Atom probe tomography and correlative microscopy: Key techniques for future planetary science studies

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Our Galaxy is vast and awe-inspiring. The stars, planets, and our sun capture our imagination as children. For many of us, that wonder never ceases. It continues to inspire us throughout our careers and prompts us to question the evolution of our Solar System, to question what our place is within it, and how we may maintain longevity in a relatively volatile environment. To answer these questions planetary scientists turn to the study of extraterrestrial material. They analyze meteorites, impact craters, and materials returned by sample return missions for the evidence of events that are known to induce crystallographic and/or elemental changes, or for evidence of extraterrestrial isotopic abundances that point to the age and the original source of the material. Through these studies, we can constrain timelines of events that have occurred throughout the Solar System's extensive history. Recently, atom probe tomography (APT) has been applied to the study of these materials. APT in correlation with larger-scale analysis techniques has provided insights into isotopic ratios or nanoscale distribution of elements, enriching our knowledge, and minimizing uncertainties in the time frame of critical cosmic events. The continued use of correlative microscopy with APT for the study of planetary science, including studies of small amounts of pristine materials delivered to the Earth by exciting sample return missions, promises to provide key information into the history of our Solar System. Here, we highlight the implications of correlative microscopy with APT for the future pursuits of planetary science, we reflect on the groundbreaking research already achieved, the challenges that have been overcome to achieve these outcomes and the challenges yet to come.

Atom probe tomography: Extending the length scale studied in planetary science

The challenges of landing a mission on another planetary body, safely collecting and returning a sample to Earth, means that the amount of extraterrestrial material delivered to the Earth is typically less than a kilogram and can be as low as a few hundred 0.1-µm-sized particles.[1] The low mass of returned material means it is imperative that the maximum scientific insight is acquired from the minimum sample volume.[2] Correlative microanalysis can be used to explore these unique samples from centimeter scales down to individual atoms.[2]

Atom probe tomography (APT) addresses the need for maximum scientific information from minimum sample volume. APT is a powerful analysis technique capable of extracting 3D chemical, structural, and isotopic information from specimen volumes of $<0.01\ \mu m^3$,[3] making it ideally suited to exploring nano-atomic scale effects, such as space weathering[4] and water rock reactions in the Solar System.[5–10] A key feature of APT is the ability to target mineral phases that are too small for traditional chemical and isotopic measurement techniques, providing high-precision isotope ratio measurements from small sample volumes.[11–15] The high resolving

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doi:10.1557/s43577-022-00375-6

power and small sample volumes make APT an ideal analysis technique to maximize the science return from pristine sample return mission materials, as well as enhance research into meteorites and impact craters, providing a unique atomic-scale perspective of extraterrestrial materials.[16] Critical to producing meaningful results from APT is the use of larger-scale analysis techniques, such as secondary ion mass spectrometry (SIMS), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and even remote sensing techniques.[17] Such techniques provide context for the atom probe results and are critical to targeting precise locations for optimizing the scientific outcome from nanoscale APT analysis volumes.

Here, we outline past and future sample return missions, current technological developments paving the way for improved analysis of minerals, and review recent high-profile results in planetary science obtained using a combination of APT and correlative microscopy techniques.

Returning pristine material from the Solar System

Planetary science and space exploration are entering a second golden age. In particular, several exciting sample return missions are underway, with spacecraft sent out into the Solar System to collect and deliver pristine rocky materials from the surface of planetary, cometary, and asteroidal bodies for study in laboratories on Earth. The direct return of samples from the surface of these bodies has three massive benefits when compared to the record of meteorite finds on planet Earth.[18,19] (1) Many minerals in extraterrestrial rocks are out of equilibrium in Earth's atmosphere and react rapidly. Carefully curated sample return mission materials preserve minerals that are typically destroyed in Earth's environment. (2) The sampling location on the planetary body is known, providing crucial geological context to the extraterrestrial rock record. (3) Analysis of extraterrestrial materials from known locations on known planetary bodies allows us to confidently link the meteorite groups we have on Earth to asteroid types in space, vastly augmenting the interpretive potential of the existing meteorite record.

