Long-term Proposal Report 1
Energy Scanning X-ray Diffraction Study of Extraterrestrial Materials Using Synchrotron Radiation

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Introduction

In order to understand the birth and early evolution of the solar system, it is essential to analyze extraterrestrial materials such as meteorites, interplanetary dust particles (IDP), lunar samples, cometary dust, and the samples of primitive asteroids such as Itokawa (which was sampled by JAXA's Hayabusa spacecraft). Because many of these samples were formed under extreme conditions quite different from the present earth, they often contain interesting minerals rarely found on the earth, but which are critical indicators of physico-chemical conditions in the early solar system. However, these extraterrestrial samples are usually available only in sub-microgram quantities, which makes it difficult to fully characterize these rare minerals by standard techniques. The analysis of extraterrestrial materials has recently focused on chemical characteristics such as chemical and isotope compositions, and unfortunately, crystallographic data of these extraterrestrial materials are lacking in many cases. This situation arises from the fact that these rare minerals are small and it is difficult to obtain diffraction data. However, such rare and small minerals carry critical and unique records of formation conditions in the early solar system, and the chemical and physical evolution of this most primitive material into protoplanets which eventually evolved into the planets. Whereas the initial formation of solid material in the solar system involved fairly simple gas-solid reactions, the resultant dust grains were almost universally altered through a combination of heat and pressure (thermal metamorphism), impact shock, and aqueous alteration (from liquid water that resulted from heating of water ice). Thus, we require a complete understanding of thermal, shock and aqueous alteration history. This goal is only achievable through detailed mineralogic characterization of surviving extraterrestrial materials. For example, some minerals can be present as one of several polymorphs formed at particular P-T conditions, and thus the particular crystal structure assumed by the phase is a record of the particular pressure and thermal history of that mineral. Thus it is essential that the crystal structures of these phases should be adequately characterized so that the thermal and pressure conditions can be properly "read". In understanding the aqueous alteration of primitive materials, we are also learning in what form water and organics, the seeds of life, arrived at Earth and other terrestrial planets. Since no one protoplanetary body records the full early history of the solar system, we must read the record in a wide cross section of available materials.

In our project the most primitive materials were represented by comet samples, which include chondritic interplanetary dust particles, the comet Wild 2 samples retuned by NASA's Stardust spacecraft, and meteorites that sample C- and S-type asteroid regolith. Metamorphosed and shocked materials are represented by the ordinary chondrite meteorites, some chondritic interplanetary dust particles, and the asteroid Itokawa samples collected by JAXA's Hayabusa spacecraft. The aqueously altered samples are represented by the carbonaceous chondrite meteorites and the salt from two ordinary chondrites which we were fortunate to have recognized a decade ago, and carefully preserved for this current investigation.

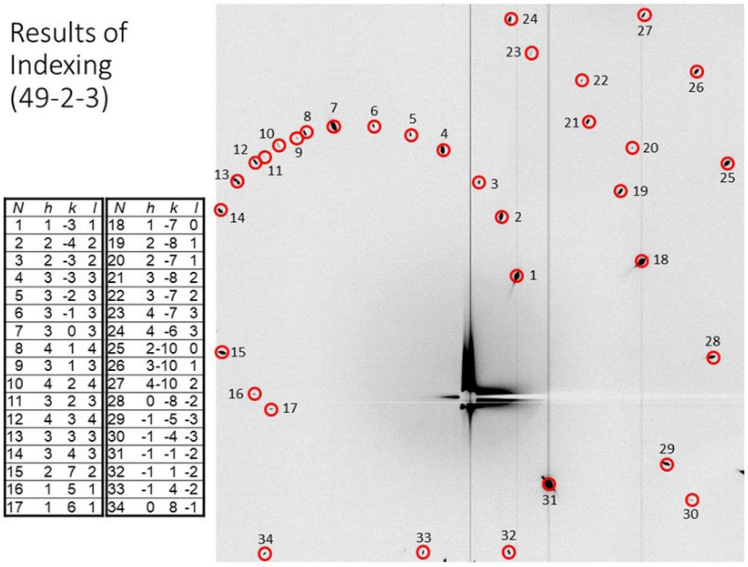
There were three main goals of our research. Goal 1: Characterizing the crystal structures of minerals in the primitive asteroid and comet regolith samples – reveals formation conditions and early solar system small body evolution. For this work we analyzed the following samples: Carbonaceous chondrite meteorites, Asteroid Itokawa samples from the Hayabusa Mission, and Comet Wild 2 samples from the Stardust Mission. Goal 2: Characterizing the effects of impact shock on asteroid and comet regolith samples, to reveal how fluids and solids migrated through the early solar system. For this work we analyzed the following samples: Carbonaceous chondrite meteorites, and the Asteroid Itokawa samples. Goal 3: Determining the origin and early history of aqueous fluids in the early solar system. For this work we analyzed the following samples: Water bearing primitive meteorites, and hydrovolcanic products preserved within two very special ordinary chondrite meteorites – Zag and Monahans.

