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Creeping Closer: Clay Separation and X-ray Diffraction for Refined K/Ar Dating of Fault Motion on the Creeping Central Section of the San Andreas Fault

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Introduction

Creeping faults are typically not associated with large earthquakes. However, new K/Ar dating and biomarker maturity data on the San Andreas Fault Observatory at Depth (SAFOD) borehole[4] present evidence that large paleoearthquakes have occurred in the central creeping section of the San Andreas Fault, California[1].

K/Ar dating leverages the fact that heat released during co-seismic slip resets the geologic age. Strain during such a slip results in diagenesis: importantly, the formation of the clay mineral illite[2]. When this authigenic illite is purified from surrounding detrital materials, its age corresponds directly to the timing of the seismic event that created it.

Previous K/Ar ages of bulk samples with evidence of coseismic heating range from 3.3 to 15.8 Ma[1]. Further argon diffusion experiments, though, suggest that these ages are only partially reset, hence actual event ages may be even younger. Here, we aim to refine these ages by leveraging a size separate approach. Anticipating that the finest fractions will contain the purest sampling of authigenic illite with minimal detrital materials, the separates should produce a mixing line that can be used to estimate the timing of authigenic formation. The resulting insights into fault rock composition and the timing and location of past earthquakes will prove crucial in assessing the region's seismic hazard.

Sample Set

The San Andreas Fault Observatory at Depth (SAFOD) core comes from a region of the central creeping section of the San Andreas Fault. Located near Parkfield, California, the drill site concluded operations in 2007[4].

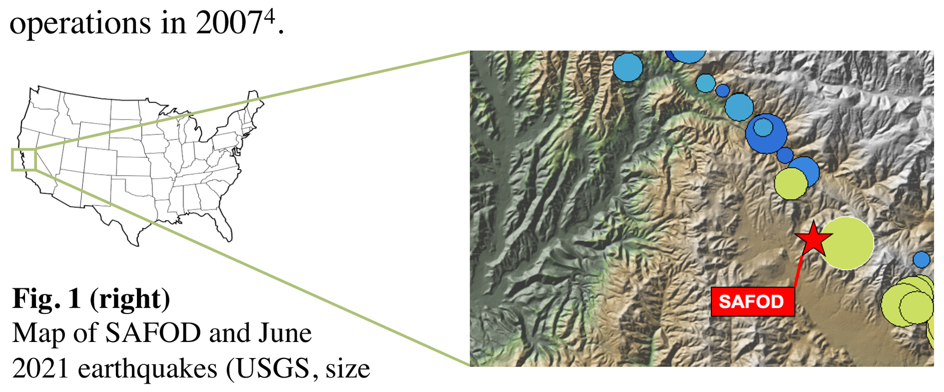

Fig. 1 (right) Map of SAFOD and June 2021 earthquakes (USGS, size = magnitude, color = depth)

Eight samples were selected from different depths along the core, both near and distant from the southern actively deforming zone.