The Moon has been the target of the majority of sample return missions (**Figure 1**). The Apollo program itself successfully delivered 382 kg of Lunar regolith and rock between 1969 and 1972,[20,21] swiftly followed by the Soviet Union's Lunar 16, 20, and 24 missions, which returned 0.326 kg.[22] Recently, China's Chang'e 5 mission returned 1.73 kg of material from the Northern Oceanus Procellarum on the Moon.[23] A key result obtained from these samples is that the Moon is not as dry as previously thought.[24]

Sample return missions have also been used to sample the Sun. In 2004, the Genesis mission returned samples of the Sun's solar wind,[25] a stream of predominantly H and He ions from the Sun that penetrate a few 100 nm into natural and synthetic materials.[26] The returned samples provided new insights into the composition of our star's Solar Wind, revealing that it had more ^{16}O and a lower $^{15}N/^{14}N$ ratio than most other Solar System materials.[27,28]

NASA's Stardust mission delivered the first samples from a comet by collecting dust samples from the coma of comet Wild 2, delivering ~1 g of cometary particles to Earth in 2006.[29] Analysis revealed the presence of glycine, a fundamental chemical building block for life within materials.[30] Also observed was the presence of refractory minerals that formed near the Sun, indicating that material was transferred across the entire protoplanetary disk in the early Solar System.[31]

The Japanese Space Agency (JAXA's) Hayabusa mission was the first to successfully sample the surface of an asteroid.[1] The Hayabusa probe visited the S-type asteroid Itokawa and successfully returned >1534 particles from the asteroid's surface.[32] Itokawa regolith is petrologically and geochemically similar to the L chondrite meteorite group and provided the first direct link between an asteroid and meteorite.[33] This success was followed by JAXA's Hayabusa2 mission to the C-type asteroid Ryugu, returning 5.4 g of regolith samples from two landing sites.[34] The results of the initial analysis of Ryugu regolith reveal that it is most similar to the CI carbonaceous chondrites,[34] which are exceedingly rare in the meteorite record, as well as being the most chemically primitive and highly aqueously altered samples.[35]

Sample return missions are not always to a planetary body or asteroid. Near earth space contains small interplanetary dust particles and micrometeorites. These extraterrestrial dust grains have been collected, typically using aerogel, by the Orbital Debris Collection experiment on the Mir Space Station, the Tanpopo mission, and the Materials International Space Station Experiment on the International Space Station, the Long Duration Exposure Facility (LDEF).[36] Dust grain samples have also been collected by regular stratospheric flights, some of which have targeted major meteor showers and cometary dust trails.[37,38] These missions have enabled the calculation of the extraterrestrial dust flux to the Earth.[39] In addition, returned satellite components such as the Hubble Space Telescope and the Mir Space Station have themselves become inadvertent sample return missions as they have been bombarded by micrometeorites and dust during their time in space.[40]

Future sample return missions are currently underway or are planned including NASA's OSIRIS-REx mission, which is due to deliver an estimated 0.4–1 kg of the C-type asteroid Bennu in 2023.[41] The planned CAESAR mission is set to return 80 g from Comet 67 P.[42] Other missions include CNSA's Chang'e 6 mission to the Lunar surface in 2024,[43] the ZhangHe mission to asteroid Kamo'oalewa, JAXA's MMX mission to Mars' moon Phobos and plans for the OKEANOS mission to return samples to Earth from Jupiter's Trojan asteroids.[42] Finally, there is also an international collaborative effort to return samples from Mars in a Mars Sample Return (MSR) campaign of missions that begin with NASA's Perseverance rover that landed in Jezero crater on Mars in

2021, and will drill and cache rock cores to be collected by a future fetch rover mission.[8] MSR aims to deliver 0.5 kg of the red planet to the blue planet in the early 2030s.[8] This pristine Martian material has the potential to deliver a step change in our understanding of the evolution of Mars, other planets, and extraterrestrial habitability.[8]

