MSIAC
Munitions Safety Information Analysis Center

Supporting Munitions Safety

Procédés de fabrication innovants pour les matériaux énergétiques

Journée technique du GTPS - Usinage et procédés innovants en pyrotechnie
23 novembre 2021

Christelle Collet
TSO Propulsion Technology
c.collet@msiac.nato.int

Chris Hollands
TSO Energetic Materials
c.hollands@msiac.nato.int

This document has been prepared for MSIAC member nations. Disclosure of the contents to other nations must be authorised by the MSIAC Steering Committee.

Neither MSIAC nor the participating Nations can guarantee nor warrant the adequacy, accuracy, currency or completeness of the Technical Information contained in this document.

Overview

I. Traditional manufacturing techniques vs AM

II. Families of Additive Manufacturing Processes & their Applicability to the Different Categories of EM

1. Binder Jetting
2. Directed Energy Deposition
3. Material Extrusion
4. Material Jetting
5. Powder Bed Fusion
6. Sheet Lamination
7. Vat photopolymerisation

III. New Aspects to Consider with AM

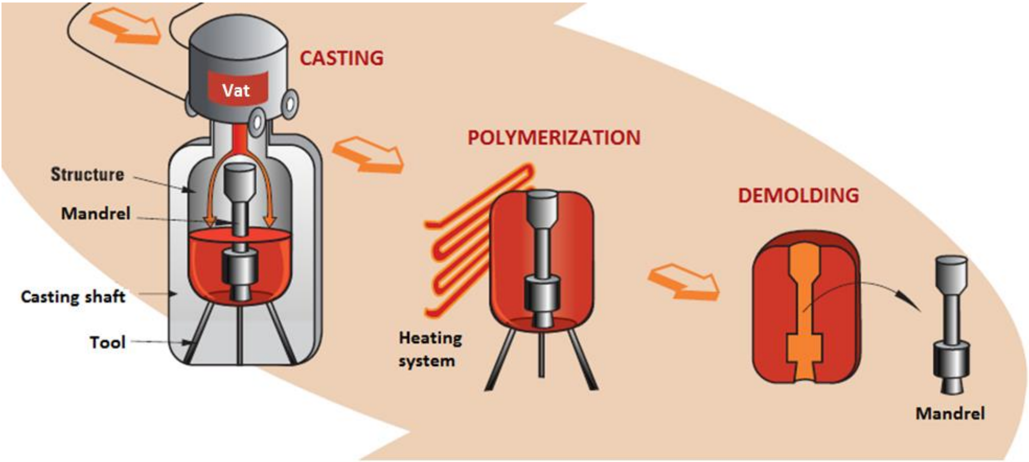
IV. Conclusion on AM

V. ResonantAcoustic® Mixing – Opportunities and Challenges

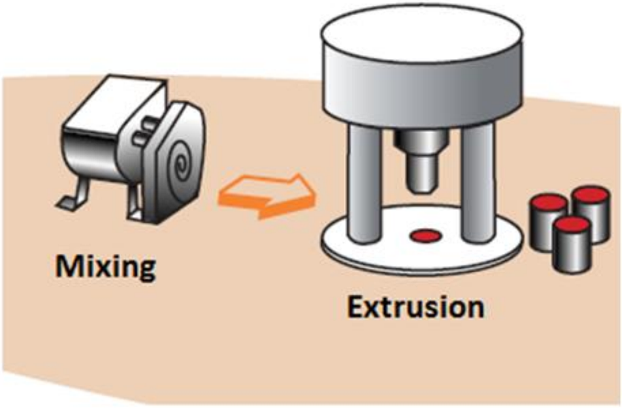
VI. Conclusion on RAM

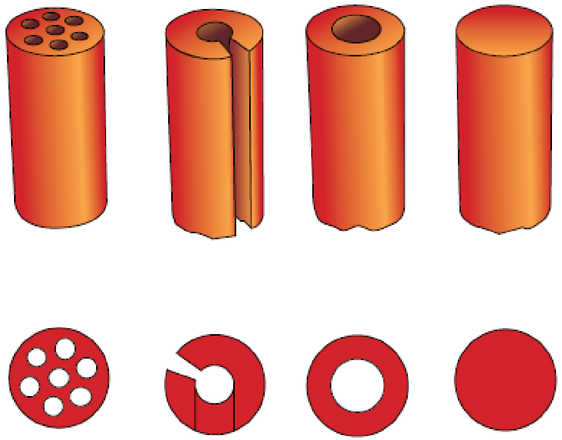
I. Traditional manufacturing techniques vs AM

Traditional Techniques – Disadvantages



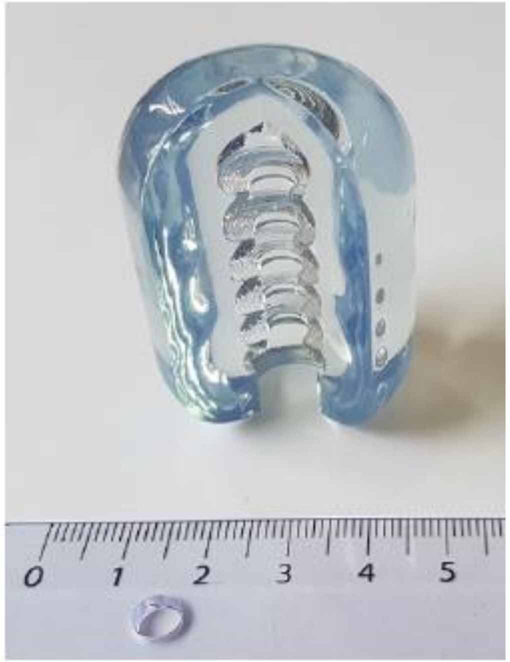
[1]



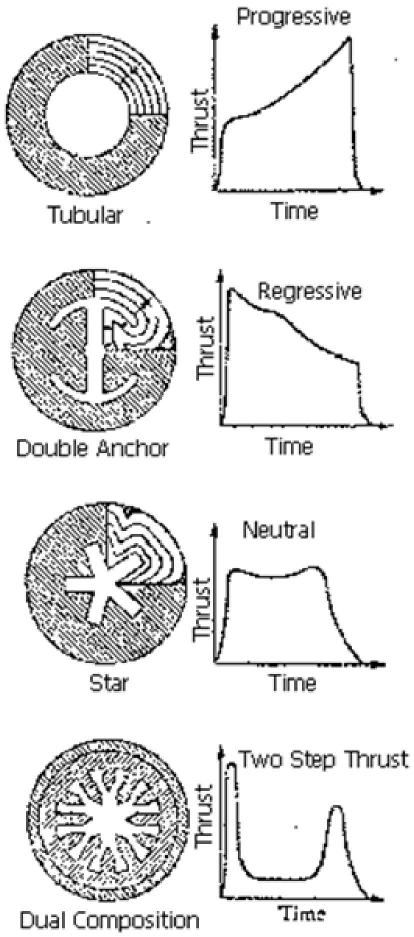


- → **Common processes cannot build complex shapes**
- → **In cast-cure processes, mandrels limit the shape of propellants in SRMs**
- → **In extruded processes, shapes are limited and it is not trivial to build multi-material parts**

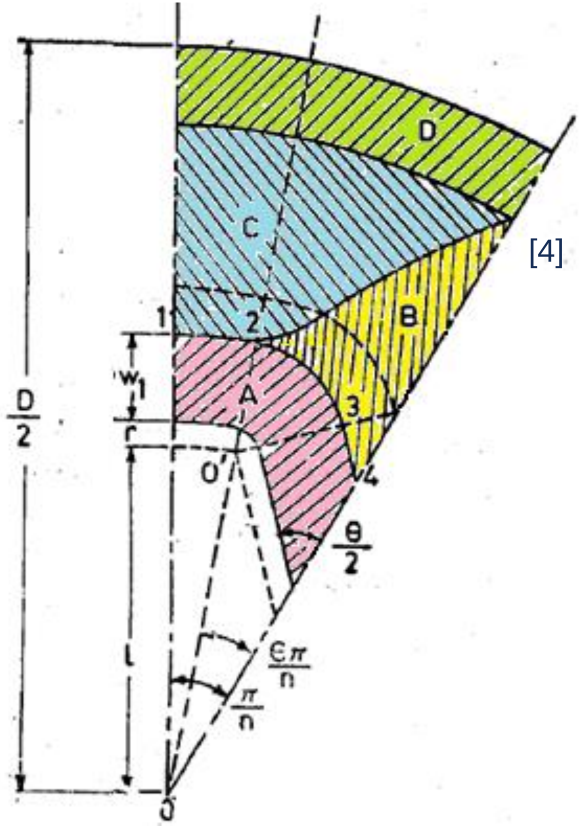
Additive Manufacturing – Advantages



[2]



[3]



[4]

