44.0 ADVANCED CHARACTERIZATION OF PARTICULATE MATERIALS SIMULATING HIGH EXPLOSIVES

Summer Camerlo (Mines)
Faculty: Amy Clarke (Mines), Kester Clarke (Mines)

This project initiated in Fall 2020 and is supported by the Center for Micromorphic Multiphysics Porous and Particulate Materials Simulations with Exascale Computing Workflows, U.S. Department of Energy (DOE), National Nuclear Security Agency (NNSA), Office of Advanced Simulation and Computing (ASC), ASC Predictive Science Academic Alliance Program III (PSAAP III). It involves collaboration with the University of Colorado Boulder and other academic partners, in addition to the three NNSA national laboratories: Los Alamos National Laboratory (LANL), Sandia National Laboratories (SNL), and Lawrence Livermore National Laboratory (LLNL). The experimental work performed during this project will serve as the basis for a Master's thesis program for Summer Camerlo.

44.1 Project Overview and Industrial Relevance

Traditional plastic bonded explosives or high explosives (HEs) require extensive safety precautions in handling and testing environments. HEs are of great interest to the NNSA, as well as other U.S. Department of Defense labs. Current knowledge of HEs is limited, from processing to thermo-mechanical behavior states for virgin and recycled HEs. Thus, there is a need for robust characterization of HEs to inform predictive modeling of HE behaviors [44.1].

By using materials that emulate characteristics of traditional HEs for simulations and other non-critical testing, a considerable amount of time and money can be saved. This project is intended to provide a robust characterization of a mock high explosive (MHE) developed by LANL that contains angular idoxuridine (IDOX) crystals suspended in an estane binder matrix. Features like particle size, shape, and distribution are of interest, in addition to the processing of these materials and their quasi-static to dynamic mechanical response. In this project, experiments will link MHE characteristics to processing and properties, building a database of information to inform multiscale computational efforts underway at the University of Colorado Boulder. Due to the highly sensitive and costly nature of experimental studies, the availability of predictive computational models will enable parametric studies of these materials under a broader range of conditions potentially including different strain rates, shock loading, cyclic loading, and their thermal dependence [44.2]. The end result will be a comprehensive database of in-situ/ex-situ quasi-static and high strain-rate compression testing data, with x-ray radiography and computed tomography (CT) whenever possible to fully characterize the particle/matrix characteristics. In this project, we will simulate with quantified uncertainty the deformation response of MHEs, which can then be modified to represent full plastic bonded explosives of interest to the NNSA labs.

44.2 Previous Work

Previous work has been completed at LANL, SNL, and LLNL. For example, different MHEs have been used to simulate traditional explosives such as PBX 9501. Previous MHE's tested include LX-17 and PBX 9502. Ferranti et al. used reverse Taylor impact experiments to measure the dynamic behavior of MHEs and to evaluate their dynamic loading response [44.3]. By establishing viable experimental techniques such as this, the development and validation of computational modeling becomes much more practical. The inclusion of even minor elastic strains becomes important for MHEs, as they are predominantly polymeric.

Although other deformation mechanisms are still being quantified, fracture in HEs and MHEs has been studied extensively and can lead to failure prediction through analysis of crack initiation. Through utilization of digital image correlation (DIC), researchers at LANL were able to quantitatively identify the location and extent of macroscopic cracks [44. 4]. A simple three-point bend test was performed; however, the specimen was not pre-cracked according to ASTM standards. Because of the extremely elastic behavior and difficulty seeing fracture in MHEs due to their heterogeneity and elastic behavior, DIC was necessary to obtain information about the local deformation.

The first objective of this new project was to develop an experimental and computational framework to study MHE. For this, baseline data must be obtained by experiments and modeling of epoxy embedded with particles to serve as model systems. This data will enable the computational framework to be developed and calibrated before moving on to the MHE of interest to NNSA. Several different epoxies were initially tested and pared down by their viscosity and ease of use. The current epoxy, Hexion EPIKOTE RIMR 135, was chosen because when heated, it flows easily. This flow is necessary when adding in the particles. The "model" particles are glass beads of varying sizes, representing simple spherical particles embedded in the epoxy matrix, as well as fine, angular sand, which better represents the IDOX crystals in size and shape found in the LANL MHE. Right cylindrical samples were created for subsequent baseline mechanical testing and characterization by x-ray radiography and tomography.

