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## RESEARCH LETTER

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### Key Points:

- One rare mm-sized granitic fragment has been identified in lunar basaltic breccia meteorite Northwest Africa 10447
- This granitic fragment has a crystallization age of 4.32 Ga, which is consistent with the ages of the oldest Apollo granite samples
- Lunar granite may occur outside of the Procellarum KREEP Terrane of the Moon (i.e., the mare region with low Th and TiO<sub>2</sub> contents)

### Supporting Information:

Supporting Information may be found in the online version of this article.

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## New Evidence for 4.32 Ga Ancient Silicic Volcanism on the Moon

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**Abstract** The currently available Apollo-returned granitic lithology is restricted to only the nearside of the Moon (e.g., Procellarum KREEP Terrane [PKT]). In contrast, lunar meteorites represent randomly ejected crustal material, and they extend our understanding of the lunar magmatic history through time and space. Here, we report a  $4,321.5 \pm 5.2$  Ma ( $2\sigma$ ) granitic fragment found in lunar basaltic breccia meteorite Northwest Africa (NWA) 10447, which is compositionally similar to the regoliths from the Moon's Non-PKT regions. This granitic fragment has crystallization age similar to the oldest (~4.32 Ga) granites sampled during the Apollo 12 and 17 missions, and adds to the growing evidence of ancient silicic volcanism on the Moon. In addition, this newly identified granitic fragment in the Th- and TiO<sub>2</sub>- poor breccia NWA 10447 provides evidence for that lunar granite may occur outside of the PKT of the Moon (i.e., the mare region with low Th and TiO<sub>2</sub> contents).

**Plain Language Summary** Lunar granite, that is, highly evolved lithologies, can provide valuable information about the magmatic evolution of the lunar crust. At present, only a few (~20) granites have been found in the Apollo samples (i.e., Apollo 12, 14, 15, and 17) and lunar breccia meteorites (e.g., NWA 4472), and they occur as rock fragments (e.g., 12,013) or mm-sized clasts within breccia (e.g., 14,321). In this paper, we report a mm-sized granitic fragment in lunar basaltic breccia meteorite NWA 10447, which has relatively low contents of Th (0.79 ppm) and TiO<sub>2</sub> (0.6 wt%). This fragment provides evidence for that lunar granite would occur outside of the PKT of the Moon, where the breccia meteorite NWA 10447 is ejected from. In addition, the granitic fragment has crystallization age (~4.32 Ga) similar to the oldest granites returned by Apollo 12 and 17 missions, providing new evidence for the existence of more ancient silicic volcanism on the Moon. This study also indicates that our understanding of lunar silicic magmatism is still insufficient. The further investigation of more silicic lithologies from other lunar meteorites (e.g., Th-poor breccias) will be helpful in filling this knowledge gap.

## 1. Introduction

On the Moon, the highly evolved lithologies that primarily consist of granophyric intergrowths of silica and K-feldspar are commonly called granite (Seddio et al., 2013). This type of rock represents a late-stage product of the extreme fractional crystallization of a magma and provides valuable information about the history of lunar magmatism (Bonin, 2012; Jolliff, 1991). Granite is rare on the Moon, but it is frequently found as either clastic fragments or as small, isolated clasts within both the Apollo-returned samples and some breccia meteorites (e.g., Apollo 12, 14, 15, and 17 samples; breccia meteorite Northwest Africa [NWA] 4,472; Jolliff et al., 1999; Joy et al., 2011; Seddio et al., 2013; Taylor et al., 1980; Warner et al., 1978). In addition, the global remote-sensing data has detected a series of silicic volcanisms distributed mostly on the nearside of the Moon within the Procellarum KREEP Terrane (PKT) (e.g., Hansteen Alpha, Gruithuisen domes, Helmet, and Lassell Massif on the nearside; Compton-Belkovich on the far side; Glotch et al., 2010; Jolliff et al., 2011). However, the currently available lunar samples collected during the Apollo and Luna missions only represent ~4.4% of the lunar surface (Warren et al., 2005), which limits our understanding of the granitic activity on the Moon, particularly that in the non-sampled regions. In contrast,

the randomly ejected lunar meteorites are thought to be more representative of the lithologies of the lunar crust, and thus, they provide valuable material for investigating the geological history of the Moon (e.g., Gross et al., 2020, 2014; Jolliff et al., 2009; Snape et al., 2011; Zeigler et al., 2017).

