Automated NanoSIMS measurements & presolar grains

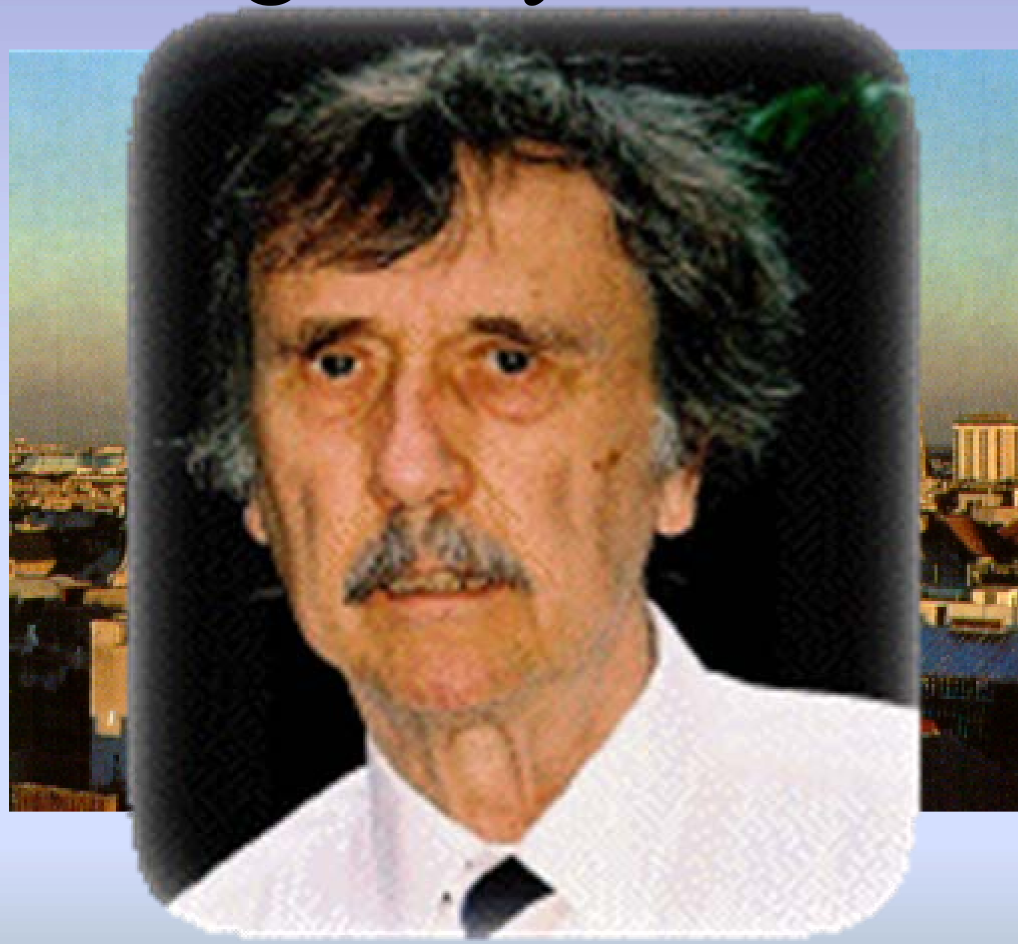
Frank Gyngard

Alain Morgand

Larry Nittler

…wasting away in Austria

Ernst Zinner



…checking for roid ragers at daughter's swim meet

Frank Stadermann

Automated NanoSIMS measurements & presolar grains

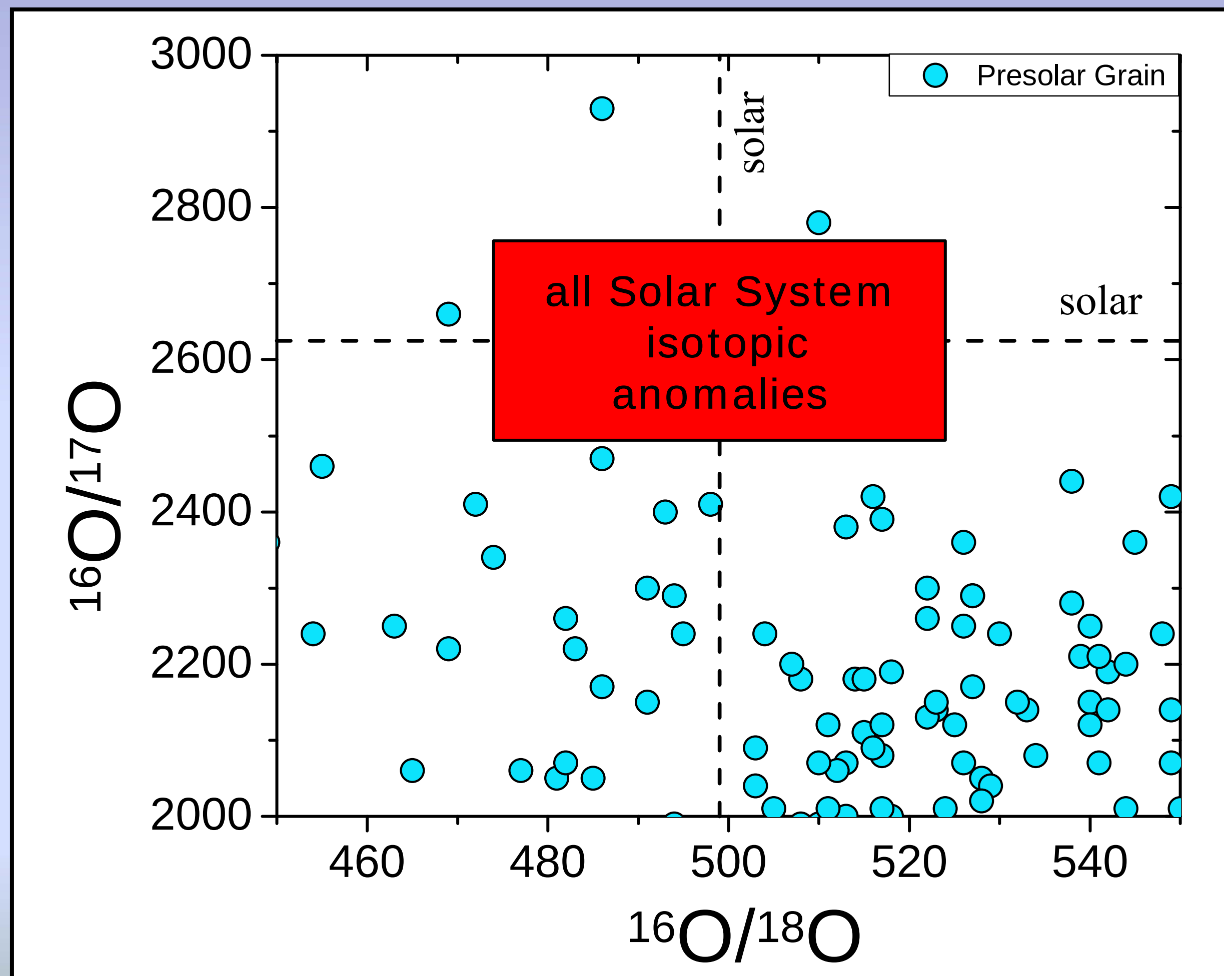
Frank Gyngard
Alain Morgand
Larry Nittler

Thanks to John Valley & Noriko Kita for the invitation!

How do we know they're presolar?

Isotopes!