AM allows complex shapes: Better control over the burn rate

AM can create multi-material parts: Better control over the burn rate

II. Families of Additive Manufacturing Processes & their Applicability to the Different Categories of EM

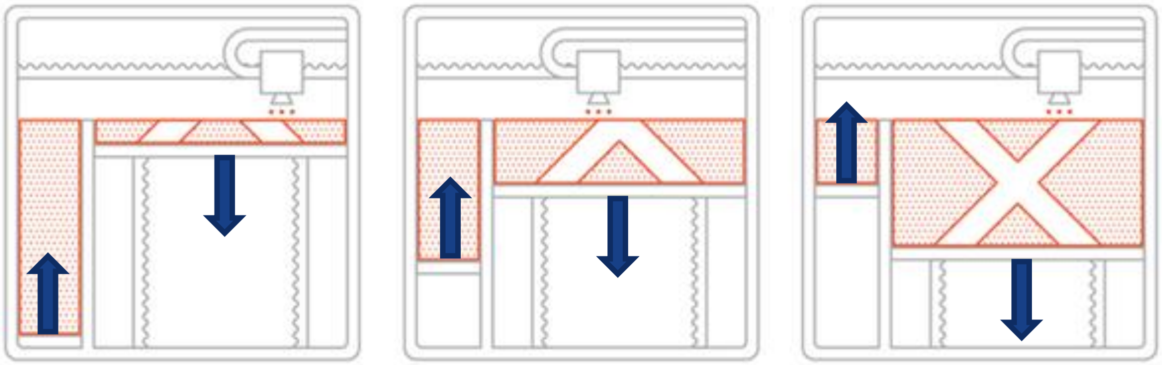
Families of Additive Manufacturing and Energetic Materials

- The families of AM are those defined in ISO/ASTM 52900:2015
- The EM categories were specifically defined for this study, with respect to their ability to be used as a feedstock for the AM techniques
- This analysis aims to fill in the cells in the below table with the following color code:
  - **Red**: the AM process is currently considered incompatible with or irrelevant to the EM;
  - **Orange**: the AM process would need a strong adaptation to be compatible with the EM or the other way around, in that the EM would need significant modification / reformulation to be compatible with the AM technique;
  - **Green**: there is literature evidence concerning the use of the AM technique to manufacture the EM, or there are no obvious reasons why the technique may not be used (NB the EM may require minor modification to be compatible);
  - **Grey**: there is insufficient information to make a definite judgement.

		AM Techniques						
		Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
EM Categories	Pure energetics / reactive mixtures	?	?	?	?	?	?	?
	Hot slurries	?	?	?	?	?	?	?
	Uncured slurries	?	?	?	?	?	?	?
	Gelatinized slurries	?	?	?	?	?	?	?
	Solid shaped energetics	?	?	?	?	?	?	?

1. Binder Jetting

- A liquid bonding agent is selectively deposited to join powdered materials that lie in a powder bed
- The powder is hardened when the printing head or the spraying system deposits a drop of binding fluid in a layering process



1. Binder Jetting

- Assessment of binder jetting for being used for EM manufacturing:
  - **Pure energetics & reactive mixtures**: No example so far, but theoretically feasible, provided that small quantities of raw materials are involved in the process
  - **Hot & Uncured Slurries**:
    - No example so far, but no theoretical limitation in the use of slurries for binder jetting, provided the bonding agent could be extruded through the nozzle
    - Possibly OK for small quantities (lab scale manufacturing or niche applications) but not for large scale volumes, as it would involve potentially large volumes of dry powder
  - **Gelatinized slurries**: Their high viscosity and sensitivity may prevent their use with this technique but there is not enough available information to make a definite judgement
  - **Solid shaped energetics**: By design, binder jetting bonds together solid particles by a binder layer which is solidified / cured immediately before another layer is deposited on top of it → not suited for the use of solid shaped energetics as a feedstock

1. Binder Jetting

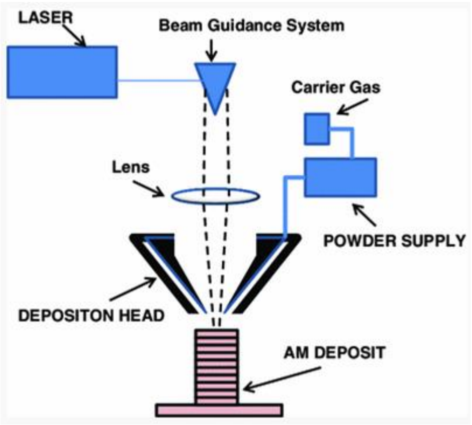
- Assessment of binder jetting for being used for EM manufacturing:

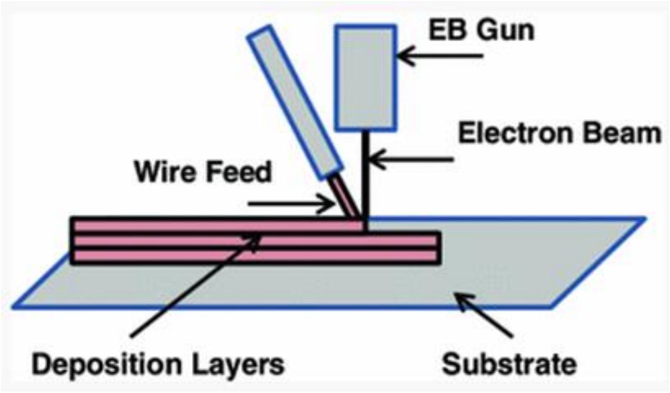
		AM Techniques						
		Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
EM Categories	Pure energetics / reactive mixtures							
	Hot slurries							
	Uncured slurries							
	Gelatinized slurries							
	Solid shaped energetics							

Red: incompatible / irrelevant
Orange: maybe with significant modification / reformulation
Green: OK
Grey: insufficient information to make a definite judgement

2. Directed Energy Deposition

- Focused thermal energy is used to fuse materials by melting them as they are being deposited.
- This process is often used with powder metal repairs where material has to be added to existing components by using a multi-axis nozzle to extrude melted material onto the printing surface.
- Many sub-categories exist, such as Powder Feed Systems and Wire Feed Systems





2. Directed Energy Deposition

- Assessment of Direct Energy Deposition for being used for EM manufacturing:

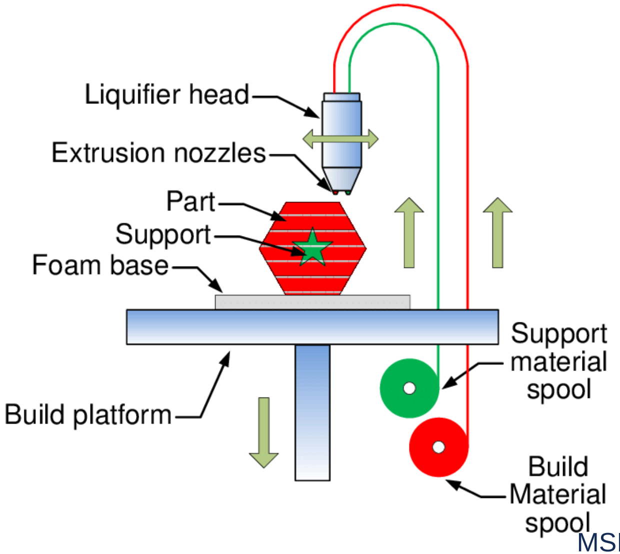
  - **all EM:** Directed energy deposition is not known to be used for EM. The technique is likely to generate unacceptable levels of thermal energy within the processed materials. For this reason, this AM technique is not considered suitable for EM, unless getting rid of the laser or electron beam.

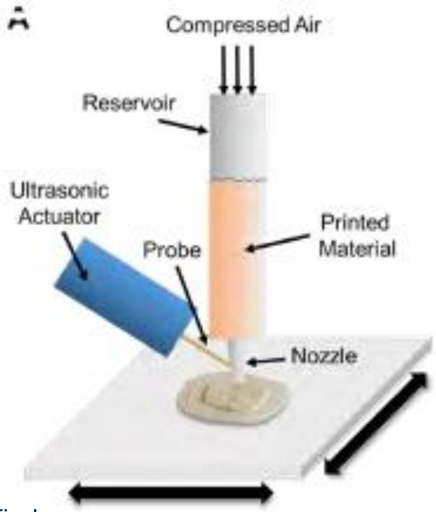
		AM Techniques						
		Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
EM Categories	Pure energetics / reactive mixtures							
	Hot slurries							
	Uncured slurries							
	Gelatinized slurries							
	Solid shaped energetics							

Red: incompatible / irrelevant
Orange: maybe with significant modification / reformulation
Green: OK
Grey: insufficient information to make a definite judgement

3. Material Extrusion

- Common AM process in which a plasticized material is extruded to form products from a sliced computer-aided design model
- It is considered as a solid wire feed technique (in opposition to powder feed systems)
- Numerous sub-categories exist including Fused Deposition Modelling (FDM) and vibration assisted direct-write

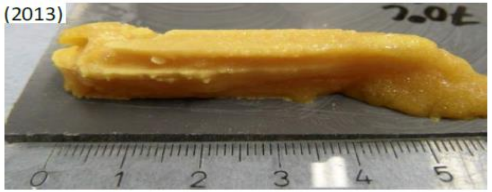




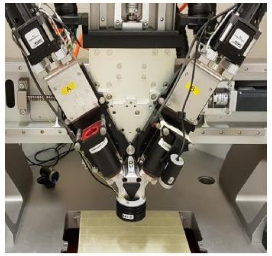
3. Material Extrusion

- Assessment of Material Extrusion for being used for EM manufacturing:
  - **Pure energetics & reactive mixtures**: By design, this process is not suited to for dry powders

[2]

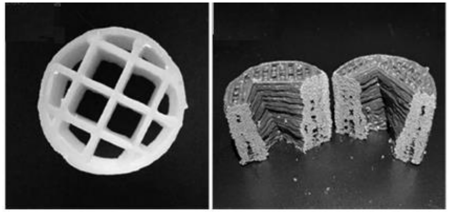


  - **Hot slurries**:
    - In 2013, TNO reported the use of pure TNT to form a 3D-printed shape
    - Despite the demonstration of feasibility, the AM works on TNT have not been pursued at TNO




[5]

  - **Uncured slurries**:
    - TNO have been developed developed an innovative FDM process since 2017: UV-FDM (hybrid process between FDM and SLA)
    - It aims to provide a higher solids loading and solve the viscosity problem of SLA. The two feeders can build parts with two different materials
    - Other efforts have been recently published in the US (US Army, US Navy, NASA) and in India
    - Attention must be paid to new kinds of defects that may be generated by this technique.



