Evaluation of Composite Propellants In RAM

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Presented by:

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What is RAM/Advantages

- Resonant acoustic mixing is a non-contact mixing technology that relies on the application of a low-frequency acoustic field to facilitate mixing.
- The RAM technology uses an oscillating resonant driver system to transfer energy to a platform that shakes a mixing vessel or processing container. This provides uniform mixing throughout the container and is a faster process than conventional mixers.
- Resonant acoustic mixers create up to 0.55-inch oscillating displacements at approximately 60 Hz and up to 100 g of acceleration.
- The operating parameters that can be tailored throughout a mix cycle are: acceleration or intensity, pressure, temperature, use of both top and bottom transducer, time at specific conditions, and power into the mix.
- RAM Advantages
    - Shorter mixing time (from 5 hours to 30 minutes)
    - Reduction of waste when propellants are mixed in end unit
    - Cost savings due to reduced mixing time and/or waste
    - Consistent product
    - Scalability

Background

- Solid composite propellants for rocket motors are made from a wide variety of substances that are selected based on the specific application. Different ingredients and their proportions will result in different physical and chemical properties, combustion characteristics, and performance.
- Currently, composite propellants are mixed either in shaker style mixers or in Baker Perkins double planetary mixers.
- Depending on the type of mixer used, the EC-10 propellant can take up to three days to make.

Scope

- Two different types of propellants were selected for evaluation
  - BC-16 Composite Propellant
    - HTPB Binder
    - Uses 2 particle sizes of AP.
    - Uses a bonding agent that doesn't react with AP. No ammonia is produced to interfere with the curing process.
    - Six small-scale mixes were done in the LabRAM to study how order of addition affects the mix and the variations in viscosity during mixing.
    - One mix in the RAM-5.
  - EC-10 Composite Propellant
    - HTPB Binder
    - Uses ultra-fine AP.
    - The bonding agent used in the propellant formulation reacts with the ammonium perchlorate during mixing, producing ammonia gas. The ammonia generated by this reaction will react with the curing agent, interfering with the propellant cure.
    - Two RAM-5 mixes were done with overnight holds.
      - Ammonia concentration was measured throughout the mixing process.

RAM Models Used

- The LabRAM at NSWC IHD used for this effort was one of the first LabRAMs made by Resodyn Acoustic Mixers™. For this specific LabRAM, the intensity of the mixer was set and the frequency and acceleration were derived variables of the mixer software. Once determined by the software, the frequency would stay constant and the acceleration would vary throughout the mix process.
- Newer RAMs are manufactured with a software that accepts acceleration, not intensity, as an input; therefore, when mixing in the RAM-5, acceleration was used as an input.

LabRAM Mixing Vessel
Photo Credit NSWC IHD Dept. E2

RAM-5
Photo Credit NSWC IHD Dept. E2

BC-16 Evaluation

- LabRAM Mixes A-D: Evaluate how ingredient order of addition affects the mix
  - 300 gram mixes
  - Mixed until mix temperature reached 74°C (165°F).
  - Order of addition was varied
  - Lessons learned:
    - Start mixing at low intensity/acceleration to prevent liquids from splashing.
    - Begin vacuum after the solids have been able to incorporate into the mix to avoid solids going into the vacuum lines.
    - Prevent liquids from touching the walls of the mixing vessel during ingredient addition to guarantee that all the ingredients are properly mixed and the solids will not adhere to the walls.
- LabRAM Mixes E-F: Evaluate viscosity changes during mixing
  - 300 gram mixes
  - A rotational viscometer was used to measure the viscosity throughout the mixing process.
  - End-of-mix viscosity was reached at around 60°C. Continued mixing until temperature reached 74°C to evaluate material's behavior. Viscosity continued decreasing as the temperature increased.
  - Viscosity significantly decreased after curative addition.

