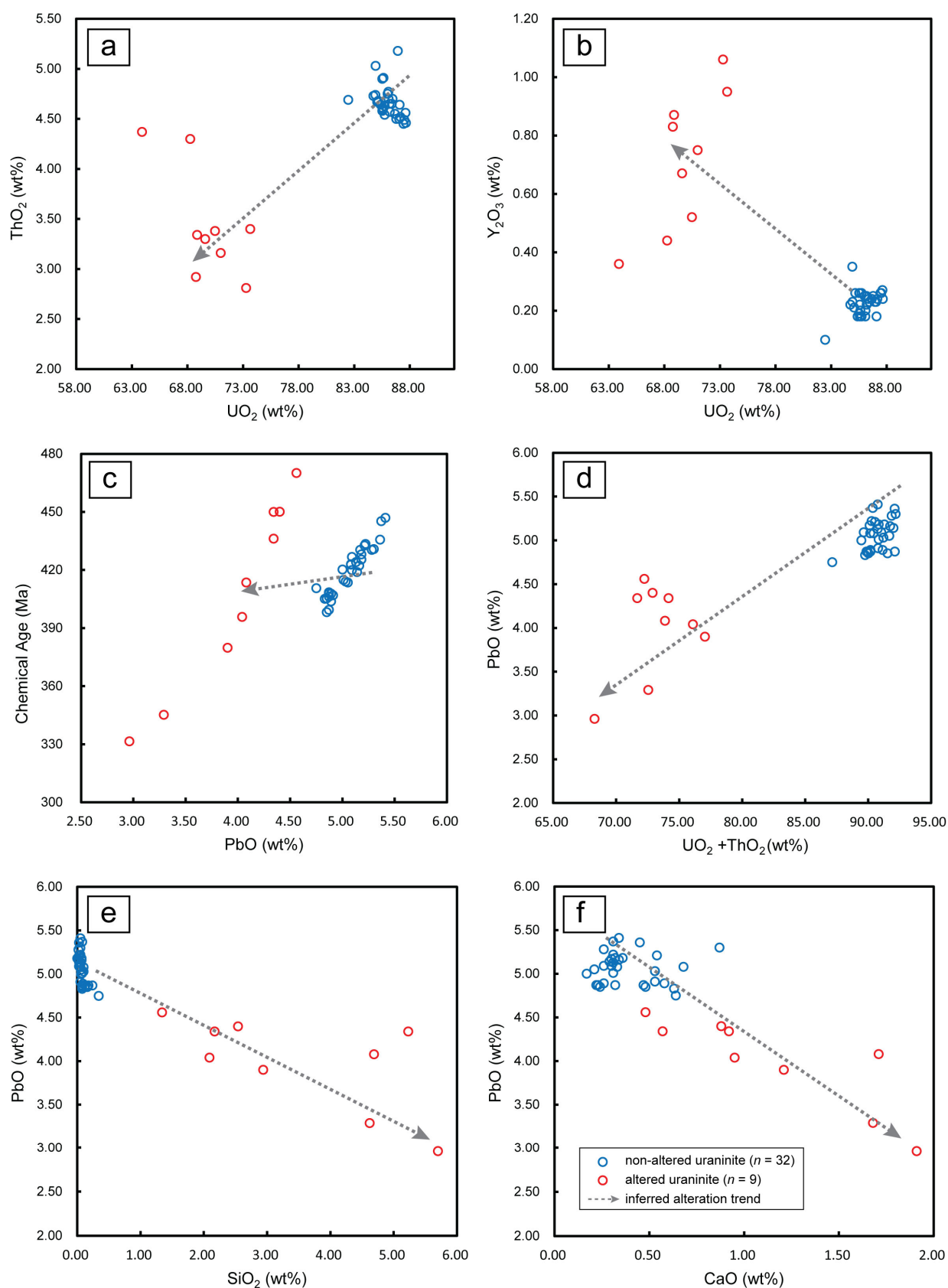


Figure 4. (a–f) Diagrams of major and minor elements displaying the chemical composition of uraninite from the Guangshigou uranium deposit. Data points with $>1\text{wt}\%$ SiO_2 are considered as altered uraninite.