- A grain from a single star will likely have an isotopic composition noticeably different from this average



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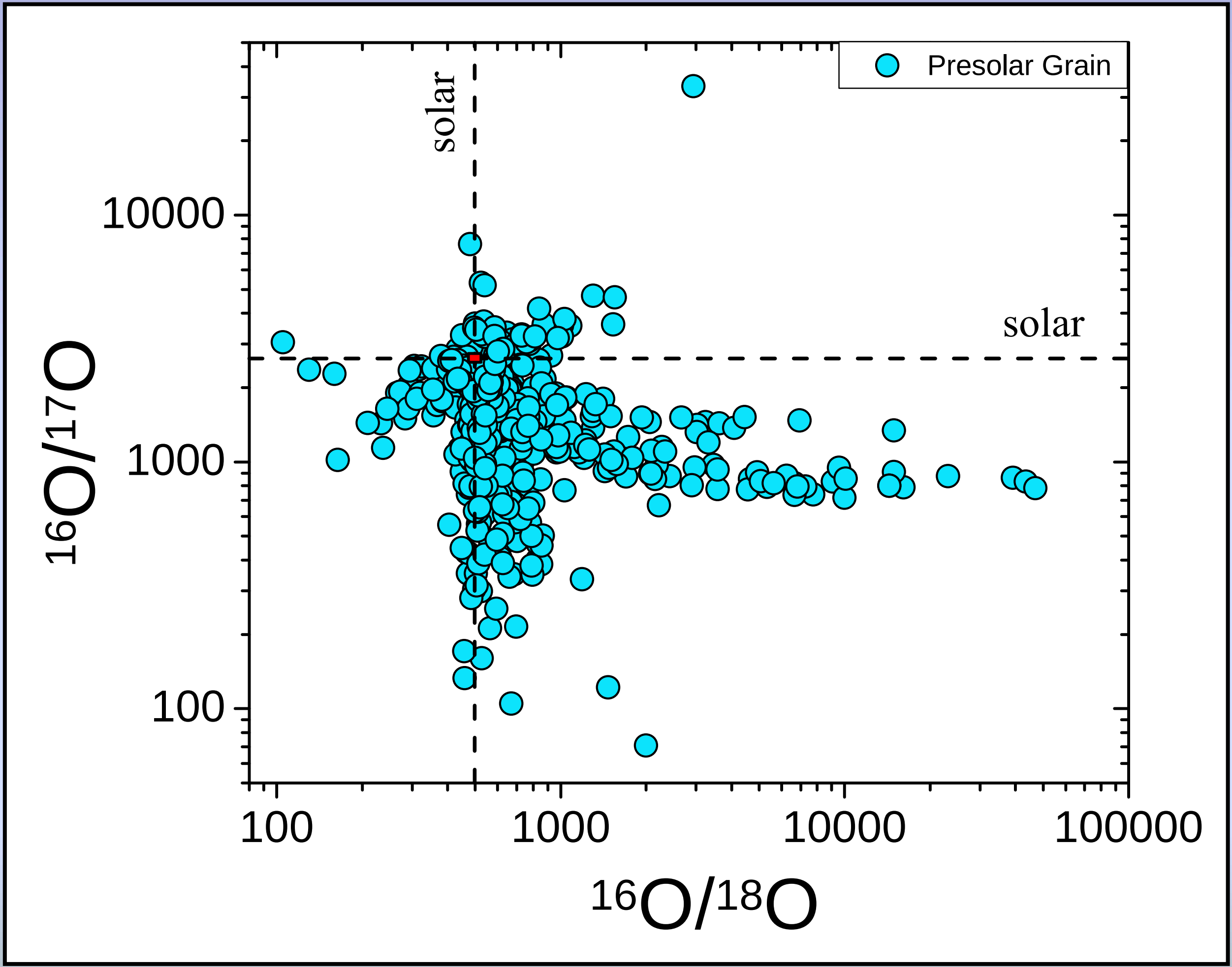
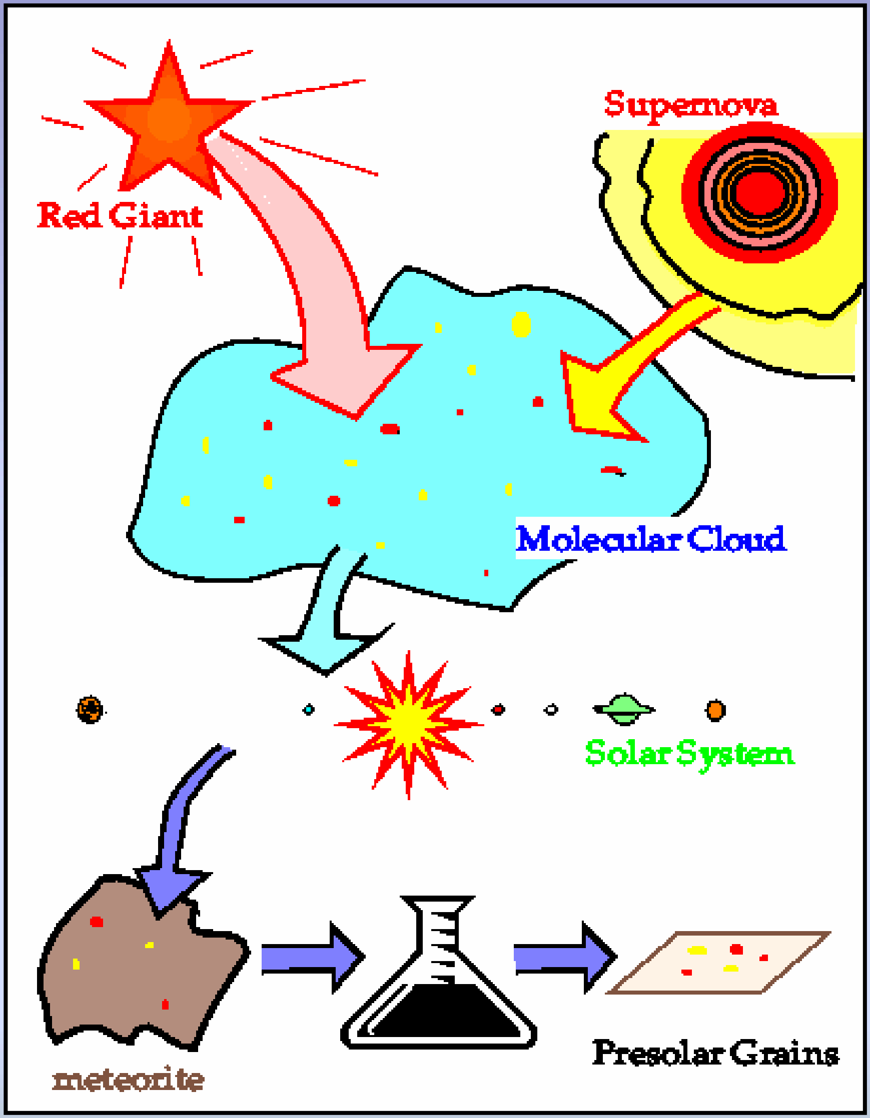



Red Giant
Supernova
Molecular Cloud
Solar System
meteorite
Presolar Grains

Presolar grains…all grown up

10,000+ grains *individually* analyzed

Carbides, oxides, silicates, nitrides, etc.

IMS-f series, SHRIMP, 1280, NanoSIMS, etc.

