A COMPREHENSIVE NANO- AND MICRO-MECHANICAL TESTING PLAN FOR BENNU SAMPLE RETURN.

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Introduction: NASA's OSIRIS-REx mission to Bennu, a carbonaceous asteroid, will return samples to Earth in September 2023 [1]. Bennu is a remnant from the protoplanetary disk that records the earliest history of the solar system [2]. As a major thrust of the mission, the OSIRIS-REx Sample Analysis Team (SAT) will uncover the characteristics and origins of the returned sample [1]. As part of this investigation, a series of tests will be performed to determine the physical and thermal properties and the effects of nano-sized inclusions on the micro-scale of the returned sample. The following overview focuses on a subset of these analyses: the nano- and micro-scale mechanical tests.

Nano- and Micro-scale Testing Program: We plan to perform (i) nano- and micro-scale indentation, (ii) compression testing, and (iii) atomic force microscopy (AFM) cohesion measurements on Bennu samples. These data will be used to develop scaling relationships to extrapolate strength values to larger scales, providing insight into the physical evolution of Bennu and its parent body [3, 4].

<u>Preliminary Results of Indentation Testing on Analog Materials:</u> Bennu's infrared spectra are consistent with carbonaceous chondrite meteorites with a high degree of aqueous alteration [5], thus the returned sample is expected to consist of abundant, carbonaceous chondrite–like clay-rich material and possible other phases such as carbonates and magnetites [6]. The expected heterogeneous nature of these samples makes the response to any excitation on larger scales a combination of the effects across multiple material phases on smaller scales. Instrumented nanoindentation, which consists of pushing a diamond Berkovich tip into the surface of a material and recording the force-displacement response, allows for the properties of the clay-like matrix and anhydrous silicates (on a scale down to ~5–10 nm) to be elucidated thanks to phase separability [7]. Micro-indentation is the same style test with larger forces and penetration depths, yielding a more homogenized response. The modulus (*M*) and hardness (*H*) of the indented material(s) are determined using the Oliver and Pharr model [8].

Typically, up to several hundred nano-indents on a single phase are desired to have a high confidence in the material property statistics. As part of preparations for sample return, several practice materials were indented, including a 30-μm thin section, prepared at NASA's Johnson Space Center (JSC), of the Lonewolf Nunataks 94102 (LON) meteorite (Fig. 1,a-c). Although the sample may contain epoxy in the pore spaces, this preparation method is well suited for testing protocol development, as the stiffness and hardness of the epoxy is typically a factor 5–7 lower than the most compliant phase of the sample — the clay matrix.

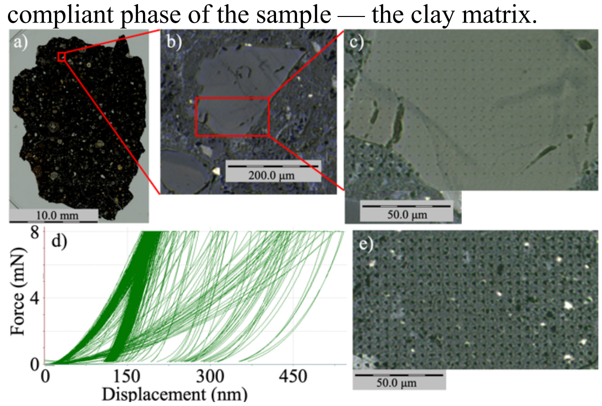

Fig. 1: (a-c) Progressively magnified view of LON meteorite thin section with 405-indents performed on embedded stiff particle (c), resulting in (d) collection of force-displacement curves (e) 400 indents in the fine-grained clay matrix.

