Response to Reviewers are shown in Blue below

Review 1:

We (Brueseke, Benowitz, Trop) found, the currently under discussion manuscript, *New Developments in Incremental Heating Detrital 40Ar/39Ar Lithic (DARL) Geochronology using Icelandic River Sand by* Odinaka Okwueze, Kevin Konrad, and Tomas Capaldi well written and a good contribution to the continued use of the DARL (Detrital Argon Lithics) geochronology approach. We agree the magmatic history of the glaciated Iceland magmatic province will benefit from applications of the DARL technique, as will other relatively remote and glaciated area such as the Cascades Arc of Northwestern United States.

We graciously recommend some key adjustments to the text, given our and others past work doing both 40Ar/39Ar step-heating and modified single grain fusion on ground mass, whole rock chips, and discrete mineral grains from gravel- and sand-sized volcanic-lithic clasts. We first reported 40Ar/39Ar ages on volcanic-lithic grains from modern river sands in the Wrangell Volcanic arc at a 2014 conference (Benowitz et al., 2014), where we demonstrated that a modified heating schedule of sand-sized volcanic lithics was more efficient and accurate for DARL analyses. This was based on incremental heating single sand-sized volcanic-lithic grains and then modifying our fusion schedule based on these results. We also recommended when applying DARL to other regions standard step-heating be performed before developing a fusion or modified (shortened) step-heat schedule. At the time we were concerned about excess 40Ar not excess 36Ar (which Okwueze et al. document). We agree that excess 36Ar is an underappreciated aspect of 40Ar/39Ar geochronology (Benowitz et al., 2018). These method details were explained in a subsequent Geosphere article (Trop et al., 2022; relevant aspects are copied below) and inasmuch, should be noted as where the DARL technique originated and was first published. Furthermore, Kenny et al. (2022) also performed 40Ar/39Ar incremental step-heats on detrital sand volcanic-lithic grains. We also performed and published (Trop et al., 2022) incremental step-heats on volcaniclithic grains when results were questionable or were of key age spans as one of our goals was to determine the age of initiation for the Wrangell Arc. VanderLeest et al (2020) also applied stepheats to detrital clasts.

Thus, we kindly suggest that Okwueze et al. revise their text and clarify that 40Ar/39Ar step-heats and modified fusions were done previously on modern river volcanic-lithic grains, consequently the contribution here builds on these prior studies. This key fact should be made clearer in this manuscript; as-is, the DARL technique as described is not new or particularly novel, especially given that it is centered on n = 15 grains (vs. n = ~2600 grains; Trop et al., 2022). Additionally, Kenny et al., 2022 performed modified step-heats on 50 grains with step counts varying from 2 to15 (?) steps to optimize number of grains vs. diffusion profile information. See their supplemental files.

We understand there is so much literature out there, that it is easy to miss aspects of past research and take no offense and based on conversations with the corresponding author know none was meant. We are genuinely excited to see more DARL work reported from this research team and others.

We thank Brueseke, Benowitz, and Trop for there constructive reviews and appreciate the effort to coordinate thoughts into a single document. I hope no offense was taken in my lack of knowledge on some of the history of the DARL method. In the revised document we have taken time to highlight the past achievements in more detail.

Specific recommended changes:
Something like the following for their introduction: *Following previous combination 40Ar/39Ar incremental step-heating and informed modified fusion procedure on modern river volcanic lithic grains (Benowitz et al., 2014, Trop et al., 2022), we developed a new DARL partial fusion procedure specific to the magmatic products of Iceland*.
Below are additional changes and information re: relevant past work we recommend the authors consider during their revision.

We thank you for the thorough review and providing a stronger background of this method. The manuscript has been adjusted to properly cite the background of the method and we clarify the new aspects of the technique we included. We expanded the introduction to capture a more comprehensive review of the method. Furthermore, we defined our contribution as being more focused on low-K glassy volcanics.

