The paper now includes the following Appendix.

**Appendix A: Calculating ESRFT, mean FT, and eU**

*ESRFT and mean FT*

The starting point calculating the equivalent-FT sphere radius (ESRFT) when FT values are provided for each decay chain is the FT equation for a sphere (Farley et al., 1996; Ketcham et al., 2011):

|  |  |
| --- | --- |
|  | (A1) |

where *R* is sphere radius, *S* is stopping distance, and *B* is an adjustment factor for the 3rd-degree polynomial term to account for *S* being the weighted mean of stopping distances along branching decay chains, rather than a single stopping distance. For U and Th decay chains *B* should be 1.31, and for single stopping distances it should be 1 (Ketcham et al., 2011).

Solving this equation for *S/R* over the FT range from 0.5 to 1 using a 3rd-degree polynomial to match the effect of the cubic term gives:

|  |  |
| --- | --- |
|  | (A2a) |
|  | (A2b) |

The polynomial in Equation (A2a) is the appropriate one to use for data to be reported in age tables; Equation (A2b) is provided for completeness, and may be useful for comparing to other calculations that use mean *S* values to represent chains.

The FT value to use is the weighted mean incorporating the separate factors FT,238, FT,235 and FT,232, accounting for different alpha productivity along each chain. Expanding the approach of Farley (2002) to account precisely for 235U, we calculate:

|  |  |
| --- | --- |
|  | (A3a) |
|  | (A3b) |

So that the weighted mean, , is

|  |  |
| --- | --- |
|  | (A4) |

Solving the result of Equation (A2) for ESRFT requires the analogous calculation to determine the weighted mean stopping distance, :

|  |  |
| --- | --- |
|  | (A5) |

where *S*238, *S*235, and *S*232 are the weighted mean stopping distances for each decay chain (18.81 µm, 21.80 µm, and 22.25 µm, respectively, for apatite, but the calculation applies to any mineral). Then, combining equations (A2) and (A5):

|  |  |
| --- | --- |
|  | (A6) |

*eU*

The earliest mention of *eU*, or effective uranium with respect to He production, we are aware of is in Shuster et al. (2006), who put forward the formula

|  |  |
| --- | --- |
|  | (A7) |

where brackets indicate composition in ppm, without a detailed description of its derivation. Converting from elemental or isotopic compositions in ppm to an equivalent alpha particle production rate requires accounting for decay constants, isotopic proportions, alpha particle production, and atomic mass. We calculate the present-day alpha production rate *R* (here calculated in  g-1 yr-1) as:

|  |  |
| --- | --- |
|  | (A8) |

where A is Avogadro’s number, is the decay constant, *p* is isotopic proportion, *N* is number of alpha particles produced in the decay chain, and *ma* is atomic mass. The eU factor is then calculated by dividing the Th and Sm by the combined U utilizing the values in Table A1, we find the *eU* equation to be slightly different:

|  |  |
| --- | --- |
|  | (A9) |

We do not know the reason for the small discrepancy with Equation (A7), but the ~1% difference in the effect of Th is not likely to be important for current uses of *eU*. The 0.238 factor has a likely uncertainty of ±0.002; the 232Th half-life currently recommended by the nuclear chemistry community has only three significant figures based on a weighted average of several determinations using different methodologies (Browne, 2006; Holden, 1990), whereas the geological community has adopted the value from the single study with the highest reported precision (Le Roux and Glendenin, 1963; Steiger and Jäger, 1977).

We include Sm for completeness, but as its alpha decay has a relatively low recoil energy it is not clear whether simply counting the particle is the most appropriate way to include its potential contribution to damage which affects helium diffusivity. An alternative formulation can be posed in terms of energy deposition (kerma; Shuster and Farley, 2009):

|  |  |
| --- | --- |
|  | (A10) |

where *E* is the mean alpha particle recoil energy for the decay chain. The revised kerma-based quantity, *eUk*, is then:

|  |  |
| --- | --- |
|  | (A11) |

This relation predicts that Sm will have an even lower relative contribution to diffusivity than indicated in Equation (A9), but that Th will be 11% more potent, due to its higher mean recoil energy compared to 238U. We do not currently recommend this approach, but it does pose a potentially testable hypothesis.