New developments in correlative microscopy techniques for APT sample preparation

The precious nature of planetary samples requires high-precision targeting of features. The most common means of capturing a feature of interest within the analyzable volume of an atom probe needle tip, <100 nm radius, is to use the focused ion beam (FIB) "lift-out" technique.[44] With the aid of correlative analysis tools, features such as grain boundaries, nanoscale precipitates, and dislocations can be located. For example, **Figure 2**a identifies four microscopy techniques that are required to identify and capture potassium (K)-rich feather-like features within the atom probe data: (1) Scanning electron microscopy is used to distinguish between three minerals, olivine (Ol), plagioclase (Pl) and troilite (Tr); (2) energy-dispersive x-ray spectroscopy (EDS) provides low-resolution information as to the distribution of potassium (K) within the plagioclase; (3) electron backscatter diffraction (EBSD) provides further information as to the crystal structure within the minerals, such as grain boundaries and the presence of twinning; (4) finally, FIB–time of flight-SIMS (FIB–ToF-SIMS) identifies a depletion of K at twin boundaries and the presence of feather-like enrichment of K extending from certain grain boundaries. The junction between these feather-like features and the grain boundary was then targeted using a newly defined "button" method. The button method uses Pt targets, or "buttons," to mark the exact location of the feature of interest, providing a fiducial marker for enhanced accuracy annular milling.[45] After depositing the button marker the sample is protected by a Pt deposition layer before being lifted out and placed on a pre-prepared microtip, ready to be prepared into a needle-shaped sample by annular milling.[46]

The standard liftout technique provides an area of approximately 2×2 μm^2 from which the final tip is prepared. This is a relatively large area compared to the feature of interest, which may only be on the order of 10 s of nm in size, such as nanoscale precipitates or the feather-like K-rich features in Figure 2a. Furthermore, the feature of interest may not always be central to the liftout post.[45] Using the original liftout method the feature of interest is often targeted using contrast between matrix and precipitate, or matrix and grain boundary; however, contrast is not always detectable and successfully capturing the feature of interest within the atom probe tip can be limited to luck. Using the button method, annular milling is focused on the button and therefore even if the feature of interest is not central to the liftout post, it can still be targeted. The atom probe map in Figure 2a shows the successful application of the button technique to the targeting of a K-rich feather-like structure. In this instance, placement of the button has not only improved the accuracy of the liftout process, but has also provided a precise correlation between the atom probe tip and the data from larger-scale analysis techniques: SEM, EDS, EBSD, and FIB–ToF-SIMS.

When preparing atom probe tips from planetary samples, it is not only necessary to improve the feature targeting accuracy but also to reduce the potential loss of material. The FIB liftout process is known to be difficult to master and has a high probability of sample loss during the liftout and placement processes. As such, alternative methods are continually being investigated to remove the need for the liftout process.[49,50] The "crater-method" (Figure 2b), which takes advantage of the substantially higher milling rates of modern plasma-FIBs, is one such method.[47] However, a significant amount of material is removed using this method and the technique may not be suitable for precious samples. A very recent development in non-FIB liftout sample preparation methods for APT is the use of ion-induced bending (IIB) (Figure 2c).[48] IIB provides a pathway to prepare atom probe tips with a planar feature, such as a grain boundary, along the vertical axis of the tip, reducing the probability of early fracture and maintaining the feature of interest within the APT field of view for longer, providing larger quantities of relevant data. Currently, this technique has only been attempted on ductile material; however, the results from cryogenic temperature experiments at –162°C[48] indicate that this technique may also be applicable to more brittle samples, such as typically analyzed in planetary science.

APT data analysis challenges

For minerals, APT is able to chemically and spatially identify trace elements, as well as to quantify isotopic abundances. However, analysis can be hindered by high levels of background noise, poor quality data, and/or overlapping peaks.[11] For example, a recent round robin study of the GJ1 zircon reference material yielded a wide range of reported compositions.[51] Variations in reporting and ranging make direct comparisons between studies difficult, encouraging community-driven efforts to standardize reporting of background selection, ranging, and acquisition parameters[52] as well as peak deconvolution for isotopic species of interest.[12]

In species such as feldspar, the ability to extract meaningful isotopic ratios (such as Pb/Pb, Ar/Ar, Rb/Sr, or Sm/Nd) from the mass-to-charge-spectra is complicated by a wide array of trace elements that lead to peak overlaps at most of the target masses (e.g., $^{40}Ar^{++}$ would be masked by $^{20}Ca^{+}$).[53] In zircon ($ZrSiO_4$), the presence of silica leads to the production of multiple SiO peaks that can mask certain target masses, such as $Si_2O_3^{+}$ masking $^{208}Pb^{++}$, and $^{96}Zr^{2+}$ masking $^{48}Ti^{+}$, preventing zircon thermometry.[14] In Ca-rich materials, such as apatite, large tails in the mass spectra after Ca peaks mask other species of interest such as Fe and Mn.[54] Along with issues of overlapping peaks and interference from background noise, isotope analysis, such as the study of C isotopic ratios, can also be influenced by instrument biases, such as undercounting of ^{12}C due to multi-ion detection events.[13]