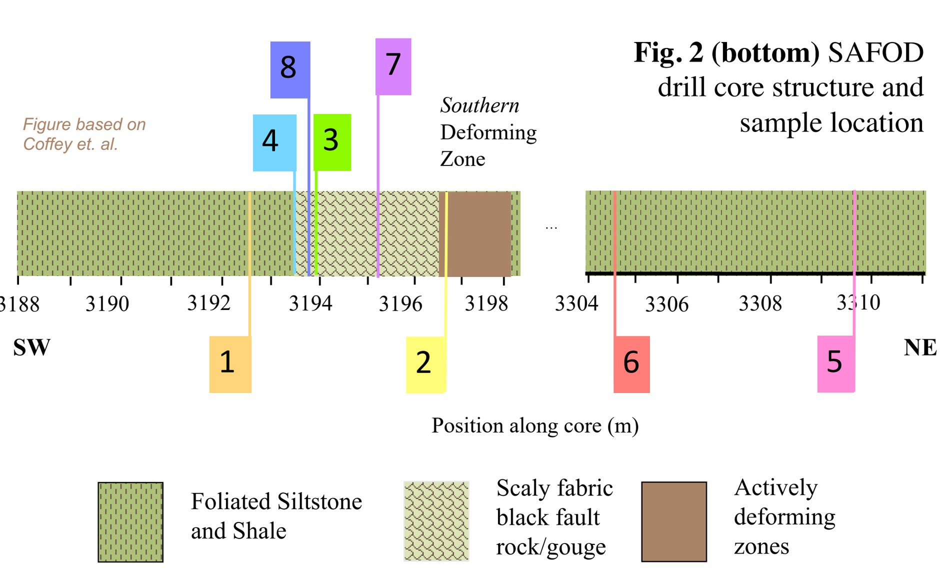

Fig. 2 (bottom) SAFOD drill core structure and sample location

Research Objectives

1. Quantify differences in clay mineral composition and illite crystallinity based on proximity to actively deforming zone
2. Quantify differences in clay mineral composition and illite crystallinity between size separates at the same depth
3. Determine illite K/Ar ages reset by friction-generated heat due to co-seismic slip during past earthquakes

Methods

Size Fraction Separation

Two hydrodynamic settling approaches were applied:

Greater than 2 micron ESD

- Settled samples in a 100 mL graduated cylinder for 3 hours and 57 minutes
- Extracted supernatant → used for separating less than 2 micron size fractions

Less than 2 micron ESD

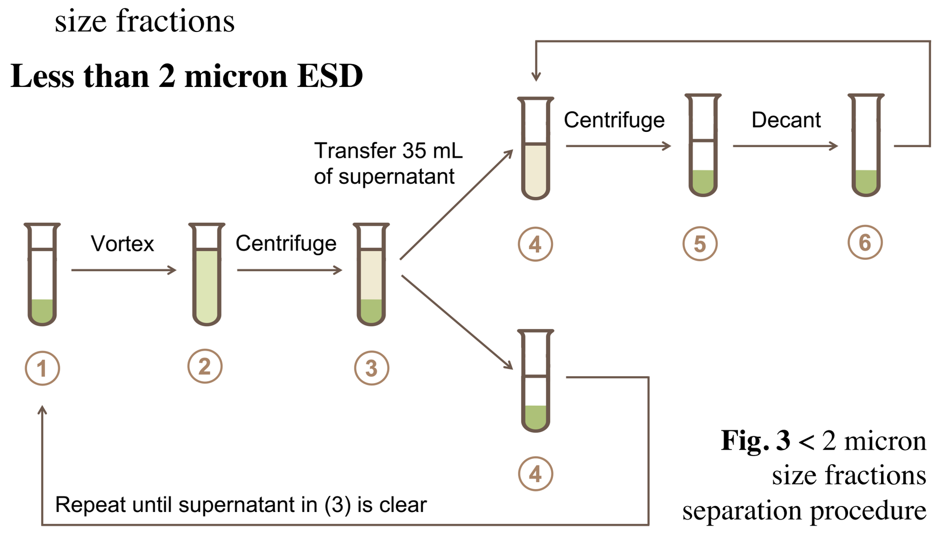

Fig. 3 < 2 micron size fractions separation procedure

- Separated based on Fig. 3 at times and speeds shown in table 1

	Size Fraction				
	< 0.2	0.2 – 0.5	0.5 – 0.8	0.8 – 1.4	1.4 – 2.0
Centrifuge Speed (RPM)	3500	2200	1360	780	550
Centrifuge Time (min)	24	10	10	10	10

Table 1 Centrifuge speeds and times for separating < 2 micron size fractions as determined by Stokes' Law

X-Ray Diffraction

- Ran each size separate through a BTX III Benchtop XRD Analyzer for 200 – 300 exposures