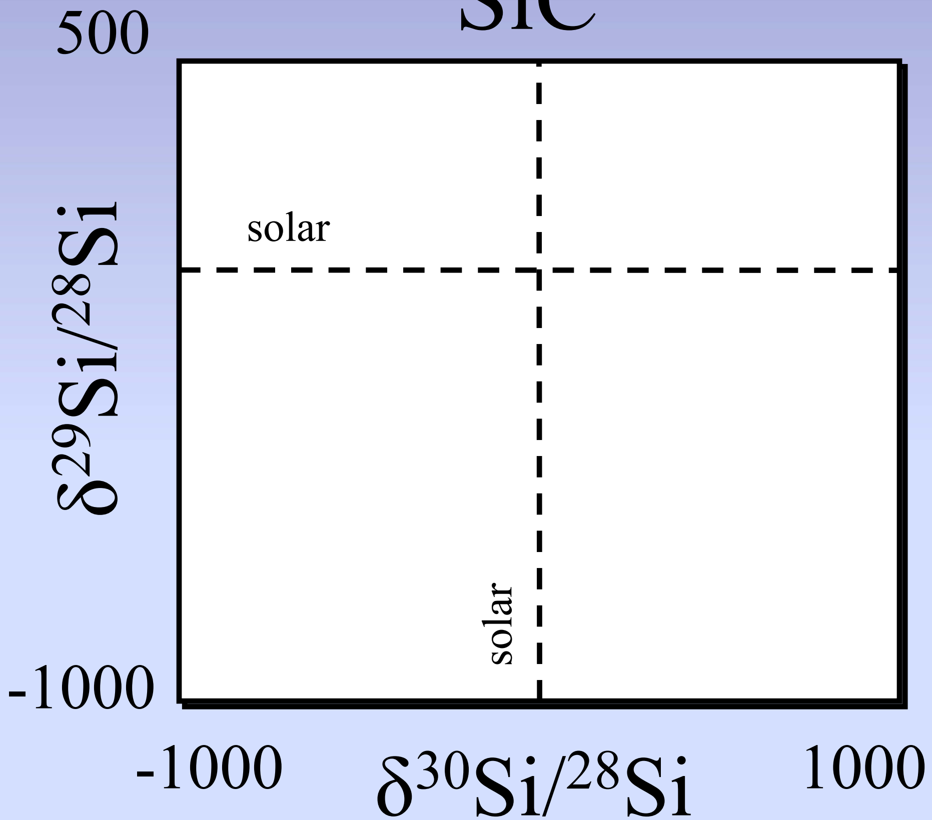
Where do we go from here?

~~Better spatial resolution: $\lesssim$10 nm~~

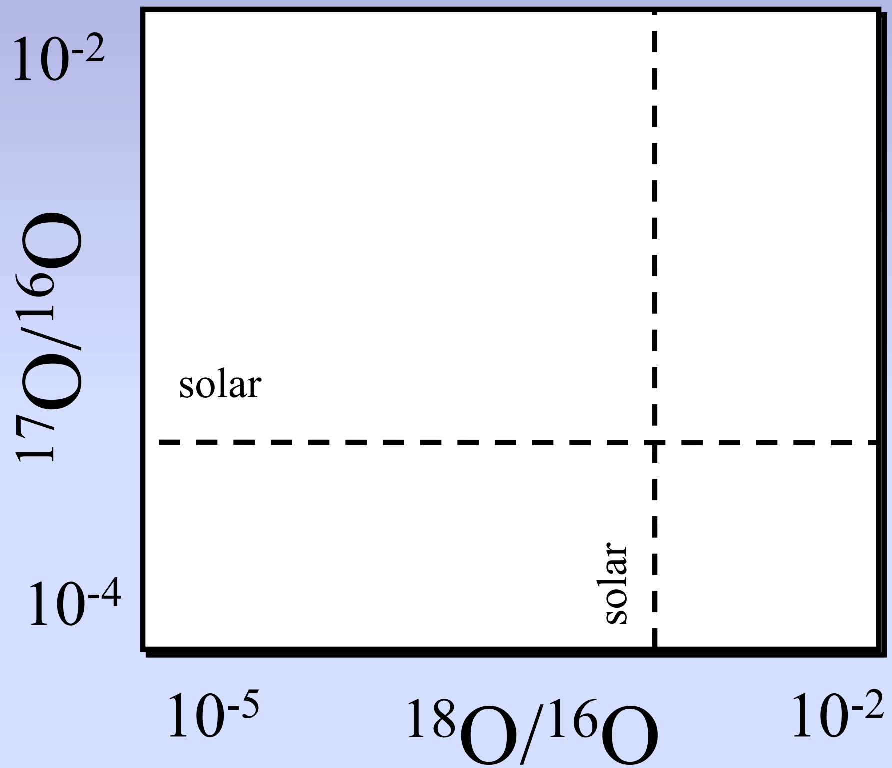
Search for rare grains

CAMECA SCIENCE & METROLOGY SOLUTIONS

SiC



Oxides/Silicates



$$\delta^{i}Si/^{28}Si = [(^{i}Si/28Si)_{measured}/(^{i}Si/^{28}Si)_{\odot} - 1] \times 1000$$

