Estimating uncertainties on α-ejection corrections relevant for the apatite (U-Th)/He method using nano-CT

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A systematic method for estimating error on α–ejection corrections doesn't exist:

Getting accurate dates from the apatite (U-Th)/He method requires corrections for α-ejection because He atoms can travel up to 20 µm[1] and can be lost from the crystal.

- These corrections are based on grain size and idealized geometries with smooth faces[1].
- α-ejection is based on number of the terminations, crystal dimensions, and a 'hexagonal' or 'elliptical' geometry.
  - We aim to expand the parameters α-ejection corrections are based on.
- Uncertainties on these corrections are poorly constrained and often not included when reporting apatite (U-Th)/He data.

	Goal	No. of Grains Analyzed	Description of Grains	Width of Grains	Micro-CT Resolution
Glotzbach et al. (2019)[3]	Develop a new method of measuring grains in 3D to reduce errors in α-ejection corrections.	24 grains	Rounded; Irregular; Hexagonal; 2 N_p, 0 N_p	50 µm < x <125 µm	1.2 µm
Herman et al. (2007)[2]	Approximate α-ejection corrections for irregular grains utilizing micro-CT for each grain.	11 grains	Detrital	>100 µm	6.3 µm

Table 1. Summary of previous studies which attempted to solve this problem using micro-CT.

Research Goals:

- Use nano-computed tomography (nano-CT) to compare actual α–ejection corrections to manually calculated α–ejection corrections.
- Formulate a classification system for describing apatite grains.
- Assess if there is a relationship between the magnitude of uncertainty and the size, degree of rounding, or amount of surface relief of an apatite.

Determine a 'rule of thumb' for estimating errors on α-ejection corrections based on specific apatite characteristics.

	Goal	No. of Grains Analyzed	Description of Grains	Width of Grains	Nano-CT Resolution
This study	Create a **realistic** and **practical** set of guidelines for estimating uncertainties for α-ejection corrections.	293	Intrusive, volcanic, detrital, basement	40 µm < x < 170 µm	0.63 µm

Table 2. Summary of this study.

Apatite Selection Parameters:

Samples:

	Type	Age
Marlborough Fault System-05	Detrital	Miocene
Marlborough Fault System-07	Detrital	Miocene
Fish Canyon Tuff	Volcanic	Oligocene
Whitehorn	Intrusive	Cretaceous
McClure	Intrusive	Cambrian
Superior	Basement	Precambrian
Bail	Basement	Precambrian
Deep Creek	Intrusive	Eocene

Table 3. Descriptions of the nine samples used to pick grains for analysis.

Grain Quality:

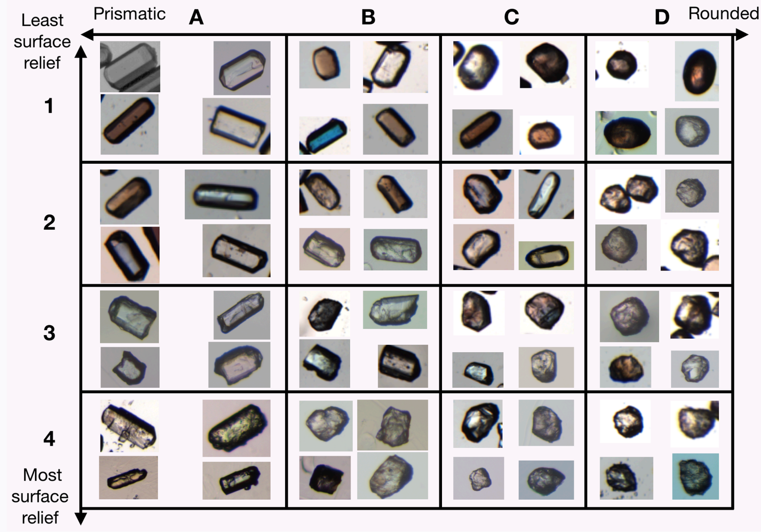


Fig 1. QUALM ("Quality Matrix"). Each apatite was graded on degree of roundness and surface relief. Apatite grains can be systemically described across studies using a QUALM.

