



Effect of Time and Acceleration on Particle Size Refinement of Pyrotechnic Constituents During Processing in a Resodyn Mixer

Presented to:

CAD/PAD Technical Workshop

Presented by:

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Abstract

In recent years, Resonant Acoustic Mixers (RAM) have been widely investigated for mixing of pyrotechnics, propellants, and explosives. Several factors, such as mixing time, acceleration, vessel temperature and pressure, as well as aspect ratio, are critical parameters affecting mixing quality.

In this R&D effort, we investigated the effect of time and acceleration in a LabRAM I on particle size refinement of pyrotechnic constituents. The focus was on refinement of two different oxidizers, having different particle size distributions, when exposed to stainless steel (SS) and yttria-stabilized zirconia (Y_2O_3 - ZrO_2) spherical particles. Specifically, SS particles with a narrow 5 to 10 μm range, Y_2O_3 - ZrO_2 microspheres with 45 – 53 μm range, as well as spherical 400 μm and 800 μm Y_2O_3 - ZrO_2 grinding media were used in our experiments.

This presentation contains the results of particle size analyses of selected powders, using MicroTrac Sync, when exposed to different processing conditions in the LabRAM I. In addition, the presentation will be supplemented with SEM photographs of processed powders.

Problem

Customer concerned that when mixing in the RAM, the harder fuels would have an effect on the particle size of a fragile oxidizer.

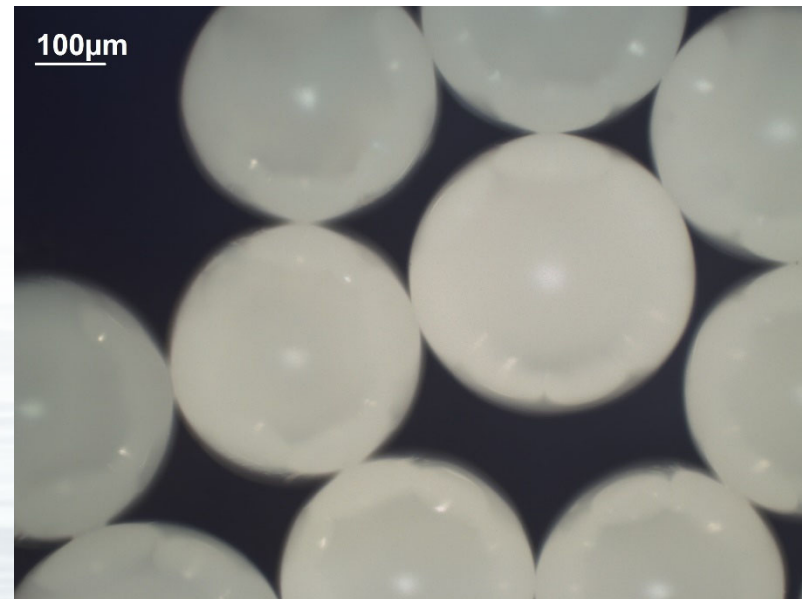
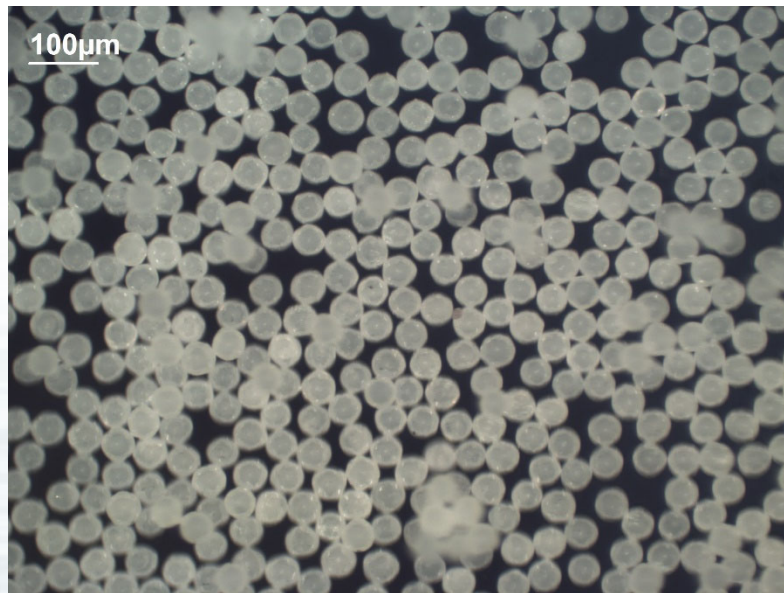
Methodology

- Cut inert media to narrow particle size distribution (PSD)
 - Measure PSD
- Accelerate media with target material of concern
 - Mixing duration = 2 hours
 - Vary acceleration from 30 g to 90 g
- Measure PSD of target at each acceleration point or time interval

Milling Material

- To examine the effect of milling in the RAM three media were chosen:
 - Cospheric Yttrium Stabilized 45 to 53 μm Spherical Zirconia
 - IMP Yttrium Stabilized 400 μm average Spherical Zirconia milling media
 - IMP Yttrium Stabilized 800 μm average Spherical Zirconia milling media

To simulate the worst case conditions, the subject material will experience in the RAM the compositions were 50:50 ratios with respect to volume

