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Short Communication

Electrospray charging of minerals and ices for hypervelocity impact research

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ABSTRACT

Microparticle impacts are an important solar system process, and laboratory experiments are essential to understanding both microcratering and the results from *in situ* cosmic dust analyzers. However, current dust accelerators can only use conductive projectiles, limiting projectile types and possibly complicating studies of microparticle impact chemistry. We present a charging method that eliminates the need for conductive projectiles by using electrospray, instead of contact charging, to electrically charge microparticles. Using this novel application of electrospray, charged microparticles of quartz, quartz–ice aggregate, and methanol–water ice have been produced and observed. These experiments also demonstrate that the quartz surface can be protonated under non-equilibrium electrospray conditions, implying that the quartz surface may have a point of zero charge. Although coupling an electrospray source to a dust accelerator presents challenges, electrospray charging of minerals, mineral–ice aggregates, and astrophysical ices may enable experiments with projectiles that more closely resemble actual solar system materials.

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1. Introduction

Impact cratering is a ubiquitous and significant solar system process, and understanding the impact process and its ramifications is a goal in several NASA guiding documents (NASA, 2006; Paulikas et al., 2007; Johnson et al., 2010; Abell et al., 2011; Committee on the Planetary Science Decadal Survey, 2011). Cratering operates at scales ranging from micrometeorites to planetesimals (e.g., Lindsay, 1976; Canup, 2004), and the details of cratering processes are scale-dependent. This paper focuses on micrometeorite cratering, the scale relevant to cosmic dust (Burchell et al., 1999b; Austin, 2003), space weathering (Brunetto, 2009), and the Stardust particles (Sandford et al., 2006; Horz et al., 2006). This paper presents a new method for charging particles for high- to hyper-velocity micrometeorite impact experiments, experiments essential to understanding microcratering, impact ionization (Sysoev et al., 1997; Burchell et al., 1998; McDonnell et al., 2001), and results from *in situ* cosmic dust analyzers like those on Stardust, Galileo, and Cassini (Burchell et al., 1999b; Shibata et al., 2001; Goldsworthy et al., 2003).

Existing dust accelerators are based on the designs of Shelton et al. (1960) and Friichtenicht (1962). They consist of a dust reservoir, charging needle, Van de Graaf or Pelletron accelerator, charge sensitive detectors, and impact region. Microparticles are charged by touching a high-voltage needle in the dust reservoir.

Charge is transferred from the charging needle to the projectile, which acquires charge of the same polarity. Charged particles then exit the dust source and are accelerated by the potential difference produced by the generator. Accelerated “dust” can reach speeds up to 100 km/s (Burchell et al., 1999b; Stübig et al., 2001).

Ideally, projectiles used in microparticle impact experiments should be identical to those involved in actual microparticle impacts (e.g., silicates, carbonaceous materials, and astrophysical ices). However, because current instruments use contact to charge projectiles, projectiles must be electrically conductive. Unfortunately, most solar system materials of interest are not electrically conductive. Although a limited range of non-conductive materials have been coated with a thin layer of conducting material and used in acceleration experiments (Burchell et al., 1999a; Goldsworthy et al., 2003; Srama et al., 2009; Mocker et al., 2010), the conductive requirement hinders attempts to use natural geological materials as projectiles. Ices, for example, have not been coated before and likely cannot be coated with a conductive layer, yet microparticle cratering experiments with ices and silicate–ice aggregates are of prime importance to comets and the outer solar system, including the Enceladus plume. Furthermore, conductive coatings may complicate efforts to interpret data from *in situ* cosmic dust analyzers because of preferential surface ionization, particularly at lower impact velocities (Sysoev et al., 1992; Sysoev et al., 1997; Burchell et al., 1998; McDonnell et al., 2001). There were early attempts (Vedder, 1978) at electrical acceleration of non-conducting particles—including quartz—in which particles were

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electrodynamically suspended, charged by a proton beam, and the mass-to-charge measured prior to multi-stage acceleration, but this technique was time-consuming, and was not amenable to ices. While existing dust accelerators have clearly contributed to understanding microscale cratering and its applications to planetary science, the use of non-conductive projectiles would enable experiments that use uncoated solar system materials as projectiles. Such experiments would more closely resemble the micro-particle impacts that actually happen in the solar system, and eliminate any complications to impact mass spectrum interpretation introduced by conductive coatings.