After determining the epoxy type to be used for initial sample preparation, characterization, and mechanical testing, a variety of sample sizes were created using a silicone molding kit. Void formation in the epoxy that could impact mechanical response was reduced by creating the samples in a positive pressure chamber, ensuring less void formation as the epoxy cures. The epoxy samples were then lightly sanded to ensure good visibility of the particles embedded in the matrix.

After being created, selected epoxy samples were characterized with computed tomography and mechanically tested. Quasi-static compression testing revealed that the epoxy samples containing glass particles fractured along ligaments between the glass beads.

Initial computed tomography was performed on an epoxy sample with 3mm aluminum beads. Preliminary samples were constructed using an acrylic tube that was filled with epoxy; however, this is no longer how the samples are prepared. Because acrylic is essentially "invisible" to the X-rays, the sample was utilized to validate the use of CT on the epoxy and produce imaging of the matrix however this method is no longer used because the acrylic tubing has a different mechanical response from the epoxy. In addition, aluminum balls were determined not viable because their mechanical response differs too significantly from IDOX.

44.3 Recent Progress

44.3.1 Creation of recycled MHE samples from machining fines recovered from pristine MHE production

Utilizing die specifications received from LANL, a die was manufactured for use at Mines. The machining fines were compacted in a 100-ton Genesis hydraulic press into 0.5" diameter by 0.25" high cylindrical pucks. These recycled samples are of interest to the NNSA laboratories because the pristine MHE is very costly to make. Showing that recycled MHE samples have similar properties to the pristine samples would allow for usage of the recycled samples and reduced costs relative to creating samples from pristine MHE. That said, the impacts of recycling must be well understood, including factors such as variations in IDOX crystal sizes relative to pristine MHE. The samples were created by following the same steps used by LANL for the creation of pristine samples, except for the initial mixing of IDOX and Estane binder. The fines were weighed before being added to the preheated die at 50°C and then compacted to 2000 lbf. The properties and microstructures of the recycled samples will be compared to pristine IDOX MHE samples received from LANL.

44.3.2 Creation of "dummy" mock HE samples

In addition to the epoxy samples covered in **44.2**, other preliminary samples produced were created by pressing FK-800 resin, a binder typically used for HMX in place of Estane, in the same way as the recycled MHE samples. FK-800 comes in powdered form and can either be chemically dissolved and formed [44.5] or compressed at intermediate temperatures. For ease and safety of creation, the latter method was chosen to produce the resin samples. Compaction of the resin required a higher temperature than that of the recycled MHE for full cohesivity. Plain resin samples were created to undergo mechanical testing to quantify the properties of the binder with no particulate. In addition, F50 sand was mixed in with the resin pre-compaction to simulate embedded IDOX crystals. The sand was added in ratios of 50 wt%, 55 wt%, 60 wt%, 65 wt%, 70 wt%, and 75 wt% in an attempt to match the binder-particulate ratio of IDOX MHEs, which is approximately 95% particulate. That ratio was not quite attainable as the sand is less cohesive compared to IDOX crystals.

44.3.3 Material characterization of recycled MHE, pristine MHE, and other materials of interest

The compressive stress/strain responses of materials of interest were collected using the Gleeble 3500-GTC and a 100 kip MTS load frame. The most significant result was that the recycled MHE specimens have a significantly higher stress response than pristine samples across all strain rates, as shown in **Fig 44.1**. Various mock material responses can be seen in **Fig 44.2**. Another response of interest is that of multiple samples of the FK-800 resin. Six samples were tested at a strain rate of 1/s, and there are significant differnces between samples, as shown in **Fig 44.3**. More testing needs to be done to determine the reason for these differences.

In addition to mechanical testing, SEM was performed on the pristine and recycled MHE samples to quantify the difference in particle size [**Fig 44.4**]. The pristine MHE samples have crystal sizes averaging ~43.6 μm^2 , while the recycled crystal sizes have an average of ~29.7 μm^2. This sizing difference is the most likely cause of the stress response discrepancy between the two sample types.