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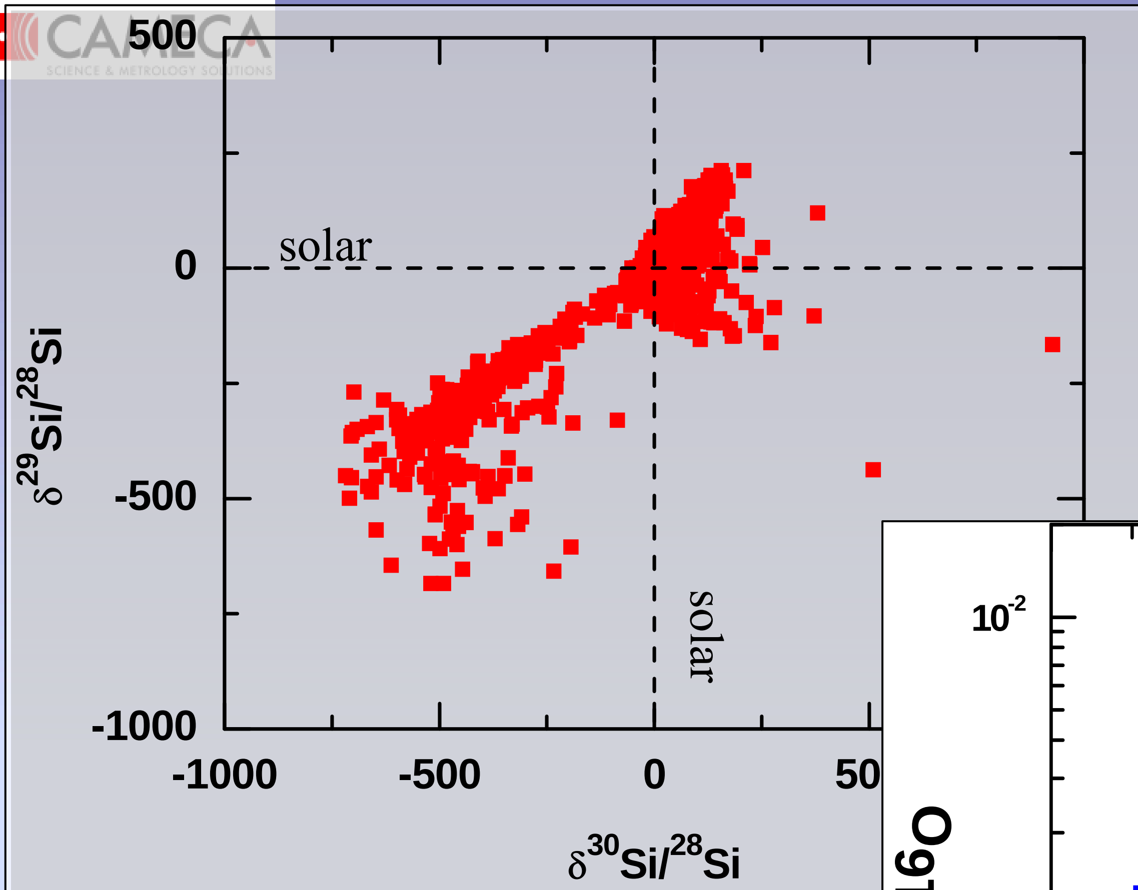
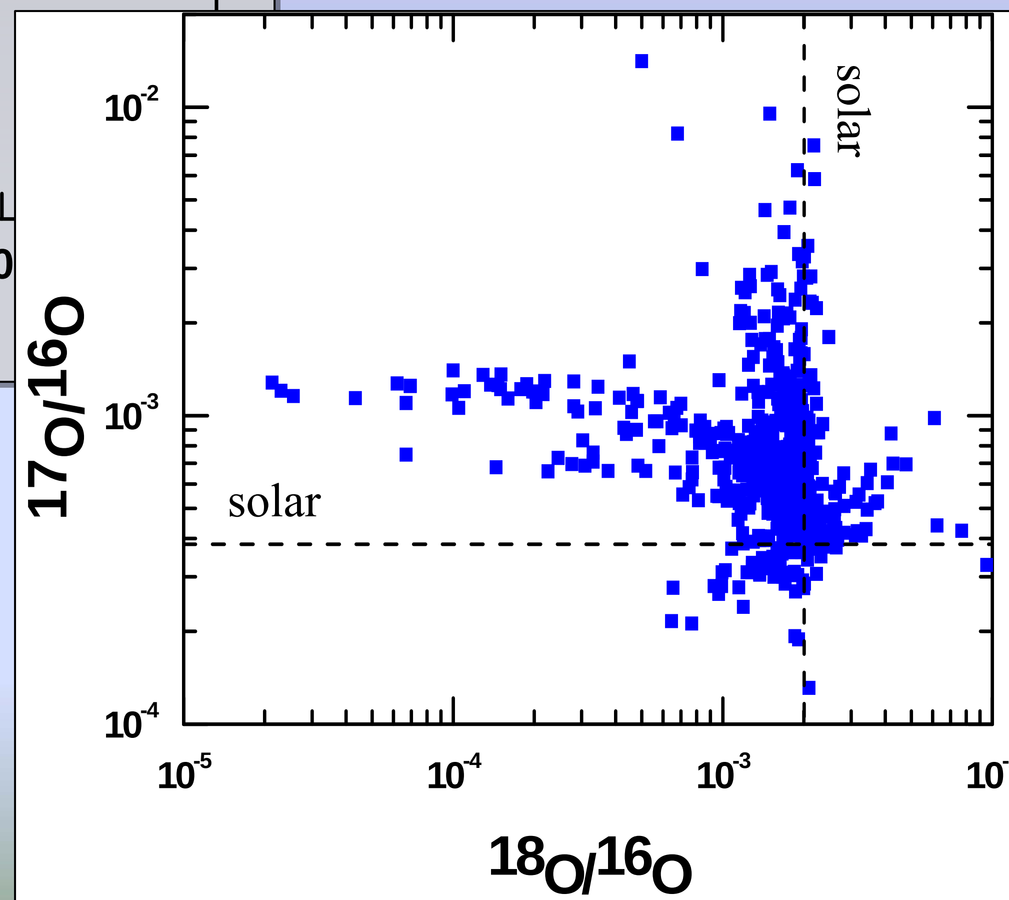
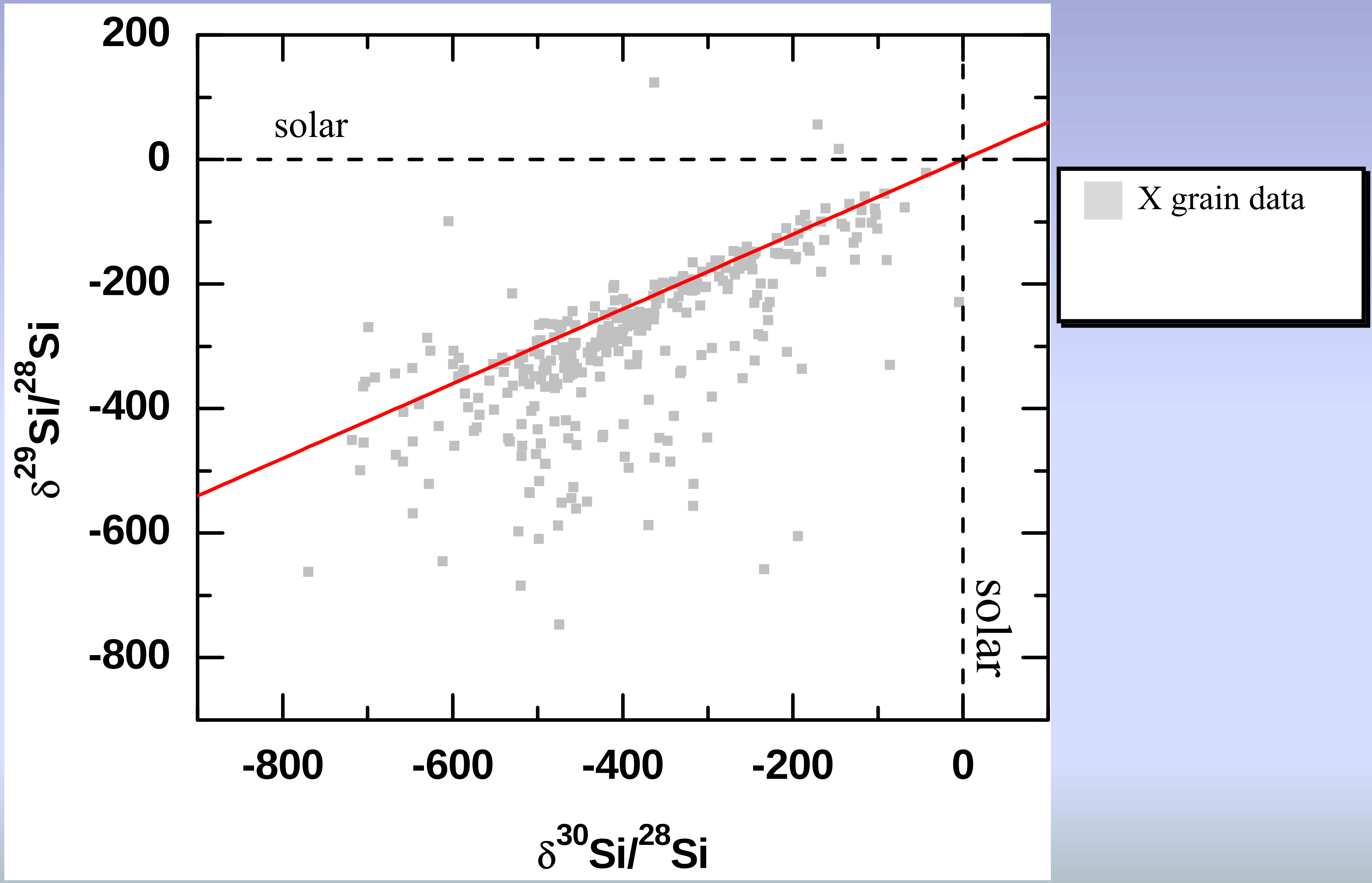


>99% of all SiC, oxides, & silicates



Please visit:
http://presolar.wustl.edu/~pgd/

SiC X grains: supernova dust!



