AVT-340 Research Workshop on Preparation and Characterization of Energetic Materials

Resonant Acoustic Mixing of Propellant Compositions

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TNO innovation for life

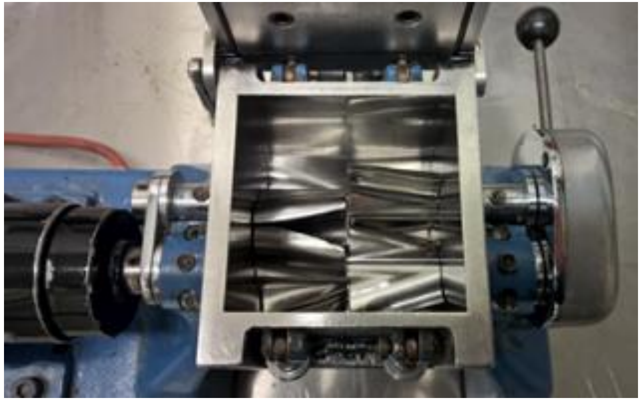
CONTENTS

- Background
- Comparative study of resonant acoustic vs. conventional mixing of a rocket propellant composition
- Other RAM developments, limitations and issues

This presentation [may] contain strategic technology that is subject to EU/Dutch export controls and may require prior written authorization from government authorities before (re)export and/or (re)transfer

BACKGROUND

- Conventional manufacturing of propellants using mixing & casting
- Processing times at least several hours, usually 1-2 days (depending on production scale)
- Limitations in processing of highly viscous mixtures (high solid load, high fraction of fine particles)

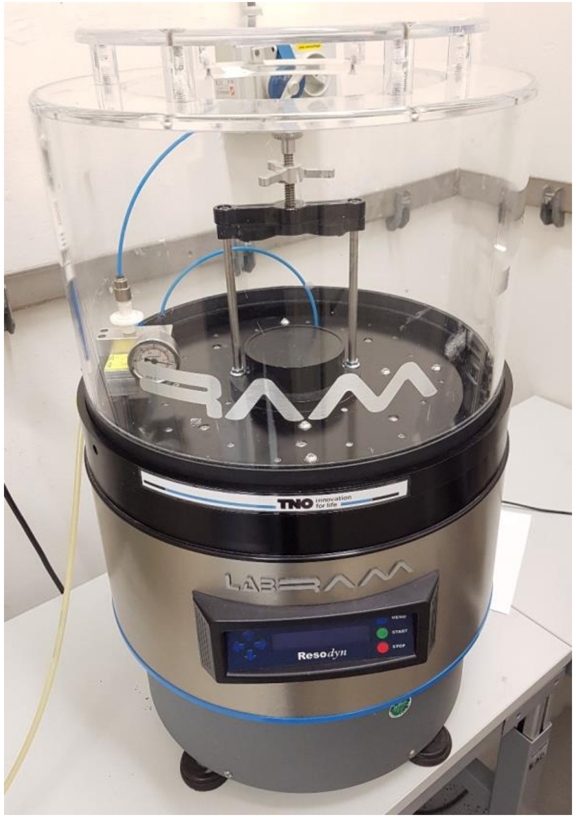


IKA high shear mixer, horizontal mixing blades, max. 350 g

HKV5 mixer, double planetary mixer, max. 4-5 kg

RESONANT ACOUSTIC MIXING

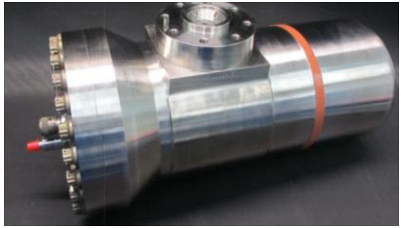
- Resodyn® LabRAM at TNO since December 2012
- Maximum mixing volume ~ 500 ml
- Maximum mixing mass ~ 500 gram