44.3.4 Computed tomography of recycled MHE, pristine MHE, and other materials of interest

Using the Zeiss Versa 820, computed tomography (CT) of recycled MHE, pristine MHE, F50 sand embedded in FK-800 resin, and glass beads embedded in epoxy was performed. In addition to stationary CT, an *in situ* Deben load frame was utilized to capture post-compression deformation of glass beads in epoxy. The CT scans will be used by the modeling team for segmentation of the IDOX crystals from the binder and visually contrast the recycled and pristine MHEs, as shown in **Fig 44.5**.

44.4 Plans for Next Reporting Period

- Internship at LANL with a focus on the creation and material characterization of new materials to simulate High Explosive and their mechanical properties
- Thorough literature review
- More *in situ* CT of recycled and pristine MHE, including CT during pressing to simulate processing
- Improve and streamline image segmentation process to acquire quantitative information about particle/binder assemblies

44.5 References

[44.1] J. Brown, A.C. Co-director, PSAAP III Statement of Work (1) 4, (2020).

[44.2] J.D. Yeager, C.S. Woznick, D.G. Thompson, A.L.H. Duque, C. Liu, A.C. Burch, P.R. Bowden, M. Shorty, D.F. Bahr, Development of a new density and mechanical mock for HMX, Proc. 16th Int. Detonation Symp. (2018) 1631–1641.

[44.3] L. Ferranti, F.J. Gagliardi, B.J. Cunningham, K.S. Vandersall, Dynamic characterization of mock explosive material using reverse Taylor impact experiments, Conf. Proc. Soc. Exp. Mech. Ser. 6 (2011) 337–346. https://doi.org/10.1007/978-1-4419-9792-0_56.

[44.4] C. Liu, C.M. Cady, P.J. Rae, M.L. Lovato, On the Quantitative Measurement of Fracture Toughness in High Explosive and Mock Materials, 836 (2010).

[44.5] 3M Specialty Materials, FK-800 Resin, (n.d.).

