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Major Goals: Granular materials are known to exhibit excellent shock and blast dissipation properties. They are also effective in stopping the high velocity projectiles. Sandbags have been used as fortification against impact and blast loading. Various penetration studies have been performed to assess the mechanisms that help in stopping the projectiles in granular materials. One of the important energy dissipation mechanisms in defeat of projectiles in sand is the fracture of particles in front of the projectile into very fine powder. Particles or granular solids of brittle materials are also subjected to compressive loading during materials processing, storage, handling, transportation, and usage. The dynamic compressive loading can lead to a wide range of deformation and fracture behaviors in the particles. In many particle size reduction processes such as milling, fracture of particles is desired to reduce the size of the particles efficiently. For some materials, small sub-particles resulting from the fracture of larger particles can be harmful to equipment and personnel. Silica dust resulting from fracture of polycrystalline silica particles can cause deadly lung diseases such as silicosis. Small particles (diameter<400 microns) composed of many organic and inorganic materials are categorized as explosive dusts. There is a high likelihood of explosion when an ignition source is present in the vicinity of the explosive dusts. Improved understanding of the particle fracture mechanisms will be immensely helpful in improving the efficiencies of processes when the particle fracture is desired. On the other hand, better understanding of failure mechanisms will help in preventing particle fracture when the resulting fragments can be harmful.

The aim of this study is to obtain the comprehensive understanding of the particle fracture processes under compression. The objective is further divided in to two complementary parts: (1) Experimental assessment of fracture of individual and contacting particles under quasi-static and dynamic compression to observe the failure processes, (2) Development of analytical and numerical models to accurately predict the observed behaviors. The experimental results will guide the development of the predictive model.

The experimental investigation is further divided into: (1) single particle fracture experiments, (2) two particle fracture experiments, and (3) multi-particle fracture experiments. For each type of the experiment mentioned previously, fracture behavior was investigated under quasi-static and dynamic compression. A lot of research has been performed to understand the fracture behavior of individual particles under quasi-static compressive loading. There are established methods in the literature to analyze the fracture energy distribution of particles using quasi-static single particle tests (crushing tests) and studying the breakage of hard particles under controlled loading conditions. However, the literature of fracture of brittle particles under controlled dynamic compressive loading is limited. Two different experimental methodologies have been used to study the behavior of particles under dynamic compression. In the first method, a single particle was impacted against a rigid target and the fracture process was studied using various in-situ and post-processing methods. In a complementary experimental method, a drop weight setup was used to dynamically compress a particle between two plates. The experimental studies present in the literature have three knowledge gaps that the present study aims to fill: (1) the reported studies focus only on the single particle compression and hence cannot account for the effects of the particle-particle contacts which are prevalent in granular materials, (2) It has been impossible to observe the in-situ failure mechanism for opaque particles, (3) Effects of multiple particle-particle contacts and subsequent confinement on the particle fracture are not studied. In this study, synchrotron X-ray imaging setups are used to observe the failure modes of both transparent and opaque brittle particles under compression. X-ray imaging offers a unique advantage of observation of sub-surface damage and cracking which cannot be observed using a traditional optical high speed imaging setup. The fracture mechanisms under dynamic compression are captured using high speed synchrotron X-ray phase contrast imaging system. Further, effects of residual stresses are investigated by performing compression experiments on annealed glass particles. The recorded fracture mechanisms will be vital in identifying the critical stress and strain parameters that initiate the fracture of the particles. In literature, two conflicting critical stress parameters are proposed for the initiation of fracture in particles under quasi-static compression. In the first theory, the maximum tensile stress inside the volume of the particle is proposed as the critical parameter causing the fracture of the particle. In the second theory, the maximum tensile stress just outside the contact area between the particle and the platen was proposed as the critical stress causing fracture. The in-situ observation of the fracture processes will be vital for identifying the correct critical stresses causing fracture. Along with the critical parameters for the initiation of fracture, the fracture mechanisms will also shed light on the fragment size distributions generated by the fracture of the particles. The observed morphology of the fractured particles and the fragment size distributions will also be useful in improving the numerical models (especially particle based discrete element based models).

While the identification of failure modes using both in-situ and post-mortem characterization techniques is beneficial, the ability to predict the fracture behavior will be truly helpful in designing granular systems for various applications including impact protection and energy absorption. Most of the currently available predictive models are not adequate for addressing the particle failure morphology and instead focus on the likelihood of particle size reduction through fracture. Here, we propose a phenomenological parameter based on various particle properties including elastic modulus, hardness, toughness, and particle size to predict the fracture mechanisms of contacting brittle particles under dynamic compression. Finite elements models were also developed for two material systems: (1) elastic-perfectly brittle material, (2) elastic-perfectly plastic material, corresponding to soda lime glass and acrylic (PMMA) particles respectively. The critical stresses were identified by correlating the experimentally observed crack initiation points to the numerically obtained stress states.