COMPARATIVE STUDY*: RAM VS. CONVENTIONAL MIXING OF A PROPELLANT

- Ammonium nitrate (AN) based igniter propellant produced by conventional process and LabRAM
- Rocket propellant samples from conventional batch were prepared and delivered by producer (Aerospace Propulsion Products, APP)
- Propellant ingredients (taken from the same lots) were delivered by producer APP
- Optimization of LabRAM process, resulting in a total mixing time of 10-15 min (depending on final temperature of the mixture; higher temperature gave better casting properties)
- Characterization: density, propellant cross-sections (SEM), burning rate

https://app.ariane.group/en/



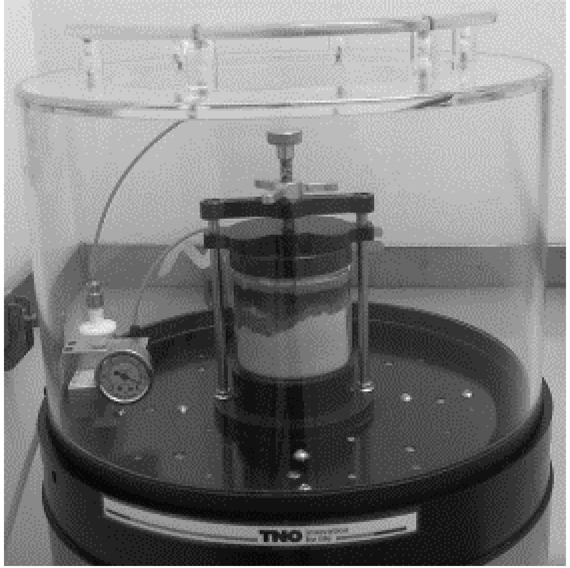
Igniter containing AN-based propellant grain

* M. Zebregs, A.E.H.J Mayer and A.E.D.M. van der Heijden, *Comparison of propellant processing by cast-cure and resonant acoustic mixing,* Propellants, Explosives, Pyrotechnics **45** (2020) 87-91

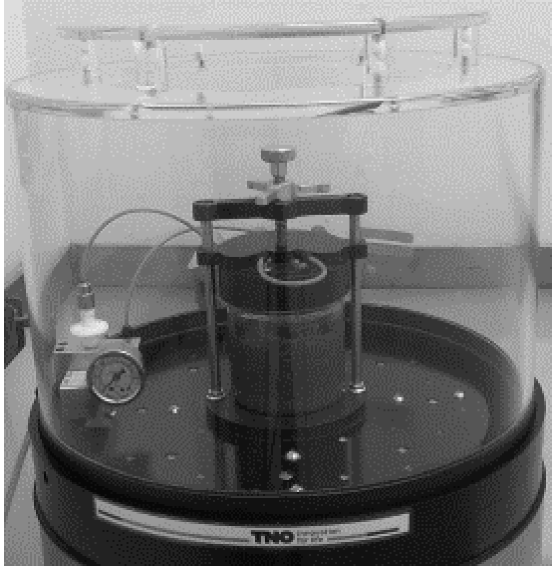
COMPARATIVE STUDY: MIXING PROCEDURE

Step	RAM-power	Time	Pressure	Remarks
1	30%	20 s	Atmospheric	Start of mixing
2	50%	2 min	Atmospheric	
3	80%	2 min	Atmospheric	Add remaining binder at end of step 3
4	80%	3 min	Alternately atmospheric/vacuum	Vary from atm. to vacuum every minute
5	100%	3 min	Alternately atmospheric/vacuum	Vary from atm. to vacuum every minute
6	100%	Variable	Alternately atmospheric/vacuum	Mix longer to increase temperature
7	4%	30 s	Alternately vacuum/atmospheric	Smoothen the mix