Preliminary results using a force-controlled testing protocol with a maximum force of 8 mN indicate that there are stiffer inclusions embedded in a more compliant clay matrix. The collection of measurements on and around the particle (Fig. 1c) resulted in the force-displacement curves (Fig. 1d). Most of the indents on the particle had shallow footprints around 150 nm deep, giving an *M* and *H* of 183.2 ± 44 GPa and 12.5 ± 3.56 GPa, respectively. Indents on the clay matrix (Fig. 1e) were deeper, for the same maximum force, due to the more compliant behavior of the clay, resulting in wider footprints and giving an *M* and *H* of 46.7 ± 7.3 GPa and 2.67 ± 0.61 GPa, respectively. Micro-indentation was also performed on a clay-rich area of a 50-micron polished thin section, also prepared at JSC, of a Bennu-like CM terrestrial simulant, similar to [9] but with a higher total porosity of ~54%. These indents reached depths of ~2.9 microns at 8 gram-force, yielding an *M* of 18.22 ± 5.93 GPa and *H* of 0.49 ± 0.14 GPa.

In summary, nanoindentation has been demonstrated to be a viable technique for identifying the mechanical properties of individual phases at very small scales, which is consistent with findings from other heterogeneous materials including cements and organic-rich source rocks [7,10,11]. Ongoing work to prepare for implementation on the returned samples includes investigating other sample preparation techniques to see whether the needed quality of polishing can be achieved without embedded epoxy stabilization.

Plans for Compression and Cohesion Testing:

Compression. Compressing a grain between two rigid metallic pistons is a common technique for quantitative determination of the mechanical behavior of individual grains at millimeter and sub-millimeter length scales [12]. The scale of this test will be on the order of a few hundred microns to millimeters, depending on the size distribution of the returned sample. The micro-indenter will be used with a "flat punch" probe to perform the test. The loading protocol will be displacement controlled up to a maximum limiting depth. The output of each test is a force versus displacement curve. The compressive, or crushing, strength is then determined from a simple function that uses particle dimensions and the max force that caused the particle to crush. The average particle diameter will be determined through a microscope with a digital caliper and confirmed using a Vernier. Preliminary tests and protocol development are ongoing.

Particle cohesion. Bennu is a rubble pile held together by a combination of gravity, friction, and cohesion that may also control its bulk shape [6, 13, 14] Identifying the relative magnitudes of these forces offers a way to help understand the stability and evolution of Bennu's shape due to external forces (like YORP spin-up), and to develop hazard-mitigation strategies for Bennu and other near-Earth asteroids. To accomplish this, we plan to directly measure the cohesive force between sample particles. Importantly, we seek to mitigate the influence of humidity, which has adversely affected cohesion measurements in the past, as discussed most recently in [15].

Particle-to-particle measurements will be tested inside of an AFM that can perform vertical approach displacement. One particle will be attached to a tipless AFM cantilever, using a micro-manipulator, and another to a substrate. The cantilever arm will be lowered, allowing the particles to touch, until zero force is registered. The cantilever will then be raised, and the particle separation force will be measured. All testing will be performed in a laboratory-grade nitrogen-purged chamber also containing desiccant to help reduce the relative humidity (RH), which will be constantly monitored and recorded by a hygrometer. Before testing, the particles will be heated up to just under 100 degrees C to help evaporate any free water while being exposed to laboratory ambient conditions and be stored in a vacuum desiccator prior to testing.

The objective is to determine the cohesion force of Bennu as a whole. We will request a random sample of fine particles and obtain the statistical distribution of cohesive/adhesive forces between different particle pairs. The total amount of particle pairs depends on the total allocated sample, but it is expected that ~40 particles (or 20 pairs) will be feasible to test. Cohesion force is governed by the amount and composition of asperities, or roughness contact points [16], so for each cohesion pair, several replicate "touches" will be performed at different locations. The surface roughness power spectra $C(q)$ will also be elucidated through linear profilometry on the grains surface.

The effect of humidity on cohesive force will also be investigated on the Bennu-like CM terrestrial simulant [9] in advance of sample return. Ambient conditions will be slowly released into the nitrogen-purged chamber while repeated particle touches are taking place, causing a change in measured force. This will allow us to understand how much time is available to expose the particles to ambient conditions before the surface tension of water governs the measurements and, conversely, how long we must purge with nitrogen gas before beginning a new test.

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