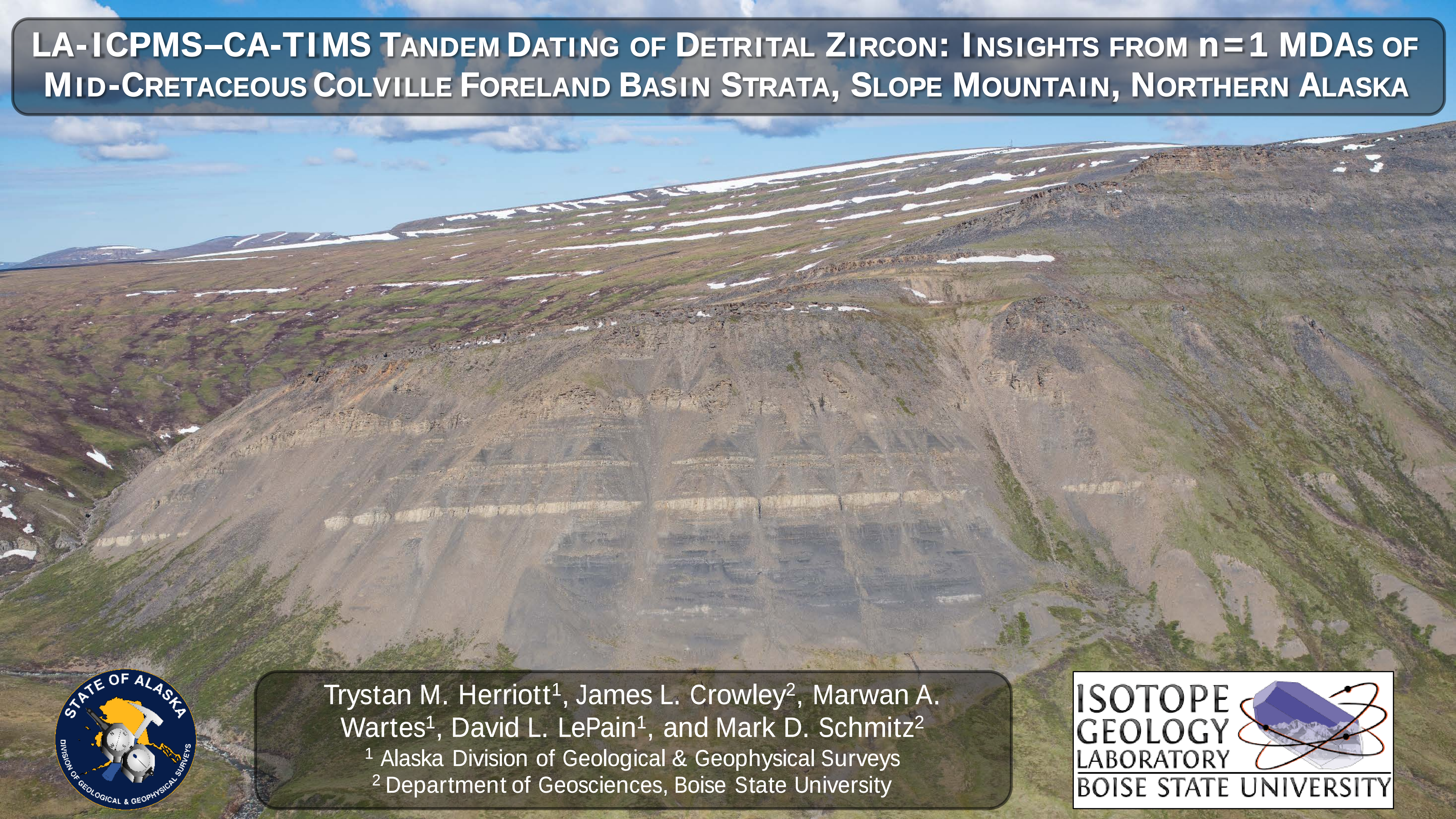
LA-ICPMS–CA-TIMS Tandem Dating of Detrital Zircon: Insights from n=1 MDAs of Mid-Cretaceous Colville Foreland Basin Strata, Slope Mountain, Northern Alaska



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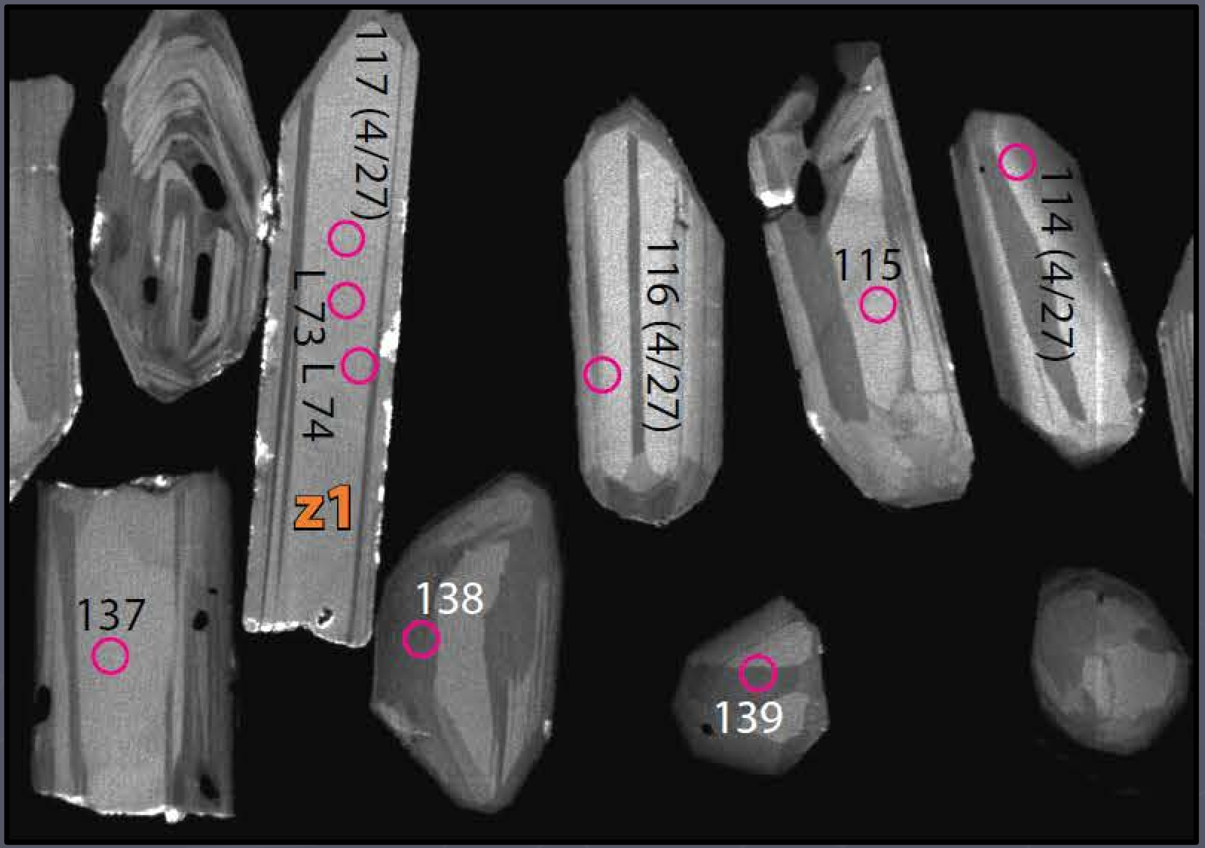
Abstract