Grain Size:

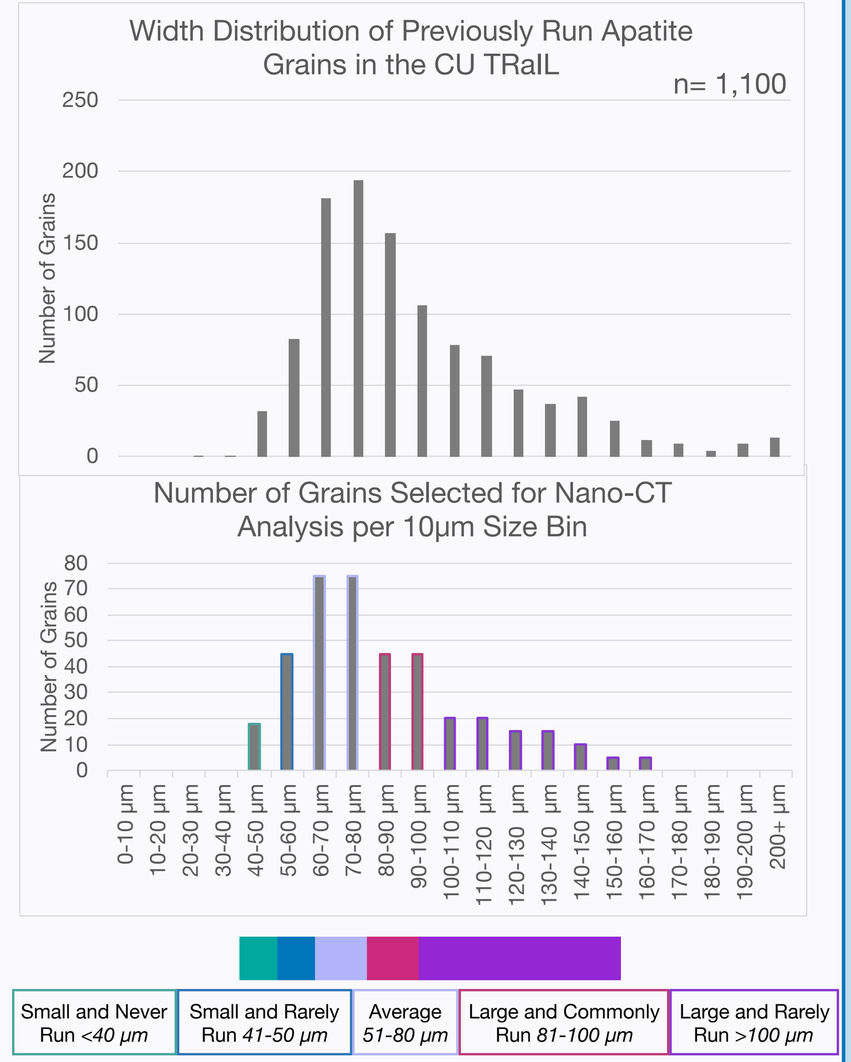
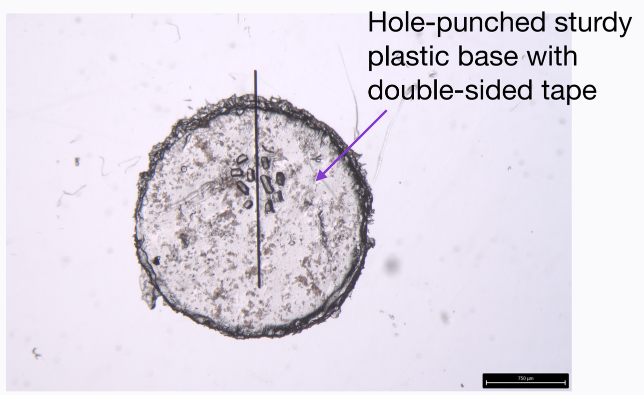
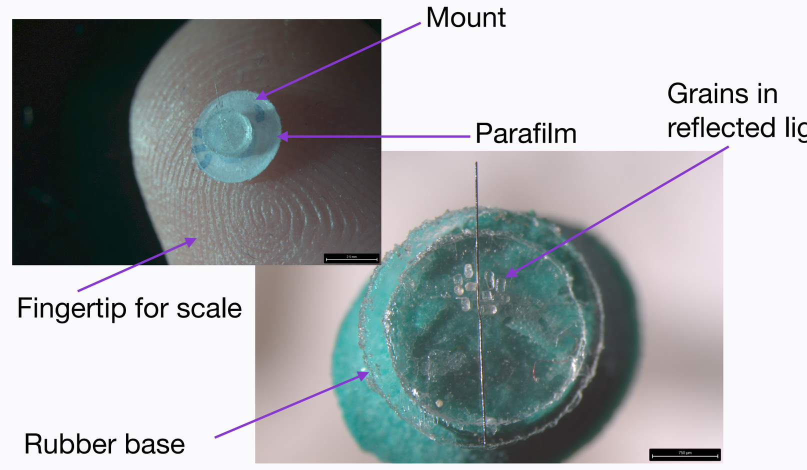