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**Table A1**: Values used for calculating eU

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | 238U | 235U | Th | Sm (total) | 147Sm |
|  (1/yr) | 1.55125E-10 | 9.8485E-10 | 4.9475E-11 | 6.539E-12 | 6.539E-12 |
| *p* | 0.9928 | 0.0072 | 1 | 0.1499 | 1 |
| *ma* (g/mol) | 238.029 | 238.029 | 232.039 | 150.36 | 147 |
| *N* (/chain) | 8 | 7 | 6 | 1 | 1 |
| /g/yr | 3.117E+12 | 1.256E+11 | 7.70E+11 | 3.93E+09 | 2.68E+10 |
| eU factor |  |  | 0.238 | 0.0012 | 0.0083 |
|  |  |  |  |  |  |
| *E* (MeV) | 5.359 | 5.946 | 5.990 | 2.247 | 2.247 |
| energy deposited/g/yr | 1.671E+13 | 7.468E+11 | 4.61E+12 | 8.82E+09 | 6.02E+10 |
| eUk factor |  |  | 0.264 | 0.0005 | 0.0034 |

1 Values for U and Th from Steiger and Jäger (1977).

**Appendix B: Evaluation of accuracy and precision in Blob3D FT calculations**

This Appendix describes a series of tests that demonstrate the accuracy and precision of the methods for FT calculations implemented in Blob3D (Ketcham, 2005). All calculations are performed in Blob3D, or with scripts in IDL, the computer language in which Blob3D is written.

*Centered spheres*

In the first set of tests, we use spheres, which Herman et al. (2007) recognized as a good test shape because its surface is poorly approximated by coarse stacked cubes. We begin with a 1283 voxel field, and select all voxels with centers within 63 voxel widths of the center of the volume, creating a 63-µm radius sphere with a 1-voxel-thick black boundary on all sides. Four additional lower-resolution versions were then created by rebinning the original data set to make volumes with 643, 323, 163, and 83 voxels; these data sets were then padded with an additional layer of black (nonselected) voxels on three sides to ensure the spheres had a black boundary on all sides for Blob3D processing. In the 8-bit data volumes, selected voxels have a value of 255 (white) and non-selected ones a value of 0 (black).

If the voxel width is 1 µm in the 1283 data set, the resulting ideal sphere radius is 63 µm, which has an FT,238 correction of 0.7777 (stopping distance 18.81 µm). Because of voxelation effects, the actual volume selected will be slightly different than the ideal case; for example, the volume in the 1283 data set corresponds to an equivalent-sphere radius (ESR) of 63.02 µm. With each rebinning step, doubling the voxel size roughly maintains the original volume, simulating lower resolution; i.e. 2-µm voxels for the 653-voxel data set, 4-µm for 333, 8-µm for 173, and 16-µm for 93. We ran an initial set of tests using these voxel sizes, and an additional set with the voxel size halved, corresponding to a 31.5-µm radius crystal, close to the lower end of the practical limit (FT,238 = 0.5655).

Because the calculation employs a Monte Carlo algorithm, answers change slightly from run to run, so for each data set and resolution results from five Blob3D runs were used to gauge precision. Results are provided in Table B1, and shown in Figure B1 as the mean measured (calculated) FT divided by the ideal value for the ESR of the volume actually selected at each resolution, with bars showing one standard error.

Results for the 63-µm sphere test are in Table B1A and Figure B1A. Solid symbols show the result of the normal Monte Carlo analysis, with results accurate to within 0.1% at up to a 4-µm voxel size, but mean errors rise to approach 1% with 8-µm voxels. Half-tone symbols show the result of altering the processing by first super-sampling the volume, subdividing each voxel into a 33 set and then smoothing the expanded data volume with a 5-voxel-wide filter, followed by re-binarizing the data with a threshold (value 127) prior to the Monte Carlo analysis. This step improves accuracy at 8-µm resolution to within 0.4% on average, and also further reduces the sub-0.1% error at the 4-µm level. However, the 127 re-threshold value is not the optimal one, as it slightly shrinks the volume due to the overall convex shape of the grain, so the algorithm finds the optimal threshold that reproduces as closely as possible the pre-super-sampled grain volume. The result improves the 8-µm calculation yet more, reducing the mean error to just over 0.2%, and even with 16-µm voxels the error is only just over 0.5%. This improvement also demonstrates that getting the volume correct is a primary control on the accuracy of the FT calculation; this principle is used to examine the case of non-centered spheres later in this Appendix.

Remaining tests use the convention that when voxel sizes are 4 µm and higher the constant-volume super-sampled approach is used; the only cost of super-sampling is slightly more computing time, which is still less than 1 second per grain (but could rise above this level if employed with smaller voxels and larger grains). The 31.5-µm sphere test (Table B1B, Fig. B1B) shows similar results as the larger case; mean errors are less than 0.5% up to voxel sizes of 8 µm.