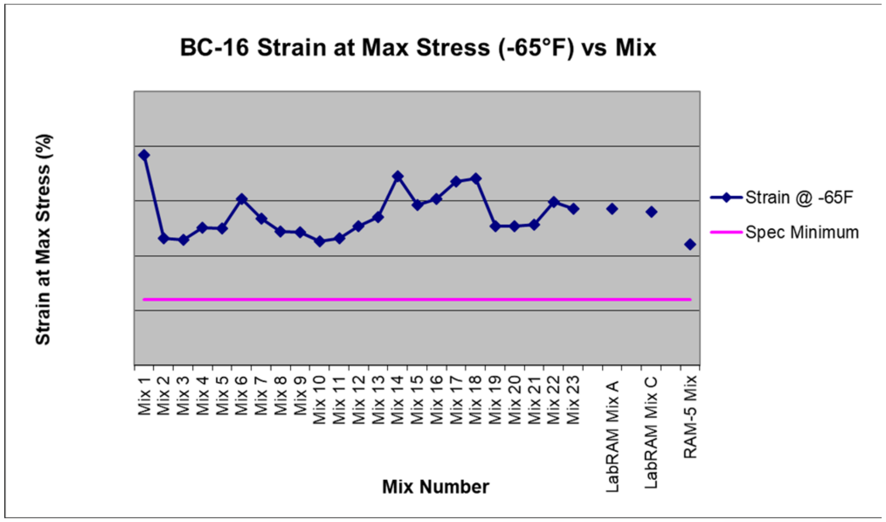
BC-16 Evaluation

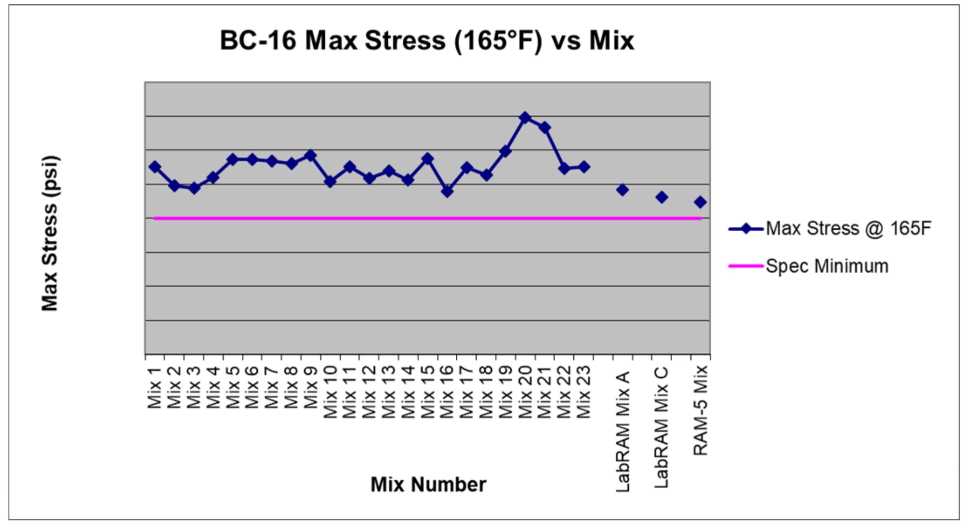
- RAM-5 Mix
  - As mentioned before, the LabRAM uses intensity as an input and the frequency and acceleration are derived variables of the mixer software; however, the RAM-5 accepts acceleration, not intensity, as the input.
  - The BC-16 LabRAM mixes exhibited proper mixing at an acceleration of around 70g, evidenced by the temperatures and the viscosities of the mixes. However, it is important to start mixing at a low acceleration/intensity to avoid splashing of the mix and to keep the solid particles from floating up into the vacuum tubes.
  - The RAM-5 mix was carried out in 10 cycles and, using the lessons learned from the small-scale mixes, the acceleration was started at 20g and was gradually increased to 70g.