Fig 2. The width distribution of 1,100 previously run apatite grains in the CU Boulder TRaIL was analyzed and normalized to 400 grains which resulted in the number of grains selected for analysis in each 10 µm size bin. The 10 µm bins were grouped into five generalized size categories.

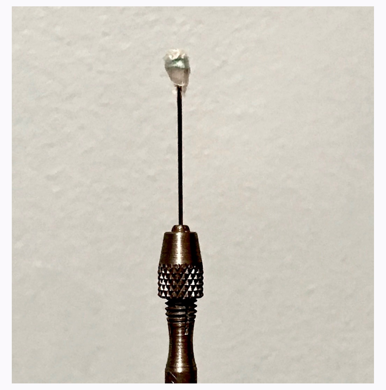
Data Acquisition Process:



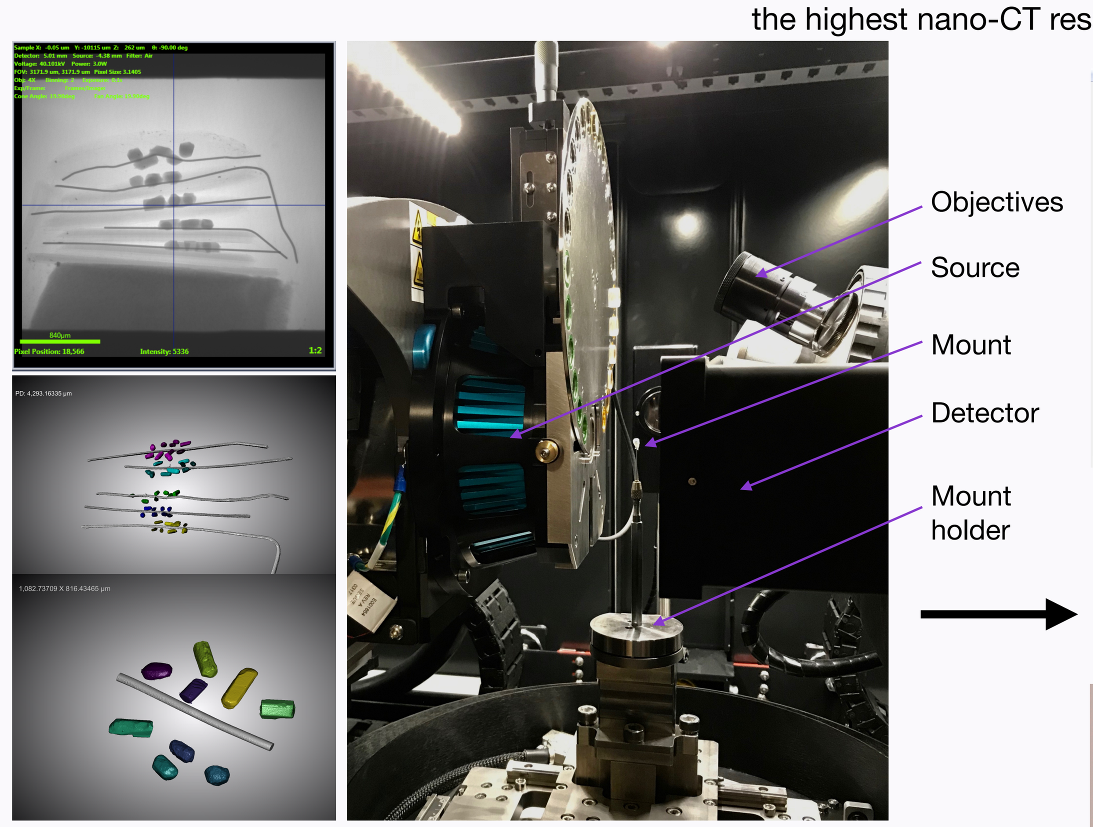
Step 1. A single 2mm round with 10 apatite grains arranged around 0.025mm stainless steel wire for orientation. (transmitted light)