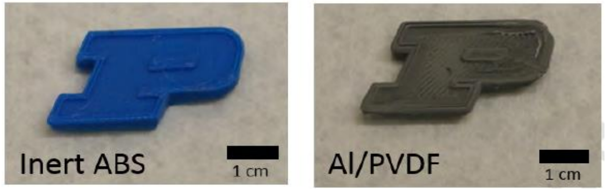
[6]

3. Material Extrusion

- Assessment of Material Extrusion for being used for EM manufacturing:

  - **Gelatinized slurries**:
    - No publication so far
    - The WP19-1246 SERDP-ESTCP project aims to investigate the feasibility of a novel vibration assisted direct-write AM technology to print extremely viscous NC/NG based mixtures with less solvent

  - **Solid shaped energetics:**
    - In a 2018 patent by Spence and Williams, filaments of 3 mm in diameter were prepared with exemplary compositions AP/TPE 74/26 and HMX/TPE 90/10 (solids fraction 25 - 95 wt.%)
    - The US reported the successful preparation of Al/PVDF filaments (20 wt.% Al) to manufacture solid shapes by a FDM technique



[7]

    - Practical and safety issues: viscosity, homogeneity during extrusion, hazards due to friction and heated nozzle, complexity of the technique (manufacturing a filament before printing a shape)

3. Material Extrusion

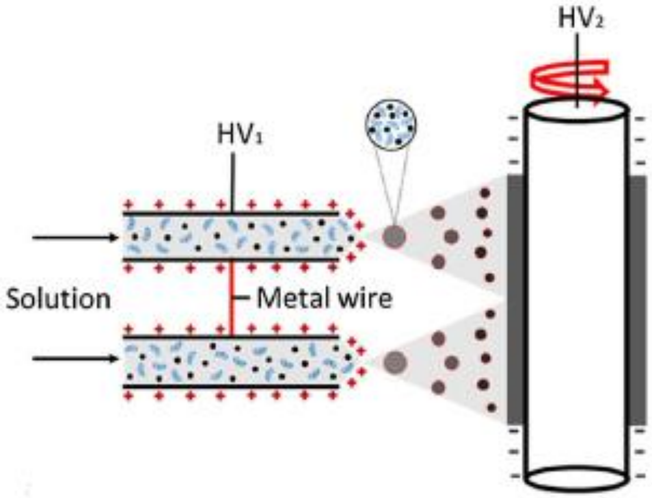
- Assessment of Material Extrusion for being used for EM manufacturing:

EM Categories	AM Techniques: Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
Pure energetics / reactive mixtures	Orange	Red	Red				
Hot slurries	Orange	Red	Green				
Uncured slurries	Orange	Red	Green				
Gelatinized slurries	Grey	Red	Orange				
Solid shaped energetics	Red	Red	Green				

Red: incompatible / irrelevant
Orange: maybe with significant modification / reformulation
Green: OK
Grey: insufficient information to make a definite judgement

4. Material Jetting

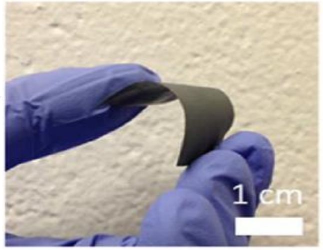
- Droplets of build material are selectively deposited on a support material
- A print head or a spraying system lays down successively solidifying layers of material
- Also referred to as Physical Vapor Deposition (PVD)
- Many derived techniques exist including Electrostatic Deposition (ESD) and electrostatic inkjet (EIJ) printing



4. Material Jetting

- Assessment of Material Extrusion for being used for EM manufacturing:
  - **Pure energetics & reactive mixtures**: many reported examples of vapor deposited EM thin films:
    - In the US, vapor-deposited thin films made of PETN, Al/MoO3 and Al/Bi2O3 nanothermites, HNAB, BNFF and HNS. Similar works conducted in the UK with TATB and LLM-105.
    - Demonstration by the US of the feasibility to prepare nanocomposite RDX in situ using an inkjet printer
    - A CL-20-based explosive ink known as EDF-11 has been qualified by the US Army
    - ESD process is suitable for manufacturing reactive microstructures based on Al/PVDF with a solids loading of 50 wt% nanoscale Al

[8]



  - **Hot and gelatinized slurries**: not enough information available in the literature
  - **Uncured slurries**: spray deposition of polyimide-titanates layers onto PTFE sheets was reported for manufacturing free standing films and structures with $SrTiO_3$ particle loadings of up to 85 wt% → could possibly be also used with uncured slurries of composite energetic materials
  - **Solid shaped EM**: by design, not suited to use solid shaped energetics as a feedstock.

4. Material Jetting

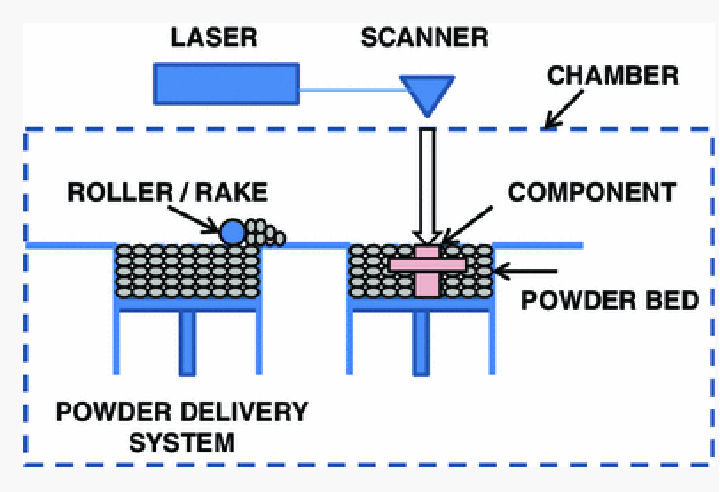
- Assessment of material jetting for being used for EM manufacturing:

		AM Techniques						
		Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
EM Categories	Pure energetics / reactive mixtures	Orange	Red	Red	Green			
	Hot slurries	Orange	Red	Green	Grey			
	Uncured slurries	Orange	Red	Green	Grey			
	Gelatinized slurries	Grey	Red	Orange	Orange			
	Solid shaped energetics	Red	Red	Green	Red			

Red: incompatible / irrelevant
Orange: maybe with significant modification / reformulation
Green: OK
Grey: insufficient information to make a definite judgement

5. Powder Bed Fusion

- A powder layer is created by spreading powder across the work area.
- The energy source (electron beam, UV light or laser beam) delivers energy to the surface of the powder bed, melting or sintering the powder into the desired shape.
- A new powder layer is then spread across the work area on top of the power bed, and the process is repeated to create a solid three dimensional component



5. Powder Bed Fusion

- **all EM**:
  - As for binder jetting, this technique is at first sight considered too hazardous due to the use of a large amount of dry powder.
  - There is lack of available information to support the suitability of this technique to EM

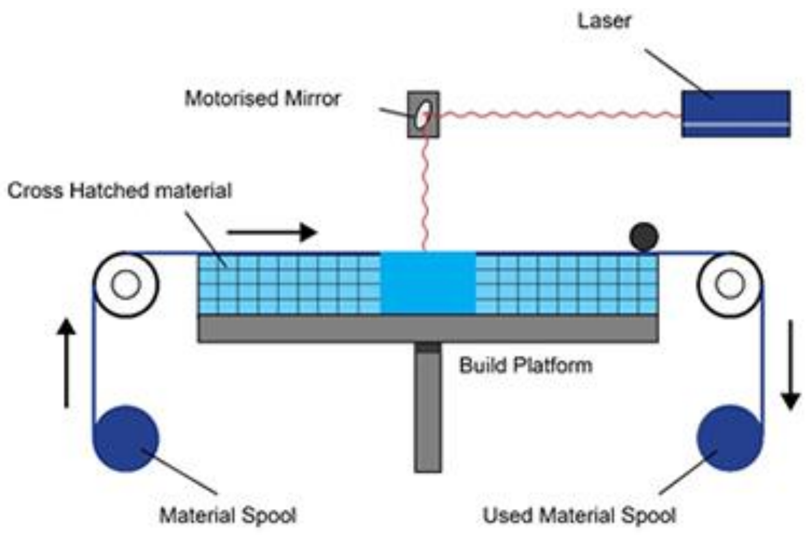
- Assessment of powder bed fusion for being used for EM manufacturing:

		AM Techniques						
		Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
EM Categories	Pure energetics / reactive mixtures	Orange	Red	Red	Green	Grey		
	Hot slurries	Orange	Red	Green	Grey	Grey		
	Uncured slurries	Orange	Red	Green	Grey	Grey		
	Gelatinized slurries	Grey	Red	Orange	Orange	Grey		
	Solid shaped energetics	Red	Red	Green	Red	Grey		

Red: incompatible / irrelevant
Orange: maybe with significant modification / reformulation
Green: OK
Grey: insufficient information to make a definite judgement

6. Sheet Lamination

- Pre-constructed sheets are bound together with glue or are fused, through ultrasonic welding or adhesive.
- The shaping is completed through further material removal processes.