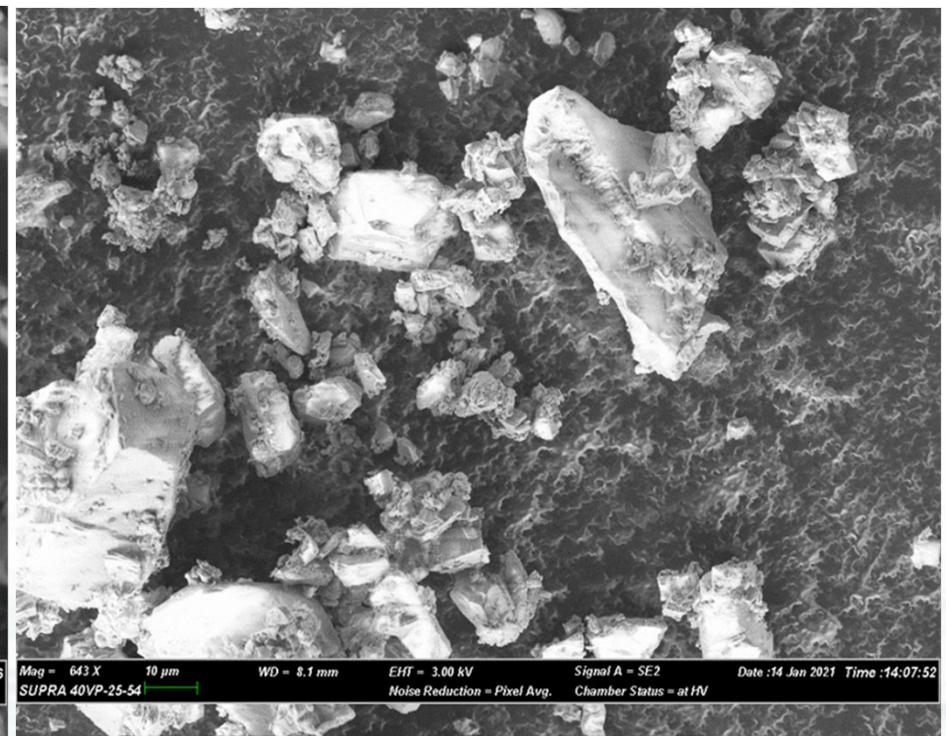
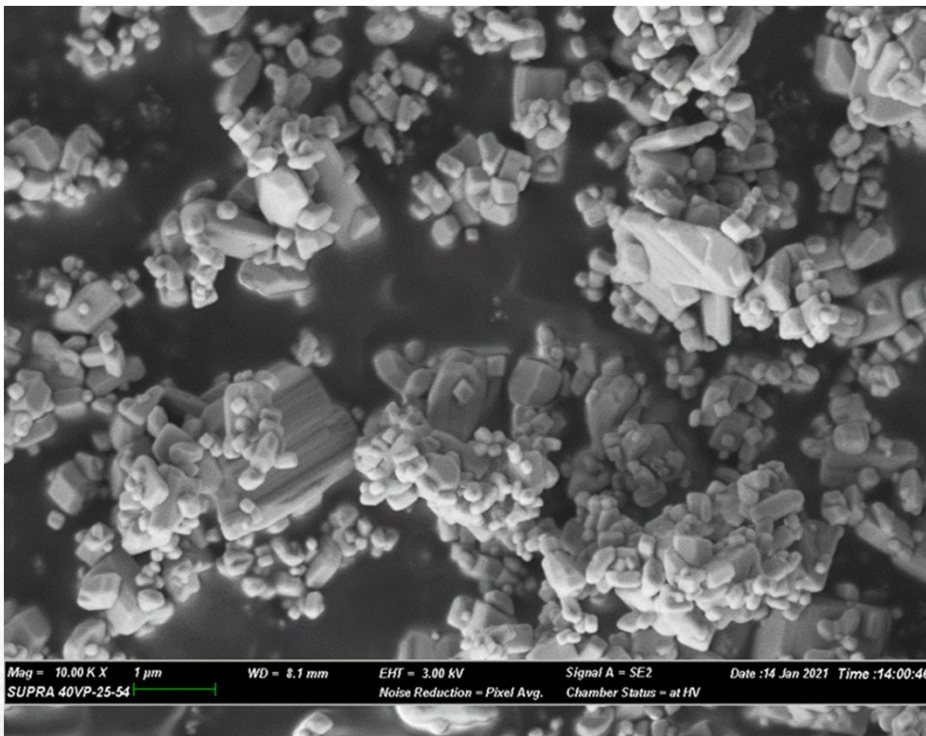


Target Materials

- BaCrO_4 (MIL-B-550 Class A) powder.
- KClO_4 (MIL-P-217 Granulation IV) powder.

Left SEM photograph

Right SEM photograph



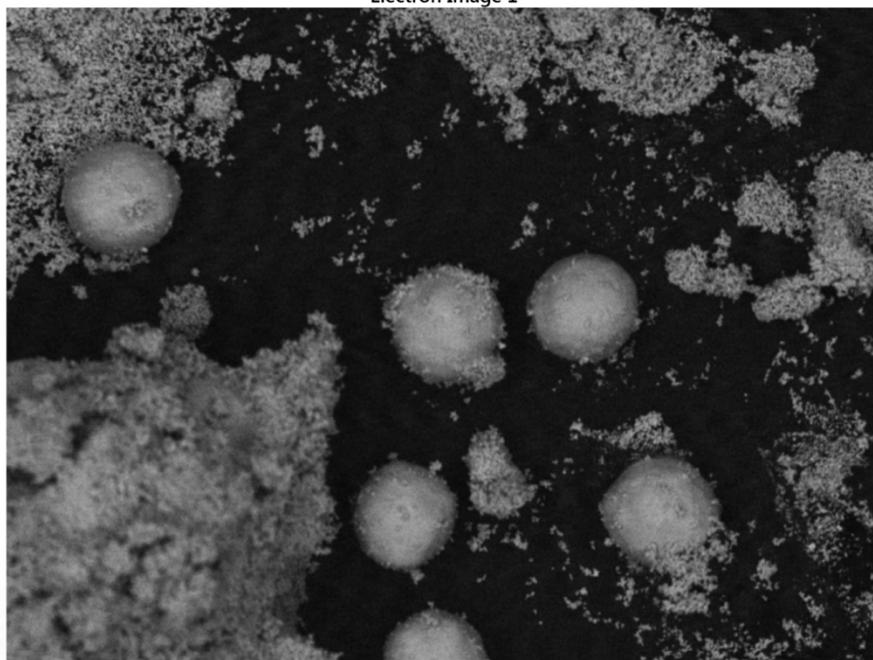
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Milling Material