Accomplishments: The fracture behaviors of individual and contacting particles are studied using experimental techniques. Six different materials were studied: soda lime glass, silicon dioxide, silicon, barium titanate glass, poly methyl methacrylate, and yttria stabilized zirconia. The fracture mechanisms were studied at quasi-static and dynamic loading rates. Single, two, and multiple contacting particle geometries were studied to investigate the effects of contact conditions on the fracture mechanisms. The experiments were performed for various diameter ranges to assess the effects of size on fracture behavior.

The quasi-static fracture behavior of particles was studied using the single particle compression experiments, performed using a servo-hydraulic machine. The nominal fracture stress-strain data is presented in Figure 1. The fracture stresses were fitted to a Weibull distribution to obtain the Weibull moduli for all materials. Two deterministic models based on the tensile stresses at the contact and near the center of the particle and a statistical model based on the Weibull weakest link theory were proposed to identify the fracture modes of the particles. An example of fracture stresses plotted against the proposed analytical models for soda-lime glass particles is presented in Figure 2. The models provided an upper and lower bounds for the fracture stresses, however, the variability in the experimental data prevented definitive identification of the fracture modes.

A modified Kolsky bar apparatus was synchronized with the high speed X-ray phase contrast imaging setup to record the in-situ fracture mechanisms of single, two, and multiple contacting particles under dynamic compression. The details of the particles used in the compression experiments are provided in Table 1 in the attached document. The failure mechanisms of the particles under dynamic compression were investigated using a combination of a modified Kolsky bar setup and high speed synchrotron X-ray Phase Contrast Imaging (PCI) at Advanced Photon Source (APS) beam line 32-ID-B at Argonne National Laboratory, Argonne, IL. The details of the experimental method are provided in previous reports and also in literature (Parab et al., Int. J. Impact Eng. 68 (2014): 8-14). A schematic and photograph of the experimental setup is presented in Figures 3 and 4 respectively. Controlled constant velocity ($v = 6$ m/s) compressive loading was applied using the Kolsky bar apparatus and the in-situ fracture mechanisms were recorded using the high speed synchrotron X-ray PCI. No significant size effects on the fracture mechanisms were observed. The experimental results obtained from the single, two, and multi particle dynamic compression experiments are presented in Figures 5, 6, and 7 respectively. Note that only one representative experimental result from at least three repeated experiments is presented here. The fracture behavior of the particles was consistent across the repeated experiments. For the single particle experiments, the crack initiation was observed to occur near the center of the particle for all materials. For two particle experiments, the crack initiation occurred near the particle-particle contact in form of angular Hertzian cracks for brittle materials. These angular cracks only separated fragments near the contact and did not ultimately fracture the particle. Angular cracks with resulting contact fragments were also observed in the multi-particle experiments for soda lime glass particles. The elastic-plastic particles did not show any contact cracks. For all particles, the cracks that caused the catastrophic fracture initiated under the particle-particle contact or near the center of the particle. For the single and two particle experiments, the cracks propagated toward the contact, thus forming meridional cracking pattern. For the multi-particle experiments, cracks spanned the contact points with one major meridional crack observed in all experiments. Regardless of the contact conditions, three major catastrophic fracture modes were observed for the particles depending on the material properties: (1) explosive fragmentation or pulverization (soda lime glass), (2) finite number of large cracks or major cracking (silicon, silicon dioxide, and barium titanate glass), and (3) single crack (poly methyl methacrylate, and yttria stabilized zirconia).

A new phenomenological model was then proposed to better describe the observed morphological fracture mechanisms under dynamic compression. This phenomenological model was represented by a pulverization parameter that was related to the hardness, elastic modulus, fracture toughness and the size of the particles assuming displacement driven loading conditions. The pulverization parameters for the particles studied here are plotted in Figure 8. High pulverization parameter values were associated with explosive fragmentation failure, low pulverization parameter values were associated with single cracking failure, and intermediate pulverization parameter values spanned a range of failure modes between explosive fragmentation and single cracking failure.

Scanning electron microscopy was then used to image the generated fragments from the dynamic fracture experiments. All particles showed some variant of the prominent brittle fracture features which included hackle lines and Wallaner lines. In some polycrystalline particles, inter-granular crack propagation was observed. The sharpness of the features were observed to be related to the magnitude of the pulverization parameter for the materials.

From the experimental and numerical results, the critical parameter for the initiation of fracture for single and two contacting particles was identified as the maximum tensile stress inside the volume of the particle for all the materials studied here. However, the subsequent propagation and fragmentation of the particle was significantly dependent on the material properties of the particle. For the materials showing plasticity (PMMA), the crack initiated at the center and propagated towards the contacts fracturing the particle in hemispheres. For brittle materials, the particles either fractured explosively (glass) or into finite number of large fragments (silica and silicon). It is clear that although the initiation of the cracking is dependent significantly on the stress states (specifically maximum tensile stress); the subsequent fragmentation is dependent on both stress states and the flaw distributions in the particle.