6. Sheet Lamination

- **Granular energetics and all kinds of slurries**:
  - By design, this process is not suited to use granular and slurry materials as a feedstock as it requires a pre-produced solid sheet of the same material

- **Solid shaped EM**:
  - Today’s designs of sheet lamination apparatus use high energy cutting or fusing which are not considered suitable for solid EM due to unacceptable levels of friction and thermal energy that the technique may generate within the processed materials
  - Should the high energy cutting be replaced by a safer cutting technique, this process could be suited to manufacture solid EM from pre-produced sheets made of the same EM but this would require significant adaptation of the process and a way to recycle the wasted parts

6. Sheet Lamination

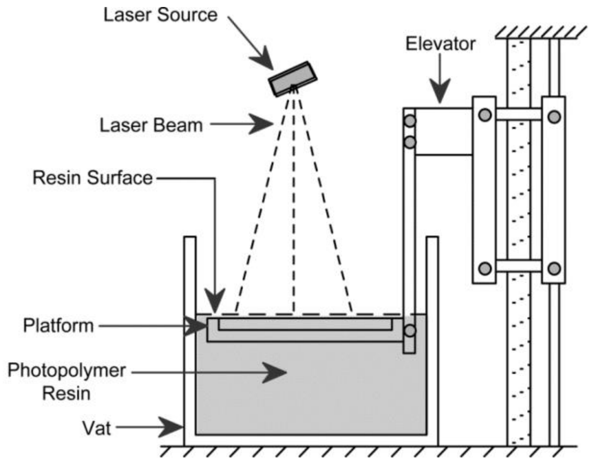
- Assessment of sheet lamination for being used for EM manufacturing:

		AM Techniques						
		Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
EM Categories	Pure energetics / reactive mixtures	Orange	Red	Red	Green	Grey	Red	
	Hot slurries	Orange	Red	Green	Grey	Grey	Red	
	Uncured slurries	Orange	Red	Green	Grey	Grey	Red	
	Gelatinized slurries	Grey	Red	Orange	Orange	Grey	Red	
	Solid shaped energetics	Red	Red	Green	Red	Grey	Orange	

Red: incompatible / irrelevant
Orange: maybe with significant modification / reformulation
Green: OK
Grey: insufficient information to make a definite judgement

7. Vat Photopolymerization

- A resin (liquid photopolymer) is selectively cured in a vat by light-activated polymerization. A photopolymer polymerizes when exposed to a selected wavelength of light e.g. UV light (190-380 mm).
- It can build parts layer by layer for a high-detail surface finish. The uncured resin has then to be removed with air pressure and the object is cut off of its support.
- This category includes Stereolithography (SLA), which is one of the earliest forms of 3D printing (patented in 1986)

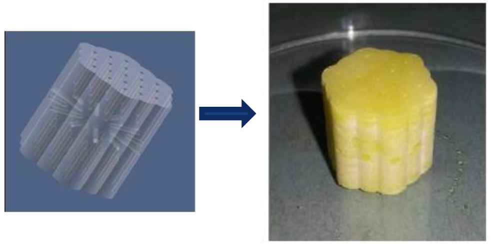


7. Vat Photopolymerization

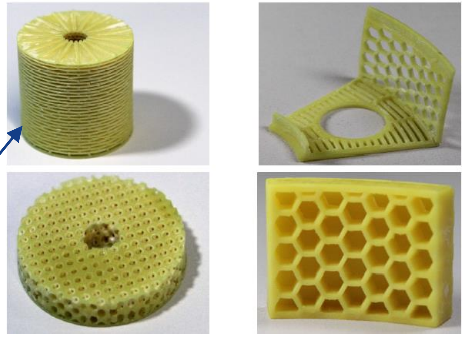
- **all EM except uncured slurries:** By conception, vat photopolymerisation can only use a slurry which is solidified by a curing process. As a result, it cannot be suited for using EM powders, hot slurries, gelatinized slurries and solid shaped energetics.

- **Uncured slurries**:
  - Vat photopolymerisation is proved to be used for EM manufacturing
  - It leads to a higher spatial resolution and requires lower temperature
  - In the TNO process, a pigment is used in the binder to limit the penetration depth of UV-light into the mixture at a depth of 2 mm, which is critical for the SLA process to work since the layers do not have to be cured underneath
  - A solids loading of 50% wt was reached (leading to 75% wt of total energetic fill when including the energetic binder)
  - TNO conducted the world’s first firing of 3D-printed gun propellant

[2]



[9]



7. Vat Photopolymerization

- Assessment of vat photopolymerization for being used for EM manufacturing:

EM Categories	AM Techniques: Binder jetting	Directed energy deposition	Material extrusion	Material jetting	Powder bed fusion	Sheet lamination	Vat photo-polymerisation
Pure energetics / reactive mixtures	Orange	Red	Red	Green	Grey	Red	Red
Hot slurries	Orange	Red	Green	Grey	Grey	Red	Red
Uncured slurries	Orange	Red	Green	Grey	Grey	Red	Green
Gelatinized slurries	Grey	Red	Orange	Orange	Grey	Red	Red
Solid shaped energetics	Red	Red	Green	Red	Grey	Orange	Red

Red: incompatible / irrelevant
Orange: maybe with significant modification / reformulation
Green: OK
Grey: insufficient information to make a definite judgement

III. New Aspects to Consider with AM

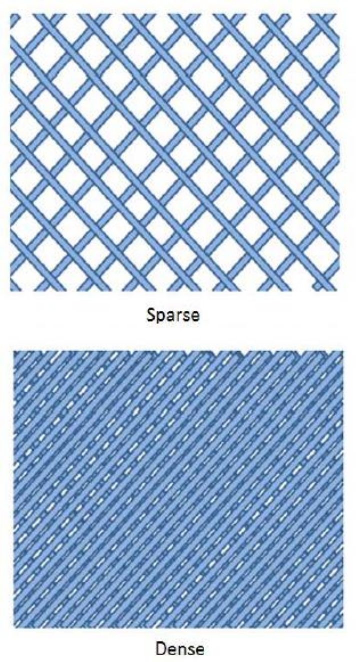
Material Extrusion: Optimization Parameters

Operation specific	Modeler specific	Geometry specific	Material specific
- Slice thickness/height - Road width - Raster angle - Envelope temperature - Contour width - Contour Number - Air gap	- Nozzle diameter - Filament feed rate - Flow rate - Head speed - Temperature of extruded material - Temperature of the bed	- Fill vector length - Support structure - Orientation - Number of layers	- Filament diameter - Viscosity - Uniformity of feed stock - Chemical composition - Coefficient of thermal expansion

Material Extrusion: Optimization Parameters

Operation specific

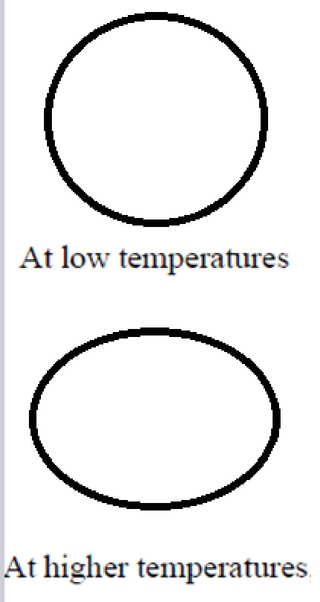
- Air gap [12]



Porosity

Modeler specific

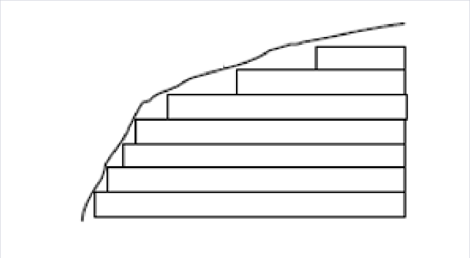
- Temperature of extruded material [13]



Interlayer defects

Geometry specific

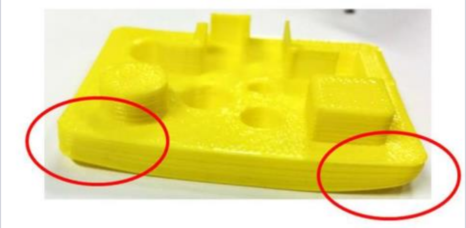
- Orientation [13]



Bad surface finish

Material specific

- Thermal expansion [14]



Voids

Material Extrusion Parameters

	[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]	[11]	[12]	[13]	[14]	[15]	[16]	[17]	[18]
Air gap			S		S					S	S					S		S
Chemical composition, set time, softening point					M			S	S			D		D	M			
Coefficient of thermal expansion				D				S	S			D			M			
Contour number					M					S						S		
Contour width			M		M					S	S					S		
Envelope temperature		S			M			S	S			D			S			S
Filament diameter	D																	
Filament feed	D																	
Flow rate	D						M							D				
Geometry	D																S	
Hardware/Softwabuild-up	D	M								D		D						
Head speed															M			
Layout optimization		S	M							D	S			D			S	
Nozzle diameter	D																	
Number of layer								D	D								S	
Orientation	D	S	S	D	S	S	S			S	S						S	S
Raster angle		M	S	D	S	M				S	S					S	S	
Road/Raster width	D		S		S	M										S		S
Road length	D							D										
Slice thickness or height	D	S	S			S												S
Support structure																		M
Temperature of extruded material		S		D				D	D			D			D			D
Uniformity of feed stock	D						M											
Viscosity, rheology, tackiness		D		D														D
Complex analysis			yes		yes	yes				yes	yes		yes			yes		yes