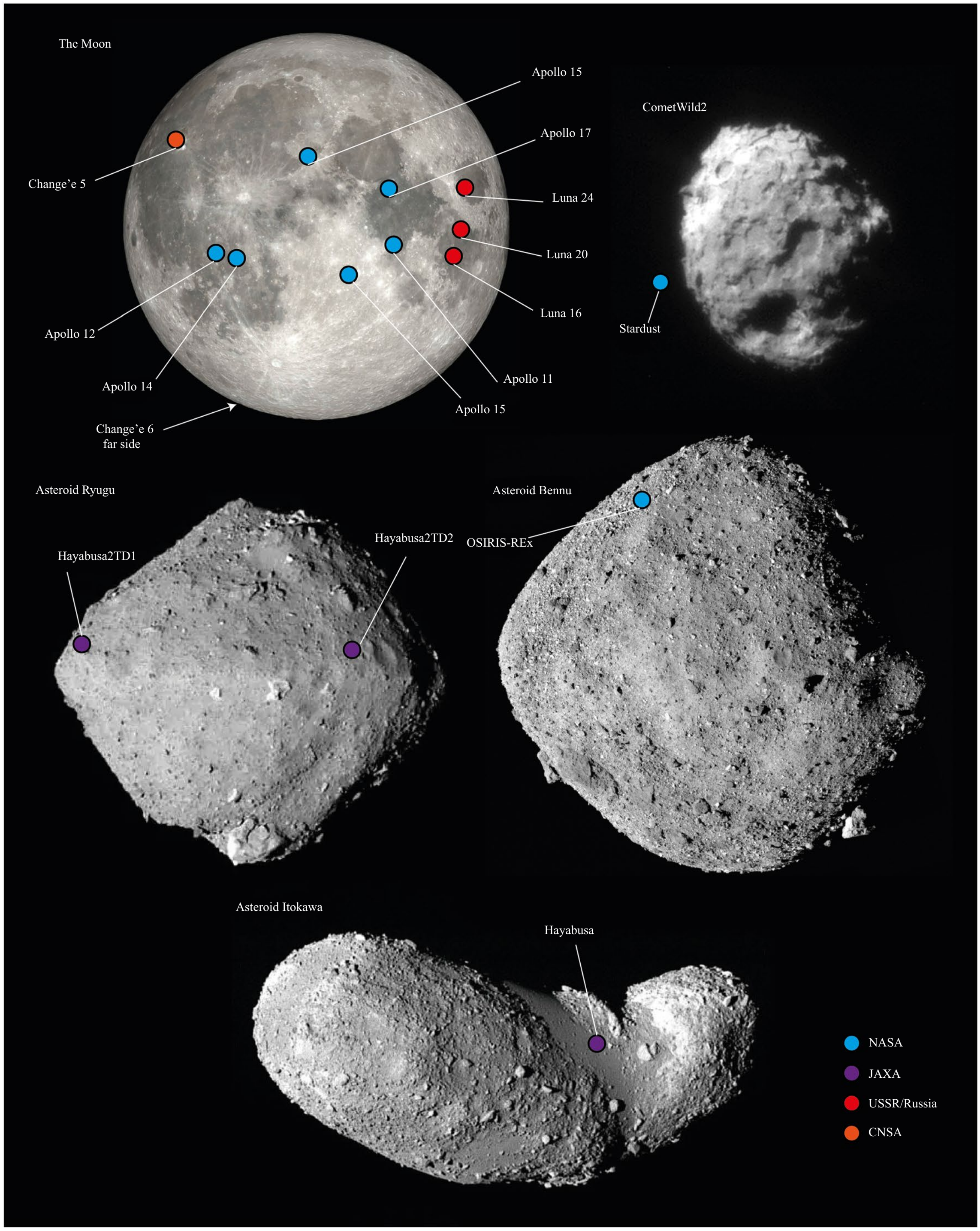

Figure 1. Images of the planetary bodies that have been or are intended to be sampled by sample return missions, including the Moon, Comet Wild 2, and asteroids Ryugu, Bennu, and Itokawa. Images from JAXA, NASA.