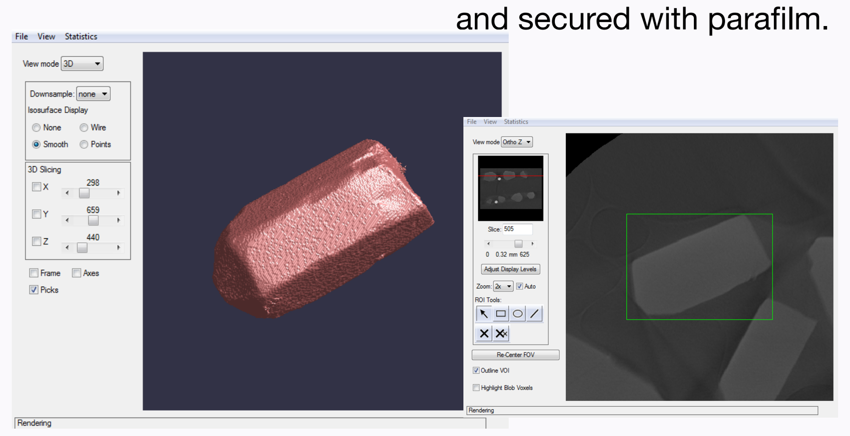
Step 2. Five rounds with 10 apatite grains each were stacked vertically with the wires aligned so the grains occupied an area ≤ 1.3mm to achieve the highest nano-CT resolution.



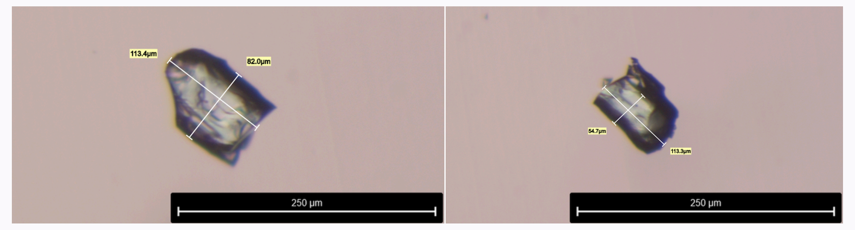
Step 3. Stacks of 5 rounds were mounted on a rubber base superglued to the head of a dressmakers pin and secured with parafilm.



Step 4. Nine mounts were scanned in a X-ray microscope using nano-CT. 'Scout and Scan' procedure was used:
-A x4 map (~3 µm pixel size) was created. (top left)
-Then, 1-3 layers were scanned at a time with x20 objective (~0.63 µm pixel size).*
Pictured: A full mount (middle left) and a single mount (bottom left) as reconstructed in Dragonfly.