[1] (Van Weeren, et al., 1995)
[2] (Knoop & Schoeppner, 2015)
[3] (Sood, Ohdar, & Mahapatra, 2009)
[4] (Said, et al., 2000)
[5] (Ahn, Montero, Odell, Roundy, & Wright, 2002)
[6] (Khan, Lee, & Abdullah, 2005)
[7] (Lee, Kim, Kim, & Ahn, 2007)
[8] (Wang, Tong, & Ye, 2007)
[9] (Bellehumeur, Gu, Sun, & Rizvi, 2008)
[10] (Montgomery, 2003)
[11] (Srivastava, Maheshwari, Kundra, Yashaswi, & Rathee, 2016)
[12] (Yaman, 2018)
[13] (Rathee, Srivastava, Maheshwari, & Siddiquee, 2017)
[14] (Srivastava, Maheshwari, & Kundra, Virtual Modelling and Simulation of Functionally Graded Material Component using FDM Technique, 2015)
[15] (Choi, Kim, Jeong, & Youn, 2016)
[16] (Wagari & Lemu)
[17] (DeCicco, 2013)
[18] (Vasudevarao, Natarajan, Henderson, & Razdan, 2000)

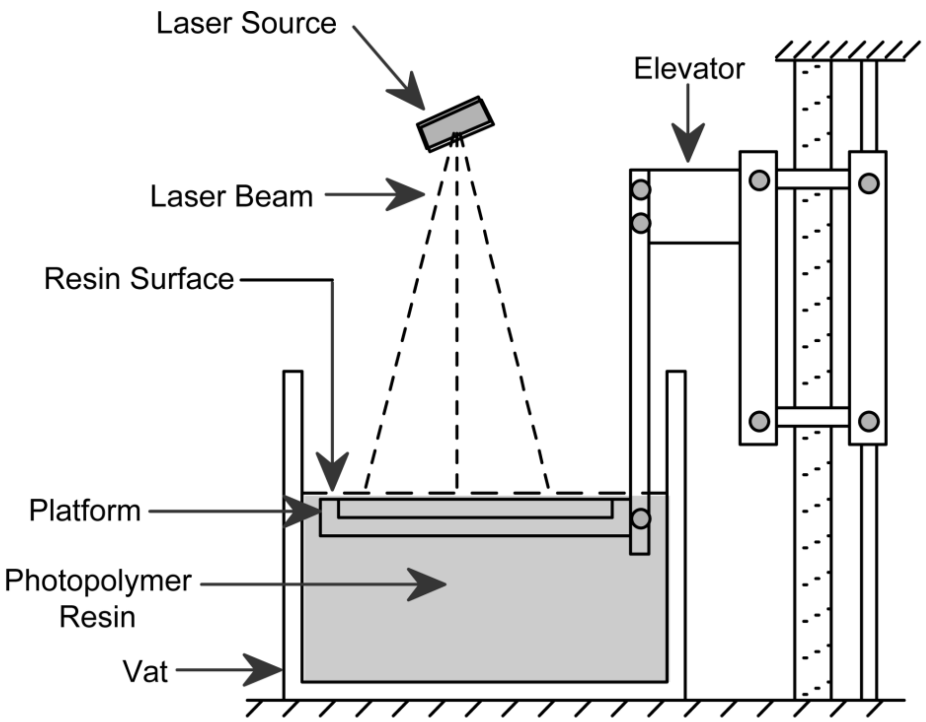
S: Studied
D: Discussed
M: Mentioned

Vat Photopolymerization: Stereolithography

Lower temperature process → safer
Good surface finish → better performance

But…

Only one material can be processed
Lower solid loading → reduced performance



[19]

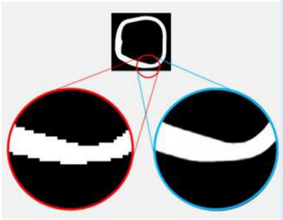
Vat Photopolymerization: DLP

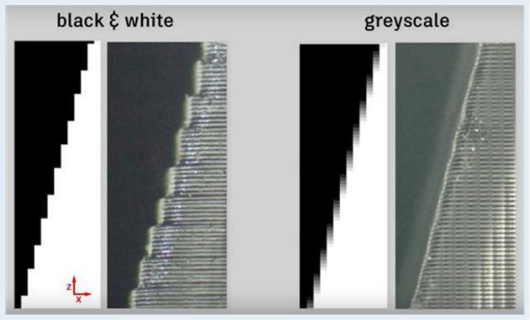
Supporting Munitions Safety

Lower temperature process → safer
Excellent surface finish → better performance

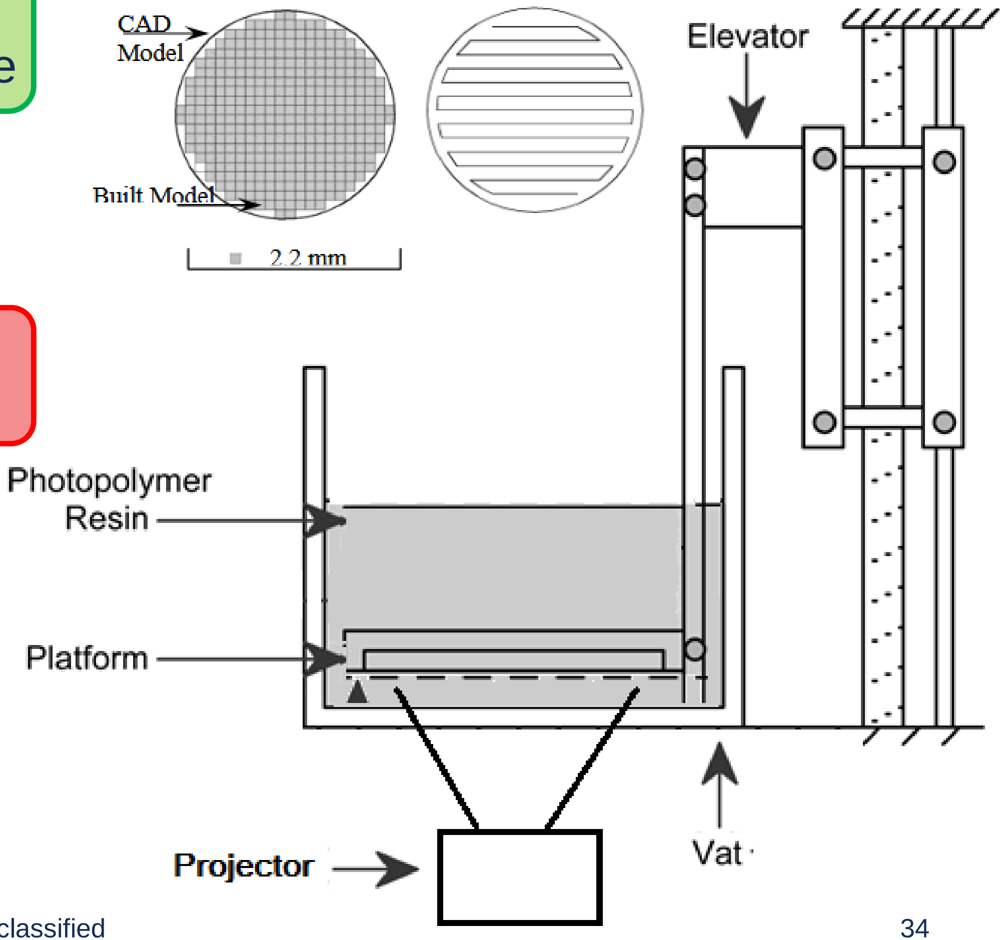
But…

Only one material can be processed
Lower solid loading → reduced performance





[15]



MSIAC
Munitions Safety Information Analysis Center

Optimization parameters: SLA and DLP

Supporting Munitions Safety

Optimization parameters for Stereolithography

Operation specific	Modeler specific	Geometry specific	Material specific
- Layer thickness - Length of layer - Number of layers - Hatch or fill spacing - Raster angle - Number of contour - Contour width - Road width	- Laser power - Beam width - Time exposure - Scan rate	- Orientation	- Penetration depth - Critical exposure - Polymerisation shrinkage - Chemical composition

Optimization parameters for DLP

Operation specific	Modeler specific	Geometry specific	Material specific
- Layer thickness - Separation distribution - Work area - Number of layers	- Time exposure - Positioning velocity - Separation velocity - Intensity	- Orientation	- Penetration depth - Critical exposure - Polymerisation shrinkage - Chemical composition

Material Extrusion vs Vat Photopolymerization

Material Extrusion:

- Print high solid loading energetic material
- Print different material in the same part

- Many defects: voids, porosities…

Vat Photopolymerization:

- High resolution final product
- Complex shapes

- Print low solid loading EM
- Only one kind of material

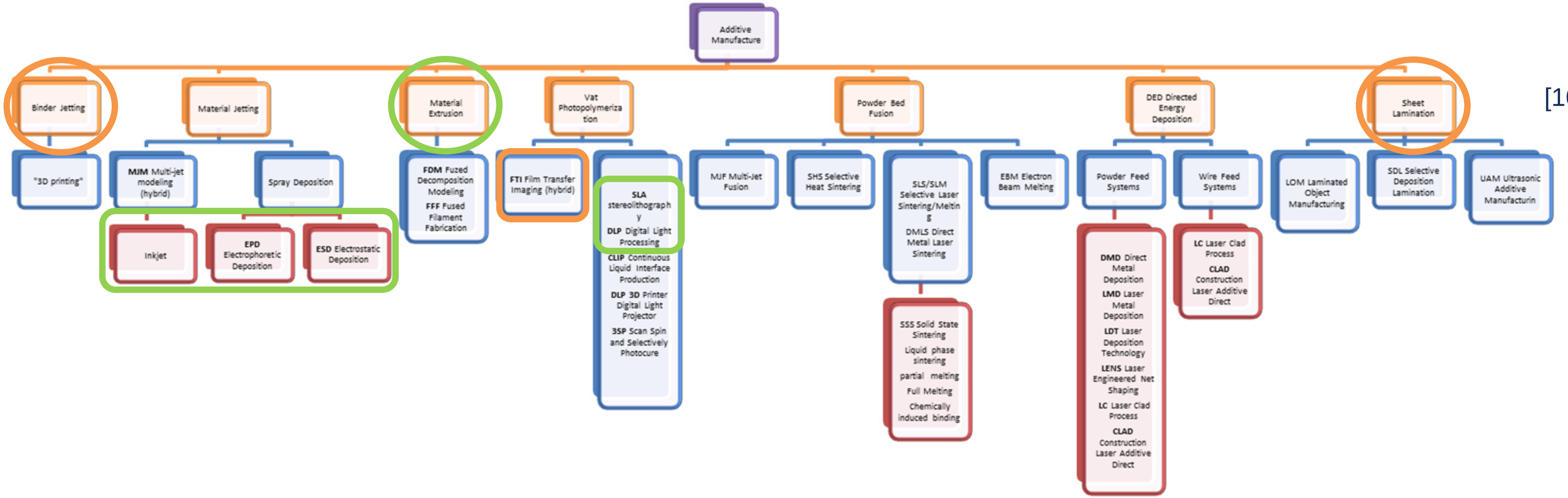
Next challenges are:

- Optimizing material extrusion to avoid defects
- Being able to increase the solid loading in VP

IV. Conclusion on AM Techniques Applied to Energetic Materials

Conclusion on AM Techniques

- From what has been openly published so far, only material jetting, material extrusion and vat photopolymerization have been exploited in the field of energetic materials
- Other AM techniques could be suited for EM production, because there are no obvious reasons why they would not be suitable, or provided an adjustment of the process or of the feedstock material be undertaken

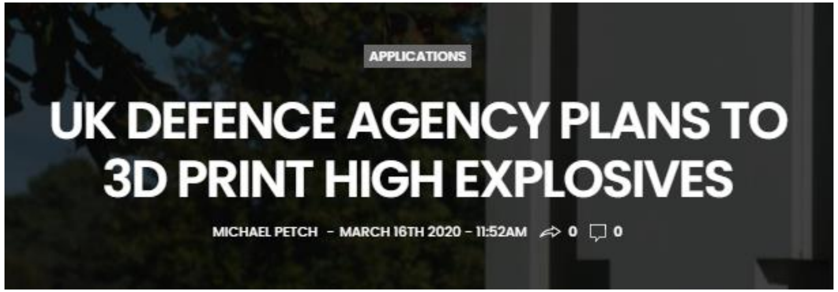


[10]

Conclusion (cont'd)

- AM techniques are rapidly evolving: an increasing number of hybrid techniques are being developed which use the combined advantages of different main techniques
- The main difficulties are 1) Applying AM for highly solids loaded materials and gelatinized slurries and 2) addressing the issues related to new kinds of defects

- For the time being, there are no examples of fielded munitions containing a 3D-printed energetic filling but thanks to the recent advances in this area, it is no longer considered to be unattainable and it benefits from significant funding:



The Defence Science and Technology Laboratory (DSTL), an executive agency sponsored by the Ministry of Defence (MOD) of the UK, has started to develop 3D printed explosives.

Further work in 3D printing explosives

To reduce cost and improve weaponry efficiency, the Australian government has awarded USD $2 million to research institutions to develop methods to 3D print energetic materials. Professor David Lewis, a polymer specialist at the awarded Flinders University, said, "The ability to develop systems like this is the next generation of additive manufacturing and is the key to this technology becoming mainstream."

Full article available at:
https://3dprintingindustry.com/news/uk-defence-agency-plans-to-3d-print-high-explosives-169082/

Bibliography Related to the AM Section

[1] **Tauzia, J. M.** (2006). À propos des matériaux énergétiques*. SNPE Matériaux énergétiques*

[2] **van Lingen, J. and Straathof, M., van Driel, C. and den Otter, A.** (2016). 3D printing of gun propellants. *43rd IPS PYROTECH 2018.*

[3] **Hill, P.G. and Peterson, C.R.,** (1992) Mechanics and Thermodynamics of Propulsion, ISBN 978-81-317-2951-9

[4] **Krishnan, S., and Tarit Bose, K**. (1976) Design of multi-propellant star grains for solid propellant rockets. *Journal of Materials Engineering and Performance*, 1917–1928

[5] **Straathof, M., van Driel, Ch., den Otter, A., van Lingen, J., Heinsius, J., Isenia, J. and Rijnders, B.** (2019) Gradient printing of energetic materials - first results. *31st International Symposium on Ballistics*, Hyderabad, India

[6] **Chandru, A.R., Balasubramanian, N., Oommen, C. and Raghunandan, B.N.** (2018) Additive Manufacturing of Solid Rocket Propellant Grains. *Journal of Propulsion and Power*, doi:10.2514/1.836734

[7] **Fleck, T.J., Murray, A.K., Gunduz, I.E., Son, S.F., Chiu G.T. and Rhoads, J.F.** (2017) Additive Manufacturing of Multifunctional Reactive Materials. *Additive Manufacturing*, vol. 17, p. 176–182

[8] **Huang, C., Jian, G., DeLisio, J. B., Wang, H., & Zachariah, M. R**. (2014). Electrospray Deposition of Energetic Polymer Nanocomposites with High Mass Particle Loadings: A Prelude to 3D Printing of Rocket Motors. *Advanced Engineering Materials*

[9] **van Lingen, J.** (2018) New production technologies for energetic materials that can reduce environmental impact; AM and solventless (co-) Extrusion. in *SERDP-ESTCP Symposium*

[10] **Wolff, A., Andrews, M. and Collet, C.** (2020) Common processes used for explosives and propellants. *MSIAC Limited Report L-240*

[11] **Bailey, A. and Murray, S.G**. (1989). *Explosives, Propellants and Pyrotechniques*

[12] **DeCicco, A.** (2013). *Effect of build parameters on additive materials*

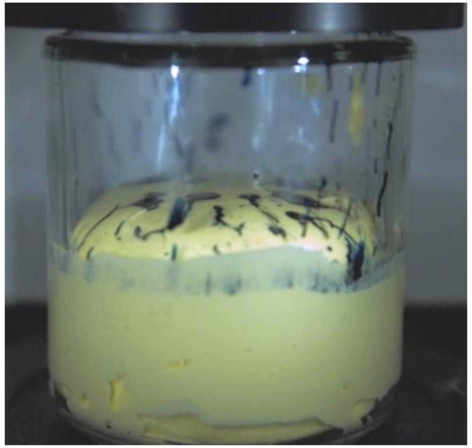
[13] **van Weeren, R., Agarwala, M., Jamalabad, V. R., Bandyophadyay, A., Vaidyanathan, R., Langrana, N. and Ballard, C**. (1996). Quality of Parts Processed by Fused Deposition. In *Rapid Prototyping Journal,* Volume 2 Issue 4

[14] **Henderson, M. R**. (2000). Sensitivity of Rp Surface Finish to process parameter variation*. Partnership for Research in Stereo Modeling and Department of Industrial Engineering Arizona State University*

[15] **van Lingen, J**. (2018). Additive Manufacturing of energetic materials at TNO. *Presentation for MSIAC*

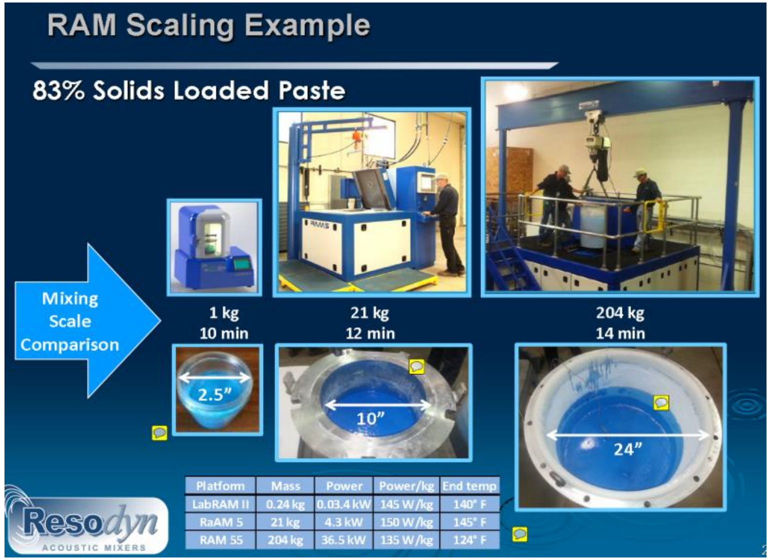
V. ResonantAcoustic® Mixing – Opportunities & Challenges

Conventional vs RAM



ResonantAcoustic® Mixing (RAM)

- Bladeless mixing, shear induced through low frequency (60 Hz)
  - Acceleration: 5 – 100 G
  - Amplitude: 2 – 12 mm
- Hailed as a disruptive technology
- Mixing of different phases needs specific theories [1]
  - Particle-particle collision (left)
  - Acoustic streaming (centre)
  - Faraday instabilities (right)
- Shown to be scalable
  - 500 g (LabRAM) to 400 kg (RAM 55)



1. Wolff, A. *L-246* (**2019**) MSIAC Report
2. Farrah, L. 2nd *EMQ RAM Tech. Meeting.* (**2018**).

Experimentation – Different Approaches

MSIAC Munitions Safety Information Analysis Center

Supporting Munitions Safety

Two categories of tests:

1) Tests on how **the apparatus** mixes

- Macro & micromixing
  - Beam line experiments [Jubb, 2018]
  - RAM provides a more homogeneous mix [Beckel, 2018] [Nelson, 2018]
  - Apparatus to monitor mixing progress [Jubb, 2018]
- These tests are needed to
  - Build models,
  - Test theory and
  - Run simulations