*Cylinders*

As most apatite (and zircon) grains are elongate, we also tested cylinders as a close-to-worst-case endmember, again because a round outline is more poorly approximated by cubes than a hexagonal or tetragonal one. We created the cylinders by stacking 510 63-voxel-radius circles with blank slices at each end to achieve an aspect ratio close to 4, and down-sampled as with the sphere test four times by powers of 2. Results are shown for the 63-µm and 31.5-µm cases, with respective ideal FT,238 values of 0.8350 and 0.6772, in Table B1C,D and Figure B1C,D. Even in the coarsest-resolution cases, the mean calculated FT,238 values are only off of the ideal by 0.3%.

*Non-centered spheres*

In their Monte Carlo FT implementation, Herman et al. (2007) report poor precision for small spheres when their centers are not centered in a voxel, with errors rising to several percent for a 40-µm radius sphere with 6.3-µm voxels across a range of center locations (calculated FT range ~0.58-0.67). Errors of this magnitude correspond to the effect of getting the radius wrong by plus or minus almost an entire voxel.

We tested for “voxelation” effects on dimensional measurement by running 100,000 trials randomizing the location of the sphere center in a voxel grid using the same radius and voxel size, once again selecting all voxels with centers within the radius of the randomized center. Converting the resulting volumes to sphere-equivalent radii, we got a mean radius error of 0%, maximum radius errors of +0.8/-1.1% and a standard deviation of 0.2%. At 40 µm (a severe case) a 1% change in radius leads to a ±0.5% change in FT,238 (range 0.6494-0.6561). Together, these results indicate that the degree to which a sphere is off-center to the CT voxel grid has only a very small effect on its measured size, and a correspondingly smaller effect on the FT determination.

There is a case where resolution is a concern, however, which is when the grain size approaches the “true” data resolution. All CT data are blurry to some extent, due to the finite size of the X-ray focal spot and detector elements, among other factors (ASTM, 2011). This blurring can be characterized as a point-spread function (PSF), which can be considered as a smoothing kernel which “blurs” reality as the CT process translates it into a voxel grid. If the smoothing function width, which can be roughly estimated as the number of voxels it takes to fully transition from one material into another across a flat interface (Ketcham et al., 2010) approaches the grain radius, it can affect grain size and shape measurement (Ketcham and Mote, 2019). Typical PSF widths are on the order of 3-5 voxels in most CT data, and so as a rule of thumb the voxel size should be limited to less than 20% of the grain shortest dimension. Even in this case accurate grain measurements are possible, but require additional steps and calibrations, as described by Ketcham and Mote (2019).

We are thus confident that our implementation provides a high degree of accuracy and precision on even very small grains at low resolutions where voxel sizes are up to 20% of the radius.