"The detrital 40Ar/39Ar lithic (DARL) method is a relatively new detrital geochronological tool that determines the 40Ar/39Ar total fusion or incremental heating age determinations on single grains or multi-grain aliquots recovered from sedimentary deposits (e.g. watersheds) (Benowitz et al., 2014, 2018; Vanderleest et al., 2020; Trop et al., 2022; Kenny et al., 2022). The technique was first reported by Benowitz et al. (2014), wherein incremental heating analyses were undertaken on fine-grained volcanic lithics to propose refined total fusion temperature ranges for rapid DARL analyses. The DARL method was employed to determine the history of the Wrangell Volcanic Arc (Alaska, USA) through 2771 analyses of grains, ranging in size from sand to cobble (Trop et al., 2022). The DARL ages matched the expected age range based on available bedrock analyses (Trop et al., 2022; Brueseke et al., 2023). The chemistry and age results from this technique allowed for novel insights into the evolution of the Wrangell Arc (Alaska, USA) that were only partially observed using traditional U-Pb detrital zircon analyses (Trop et al., 2022; Brueseke et al. 2023). Similarly, Vanderleest et al. (2020) performed incremental heating experiments on igneous clasts separated from a conglomeratic formation (n=7), which provided detrital chronologic constraints on the evolution of the Magallanes-Austral basin within the southern Patagonian Andes. More recently, Kenny et al. (2023) employed the DARL method on 50 sand-sized grained collected from the drainage basin of the sub-glacial Hiawatha impact structure in Greenland. Although none of the grains produced traditionally concordant heating spectrum (e.g. >50% of 39Ar released with more than five consecutive steps), two mini-plateau ages matched resetting ages for detrital zircon. The DARL method has potential limitations due to the lower closure temperatures of Ar and greater susceptibility of age disturbances due to alteration as compared to the detrital zircon method. However, in environments that contained mixed mafic and felsic lithologies (e.g. volcanic arcs) or consist primarily of fine-grain extrusive volcanics (e.g. Iceland), the DARL method allows for novel insights not obtainable by the traditional detrital mineral phases. Here we expand upon the method through incremental heating experiments on

single coarse sand or fine gravel grains of volcanic lithic fragments from Icelandic rivers. These sedimentary deposits primarily consist of glassy or fine-grained low-K mafic lava flows and if ages can be reliably constrained with the DARL method, then other low-zircon fertility terrains such as arc and intraplate ocean islands can be constrained. Based on the incremental heating results we propose a methodology for rapid fusion analyses of glass-rich volcanic lithics.”

Around line 15 (Benowitz et al., 2014; VanderLeest et al., 2020; Kenny et al., 2022; Trop et al., 2022 did 40Ar/39Ar incremental step-heats on detrital cobbles and/or sand). Here we present a new methodology for capturing the magmatic history of fine grained extrusive volcanic rocks using single grain detrital 40Ar/39Ar incremental heating geochronology. The DARL (or Detrital Argon Lithics) **method thus far** has consisted of 40Ar/39Ar total fusion analyses, which pose a problem in the case of Iceland, due to the nature of its young glassy lava flows commonly displaying subatmospheric 40Ar/36Ar isochron intercepts and low 40Ar*.

Changed to: “The DARL (or Detrital Argon Lithics) method has consisted of ^{40}Ar/^{39}Ar incremental heating and total fusion analyses, which has not yet been applied to predominantly mafic terrains composed of young glassy lava flows, which commonly display subatmospheric ^{40}Ar/^{36}Ar isochron intercepts and low ^{40}Ar*.”

Around line 25 Benowitz et al., 2014**;** Trop et al., 2022 did both a combination of informed single grain fusions based on incremental heating results; n = ~2600 grains are what was eventually analyzed and reported in Trop et al (2022) For this reason, **we propose combining the aspects of the total fusion and incremental heating** DARL methodologies to acquire age data for the large N values needed for detrital studies while improving the accuracy of total fusion DARL analysis.

Changed to: “For this reason, we build upon a previously proposed method that combines total fusion and incremental heating DARL methodologies to acquire age data for the large N values needed for detrital studies of mafic volcanic terrains.”