Recent advances in detrital zircon (DZ) maximum depositional age (MDA) research are notable, yet the persistent array of laser ablation-inductively coupled plasma mass spectrometry (LA-ICPMS)-based DZ MDA algorithms portends opportunity for further progress. Sound geologic reasoning must underpin MDAs, but interpreting youthful date distributions requires many analytically and statistically driven decisions, including whether to use single- or multi-grain MDAs. Furthermore, the reliability of MDA algorithms is usually assessed by chronostratigraphic benchmarking that does not ascertain MDA accuracy, and even careful consideration of known challenges can still lead to inaccurate MDAs. Here, we present tandem LA-ICPMS–chemical abrasion-thermal ionization mass spectrometry (CA-TIMS) U–Pb results for five DZ samples from a ~950-m-thick section of mid-Cretaceous Torok and Nanushuk Formations at Slope Mountain. Youthful DZ yields are extremely sparse and only permit n=1 MDAs. LA-ICPMS dates are 0.3–6.4% younger than CA-TIMS ages from the same grains. The biostratigraphy suggests ~110–94 Ma sedimentation; the CA-TIMS-based MDAs reduce this window by ~8.5 Ma. These MDAs and a new CA-TIMS tephra zircon age correlated to the section's top render reasonable minimum sedimentation rates (100s m/Ma). However, using the youngest single-grain LA-ICPMS dates as MDAs yields an improbably rapid rate (~5 km/Ma) that affirms their inaccuracy. We examine the new results and two published tandem DZ datasets to assess whether analytical dispersion and Pb-loss are formidable or discountable. These tandem dates indicate pitfalls for LA-ICPMS MDAs, with too-young offsets per study that are impactful (~2–3% avg.) and pervasive (~85–100% of pairs). Tandem date-pair plots reveal relations that DZ MDA algorithm comparisons can obscure, and too-young biases in LA-ICPMS dates likely reflect complex combinations of analytical scatter, matrix effects, and low-temperature Pb-loss. The youngest date in an LA-ICPMS distribution maximizes any too-young bias regardless of the contributing sources, but multi-grain MDAs are also prone to negative offset. We demonstrate the value of tandem LA-ICPMS–CA-TIMS geochronology for establishing DZ MDAs—and assessing the validity of MDA algorithms—in a demanding, n=1 application.

*Authors' note: These slides were presented at the Geological Society of America (GSA) Connects annual meeting in Denver, Colorado, on 10 October 2022, during a session honoring the life, career, and mentorship of Charles "Gil" Mull. The original abstract is available here: https://doi.org/10.1130/abs/2022AM-381642. Formal publication of this work is in preparation.

DZ Maximum Depositional Ages

- <u>Law of detrital zircon</u> (Gehrels, 2014): A sedimentary rock cannot be older than its youngest zircon (Houston and Murphy, 1965)
- <u>LA-ICPMS</u> is most common method in DZ studies
  - Uncertainties present notable challenges
- <u>CA-TIMS</u> is becoming more common in DZ studies (e.g., see GSA Connects 2022 Program)
- <u>Tandem dating</u>: LA-ICPMS with follow-up CA-TIMS
- DZ MDA foundation: Dickinson and Gehrels, 2009
- Recent DZ MDA insights: e.g., Coutts et al., 2019; Herriott et al., 2019; Johnstone et al., 2019; Copeland, 2020; Gehrels et al., 2020; Sharman and Malkowski, 2020; Rasmussen et al., 2021; Vermeesch, 2021



Progress, but the LA-ICPMS MDA algorithm alphabet soup persists:

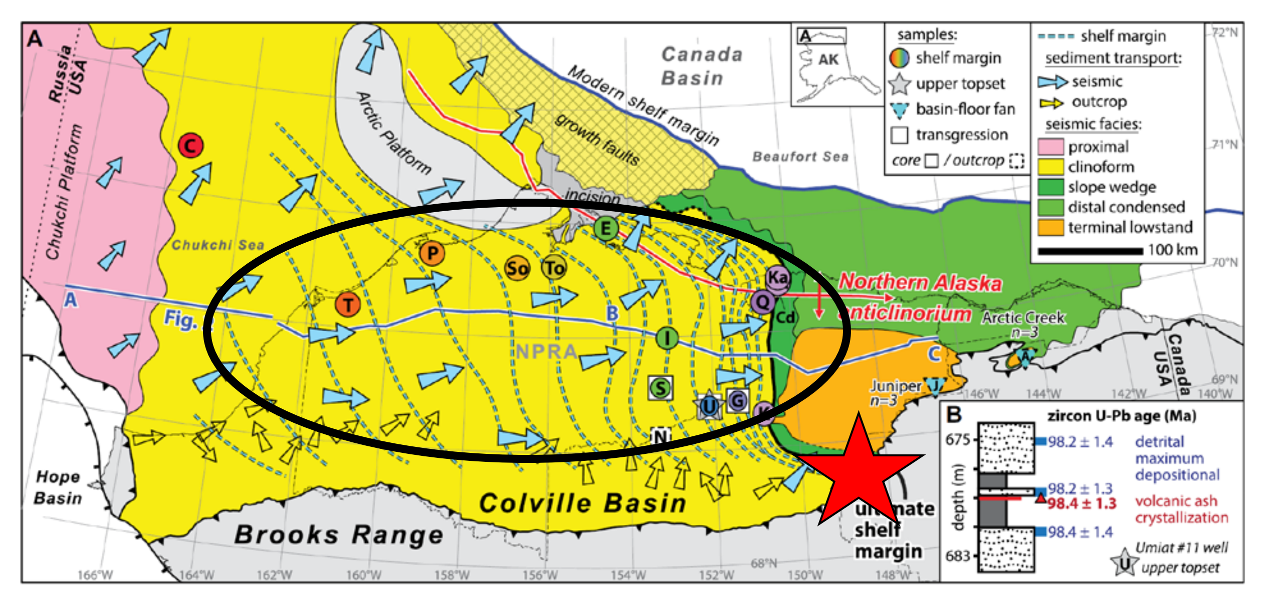
YSG, YDZ, YSMGA, YC1σ, YC2σ, YSP, MLA, YPP, YMKDE, T, Unmix, TuffZirc, Y3Zo, etc.