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**Table B1**: Results of Blob3D measurement of synthetic spheres and cylinders.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Resolution  (µm) | Sampling1 | ESRm2 (µm) | FT,238,ideal3 | FT,2384 | FT,238 | FT/FT,ideal |
| 1. Sphere radius = 63 µm | | | | | | |
| 1 | normal | 63.02 | 0.7778 | 0.7776(03) | 0.0006 | 0.9997(03) |
| 2 | normal | 63.02 | 0.7778 | 0.7773(03) | 0.0006 | 0.9994(03) |
| 4 | normal | 63.06 | 0.7779 | 0.7774(05) | 0.0011 | 0.9993(06) |
| 4 | super | 63.06 | 0.7779 | 0.7779(05) | 0.0012 | 0.9999(07) |
| 8 | normal | 63.03 | 0.7779 | 0.7707(04) | 0.0009 | 0.9909(05) |
| 8 | super | 63.03 | 0.7779 | 0.7754(02) | 0.0004 | 0.9969(03) |
| 8 | super,cv | 63.03 | 0.7779 | 0.7763(06) | 0.0013 | 0.9980(08) |
| 16 | super,cv | 62.61 | 0.7764 | 0.7723(03) | 0.0007 | 0.9947(04) |
| 1. Sphere, radius = 31.5 µm | | | | | | |
| 0.5 | normal | 31.51 | 0.5656 | 0.5651(01) | 0.0003 | 0.9991(02) |
| 1 | normal | 31.51 | 0.5656 | 0.5657(05) | 0.0011 | 1.0002(09) |
| 2 | normal | 31.53 | 0.5658 | 0.5649(09) | 0.0019 | 0.9983(15) |
| 4 | super, cv | 31.52 | 0.5657 | 0.5650(04) | 0.0008 | 0.9989(06) |
| 8 | super, cv | 31.31 | 0.5629 | 0.5607(05) | 0.0011 | 0.9961(09) |
| 1. Cylinder, radius = 63 µm, height = 510 µm | | | | | | |
| 1 | normal | - | 0.8350 | 0.8346(06) | 0.0003 | 0.9995(07) |
| 2 | normal | - | 0.8350 | 0.8352(03) | 0.0007 | 1.0002(04) |
| 4 | super, cv | - | 0.8350 | 0.8350(05) | 0.0010 | 1.0000(06) |
| 8 | super, cv | - | 0.8334 | 0.8318(05) | 0.0011 | 0.9981(06) |
| 16 | super, cv | - | 0.8287 | 0.8267(04) | 0.0008 | 0.9976(04) |
| 1. Cylinder, radius = 31.5 µm, height = 255 µm | | | | | | |
| 0.5 | normal | - | 0.6772 | 0.6774(04) | 0.0009 | 1.0003(06) |
| 1 | normal | - | 0.6772 | 0.6781(04) | 0.0009 | 1.0014(06) |
| 2 | normal | - | 0.6770 | 0.6767(07) | 0.0015 | 0.9995(10) |
| 4 | super, cv | - | 0.6740 | 0.6732(04) | 0.0009 | 0.9987(06) |
| 8 | super, cv | - | 0.6651 | 0.6632(07) | 0.0016 | 0.9971(10) |

1Sampling is either normal, super-sampled, or super-sampled maintaining constant volume

2ESRm: measured equivalent sphere radius, as the voxelated spheres had slightly different volumes than ideal ones.

3FT,238,ideal: FT,238 value (for the 238U stopping distance for apatite) for the given shape with the voxelated volume and, for cylinders, aspect ratio.

4FT,238: mean measured FT,238 value over 5 trials, with estimated precision in parentheses.

**Table B2**: IDL code for conducting off-center sphere volume test

; TestSphereVolumes

;

; Generates a series of voxelated spheres with centers at random points within the

; central voxel, and evaluates the effect on apparent sphere size.

;

; For each test, a voxel field is generated, and a random 3D coordinate is generated

; within the central voxel. The routine then determines all voxels whose centers

; are within the sphere radius of the central coordinate, and reports the resulting

; volume and sphere-equivalent radius.

;

; INPUT PARAMETERS:

; None (only through Keyword Parameters)

;

; OUTPUT:

; Prints to console a tab-delimited table.

; Columns correspond to:

; Volume: Volume of voxels in selected region

; Volume/TrueVol: Denominator is true volume for sphere with given radius

; Abs(1-Vol/TrueVol): Normalization to show fractional error

; Radius: Equivalent spherical radius of selected region

; Radius/TrueRad: Denominator is true radius, as entered by user

; Abs(1-Rad/TrueRad): Normalization to show fractional error

; Rows correspond to mean, standard deviation, maximum, and minimum of each

;

; KEYWORD PARAMETERS:

; NUMTESTS: Number of random spheres to generate; default = 100

; VOXELSIZE: Voxel edge length; units arbitrary, but same as radius; default = 6.3

; SPHERERAD: Sphere radius; units arbitrary, but same as voxel size; default = 40.

; SHOW: Set to show an animation of central slice through each voxelated sphere.

;

; CALLING SEQUENCE:

; TestSphereVolumes, /SHOW, NUMTESTS=100000L, VOXELSIZE=6.3, SPHERERAD=40.

;

; MODIFICATION HISTORY:

; Written by: Rich Ketcham, 6 February 2019

**Pro** **TestSphereVolumes**, SHOW=show, NUMTESTS=numTests, VOXELSIZE=voxelSize, $

SPHERERAD=sphereRad, PSF\_RAD=psfRad, THRESH\_VAL=threshVal

**if** **NOT** **Keyword\_Set**(numTests) **then** numTests = **100**

headings = ["Volume","Vol/TrueVol","Abs(1-Vol/TrueVol)","Radius", $

"Rad/TrueRad","Abs(1-Rad/TrueRad)"]

results = **FltArr**(**N\_Elements**(headings),numTests)