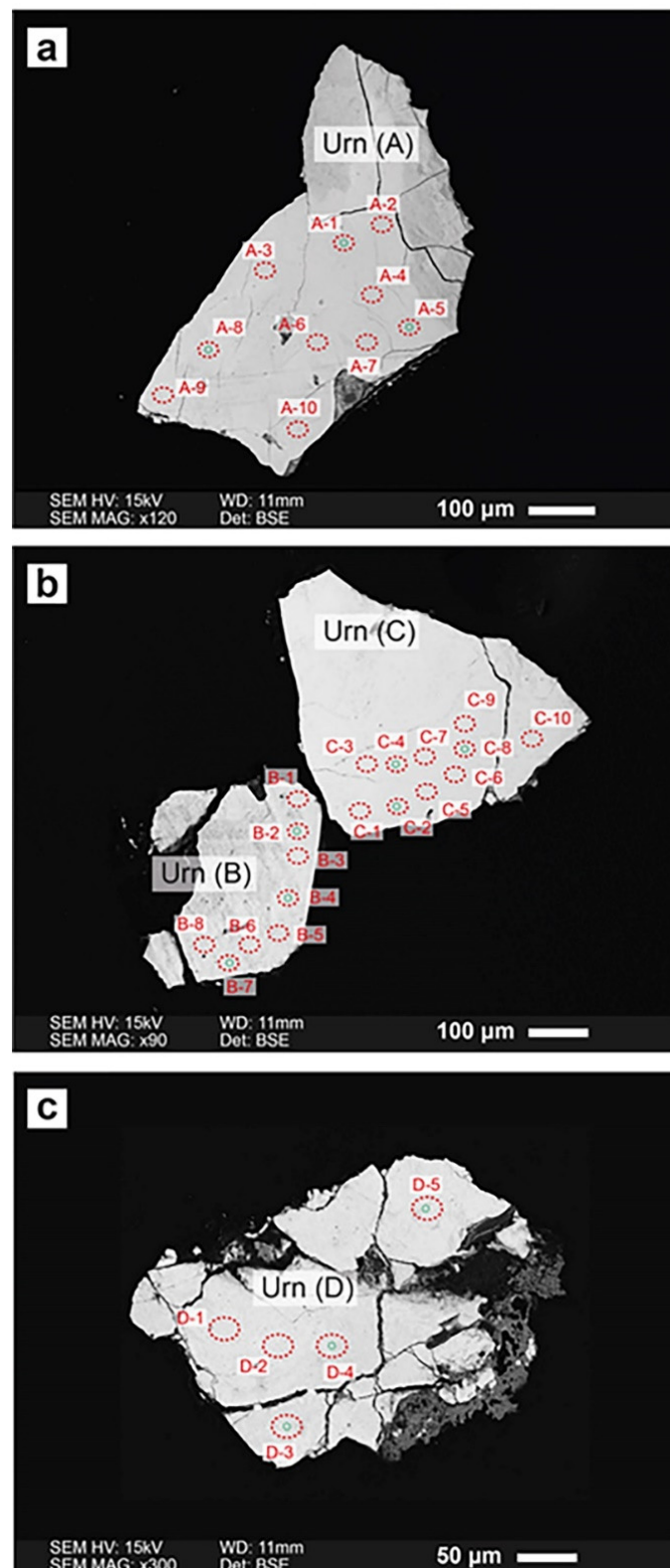


Figure 5. (a–c) Back-scattered electron images of uraninite grains selected for EPMA and Secondary-Ion Mass Spectrometry (SIMS) measurements. The dashed green circles indicate the location of EPMA (see the corresponding data points in Table 3) and the dashed red ellipses the location of SIMS data points.