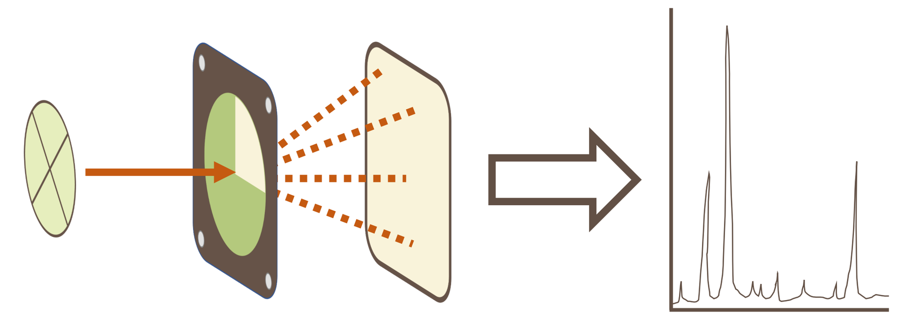

Fig 4 How the Transmission XRD Functions

- Carried out Rietveld (polytype) refinement with Profex

K/Ar Dating

- Loaded 0.2 – 0.6 mg of sample into micro furnaces
- Used a mass spectrometer to determine Ar ratios
- Estimated ages of samples from the Ar ratios determined here and the K ratios either determined through XRD (above) or isotope dilution (below)

Precise Determination of K Concentration by Isotope Dilution

- Column chromatography to isolate Potassium

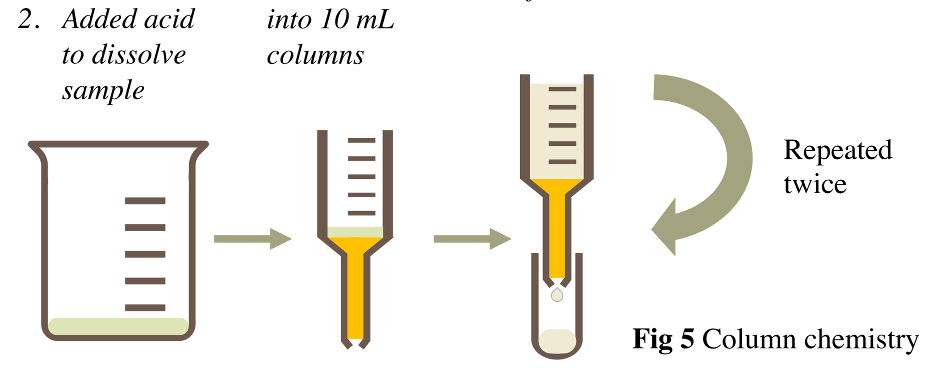

Fig 5 Column chemistry

- Measured isotopic ratios using a Multicollector-Inductively Coupled Plasma Mass Spectrometer (MC-ICPMS)

Results

Sample 4

- The age of the authigenic illite (lower intercept) indicates formation during seismic activity in the last 1.08 Ma years
- Age of detrital minerals (upper intercept) is 49.39 Ma, suggesting that the country rock has been minimally reset by recent earthquakes