2. Materials and Methods

We present a method that uses electrospray to charge microparticles, eliminating the need for conductive projectiles and allowing the use of bare, uncoated minerals, ices, and mineral-ice aggregates as projectiles. Developed as a tool for charging large biomolecules (Fenn et al., 1989), electrospray has since expanded to inorganic compounds (Cole, 1997). However, no previous study reports electrospray charging of mineral grains. Based on our review of the literature, this is the first time that minerals and mineral-ice aggregates have been charged using electrospray.

2.1. Electrospray charging of microparticles

Electrospray relies on the strong electric field between a highly charged needle and a grounded orifice (Fig. 1). For positive-mode electrospray, a suspension containing particles of interest is acidified and slowly pumped through a positively charged needle. The potential gradient between needle and orifice results in a Taylor cone at the needle's tip, where the suspension breaks into tiny, charged droplets—each with excess protons—that form a plume extending toward the orifice. Solvent evaporates from the charged droplets as they move into and through the vacuum system, increasing the ratio of surface charge density to droplet size. When Coulombic repulsion exceeds surface tension, a condition known as the Rayleigh limit, droplets undergo fission to form smaller droplets, each carrying some of the original droplet's mass and charge. This process occurs repeatedly, and positive-mode electrospray ultimately produces gas phase, positively charged (protonated) solute particles (Cole, 1997).

The electrospray suspensions are acidified to improve particle charging and electrospray quality. We created suspensions at three different pH values and evaluated their performance. These values were pH 0.95, 2.01, 3.60. pH 3.60 yielded the best electrospray performance. The moderately acidic pH that our experiments show

are effective for electrospraying mineral microparticles should not significantly modify microparticle surfaces. Even complex organic compounds can be electrosprayed without suffering ill effects. Viruses, for example, have been shown to survive the electrospray process intact (Fuerstenau, 2003). Fragile carbonaceous material, like that in carbonaceous chondrites, should be practically unaffected by electrospray. The electrospray setup used in this study consisted of a KD Scientific KDS100 syringe pump, Hamilton 0.5 mL syringe, 63.5 μm inner diameter hollow needle (produced in-house), and Stanford Research Systems PS350 high voltage power supply. The syringe pump dispensed the suspension to be electrosprayed at rates between 100 and 300 $\mu\text{L/hr}$. The needle was kept between +3000 and +3500 V and was located 1.3 cm from the orifice into vacuum.

2.2. Instrument design

A new instrument was designed and built for this study, consisting of an electrospray source (described above), two-stage differential pumping system, beam tube, and image charge detector (Fig. 2). Electrosprayed particles entered the first stage of the vacuum system (pressure ~ 1 Torr) through a 150 μm diameter orifice, traversed a 30 cm-long beam tube, passed through a skimmer, and entered the second stage of the vacuum system (pressure between 10^{-1} and 10^{-2} Torr), where they were measured by an image charge detector.

2.3. Image charge detection

The charge and velocity of electrosprayed particles were determined with an image charge detector consisting of a copper tube (5.715 cm long; 0.318 cm inner diameter) connected to a charge-sensitive pre-amplifier (Amptek A250) and three pulse shaping amplifiers (Amptek A275), all housed in copper shielding. The detector and associated electronics were located in vacuum.

A charged particle entering the charge detector induced an equal but opposite image charge on the copper cylinder. The detector recorded a down peak as a positively charged particle entered the tube and an up peak as the positively charged particle exited the tube (Fig. 3). Peak area is directly proportional to particle charge (Maze et al., 2006; Mabbett et al., 2007; Zilch et al., 2008; Smith et al., 2011). The charge detector was calibrated by pulsing a known voltage pulse through the 2 pF test capacitor of charge detector and measuring the peak area of the resulting signal. A peak area of 1 μVs corresponds to 168,000 protons. Signals were processed and recorded on a LeCroy WaveSurfer 42Xs oscilloscope.