Despite these complications, careful reduction and correction of measured isotopic abundances can produce reliable results. For example, comparison of measured isotopic abundances and ratios with other techniques such as SIMS[14,55,56] or NanoSIMS[54] shows strong agreement between geoanalytical techniques, typically within 2σ counting statistics uncertainties (95% confidence interval) of the values calculated by APT.

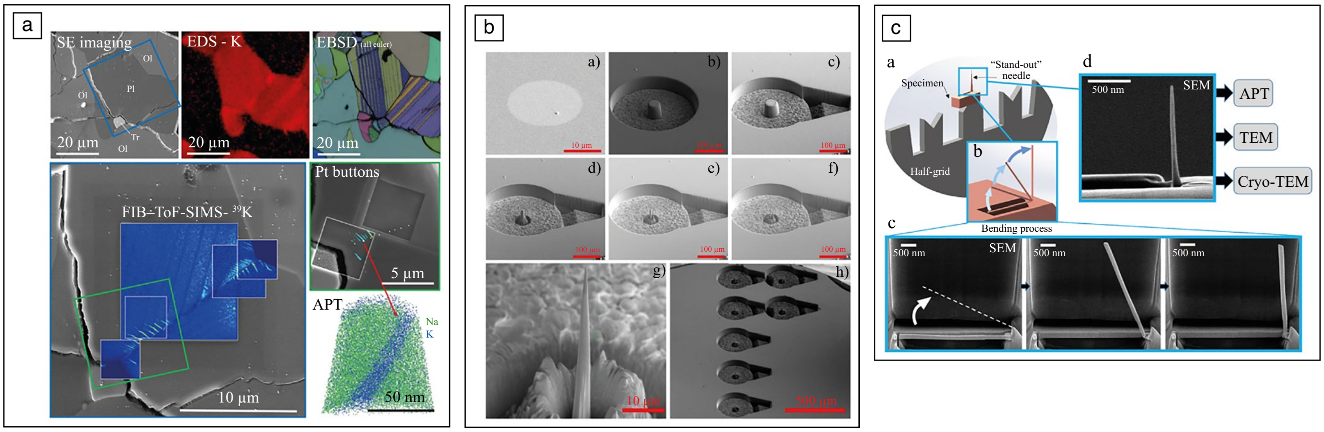

Figure 2. (a) Correlative microscopy workflow for targeting nanoscale features incorporating focused ion beam-time of flight-secondary ion mass spectrometry (FIB–ToF-SIMS) and the button targeting method. Image from Reference 45. (b) Preparing multiple atom probe tips using the new crater method. Reprinted from Reference 47. (c) Proposed ion beam bending method for the preparation of atom probe tips. Reprinted with permission from Reference 48. © 2022 American Chemical Society. EDS, energy-dispersive x-ray spectroscopy; EBSD, electron backscatter diffraction; APT, atom probe tomography; TEM, transmission electron microscopy; SEM, scanning electron microscopy.

For many meteoritic samples, the concentration of O and H species is of particular interest.[6,16] However, quantification of both O and H presents challenges in APT studies. H contamination within the analysis chamber results in background H signals that can be difficult to differentiate from native H.[6] Likewise, O is often underrepresented in APT data due to the development of neutral ions during field evaporation processes.[57] Furthermore, FIB sample preparation influences the presence of volatile species, such as O and H, within the samples prior to APT analysis.[58]

Addressing analysis challenges through new technology developments

A goal to address the aforementioned challenges is the development of atom probe instrumentation that enables the collection of significantly improved raw data, negating the need for intricate validation methods.[3] Cryo/vacuum transfer systems and a new H-free Ti analysis chamber have already been developed and have been used to address issues of H contamination[59–62] and provide scope for improved accuracy in the study of hydrous minerals. A proof of concept energy-position-sensitive detector has also recently been developed.[63] The incorporation of energy sensitivity to APT studies would facilitate the differentiation of overlapping peaks,[64] such as $^{40}Ar^{++}$ and $^{20}Ca^{+}$, enabling the calculation of more accurate isotopic ratios.