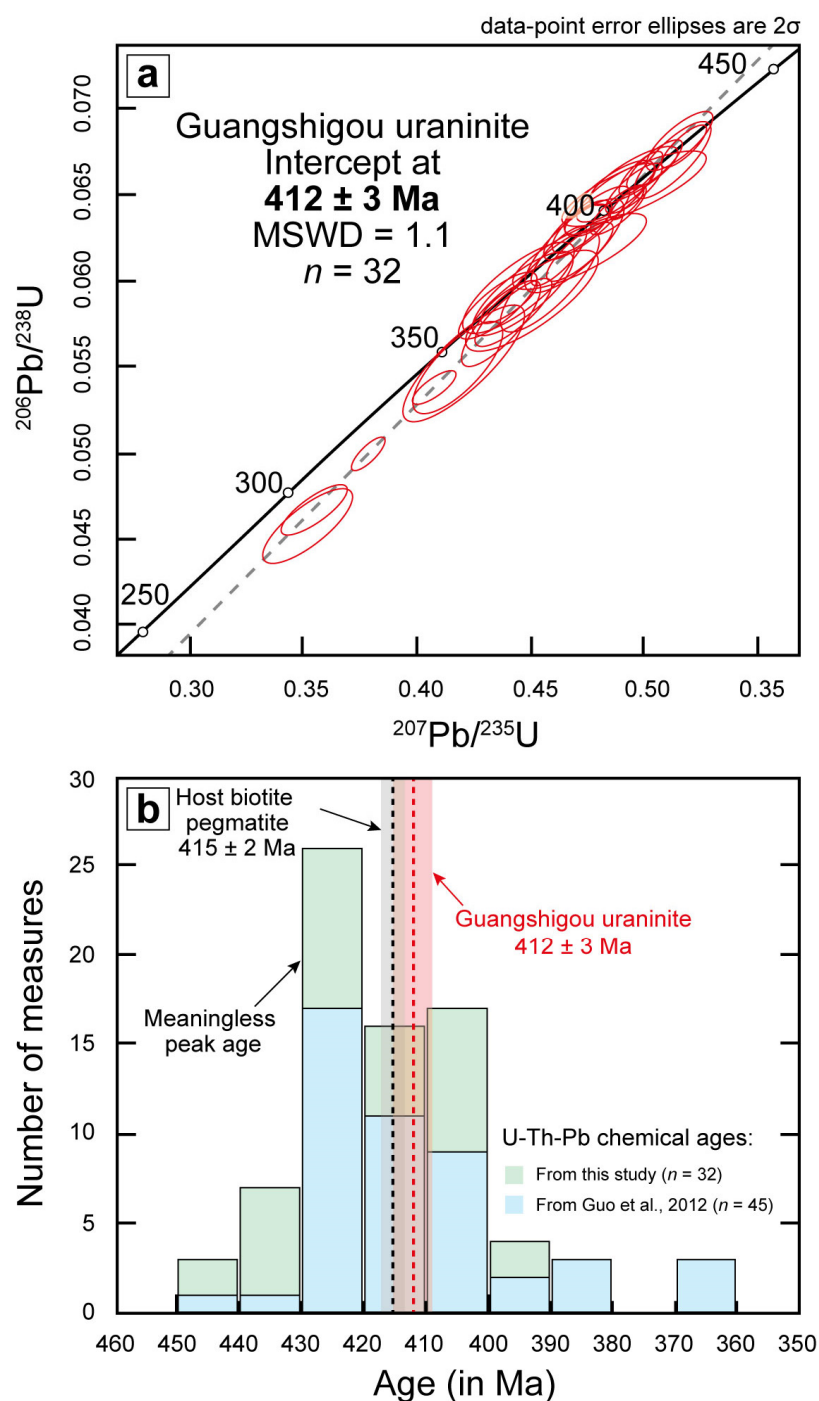


Figure 6. (a) Wetherill Concordia plots of U-Pb data for uraninite from the Guangshigou uranium deposit. The data error ellipses are at 2σ . (b) Histogram of U-Th-Pb chemical ages on uraninite (calculated according to this study and [28]). The black dashed line indicates the crystallization age of zircon from the Guangshigou pegmatite (from [22]) and the area in grey is representative of the error. The red dashed line indicates the crystallization age of uraninite and the area in light red is representative of the error.

For comparison, U-Th-Pb chemical age estimates were calculated from the composition of non-altered uraninite presented in this study and also compiled from Guo et al. [28]. The age estimates broadly range from 446 to 366 Ma and display a bimodal distribution with two meaningless peak ages in the ranges 430–420 Ma and 410–400 Ma (Figure 6b), which indicate either older or younger ages than the one determined by SIMS at 412 Ma. Older

chemical ages maybe related to small amounts of common Pb contained in uraninite or due to radiogenic Pb diffusion [75], while younger ages reflect radiogenic Pb loss during post-crystallization alteration.

5. Discussion

5.1. Magmatic Origin of Uraninite from the Guangshigou Deposit

Uranium in silicate magmas exhibits a strongly incompatible behavior because of its large ionic radius and high valence, which prevents its incorporation into the structure of the main rock-forming silicates. As a result, during partial melting and crystal fractionation, U is preferentially fractionated into silicate melts [19]. However, despite its strongly incompatible behavior, deposits dominantly resulting from magmatic processes are rare. For instance, in an average granitoid with a U enrichment of 3 to 4 ppm, uranium is dominantly hosted by refractory accessory minerals such as zircon, apatite, monazite (etc.). Only some specific granitic igneous rocks with higher U contents, including metaluminous high-K calc-alkaline and peraluminous igneous rocks, may allow crystallization of uraninite [19,75]. Peralkaline magmas may however be excluded as individual crystals of uraninite are generally not able to crystallize despite the strong U enrichment of these melts [19].

In the Guangshigou deposit, the U mineralization is dominantly represented by disseminated uraninite crystals hosted in granitic biotite pegmatites. The following lines of evidence support the argument that uraninite from the Guangshigou deposit has a magmatic origin.