Here are the investigations we made during the proposal runs:

1. We analyzed the crystal structures and/or deformation of pyroxene, olivine and albite crystals in regolith particles from asteroid Itokawa, returned by JAXA's Hayabusa spacecraft.
2. Structures of deformed phases in chondritic interplanetary dust particles.
3. Crystal structure of olivine crystals replacing matrix phyllosilicates, and associated sulfides in carbonaceous chondrite meteorites.
4. Crystal structures of carbon crystals in two ordinary chondrite meteorites, due apparently to repeated parent body shock metamorphic events, possibly related to the impact that formed earth's moon.
5. Structures of shock-deformed olivine and calcium phosphates in five ordinary chondrite meteorites.
6. Measurement of cell dimensions of olivine in the coarse-grained terminal particles of coma grains returned from comet Wild 2 by NASA's Stardust spacecraft.
7. Crystal structure of calcium carbonates from the meteor crater in Arizona.
8. Crystal structures of mineral grains separated from within salt crystals found in an ordinary chondrite meteorites (Zag and Monahans), which must come from the mantle of a hydrovolcanically-active early solar system body (possibly Ceres).

X-ray Diffraction at SPring-8

We have been working for the past three years on synchrotron radiation X-ray diffraction (SXRD) studies at SPring-8 beamline BL37XU (Proposal 2014B0113 - 2017A0113), employing a micro-beam diameter as small as 1 μm. SXRD is most useful when combined with other analytical techniques such as synchrotron X-ray fluorescence (SXRF) and synchrotron based X-ray computed microtomography (SXRCT), determining chemical compositions and physical properties at the nano scale. We use a stationary sample method and polychromatic X-rays because the irradiated area of the sample is always the same and fixed, meaning that all diffraction spots occur from the same area of the sample. This aspect is essential for nanogram-sized samples. In beamline BL37XU an undulator is installed and its radiation is further monochromatized using a Si(111) double-crystal monochromator. The X-ray energy is automatically adjusted by changing the undulator gap and the angle of a monochromator. A Kirkpatrick and Baez mirror is situated upstream of the sample giving a beam size of 0.7(V) × 2(H) mm^2 at the sample position. Diffraction patterns are collected on a two-dimensional detector (CMOS Flat panel detector, Hamamatsu Photonics K.K.). The samples are attached to an XYZ-stage, and the target micro area in the sample was adjusted on the micro-beam position under an optical microscope. We applied energies from 30 to 20 keV (λ = 0.4133 - 0.6199 Å) at increments of 40 eV with each exposure time being 0.5 seconds. In total we used 72 shifts over the three-year period 2014 - 2017.