The release of a new generation of CAMECA atom probes, the LEAP6000 and the Invizo 6000, also present new opportunities for the acquisition of significantly improved raw data.[65,66] Deep UV lasers have been included to improve data quality and yield. Addressing concerns of peak identification and background noise is the integration of a synchronous voltage and laser pulsing system. Furthermore, the Invizo 6000 incorporates two incident lasers to minimize preferential evaporation from a single side, as well as a new electrode that facilitates a much wider field of view, enabling the entire tip to be captured by the position-sensitive detector, increasing the volume of data collected from a single tip.

Application of correlative microscopy with APT to planetary studies

Correlative microscopy with APT has been successfully applied in a number of planetary science studies. Within these studies, APT has provided critical 3D information about segregation, diffusion, and isotopic ratios, beyond the resolution of other imaging techniques (**Figure 3**). Here, we provide an overview of studies enhanced by the addition of APT to existing microscopy techniques, and the implications for our understanding of solar disk evolution, planetary evolution and bombardment, and alteration and weathering.

Solar disk evolution

A powerful example of the application of correlative microscopy with APT for planetary science has been in aiding the search for the host phase of a pre-solar noble gas signature within meteoritic nanodiamonds.[13,67–69] Bulk geochemical analysis of nanodiamonds within chondritic meteorites revealed an anomalous Xe isotopic component suggesting that at least some nanodiamonds have a pre-solar origin.[67] To determine if a nanodiamond is pre-solar requires the measurement of nucleosynthetic C isotope signatures, which is challenging to measure as nanodiamonds are 2.7 nm in diameter on average.[70] Thus, extracting C isotope ratio measurements from an individual nanodiamond is only possible with APT. One hundred nanodiamonds have so far been prepared by FIB lift-out methods and measured by APT, all of which formed in our solar system. Given the small statistics and the likely abundance of pre-solar nanodiamonds being 1 in 10^5,[71] many more nanodiamonds need to be measured in order to find the pre-solar carrier phase representing the anomalous Xe isotope signal.

Correlative microscopy with APT has also been applied to the analysis of refractory metal nuggets (RMNs), revealing migration of particles early in our Solar System with implications for grain transfer in the protoplanetary disk.[72] RMNs are submicrometer metal alloys and are interpreted to form as one of the first solid phases in the Solar System during high temperature (>1800 K) condensation near the young Sun in the inner Solar System.[73–75] APT analysis of RMNs reveal that they contain trace to minor amounts of S.[72] Sulphur is not stable as a solid in the high-temperature region where RMNs would form. This suggests that RMNs must have been mixed into and out of the cooler outer portions of the Solar System before mixing back into the high-temperature region to become incorporated into Ca–Al-rich inclusions.[72] However, such efficient mixing of particles in the disk would effectively result in all meteoritic materials experiencing a high-temperature history, which is not the case, thus the region where RMNs are forming and mixing must be isolated by a barrier or gap from the remainder of the protoplanetary disk[72] such as are observed by the ALMA telescope in protoplanetary disks forming in our Galaxy today.[76]

Planetary evolution and bombardment

Constraining the timing, duration, and intensity of asteroid and cometary bombardment is critical to understanding the evolution of planetary crusts, and potentially the delivery, extinction, and sterilization of life in the early solar system.[77] Isotopic and structural evidence for these events are recorded within ancient (>4 billion year old) meteorites from the Moon, the asteroid belt,and rare Martian breccias, though primary mineralogical evidence is overprinted by later metamorphism and shock ejection, significantly complicating interpretation.[78]

Correlative microscopy with APT has provided unique new ways to analyze nanoscale evidence of the earliest planetary processes. Within lunar meteorite Northwest Africa (NWA) 3163, grains of baddeleyite (monoclinic ZrO_2) on the order of <10 µm in size record evidence of crystallization and impact deformation within nanoscale domains. Correlated EBSD and SIMS with APT have facilitated targeted measurement of the U–Th–Pb systematics