1. Most uraninite grains show well-shaped euhedral to subhedral crystal morphology (Figure 3), which can be considered as a result of high-temperature precipitation [10]. Furthermore, their occurrence as inclusions in major rock-forming minerals (Figure 3a,b), along cracks or interstitial at the grain boundaries between rock-forming minerals (Figure 3c,d), or as intergrowth with magmatic zircon, apatite, monazite, and xenotime (Figure 3a–c) suggest that uraninite is coeval of the main rock-forming and accessory minerals constituting the host pegmatite.
2. Non-altered uraninite in the Guangshigou deposit is characterized by low to relatively high Th, REE and Y contents (up to 4.64 wt% ThO₂, 0.93 wt% Σ REE₂O₃ and 0.23 wt% Y₂O₃; Figure 4), and low U/Th ratios with a mean value of 18.54 (Table 3). The chemical composition and U/Th ratios of uraninite generally have been used to constrain its origin [76]. For instance, uraninite with U/Th ratios greater than 1000 is likely of low-temperature, hydrothermal origin, whereas higher temperature metamorphic or magmatic uraninite typically has U/Th ratios on the order of 10–100 [77]. Hence, U/Th ratios (15 to 26) of uraninite from the Guangshigou deposit suggest a magmatic origin. In addition, their Th, REE, and Y enrichments indicate that they most likely crystallized from a fractionated high-K calc-alkaline pegmatitic magma [19,75,78–80]. Indeed, when the high-K calc-alkaline melts become slightly peraluminous and/or when their temperature and Ca content has decreased sufficiently, monazite may become stable and Th-bearing accessory minerals start to fractionate to induce a decrease in Th/U ratios and allow uraninite crystallization [19], which is consistent with cogenetic monazite observed together with uraninite in the Guangshigou deposit (Figure 3c). Moreover, when uraninite crystallizes in equilibrium with Th-rich minerals, it is commonly characterized by high Th contents [81], which is also consistent with the relatively high Th concentrations (mean = 4.64 wt% ThO₂, Figure 4a) measured in uraninite by the EPMA. Nevertheless, the proportion of uraninite is generally small in such fractionated igneous rocks [19], hence suggesting that U-rich pegmatites from the Guangshigou deposit have likely resulted from additional magmatic processes possibly involving the anatexis of parts of the metamorphic country rocks in which the pegmatites intruded. In addition, it is also excluded that U-rich pegmatites from the Guangshigou deposit only formed by low degree of partial melting of metamorphic rocks belonging to the Qinling Group. First, because the fraction of U, Th and REE

hosted in accessory minerals such as monazite, zircon, and apatite from the country rocks, cannot contribute to the enrichment of the melts because these accessories are only weakly soluble in low-temperature peraluminous silicate melts [19]. Second, because the fractionation of monazite, the main Th- and REE-bearing mineral in peraluminous magmas, depletes melt in Th and REE. Uranium is not depleted because accessories such as monazite, zircon, and apatite incorporate only minor amounts of U. Consequently, the U remaining in the melt continues to be enriched during fractionation until the silicate melt reaches uraninite saturation enabling the crystallization of Th-poor uraninite [82].

3. SIMS U-Pb dating on uraninite yielded a crystallization age of 412 ± 3 Ma, which is concomitant (within errors) with the emplacement age (415 ± 2 Ma, [58]) of the granitic biotite pegmatite hosting the U mineralization in the Guangshigou deposit.

5.2. Geochronological Constraint and Comparison with the Previous Ages of Uraninite

The Shangdan domain has been interpreted as an early Paleozoic uraniferous province in the NQOB [23]. In this region, U-bearing granitic pegmatites are widespread and several pegmatite-type U deposits including the Guangshigou deposit have been discovered [22,24,25]. The basement of the Guangshigou U mining district belongs to the third member of the Qinling Group, which is composed of biotite-plagioclase gneiss, amphibolite, amphibole-biotite schist, leptynite and migmatite, and marble [22,24,51]. Zircon U-Pb ages of basement rocks from the Qinling Group recorded two major magmatic events during the Mesoproterozoic (ca. 1600–1400 Ma) and Neoproterozoic (ca. 950–850 Ma), and an Early Paleozoic episode of metamorphism with a peak age at 477 ± 18 Ma, indicating that the Qinling Group had undergone metamorphism during the evolution of the NQOB [83]. Then, the metamorphic rocks of the Qinling Group were intruded by a series of Caledonian granitic igneous rocks during two main stages [22]: (i) the Early Silurian (e.g., the Huichizi granite batholith at 441–422 Ma [22,70]) and (ii) the Late Silurian–Early Devonian (e.g., the Damaogou granite stock at 420–418 Ma [22,27] and various granitic pegmatites at 415.6–412.1 Ma [22]). The granitic biotite pegmatite that hosts the U mineralization in the Guangshigou deposit was dated at 415 ± 2 Ma [58] and therefore, emplaced during the second stage of the Caledonian magmatic event, which occurred during the late tectonic post-collisional evolution of the NQOB [66–68]. Moreover, the Guangshigou pegmatite-type U deposit was interpreted as a typical magmatic deposit, and the U-Pb age of zircon from the host pegmatite is often regarded as the U mineralization age.