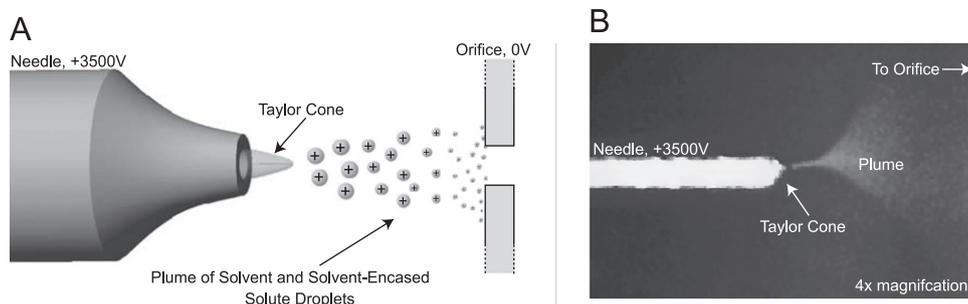


Fig. 1. The electrospray process. (a) A suspension of microparticles is slowly pumped through a hollow needle. The electric field between needle and grounded orifice causes a Taylor cone to form at the needle's tip, where the suspension breaks into tiny, positively charged droplets. Solvent evaporates as the particles travel from the needle into vacuum, and droplets fission upon reaching the Rayleigh limit. At this point, evaporation and fission could continue until a charged, solvent-free solute particle is produced. Or, the solvent could freeze before the particle is completely desolvated, forming a mineral-ice aggregate. (b) Photomicrograph showing electrospray needle, Taylor cone, and droplet plume before entering vacuum.

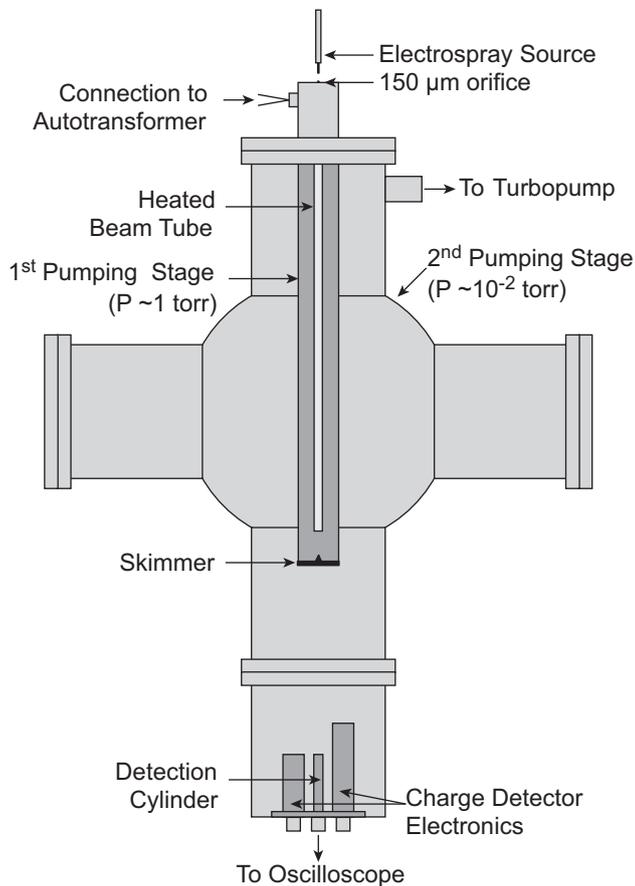


Fig. 2. Instrument for electro spraying and detecting mineral and ice microparticles, consisting of an electro spray source, two-stage differential pumping vacuum system, heated beam tube, and image charge detector. Electro sprayed microparticles enter vacuum, where they pass through a heated beam tube and skimmer before entering the second pumping stage and being detected by an image charge detector. Charge detector signals are amplified and then recorded by an oscilloscope.