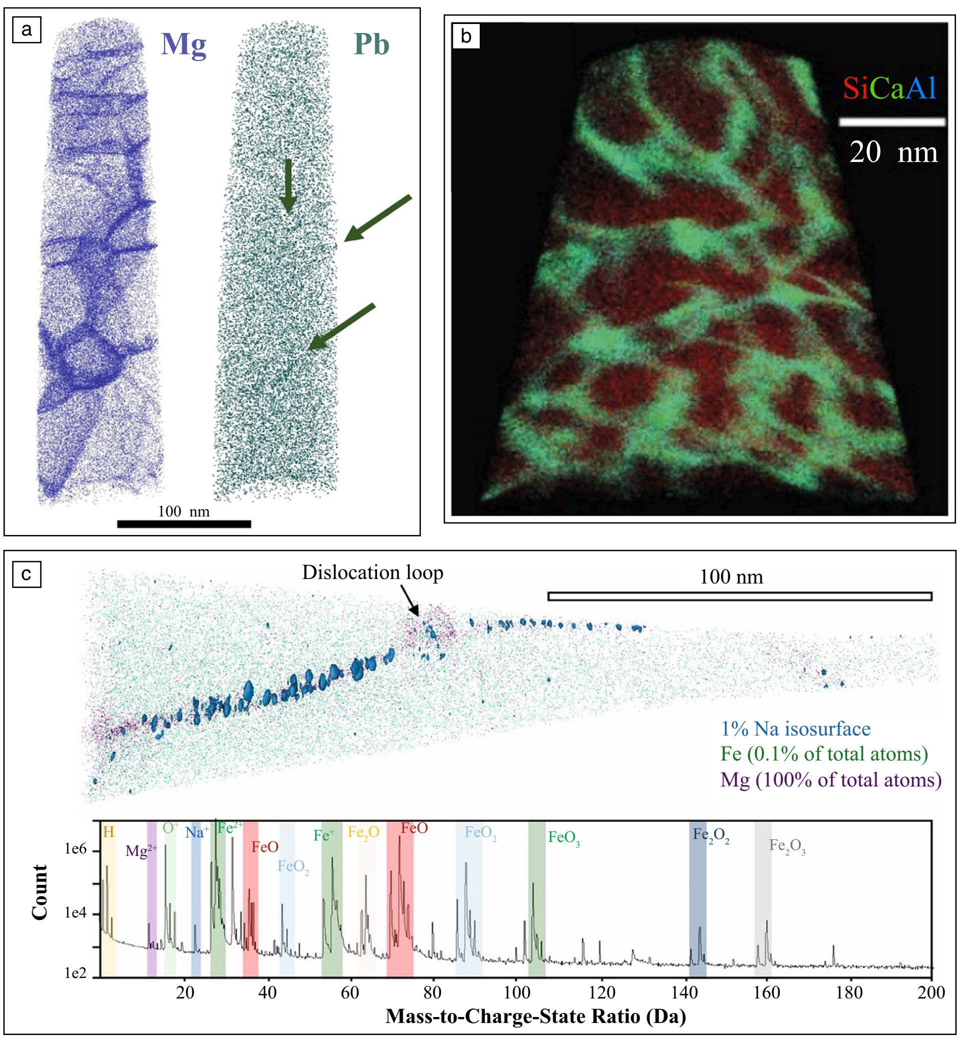

Figure 3. (a) Evidence of Mg and Pb segregation to grain boundaries in shocked Lunar apatite enabled a new age to be placed on the Serenitatis crater.[17] (b) Identification of a heterogeneous material at the nanometer scale, where other techniques such as transmission electron microscopy and scanning electron microscopy indicate a homogeneous structure. 1 × 5 nm tubular structures of Ca–Al silicate within a silicate matrix from a Martian meteorite.[2] (c) Evidence of Mg segregation to magnetite framboid grain boundary and Mg-, Na-, and Mn-rich dislocation loops in the Tagish Lake meteorite provides evidence of an alkaline formation liquid.[7]

of these crystal domains to reveal discrete age reservoirs recording the crystallization (4.3 billion years (Gyr) ago) and impact deformation (2.1 Gyr ago) of the grain.[53] Such ages would become homogenized when analyzed by other techniques, preventing a robust measurement for the age of crustal formation recorded by the meteorite.