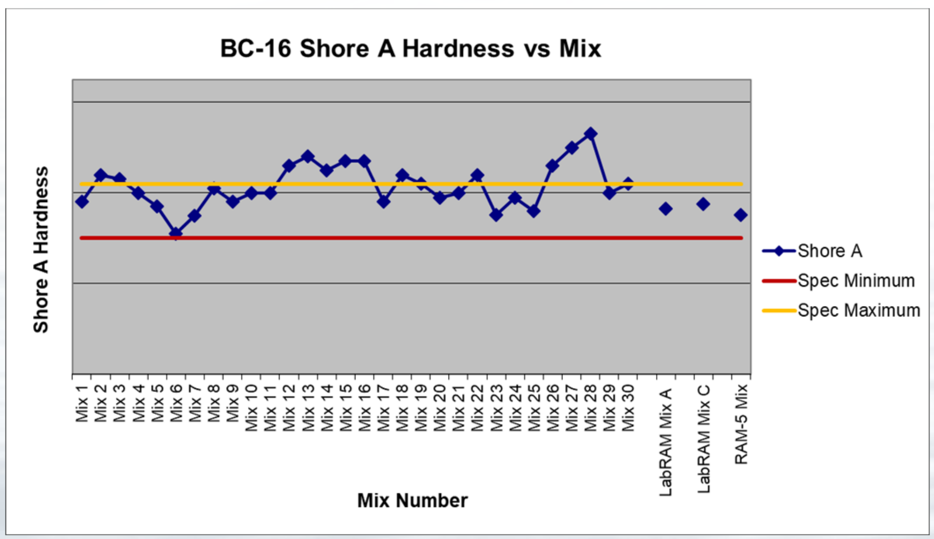
NAVSEA WARFARE CENTERS Indian Head

BC-16 Results

- Mechanical Properties

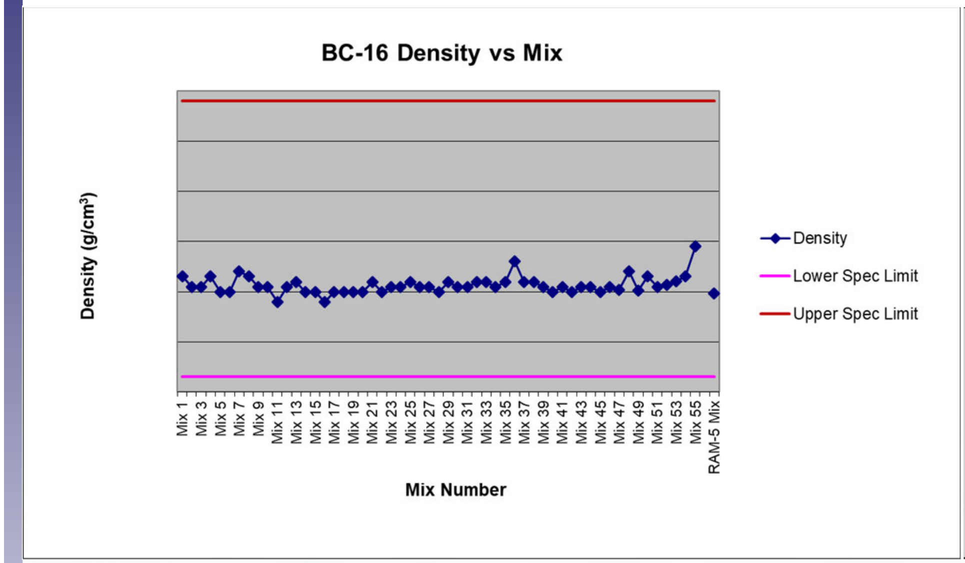


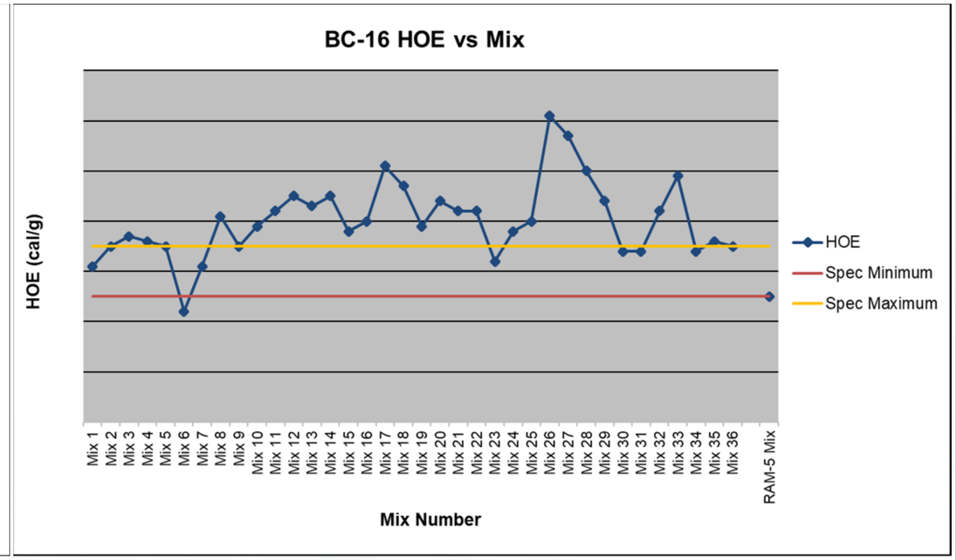




BC-16 Results

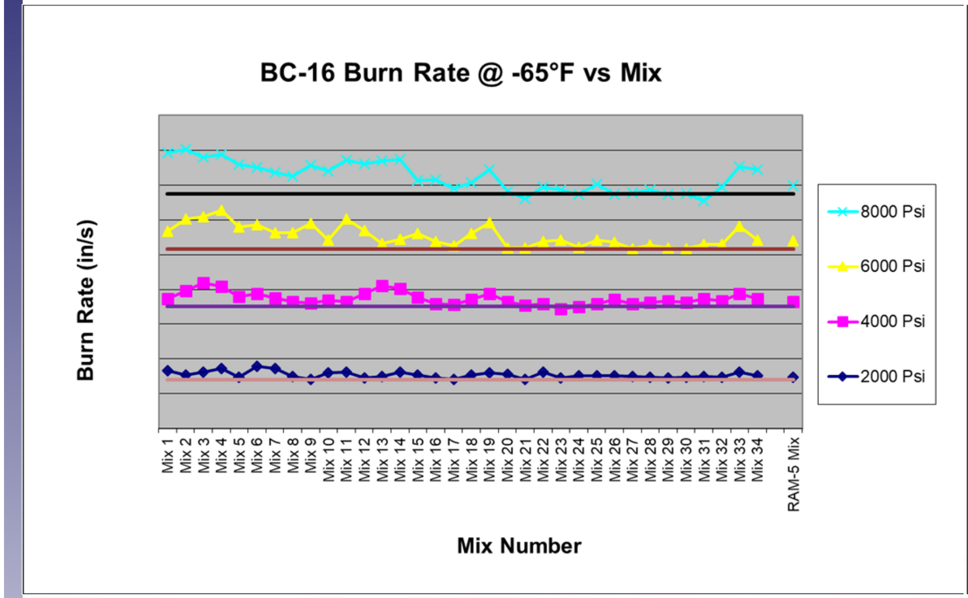
- Density and Heat of Explosion Results

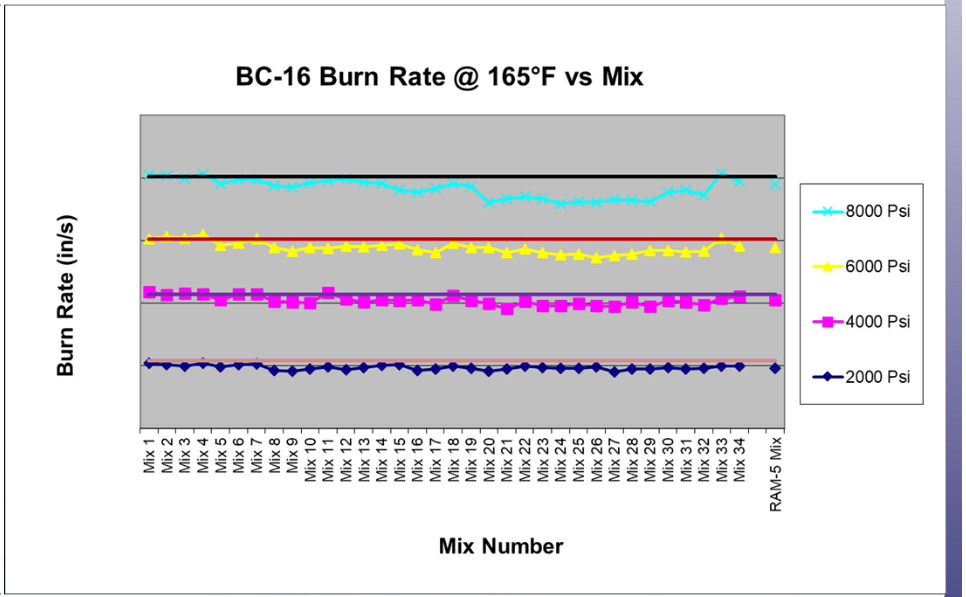




BC-16 Results

- Burn Rates Results





BC-16 Evaluation Summary

- The BC-16 mixes in the LabRAM produced acceptable propellant that met the performance requirements.
  - The LabRAM propellant mixes had a typical appearance and looked homogeneous before and after curing, with the exception of Mix C which had a visible spot of carbon black.
    - The spot of carbon black can be explained by evidence of unmixed carbon black in several places along the wall of the mixing vessel around the place where the carbon black was added. Possibly stuck to liquid on side of mixing vessel and didn't move/mix.
    - This highlights the importance of adding the ingredients properly to ensure homogeneous mixing in RAM.
- The large scale mix of BC-16 also met the performance requirements.