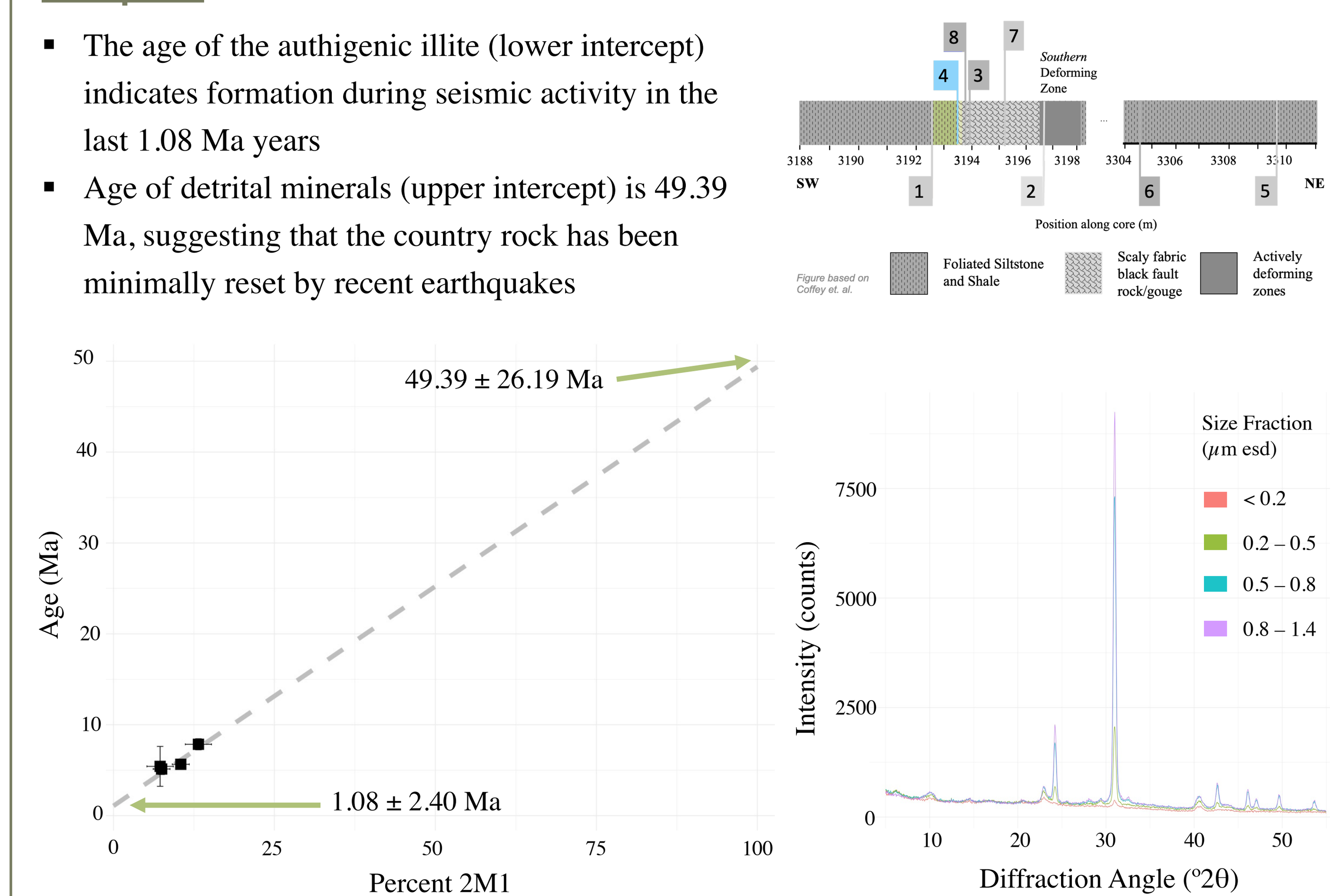

Fig 6 Phase analysis from XRD (right) and formation ages based on a York regression of age vs percent detrital materials (left). Some errors are smaller than the spot size

Sample 8

- The age of authigenic illite (lower intercept) indicates seismic activity has occurred as recently as 0.88 Ma
- Age of detrital minerals (upper intercept) is also young at 5.72 Ma, suggesting recent earthquakes have resulted in partial resetting of the country rock