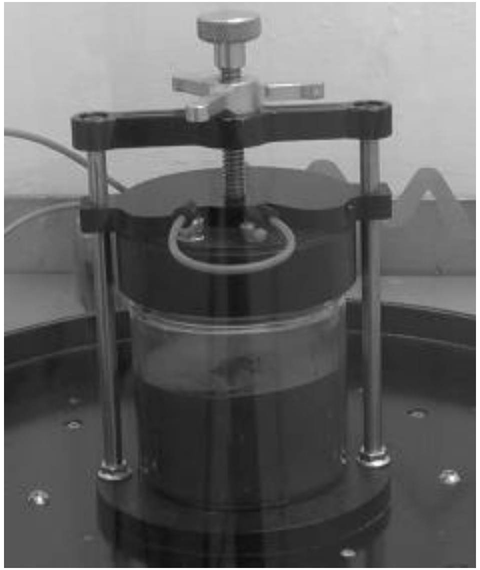
COMPARATIVE STUDY: MIXING PROCEDURE



Start LabRAM process



After 4 minutes (step 3 completed)



After ca. 10 minutes (final step): homogeneously mixed propellant

COMPARATIVE STUDY: DENSITY

- Helium gas pycnometry Micrometrics AccuPyc 1340

Propellant	Sample	Density [g/cm^3]
RAM-processed	Small sample [a]	1.375
RAM-processed	Large sample [b]	1.415
Conventional cast-cure	Small sample [a]	1.374
Conventional cast-cure	Large sample [b]	1.424

[a] Small part cut from a left-over chimney burner test sample.
[b] Sample taken from the remaining cured block of propellant.

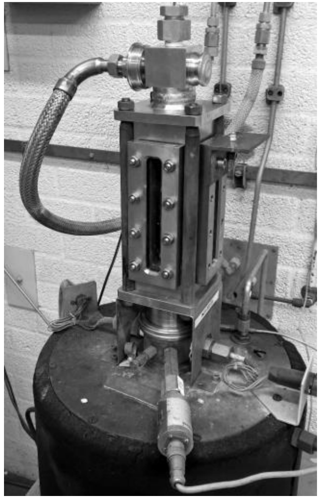
COMPARATIVE STUDY: DENSITY

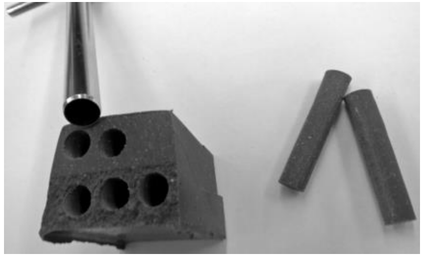
- Densities as measured for either the small or large samples are practically the same
- Lower density found for the small samples might be due to the larger surface-to-volume ratio of these samples compared to the larger samples (assuming the same degree of surface porosity)

➔ *difference in mixing and processing hardly affects propellant density*

COMPARATIVE STUDY: BURNING RATE

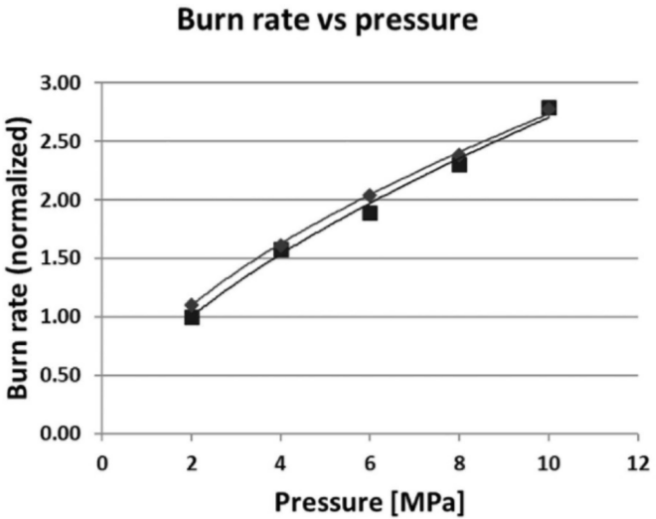
- Samples were prepared to determine burning rate: chimney burner (CB) tests
- Sample dimensions: ca. ø 10 mm, height ca. 40-50 mm
- Burning under nitrogen atmosphere
- High-speed camera IDT Vision, MotionPro Y4, frame rate typically 500 to 5,000 fps
- Tests were executed at 2, 4, 6, 8 and 10 MPa (in duplicate)





