Measuring Shock Stage of Itokawa and Other Asteroid Regolith Grains by Electron Back-Scattered Diffraction

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Introduction: One of the fundamental aspects of any astromaterial is its shock history, since this factor elucidates critical historical events, and also because shock metamorphism can alter primary mineralogical and petrographic features, and reset sample chronologies [1-3]. Failure to take shock history into proper account during characterization can result in seriously incorrect conclusions being drawn regarding the formation and geological history of a body. Thus the Hayabusa Preliminary Examination Team (HASPET) made shock stage determination of the Itokawa samples a primary goal. However, we faced several difficulties in this particular research. The shock state of ordinary chondrite materials is generally determined by simple optical petrographic observation of standard thin sections, sometimes doubly polished but always of a uniform, set thickness. The Itokawa samples available to the analysis team were sometimes attached to carbon fibers, but more generally mounted into plastic blocks which were polished on only one side, and were of non-standard and greatly varying thickness, all of which significantly complicated petrographic analysis (but did not prevent it).

Shock State by EBSD: We made determination of the sample shock state of several Itokawa regolith grains by electron back-scattered diffraction (EBSD) [1-5]. Since EBSD is probably going to become the tool of choice for shock determination of regolith grains, we made a special effort to provide a solid foundation for this technique. Thus one goal of this work is to devise a bridge between shock determinations by standard light optical petrography, crystal structures as determined by electron and X-ray diffraction techniques [1-4]. We are comparing the Itokawa samples to L and LL chondrite meteorites chosen to span the shock scale experienced by Itokawa, specifically Chainpur (LL3.4, Shock Stage 1), Semarkona (LL3.00, S2), Kilabo (LL6, S3), NWA100 (L6, S4) and Chelyabinsk (LL5, S4). In this presentation we will concentrate on the EBSD work.

An important subtask of the EBSD work was to determine if shock state “standards” (meteorite samples of accepted shock state) show strain measurements that may be statistically differentiated, using a sampling of particles (number and size range) that may be expected from an asteroid sample-return mission. We are initially seeking “Indirect” evidence of impact shock since we are not seeking actual strain values, rather indirect strain-related measurements such as extent of intra-grain lattice rotation.

Our ultimate goal is to establish and then to apply the EBSD method, in particular, to regolith grains from near-earth asteroid Itokawa returned to Earth by the Hayabusa spacecraft, and ultimately add this capability to the planetary science took kit for subsequent missions and their resultant returned astromaterial particles.

Our research will improve our understanding of how small, primitive solar system bodies formed and evolved, and improve understanding of the processes that determine the history and future of habitability of environments on other solar system bodies. The results will directly enrich the ongoing asteroid and comet exploration missions by NASA and JAXA, and broaden our understanding of the origin and evolution of small bodies in the early solar system, and elucidate the nature of asteroid and comet regolith.

Techniques: This work was begun under the auspices of the Hayabusa Sample Preliminary Examination Team (HASPET) activity, where many analyses were made in carefully planned sequential order, under very severe time constraints. One effect of the time constraint was that we frequently had only one opportunity to see a particular sample before it was partially or entirely consumed by a subsequent analysis. Since EBSD requires exceptionally well-polished samples we had to find a new procedure for the final polish. Rather than using water and colloidal silica, as is traditional for EBSD, we used a mixture of ethylene glycol, ethanol, glycerol, and 0.05 µm alumina, as recommended by George Vander Voort (personal communication, 2010). The resulting sample finish was slightly inferior to what could have been achieved using colloidal silica, but was adequate for our purposes.

We employed JSC’s Supra 55 variable pressure FEG-SEM and Bruker EBSD system. We were not seeking actual strain values, but rather indirect strain-related measurements such as extent of intra-grain lattice rotation, and determining whether shock state "standards" (meteorite samples of accepted shock state, and appropriate small grain size) show strain measurements that may be statistically differentiated, using a sampling of particles (number and size range) typical of asteroid regoliths.

In order to usefully obtain and compare EBS patterns from astromaterial samples, we had to undergo a rather extensive optimization program for sample preparation and analysis settings. Unfortunately, we anticipate that each EBSD user must perform a similar procedure, especially for different SEM models. Using our system we determined that a column pressure of 9 Pa and no C-coating on the sample was optimal. We varied camera exposure time and gain to optimize mapping performance, concluding that 320x240 pattern pixilation, frame averaging of 3, 15 kV, and low extractor voltage yielded an acceptable balance of hit rate (>90%), speed (11 fps) and map quality using an exposure time of 30 ms (gain 650). We found that there was no strong effect of step size on Grain Orientation Spread (GOS) and Grain Reference Orientation Deviation angle (GROD-a) distribution; there was some effect on grain average Kernel Average Misorientation (KAM) (reduced with smaller step size for the same grain), as expected. We monitored GOS, Maximum Orientation Spread (MOS) and GROD-a differences between whole olivine grains and sub-sampled areas, and found that there were significant differences between the whole grain dataset and subsets, as well as between subsets, likely due to sampling-related "noise". Also, in general (and logically) whole grains exhibit greater degrees of cumulative lattice rotation. Sampling size affects the *apparent* strain character of the grain, at least as measured by GOS, MOS and GROD-a. There were differences in the distribution frequencies of GOS and MOS between shock stages, and in plots of MOS and GOS vs. grain diameter. These results are generally consistent with those reported by A. Ruzicka [5]. However, it is unknown whether the differences between samples of different shock states exceeds the clustering of these values to the extent that shock stage determinations can still be made with confidence. We are investigating this by examination of meteorites with higher shock stage 4 to 5.

Grain size vs. GOS/MOS distribution: We compared the grain size vs GOS and MOS distributions for Semarkona and Chelyabinsk, which have significantly different shock histories. Significant differences between plots of GOS and MOS vs grain size are apparent, and can be proposed as factors to be used to discriminate between different shock histories. It remains to be proven that these are due to differences in the inherent properties of these samples rather than due to differences in settings used for data acquisition, or to deconvolve these two and other potential sources. Notably, MOS shows considerably less difference in "illegal zone" limit position.

Conclusions: A major question we are addressing is: Do small fragments properly represent the strain state of larger rocks? And if so, how to measure that strain? Along these lines there are some important factors to consider. Subsets of larger grains generally don't fully represent the full range of strain behaviors exhibited by the largest grains in our EBSD maps by the strain-related measurements we're using (GOS, MOS, GROD-a, KAM and grain averaged KAM). There is not a 100%, completely clear relationship between grain size and GOS/MOS. However, a very preliminary comparison between Semarkona and Chelyabinsk indicated a significant strain difference, using GOS. Therefore, it appears that GOS can be used to reveal the impact shock histories of asteroidal samples, assuming that a sufficient number of samples or map areas are interrogated. Since there's no strong effect of grain size on some of the most important strain-related measures (e.g., GOS and GROD-a), perhaps small particles are may be reasonably characterized by relatively pixelated maps. Our results suggest that shock strain can be best elucidated by collection of especially slow, high quality EBSD maps (e.g., 640 x 480 camera binning, >1 frame averaging), even if they're only incrementally better than the standard balanced settings we used (320x240, 1 frame). The best results are obtained when completely comparable maps are collected. This means using identical (1) sample preparation techniques, (2) identical instrument and instrument conditions, (3) identical map settings, (4) similar mapping areas and/or number of grains. Since there are numerous SEM models being used by meteoriticists, it would be a best for a set of standard materials to be prepared and distributed in "round robin" fashion to all interested labs.

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