Around line 40 (DARL has been applied to sand and pebble grains and cobbles, and as a combination of modified fusion and incremental step-heating…. Benowitz et al., 2014; VanderLeest et al 2020; Kenny et al., 2022; Trop et al., 2022)
The detrital 40Ar/39Ar lithic (DARL) method is a relatively new detrital geochronological tool that **thus far** employed 40Ar/39Ar total fusion analyses on single grains or multi-grain aliquots recovered from cobble sized (>10 cm) volcanic sediments (Trop et al., 2022; Brueseke et al., 2023).

Changed to: “The detrital ^{40}Ar/^{39}Ar lithic (DARL) method is a relatively new detrital geochronological tool that determines the ^{40}Ar/^{39}Ar total fusion or incremental heating age determinations on single grains or multi-grain aliquots recovered from sedimentary deposits (e.g. watersheds) (Benowitz et al., 2014, 2018; Vanderleest et al., 2020; Trop et al., 2022; Kenny et al., 2022).”

Around line 50 (this has already been done…Benowitz et al., 2014; Kenny et al., 2022; Trop et al., 2022**)** Here **we expand** upon the method through incremental heating experiments on single coarse sand grains of volcanic lithic fragments from Icelandic rivers.

This section of the paragraph was changed considerably to reflect this, see first comment response above.

Line 220 (This seems a little overstated given ~10 years of DARL step-heating work and the orders of magnitude larger number of individual DARL analyses from Trop et al., 2022 and the combination of geochemistry and DARL dating in VanderLeest et al 2020 and Brueseke et al., 2023).

Provided the level of difficulty, the incremental heating DARL experiments worked well and **represent an advancement in the field of detrital geochronology.**

Sentence removed for simplicity.

Around line 250 (this was sort of done -Trop et al., 2022- to evaluate alteration and excess 40Ar and for sure the DARL method has been applied to dominantly mafic bedrock sources.) Although the internal concordance test afforded by the incremental heating method has many advantages, the long analyses time hinders the method's use for detrital geochronology studies, which rely on high= N values. Therefore, **we propose that a subset of grains from a sampling site be analyzed with the incremental heating method** in order to define the best partial fusion temperature ranges and appropriate assumed 40Ar/36Ar0. More work is required to assess the validity of the method in different geologic settings, but the primary data from this study indicates the method is valid and **allows for detrital geochronology studies of dominantly mafic bedrock sources.**

Changed to: "A single incremental heating experiment using a vacuum furnace takes ~12 hours to complete. Therefore, a rapid analyses method is required to obtain the large N values needed for a successful detrital geochronology study. Trop et al. (2022) used incremental heating on a subset of grains to assess for alteration or excess argon. Thereafter, they employed the total fusion method wherein individual grains or multi-grain aliquots were fused in a single step (Trop et al., 2022). An atmospheric $^{40}Ar/^{36}Ar_0$ was assumed with the age calculations and the results were ~equivalent to K/Ar ages collected from the region."

Around Line 260 (at the time we used 295.5 for atmospheric 40Ar/36Ar0)…which now is not standard…but does make the reference to our work a bit confusing…perhaps remove?). An atmospheric 40Ar/36Ar0 was assumed with the age calculations and the results were ~equivalent to K/Ar ages collected from the region.

That shouldn't make a significant difference as the atmospheric to subatmospheric shift will be the same degree but show lower values since your mass discrimination factors on air standards were normalized to 295.5 instead of 298.6. E.g. this Iceland sample set would have a $40Ar/36Ar_0$ mean around 292 if I used 295.5 for my MDF corrections.

Around line 295
The DARL method provides a novel means of constraining the volcanic history of a region through detrital geochronology of lithic grain sand samples.
Please Add the reference to Trop et al. (2022), given that is where the DARL technique originated and was first published:
The DARL method (***Trop et al., 2022***) provides a novel means of constraining the volcanic history of a region through detrital geochronology of lithic grain sand samples.

Changed to: "The DARL method (Trop et al., 2022) provides a novel means of constraining the volcanic history of a region through detrital geochronology of lithic grain sand samples."