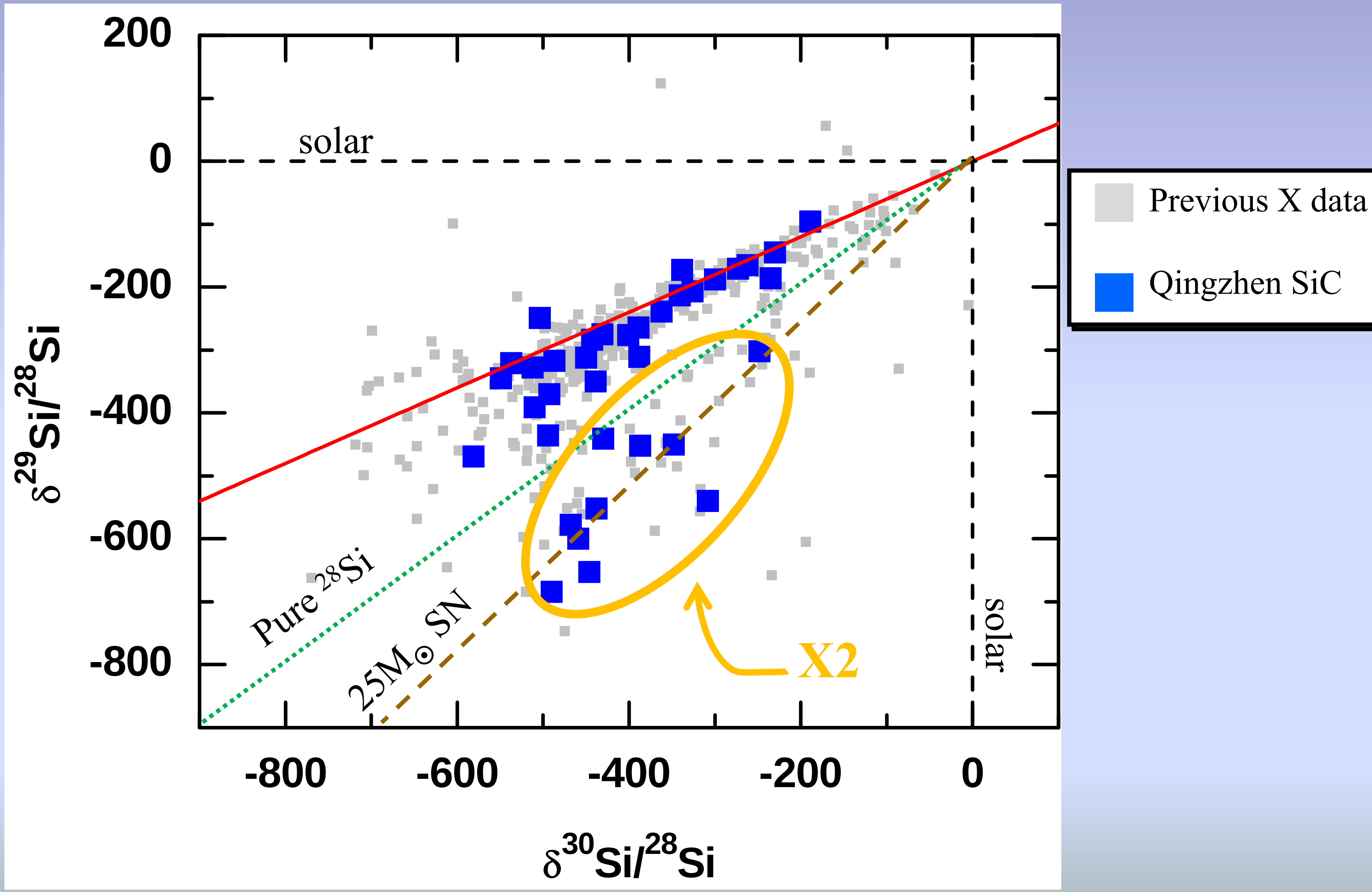
Example: SiC from Qingzhen meteorite

X grains: $\lesssim$ 0.25%

X2 grains: ~1/4 of all Qingzhen X grains

Abundance of X2 grains: 0.063%

SiC X grains: supernova dust!



Search for rare grains

Automated, high-throughput measurements

Lots of instrument time, which is costly

Efficient grain definition & sorting algorithms

Direct Imaging

Defocus ~50-100 µm static primary beam over area to analyze

Send entire ion image through mass spectrometer

Use a CCD camera to image channel plate/fluorescent screen…or SCAPS

Raster Imaging

Raster small (~1-0.1 µm) primary beam over an area

Synchronize secondary ions w/primary ion raster

Reconstruct original location of sputtered ions

Use electron multipliers

"…what one fool can do, another can."

Available online at www.sciencedirect.com
SCIENCE @ DIRECT
Applied Surface Science 252 (2006) 7148–7151

ELSEVIER

Automated ion imaging with the NanoSIMS ion micropr

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Received 12 September 2005; accepted 15 February 2006
Available online 23 May 2006

Mem. S.A.It. Vol. 77, 903

Memorie della

Helium and neon in single presolar grains from the meteorites Murchison and Murray

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Abstract. In this study we present single grain analyses of He and Ne in presolar SiC extracted from the meteorites Murchison and Murray and find a larger fraction of noble gas-rich grains using an improved instrumental detection limit. We combine our study with NanoSIMS ion microprobe analyses to classify the grains, and compare our isotopic data with new AGB star nucleosynthetic model predictions.

Key words. dust, extinction — circumstellar matter — methods: laboratory — methods: analytical — nuclear reactions, nucleosynthesis, abundances

1. Introduction

Helium and neon are prominent nuclear fusion products of AGB stars and can be implanted by stellar winds into circumstellar condensates

2. Methods

Presolar grains were extracted from the carbonaceous chondrites Murchison and Murray at the MPI for Chemistry in Mainz with

Pergamon

Geochimica et Cosmochimica Acta, Vol. 67, No. 24, pp. 4961–4980, 2003

0016-7037/03 $30.00 + .00

doi:10.1016/S0016-7037(03)00485-X

Automated isotopic measurements of micron-sized dust: Application to meteoritic presolar silicon carbide

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(Received February 4, 2003; accepted in revised form July 3, 2003)

Abstract—We report the development of a new analytical system allowing the fully automated measurement of isotopic ratios in micrometer-sized particles by secondary ion mass spectrometry (SIMS) in a Cameca ims-6f ion microprobe. Scanning ion images and image processing algorithms are used to locate individual particles dispersed on sample substrates. The primary ion beam is electrostatically deflected to and focused onto each particle in turn, followed by a peak-jumping isotopic measurement. Automatic measurements of terrestrial standards indicate similar analytical uncertainties to traditional manual particle analyses (e.g., ~3‰/amu for Si isotopic ratios). We also present an initial application of the measurement system to obtain Si and C isotopic ratios for ~3300 presolar SiC grains from the Murchison CM2 carbonaceous chondrite. Three rare presolar Si_3N_4 grains were also identified and analyzed. Most of the analyzed grains were extracted from the host meteorite using a new chemical dissolution procedure. The isotopic data are broadly consistent with previous observations of presolar SiC in the same size range (~0.5–4 μm). Members of the previously identified SiC AB, X, Y, and Z subgroups were identified, as was a highly unusual grain with an extreme ^{30}Si enrichment, a modest ^{29}Si enrichment, and isotopically light C. The stellar source responsible for this grain is likely to have been a supernova. Minor differences in isotopic distributions between the present work and prior data can be partially explained by terrestrial contamination and grain aggregation on sample mounts, though some of the differences are probably intrinsic to the samples. We use the large new SiC database to explore the relationships between three previously identified isotopic subgroups—mainstream, Y, and Z grains—all believed to originate in asymptotic giant branch stars. The isotopic data for Z grains suggest that their parent stars experienced strong CNO-cycle nucleosynthesis during the early asymptotic giant branch phase, consistent with either cool bottom processing in low-mass ($M < 2.3M_\odot$) parent stars or hot-bottom burning in intermediate-mass stars ($M > 4M_\odot$). The data provide evidence for a sharp threshold in metallicity, above which SiC grains form with much higher $^{12}C/^{13}C$ ratios than below. Above this threshold, the fraction of grains with relatively high $^{12}C/^{13}C$ decreases exponentially with increasing $^{29}Si/^{28}Si$ ratio. This result indicates a sharp increase in the maximum mass of SiC parent stars with decreasing metallicity, in contrast to expectations from Galactic chemical evolution theory. *Copyright © 2003 Elsevier Ltd*

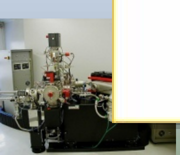
1. INTRODUCTION

…otopic measurements of micron-sized materials tradition- …have been sparse, because the relatively small number of …s available limits the achievable measurement precision. …ever, scientific interest in the isotopic compositions of …on-sized dust grains has greatly increased in recent years, … the advent of highly sensitive measurement techniques …, secondary ion mass spectrometry [SIMS]) and the dis- …ry of relatively large isotopic variations in some micro- …c materials. For example, primitive meteorites contain a …fraction of mineral grains with highly unusual isotopic …positions, compared to any other meteoritic or terrestrial …rials. These presolar grains are believed to have originated …e cooling outflows from ancient stars and supernova ex- …ons before the formation of the solar system. Since their …very in 1987, presolar grains have yielded a wealth of …mation on astrophysical and cosmochemical processes

from nuclear enrichment facilities are used to monitor nuclear treaty compliance (Simons et al., 1998; Tamborini and Betti, 2000). Finally, there is recent interest in the isotopic composition of aerosol particles collected from the Earth's atmosphere (Aléon et al., 2002).