Step 5. Exported nano-CT data as .tiff stacks which were manually processed by separating apatite 'blobs' from the matrix. Blob 3D calculated 'actual' α-ejection corrections, sphere normalized surface to volume ratio, and surface area.



Step 6. Photomicrographs of each grain were taken and manually measured by two people using a Leica stereomicroscope. Number of terminations, geometry, and QUALM value were also recorded for each grain.

Fig 3. Steps 1-6 outline the data acquisition process.

* Xradia Versa XRM-520, Zeiss, Dublin, CA. Energy settings: 40V, 3.0W; air filter; 3,201 projections; 2.5-2 sec exposure time to achieve ~5,000 intensity values; source (-4.3mm), detector (4.9mm).

Preliminary results suggest uncertainties on α-ejection corrections for the most commonly analyzed grains of ~10%:

Error for α-ejection Corrections Based on QUALM Value

Prismatic → Rounded; Least Surface Relief ↓ Most Surface Relief; Increasing Percent Error

	A	B	C	D
1	8.7%	7.1%	7.7%	9.4%
2	10.1%	9.4%	12.7%	3.9%
3	5.2%	17.3%	15.7%	5.5%
4	9.0%	4.9%	19.7%	19.9%

Fig 4. Average percent error for each QUALM value between the 'actual' α-ejection and manual α-ejection corrections. Percent error increases towards the bottom right corner (D4).

Number of Grains Analyzed in Each QUALM Value

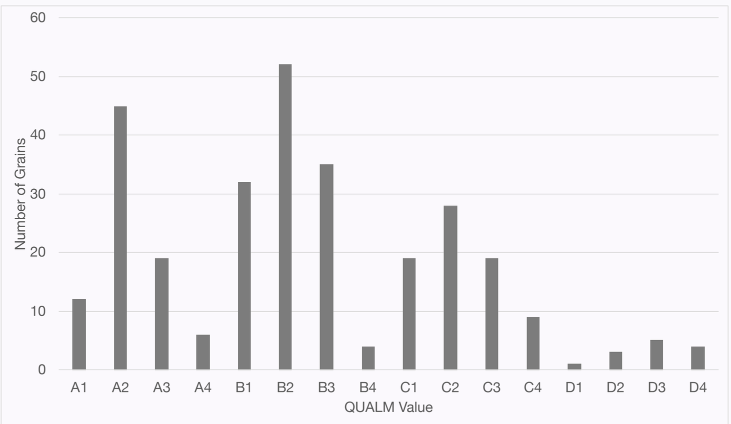


Fig 5. Low numbers of grains at higher degrees of surface relief and rounding is at least partly due decreased ability to identify apatite visually.

Error for α-ejection Corrections Based on Grain Size

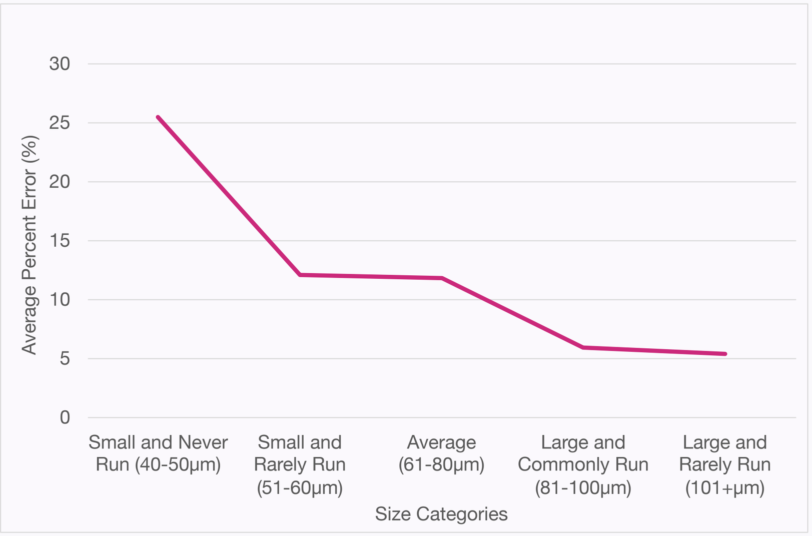


Fig 6. Average percent error for each of the 5 generalized size categories between the 'actual' α-ejection and manual α-ejection corrections[4]. Larger grains have less error associated with them.