Other manuscript notes that need to be addressed:
Please define what you mean by discordant: We think we know what you are referring, but it is never defined/explained how you are applying this broad term.

Added to the methods (~line 122): "We define a successful age plateau as containing five or more consecutive heating steps that incorporate over 50% of $^{39}Ar_K$ and have a probability of fit factor >5%. If a heating step is not within uncertainty of the plateau than we refer to that as a discordant step."

Table 1: Please add the known age range for magmatism for each sample/drainage.

Added. Caption updated: **Table 1:** "The location and general geomorphology of each sampling site location. Age ranges are approximated from available outcrop $^{40}Ar/^{39}Ar$ and K/Ar age determinations collated in Jóhannesson and Sæmundsson (2009). "

How often did you measure mass discrimination? Did it drift? Could applying the "incorrect" mass discrimination explain your excess 36Ar (and excess 40Ar) measurements? 36Ar was measured on a more sensitive electron multiplier? Where 40Ar was measured on a sensitive (but less so?) faraday? Is this a factor in the excess 36Ar measurements?

We doubt these are controlling factors on the excess 36Ar measurements…but these factors should be at least documented and mentioned-dismissed in the text/methods.

Some of these factors were mentioned in the methods and the raw data is provided in the new supplements. We have adjusted the methods to expand on these calibration procedures. "Five air standards (for mass discrimination factors; MDF) and collector calibrations (for faraday-ion multiplier calibration) were run prior to every experiment. The MDF (assuming a 40Ar/36Aratmo = 298.56 ± 0.31; Lee et al. 2006) and calibration factors for an individual experiment were determined by fitting a polynomial curve to the results over two weeks and interpolating the values for when the experiment was run. Collector calibrations are done by putting 36Ar (from air) on the multiplier then on the faraday by adjusting the magnet. This is repeated 75 times per analyses to determine the multiplier/faraday offset. Five collector calibrations were run per day (immediately after the MDF analyses before pumping the air out of the mass spec). Neither the collector calibrations nor MDF results varied significantly over the course of the project”

It also important to note that other samples (not related to this project) run during the same time consistently produced atmospheric intercepts so I believe the phenomenon is related to the geology of the samples as opposed to analytical factors.

Perhaps more discussion on how modern mass spectrometer instrumentation allows for the clearer identification of excess 36Ar could be added?

Although this could be valuable I do not see a clear location to include this discussion in the manuscript.

Around Line 275

Therefore, we can calculate the partial-fusion age between those temperature steps, using an 40Ar/36Ar0 that is representative of our dataset (296 ± 4; Figure 8).

What was the range of determined 40Ar/36Ar0 for all the grains analyzed?
289.7 to 300.3….Is it really sensible to assume a single subatmospheric 40Ar/36Ar0 for all samples?

The justification for using a value of 296 +/- 4 is that the larger uncertainty covers the range from subatmospheric to slightly supra atmospheric. The nature of the TF DARL method comes with inherent uncertainties (not unlike detrital zircon) but the high precision at which the atmospheric value is known (+/- 0.31) is clearly too precise to cover the natural range observed in these young volcanic products. However, if we employ the total range (287.1 – 304.3 or 295.7 +- 8.6) then each age uncertainty will become unusable.

Given most results approximated or were greater than 298.56 ± 0.62 (Lee et al., 2006)? The 296 ± 4: Is that a weighted average? The uncertainty is propagated during the age calculations?

Correct, the 296 is from the weighted average. The +- 4 uncertainty was propagated during the ‘partial fusion’ calculations, hence the larger uncertainties throughout. Those values are now provided in Table 3.

Isochron plots:

Are the same steps used for the plateau age determinations used for the isochron age determinations? They should be. It seems for some of the samples this is not the case? It is hard to tell given the number of steps used in the isochron determinations are not listed in table 2. If always the same number of steps/same steps are used for isochron regressions as were used for the plateau age determinations (as they should be? Unless justified), please mention in text.