N	h	k	l	N	h	k	l
1	1	-3	1	18	1	-7	0
2	2	-4	2	19	2	-8	1
3	2	-3	2	20	2	-7	1
4	3	-3	3	21	3	-8	2
5	3	-2	3	22	3	-7	2
6	3	-1	3	23	4	-7	3
7	3	0	3	24	4	-6	3
8	4	1	4	25	2	-10	0
9	3	1	3	26	3	-10	1
10	4	2	4	27	4	-10	2
11	3	2	3	28	0	-8	-2
12	4	3	4	29	-1	-5	-3
13	3	3	3	30	-1	-4	-3
14	3	4	3	31	-1	-1	-2
15	2	7	2	32	-1	1	-2
16	1	5	1	33	-1	4	-2
17	1	6	1	34	0	8	-1

Figure 1 Indexed single crystal X-ray diffraction pattern of Hayabusa sample RA-QD02-0049-02.

Hayabusa Mission Samples from Asteroid Itokawa

We analyzed the crystal structure of pyroxene, olivine and albite crystals in 8 asteroid Itokawa particles returned to Earth by the Hayabusa spacecraft[1-10]. One research goal was to determine the thermal metamorphic peak equilibration temperature witnessed by the asteroid using the low-calcium pyroxene structure, and albite structures. Another critical goal was to understand the range of impact shocks experienced by the regolith of Itokawa after it reaccreted (Itokawa is a second generation asteroid, which formed, was disrupted, and then partially reaccreted). The results from our study of Itokawa samples will permit shock effects to be understood from similarly micron-sized grains which will soon be returned from asteroids Ryugu and Bennu[11,12] by the Hayabusa2 and OSIRIS-Rex spacecraft, respectively. The results from our study of Itokawa sample 49-1 are very interesting, since this particular sample was essentially unshocked, despite the fact that the vast majority of the returned Itokawa samples have a record of moderate shock deformation. Only continued investigation will reveal whether this one sample is unique, or a member of a previously unrecognized grain population in the Itokawa regolith. In general we found that individual, nanogram-sized Itokawa regolith grains recorded individual shock levels below that for the bulk regolith. This means that studies of the samples that will be returned by the Hayabusa2 and OSIRIS-Rex spacecraft will have to be carefully performed to avoid underestimating the shock history of the target asteroids.

We also invalidated an earlier report that impact shock changes the cell dimensions of olivine. See Figures 1 and 2. Analysis of Itokawa sample RA-QD02-0049-02 olivine, which had been moderately shocked, revealed that cell constants were not measurably changed by this process. Figure 1 shows the indexed single crystal X-ray diffraction pattern and Figure 2 shows the graphical comparison of the derived unit cell constants with the "ideal" ones calculated using Vegard's law[7-10].

Interplanetary Dust Particles (IDPs)

IDPs are asteroid and comet dust collected in Earth's stratosphere by a NASA program managed by Zolensky[1]. We attempted to make crystallographic analyses of silicates and

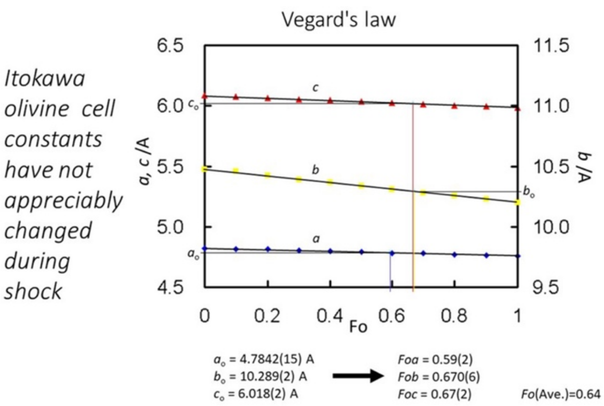


Figure 2 Graphical comparison of the derived unit cell constants of moderately-shocked olivine from Hayabusa sample RA-QD02-0049-02 with the "ideal" cell constants calculated using Vegard's law[7-10].

sulfide minerals in IDPs, especially those that are believed to drive from Comet Grigg-Skjellerup and Comet Giacobini-Zinner. Unfortunately, the samples proved to be mounted in such a manner that precluded analyses by this technique. We believe that our experience will permit future studies of these samples to be more successful.