Lunar Apollo-granites have been dated using a large range of isotopic methods (e.g., the  $^{40}\text{Ar}$ - $^{39}\text{Ar}$ , Sm-Nd, U-Pb, and Pb-Pb systems), which have yielded a range of dates from 3,880 Ma to 4,320 Ma (i.e., from Pre-Nectarian to Nectarian; Meyer et al., 1996; Nyquist and Shih, 1992; Thiessen et al., 2018; Turner, 1970). These ages indicate that the episode of granitic activity on the Moon occurred over about 450 million years (Bonin, 2012). Subsequently, with the acquisition of new data from lunar meteorites, the oldest lunar basaltic rock ( $\sim 4,370$  Ma, Kalahari 009) and the oldest (i.e.,  $\sim 4,380$  Ma) zircon evidence for lunar immiscible silica-rich melts have been discovered (Snape et al., 2018; Zeng, Joy, et al., 2020). These findings suggest that it is very likely that ancient magmatic processes occurred outside of the Apollo landing areas, and these magmatic events were capable of producing highly evolved granites. To address this hypothesis, identifying and studying ancient lunar granite is critical to revealing the granitic activity and magmatic processes that occurred on the Moon.

Regolith breccia NWA 10447 is a lunar basaltic meteorite that has not been well characterized. It is Th-poor (i.e., Th = 0.79 ppm; Korotev and Irving, 2021) and is distinctly different from the Apollo-returned samples from the PKT regions (i.e., Th > 2–3 ppm; Korotev, 2005; Lucey et al., 2006). Recently, one granitic fragment was found in the matrix of this breccia meteorite (Figure 1), providing us with an opportunity to investigate the ancient granitic activity on the Moon. Here, we report the petrology, mineralogy, and chronology of this lunar granitic fragment with the aims of (1) characterizing the petrological features of granitic fragment occurred in the Th-poor lunar basaltic breccia; and (2) discussing the provenance and implications of this granitic fragment from NWA 10447.