2) Tests on the **produced materials**

- Similar density can be found [Zebregs, 2018] and less voids are observed with the RAM,
- Similar safety properties are observed in terms of impact, friction and ESD [Beckel, 2016]
- Similar performance can be seen with RAM [Jubb, 2018]
- Similar sensitivity of the final product [Komansechek, 2018]

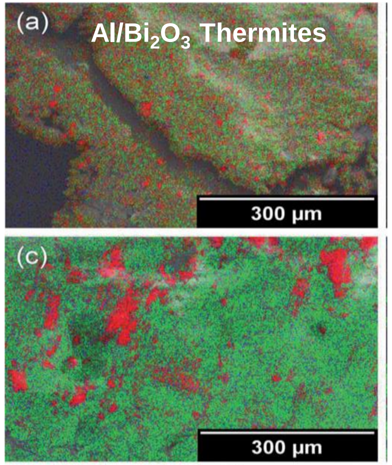
Driver is **performance**

- Comparison of RA mixed against standard mixers is difficult
- RA mixed unique materials will require different analytical approach

Experimentation - Energetics Being Processed

- Co-crystalisation
- Rocket motor propellants
- Plastic explosives
- High solids loaded PBX
  - Cast cured
  - Moulding powders
- Thermites
- Flare compositions

- Gun propellant still requiring safety assessments
  - Energetic liquids

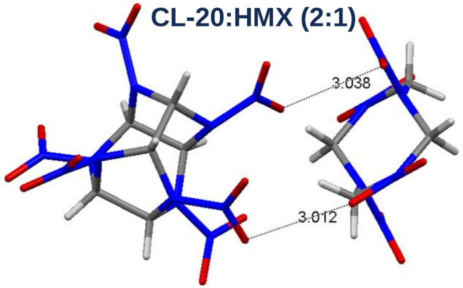


*Red(Al), Green(Bi), Blue(O)

Nellums, 2013

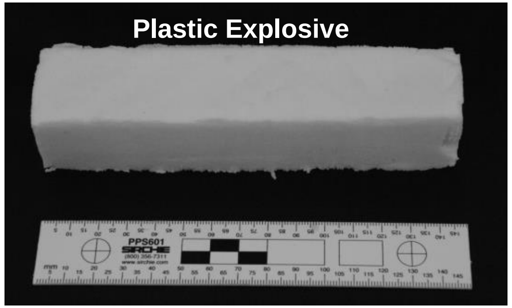
Nelson, 2018

Miklaszewski, 2018



am Ende, 2015

Provatas, 2017



Provatas, 2017

Major Questions Related to EM Qualification

Processing

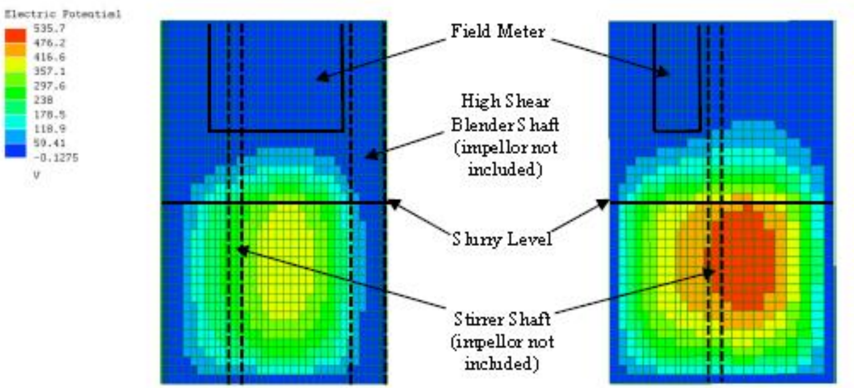
- General
  - How to determine end of mix?
- Mix In Case
  - What is considered a batch size?
  - What will be considered lot acceptance?
- Continuous Mixing
  - What is a batch?
  - How and when to sample?
  - Acceptance criteria
- Scaling
  - Material quality
  - Safety

Qualification

- Material Specifications
  - Are they suitable and sufficient for RAM applications?
- Lot and batch sizes
  - Should there be a change in definition for in-case and continuous mixing?
- Current Qualification standards
  - Are they suitable and sufficient for RAM?

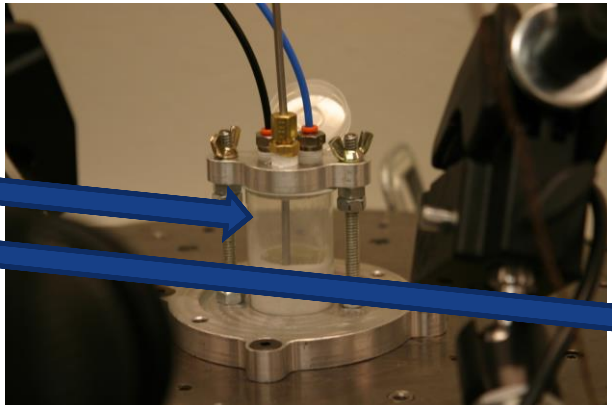
Hazards for RAM - Electrostatic

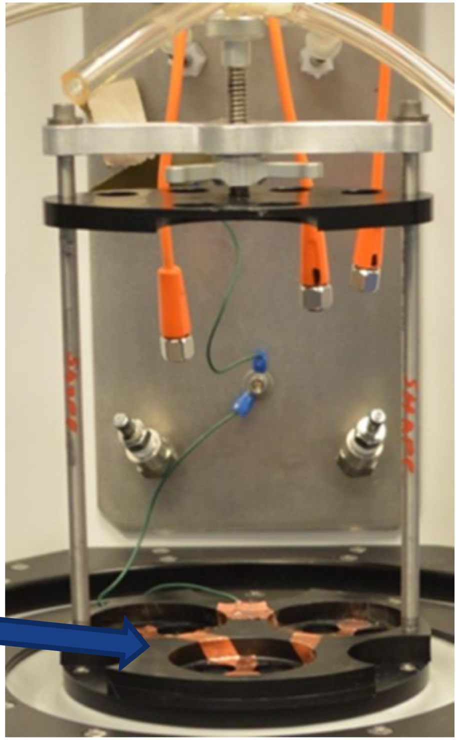
- Experimentation
  - Few RAM examples
  - High shear mixers
- Measuring
  - Field meter



Pavey, 2015

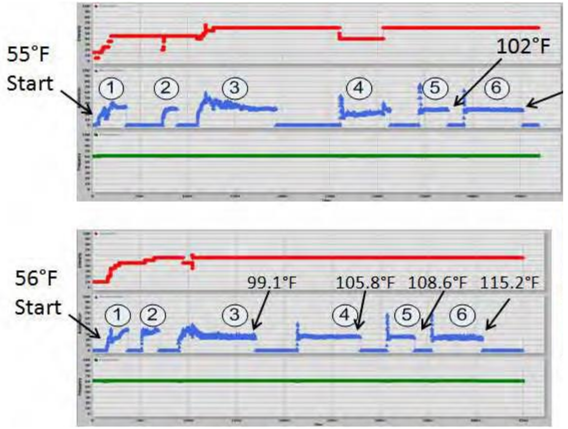
- Controlling
  - Grounding of product through thermocouples
  - Grounding of vessel
  - Grounding of resonator
  - Monitoring grounding pathway


Miklaszewski, 2018

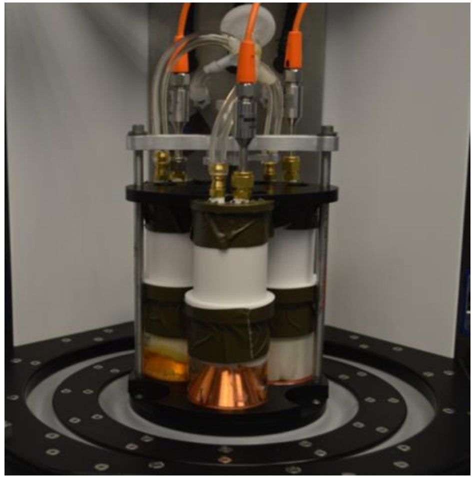

Nelson, 2018

Hazards for RAM - Thermal

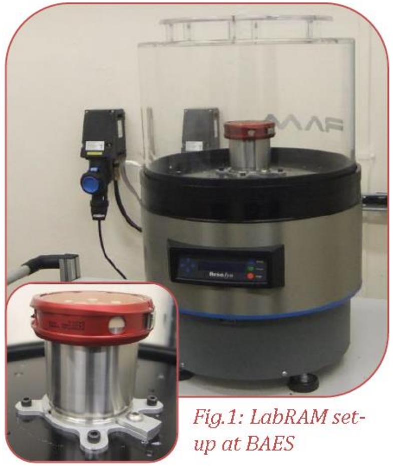
- Experimentation
  - Observed temperature increase due to shear from mixing
- Measuring temperature
  - K-type thermocouples in product
    - Multiple, varying heights
  - IR thermometer
  - FTIR camera post mix
- Control of temperature
  - Designing of water-jacketed vessels