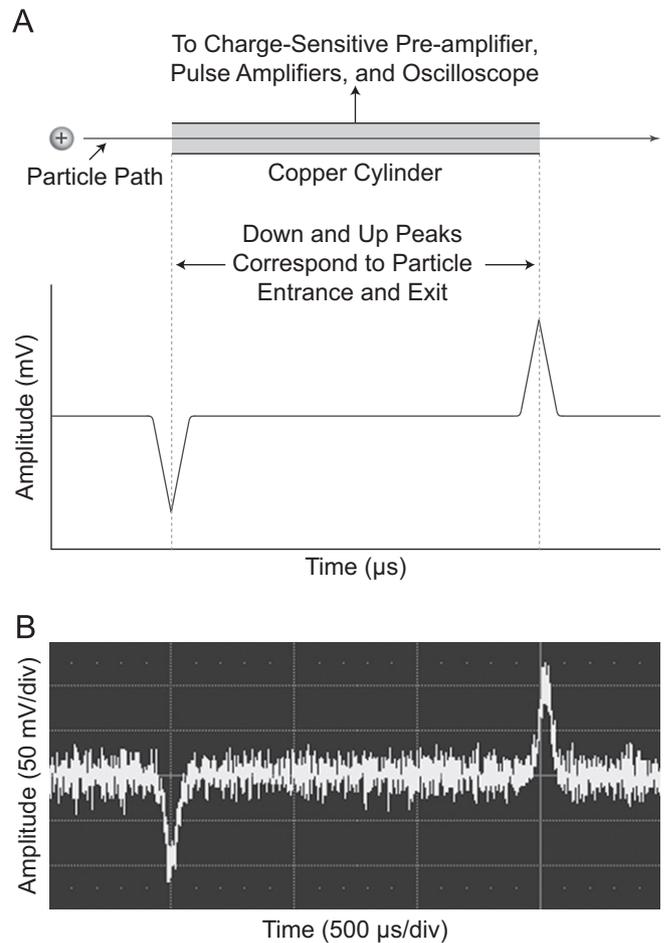


Fig. 3. Image charge detection. (a) A charged particle induces an equal but opposite image charge as it enters and exits the cylinder: a down peak when a positively charged particle enters and an up peak as a positively charged particle exits. The signal is amplified and recorded by an oscilloscope. (b) Charge detector signal from a typical charged quartz microparticle.

2.4. Particle types

Experiments were done with a variety of particle types, including 2.2- μm diameter amino-terminated polystyrene spheres (Spherotech), $\sim 2 \mu\text{m}$ diameter quartz microparticles, quartz-ice aggregates, and methanol-water ices. The recent detection of silica and hydrated particulates in the Enceladus plume makes the quartz and quartz-ice aggregates particularly relevant to planetary science.

Amorphous silica microparticles are commercially available, but because SiO_2 surface charging is phase dependent (Zhukov et al., 2000), quartz microparticles were made in-house. Transparent quartz points (Ward's Natural Science) were ground to a fine powder with mortar and pestle and then processed for 30 minutes in a McCrone Micronizing Mill (Fig. 4). The quartz microparticles were suspended in distilled water and centrifuged four times to narrow the particle size distribution, resulting in an average diameter of $\sim 2 \mu\text{m}$ (Fig. 4). We prepared 0.005% w/v suspensions of all particle types in a 4:1 mixture of methanol and water, following the procedure of Cai et al. (2002). 4:1 mixtures of methanol and water without particles served as controls.

The pH of an electro spray suspension affects the spray quality produced by that suspension (Cai et al., 2002). Suspension pH was adjusted with formic acid.

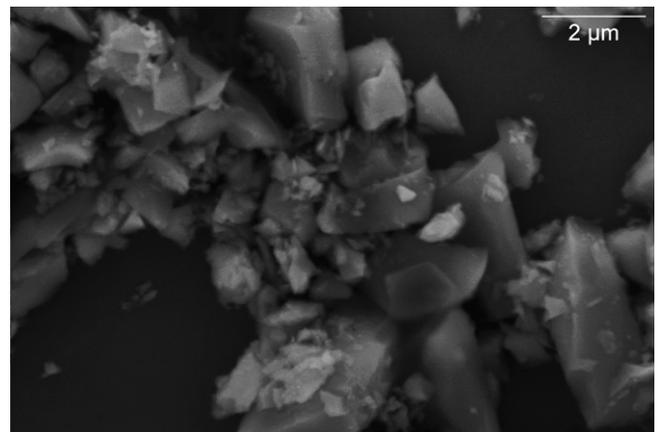


Fig. 4. SEM image of quartz microparticles after 30 minutes in the micronizing mill. The particle size distribution was narrowed before using microparticles in experiments.