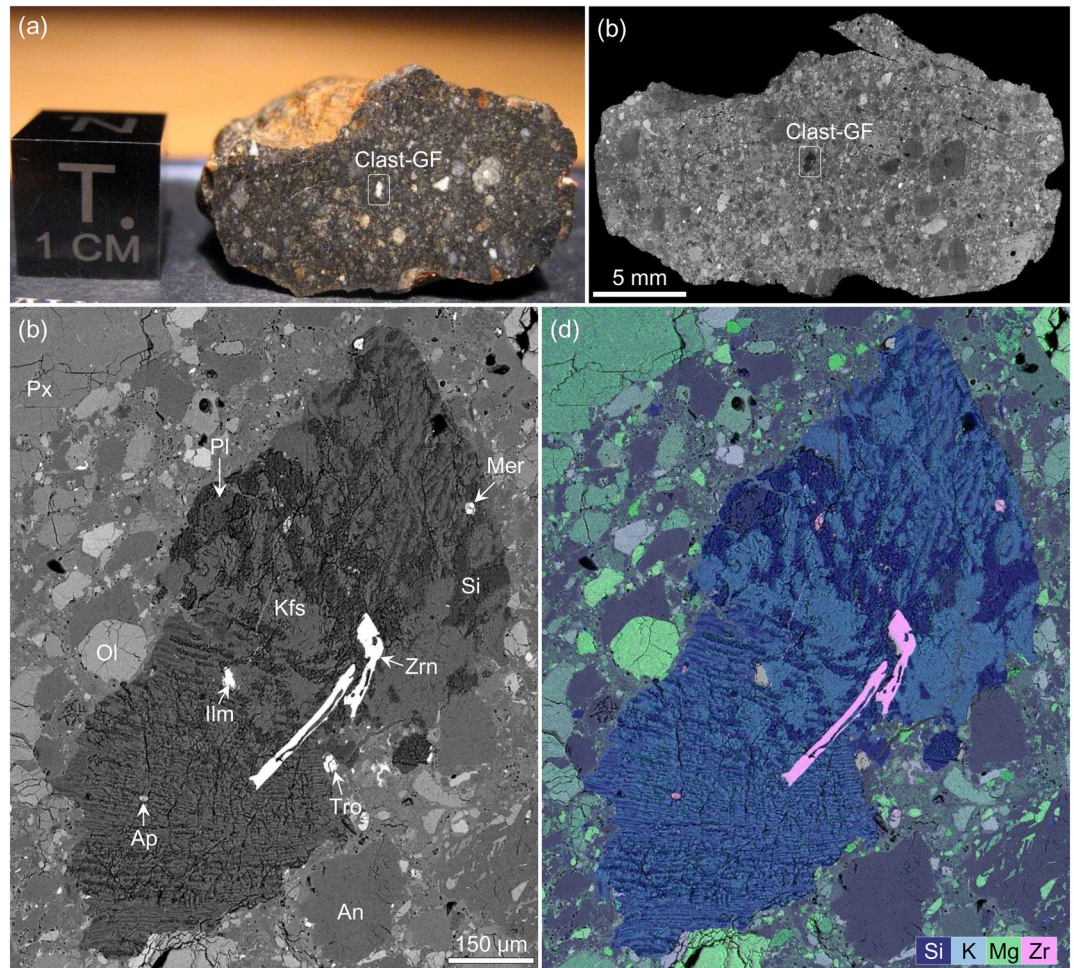
## 2. Sample and Analytical Methods

The breccia meteorite NWA 10447 was provided by Fabien Kuntz, who holds the main mass of this sample (Bouvier et al., 2017). In this study, the polished thick sections of NWA 10447,  $\sim 1.5 \times 2.5$  cm in size, was prepared at the Institute of Geochemistry, Chinese Academy of Sciences (e.g., Figure S1). Then, the studied granitic fragment (i.e., Clast-GF; Figure 1) in this section of NWA 10447 was characterized using a wide range of in situ analytical techniques.

Back-scattered electron (BSE) images of Clast-GF were taken using the FEI Scios dual-beam focused ion beam/scanning electron microscope (FIB/SEM) at the Institute of Geochemistry, Chinese Academy of Sciences. The conditions were as follows: a 15 kV accelerating voltage, a 3.2 nA beam current, and a 7–8 mm working distance. X-ray elemental mapping of Clast-GF was performed using the energy-dispersive spectrometer attached to the FEI Scios FIB/SEM, with the same current and voltage setting noted above. Based on the BSE and X-ray mapping images, the modal mineralogy (vol%) of Clast-GF was estimated. The pixels of each mineral phases in this clast were counted using Photoshop. In addition, the quantitative mineral composition of Clast-GF were determined using the JXA 8230 electron microprobe analyzer (EMPA) at the Institute of Geochemistry, Chinese Academy of Sciences (Table S1). The operating conditions were as follows: a 15 kV accelerating voltage, a 20 nA beam current, and a 1  $\mu\text{m}$  focused beam. Natural minerals and synthetic minerals were used as standards. The typical detection limits for most of the elements were 0.02–0.03 wt%.

Micro-Raman spectroscopy was performed using the Renishaw (RM 2000 and inVia Plus) micro-Raman spectrometer at the Institute of Geochemistry, Chinese Academy of Sciences. The laser (532 nm) energy was 50 mW. Silicon ( $520.7\text{ cm}^{-1}$  Raman shift) was used as the standard to calibrate this instrument. The Raman spectra for the silica phases in Clast-GF were collected under the following conditions: (1) the  $\times 50$  objective was used to focus the excitation beam on a  $\sim 1\text{ }\mu\text{m}$  spot; (2) the acquired spectral range was  $100\text{--}1,200\text{ cm}^{-1}$ ; (3) the exposure time for each spectrum was 15–50 s (1 times); and (4) the power of the laser energy (i.e., 0.5–5 mW) was 1%–10%.

