INVESTIGATING ZIRCON SHOCK MICROSTRUCTURES WITH NANOSIMS. C. A. Crow[1], B. Jacobsen[2], K. D. McKeegan[1], and D. E. Moser[3] [1]University of California, Los Angeles (Department of Earth, Planetary, and Space Sciences, 595 Charles Young Drive E., Box 951567, Los Angeles, CA 90095; ccrow@ucla.edu), [2]Lawrence Livermore National Laboratory, Livermore, CA, [3]University of Western Ontario, London, Canada.

Introduction: The presence of impact shock microstructures has been used to interpret the significance of lunar and terrestrial zircon Pb-Pb and U-Pb ages [eg. 1, 2]. In end member scenarios of no shock deformation and complete impact induced recrystallization, the ages are generally interpreted as the crystallization age and the impact age, respectively [1, 2]. For cases of intermediate shock deformation, the effects of microstructure formation on the measured isotopic ages are not clear, especially in the lunar zircon population.

In some terrestrial and lunar zircons, shock microtwins (usually ~1-2 μm in width) have been associated with Pb-loss, but the fact that not all microtwins have this association leaves their effect on the ages open to debate [e.g. 1, 2]. Moreover, the small size of these features has made it difficult to directly investigate their effects on U-Pb ages by conventional SIMS methods.

Here we report preliminary U-Pb and Pb-Pb analyses of a set of ~5 μm lunar zircon microtwins made with the Lawrence Livermore National Lab (LLNL) NanoSIMS 50. These analyses were undertaken to both (1) test the spatial limits and feasibility of U-Pb age analyses of zircons using the Hyperion primary beam source at LLNL, and (2) to investigate the distribution of U, Pb, and Fe across microtwin structures.

Sample: NanoSIMS analyses were conducted on a zircon separated from lunar sample 14305 - a clast-rich, crystalline matrix breccia. The zircon, Z34 "Disco", was selected for analysis based on scanning electron microscopy (SEM) electron backscatter diffraction (EBSD) images collected at the University of Western Ontario (Figure 1).

The EBSD analyses revealed that Z34 is a highly shocked grain containing two crosscutting sets of shock microtwins, one of which is curved and larger than typical structures (in some places up to ~ 6 μm in width). The curved microtwins are also associated with impact melt glass inclusions, typically ~ 1μm in diameter, which are easily resolved by the NanoSIMS analyses (Fig. 2). Prior to NanoSIMS analyses, an unshocked region of Z34 was dated with the UCLA Cameca IMS1270 giving a ^{207}Pb-^{206}Pb age of 4324 ± 5 Ma (1σ) and a ^{206}Pb-^{238}U age of 4231 ± 94 Ma (1σ).

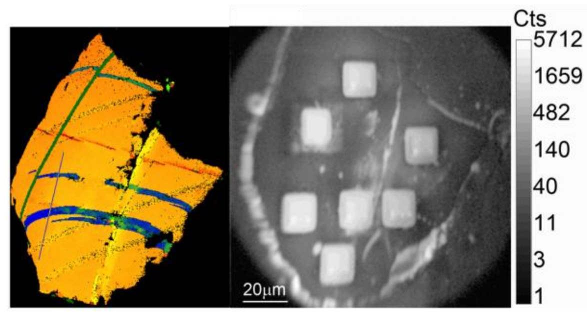

Figure 1: (Left) EBSD Map of 14305 Z34 "Disco". Blue features are curved microtwins; green is a second crosscutting microtwin; pixilated bands are zones of less crystalline zircon, most likely high U concentration primary zoning. (Right) NanoSIMS total ion image showing location of analyses.

Methods:

Instrumental Setup. We used a 600 pA $^{16}O^-$ primary beam to obtain a 2x3 μm spot size. Previously reported U-Pb NanoSIMS analyses used a 15 μm spot size with a 7 to 9 nA beam [3]. Analyses were collected in mapping mode where the beam was rastered over a 13 μm box, and data was collected from the inner 10 μm to exclude edge effects. Each analysis took ~ 2 hours in order to achieve sufficient counting statistics.

Tuning and peak identification was conducted on NIST610 glass and AS3 zircon standards. Ion intensities were measured on five electron multipliers (EM) by stepping the magnet over four B-field settings. The configuration of the trolley system is shown in Table 1. A high mass resolution scan of ^{30}Si gave a mass resolving power of ~4000 (6000 Cameca units).

Table 1: NanoSIMS Trolley Configuration

T1	T2	T3	T4	T5
^{30}Si	^{56}Fe		^{204}Pb	^{238}U
			^{206}Pb	
			^{207}Pb	
		$^{90}Zr_2{}^{16}O$	^{208}Pb	$^{238}U^{16}O$

Calibration. Instrumental U-Pb relative sensitivity was determined by analyzing AS3 standard zircons under the same measurement conditions; this is similar to the protocol used for the IMS-1270 at UCLA and elsewhere [4]. AS3 zircons have a known, homogeneous age of 1099.1 ± 0.2 Ma although the uranium concentration is heterogeneous [5]. A linear fit of the UO+/U+ versus ^{206}Pb+/^{238}U+ relative sensitivity factor (measured ratio/true ratio) for the AS3 zircons was used to infer the RSF of the unknown analyses. The standards and unknowns had a similar range of UO/U values, so no extrapolation was necessary.