COMPARATIVE STUDY: BURNING RATE

- Equal burning rate performances
- Pressure exponents 0.57 and 0.62 for conventional and RAM-processed propellants, respectively
- Results within batch-to-batch variation of pressure exponents generally measured for conventionally processed propellants and in line with values mentioned in literature



Normalized burning rate vs. pressure

■ RAM-processed

♦ Conventionally processed

➔ difference in mixing and processing hardly affects burning rate characteristics

COMPARATIVE STUDY: CROSS-SECTION

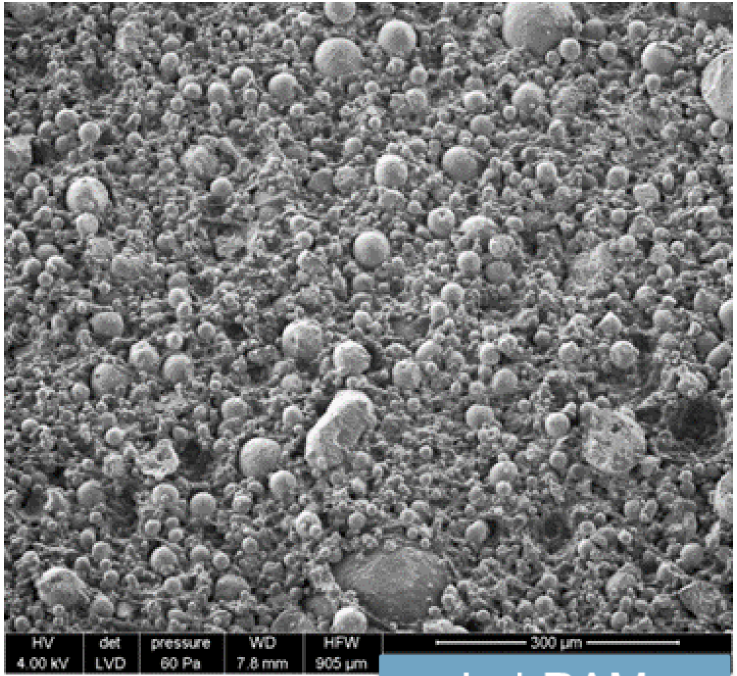
- Homogeneity of samples from both batches was visually determined by analyzing propellant cross-sections using scanning electron microscopy (SEM)

- SEM type: FEI NovaNanoSEM 650
  - Two magnifications resulting in a horizontal field width of ~1 mm and 128 μm, respectively
  - Accelerating voltage 5 kV
  - Low vacuum mode (50 Pa)