The in-situ U-Pb isotopic system of the zircon in Clast-GF was measured using the CAMECA IMS 1280-HR secondary ion mass spectrometer (SIMS) at the Guangzhou Institute of Geochemistry, Chinese Academy



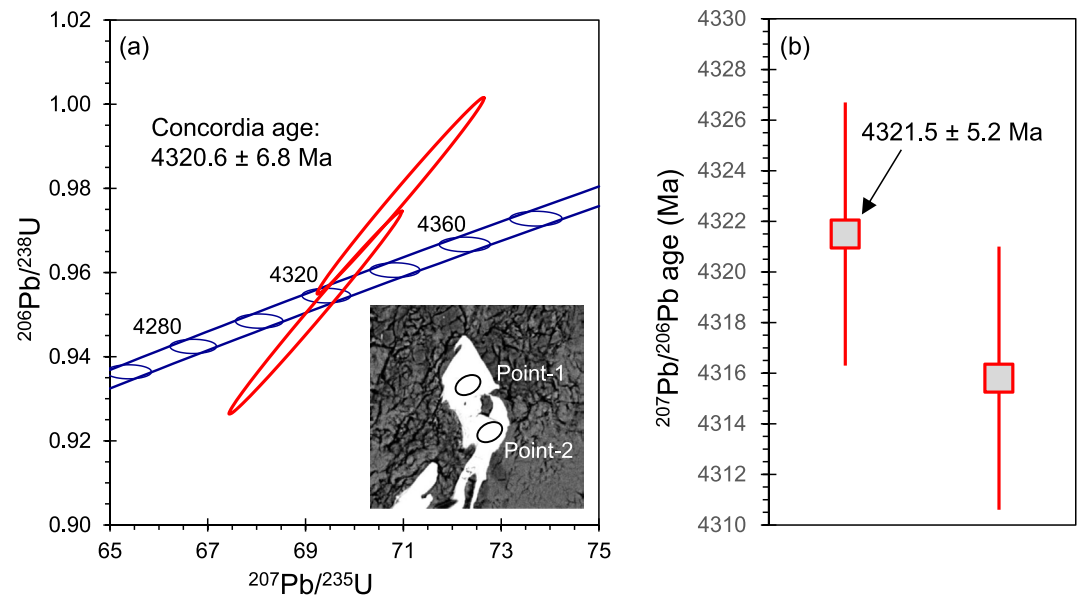
**Figure 1.** Occurrence, texture, and mineralogy of lunar granite fragment Clast-GF in breccia meteorite Northwest Africa (NWA) 10447. (a–b) Hand sample (Courtesy of F. Kuntz) and Back-scattered electron (BSE) images of lunar breccia meteorite NWA 10447. The studied granite fragment (Clast-GF) was marked in these images. (c) BSE image of Clast-GF. (d) X-ray element distribution maps (i.e., Si, K, Mg, and Zr) overlay on the BSE image of Clast-GF. Mineral phases in Clast-GF were labeled: Kfs = K-feldspar, Si = silica, Pl = plagioclase, Px = pyroxene, Ol = olivine, An = anorthosite, Zrn = zircon, Ilm = ilmenite, Tro = troilite, Ap = apatite, and Mer = merrillite.

of Sciences. The detailed analytical procedures were same as those described by Li et al. (2009) and Xu et al. (2020). The  $O^{2-}$  primary ion beam was used, with a spot size of  $20 \times 30 \mu\text{m}$  and a beam intensity of  $\sim 10 \text{ nA}$ . Zircon standard Plesovice was used to calibrate this instrument (Sláma et al., 2008). Another standard zircon (SA 01; Huang et al., 2020) was also analyzed as an unknown sample to monitor the reliability of the entire procedure. The measured  $^{204}\text{Pb}$  was used for the common Pb correction, which was assumed to be surface contamination from the sample preparation, and the composition of the sample was calculated according to the two-stage evolution model (Stacey and Kramers, 1975). The U-Pb dating results are presented in Table S2.