In this study, the in situ U-Pb isotopic dating by SIMS on non-altered uraninite from the Guangshigou deposit yielded a crystallization age of 412 ± 3 Ma. This age constrains the U ore genesis in the Guangshigou deposit to the Early Devonian, which corresponds to the second stage of the Caledonian magmatic event in the NQOB. Moreover, this age on uraninite is very similar (within errors) to the U-Pb isotopic age that was determined on zircon (i.e., 415 ± 2 Ma, [58]) from the granitic biotite pegmatite hosting the U mineralization of the Guangshigou deposit (Figure 7), hence confirming the magmatic or late magmatic origin of the uraninite and that the Guangshigou deposit is a typical pegmatite-type U deposit. In comparison with the previous investigations for uraninite dating that were performed by ID-TIMS (427–380 Ma, [23]), or in situ EPMA U-Th-Pb chemical dating (441–330 Ma, [22,28]) and LA-ICP-MS isotopic dating (405 ± 3 and 403 ± 3 Ma, [22,43]), our crystallization age determination by SIMS at 412 ± 3 Ma represents very precise result and consistent age of the U mineralization with respect to the emplacement age of the host pegmatite in the Guangshigou deposit. It is also very consistent with ID-TIMS isochron U-Pb ages of 414 and 418 Ma obtained by Feng et al. [23]. However, in situ U-Th-Pb chemical ages calculated from uraninite composition analyzed by EPMA show a broad distribution, from 446 to 398 Ma in this study, and meaningless peak ages (Figure 6b) either older or younger than the crystallization age determined by SIMS, due to small amounts of common Pb contained in uraninite and/or radiogenic Pb diffusion [75], or radiogenic Pb loss during post-crystallization alteration [33], respectively. Finally, U-Pb

isotopic ages obtained by LA-ICP-MS yielded crystallization ages of uraninite about 10 Ma younger than the age presented in this study and the emplacement age of the host pegmatite. These younger ages are most probably related to the fact that zones of uraninite affected by post-crystallization alteration associated with radiogenic Pb loss [33] were also ablated during the LA-ICP-MS experiments. This hypothesis is further corroborated by the observation of metamict uraninite in the Guangshigou deposit and the high Si contents (i.e., coffinitization) measured in some uraninite that show evidence for post-crystallization alteration (Figure 4). The radiation-induced destruction of the uraninite structure (i.e., metamictization), as observed in Figure 3e,f, can cause U and Pb depletion and/or enrichment, which may result in significant uncertainties for the U-Pb isotopic ratios [84–88]. Moreover, radiogenic Pb loss due to post-crystallization alteration of uraninite [33] is a common feature that needs to be taken into account for U-Pb isotopic dating as it can lead to the determination of discordant and meaningless ages, and was encountered in various magmatic to hydrothermal U systems [78–80].