Training Opportunities: Training opportunities: One undergraduate student worked on this project under the Purdue Summer Undergraduate Research Fellowship and subsequent independent research project. One graduate student completed his PhD degree with thesis based on the work described here.

Professional Development: The PI and the graduate student attended and presented at several conferences in the area of structural mechanics. These conferences are:
1. Society of Engineering Science Annual Technical Conference 2014, West Lafayette, IN
2. US National Congress for Theoretical and Applied Mechanics 2014, East Lansing, MI
3. Society of Experimental Mechanics Annual Conference and Exposition on Experimental and Applied Mechanics 2014, Greenville, SC
4. APS Topical Conference on the Shock Compression of Matter 2015, Tampa FL.
5. ASME International Mechanical Engineering Congress and Exposition 2015, Houston TX.
6. SEM XIII International Congress and Exposition on Experimental and Applied Mechanics 2016, Orlando, FL.

Along with these conferences, the graduate student presented an invited guest research talk at the Advanced Photon Source, Argonne National Laboratory in February 2016 and July 2017. The graduate student also attended the workshop on dynamic fragmentation in brittle materials at the SEM 2016 conference listed above.

Results Dissemination: The results were disseminated primarily through the conference talks and papers at the events listed in the previous section.

Honors and Awards: Weinong Wayne Chen (PI), Honorary Doctorate of Science in Technology (HDR), Tampere University of Technology, Tampere, Finland, 2017

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Table 1: Particle size, material properties and vendor for each type of particle

Material	Particle size ranges (μm)	Elastic Modulus (GPa)	Hardness (GPa)	Toughness ($MPa.m^{0.5}$)	Density (Kg/m^3)	Vendor
Polycrystalline silicon[1]	800-1200	164 ± 4	9.7 ± 0.4	0.60 ± 0.05	2330	REC Silicon (Moses Lake, WA)
Soda-lime glass (SLG)[1]	1000-1180	83 ± 6	6.5 ± 0.3	1.28 ± 0.08	2500	Cospheric LLC (Santa Barbra, CA)
Yttrium stabilized Zirconia (YSZ)[1]	780-850	255 ± 7	16 ± 0.7	5.69 ± 0.16	6020	Cospheric LLC (Santa Barbra, CA)
Polycrystalline silicon dioxide[1]	1700 - 2000	111 ± 5	13.8 ± 0.6	1.07 ± 0.04	2650	U. S. Silica (Ottawa, IL)
Barium titanate glass[1]	850 - 1000	77 ± 5	5.3 ± 0.3	1.10 ± 0.07	4140	Cospheric LLC (Santa Barbra, CA)
Poly-Methyl Methacrylate (PMMA)[2,3,4]	800, 1600	2.5 ± 0.7	0.24 ± 0.07	1.03 ± 0.16	1170	Engineering Laboratories (Oakland, NJ)

[1] Zbib, Parab, Bahr, Chen, 'New pulverization parameter derived from indentation and dynamic compression of brittle microspheres', Powder Technology, Vol. 283, pp. 57-65, 2015