### 3. Results

#### 3.1. Occurrence, Petrology, and Mineralogy

NWA 10447 is a basaltic regolith breccia, which is mainly composed of basaltic clasts, impact melt breccia clasts, volcanic glass fragments, impact glass fragments, and mineral phrases (Figures S1 and S2). Geochemically, this breccia meteorite has a bulk FeO content of 15.15 wt% and relatively low incompatible trace element contents (e.g., Th = 0.79 ppm, Sm = 2.59 ppm, and  $\text{TiO}_2 = 0.6 \text{ wt\%}$ ), which are chemically



**Figure 2.** (a) U-Pb data for the zircon in lunar granite Clast-GF in Northwest Africa 10447. Back-scattered electron image was provided showing the two analyzed points. (b) Concordant  $^{207}\text{Pb}/^{206}\text{Pb}$  ages of the analyzed zircon. Error bars for  $^{207}\text{Pb}/^{206}\text{Pb}$  ages are  $2\sigma$ .

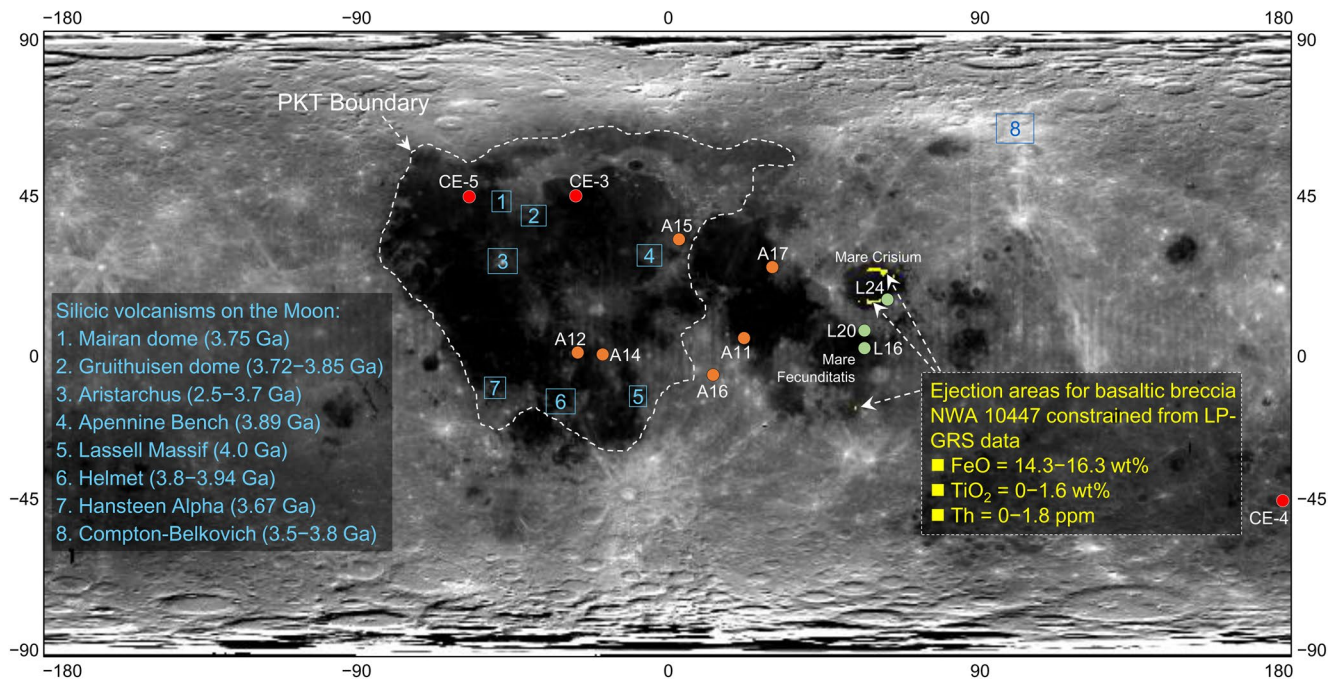
consistent with other lunar basaltic samples ( $\text{FeO} = 16\text{--}22$  wt%) and are distinctly different from the composition of KREEP-rich ( $\text{Th} > \sim 2\text{--}3$  ppm and  $\text{Sm} > \sim 7$  ppm) Procellarum KREEP Terrane (PKT) materials (Figures S3 and S4). The studied lunar granite fragment in NWA 10447 (i.e., Clast-GF) was to be located in the matrix of this meteorite, and the clast is  $\sim 1.2 \times 0.5$  mm in size (Figures 1a and 1b). Texturally, Clast-GF is characterized by micrographic intergrowths of K-feldspar ( $\text{An}_{3.9\text{--}5.7}\text{Ab}_{16.4\text{--}23.2}\text{Or}_{71.0\text{--}78.7}$ ;  $\sim 60$  vol%) and silica ( $\sim 30$  vol%) (Figures 1c and 1d). A few plagioclase grains ( $\text{An}_{33.7\text{--}36.4}\text{Ab}_{53.0\text{--}54.4}\text{Or}_{10.6\text{--}11.9}$ ) are present in minor quantities (i.e.,  $< 2$  vol%) and are associated with the feldspar-quartz intergrowths. Notably, Clast-GF contains two strip-shaped zircons that are  $\sim 20 \times 300$   $\mu\text{m}$  and  $\sim 35 \times 200$   $\mu\text{m}$  in size. In addition, a few ( $< 2$  vol%) accessory mineral phases were also observed in Clast-GF as micron-sized ( $< 15$  micron) grains, including ilmenite, troilite, apatite, and merrillite (Figures 1c and 1d).