  - There were no visual anomalies before or after curing.

EC-10 Evaluation

- Mix #1
  - 25 pound mix
  - Ammonia readings were taken throughout the mixing process.
  - All AP was added prior to cycle 2 of mixing.
  - High temperature alarm went off during cycle 3. Vessel was warm to the touch, propellant was very dry and “chunky”, ammonia smell was very potent.
  - High temperature alarm went off again during cycle 5. Mix was fluid but thick when checked.
  - Stopped mixing for the day after cycle 6.
  - After discussions with Resodyn regarding the high temperature alarms, they advised that the Resistance Temperature Detector (RTD) could be faulty.
  - Mix was continued the next day. Mix temperature went up considerably, getting too hot to add curative. It was also noted that temperature readings were not accurate during the because the longest RTD was not making actual contact with the mix.
  - It was decided to stop the mix for the day and add another hold period. It was also decided that in order to mitigate the lack of temperature feedback during mixing, the mix time would be limited to a maximum of 10 minutes per cycle.

EC-10 Evaluation

- Mix #1 continued
  - After cycle 8, the propellant looked fluid throughout.
  - There was a strong smell of ammonia after cycle 11. During cycle 12, the mix was under vacuum for 30 minutes with alternating accelerations between 0g for 5 minutes and 80g for 30 seconds, giving a total cycle mix time of 2.5 minutes.
  - Cycle 12 was repeated. There was no smell of ammonia after cycle 13 and the curative was added to the mix.
  - Cycle 14: when acceleration was set to 40g, RAM jumped to very high acceleration momentarily before settling at 40g. Acceleration was then increased to 90g. At this point RAM shut down with accelerometer alarms and would not restart.
  - When the mix was checked, curative was found splattered on the mixing vessel wall and lid, most likely caused by the momentary spike in acceleration. It was later found that the clamp on the bottom discharge valve came off during mixing and was loose in the mixing vessel holder. Resodyn Inc. confirmed that this could have caused the activation of the accelerometer alarms and shutdown of cycle 14.
  - The mix time totaled to around 1 hour and 15 minutes.
  - Mix did not cure and remained in a gel-like state.