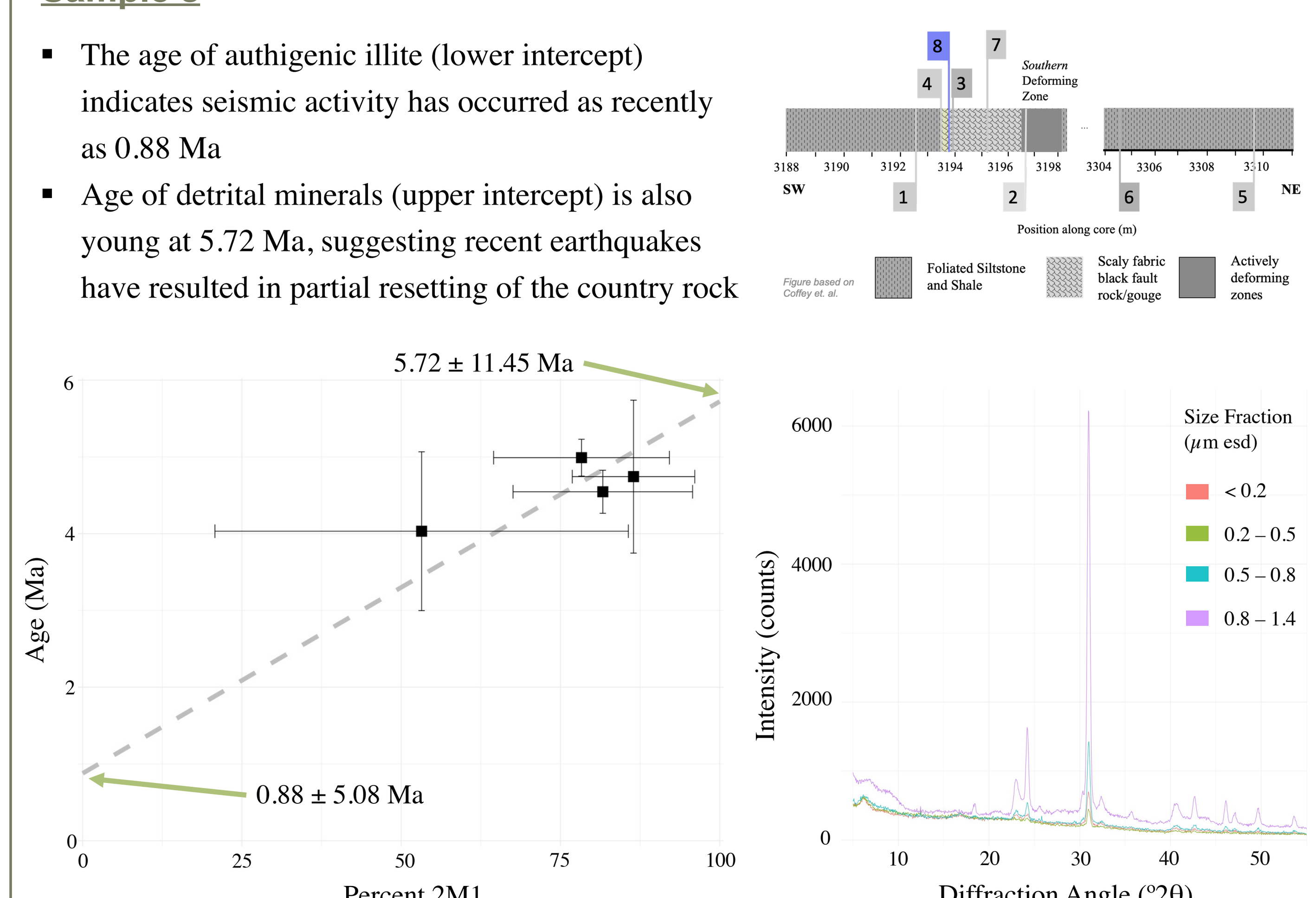

Fig 7 Phase analysis (right) and formation ages based on a York regression (left).

Potassium

- The difference between the potassium measurements and the estimates was greater for the smaller size fractions
- For the smaller size fraction, XRD overestimated potassium for sample 4 and underestimated potassium for sample 8