### 3.2. Characteristics of Silica Phase

The silica phases in Clast-GF were characterized using the micro-Raman and EPMA techniques. The Raman spectra for these silica phases show that they have Raman peaks at  $127$   $\text{cm}^{-1}$ ,  $202$   $\text{cm}^{-1}$ ,  $353$   $\text{cm}^{-1}$ , and  $464$   $\text{cm}^{-1}$ , which are consistent with the Raman spectra of quartz (Figure S5). Texturally, the quartz grains in Clast-GF are fractured in a hackled pattern (Figure S6), which are similar to the micro-morphology of the quartz particles in the Apollo 12 samples (e.g., 12023,147–10, 12001,909–14, and 12033,634–30; Jolliff et al., 1999; Seddio et al., 2015). The minor-oxide (e.g.,  $\text{FeO}$  and  $\text{TiO}_2$ ) contents of the quartz grains in Clast-GF were determined. The  $\text{FeO}$  and  $\text{TiO}_2$  contents of the quartz grains are  $0.04\text{--}0.14$  wt% and  $0.1\text{--}0.18$  wt%, respectively (Figure S7 and Table S1).

### 3.3. U-Pb Isotopic Compositions and Age of Zircon

Two U-Pb analyses were performed on a relatively large zircon grain ( $\sim 35$   $\mu\text{m}$  in width) in Clast-GF (Figure 2). This zircon has U and Th contents of  $141\text{--}241$  ppm and  $79\text{--}149$  ppm, respectively (Table S2). The SIMS results show that the two U-Pb analyses are concordant, yielding a concordia age of  $4,320.6 \pm 6.8$  Ma ( $2\sigma$  uncertainty; Figure 2a). The measured  $^{207}\text{Pb}/^{206}\text{Pb}$  ages are  $4,321.5 \pm 5.2$  Ma ( $2\sigma$  uncertainty) and  $4,315.8 \pm 5.2$  Ma ( $2\sigma$  uncertainty), which are identical within the uncertainties (Figure 2b). Since the U-Pb system of the zircon within Clast-GF could have been disturbed by impact or thermal events, which are



**Figure 3.** Possible ejection areas for lunar breccia meteorite Northwest Africa 10447 based on its bulk composition (i.e.,  $\text{FeO} = 15.15 \pm 1 \text{ wt\%}$ ,  $\text{TiO}_2 = 0.6 \pm 1 \text{ wt\%}$ , and  $\text{Th} = 0.79 \pm 1 \text{ ppm}$ ). Locations and the ages of silicic volcanisms identified on the Moon have also been shown in this image (see Table S4 for details).

pervasive on the Moon, we took the oldest concordant  $^{207}\text{Pb}/^{206}\text{Pb}$  age (i.e.,  $4,321 \pm 5.2 \text{ Ma}$ ;  $2\sigma$  uncertainty) as the minimum crystallization age of Clast-GF.