EC-10 Evaluation

- Mix #1 Discussion
  - A possible reason for the uncured propellant is that there was not enough material in the mixing vessel, resulting in inadequate mixing. A major component of the mixing action in resonant acoustic mixing is the energetic material colliding with the walls and ceiling of the vessel to break up clumps and large particles. The lower volume of propellant in the first mix could have resulted in incomplete mixing which prevented the propellant from curing.
  - Another potential reason for the may have been due to moisture contamination of the ingredients.
  - AP was added all at once, increasing the possibility of conglomeration and poor mixing.

Uncured EC-10 Mix #1
Photo Credit NSWC IHD Dept. E2

EC-10 Evaluation

- Mix #2
  - Based on the observations made after the first mix:
    - The weight of the second mix was increased from 25 pounds to 40 pounds.
    - All of the ingredients were tested for moisture percent prior to mixing, all weigh-out operations were conducted in low humidity conditions, and ingredient containers were purged with nitrogen every time they were opened.
    - AP additions were done in multiple cycles to limit solids conglomeration.
    - Mixing time was increased.
  - After cycle 4, propellant appeared “powdery” and AP was found on mixing vessel lid.
  - Started vacuum during cycle 5. Temperature increased to 73°C so the acceleration was lowered to lower the mix temperature. Mixing continued for 20 more minutes and was stopped for overnight hold.

EC-10 Evaluation

- Mix #2 continued
  - On the second day of mixing, a cycle was run before adding the curative.
  - The propellant was mixed for 28 minutes after curative was added.
  - Total mix time was 2 hours and 15 minutes.
  - Mix #2 did not cure. It is assumed that ammonia gas was trapped in the propellant mix and did not have an opportunity to escape during the RAM mixing process. If there is ammonia trapped in the middle of the mix, then the curing reaction will be inhibited, resulting in poor or no curing.

Ammonia Reading During Mixing
Photo Credit NSWC IHD Dept. E2

EC-10 Evaluation

- Mix #2 Discussion
  - EC-10 needs a significant amount of mixing time to ensure all ammonia is evacuated from the mix.
  - It is assumed that ammonia gas was trapped in the propellant mix and did not have an opportunity to escape during the RAM mixing process. If there is ammonia trapped in the middle of the mix, then the curing reaction will be inhibited, resulting in poor or no curing.

EC-10 Mix #2
Photo Credit NSWC IHD Dept. E2

Conclusions

- BC-16
  - The successful small-scale and large scale mixes of BC-16 demonstrated that the RAM mixing method is suitable for propellant formulations without a bonding agent that generates ammonia.
  - The results of mechanical properties, burn rates, density, heat of explosion, hardness, and sensitivity were within specification requirements and were comparable to previous mixes done in the conventional mixer.
  - Mixing in the RAM reduced the total mix time from 335 minutes to 31 minutes without diminishing the performance of the propellant.
- EC-10
  - Mixing EC-10 using RAM is more complex because of the ammonia generation caused by the bonding agent's reaction with ammonium perchlorate. It was determined that the propellant mixes did not cure due to ammonia not being fully removed.
  - Experienced increase temperatures during mixing of EC-10 possibly due to the propellant's high solids loading.

Recommendations

- In order to refine the mixing process, it is recommended to start with small scale mixes of EC-10 in the LabRAM.
  - Use a cooling mechanism such as a cooling jacket
  - Implement a nitrogen sweep overnight
  - Longer mix cycle on the second day to determine how long the propellant needs to be mixed to eliminate the ammonia
- After refining the process, it should be scaled up to the RAM-5. A nitrogen sweep overnight, cooling mechanism and longer mixing time before curative addition should also be considered at the large scale level.