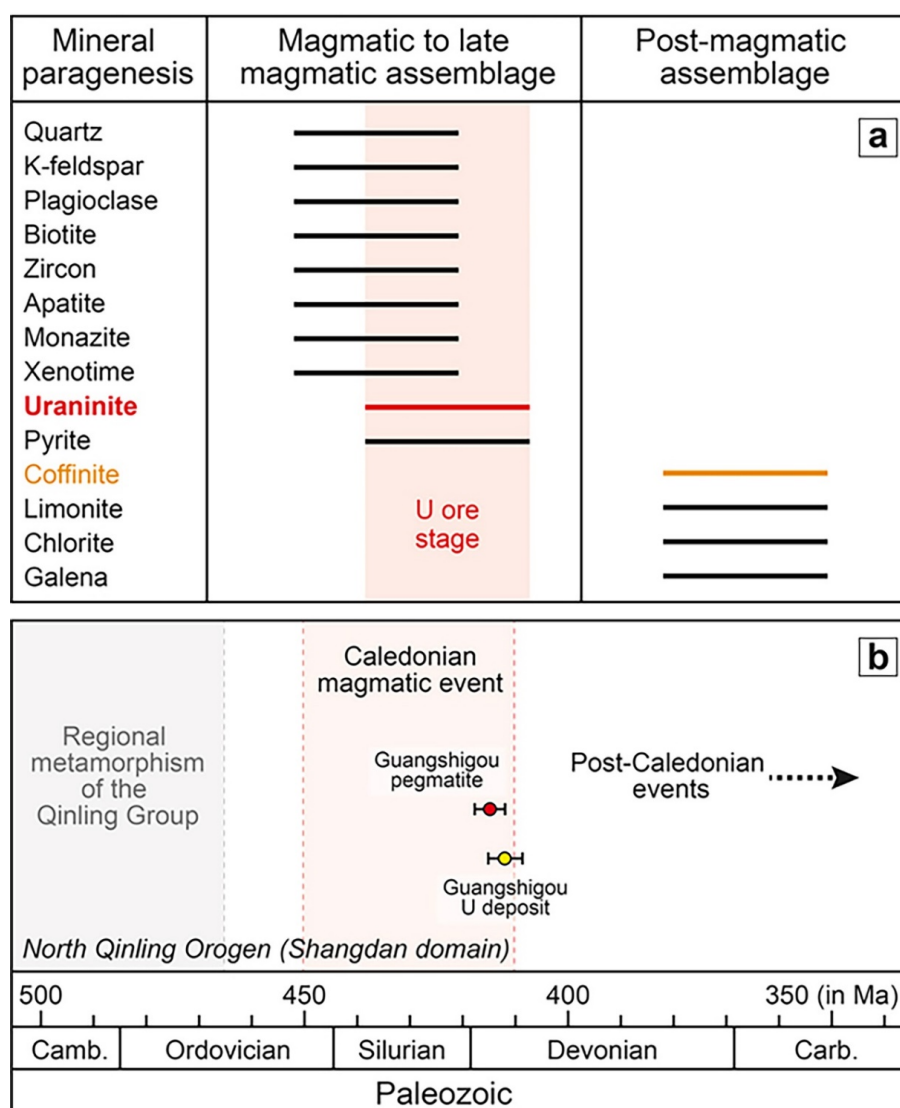


Figure 7. (a) Mineral succession diagram and metallogenic model related to the U ore formation in the Guangshigou deposit. (b) Regional geodynamic–metallogenic evolution of the Shangdan domain in the North Qinling orogen. Information regarding the regional metamorphism of the Qinling Group is after [55,83], and the Caledonian magmatic event is after [44,56]. Age of the Guangshigou pegmatite is from [22,23,28,43]. Camb. means Cambrian, Carb. means Carboniferous.

In addition, a second group of younger uraninite with U-Th-Pb chemical ages in the range of 390–340 Ma was also identified by Yuan et al. [22]. This uraninite with depleted Th contents and enriched Si, Ca and Fe concentrations compared with the primary uraninite in the Guangshigou deposit was interpreted as a result of post-Caledonian hydrothermal alteration, which is relatively consistent with the chemical composition of altered uraninite that was analyzed by EPMA in this study (Figure 4), and the post-magmatic mineral assemblage characterized by coffinite, chlorite and limonite observed in the alteration halo of uraninite (Figures 3 and 7).

5.3. Implications for Paleozoic Pegmatite-Type Uranium Ore Genesis in North Qinling

The NQOB is an important component of the Qinling composite orogen in Central China, which has experienced multiphase metamorphic and magmatic events. High-pressure and ultra-high pressure (HP-UHP) metamorphic rocks occurred as lenses or layers at the contact with Proterozoic gneisses in the NQOB, and were considered to have undergone deep subduction/collision, slab-breakoff and crustal thinning during post-collisional extension [89]. The Early Paleozoic granitic rocks that intruded in the NQOB are widely developed and considered to derive from arc magmatism which was associated with the northward subduction of the Shangdan Ocean [44]. Therefore, the magmatic evolution of the North Qinling area is the key to understanding the magmatic processes related to the formation of the pegmatite-type U mineralization in the province. Four distinct stages of Early Paleozoic felsic magmatism in the NQOB were identified at ca. 500 Ma, 470–450 Ma, 450–420 Ma, and 420–400 Ma, respectively [22,59]. Among these magmatic events, an early stage of granitoid intrusion (at ca. 500 Ma) was related to the regional HP-UHP metamorphism, while the second stage (at ca. 470–450 Ma) occurred in a post-collisional setting that preceded the Caledonian magmatic event in the NQOB [22,23,44,52,59,60,90]. Here, the third and fourth stages of granitoid emplacement correspond with the two episodes of the Caledonian magmatic event that was recorded in the NQOB [22]. Hence, the third stage (at ca. 450–420 Ma) is mainly represented by Early Silurian granitic plutons formed in a syn-tectonic environment [52,59,60], whereas the fourth stage (at ca. 420–400 Ma) is represented by Late Silurian–Early Devonian high-K calc-alkaline granitoids and pegmatites formed in a late-tectonic post-collisional context [66–68] (Figure 7b). The granitic pegmatite dykes emplaced during the late Caledonian magmatic event and that host the U mineralization in the province were interpreted as a result of crust-derived magma produced during an episode of post-collision extension in the NQOB [22,25].

In the Shangdan domain, Caledonian granitoids and pegmatites show I-type and A-type granite affinities, respectively [22]. Their whole-rock geochemical, mineralogical and Sr and Nd isotopic characteristics presented in detail in Yuan et al. [22] demonstrated that Caledonian intrusions shared a common source and were derived from the partial melting of ancient basaltic lower crust (e.g., Neoproterozoic metabasalts in the NQOB) with some addition of mantle-derived materials. Hence, it was proposed that Early Silurian granitoids (e.g., the Huichizi granite) were formed by low degree of partial melting of thickened basaltic lower crust combined with mantle-derived materials following the northward subduction of the Shangdan Ocean beneath the NQOB, and that Late Silurian–Early Devonian high-K calc-alkaline granitic rocks (e.g., the Damaogou granite) derived from the same source and emplaced during a post-collision relaxation period.