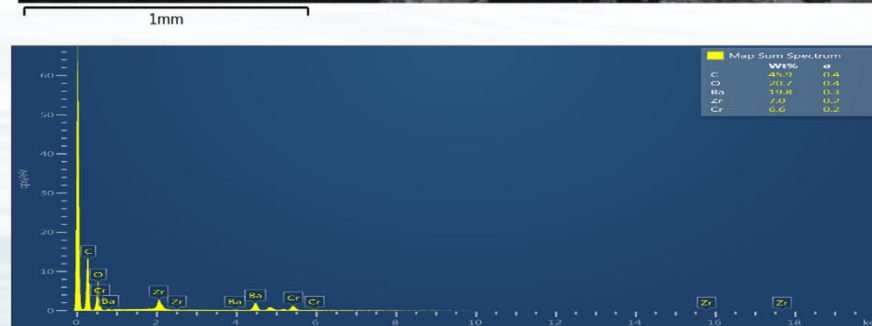
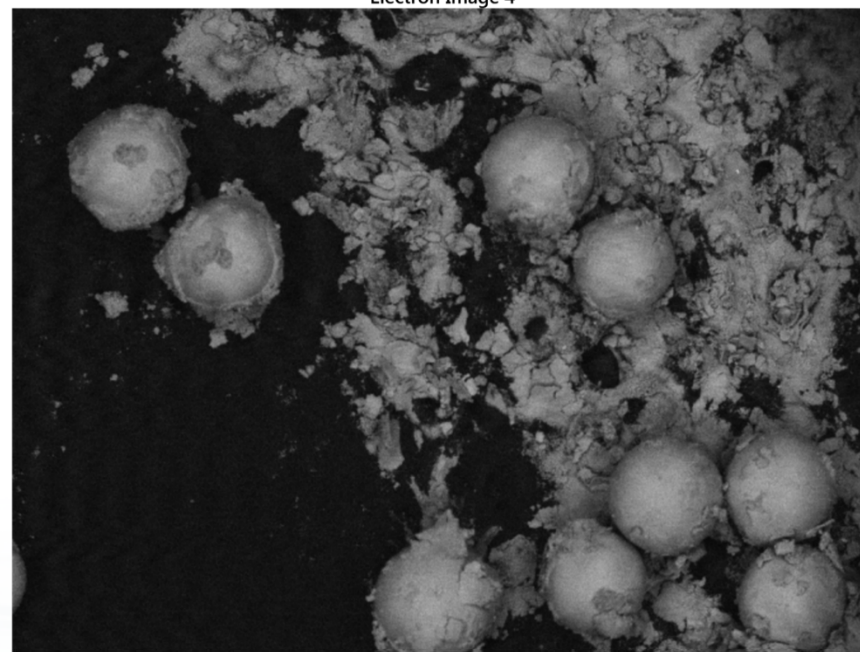
**BaCrO₄ and ZrO₂ spherical media
(45-53 μm) 50-50% by volume.**

Electron Image 1



**BaCrO₄ and ZrO₂ spherical media
(400 μm) 50-50% by volume.**

Electron Image 4



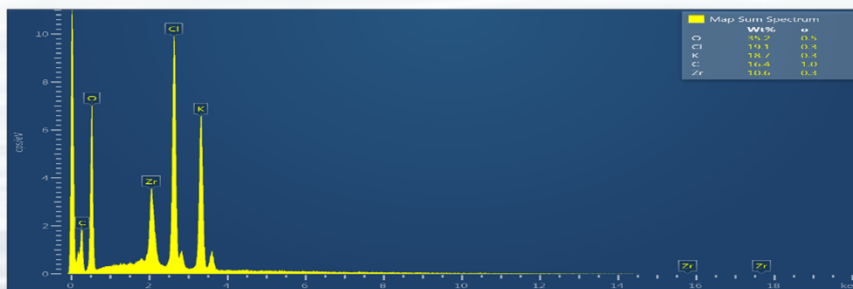
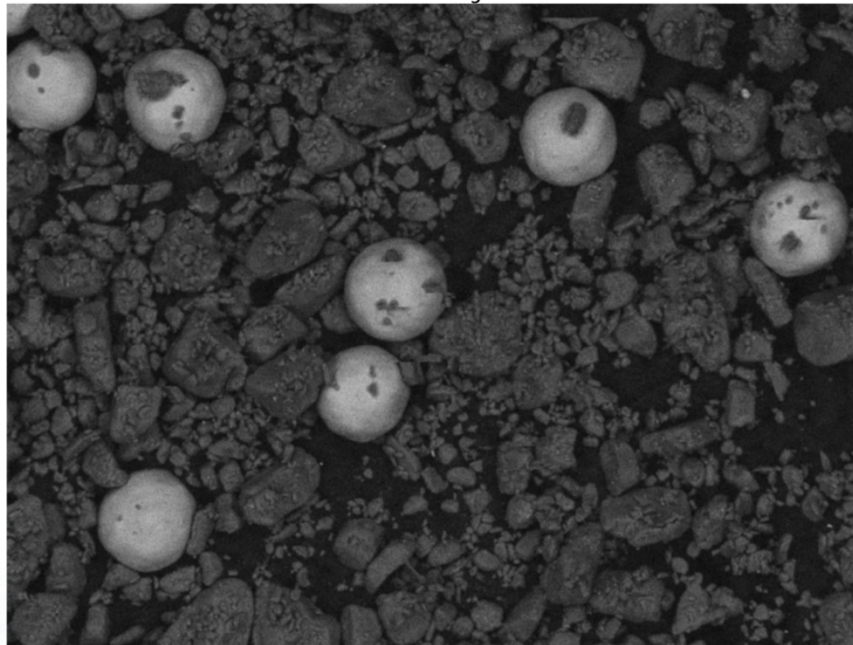
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Milling Material