C-Type Asteroid Regolith Samples

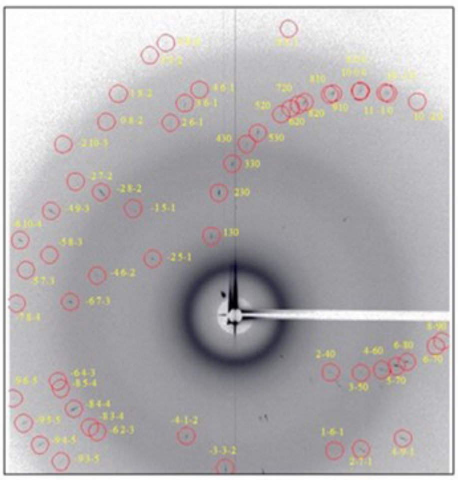
We made measurements of the crystal structures and cell parameters of secondary alteration minerals in unusual xenoliths in the Allende CV meteorite. The phases were revealed to be olivine, formed probably by the thermal degradation and recrystallization of phyllosilicates[6,14-22]. This material is now proposed to be the most abundant phase on asteroids Ryugu, the target of JAXA's Hayabusa2 mission.

We analyzed the crystal structures of secondary minerals in brecciated meteorites from C-complex asteroids, including Jbilet Winselwan, Sutter's Mill and most recently the Kaidun breccia of (principally) C and E chondrites[13,14]. The latter meteorite contains the best samples we have of hydrous, very reduced extraterrestrial materials, giving us a unique ability to determine the physico-chemical conditions of aqueous alteration on primitive asteroids. We determined that olivine crystals replaced matrix phyllosilicates in the Jbilet Winselwan, and Sutter's Mill meteorites, due apparently to parent body (asteroidal) shock metamorphism. Again, we expect to see these meteorites among the material that will be returned from Asteroid Ryugu by the Hayabusa2 Spacecraft, based on spectroscopy of that asteroid already performed by Hayabusa2 spectrometers.

We also collected crystallographic data on the mineral tochilinite, a coherently interstratified iron-nickel-magnesium sulfide hydroxide, which may be a common phase on Ryugu. Tochilinite has an incommensurate structure and is a useful cosmothermometer of early solar system, post-aqueous alteration metamorphism: Crystal structures of secondary minerals resulting from late-stage high-temperature aqueous alteration (common as small cognate clasts in otherwise hydrous meteorites) will inform us about the end-stages of C-complex asteroid metamorphism. These analyses are critical to understanding the results of the aforementioned missions to asteroids Ceres, Bennu, and Ryugu.

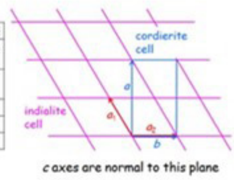
We continued our examination of the crystal structure of Fe sulfides in CM chondrites (a very abundant type of carbonaceous chondrite meteorites) with different cosmic ray space exposure ages[22,24]. Cosmic ray exposure ages date the amount of time a small sample has been traveling in space – i.e. the time since it was removed from its parent asteroid or comet. We have seen that the CM chondrite meteorites exposure ages that define at least three discreet groups, possibly indicating separate parent asteroids. We have found that these three groups have different phyllosilicate compositions, and this suggested that the sulfides present may also have different crystal structures.

We performed an XRD analysis of indialite in the Y-82094 ungrouped carbonaceous chondrite[17] (Figure 3).



XRD pattern of the cordierite-like phase in Al-rich chondrule in Y-82094 with indices (yellow numbers next to red circles) using the indialite crystal structure. Successful indexing by the indialite structure reveals that this phase is indialite rather than cordierite

	Indialite [6]	Cordierite [5]
Crystal system	Hexagonal	Orthorhombic
Space group	P6/mcc	Cccm
a (Å)	9.800(3)	17.079(3)
b (Å)		9.730(2)
c (Å)	9.345(3)	9.356(2)



Unit cell relationship between indialite and cordierite. Distortion of a hexagonal cell (indialite) gives an orthorhombic cell (cordierite).