Size Category	Number of Grains
Small and Never Run (40-50 µm)	15
Small and Rarely Run (51-60 µm)	35
Average (61-80 µm)	124
Large and Commonly Run (81-100 µm)	62
Large and Rarely Run (>100 µm)	57

Table 4. Total number of grains analyzed per size category.

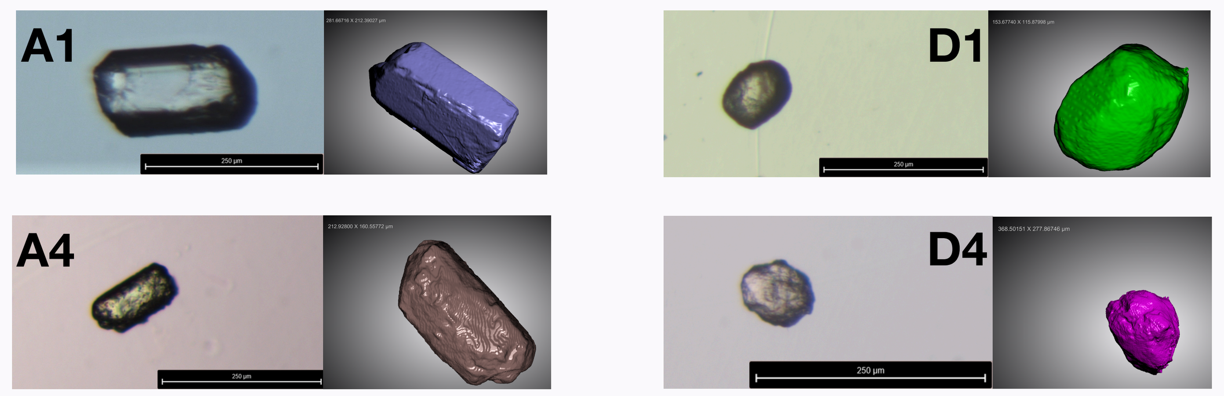


Fig 7. Examples of endmember apatite grains classified with the QUALM in 2D (photomicrograph) and in 3D (reconstructed in Dragonfly).

Ongoing Work:

- Post-process images in Dragonfly using size-exclusion to remove artifacts of the scanning process/noise, binarize the file, and get dimensions, surface area, and volume to compare to Blob 3D α-ejection corrections.
  - Assign QUALM values to grains in 3D and compare to 2D QUALM assignments to assure consistency.
- Have 1-2 more people measure the grains in the dataset and assign QUALM values (in 2D) using the matrix to assure consistency.
- Increase the number of grains with QUALM values B4, C4, D1, D2, D3, D4.
- More thoroughly quantify the controls on uncertainty associated with α-ejection corrections.
- Consider the implications for uncertainties on apatite effective Uranium concentration values.

References: [1] Farley et al. (1996) *Geochimica et Cosmochimica Acta* DOI: 10.1016/S0016-7037(96)00193-7 [2] Herman et al. (2007) *Chemical Geology* DOI: 10.1016/j.chemgeo.2007.03.009 [3] Glotzbach et al. (2019) *Chemical Geology* DOI: 10.1016/j.chemgeo.2018.12.032 [4] Calculated from: Ketcham et al. (2011) *Geochimica et Cosmochimica Acta* DOI: 10.1016/j.gca.2011.10.011

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