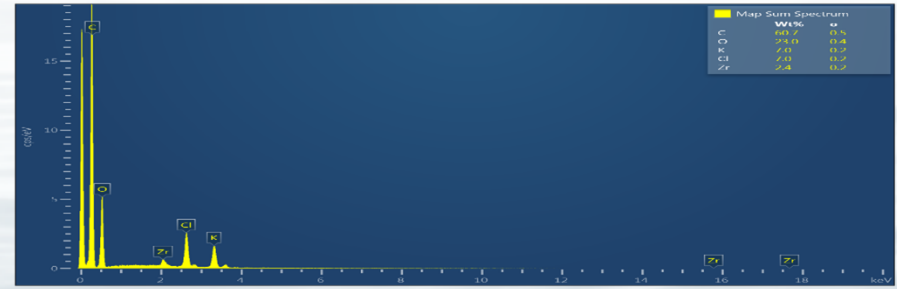
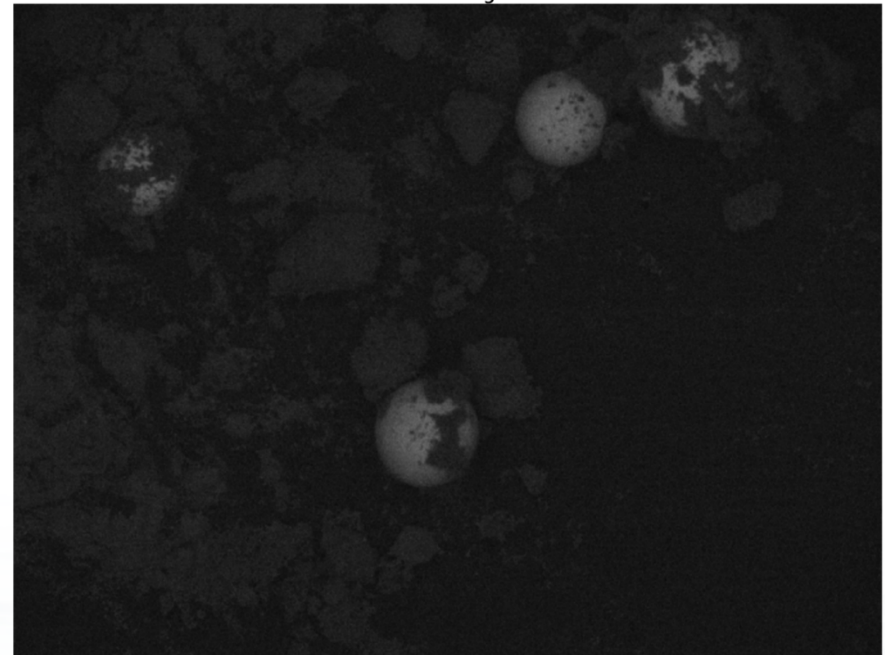
**KClO₄ and ZrO₂ spherical media
(45-53 μm) 50-50% by volume.**