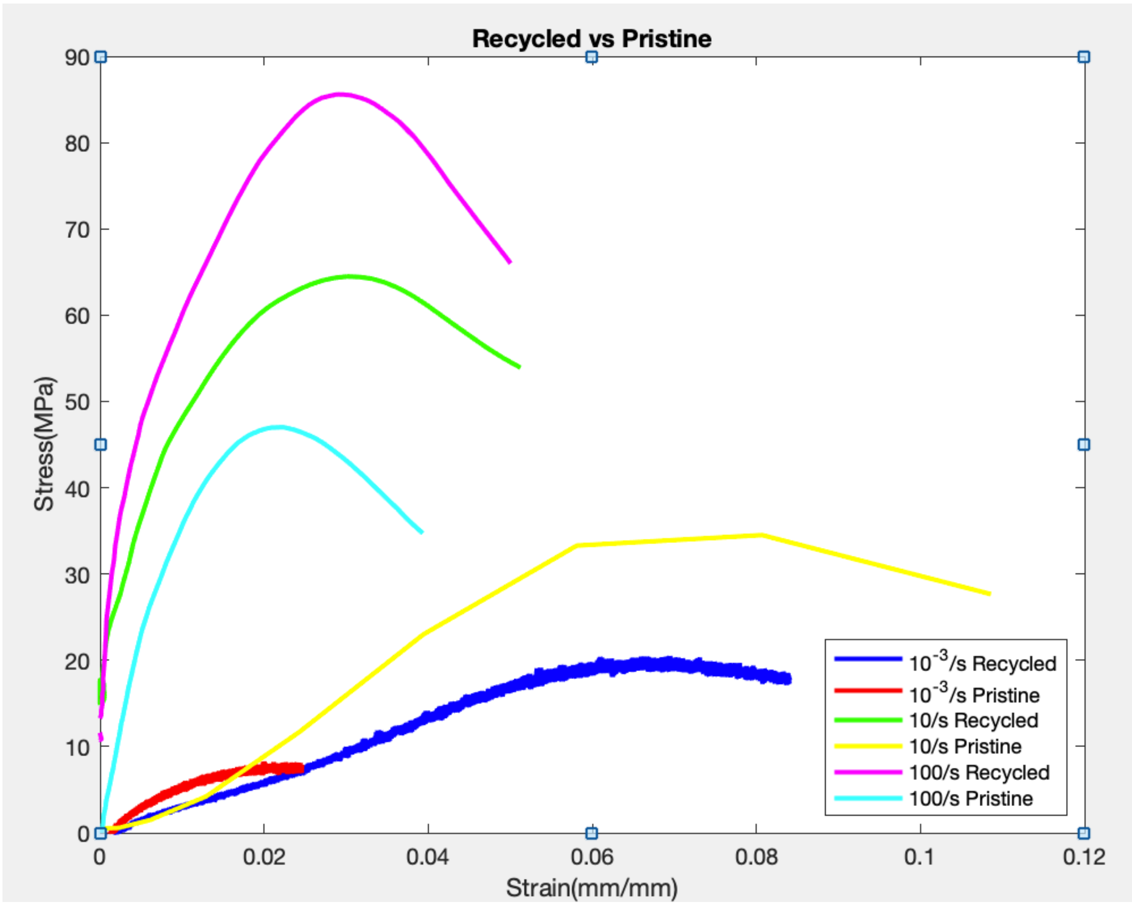

Figure 44.1: Stress v. Strain plots from compression testing of pristine LANL MHE and recycled MHE

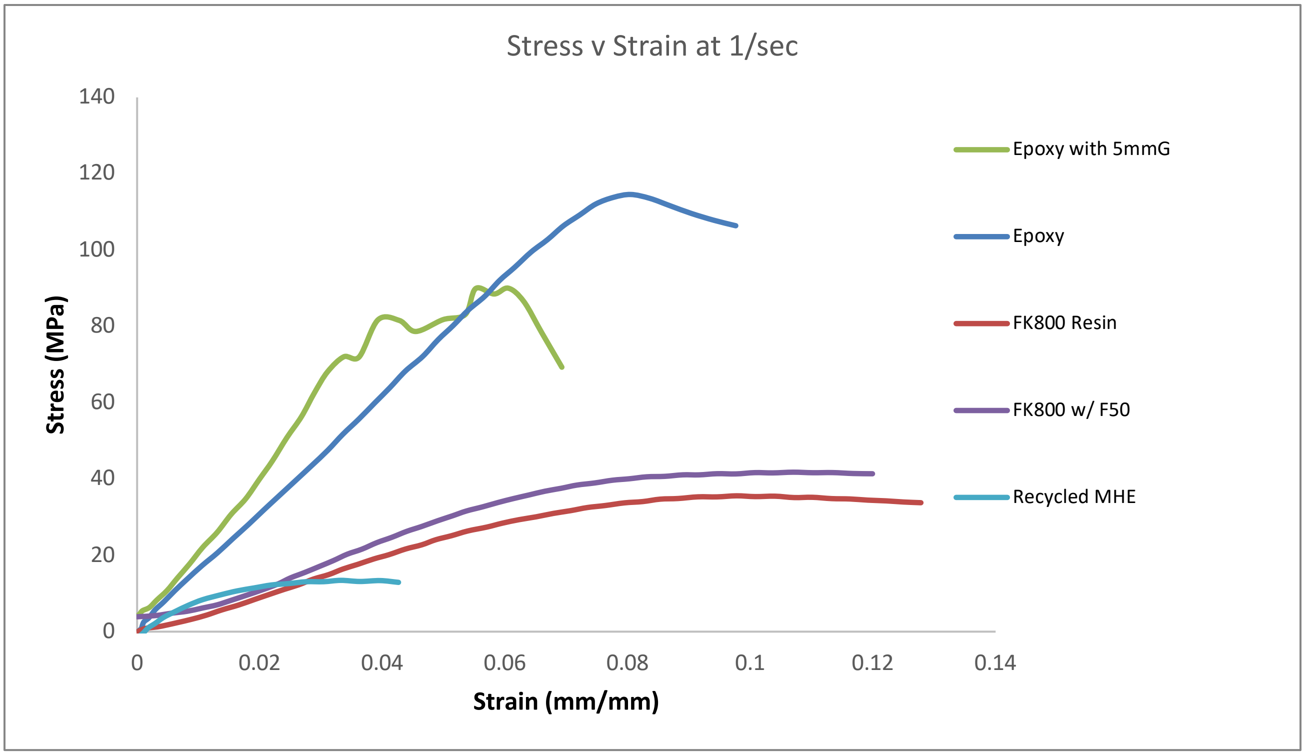

Figure 44.2: Stress v. Strain plots from compression testing of preliminary mock HE materials and recycled MHE