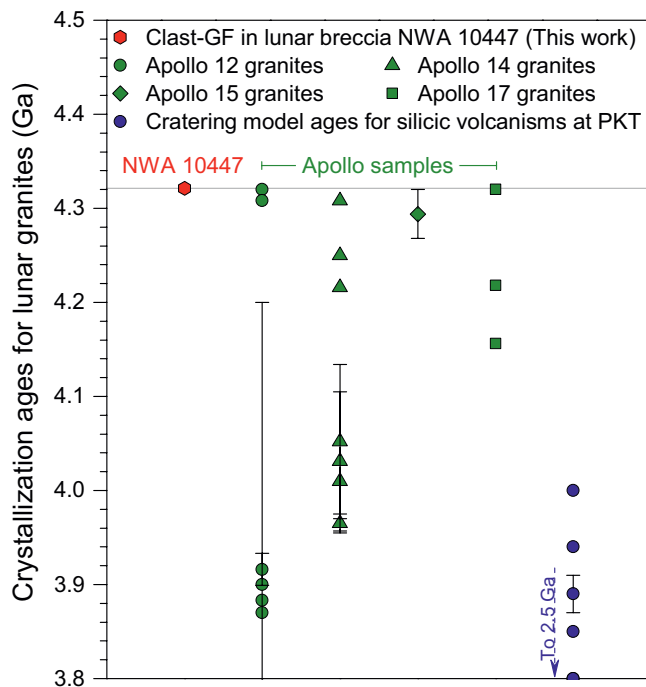
## 4. Discussion

### 4.1. Non-PKT Origin of Lunar Breccia NWA 10447

NWA 10447 is a regolith breccia meteorite consolidated from the surface materials on the Moon. By comparing this meteorite's composition with the global gamma ray spectroscopy geochemical data, the possible ejection regions of the lunar meteorite have been constrained (e.g., Calzada- Diaz et al., 2015; Gross & Joy, 2016; Joy et al., 2010; Zeng et al., 2018; Zeng, Li, et al., 2020). In this study, we used this geochemical data (i.e.,  $\text{FeO} = 15.15 \pm 1 \text{ wt\%}$ ,  $\text{TiO}_2 = \pm 1 \text{ wt\%}$ , and  $\text{Th} = 0.79 \pm 1 \text{ ppm}$ ; Korotev and Irving, 2021; Table S3) as input parameters to locate regolith samples that are compositionally similar to NWA 10447 (see Text S1 for details). The resulting map shows that lunar breccia meteorite NWA 10447 is compositionally similar to the regoliths from the south of Mare Fecunditatis and the Mare Crisium outside of the Non-PKT regions (Figure 3). This implies that Clast-GF within NWA 10447 is most likely a lithic clast from the lunar Non-PKT region where this meteorite was consolidated from. This assumption is further supported by the commonly occurrence of granitic clasts in NWA 10,447: other six granitic clasts (see Figure S2) were observed in this breccia meteorite, indicating that the granitic Clast-GF is more likely local origin rather than the lithology that was ejected from the PKT region by impact events.

### 4.2. Petrogenesis of Clast-GF

The identification of a silica polymorph would give clues to the petrogenesis of the lunar granite samples (e.g., Seddio et al., 2015). Quartz grains with a hackle fracture pattern have been commonly observed in lunar granites, indicating that they were inverted from a high-temperature, low-pressure polymorph (either tridymite or cristobalite; Seddio et al., 2015). The silica phases in Clast-GF were identified as quartz grains with a hackled pattern (Figure S6), which is consistent with an orderly transformation from tridymite or cristobalite (indicative of rapid cooling) at a later time. Thus, it is reasonable to suggest that Clast-GF represents an extrusive lunar lithology (i.e., extrusive silicic volcanism).



**Figure 4.** Comparison of the crystallization age ( $4.32 \pm 0.0052$  Ma;  $2\sigma$ ) of Clast-GF in Northwest Africa 10447 and the crystallization ages of the Apollo granitic samples and the cratering model ages of lunar silicic volcanism identified by remote sensing data (see Table S4 for details). Most error bars in this image are smaller than the symbols.