**if** **NOT** **Keyword\_Set**(voxelSize) **then** voxelSize = **6.3** ; microns

**if** **NOT** **Keyword\_Set**(sphereRad) **then** sphereRad = **40.** ; microns

trueVol = (**4.**/**3.**)\***!PI**\*sphereRad^**3**

voxelVol = voxelSize^**3**

arrayDim = **Ceil**(sphereRad/voxelSize)\***2**+**1**

dv = **RandomU**(seed,**3**,numTests)-**0.5** ; Displacements inside central voxel

**for** testNum=**0**,numTests-**1** **do** **begin**

; Calculate distance from every voxel to sphere center

mid = dv[\*,testNum] + arrayDim/**2.0**

sph = **FltArr**(arrayDim, arrayDim, arrayDim)

**for** i=**0**,arrayDim-**1** **do** $

**for** j=**0**,arrayDim-**1** **do** $

**for** k=**0**,arrayDim-**1** **do** **begin**

sph[i,j,k] = (i-mid[**0**])^**2** + (j-mid[**1**])^**2** + (k-mid[**2**])^**2**

**endfor**

sph = **sqrt**(sph)

; Accept all voxels with centers closer to midpoint than radius (in voxels)

sph = (sph **LE** sphereRad/voxelSize)

**if** **Keyword\_Set**(show) **then** **begin**

**tvscl**, **Congrid**(sph[\*,\*,arrayDim/**2**],**512**,**512**)

**Wait**, **0.1**

**endif**

vol = **Total**(sph)\*voxelVol

esRad = (vol\***3.**/(**4**\***!PI**))^(**1.**/**3.**)

**if** **Keyword\_Set**(show) **then** **print**, **Total**(sph), esRad, vol/trueVol

results[\*,testNum] = [vol, vol/trueVol, **Abs**(**1.**-vol/trueVol), esRad, $

esRad/sphereRad, **Abs**(**1.**-esRad/sphereRad)]

**endfor**

tab = **String**(**9B**)

**print**, "Category",tab,"Mean",tab,"StDev",tab,"Max",tab,"Min"

**for** i=**0**,**N\_Elements**(headings)-**1** **do** **Print**, headings[i],tab,**Mean**(results[i,\*]), $

tab,**StdDev**(results[i,\*]),tab,**Max**(results[i,\*]),tab,**Min**(results[i,\*])

**End**



**Figure B1:** Results of Blob3D measurement of synthetic spheres and cylinders.

**Appendix C: Blob3D shape calculations**

This Appendix briefly describes how 3D shape calculations are conducted in Blob3D software (Ketcham, 2005; Ketcham and Mote, 2019), as they apply to measuring grain shape for apatite (or any mineral grain for which a shape analysis is conducted).

The measurement process is illustrated in the animation 97BS-CR8C.mp4 in the supplemental material, which illustrates the shape calculation on several apatite grains in sample 97BS-CR8. The measurement process consists of generating a 3D shape and measuring the area of its projection (i.e. outline or shadow) over various angles. The procedure first finds the mean projected area by projecting the shape over a uniform distribution of orientations. It then uses the minimum and maximum projected area found in that sampling as starting points to find the true minimum and maximum projected areas, via an optimization algorithm (which looks like “jiggling” the shape in the animation). It then calculates the circularity as the ratio of the maximum projection perimeter to a circle with the same area. The routine then finds the longest caliper dimension (ShapeA), or in other words the longest dimension that would be measured in 3D using a caliper. After finding the projection with the longest caliper dimension, the object is rotated around the long axis to find the longest caliber dimension orthogonal to it (ShapeB). The thirds shape parameter (ShapeC) is the caliper dimension orthogonal to the first two, which is found by rotating the object 90 degrees. Finally, the procedure uses the same procedure but in the opposite order, finding the shortest caliper dimension (BoxC), the shortest dimension orthogonal to it (BoxB), and the caliper dimension orthogonal to those (BoxA).

The ShapeABC parameters correspond to the long-standing traditional shape measurement method for rounded or irregular particles (Sneed and Folk, 1958; Wilson and Huang, 1979), but the BoxABC parameters (Blott and Pye, 2008) are more appropriate for regular shapes. For example, for a perfect cube, ShapeA is the longest corner-to-corner distance, which will be longer than ShapeB and ShapeC, while BoxA, BoxB, and BoxC will all have the same value, the cube edge length. When measuring an apatite grain, BoxC will usually be the “flattest” part of the hexagonal cross section, BoxB will be the orthogonal corner-to-corner distance of the hexagon, and BoxA will be the length in the prismatic direction, unless it is fragmented or has a very low aspect ratio.

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