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Gradient Additive Manufacturing of Energetics Research (GAMER)

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Final Technical Report

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PROGRESS MEETING 2

29 October 2019 | M.H. Straathof, M.F.J. Koolloos

innovation for life

CONTENTS

- Overview of original project plan, progress and financial status
- Technical progress report
  - WP1: Selection printable compositions
    - Conclusions from D1.1 (literature survey)
    - Summary of D1.2 (range of suitable extrusion compositions)
  - WP2: Layered printing
    - Progress on activities
  - WP3: Hardware adaptation
    - Summary of all hardware and software developed
  - WP4: Gradient printing
    - First gradient print trials
- How to proceed?

OBJECTIVES GAMER

- Build increased understanding of *gradient printing* in the domain of energetic materials in general

- Raise the TRL for 3D-printing of *rocket propellants* by *extrusion based fused deposition modeling* in particular

WORK BREAKDOWN

WP1: Selection printable compositions

- Literature survey on suitable materials (M1.1) and feasible options in extrusion technology for FDM (M1.2) **D1.1**
- Preparation of several batches “printable compositions”
- Initial printability tests of compositions **D1.2**

WP2: Layered printing

- Co-layered 3D-printing of batches of different composition **D2.1**
- Experiments: Bonding strength and burning behavior **D2.2**

WP3: Hardware Adaption

- Requirements, design and construction of special nozzle with two feeds, a static mixing section and pressure sensors (M3.1)
- Control strategy (M3.2)
- Experimental verification (M3.3) **D3.1**

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WP4: Gradient Printing

- Graded 3D-printing of batches of different composition
- Assessment printing performance
- Experiments: Microscopic examination, bonding strength and burning behaviour **D4.1**

WP5: Proof of Principle 3D Object Printing

- 3D object is made **D5.1**
- Burning behavior is predicted based on experience gained during previous tasks
- Printing is assessed
- Burning behavior of 3D object is assessed

WP6: Management and Reporting

- Intermediate reports after Y1 and Y2
- Final report at completion including future outlook on printing EM objects
- Scientific paper

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TECHNICAL PROGRESS

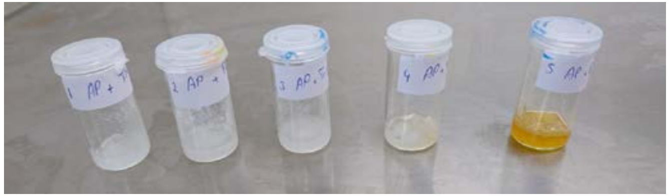
WP1 - SELECTION PRINTABLE COMPOSITIONS

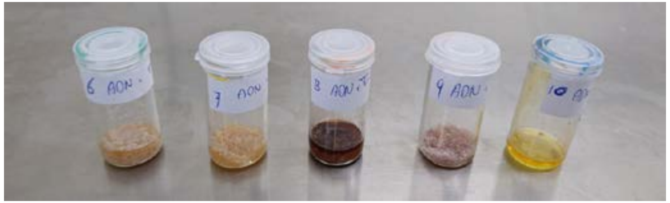
WP1 SELECTION PRINTABLE COMPOSITIONS

- Main conclusions from the literature survey:
  - Rocket propellants
    - Wide range of rocket propellant applications exists where the use of AM FGM can offer advantages.
    - Realization of these benefits requires design methods and software tools for unconventional designs.
  - Propellant compositions
    - Wide variety of possible propellant compositions provide room for incorporating UV -curable binders.
    - Knowledge about rocket propellants and UV-curable binders should lead to multiple printable propellant candidates.
  - Additive manufacturing and gradient printing
    - Numerous printers appear to be available that can combine multiple materials but in many cases this is restricted to discrete gradients or to outer surface modifications. Printers that can truly create gradients by mixing materials are scarce.
    - On the software side there seems to be a lack of available tools or file formats to create and handle designs utilizing a gradient.
    - It is concluded that FDM printing using a UV curing step to harden the printed object is most suitable for AM FGM with energetic materials.

WP1 SELECTION PRINTABLE COMPOSITIONS

- Summary of D1.2: Range of suitable extrusion compositions
  - AP and RDX most promising oxidizers based on compatibility studies
  - Solid load of 70-75 wt% was found to be optimal for extrudability
  - Acrylate content optimized for minimal morphology changes upon deposition and curing
  - Range of compositions recommended for use in the remainder of the project:
    - 43-50 wt% ammonium perchlorate
    - 22-25 wt% RDX
    - 4 wt% ethyl-NENA
    - 21 wt% acrylates
    - 0-10 wt% melamine
    - < 1 wt% additives (photo initiator, colorant, stabilizer, etc.)