### 4.3. Implications for the Silicic Volcanism on the Moon

Compared with the granites collected during the Apollo missions, the newly discovered granitic fragment (i.e., Clast-GF) in NWA 10447 exhibits similar textural and mineralogical characteristics to some Apollo granites. Specifically, Clast-GF is an unbrecciated lithology with an igneous texture (i.e., micrographic intergrowths of K-feldspar and silica). This feature is consistent with texture of Apollo granites 12,032,366–19 and 12,032,147–10 (Seddo et al., 2014, 2013). In addition, Clast-GF mainly consists of  $\sim 60$  vol% K-feldspar and  $\sim 30$  vol% silica, with less amounts of accessory mineral phases (i.e., plagioclase, zircon, ilmenite, troilite, and phosphate). Such mineral assemblage is similar to the mineralogy of Apollo granite 12,033, 507 (i.e.,  $\sim 49$  vol% K-feldspar,  $\sim 33$  vol%, and a few amounts of troilite, ilmenite, plagioclase and phosphates; Warren et al., 1987).

Previous studies have shown that lunar magmatic processes that are capable of producing highly evolved granites or silicic volcanism occurred over an extended period of time. Specifically, the Apollo-returned granite (or felsite) samples have yielded isotopic dates ranging from  $4,320 \pm 3$  Ma (Pre-Nectarian) to  $3,884 \pm 3$  Ma (Nectarian) (e.g., Meyer et al., 1996; Thiessen et al., 2018; Zhang et al., 2012). Furthermore, the ages of the silicic volcanism on the Moon ( $\sim 2,500$  Ma to 4,000 Ma) (Figure 3) have also been estimated using the crater-counting method. Compared with these results, the studied granitic fragment (Clast-GF in NWA 10447) has a crystallization age similar to the oldest granites sampled by the Apollo 12 and 17 missions (i.e.,  $\sim 4.32$  Ga; Figure 4). The existence of ancient lunar granite Clast-GF provides new evidence for the existence of more ancient (i.e.,  $\sim 4.32$  Ga) silicic volcanism on the Moon, which produced highly evolved lithologies such as Clast-GF.

The occurrence of lunar silicic volcanism has been observed to be mainly distributed in the PKT region on the nearside of the Moon and in Compton-Belkovich on the far side of the Moon (Figure 3). Similarly, all of the Apollo granites were sampled in/near the PKT regions during the Apollo 12, 14, 15, and 17 missions. Few lunar granite samples have been found in the Non-PKT area of the Moon. Clast-GF in breccia meteorite NWA 10447 may represent a rare lunar granitic fragment from the Non-PKT region of the Moon (i.e., the mare region with low Th and  $\text{TiO}_2$ ).

## 5. Conclusions

1. In lunar basaltic breccia meteorite NWA 10447, a mm-sized granitic fragment (Clast-GF) was recognized. Clast-GF have similar petrological and mineralogical feature similar to some granites (e.g., 12032,366–19) returned by Apollo missions. This granitic fragment has a crystallization age of  $4,321.5 \pm 5.2$  Ma ( $2\sigma$ ), which is consistent with the age of the oldest granite sampled during the Apollo 12 and 17 missions.
2. Unlike the regoliths from Apollo landing sites within PKT, the basaltic breccia NWA 10447 has relatively low contents of Th and  $\text{TiO}_2$  and is compositionally similar to the regoliths from the Moon's Non-PKT regions (e.g., Mare Crisium). The identification of granitic Clast-GF (and other granitic clasts) in this meteorite provides evidence for that lunar granite most likely occurs outside of the PKT of the Moon (i.e., the mare region with low Th and  $\text{TiO}_2$ ).
3. The granitic Clast-GF adds to the growing evidence of oldest ( $\sim 4.32$  Ga) silicic volcanism on the Moon. This may provide further constraints on lunar ancient silicic magmatism that was not recognized by the Apollo missions and remote-sensing observations.
4. This work implies that much is unknown about lunar granite and the silicic magmatic activity in the Non-PKT region of the Moon. In the future, investigation of more granitic lithologies from other lunar

meteorites (e.g., the Th-poor breccias) will be helpful in extending our understanding of the silicic volcanism across the Moon

### Data Availability Statement

Lunar Prospector data used for the provenance of NWA 10447 is accessible at the NASA Planetary Data System (<https://pds-geosciences.wustl.edu>). The petrological, geochemical, and chronological data used in this manuscript are available online (<https://doi.org/10.6084/m9.figshare.13571423>).

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