Figure 1: Nominal fracture stress-strain data for quasi-static compression

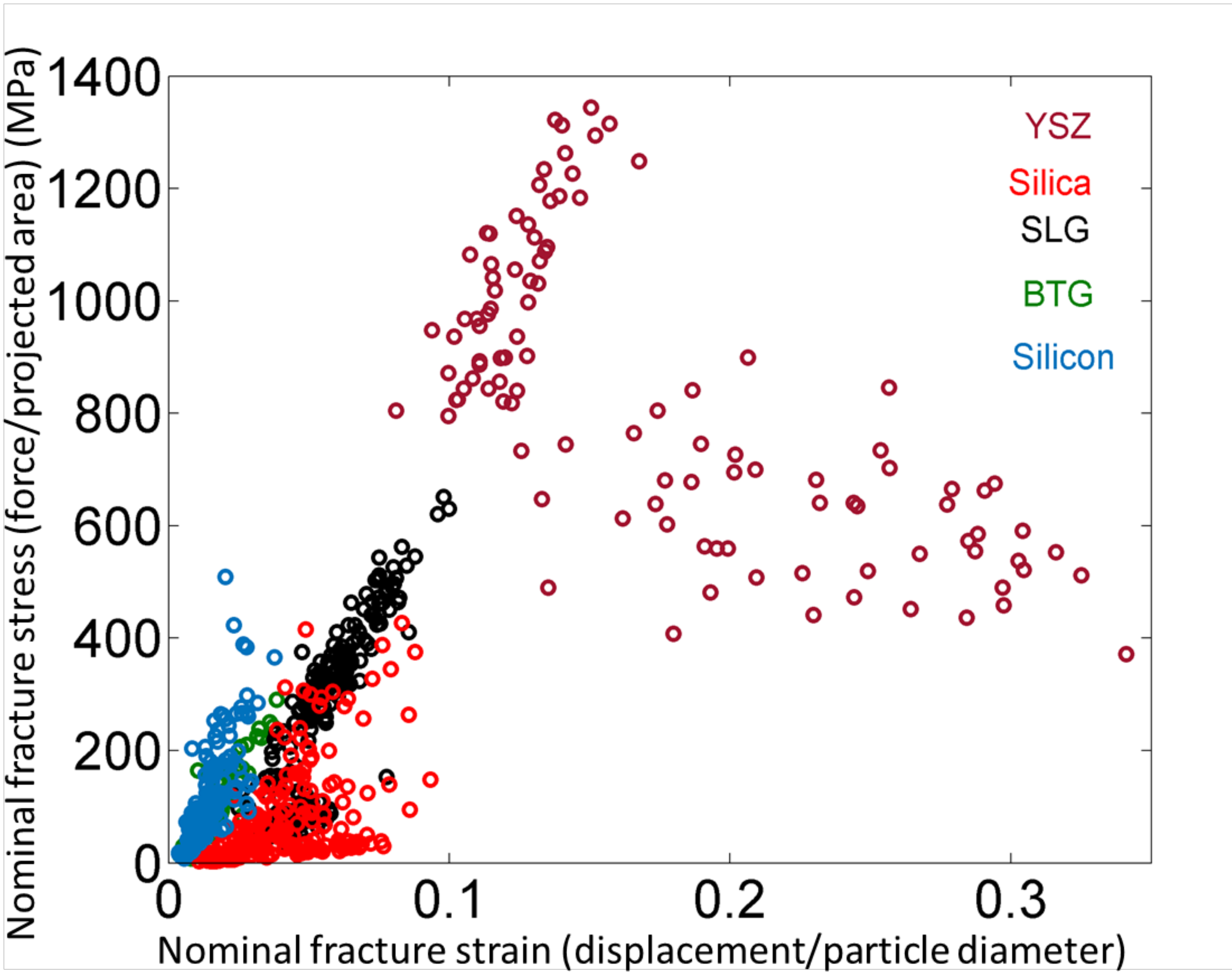

Figure 2: Fracture modes for soda lime glass particles

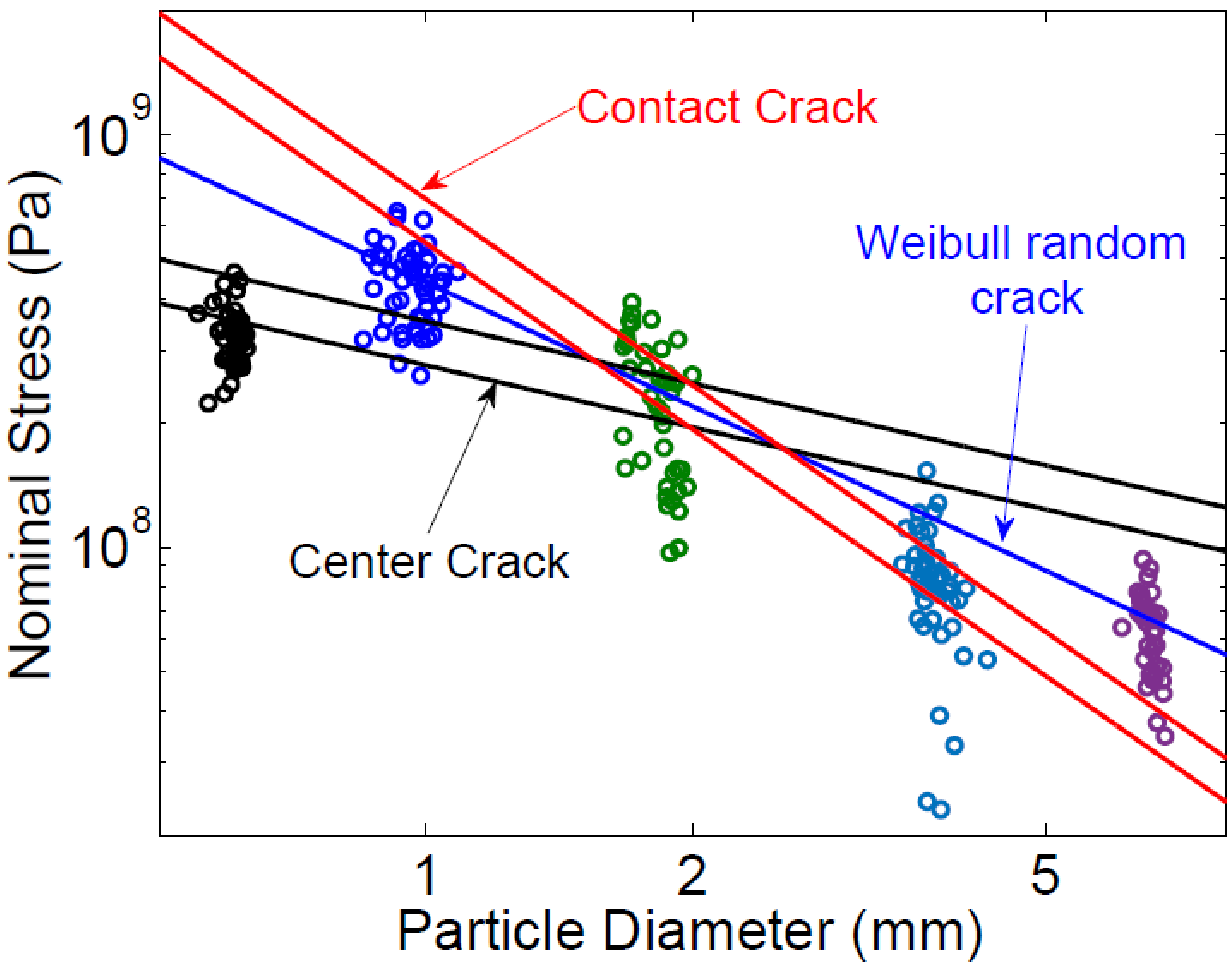

Figure 3: Schematic of high speed X-ray PCI experiments to observe the failure modes in particles under dynamic compression

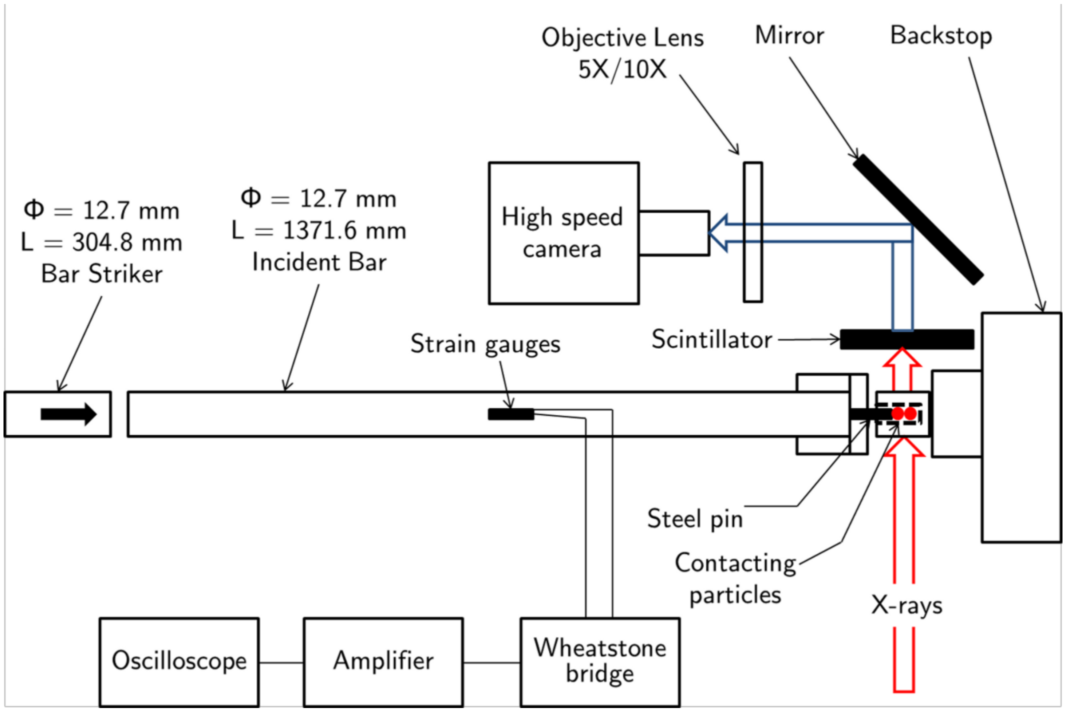

Figure 4: Photograph of the experimental setup. (1) Gas gun barrel that is used to propel the striker bar towards incident bar. (2) Incident bar. (3) Sample mounting assembly. (4) High speed camera. (5) Scintillator-mirror-optical lens assembly. (6) Wheatstone bridge. (7) Oscilloscope. (8) Differential amplifier for the strain gauge signal. (9) Differential amplifier for the load cell signal. **Inset:** Details of pin-holder-load cell assembly (a) Steel pin used for compressing the particles. (b) Particle holder. (c) Load cell. (d) Back stop