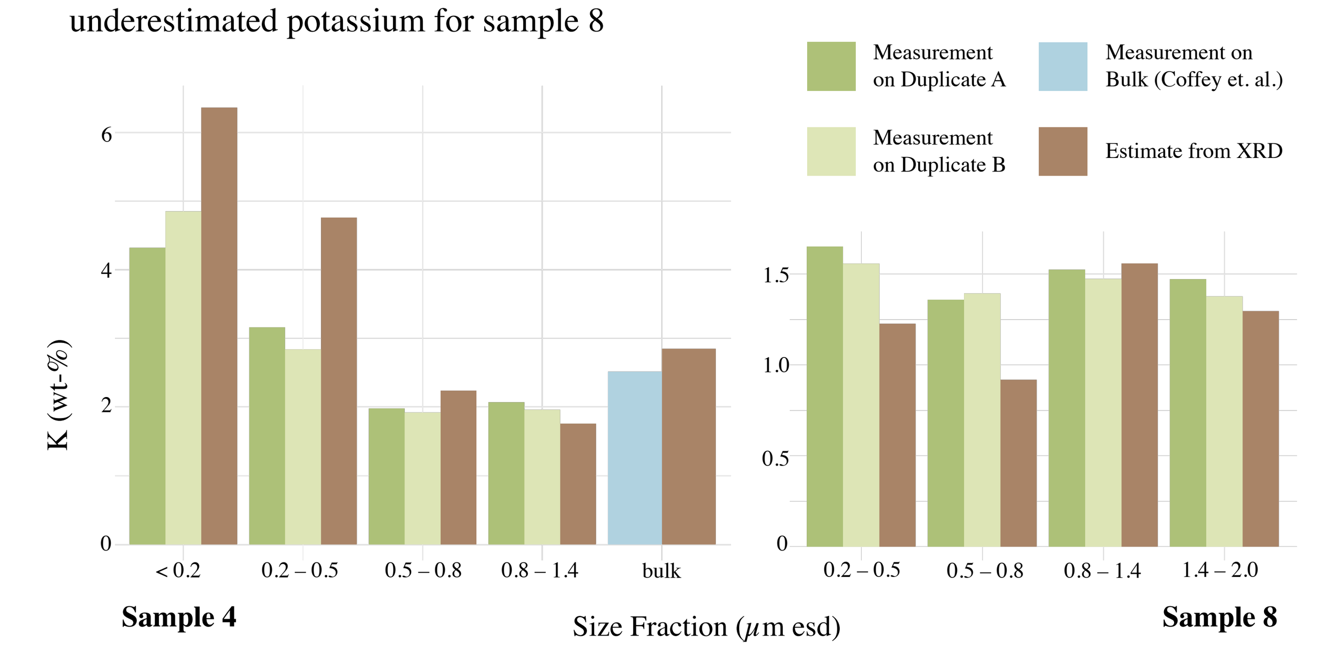

Fig 8 Comparing potassium measurements from isotope dilution to estimates from XRD

Conclusions and Next Steps

Mineralogy:

- There are quantifiable differences in the clay mineral composition and illite crystallinity, both between samples and between size fractions. These differences could be used to infer slip mechanisms.
- In general, finer size fractions tend to contain greater amounts of authigenic illite than larger size fractions and bulk aliquots.

Potassium Estimates from XRD:

- Potassium estimates were more accurate for larger size fractions. This could be the result of two characteristics of these aliquots:
  i. Comparably, a greater mass of the larger size fractions was able to be separated, allowing the XRD to be run with the suggested 15 mg of sample. Smaller size fractions were analyzed with smaller, suboptimal quantities that resulted in diffraction patterns with significant noise, complicating potassium concentration extrapolation.
  ii. The larger size fractions contained proportionately lesser amounts of the clay minerals that produce low angle peaks. 2θ values less than 5 are outside the range of BTX III Benchtop XRD Analyzer, and the abundance of these minerals in the smaller size fractions appears to generate noise in the final diffraction pattern.

K/Ar Dating:

- There has been recent seismic activity in the central creeping section of the San Andreas Fault within the last million years. This is the age of the authigenic illite, which forms in the deformation zone of faults during fault slip[3].
- Sediments from the same region of the core host similar age signals. Sample 4 and Sample 8 are samples of black fault rock located within 30 centimeters of each other and were measured at similarly young ages of 1.08 Ma and 0.88 Ma respectively.

Next Steps

1. Perform isotope dilution with a spike approach[4] on the six remaining samples, using the same aliquots that have already been used for Ar mass spectrometry.
2. Refine clay mineral composition and illite crystallinity data. In-house XRD results will be compared to those obtained through the sample mail-in service for the Advanced Photon Source (APS) at the DOE National Argonne Lab
   - Their 11-BM Beamline performs high resolution powder diffraction that is capable of processing:
     a. Small quantities of samples
     b. Clay minerals which produce similar low angle peaks

References

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