In all scenarios the isochron points and plateau points are the same heating steps. This was addressed in the methods “When a sample contained a concordant isochron with a non-atmospheric $^{40}Ar/^{36}Ar_0$ intercept (following the same statistical criteria as the described for the plateau), the plateau was recalculated using the intercept and uncertainty (e.g. Heaton and

Koppers, 2019). When a plateau was recalculated, no additional heating steps were added — even if they became concordant due to the increased intercept uncertainty."

We added the following line to the table 2 caption for clarity:
"n=heating steps used in age calculations for both plateau and isochron"

RPJSO1-e

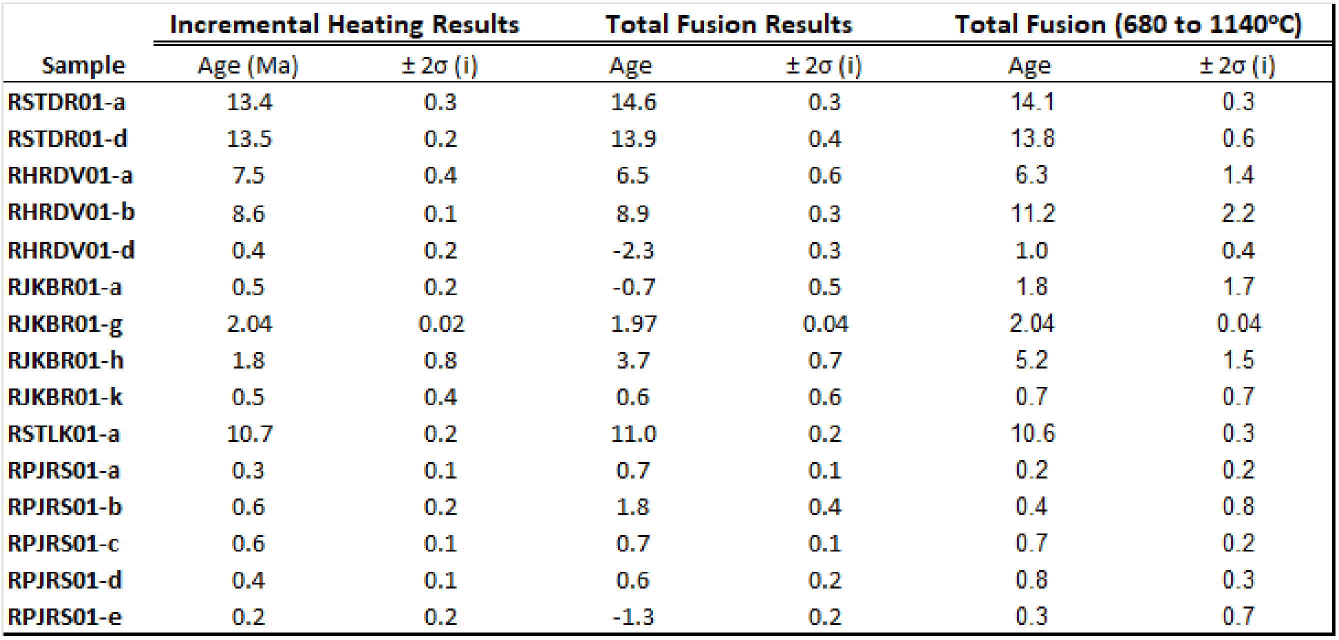
Would you consider this stepping up age spectrum indicative of loss? If so, is it appropriate to perform a regression back to initial 40Ar/36Ar (Isochron plot) given the documented loss?

I would consider this result indicative of partial degassing (which is common in terrains with overlapping lava flows). However, the concordant plateau at higher temperature (and corresponding isochron) is perfectly reasonable within standard 40Ar/36Ar practice. The atmospheric regression at mid-high temperature is further evidence this sample formed in equilibrium with atmosphere. There is no reason to remove this isochron.

Figure 10

This is a key figure…but we don't see the negative original age determinations in Table 2 and there are no supplemental isotopic files. Please add the negative (original) age determinations to Table 2 and add full supplemental files. Schaen et al. (2021) community based (dozens of noble gas lab authors) makes a strong case and sets out examples of how 40Ar/39Ar isotopic information should be documented in scientific manuscripts. Regardless if Schaen et al. (2021) is followed to the "T", detailed isotopic tables are required to be included with 40Ar/39Ar geochronology publications to be able to evaluate the authors results/interpretations/methods.