- Geologically, analytically, and statistically driven decisions: Single-grain or multi-grain MDAs? (Choose carefully)
- Do analytical dispersion, Pb-loss, and matrix effects matter? (Yes; cf., Copeland, 2020, and Vermeesch, 2021)
- Is YSG one of "the most successful and accurate" (Coutts et al., 2019) algorithms or the "the way to go" (Copeland, 2019) or "best estimate" (Copeland, 2020) of MDA? (No)

Even careful consideration of known geologic and geochronologic challenges can still render results that are potentially inaccurate (e.g., relying on single, low-probability-tail dates that are younger than crystallization age) or are ostensibly indefensible (e.g., pooling together DZ dates of unknown geologic relatedness and/or that bear systematic and/or geologic biases)

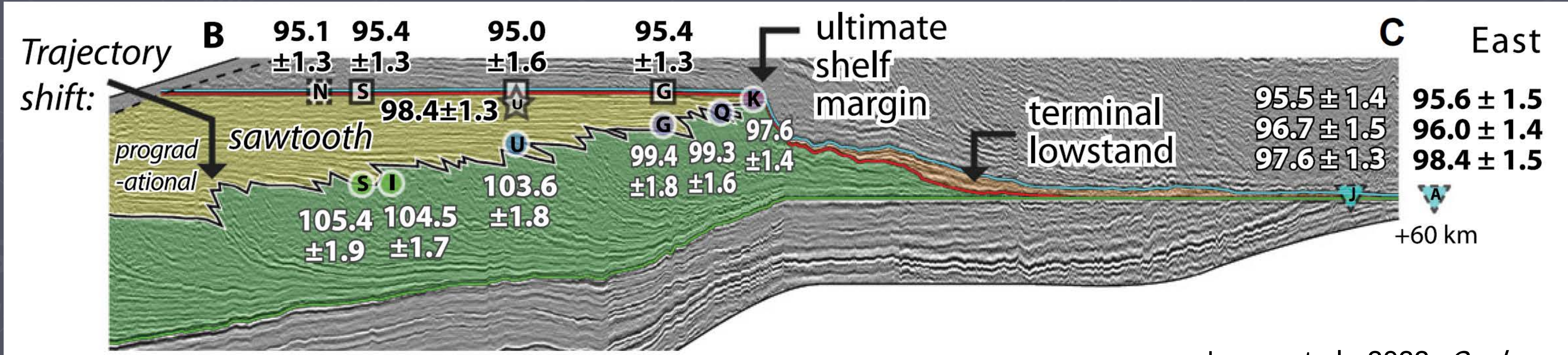
Colville Foreland Basin

- Brookian orogeny: Middle Jurassic–Early Cretaceous emplacement of thick stack of allochthons, loading the crust and driving foreland basin subsidence
- Nanushuk–Torok: Major Early–mid-Cretaceous filling cycle, forming massive, long-lived clinothem
- Lease et al. (2022): ~115–95 Ma LA-IPCMS DZ MDAs
- Time-transgressive progradational couplet
- When did Nanushuk arrive at Slope Mountain? Nanushuk perhaps earliest middle Albian–Cenomanian (~110–94 Ma)



Lease et al., 2022, *Geology*

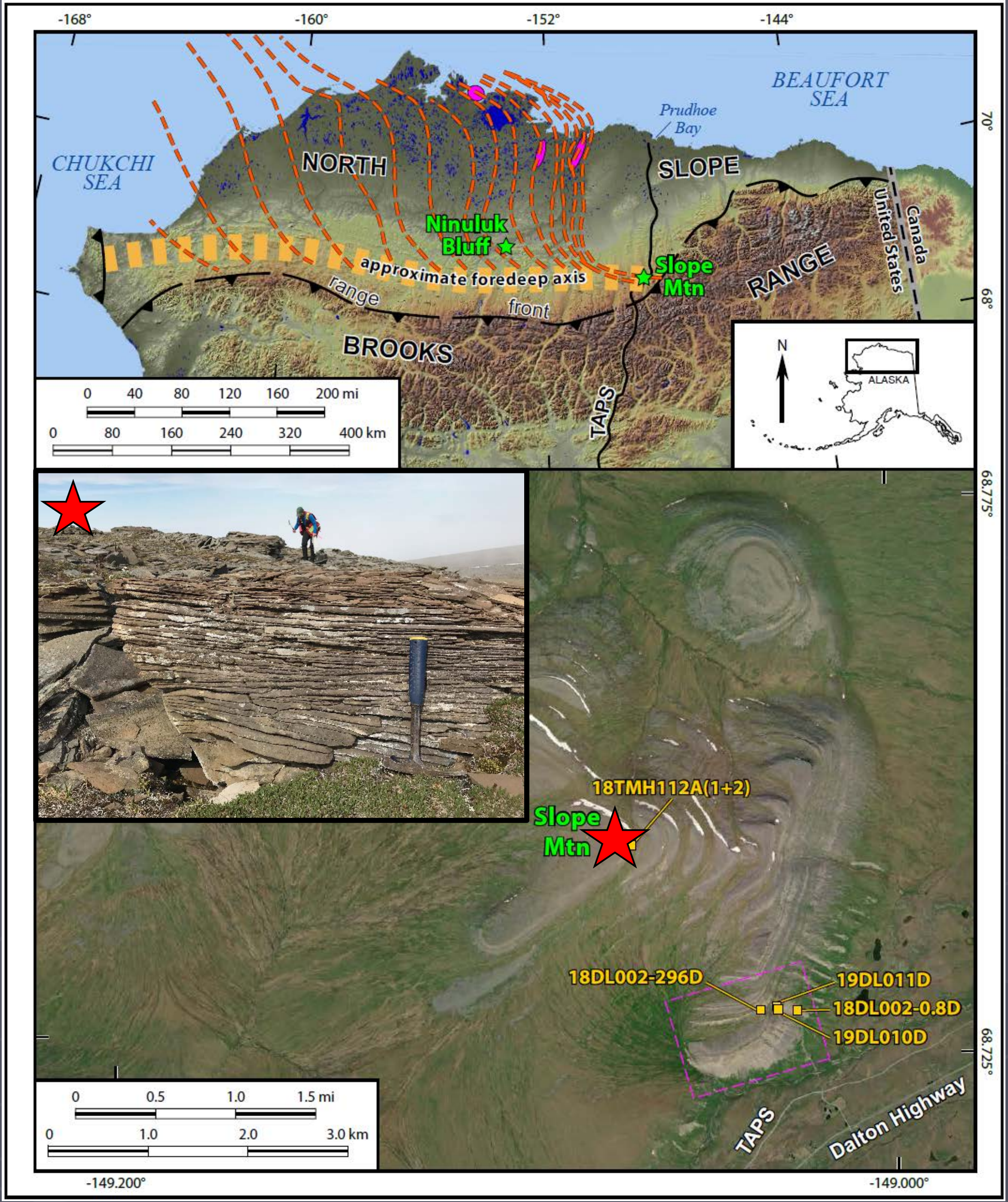
- Probably ~synchronous terminal transgressive cessation of Nanushuk–Torok at ~95 Ma