Electron Image 4



**KClO₄ and ZrO₂ spherical media
(400 μm) 50-50% by volume.**

Electron Image 8



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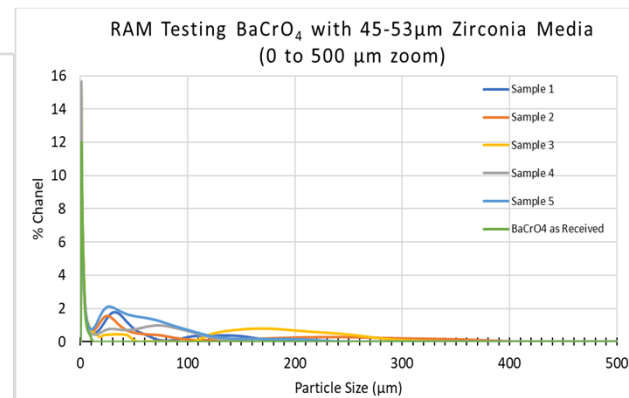
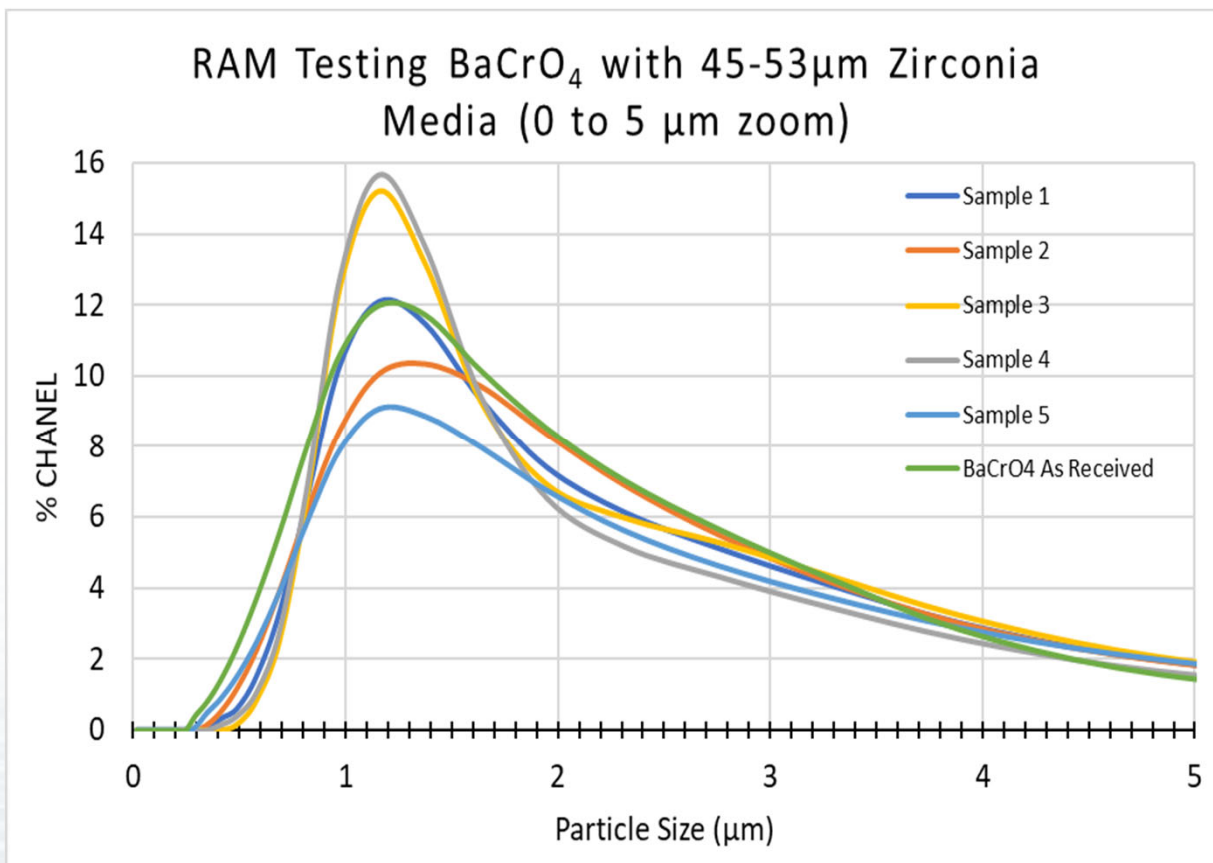
Photo Credits: Innovative Materials and Processes

Duration Testing

- A LabRAM 1 was used to test milling of particle sizes of BaCrO_4 and KClO_4 .
- The composition was loaded into a small stainless steel mixing vessel and installed onto the RAM.
- The use of cooling water was utilized to keep the vessel temperature held at 18 °C.
- Each of four currently tested compositions were subjected to 2 hours of near 100 times gravity of acceleration (g). The RAM recipe was programmed as follows.
 - 90% power for 10 minutes
 - Sample 1 retrieval interval – 0% power for 30 seconds (10 min total test time)
 - 90% power for 20 minutes
 - Sample 2 retrieval interval – 0% power for 30 seconds (30 min total test time)
 - 90% power for 30 minutes
 - Sample 3 retrieval interval – 0% power for 30 seconds (60 min total test time)
 - 90% power for 30 minutes
 - Sample 4 retrieval interval – 0% power for 30 seconds (90 min total test time)
 - 90% power for 30 minutes
 - Sample 5 retrieval (120 min total test time)

Duration Testing; BaCrO_4 with 45-53 μm zirconia media

Plot from 0 to 5 μm showing BaCrO_4 particle size distributions. Five samples: 1 at 10 minutes, 2 at 30 minutes, 3 at 60 minutes, 4 at 90 minutes, and 5 at 120 minutes. Also included is the PSD of BaCrO_4 as received from the manufacturer.