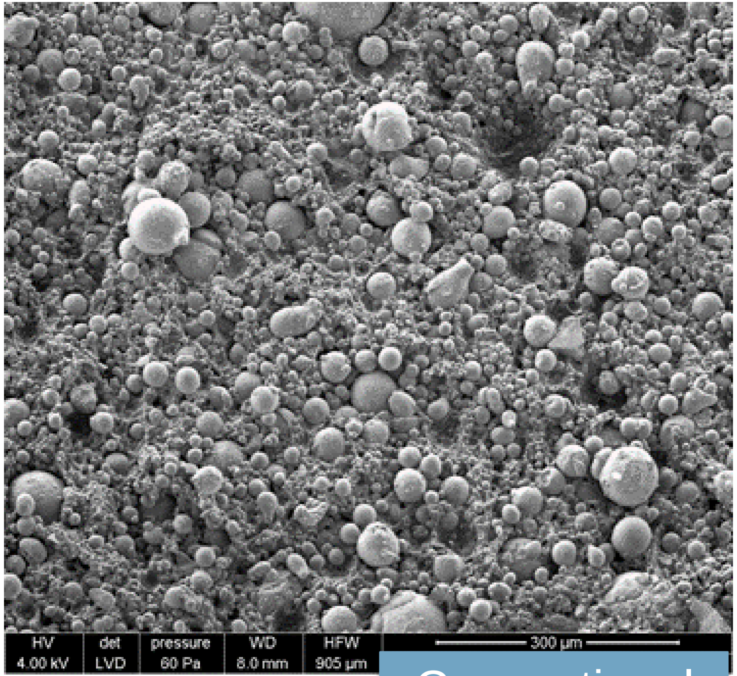
- Over 100 SEM images analyzed

Scanning electron microscope

COMPARATIVE STUDY: CROSS-SECTION

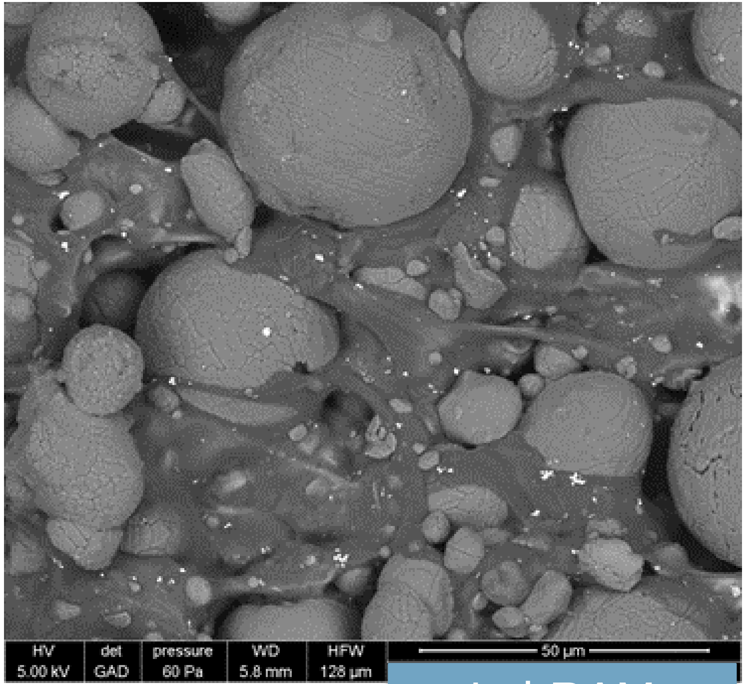


LabRAM

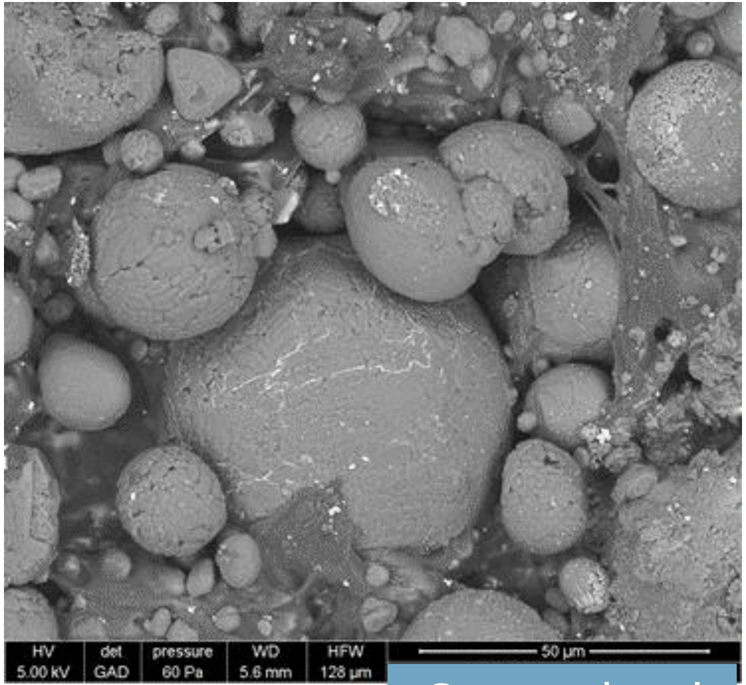


Conventional

COMPARATIVE STUDY: CROSS-SECTION



LabRAM



Conventional

COMPARATIVE STUDY: CROSS-SECTION

- No differences observed in sample homogeneity between conventional and RAM-processed propellants
- No damaging or breakage of particles observed