SIMS has been the most widely used analytical technique for measuring isotopic ratios in small particles, due to its high sensitivity and spatial resolution (Zinner, 1989; Zinner et al., 1989). Traditional SIMS measurements of single micron-sized particles are quite time-consuming, however, requiring a minimum of several minutes to locate a sample, align the primary beam with it, and analyze it. This relative inefficiency makes it difficult to obtain statistically significant datasets, especially for rare grain populations. Automated techniques in these cases are thus highly desirable.

Previously, direct ion imaging with Cameca ims-3f and ims-4f ion microprobes has been used with considerable suc-



Automatic measurement technique

1) Sputter clean sample surface

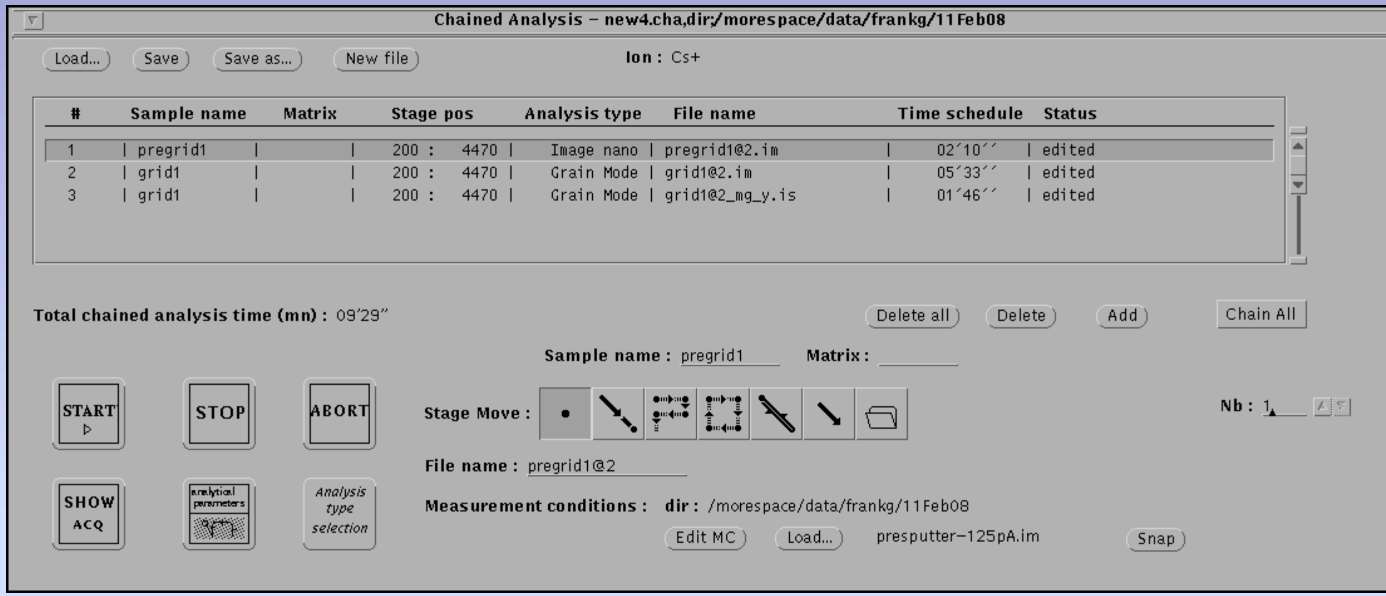
2) Acquire ion images

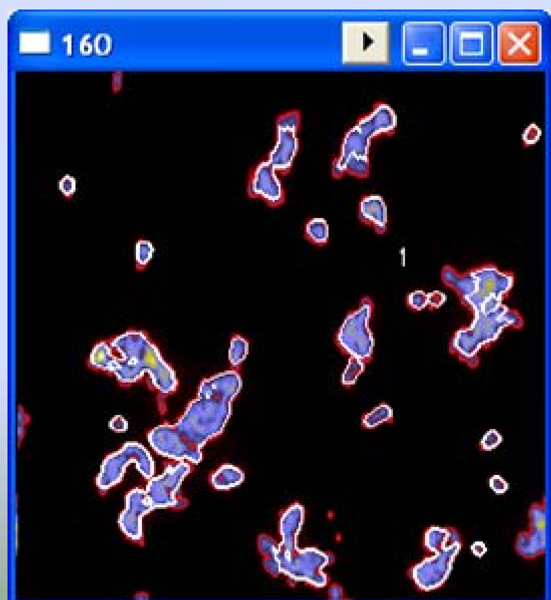
3) Automatically define particles to measure

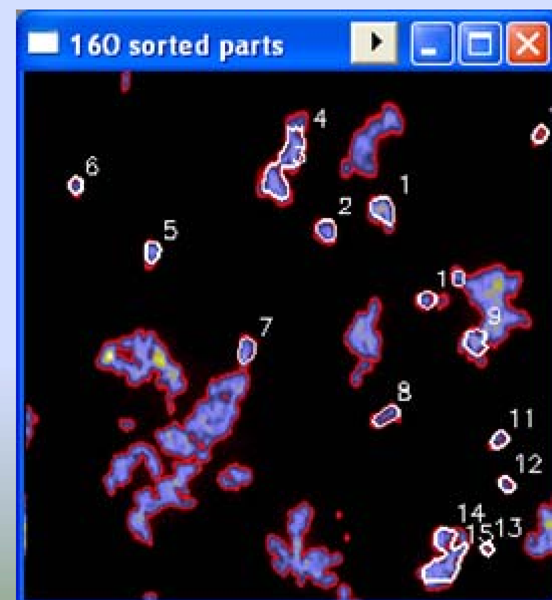
4) Make high mass resolution (HMR) measurements