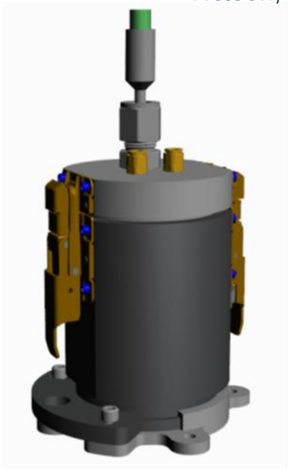
McPherson, 2014



Nelson, 2018



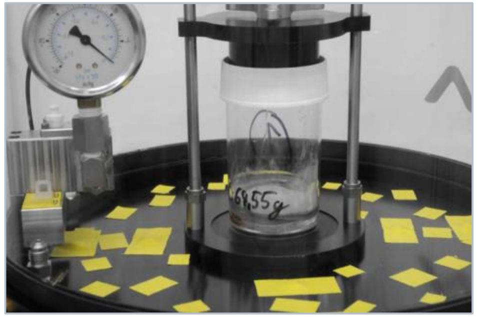
Davey, 2018



Miklaszewski, 2018

Other Hazards for RAM

Chemical
- Vapours
- Controls
  - Fume hoods
  - Closed vessels

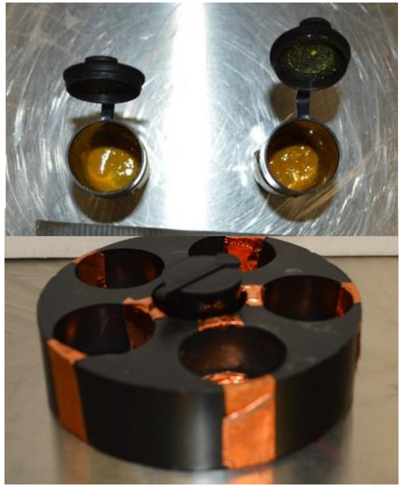


Impact / nipping
- Dust hazard
- Controls
  - Resonator covers

Electrical Power
- Isolation of high power sources
- Controls
  - Separate location for handling energetic materials
  - Aim for ATEX (EU) approved equipment

Overpressure
- Reaction – burn/deflagration
- Controls
  - Burst disc

Quantity
- Reaction – burn/deflagration
- Experimentation
  - Range test mix at harshest conditions (high acceleration; long time)
- Controls
  - Reduce mass of mix
  - Replaces hand mixing



Nelson, 2018

VI. Conclusion on RAM Applied to Energetic Materials

Conclusion on RAM

- The method of mixing differs across the techniques but the hazards for the energetic materials are similar
- Users coming together have benefited from shared experiences
- Shown how RAM users had provided safety assurance in using the technology with energetic materials
- Generation of electrostatic charge and subsequent discharge was the biggest concern for users
  - This is very much material/process condition specific
- Understanding
  - Experiments showed satisfying results for performance
  - Theory for process is young
- The RAM community must use fundamental and applied research to continue to understand the technology

The radical changes in our way of designing & processing new energetics **must not** be done at the expense of **safety**

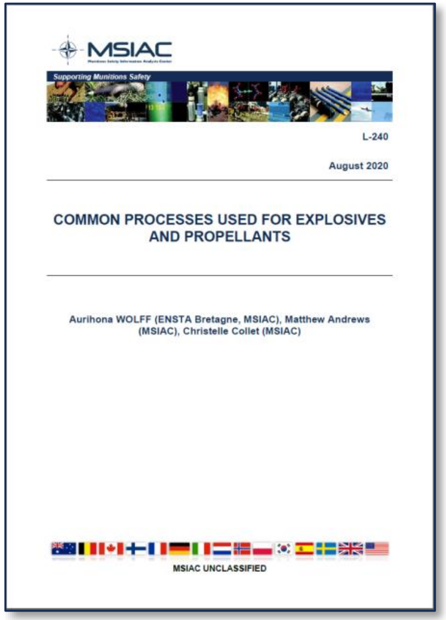
Bibliography Related to the RAM Section

- am Ende, D., Anderson, S., & Salan, J. (2014). Development and Scale-Up of Cocrystals Using Resonant Acoustic Mixing. *Organic Process Research & Development, 18*(2), 331-341. doi:10.1021/op4003399
- Beckel, E., Lee, K., Marin, J., & Shah, A. (2016). Processing of Explosives at ARDEC Using the LabRAM. *2016 Technical Interchange Resonant Acoustic Mixing.*
- Beckel, E. (2018). Environmentally Friendly Energetic Processing Via Resonant Acoustic(R) Mixing. *SERDP & ESTCP Webinar Series.*Deacon, P. (2016). Nitrocellulose Processing in a Flammable Solvent: Designing a Safer Process. *7th Nitrocellulose Symposium*. Montréal, Canada.
- Davey, R., Wilgeroth, J., & Burn, A. (2018). New Age of PBX Manufacturing: Optimisation of RAM. *49th International Annual Conference of ICT*, (p. 5). Karlsruhe, Germany.
- Guymon, C. (2018). In-Process Classification of Explosives. *Improved Explosives & Munitions Risk Management (IEMRM).* Granada, Spain: MSIAC
- Jubb, D. (2018). The Falcon Project: MSIAC: Impact of Resonant Acoustic Mixing (RAM) on Munitions Safety and Suitability for Service. *1st RAM Technical Meeting.* Portland, USA.
- Komanschek, V. (2018). Comparison of Gap Test Results of PBX KS 33: RAM vs. Planetary Mixer. *2nd RAM MSIAC Technical Meeting.* Shrivenham.
- McPherson, M. (2014). Resonant Mix Process Development for Castable Propellants and Related Energetics. *RAM Energetics Conference and Workshop.* Butte, MT.
- Miklaszewski, E. (2018). Resonant Acoustic Mixing of Pyrotechnics at NSWC Crane. *SERDP.ESTCP Symposium*.
- Nellums, R., Terry, B., Tappan, B., Son, S., & Groven, L. (2013). Effect of Solids Loading on Resonant Mixed Al-Bi2O3 Nanothermite Powders. *Propellants, Explosives, Pyrotechnics, 38*, 605-610.
- Nelson, A., Miller, M. (2018). Resonant Acoustic Mixing of High-Energy Composite Materials. *SERDP.ESTCP Symposium*
- Pavey, I.D. (2015). Hazard Assessment of High Speed Slurry Blending Using Computer Modelling of Electric Fields and Potentials. *Journal of Physics: Conference Series*, 646, 012023
- Provatas, A., & Wall, C. (2017). Development, Characterisation & Ageing of an Alternative Plastic Explosive. *48th International Annual Conference of ICT*, (p. 11). Karlsruhe, Germany.
- Thomas, G.W., Prickett, S.E., Richman, S.A., Radack, C.M., Cassell, E., Michienzi, M., Murphy, C.M., Newton, W. (2006). Environmental Security Technology Certification Program 2.75-Inch Motor Manufacturing Waste Minimization Project. *ESTCP WP-9804*
- Zebreg, M. (2018). RAM work at TNO. *2nd MSIAC Technical Meeting: Impact of Resonant Acoustic Mixing (RAM) on Munition Safety and Suitability for Service.* Shrivenham, UK.
- A. Wolff. (2019). L-245: Resonant Acoustic Mixing Applied to Energetic Materials. *Limited Report, L-245*, MSIAC, Brussels, Belgium, 2019.
- A. Wolff. (2019). L-246: Resonant Acoustic Mixing - Performance and Optimization for Energetic Materials. *Limited Report, L-246,* MSIAC, Brussels, Belgium, 2019

More Information

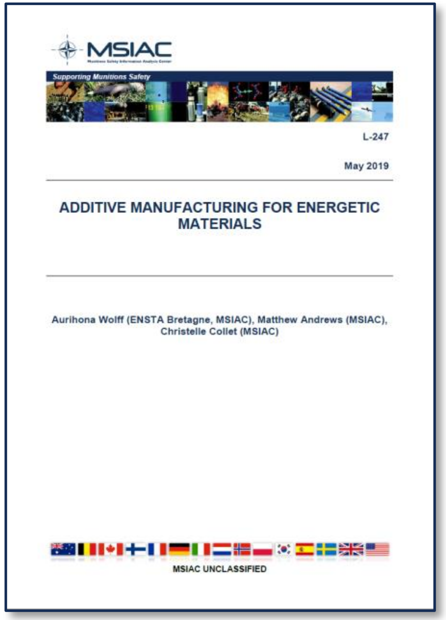
Further information can be found in:

- L-240 for common manufacturing processes,
- L-245 and L-246 for RAM
- L-247 for AM techniques and optimization parameters


MSIAC
Supporting Munitions Safety
L-240
August 2020
COMMON PROCESSES USED FOR EXPLOSIVES AND PROPELLANTS
Aurihona WOLFF (ENSTA Bretagne, MSIAC), Matthew Andrews (MSIAC), Christelle Collet (MSIAC)
MSIAC UNCLASSIFIED


MSIAC
Supporting Munitions Safety
L-245
May 2019
RESONANTACOUSTIC® MIXING APPLIED TO ENERGETIC MATERIAL
Aurihona Wolff (ENSTA Bretagne, MSIAC) Matthew Andrews (MSIAC), Christelle Collet (MSIAC)
MSIAC UNCLASSIFIED


MSIAC
Supporting Munitions Safety
L-246
May 2019
RESONANT ACOUSTIC MIXING PERFORMANCE AND OPTIMIZATION FOR ENERGETIC MATERIALS
Aurihona WOLFF (ENSTA Bretagne, MSIAC) Matthew Andrews (MSIAC), Christelle Collet (MSIAC)
MSIAC UNCLASSIFIED


MSIAC
Supporting Munitions Safety
L-247
May 2019
ADDITIVE MANUFACTURING FOR ENERGETIC MATERIALS
Aurihona Wolff (ENSTA Bretagne, MSIAC), Matthew Andrews (MSIAC), Christelle Collet (MSIAC)
MSIAC UNCLASSIFIED

Any Questions?

https://www.cartoonstock.com/cartoonists/jimallen