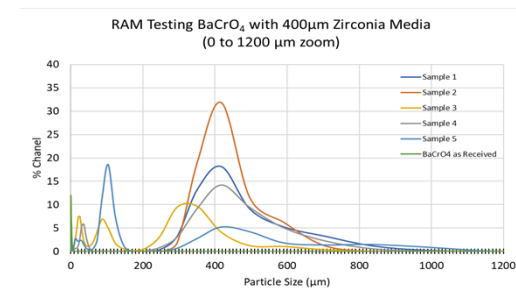
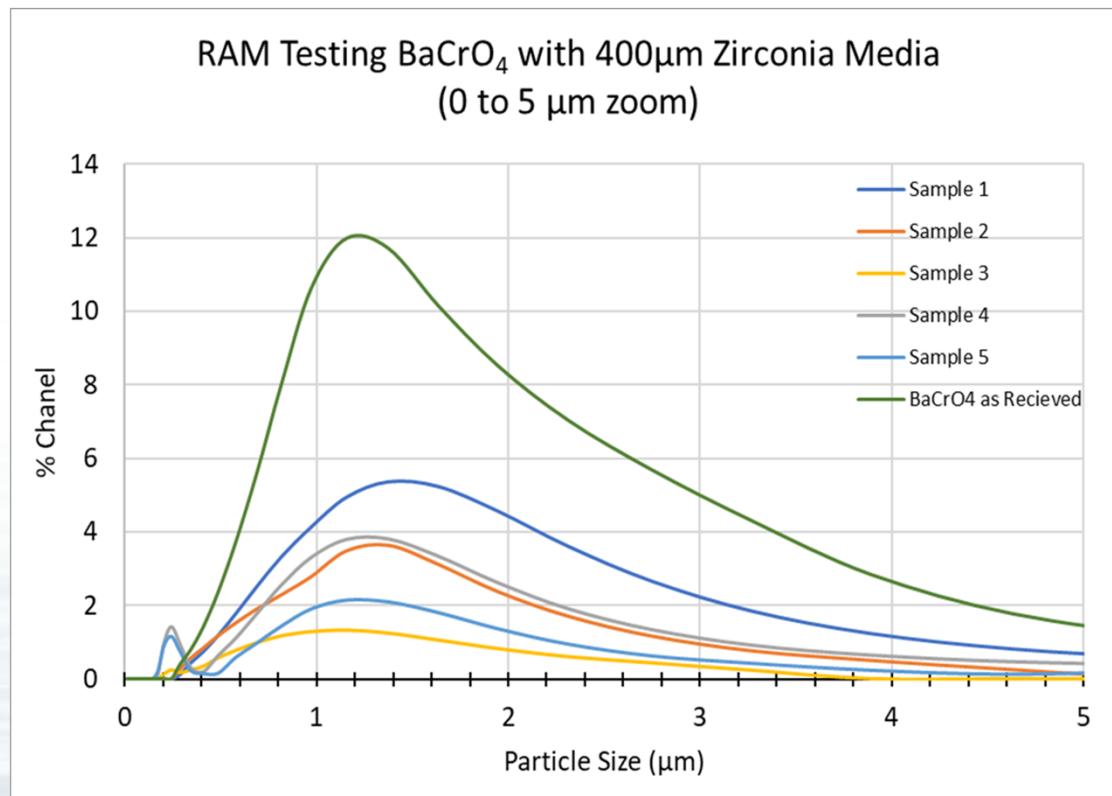


Time (m)	APS (D50)	Min (μm)	Max (μm)
10	23.87	0.972	124.5
30	17.52	1.375	124.5
60	22.78	1.635	124.5
90	23.11	1.945	124.5
120	20.97	1.375	124.5

10
*Particle size distribution
is maintained*

Duration Testing: BaCrO_4 with 400 μm zirconia media

Plot from 0 to 5 μm showing BaCrO_4 particle size distributions. RAM milling test 2; BaCrO_4 with 400 μm zirconia media. Five samples: 1 at 10 minutes, 2 at 30 minutes, 3 at 60 minutes, 4 at 90 minutes, and 5 at 120 minutes. Also included is the PSD of BaCrO_4 as received from the manufacturer.



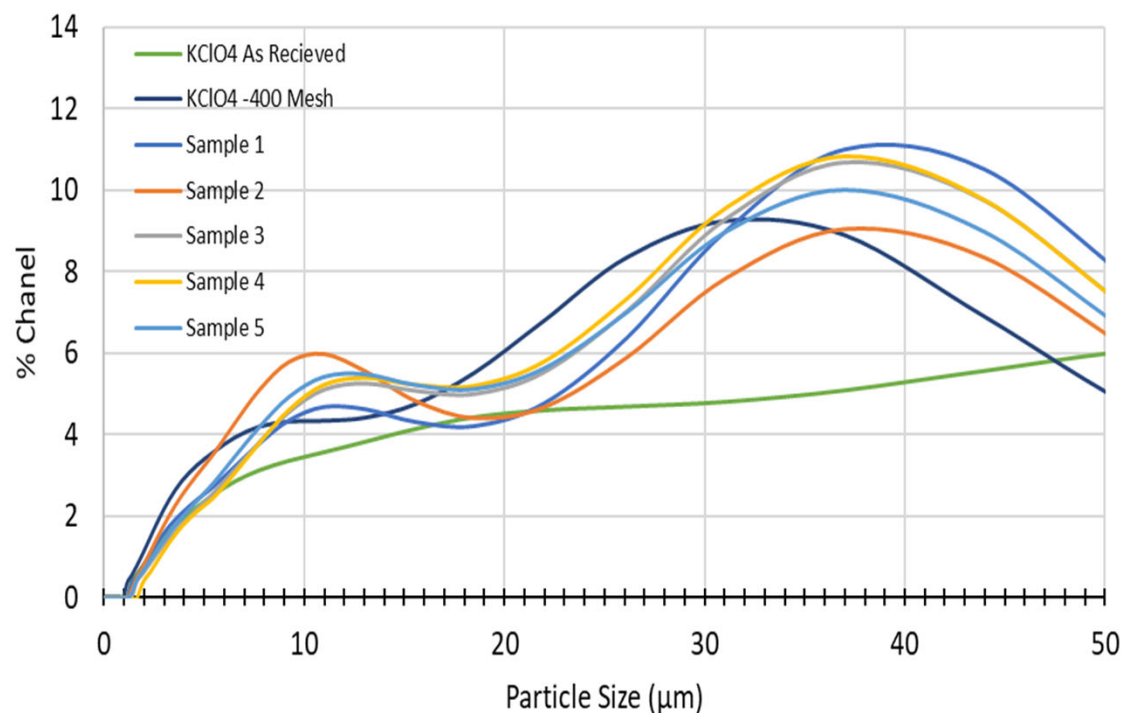
Time (m)	APS (D50)	Min (μm)	Max (μm)
10	6.56	0.972	1184
30	7.30	0.972	995.6
60	310.1	1.375	995.6
90	6.27	0.972	995.6
120	5.69	1.156	1184