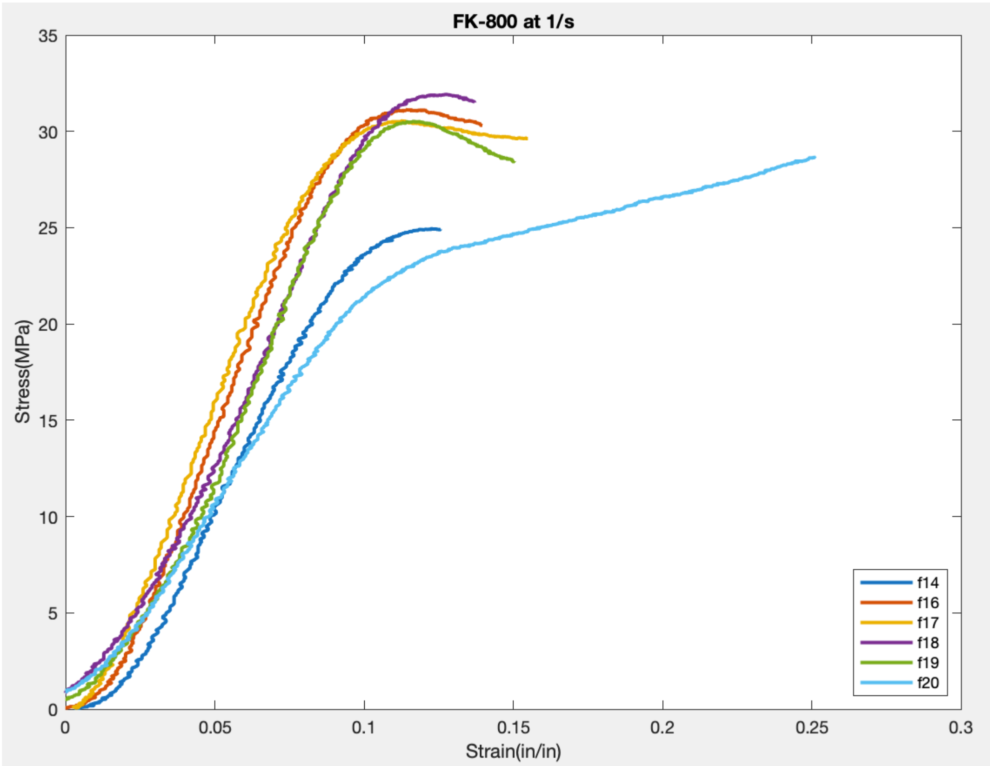

Figure 44.3: Stress v. Strain plots from compression testing of geometrically identical FK-800 samples (cylindrical, 0.25” high, 0.5” diameter)