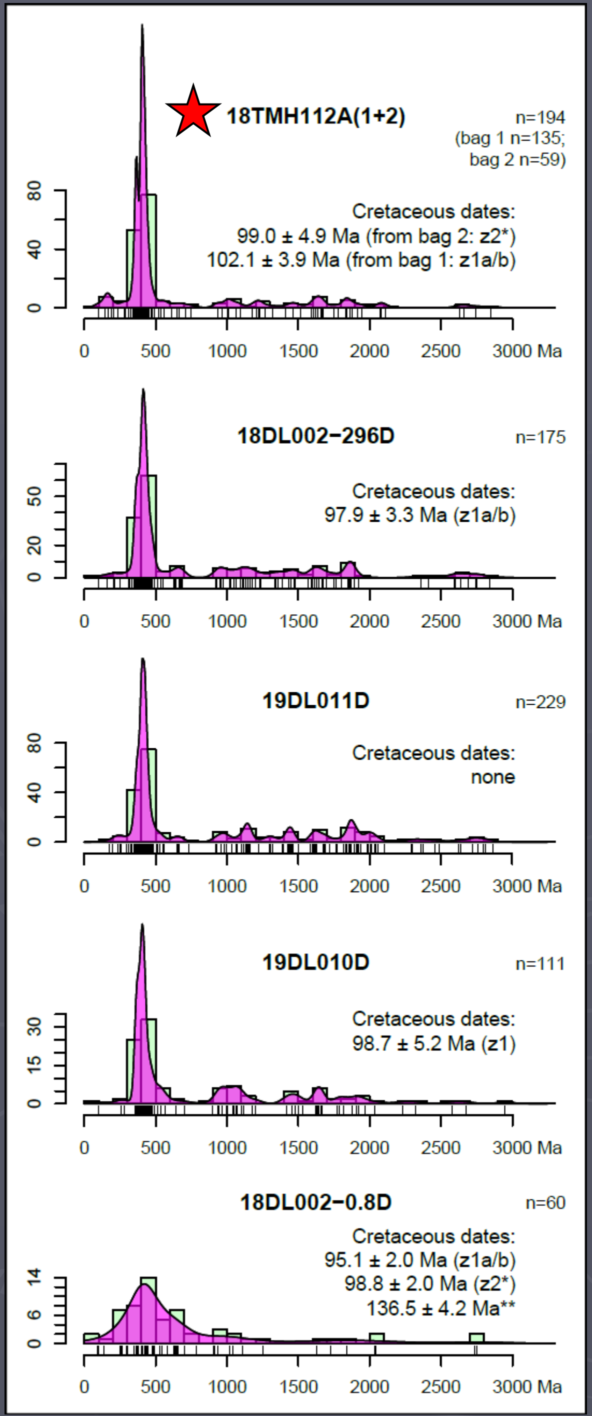
Lease et al., 2022, *Geology*

Slope Mountain Stratigraphy

- Torok–Nanushuk contact crops out at Slope Mountain
- ~935 m of Nanushuk: marine–non-marine–marine (Huffman et al., 1981)
- Confirmed that marine Nanushuk strata do occur at top of Slope Mountain
- Overlying Seabee does not crop out at Slope Mountain, but reasonable to presume it is stratigraphically nearby