5) Move sample stage and repeat

Integrated into Cameca software





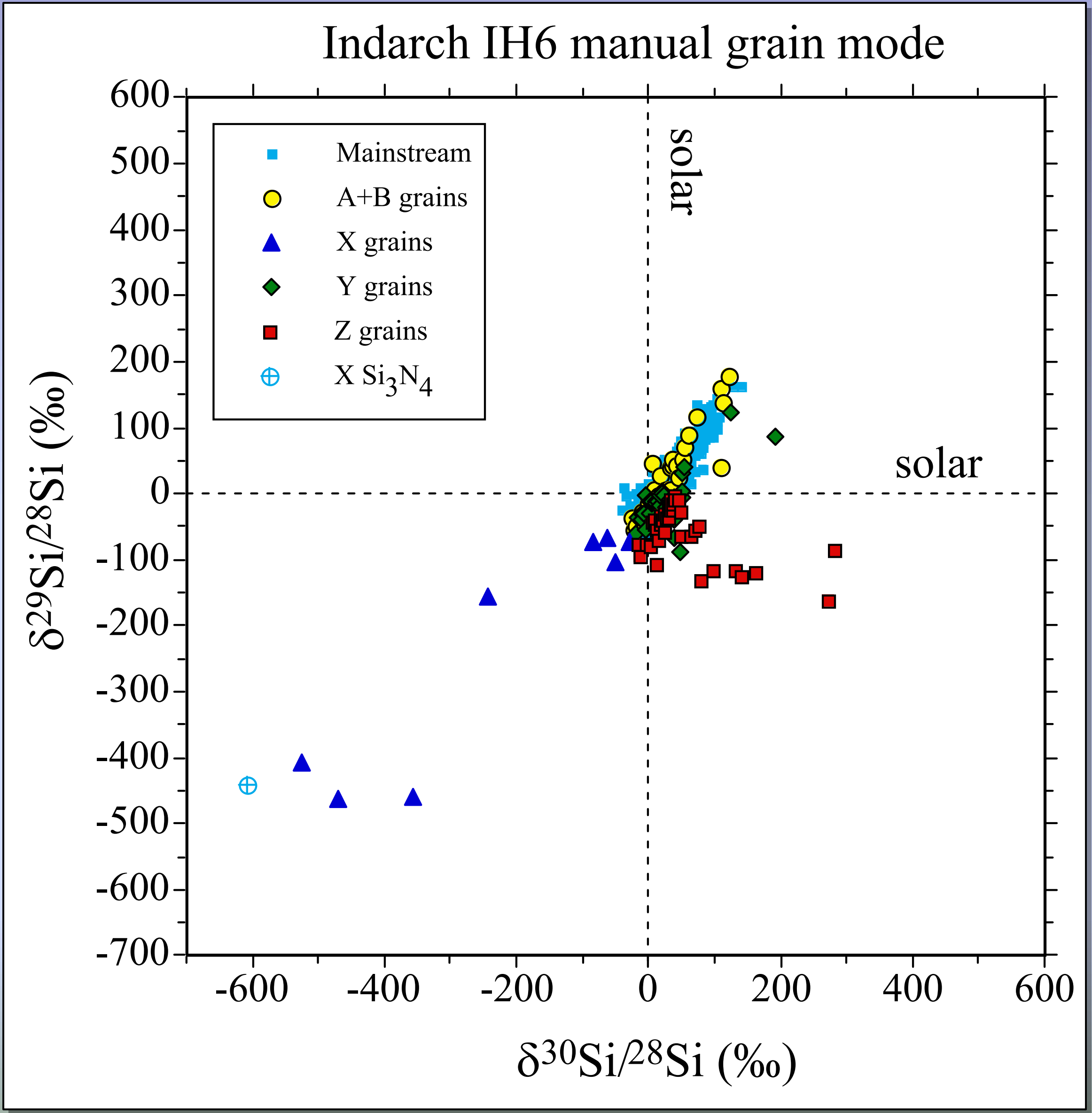


SiC grains from Indarch meteorite

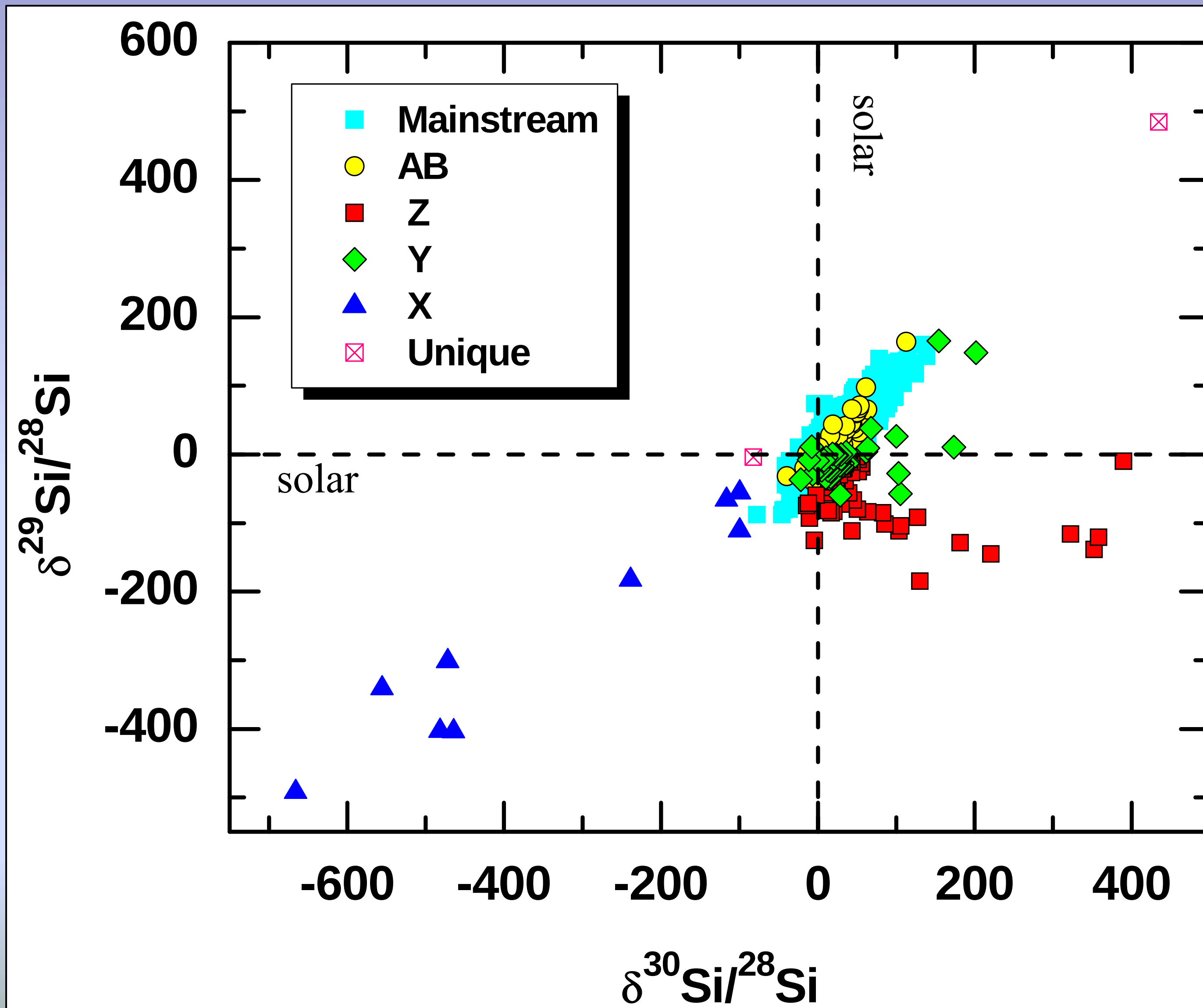
Grain size: 0.25-0.45 μm

As size goes ↓, number of Z grains goes ↑

One week of manual measurements



One week of automatic measurements



Automatic vs Manual*

Group	Number		Percentage		Criterion
Mainstream	670	437	82.3	79.6	$^{12}C/^{13}C$=10 - 100 & $\delta^{29}Si \approx 1.4\ \delta^{30}Si$
AB	39	27	4.8	4.9	$^{12}C/^{13}C<10$
X	9	8	1.1	1.5	$^{12}C/^{13}C>100$
Y	40	35	4.9	6.4	$\delta^{29}Si$ or $\delta^{30}Si<-100$‰
Z	54	42	6.6	7.6	$\delta^{29}Si<0$ & $\delta^{30}Si$ 25‰ from MS line
Unique	2	0	0.2	0	Don’t fit into well defined groups
Total	814	549			Instrument time: Roughly Equal (1 week) Man hours: Auto << Manual