➔ *difference in mixing and processing hardly affects propellant homogeneity*

COMPARATIVE STUDY: SUMMARY

- AN-based solid composite rocket propellant produced using resonant acoustic mixing shows similar properties compared to the same propellant mixed by a conventional process
- Preparation time was reduced considerably
- Difference in mixing and processing hardly affects:
  - Propellant density
  - Propellant homogeneity
  - Burning rate characteristics
- Resonant acoustic mixing is a very promising, advanced processing technique that can replace conventional mechanical mixing

OTHER RAM DEVELOPMENTS

- Other developments using (Lab)RAM in combination with energetic materials:
  - Powder mixing: Nellums et al., PEP 38 (2013) 605-610 (thermites); Yamamoto et al., 43rd IPS (2018) (flares); Puszynski et al., 43rd IPS (2018) (primary explosives)
  - Co-crystals: Anderson et al., PEP **39** (2014) 637-640; PEP **41** (2016) 783-788
  - Milling: Kotter and Groven, PEP **44** (2019) 908-914
  - Pre-mix prior to 3D printing (e.g. @TNO)
  - Scale-up: LabRAM I (0.5 kg) – LabRAM II (1 kg) – OmniRAM (5 kg) – RAM 5 (36 kg) – RAM 55 (420 kg) – continuous acoustic mixing (CAM, configurable for OmniRAM, RAM 5 and RAM 55)

LIMITATIONS & ISSUES

- Limitations & issues
  - NC/NG-based gun propellants (solventless) → too high viscosity
  - Processing and safety were recently reviewed and discussed by Andrews et al. (PEP **45** (2020) 77-86); safe processing requires understanding of:
    - How the energy from the mixer is transferred to the mixed media
    - What are the modes of initiation
    - What is their level of response

Full Paper

Propellants, Explosives, Pyrotechnics

DOI: 10.1002/prep.201900280

Resonant Acoustic® Mixing: Processing and Safety

Matthew R. Andrews,*[a] Christelle Collet,[a] Aurihona Wolff,[b] and Chris Hollands[c]

  - Work on computational simulations (multiphase flow) is being conducted on LabRAM level to move from a trial and error process to a scientific-based assessment to be able to optimize RAM technology; simulations still need to be improved and scaled up for larger units

LIMITATIONS & ISSUES

Full Paper
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Resonant Acoustic® Mixing: Processing and Safety

Matthew R. Andrews,*[a] Christelle Collet,[a] Aurihona Wolff,[b] and Chris Hollands[c]

Issue / concern	Remedy / hazard reduction
Electrostatic charging / discharging	Use liquid phase and proper grounding of device
Temperature	Monitor and control
Over-pressure in case of burn / deflagration events	Redesign vessel with weak points to rapidly reduce confinement
Impact / pressure	Calculations point at large safety factor for RDX; requires consideration for other material mixtures; add phlegmatizing liquid
Accidental energetic material release into RAM vessel	Cover exposed areas or locations where material could enter the vessel; redesign vessel clamping system to minimize probability of spillage
Adiabatic compression of gaseous bubble	Estimated roughly an order of magnitude less than the case for NG initiation

SUMMARY

- AN-based solid composite rocket propellant produced using resonant acoustic mixing shows similar properties compared to the same propellant mixed by a conventional process

- Literature reported on safe RAM processing of energetic materials including:
  - Highly filled binder systems (PBX, gun/rocket propellants)
  - Pyrotechnic compositions (nanothermites, flares)
  - Primary explosives

- Issues and concerns (as reviewed by Andrews et al.) were summarized; although satisfying results in terms of performance were found experimentally, fundamental and applied research is needed to continue to understand the technology

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Co-authors on this work:

- Martijn Zebregs
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THANK YOU FOR YOUR ATTENTION

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