Pegmatite genesis has been under controversial debate for decades. Two contrasting hypotheses, involving continuous fractional crystallization and partial melting models, have been proposed to explain the origin of pegmatite-forming melt [4,10]. A prevalent point of view is that pegmatite originates from highly water- and flux-enriched residual magma through extended fractional crystallization of granitic magma [22]. The alternative point of view is that partial melting (anatectic pegmatoids) may be favored where pegmatite dykes are spatially isolated from granitic plutons. However, U mineralization purely resulting from magmatic processes such as extreme fractionational crystallization (only

known in peralkaline igneous rocks) or anatexis (low degree of partial melting of U-rich metasedimentary rocks) are rare [75]. In the Guangshigou area, the Late Silurian–Early Devonian high-K calc-alkaline Damaogou granite (ca. 420–418 Ma) and biotite pegmatites (415 Ma) have close spatial and temporal relationships (Figure 1b) and display consistent ion-exchange modes of mica and similarities in their mineral assemblages [22]. Moreover, their geochemical signatures showing that fractional crystallization took place during the emplacement of the granite-pegmatite system and similar Sr and Nd isotopic compositions provide several lines of evidence supporting that the pegmatites formed from residual magma through the differentiation of the Damaogou granite at depth [22,25]. However, partially assimilated xenoliths within biotite pegmatite, the irregular contact between pegmatites and gneisses of the Qinling group (Figure 2) and mineralogical heterogeneity in pegmatite contact zones indicate that the pegmatitic magma experienced various degrees of crustal material contamination during ascent through the country rocks or at the site of emplacement [22]. Moreover, the relative abundance of old country rock-derived xenocrystic zircon in U-rich biotite pegmatite also argues in favor of substantial crustal contamination of the magma. Therefore, regarding the broad compositional variation and heterogeneous mineral composition of the U-rich pegmatites, Yuan et al. [22,58] and Chen et al. [25] proposed a model of hybridized pegmatite where the incorporation of elements from an external source occurred at the pegmatite-gneiss contact. For instance, the zircon crystals from these pegmatites show broad variations of Ti, Y, U, Th, Hf, and REE abundances from grain to grain in individual samples and from area to area within individual grains, which indicates a fluctuating crystallization environment for zircon which is further supporting the model of hybridized pegmatite resulting from interaction between the pegmatitic melt and country rocks [58]. Hence, enrichments in incompatible elements such as K, U and Th increase with the degree of contamination (hybridization index) of the U-rich biotite pegmatites by the host gneiss of the Qinling Group, which characterizes assimilation-contamination between country rocks and pegmatitic magma as a key process in the generation of hybridized U-rich pegmatites [12,22,25,58]. Within these U-rich pegmatites, U concentration tends to be higher with increasing biotite content and uraninite is preferentially associated with or in the vicinity of peritectic biotite [22] (Figure 3), which has been employed as an exploration guide. Nevertheless, uraninite is rarely enriched to economic grades through only fractional crystallization of a pegmatitic melt [10,19]. It requires a physico-chemical interface provided by country rocks to be sufficiently saturated and enriched. Indeed, a broad variety of factors such as redox conditions, country rock composition or volatile components may be involved in U precipitation [75]. In the Guangshigou deposit, uraninite enrichment was favored in hybridized zones at the contact between the biotite pegmatite and biotite gneiss of the Qinling Group. The relatively high F contents of biotite from U-rich pegmatite compared with those of barren pegmatite indicate that F migrated into the hybridization zone during the crystallization of peritectic biotite. Thus, Yuan et al. [22] proposed that U-F complexes were destabilized by reducing fluorine activities during the formation of peritectic biotite triggering decrease of U solubility and saturation, and ultimately co-precipitation of uraninite. However, although it was not clearly demonstrated in the studies of the Guangshigou deposit (e.g., [22,25]), sulfide- and graphite-bearing gneisses of the Qinling Group hosting U-rich pegmatite could be considered as a reducing barrier, which may have prevented the fractionation of U in magmatic fluids and promoted the precipitation of uraninite at the pegmatite-gneiss contact zone, as it was also characterized in the Rössing deposit [19]. Due to previous crystallization of uraninite in granitic biotite pegmatites, late-stage two-micas and muscovite pegmatites (ca. 412 Ma) respectively located in the intermediate and outer zones from the Damaogou granite are barren [22,25].