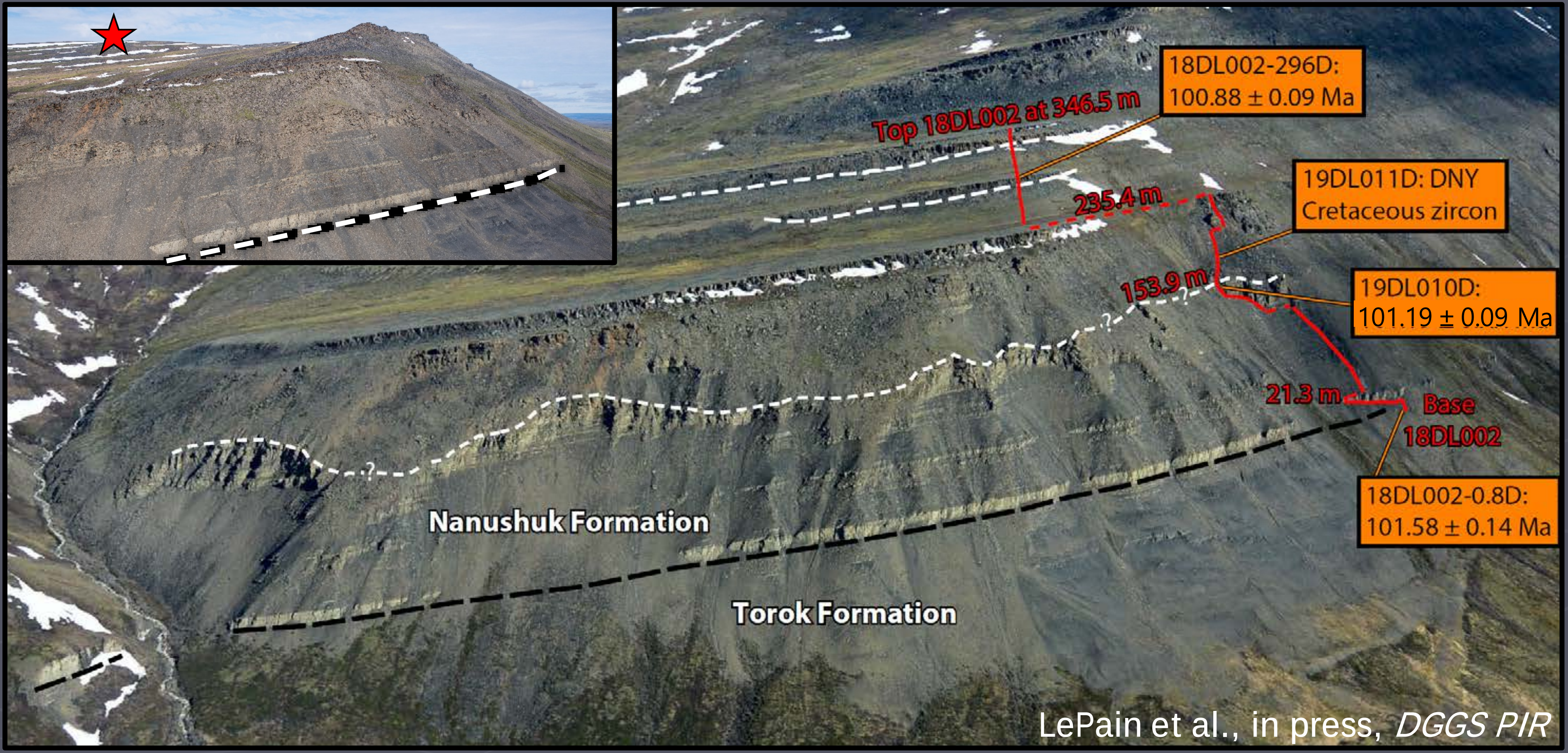
Slope Mountain DZ Results



- Abundant Devonian and older zircon: Transverse sediment routing
- Youthful zircon yields are extremely sparse (n=0 or 1 or 2)
- All LA-ICPMS dates are too young
  - Range: –0.3% to –6.4%
  - Average: –3.0%

- Top Torok LA-ICPMS YSG: 95.1 ± 2.1 Ma

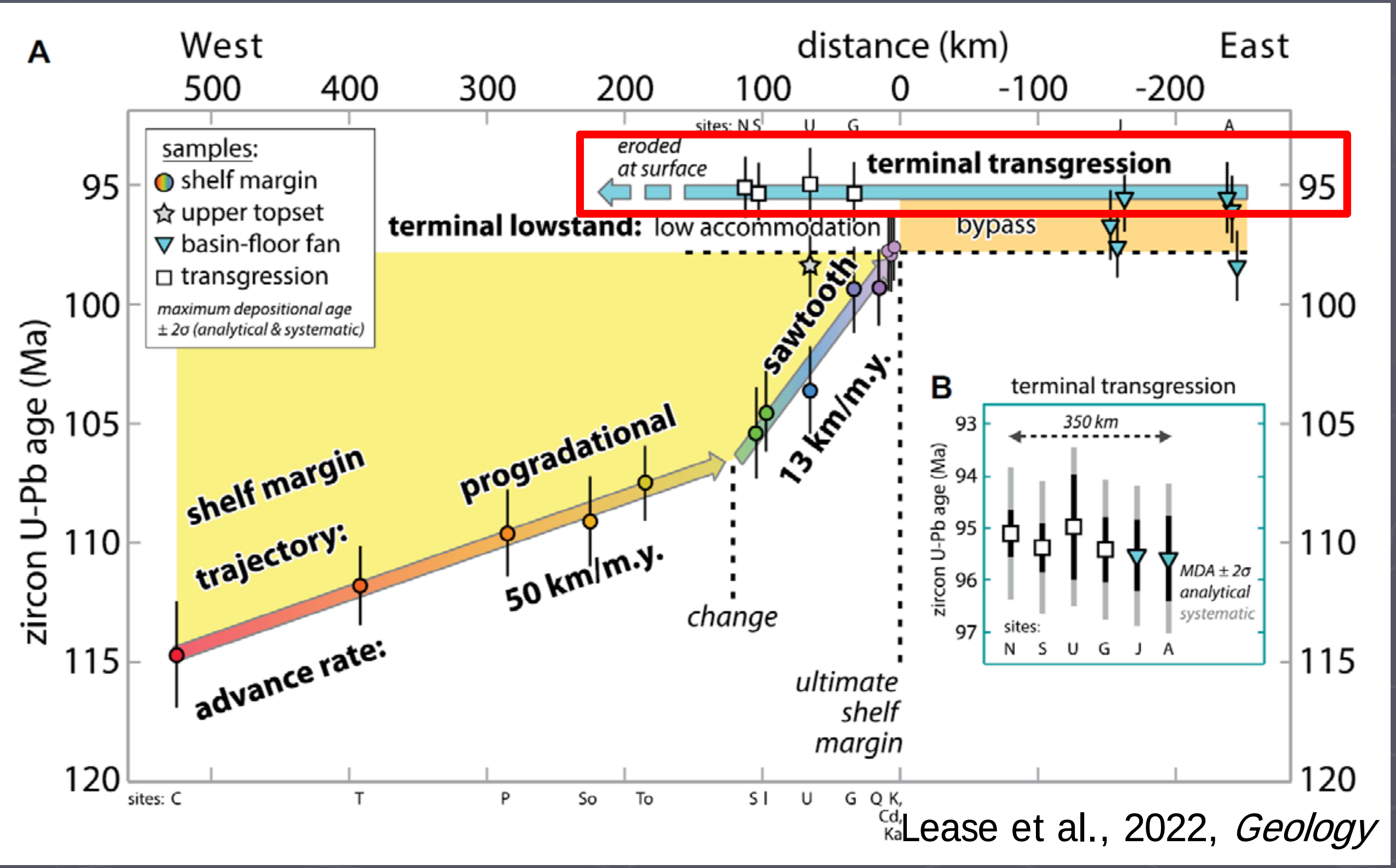
  CA-TIMS MDA: 101.58 ± 0.14 Ma
- Nanushuk at Slope Mountain is not older than latest Albian (~8.5 Myrs younger)
- ★ Top Nanushuk CA-TIMS MDA: 102.41 ± 0.06 Ma