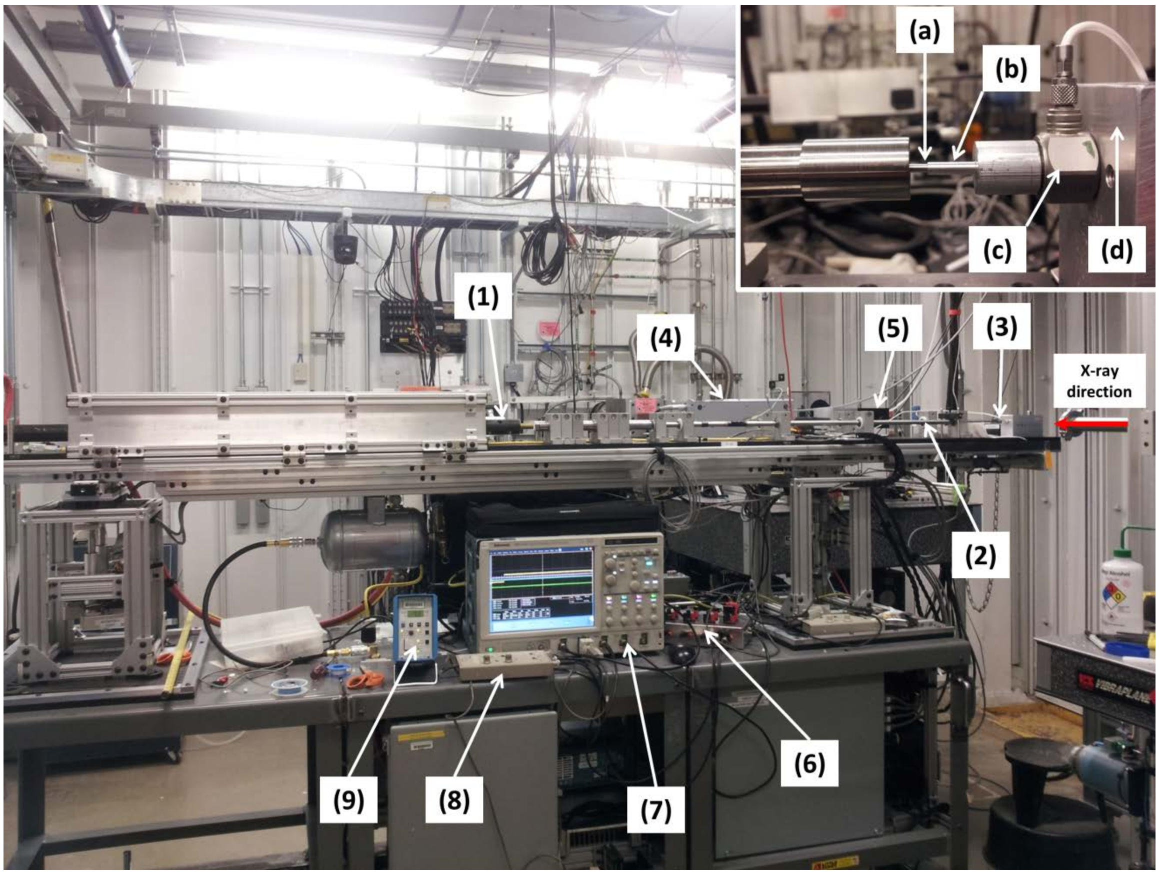

Figure 5: The high speed X-ray images for single particle compression. For all particles, the cracking initiates near the center of the particle. The crack propagation and subsequent fragmentation behavior differs based on the material of the particles. In soda lime glass particles, the cracks bifurcate rapidly as they propagate thus fragmenting the particles in large number of small fragments. The silicon dioxide, silicon, and barium titanate glass particles fragmented in roughly hemispherical fragments which further cracked in finite number of large fragments. PMMA particle also separated in roughly hemispherical fragments.