Figure 3 Indexed single crystal XRD pattern of indialite, and derived unit cell relationship between this phase and the structurally related mineral cordierite[17].

Carbon in Asteroid Impact Melts

We determined the crystal structures of carbon crystals in two ordinary chondrite meteorites, apparently formed by repeated parent body shock metamorphic events[12,23,24]. We are hypothesizing that these samples derived from the impact that disrupted the proto-earth and created earth's moon. We determined that this particular carbon was a well-crystalline form of graphite. Now we are making O isotope measurements of these samples to further test our theory for their origin.

Shock Recorded by Ordinary Chondrite Meteorites Compared to Itokawa Regolith Grains

The Itokawa samples returned by the Hayabusa Spacecraft are almost identical to the LL ordinary chondrite meteorites[4-10,13,25,26]. However, during our earlier investigation (and as mentioned above) we found that there was a significant difference in the apparent shock history of the Itokawa samples as recovered by mineral optical properties and those recorded by the fine-scale crystal state of the samples as revealed by electron back-scattered diffraction (EBSD)[2] (Figure 4). To resolve the nature of this phenomenon we examined four LL chondrite meteorites, which record the same shock histories, in addition to seven Itokawa samples (which are not available in large numbers – only 5 may be allocated to an investigator at a time). We completed our examination the shocked olivine (the most abundant phase) in the Semarkona, Chainpur, Kilabo and Chelyabinsk meteorites, which belong to the same meteorite class as the asteroid Itokawa samples, and span the probable shock impact record of that asteroid's regolith since reaccretion. In the course of this work we examine both the unit cell parameters as well as aberrations in the diffraction spot shapes. We are now using the degree of spot broadening to develop a new shock index scale for particulates such as those returned from asteroid regoliths by the Hayabusa and Hayabusa2 missions.

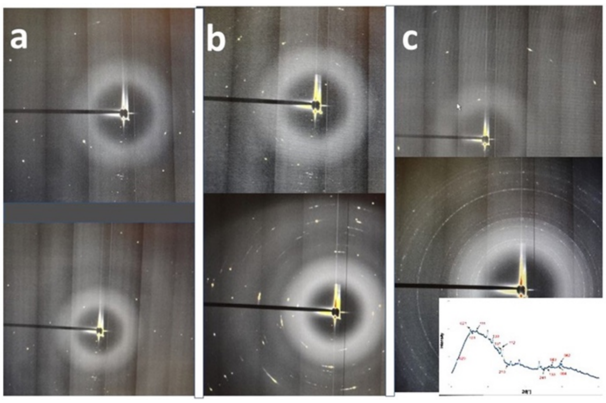


Figure 4 Single crystal XRD patterns of two olivine grains from each of (a) the Semarkona LL3 ordinary chondrite meteorite, with relatively condensed diffraction spots indicative of low degree (S2) of impact shock effects; (b) the Kilabo L6, ordinary chondrite meteorite, with diffuse diffraction spots indicative of a moderate degree (S3) of impact shock effects; and (c) the Chelyabinsk LL5 ordinary chondrite meteorite, with very diffuse (essentially polycrystalline) diffraction spots indicative of a relatively high degree (S4) of impact shock effects. The measured diffraction profile of one of these XRD patterns is shown at lower right.

We then collected similar diffraction patterns for seven Itokawa olivine grains: RA-QD02-0103, RB-CV-0024, RA-QD02-0138, RA-QD02-0028, RA-QD02-0179, RA-QD02-0127, and RA-QD02-0097 (Figure 5). Very surprisingly, the diffraction maxima peak sharpness of these patterns matched the low shock level Semarkona patterns (Figure 4a), rather than the moderate shock pattern of Kilabo olivine, despite abundant other evidence that Itokawa suffered moderate shock levels. This surprising result reveals that the single crystal diffraction patterns of regolith grains do *not* faithfully record the actual shock level experienced by an asteroid.