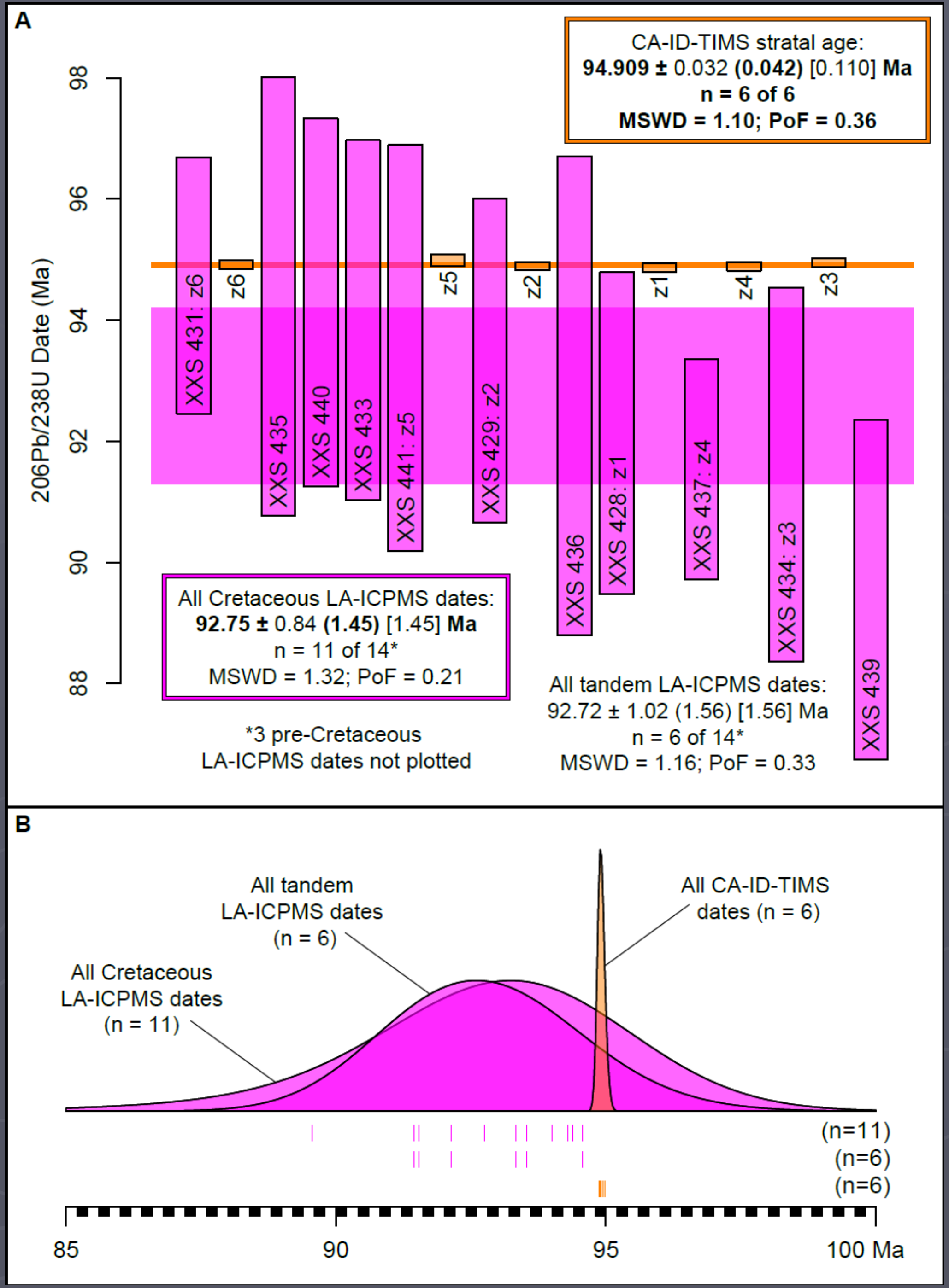
LePain et al., in press, *DGGS PIR*

Ninuluk Bluff Stratigraphy and TZ Results

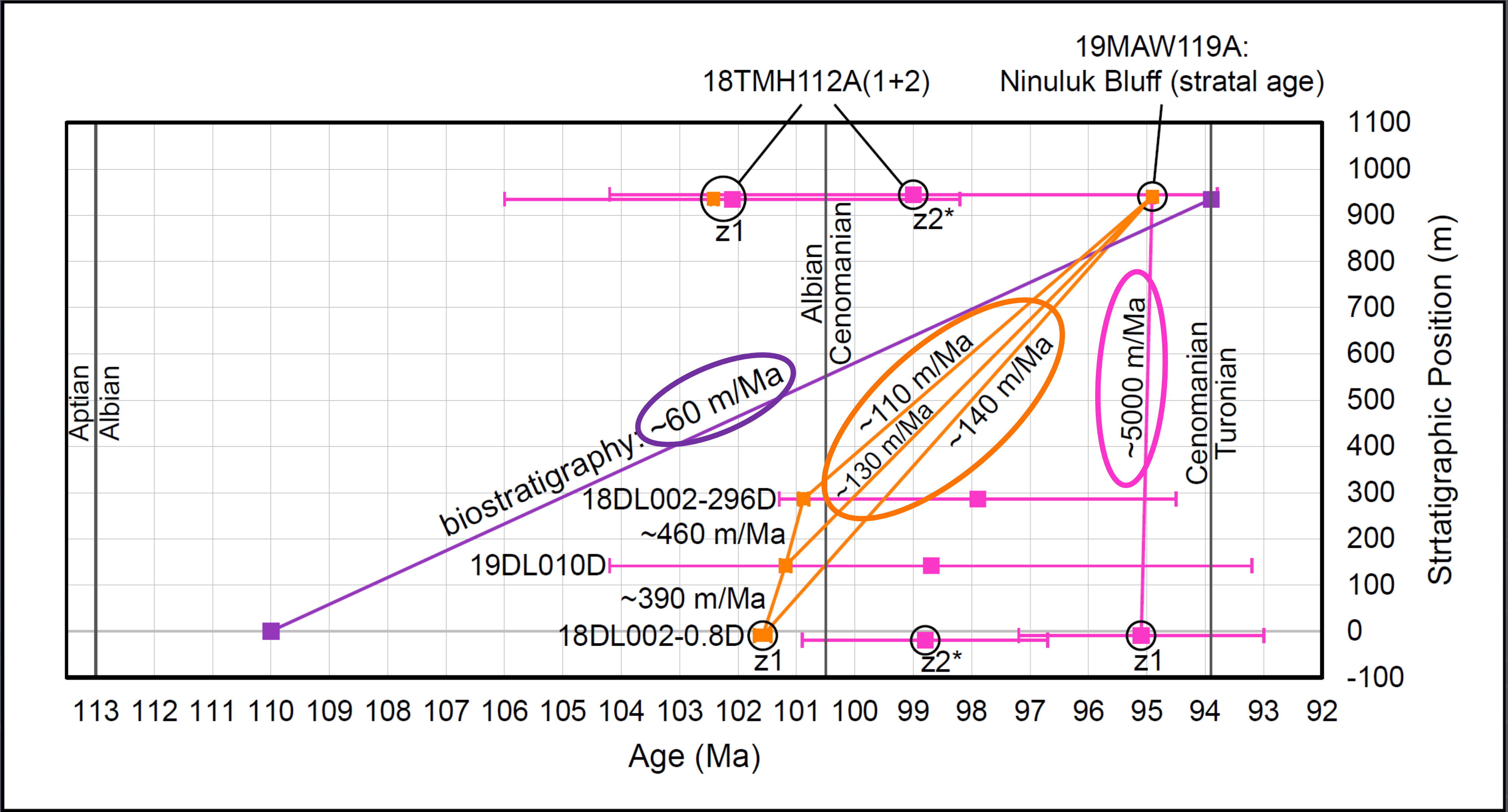
- Type locality for Ninuluk sandstone (late-stage marine Nanushuk)
- Nanushuk–Seabee contact crops out
- Tephra zircon (TZ) sampled from 4.2 m above top Nanushuk
- Minimum age for top Nanushuk at Slope Mountain:
  94.909 ± 0.042 Ma
- All LA-ICPMS tandem dates are too young: Avg. offset = –2.3%