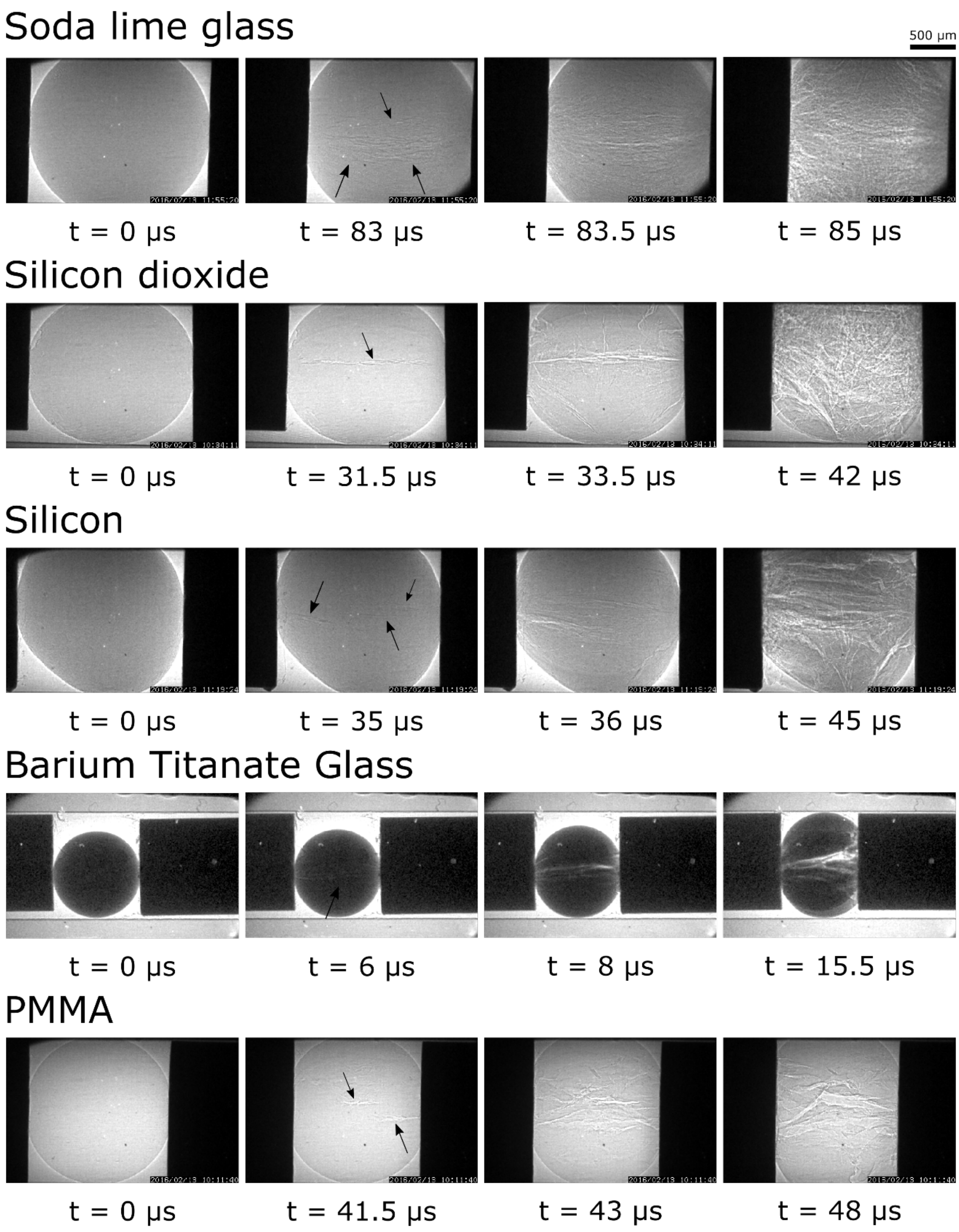

Figure 6: The high speed X-ray images for two particle compression. For all materials except the PMMA and YSZ, the cracking initiated near the particle-particle contact in the form of Hertzian cone crack. In soda lime glass particles, the cone crack propagated to separate a fragment from the particle. On further compression, large numbers of cracks initiated in the volume of the particle and rapidly bifurcated thus fragmenting the particles in large number of small fragments. The silicon dioxide, silicon, and barium titanate glass particles fragmented in finite number of fragments. Cracking initiated near the center of one of the particles for the PMMA and yttria stabilized zirconia. The crack bifurcated near the particle-particle contact thus fracturing the particle in two approximately hemispherical fragments and conical fragments near the contact