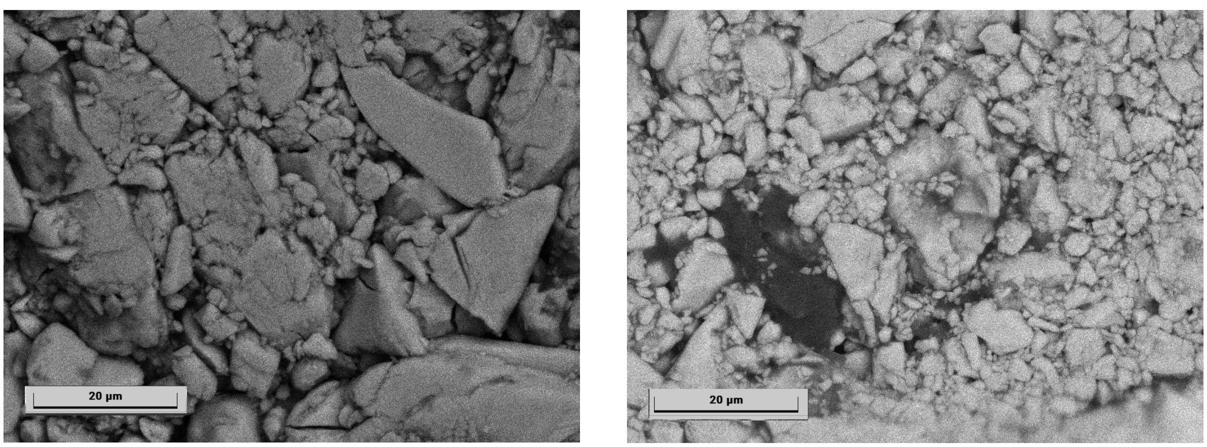

Figure 44.4: SEM imaging of pristine MHE (left) and recycled MHE (right)

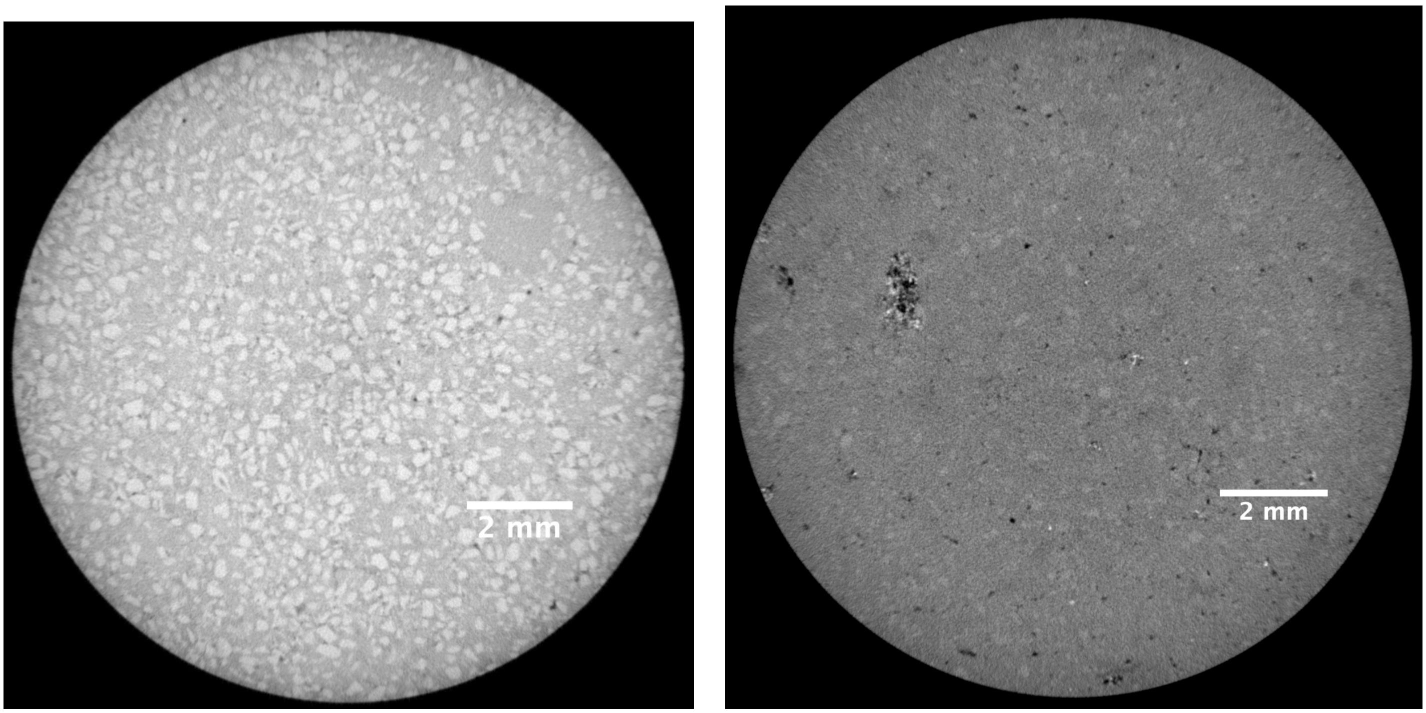

Figure 44.5: Cross section through a CT image of pristine MHE (left) and recycled MHE (right)