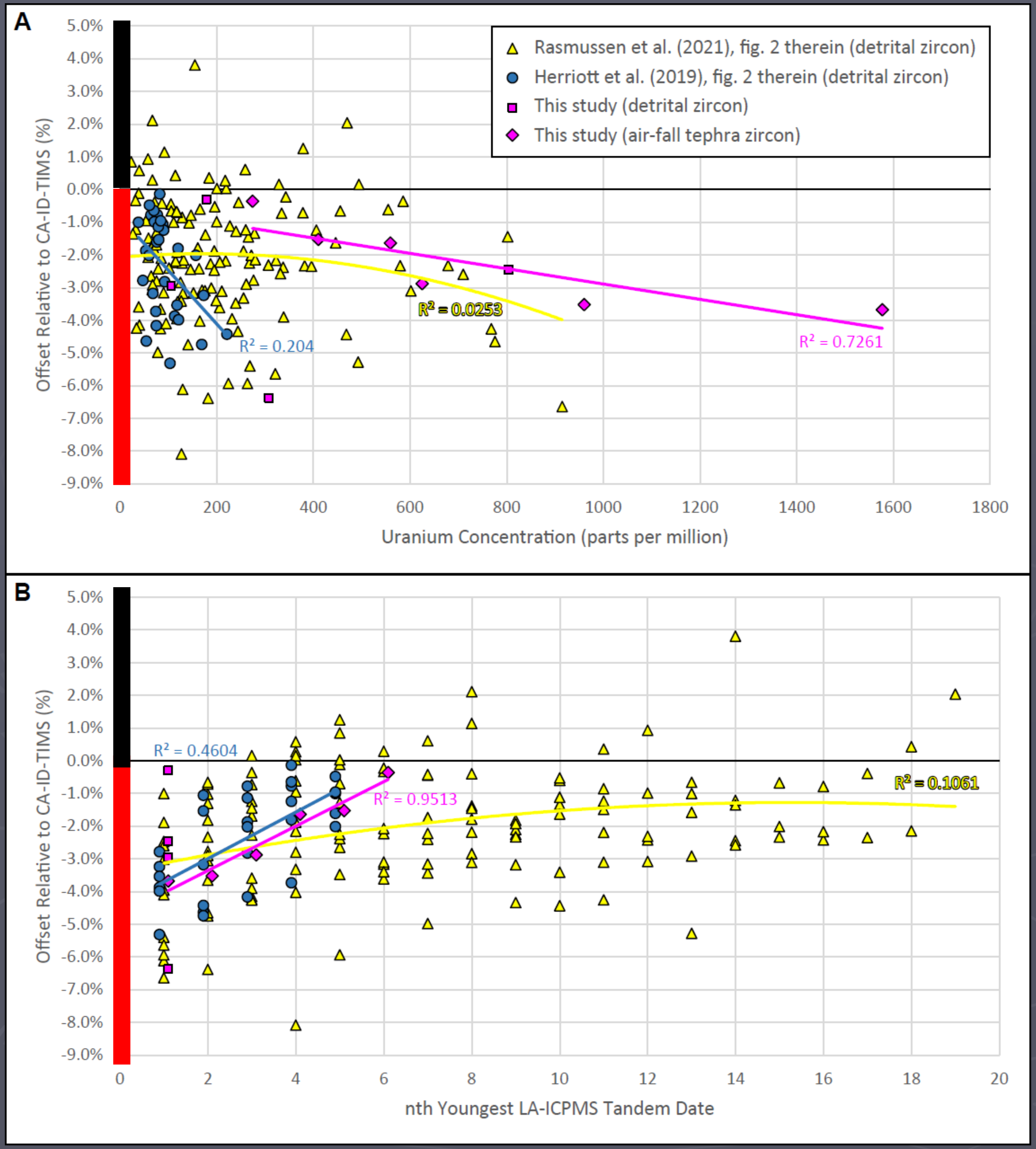


Lease et al., 2022, *Geology*



Depth–Age Plot: Slope Mountain DZ and Ninuluk Bluff TZ



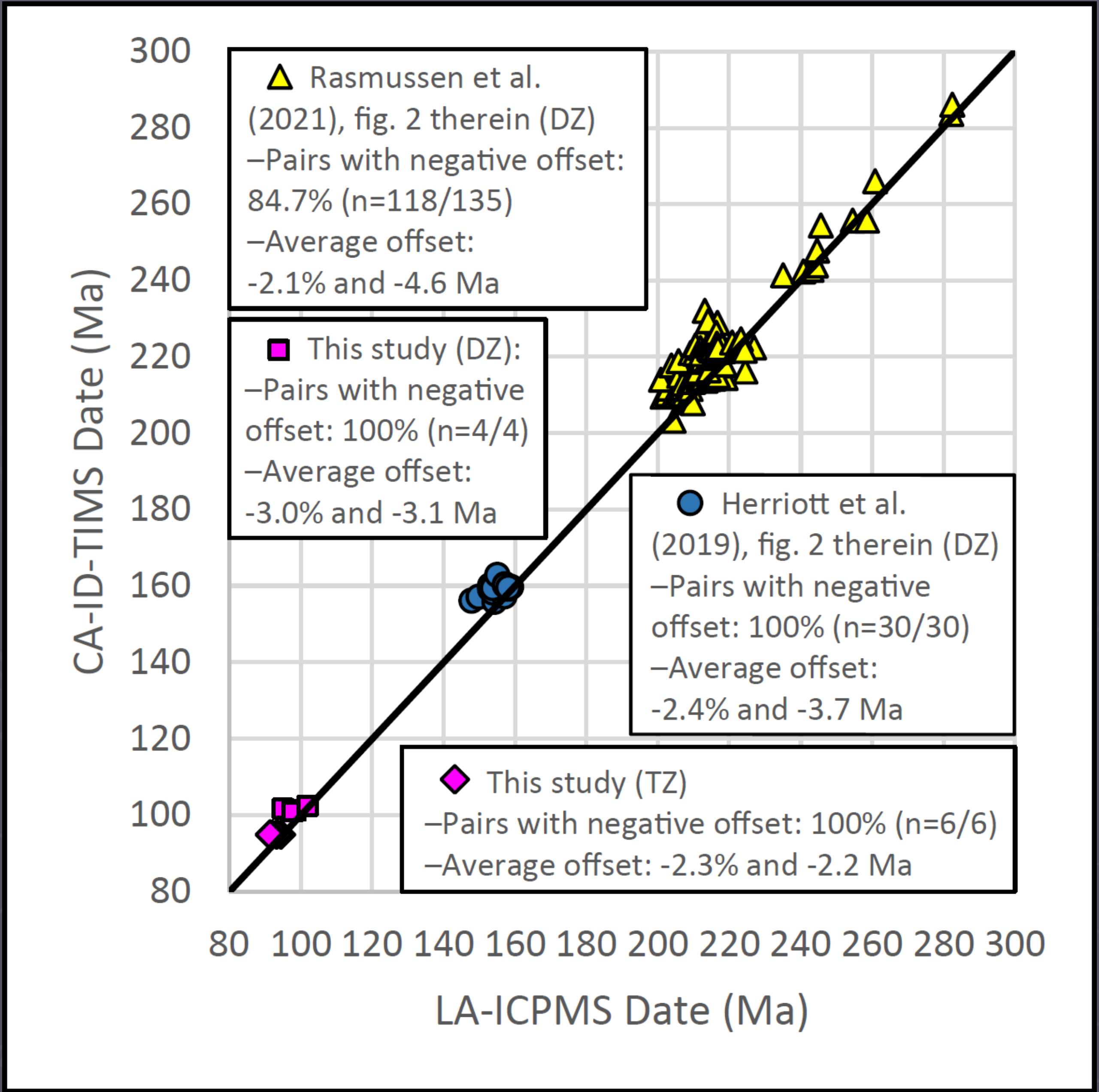


Tandem Date-Pair Plots

- This study (Slope Mtn. DZ and Ninuluk Bluff TZ)
- Rasmussen et al. (2021, *GSA Bulletin*)
- Herriott et al. (2019, *Geology*)

- Does analytical dispersion matter? Yes
- Is Pb-loss a problem? Almost certainly
- Do matrix effects affect unknowns? Almost certainly

- CA-TIMS diminishes, mitigates, and/or eliminates these uncertainties:
  - Potential for ~50X improvement of analytical precision (<0.1% precision and accuracy)
  - Pb-loss mitigation via chemical abrasion protocol
  - Inter-elemental mass fractionation corrections thru synthetic tracer solution rather than sample–standard bracketing of microbeam techniques, eliminating matrix effects



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Too-young biases ~2–3%

~85–100% pairs per study

Conclusions

- Obtaining accurate YSGs is improbable: Youngest date in an LA-ICPMS distribution maximizes total cumulative too-young bias regardless of source(s)
- Letter of the law application of the law of DZ effectively requires CA-TIMS
- YSG "successes" likely reflect crystallization to sedimentation lag and incorrectly benchmarking MDAs with existing stratal age constraints
- The benchmark for MDAs must be the crystallization age of the youngest zircon population sampled; tandem dating can provide that benchmark, whereas existing stratal age constraints, even if they are highly precise and accurate, cannot set the benchmark
- Multi-grain LA-ICPMS metrics are not poised to get the right answers for the right reasons if the underlying data bear geologic or systematic bias; low-temperature Pb-loss and/or matrix effects are capable culprits for these offsets

- Date-pair plots suggest that tandem dating should and will become more common for DZ MDA case studies
- Tandem date-pair plots can also shed light on the factors that render too-young bias on LA-ICPMS MDAs
- ~2–3% too young is a problem even at ± 2–3% (2σ)
- Expanding collaborative opportunities for tandem dating DZ MDA studies will become increasingly important

Acknowledgments

Thank you. Questions?

Lands: Alaska Native peoples have since the latest Pleistocene lived on the lands that we now study. Arctic Slope Regional Corporation granted access to their lands at Ninuluk Bluff

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Here's to Gil

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