Particle size distribution is maintained until longer than 90 minutes

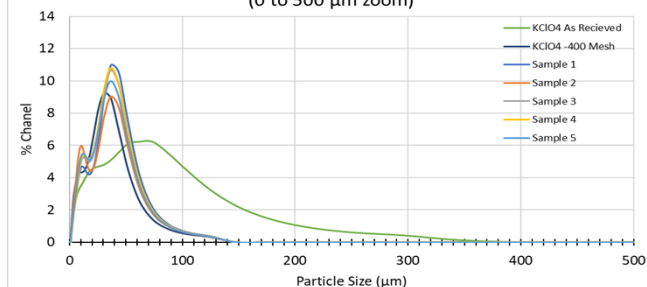
Duration Testing; KClO_4 with 45-53 μm zirconia media

Plot from 0 to 5 μm showing KClO_4 particle size distributions. Five samples: 1 at 10 minutes, 2 at 30 minutes, 3 at 60 minutes, 4 at 90 minutes, and 5 at 120 minutes. Also included is the PSD of KClO_4 as received from the manufacturer.

RAM Testing KClO_4 with 45-53 μm Zirconia Media
(0 to 50 μm zoom)



RAM Testing KClO_4 with 45-53 μm Zirconia Media
(0 to 500 μm zoom)

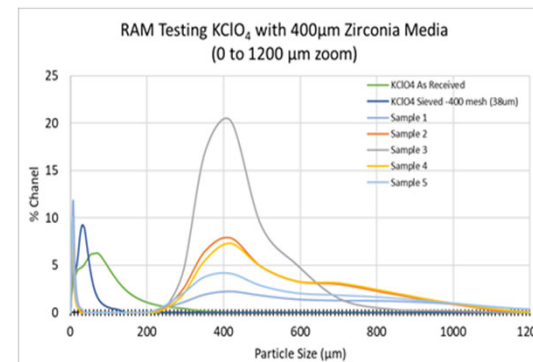
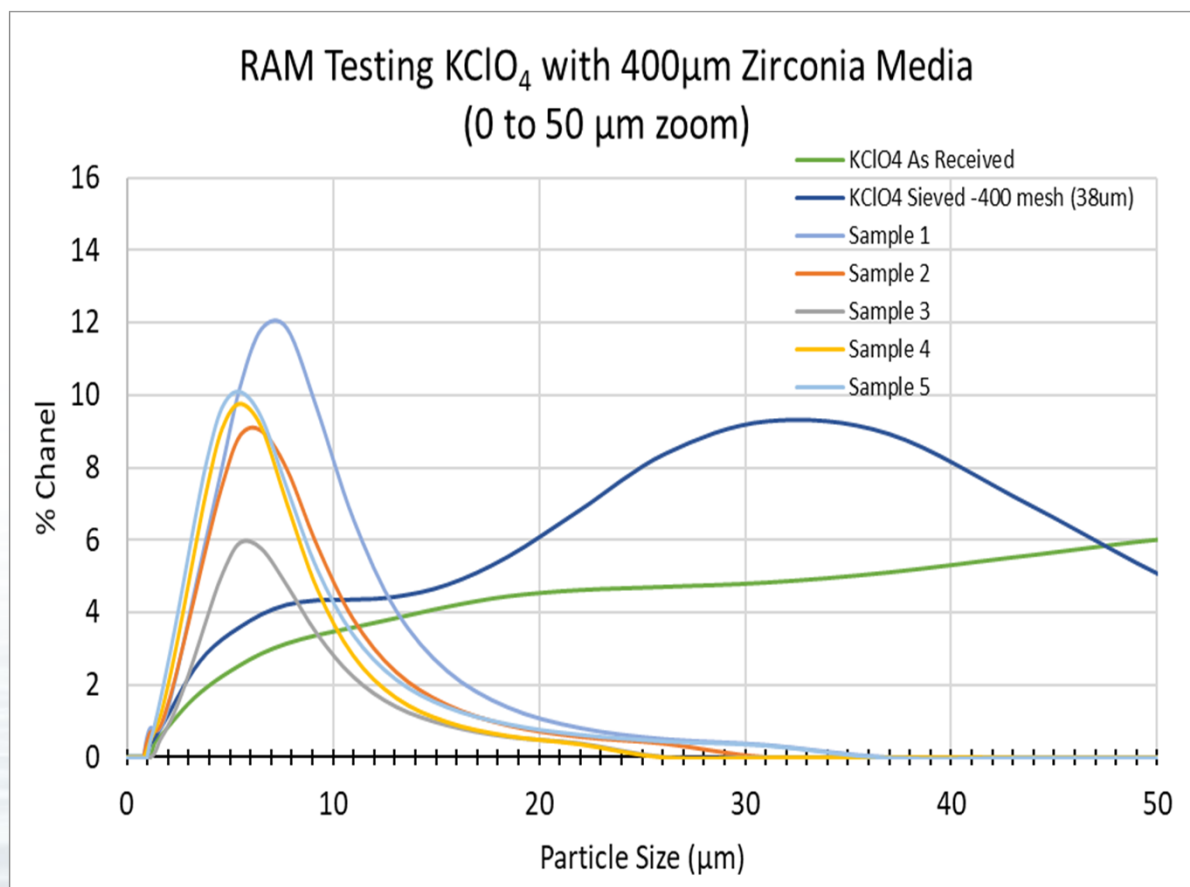


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90	23.11	1.945	124.5
120	20.97	1.375	124.5

Particle size distribution is maintained

Duration Testing; BaCrO_4 with 400 μm zirconia media

Plot from 0 to 5 μm showing KClO_4 particle size distributions. RAM milling test 2; KClO_4 with 400 μm zirconia media. Five samples: 1 at 10 minutes, 2 at 30 minutes, 3 at 60 minutes, 4 at 90 minutes, and 5 at 120 minutes. Also included is the PSD of KClO_4 as received from the manufacturer.