In addition to providing a means to directly measure mineral ages, correlative microscopy with APT also provides evidence for the mobilization and diffusion of trace element species that can aid chronological interpretation. In ancient Martian breccias, pristine zircon and baddeleyite (as revealed by EBSD and APT) place further confidence that the 4.5 Gyr ago ages yielded by the minerals record magmatism, and not impact deformation, on Mars.[79] Reanalysis of returned Apollo samples provides similar insights, with the diffusion of incompatible trace elements such as Mg to grain boundaries in apatite (Figure 3a), providing new evidence that the 4.2 Gyr age recorded by the grain represents a large impact event, rather than magmatic activity.[17] This observation has allowed a new age to be placed on the adjacent Serenitatis crater, supporting recalibration of the crater counting record for the inner Solar System.[17]

Within Apollo soils (Apollo 16, sample 61,500), silicon metal and iron silicides are indicative of highly reduced conditions on the lunar surface. The combination of low kiloelectron volt electron probe microanalysis and APT has revealed an intrinsic link between reduced Fe metal and C in Fe–Si samples with a composition close to $(Fe,Ni)_3Si$.[80] The low oxygen fugacity has previously been ascribed to O loss from the lunar surface, but these nanoscale observations provide the first evidence for high-temperature mechanisms driven by a meteorite impact. In concert, these studies reveal the strength of correlative microscopy with APT to provide new insights into historic samples and provide a basis for future analyses of returned samples.

Alteration and weathering

Space weathering is common on all airless bodies, with soil and regolith on planetary surfaces being exposed to micrometeorite bombardment, solar wind implantation, and cosmic and solar rays.[4] These effects lead to shallow (<100 nm) structural and chemical changes in the constituent grains of the regolith, which can represent a significant total mass for soils of small grain size.[6] Currently, the products of these weathering processes are poorly understood.

APT has already provided numerous new insights into the interaction between mineralogical materials and space weathering phenomena, particularly in lunar soils and regolith on asteroid parent bodies. Initial studies utilized the atom probe field-ion microscope (APFIM) to study space weathering in the Santa Catharina iron meteorite, revealing ca. 12-nm-diameter precipitates of enriched nickel within the metallic matrix.[81] Further work, utilizing correlated APT and TEM analysis of the Bristol IVA iron meteorite, highlighted the distribution of Cu, Co, P, Cr between kamacite and tetrataenite, placing empirical new constraints on the portioning of trace elements during martensitic phase transformation in iron meteorites, as well as reinforcing the presence of Ni-enriched precipitates within constrained domains of the iron matrix.[82]

Constraining the mechanisms for water delivery to the inner solar system is critical to understanding the origin and evolution of life, as well as developing our understanding of planetesimal mobility within the nascent protoplanetary disk. Precious little evidence remains for the extent and composition of water from 4.5 Gyr ago, with many reservoirs reacting with surrounding mineralogical material to produce alteration products or reaction rims, often on the submicrometer scale, as identified by TEM analysis of the sample surface layer. Careful ranging of H and hydride peaks (as well as other volatile species such as Cl and F) from atom probe data in tips extracted from the surface layer provides first-order insights into the distribution of water in surrounding minerals, providing a truly unique insight into the localized alteration produced by water reservoirs throughout the history of the Solar System.[6]

In the 4.5 Gyr old carbonaceous chondrite Tagish Lake, magnetite framboids previously assumed to form in droplets of solution on the parent body, record the composition of the residual mineralizing fluid within <5-nm-wide amorphous layers between the crystals.[7] Measurement of these domains by APT reveal localized clusters of Na within the amorphous layer, and Ca- and Na-enriched surficial coatings in the adjacent crystal faces (Figure 3c). Together, these observations provide evidence that the formation liquid was alkaline ($pH > 7$), reconciling previous models for the composition and acidity of water on the Tagish Lake parent body.

Conclusion

Advances to our knowledge of our solar system have occurred in waves, generally related to technical advancements or the return of substantial quantities of extraterrestrial material. We currently sit on the precipice of a potential new leap in our knowledge, due to the planning of multiple sample return missions.

Coinciding with the return of these samples are new developments in microanalysis techniques. In particular, the use of other techniques in combination with APT (correlative microscopy) enables targeted nanoscale studies of scientifically significant materials. APT has been shown to resolve nanoscale variations of planetary significance, robustly measuring composition, trace element composition, and isotopic variations, including those important for chronology. Combining APT with larger-scale analysis techniques enables maximization of science yield from returned samples. Forthcoming developments in APT such as energy-sensitive detectors, lower background H chambers, and the application of DUV lasers could again present a step advance in APT's application to planetary materials.

Funding

Open access funding enabled and organized by CAUL and its Member Institutions.

Conflict of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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