We acknowledge that the omission of the original full of set of isotopic information was a mistake. We have appended a supplemental document to the manuscript that includes full information suites for all analyses as individual tabs in an excel file. We also added Table 3 to show the age information used in Figure 10.



	Incremental Heating Results		Total Fusion Results		Total Fusion (680 to 1140°C)	
Sample	Age (Ma)	± 2σ (i)	Age	± 2σ (i)	Age	± 2σ (i)
RSTDR01-a	13.4	0.3	14.6	0.3	14.1	0.3
RSTDR01-d	13.5	0.2	13.9	0.4	13.8	0.6
RHRDV01-a	7.5	0.4	6.5	0.6	6.3	1.4
RHRDV01-b	8.6	0.1	8.9	0.3	11.2	2.2
RHRDV01-d	0.4	0.2	-2.3	0.3	1.0	0.4
RJKBR01-a	0.5	0.2	-0.7	0.5	1.8	1.7
RJKBR01-g	2.04	0.02	1.97	0.04	2.04	0.04
RJKBR01-h	1.8	0.8	3.7	0.7	5.2	1.5
RJKBR01-k	0.5	0.4	0.6	0.6	0.7	0.7
RSTLK01-a	10.7	0.2	11.0	0.2	10.6	0.3
RPJRS01-a	0.3	0.1	0.7	0.1	0.2	0.2
RPJRS01-b	0.6	0.2	1.8	0.4	0.4	0.8
RPJRS01-c	0.6	0.1	0.7	0.1	0.7	0.2
RPJRS01-d	0.4	0.1	0.6	0.2	0.8	0.3
RPJRS01-e	0.2	0.2	-1.3	0.2	0.3	0.7

Table 3: A comparison of concordant plateau age, total fusion ages assuming all gas released and an atmospheric $^{40}Ar/^{36}Ar_0$ value and total fusion results for steps between 680° and 1140°C assuming a $^{40}Ar/^{36}Ar_0$ of 296 ± 4. Steps with a discordant heating spectrum are excluded from the table.

We see on table 2 you correct for excess 36Ar, but don't correct for excess 40Ar. Would it be better to use the original isochron age determinations for all analysis instead of plateau ages?

The corresponding ages when we correct for a non-atmospheric $^{40}Ar/^{36}Ar_0$ and if we use the isochron ages are the same with slightly lower errors for the plateau (as a function of the weighted mean calculation). For simplicity for the non-argon geochronologist reader we prefer to just recalculate the plateau age in order to reduce the likelihood of someone using the wrong age.

Figure 10….B looks stretched? i.e., Why are uncertainties so big?
Or are uncertainties blown up with the applied 296 ± 4 40Ar/36Ar0, hence MSWD goes down simply because of the larger uncertainties? Compared to 298.56 ± 0.62 40Ar/36Ar0 (Lee et al., 2006).

Correct, the samples are recalculated with a larger $^{40}Ar/^{36}Ar_0$ intercept uncertainty, which results in large age uncertainties. One advantage with this method is that the 'geologic' uncertainty on the total fusion age is much larger compared to the analytical uncertainty. Using this larger intercept values makes the single age determinations uncertainties more realistic given the nature of the method.

What would be the MSWD for graph A be if the youngest three ages were parsed? Seems those are biasing everything and for graph B all the ages are being modified (some far away from there "actual ages"!!!).

The modification from the actual ages is a key feature of this method. Although some of the samples will have their absolute ages become less accurate, the larger uncertainties account for it and allow for the other ages to fall in line. The goal is to generate a more accurate dataset at the cost of precision. For panel A, the MSWD stays high if the youngest ages are removed due to other the two large age offsets.