We also made an unsuccessful search for high-pressure calcium phosphates within the Chelyabinsk meteorite, formed during repeated high-energy impacts to the parent asteroid.

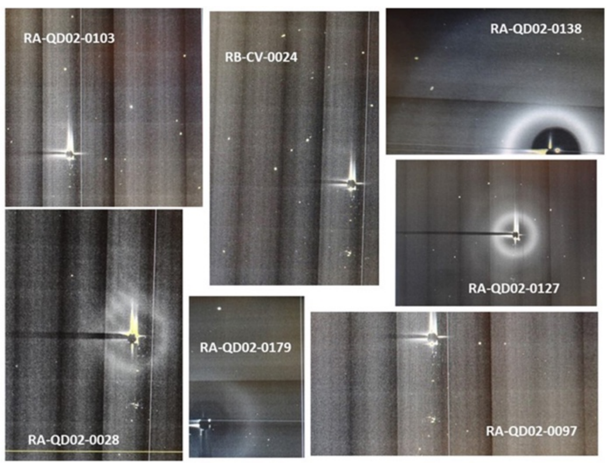


Figure 5 Single crystal XRD patterns of one representative olivine crystal from each of seven different Itokawa regolith grains. In general the diffraction maxima are relatively sharp, best resembling relatively condensed diffraction spots indicative of low degree (S2) of impact shock effects.

These have recently been reported, but we were unable to verify these claims.

We also performed an associated analysis of the cooling history and redox state of the NWA 8694 chassignite[13,26], a meteorite from Mars.

Stardust Mission Samples from the Coma of Comet Wild 2

We measured cell dimensions of olivine in the coarse-grained terminal particles of Wild 2 aerogel tracks and a comprehensive dataset of analogous olivine grains (5 - 30 μm) isolated in chondrite matrix[4]. This tested a proposal that olivine cell dimensions were somehow altered by shock. We found this assertion to be incorrect.

Shocked Carbonates from a Terrestrial Meteorite Crater

We attempted to determine the crystal structures of carbonates from the rim of meteor crater in Arizona. The goal was to determine whether impacted carbonates can form carbonate glasses. We found that amorphous carbonate phases are not present, which was counter to the proposal by the scientist who provided the samples (Fred Horz, NASA JSC). This information is critical to understanding the samples that will be returned by Hayabusa2 from C-class asteroid Ryugu, which is likely to include shocked carbonates.

Samples of Early Solar System Hydrovolcanism

We attempted to determine the crystal structures of mineral grains separated from within salt crystals found in ordinary chondrite meteorites Zag and Monahans[11,25,27-29]. These are derived from the mantle of a hydrovolcanically active early solar system body - possibly asteroid 1/Ceres. These analyses will be critical to understanding the results of NASA's Dawn Mission to Ceres, and are our only samples of hydrous volcanism. Thus far we have been able to determine cell dimensions of olivine and low-Ca pyroxene and assess the shock state of the solids erupted along with the brine fluids. We have proposed that these phases formed on the large asteroid Ceres during diverse parent body reactions, followed by hydrovolcanism. Our efforts to characterize additional, non-silicate phases were unsuccessful.

Summary of Our Results

The main goal of our research was to characterize the crystal structures of minerals in the primitive asteroid and comet regolith samples maintained by NASA and JAXA, to better reveal formation conditions and early evolution of small bodies in the early solar system. For this work we mainly analyzed carbonaceous chondrite and ordinary chondrite meteorites, Asteroid Itokawa samples, and Comet Wild 2 samples. Our analysis of the effects of impact shock on asteroid and comet regolith samples revealed new details about the origin and early history of aqueous fluids in the early solar system and how these fluids migrated through the early solar system. We also determined that the shock impact history of asteroids could be revealed by study of individual regolith grains, which will greatly expand the usefulness of the asteroid Ryugu and Bennu samples that will be returned to Earth by JAXA and NASA spacecraft over the next five years.

Acknowledgements

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