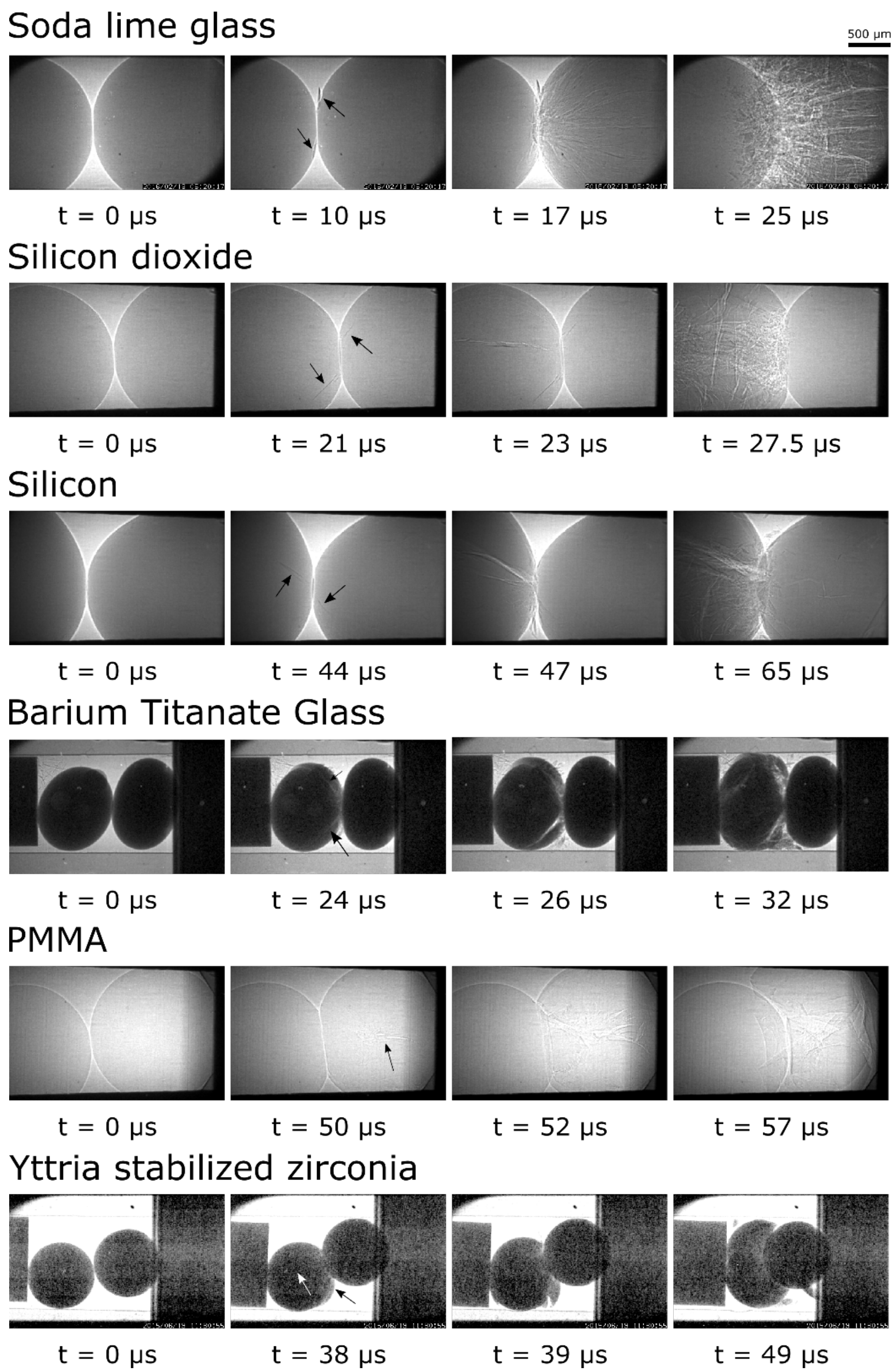

Figure 7: The high speed X-ray images for multi-particle compression. The cracking initiated near the contact points for soda lime glass particles and near the center for PMMA particles. The particles in glass particles spanned the contact points and ultimately particle with maximum particle-particle contacts fragmented explosively. For PMMA particles, the particle fractured in two hemispherical fragments.

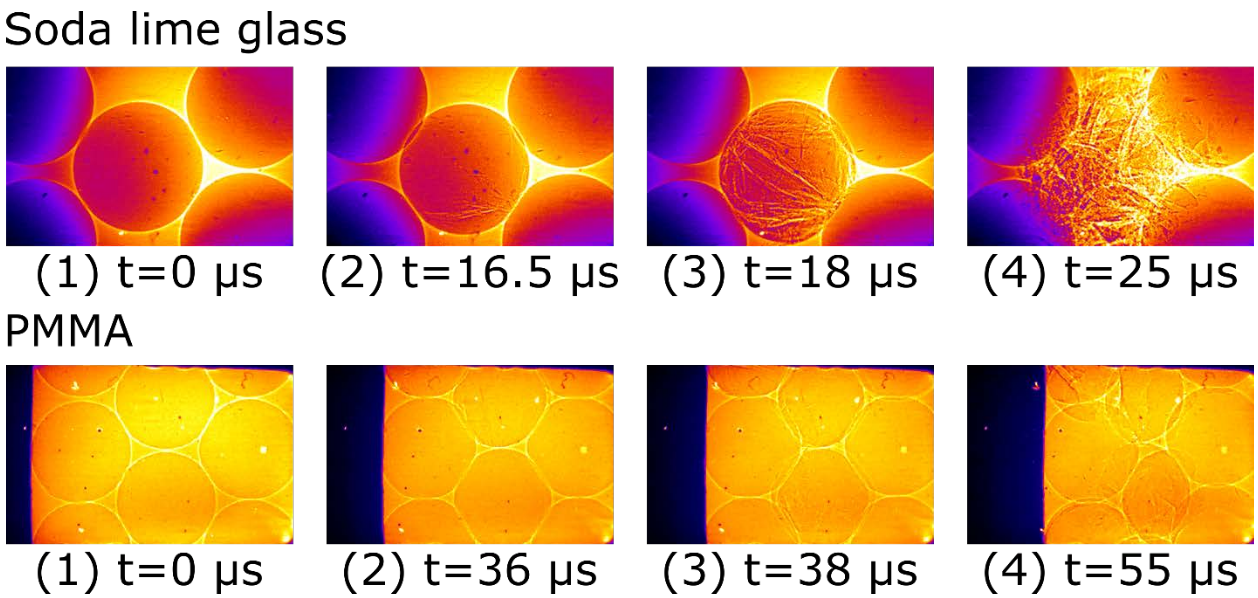

Figure 8: Pulverization parameter plotted for all particles studied here. The pulverization parameter decreases as the severity of cracking is decreased (from soda lime glass to YSZ)