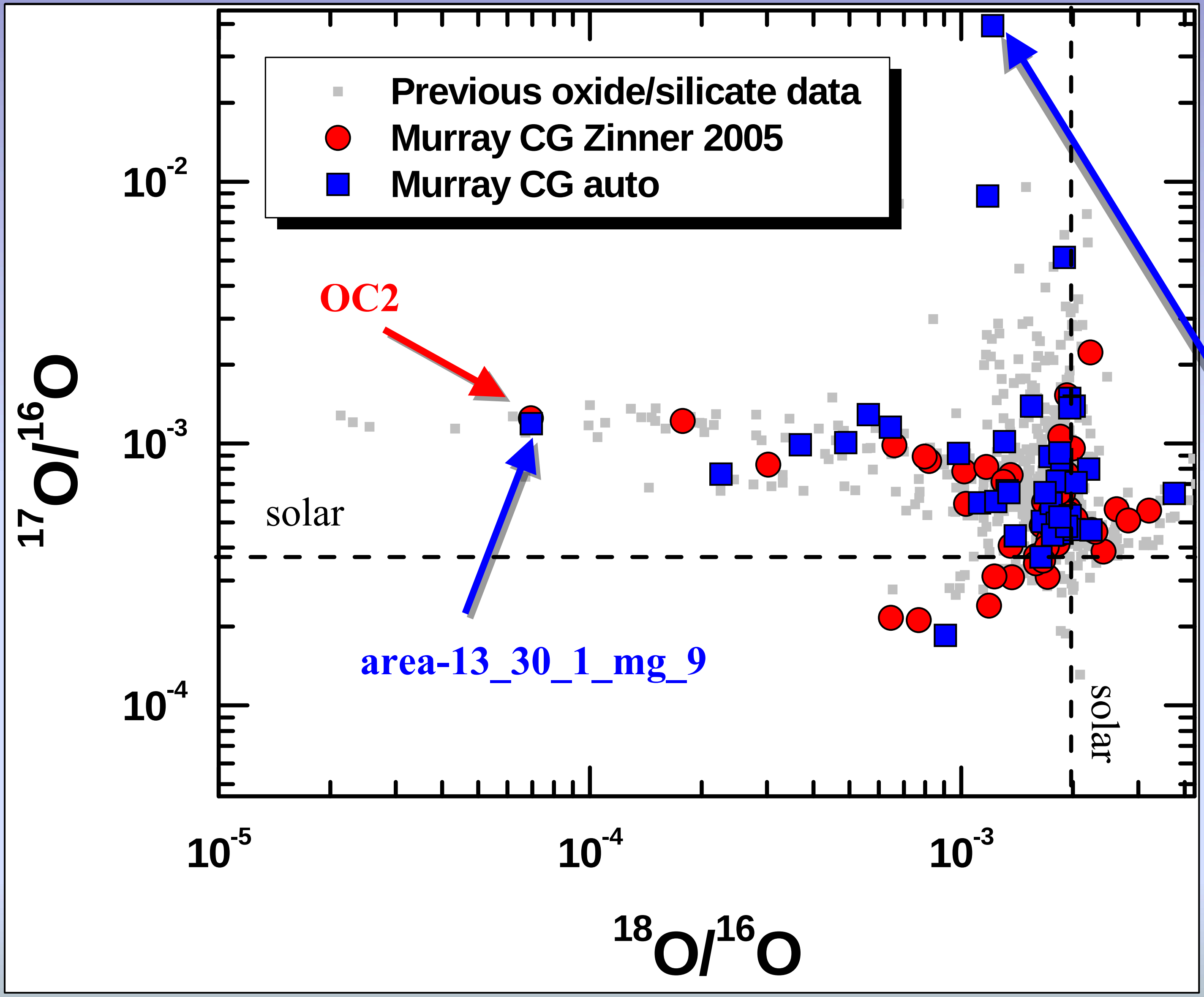
* Zinner et al, GCA, 71, 4786-4813, 2007

Spinel grains from Murray meteorite

Spinel ($MgAl_2O_4$) rich fraction

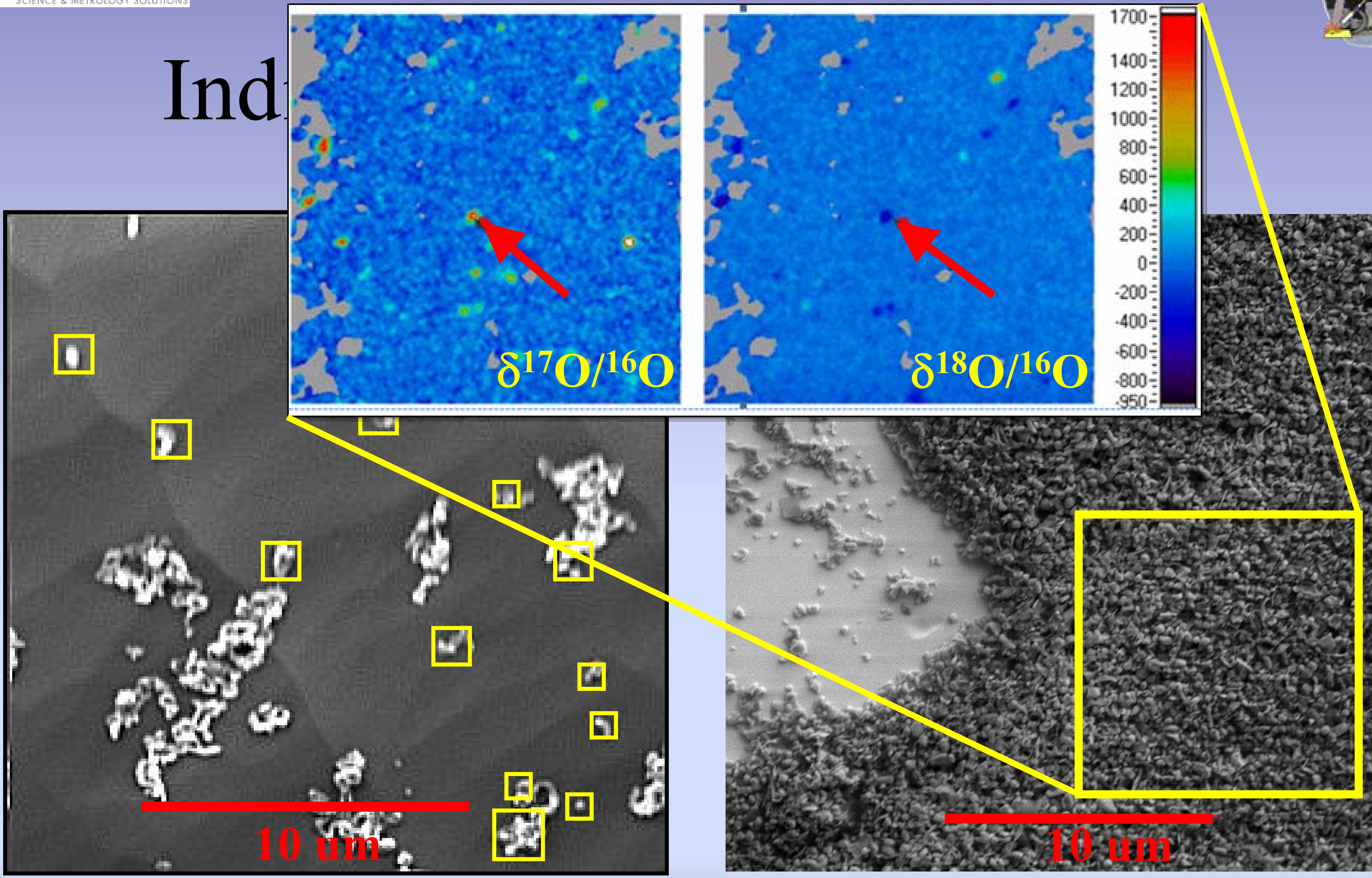
Average grain size: 0.45 μm

As size goes ↓, number of presolar spinel goes ↑



Previous oxide/silicate data
Murray CG Zinner 2005
Murray CG auto
OC2
area-13_30_1_mg_9
WOWSA!
solar
solar
10^{-2}
10^{-3}
10^{-4}
10^{-5}
10^{-4}
10^{-3}
$^{17}O/^{16}O$
$^{18}O/^{16}O$

Ind



NanoSIMS SE

FE-SEM

Advantages

No operator fatigue

Customizable: integrated into instrument software

Can be used for other applications!

Conclusion

Absent new hardware, software is key

Automated, high-volume measurements required

User's constant presence unnecessary

Fully integrated into Cameca instrument software

Drawbacks: Time consuming

	Presputter	2 min
	Acquire image (400μm^2)	5 min
+	HMR measurement	2 min (x 10)
	Time per area	27min
	Number of areas	x 144 (225 x 225 μm)

Total time: 2.7 days!