Therefore, our new crystallization age of uraninite determined by SIMS constrained the genesis of the U mineralization in the Guangshigou deposit to the Early Devonian, which is concomitant with the emplacement age of the host granitic biotite pegmatite. During the late stage of the Caledonian magmatic event that occurred in the NQOB, high-K

calc-alkaline granitic igneous rocks intruded the metamorphic rocks of the Qinling Group in a post-collisional extension environment. Fractional crystallization of the Damaogou granitic magma produced a set of fractionated biotite pegmatites, which were subsequently enriched in U, Th, and other elements by assimilation-contamination along contact zones with the Proterozoic biotite gneiss of the Qinling Group. Interlayer slipping of anticline, which has developed during early Caledonian compressive folding of the Qinling Group, provided space for pegmatitic magma emplacement. Finally, the U ores were the products of assimilation-fractional crystallization of pegmatitic magma [22,25,58]. Thus, the late Caledonian magmatic event in the NQOB produced favorable intrusive host rocks and U sources for the genesis of pegmatite-type U mineralization in the province. Therefore, late Caledonian high-K calc-alkaline granites and their associated biotite pegmatites represent highly valuable exploration guides for the discovery of similar pegmatite-related U deposits in the NQOB. Moreover, regional extension of pegmatite-gneiss contact zones associated with hybridized pegmatites, pegmatites displaying high biotite contents and anticline axes within the deformed metasediments of the Qinling Group represent additional structural–lithological–mineralogical guides that can be employed in U exploration.

5.4. Comparison with the World-Class Rössing Deposit

The genetic model proposed for pegmatite-related U ore genesis in the NQOB shows some similarities but also has significant differences with the predominant model proposed for other pegmatite-type U deposits, and particularly with the largest deposit in the world, the Rössing deposit in Namibia [6–9,75,91,92]. For example in the Guangshigou deposit: (1) the U mineralization is hosted in granitic biotite pegmatites that intruded the deformed Proterozoic metasediments of the Qinling Group along structures; (2) the Early Devonian U-rich pegmatites emplaced during the late Caledonian stage in a post-collisional extension setting; (3) the U ore occurring as Th- and REE-rich uraninite was the product of assimilation-fractional crystallization of pegmatitic magma [22,25,58] which formed from residual magma through the differentiation of the Damaogou granite combined with various degrees of crustal material contamination (hybrid zone); (4) uraninite enrichment was favored in hybridized zones at the contact between the biotite pegmatite and biotite gneiss of the Qinling Group [22]; and (5) sulfide- and graphite-bearing gneisses of the Qinling Group likely played a role as reducing barrier promoting U precipitation. In the Rössing deposit, the emplacement of pegmatite dykes was also structurally controlled in metamorphosed sedimentary units during the late kinematic evolution of the Rössing Dome in the Central Damara orogen [19], and the crystallization of uraninite was also favored by a reducing barrier represented by the sulfide- and graphite-bearing Rössing Formation, hence preventing the fractionation of U in magmatic fluids and promoting its entrapment. However, at Rössing, the large accumulation of U-rich alaskite dykes characterized by the crystallization of Th-poor uraninite mainly resulted from a low degree of partial melting of U-rich metasediments and the existence of a chemical barrier, which was able to stop the rise of the alaskitic melts which reacted with enclosing marbles of the Rössing Formation or calcsilicate rocks of the Khan Formation to form skarns [19]. This combination of factors for the formation of the Rössing deposit likely explains its very large tonnage (246,500 t) compared with the much smaller resources in the Guangshigou deposit.

6. Conclusions

The Guangshigou U deposit located in the North Qinling Orogenic Belt, Central China, represents the largest pegmatite-type U deposit of the province. The U mineralization mainly occurs as uraninite disseminated in Caledonian U-rich granitic biotite pegmatites which formed by assimilation-fractional crystallization magmatic processes.

1. Petrographic evidence for cogenetic crystallization of uraninite and other rock-forming minerals of the host pegmatite, the Th, REE, and Y enrichments in uraninite also characterized by low U/Th ratios (~ 19), and the concomitant ages between the emplacement of the host pegmatite (415 ± 2 Ma) and the crystallization of uraninite

(412 ± 3 Ma), indicate that uraninite from the Guangshigou deposit has a magmatic origin and likely originated from fractionated high-K calc-alkaline pegmatitic magma that experienced various degrees of crustal material contamination mostly derived from the local biotite gneiss of the Qinling Group.

2. In situ U-Pb isotopic dating by SIMS on uraninite from the Guangshigou deposit yielded a crystallization age of 412 ± 3 Ma, hence constraining the U ore genesis in the Guangshigou deposit to the Early Devonian, which corresponds to the late Caledonian post-collisional extension in the North Qinling area.
3. The characteristics of the pegmatite-related Guangshigou deposit exhibiting Th-rich uraninite which was the product of assimilation-fractional crystallization of pegmatitic magma showed important differences with the world-class Rössing deposit characterized by Th-poor uraninite hosted in alaskite dykes formed by low degree of partial melting of U-rich metasediments.

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