3. Results

Before beginning experiments with quartz microparticles, the instrument was tested by reproducing an experiment from Cai et al. (2002) using a pH 3.9, 0.005% w/v suspension of 2.2 μm amino-terminated polystyrene spheres in a 4:1 methanol-water

solution. We successfully reproduced their experiment, and all components of the instrument functioned as planned. We then proceeded with other experiments, and data indicate successful electrospray charging of all particle types, including quartz microparticles, quartz–ice aggregates, and methanol–water ices. This paper discusses results from 3975 particles of varying types, suspension pH, and beam tube temperatures (see Table 1).

Initial experiments indicated the quartz particles were not completely desolvated by an unheated beam tube. As a result, the beam tube was incrementally heated to aid particle desolvation. At lower temperatures, incompletely desolvated particles freeze in high vacuum due to evaporative cooling, forming charged particles of ices and mineral–ice mixtures. High beam tube temperatures result in bare, charged mineral particles.

We performed desolvation experiments with both quartz microparticles and solvent droplets to determine when the beam tube was hot enough to completely evaporate solvent. In an experiment using solvent without any particles added to it, the charge detector stopped detecting solvent droplets when the exterior of the beam tube was 609 °C, suggesting that quartz microparticles are also completely desolvated at this temperature. Although we infer that the quartz particles are desolvated at this temperature, our experiments do not rule out the possibility that some water molecules remain on the particles detected. Data show that particle charge, on both quartz microparticles and solvent droplets decreases with increasing beam tube temperature (Fig. 5). Least squares regression on the data yielded strong correlation coefficients; the slopes of all regression lines are significant at $P < 0.001$.

4. Discussion

4.1. Possible causes of desolvation relationships

Desolvation data reveal a statistically significant linear relationship between beam tube temperature and particle charge. One explanation for this relationship is that solvent evaporation

Table 1
Summary of electrospray experiments.

Particle type	pH	Beam tube temperature (°C)	Number of particles
Polystyrene	3.9	23	1805
Quartz	2	23	274
Quartz	1	23	79
Solvent	3.6	25–38	305
		52–115	314
		141–245	279
		285–432	229
Quartz	3.6	25–38	175
		52–115	167
		141–245	179
		285–432	169

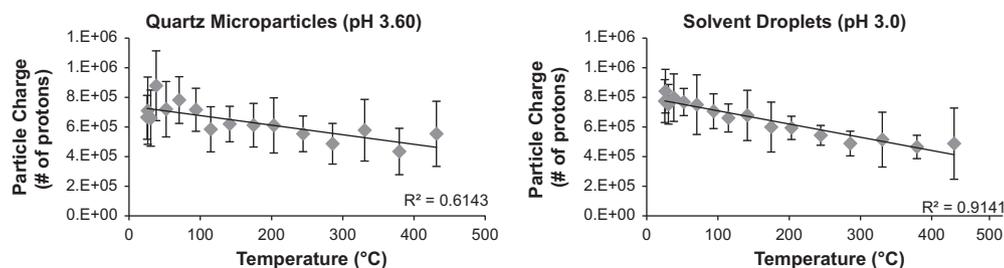


Fig. 5. Effects of desolvation on particle charge for (a) quartz microparticles and (b) solvent droplets. The slopes of the regression lines are significant at $P < 0.001$.

increases with beam tube temperature, leading to small solvent droplets budding off and carrying away some of the original particle's charge. This process would reduce the original particle's size and, assuming constant surface charge density, particle charge would decrease. However, if budding droplets did not carry charge away with them, then particle charge would remain constant despite decreased particle size. Given the debate about how droplet fission happens (e.g., Maze et al., 2006; Mabbett et al., 2007; Zilch et al., 2008; Smith et al., 2011), this hypothesis is, at best, tentative.

4.2. Implications for quartz surface chemistry

The quartz surface, which consists of silanol groups in various configurations, accepts charge differently from most metal oxides, and there is debate about whether quartz has a well-defined point of zero charge (PZC) (Kosmulski, 2001). The PZC is the pH value where a mineral surface has a net neutral surface charge, and the PZC is often determined during a potentiometric titration of a colloidal dispersion of the mineral of interest. If quartz has a PZC, it is thought to lie between pH 1 and 2 (Kosmulski, 2001). Most surface complexation models treat quartz with three types of surface groups: SiO^- , SiOH , and SiOH_2^+ groups. The SiOH_2^+ group was recently observed (Duval et al., 2002), but the Duval et al. (2002) data were not collected in aqueous solution.