Time (m)	APS (D50)	Min (μm)	Max (μm)
10	6.56	0.972	995.6
30	7.30	0.972	995.6
60	310.1	1.375	995.6
90	6.27	0.972	995.6
120	5.69	1.156	995.6

*Particle size distribution
modified within the first 5
minutes*

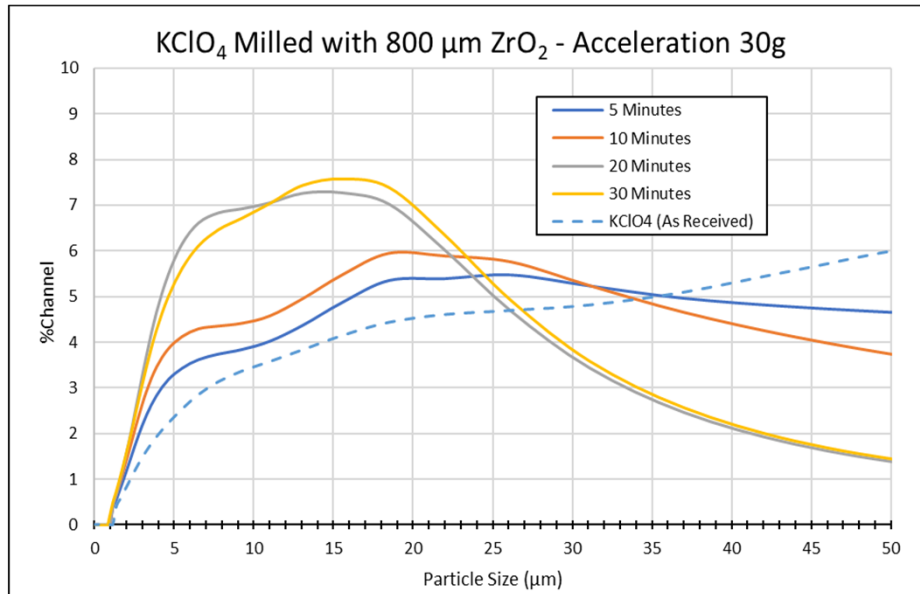
Acceleration Testing

- A LabRAM 1 was used to test milling of particle sizes of BaCrO_4 and KClO_4 .
- The composition was loaded into a small stainless steel mixing vessel and installed onto the RAM.
- The use of cooling water was utilized to keep the vessel temperature held at 18 °C.
- Each composition was subjected to 30 minutes of the specified acceleration (g).

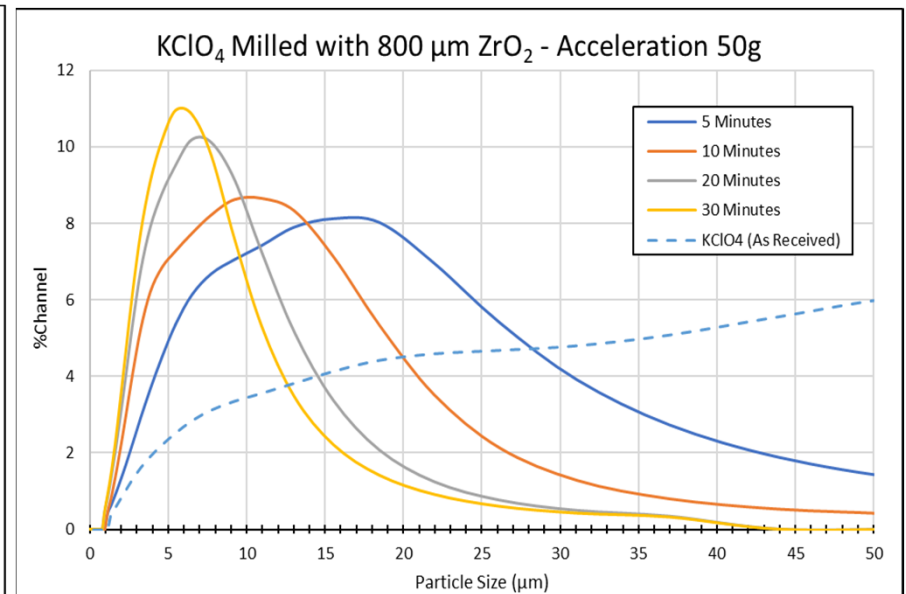
The RAM recipe was programmed as follows:

- 5 minutes
- Sample 1 retrieval interval
- 10 minutes
- Sample 2 retrieval interval – 0% power for 30 seconds (30 min total test time)
- 20 minutes
- Sample 3 retrieval interval – 0% power for 30 seconds (60 min total test time)
- 30 minutes
- Sample 4 retrieval interval – 0% power for 30 seconds (90 min total test time)

Acceleration Testing; KClO_4 with 800 μm zirconia media



Particle size distribution somewhat maintained until after 10 minutes



Particle size distribution begins changing immediately

Conclusions

- Particle size modification does not occur
 - When different materials with average particle sizes less than 10x
 - When realistic duration of mixing is used
 - At any acceleration when the above are maintained
- Particle size modification does occur
 - When different materials with average particle sizes less than 10x
 - With extremely long mixing times and large media