Data Reduction & Age Calculation. We applied an EM deadtime correction, a background subtraction, and a common ^{204}Pb correction. Terrestrial common Pb values were used for both lunar and terrestrial zircon corrections because it has previously been shown that the U-Pb ages of lunar zircons in grain mounts are not affected by the use of terrestrial vs. lunar common Pb values [e.g. 6 Supplementary Materials].

The ^{207}Pb-^{206}Pb ages were calculated from these corrected values using an iterative Matlab code. Uncertainties on these ages are dominated by counting statistics. In contrast, uncertainties on U-Pb ages are a function of both counting statistics and the uncertainty in the determination of the RSF value.

Results: We collected six analyses of 14305 Z34 - three which contained shock microtwins and three in unmetamorphosed areas. Impact melt glass inclusions ~1 μm in diameter (Fig 2), which are associated with the curved shock microtwins in SEM images, were resolved in the ^{54}Fe/^{30}Si maps (the inclusions were removed for U-Pb and Pb-Pb age calculations). We also resolved a <5 μm high U concentration band (Fig 2), which most likely results from primary igneous zoning. The identification of these two features suggest that the spatial resolution is sufficient to resolve the ~5 μm shock microtwins in the U-Pb age images.

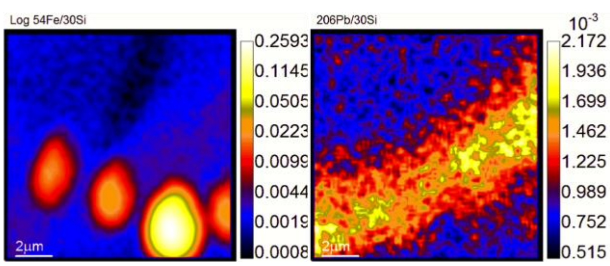

Figure 2: NanoSIMS map of (Left) ^{54}Fe/^{30}Si showing resolved ~ 1 μm impact melt glass inclusions, and (Right) map of ^{238}U/^{30}Si showing resolved primary growth band of higher U concentration. Both images are 10 x 10 μm and have a binning size of 3 pixels.

In each of the ^{206}Pb/^{238}U and ^{207}Pb/^{238}U maps we find no resolvable variation across the twins. This suggests that the process by which these shock microtwins formed was too rapid to cause appreciable Pb mobility. Alternatively, the timing of the impact event that produced the microtwins was sufficiently close to the crystallization age of the zircon that we cannot resolve the age difference within our measurement uncertainties. The former interpretation would be consistent with U-Pb results from terrestrial 'cold shock' micro-twinned zircons [1].

The average ^{206}Pb-^{238}U age of the NanoSIMS analyses is 4156 ± 77 (1σ) Ma, which is within error of the 4231 ± 94 (1σ) Ma age measured by the IMS1270 at UCLA. Similarly, the ^{207}Pb-^{206}Pb ages range from 4318 ± 39 to 4237 ± 58 Ma (1σ) which is also with in error of the IMS1270 age of 4324 ± 5 (1σ). Figure 3 shows the Concordia diagram for the NanoSIMS analyses of Z34 "Disco".

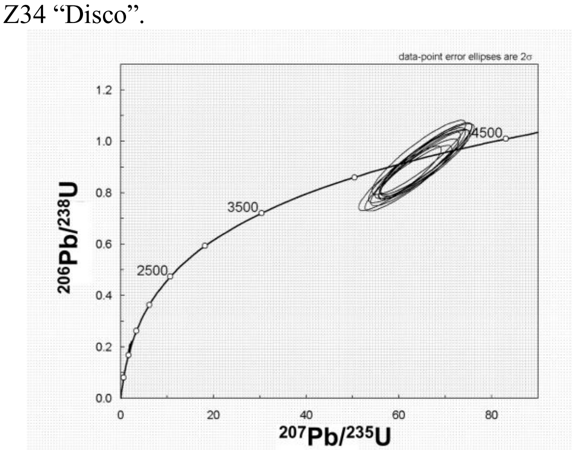

Figure 3: Concordia diagram for NanoSIMS analyses of lunar zircon 14305 Z34 "Disco" including measurements of regions containing shock microtwins and those areas devoid of microtwins.

Future analyses will aim to reduce the errors on individual U-Pb and Pb-Pb ages, but our preliminary conclusion is that formation of these sorts of curviplanar microtwins does not locally result in large degrees of Pb-loss or U-Pb isotopic age disturbance of impacted lunar zircons. Higher precision measurements should allow us to test whether other microstructural domains in this complex grain exhibit clear impact-related discordance.

References:

[1] Moser D. E. et al. (2011) *Can. J. Earth Sci., 48,* 117-139. [2] Grange M. L. et al. (2013) *Geochim. Cosmochim. Acta, 101,* 112-132. [3] Takahata N. (2008) *Gondwana Research, 14* 587-596. [4] Compston W. et al. (1984) *LPSC XIV, J. Geophys. Res., 89,* B525-B534. [5] Paces J. B. and Miller J. D. (1993) *J. Geophys. Res., 98,* 13997-14013. [6] Taylor D. J. (2009) *EPSL, 279,* 157-164.