This study's charge detector data clearly show positively charged quartz microparticles. Measured particle charges correspond to protonating roughly 0.007% of the silanol sites on a spherical, 2 μm diameter quartz microparticle, assuming 4.5 silanol sites per nm^2 . This small protonation fraction suggests that methodological refinements could increase protonation, charge-to-mass ratio, and acceleration potential.

In these experiments, quartz surfaces are positively charged by (1) protonation of silanol groups or (2) attachment of hydronium ions (H_3O^+) with or without additional residual solvent. The energy required to dissociate a water molecule from a protonated silanol group is similar to the dissociation occurring in charged, solvent-only droplets, which are not observed at high temperatures. Thus, the second option is less likely at high beam tube temperatures, and protonated silanol groups are the preferred explanation for these results.

Data indicating protonated silanol groups imply that quartz has a PZC. However, electrospray is not an equilibrium process (unlike potentiometric titrations), so the PZC of quartz cannot be specified from these data. However, the PZC is likely at or below 3.9, the pH of the quartz microparticle suspension used in desolvation experiments. The PZC is most likely below 3.9 because the ratio of protons to solute molecules increases during evaporation and fission, effectively decreasing the pH of the solvent droplets that surround quartz particles. Data from this study, along with results from (Shchukarev et al., 2004), are one of the few direct, experimental evidences of quartz surface protonation at low pH.

4.3. Implications for hypervelocity impact experiments

An important potential application of mineral electrospray is as a particle source for microparticle accelerators, with applications to planetary science. The results of this study show that electrospray charges quartz microparticles, quartz–ice aggregates, and methanol–water ices. These successes suggest that electrospray might charge other types of mineral microparticles, mineral–ice aggregates, and astrophysical ices of variable composition. Ongoing experiments in our research group involve olivine and ordinary chondrite microparticles (Kerby et al., 2012; Daly et al., 2012).

The ability of electrospray to charge completely bare mineral particles, partially desolvated particles, and pure solvent droplets enables charging of projectiles that conventional dust sources cannot charge, including particles similar to cometary and planetary ices. These charged particles could then be used as projectiles in hypervelocity microparticle impact experiments. Such projectiles would be better analogs for actual solar system projectiles and facilitate progress in understanding cratering at the microscale.

In addition, an electrospray-based particle source maintains an important advantage of existing dust sources over light-gas guns: individual particles are charged and introduced into the instrument. The very low concentration of particles in suspension (0.005% weight-to-volume) results in only one particle per electrospray droplet (Cai et al., 2002). Existing particle selection systems, like that at the Heidelberg dust accelerator, should still work with particles charged by electrospray. This is important for experiments that strive to characterize *in situ* cosmic dust analyzers, like the Cassini CDA.

Clearly, coupling an electrospray-based particle source to existing dust accelerators is not trivial, but it may be possible. Issues such as electrical insulation, electrophoretic effects, and sample access must be addressed. In particular, differential pumping and liquid sample lines must be either totally contained within the high voltage region or fed in a way that prevents electrical discharge. These issues are discussed in more detail in Daly et al. (2012) and Kerby et al. (2012). Ongoing work focuses on finding solutions to these challenges.

5. Conclusion

We have successfully used electrospray to charge quartz microparticles, quartz–ice aggregates, and methanol–water ices. These results suggest electrospray may charge other uncoated mineral grains, mineral–ice aggregates, and astrophysical ices, which open avenues for experiments that are highly relevant to planetary science. In addition, electrospray allows particle composition to be easily varied. Current work includes positive electrospray of olivine and ordinary chondrite microparticles, as well as investigating coupling of an electrospray source to a dust accelerator.

This study's results are applicable to both quartz surface chemistry and dust accelerators. Charge detector data indicate a protonated quartz surface and suggest quartz has a PZC that lies below pH 3.9, consistent with the ranges of values compiled by Kosmulski (2001). In addition, electrospray could serve as a new particle source for dust accelerators. Although existing accelerators would need to be modified to use an electrospray source, electrospray charging may provide new classes of geologically relevant projectiles for hypervelocity microparticle impact experiments, which may lead to better calibration of *in situ* cosmic dust analyzers, a clearer understanding of impact ionization processes, and a deeper understanding of the role of microcratering in the evolution of solar system bodies.

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