Can you please add a table of original ages/uncertainties for all samples vs. modified ages/uncertainty with the assumed 296 ± 4 40Ar/36Ar0 determination. We think this is a key aspect…Yes you are shifting the youngest ages, but you are also shifting the other ages, Is that appropriate given the large variations in actual measured/calculated 40Ar/36Ar0?

Table 3 added. See above.

For example, on figure 10….sample RHDRV01-b gets shifted from a total fusion age of 8.9 Ma (± 0.03) with a small uncertainty and becomes >11.0 Ma on 10B with a huge uncertainty (± ~5 Ma?).

Is this an improvement over the original accurate and precise age determinations?

The age shifts from 8.9 ± 0.3 Ma to 11.2 ± 2.2 Ma. Although the total fusion age and plateau age are within uncertainty of each other there would be no way to know this if only a TF age was calculated. Therefore, this method shirks precision for the likelihood of increased accuracy (through more conservative uncertainties). This will always be a trade off with the DARL method.

Can you get negative ages simply due to statistics? i.e. An age result of 10 ka ± 20 ka on a lava means given enough analyses you would get a negative age from the same sample.
We are not sure if trying to make “exact” geologic interpretations from modified negative 40Ar/39Ar follows best practices. Yes these grains are young and the authors can robustly state that, but we are not sure applying a 296 ± 4 40Ar/36Ar0 to a negative age with a measured 289.67 40Ar/36Ar0 makes for a geologically more meaningful age.

I see what you are saying about the negative ages due to statistics but would counter that the negative ages in our case have statistically concordant isochrons. As for whether a mean of the measured intercept values should be used or the full range will be a difficult question for future researchers to decide.

Rough figure showing large shift from measured to modeled ages.
Is 1.79 Ma age on Figure 10?
(RJKBR01-h)? might be….
forgive us if it is.

Yes, it the large offset right above the young sub-atmospheric cluster on the figure.

Line 80 (>2 mm sized grains are granule sized gravel as opposed to sand sized grains, so the text should state that fine gravel (or granules) and sand was analyzed**).**

The bulk sediment samples were sieved and grains from the **2-3 mm size fraction** were selected for all sites except RJKBR01, where the 1-2 mm size fraction was used. Each selected grain was separated and given a unique identifier (i.e. -A; Figure 2).

Thanks for the clarity. Sands changed to sands/fine gravel throughout.

Data Availability
Please include a link to all isotopic information (preferably in excel format) and supplemental figures using a file-sharing site like https://zenodo.org/records/802100. As is, it is impossible to replot the presented data, evaluate the results, etc.

All data made available through the supplement now.

Summary Suggestion:
Perhaps a better DARL method for Iceland would be to: Degass/not measure/pump out lower temperature steps (below 680 °C). And then a apply a 296 ± 4 40Ar/36Ar0 for the negative age determinations: but acknowledge these modeled age determinations are approximations and not indicative of exact geological eruptive events.

This is a good suggestion and the following line was added to the discussion: "Alternatively, since the sensitivity to the sub atmospheric intercepts seems greater in the youngest samples, perhaps the alternate 40Ar/36Ar0 (296 ± 4) should only be used when a sample produces a negative age result."

Review References:

Benowitz, J.A., Davis, K.N., Brueseke, M.E., Trop, J.M., and Layer, P., 2014, Investigating the lost arc: Geological constraints on ~25 Million years of magmatism along an arc-transform junction, Wrangell Volcanic Belt, Alaska, Geological Society of America Abstracts with Programs, Vol. 46, No.6, p.363.
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Rivera, T.A., Jourdan, F., Matchan, E.L. and Hemming, S.R., 2021. Interpreting and reporting 40Ar/39Ar geochronologic data. *GSA Bulletin*, *133*(3-4), pp.461-487
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Arc: Spatial-temporal evolution of slab-edge magmatism along a flat-slab, subduction-transform transition, Alaska-Yukon. *Geosphere*, *18*(1), pp.19-48.

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Oligocene–early Miocene mafic volcanic arc in the southern Patagonian Andes (~ 51° S) from single-clast geochronology and trace element geochemistry. *Journal of Geodynamics*, *138*, p.101751.