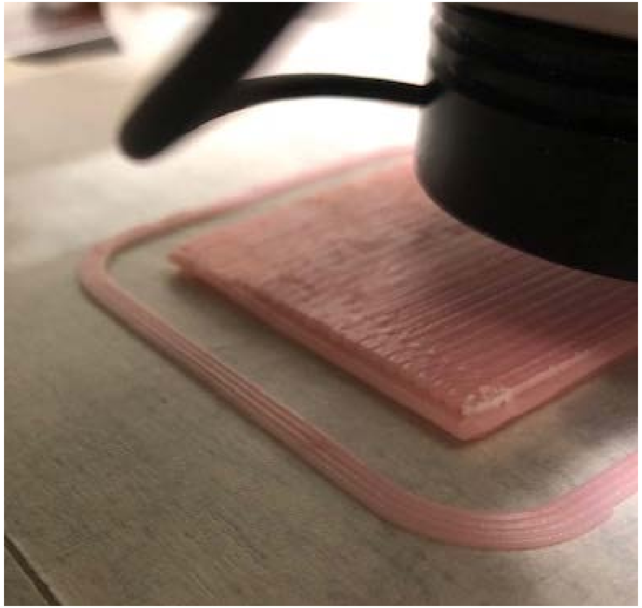


TECHNICAL PROGRESS

WP2 – LAYERED PRINTING

WP2 LAYERED PRINTING

- Progress so far:
  - Printer initiation
    - Dealt with many teething problems
    - Path deposition tests
    - Material optimization
    - Process optimization
    - Print trials
  - Scaling up to larger items
  - Microscopic analysis of prints
  - Tensile testing (still in progress)
  - Chimney burn testing (still in progress)
- Deliverables D2.1 and D2.2 not yet finished



WP2 LAYERED PRINTING

- Dealing with teething problems of printer hardware
  - Bugs in firmware / user interface
    - Freezing of controls

      > solved through firmware update
    - Sudden uncontrolled movements

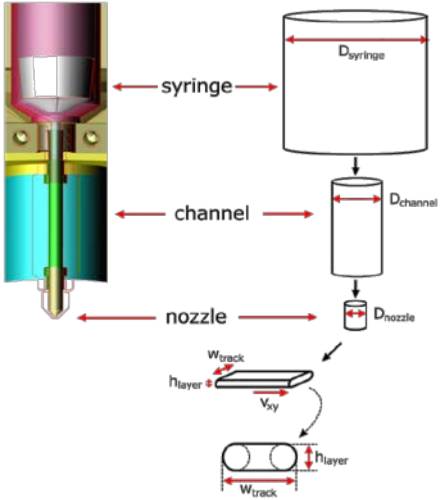
      > not yet solved, but under control
  - No UV source control in G-code

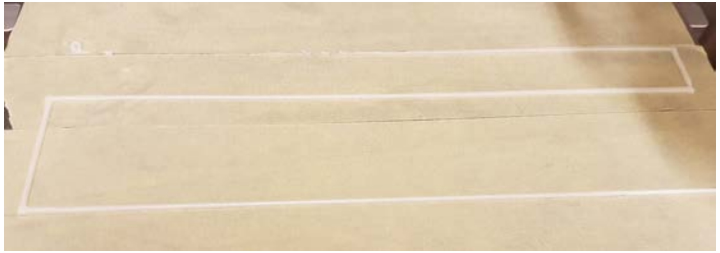
    > solved by implementing UV source control
  - Interference issues between active and inactive nozzle during printing

    > circumvented by using different extruder configuration

WP2 LAYERED PRINTING

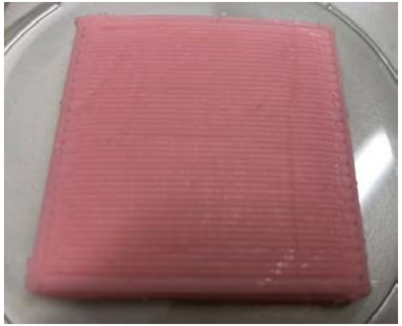
- Path deposition tests
  - Three main printing parameters to be set:
    - Piston speed
    - Print head speed
    - Layer height
  - The extruded path width follows
  - In theory, the relationship between these parameters is fixed for incompressible fluids
  - In practice, effects like die-swell and shrinkage alter the relationship
  - Path deposition tests are used to characterize the relationship for each new material





WP2 LAYERED PRINTING

- Material optimization
  - Further material optimization was required to improve the printability of the compositions selected in WP1
  - Printability problems encountered:
    - Too low viscosity, resulting in material leaking from nozzle
    - Demixing, resulting in slow clogging of nozzle
    - Shrinkage of deposited material
  - Viscosity problem solved by keeping solid load content constant using melamine to replace oxidizer (essentially changing the fuel:oxidizer ratio and thus the burn rate)
  - This also solved shrinking problem
  - Demixing continuous to be an issue, mostly with larger objects



WP2 LAYERED PRINTING

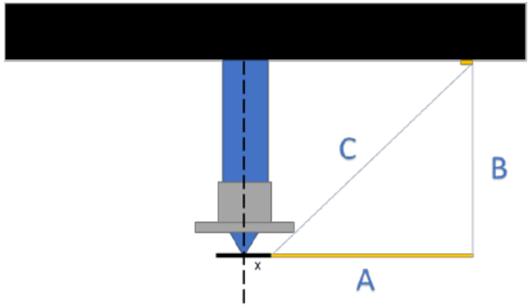
- Process optimization
  - The printing process itself also required further optimization
  - Problems encountered included:
    - Curing of material inside active nozzle

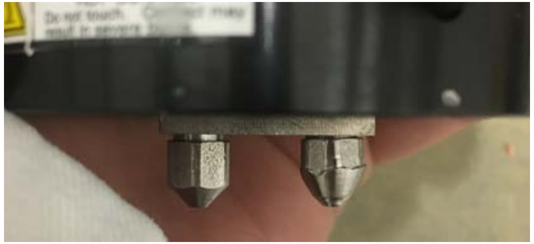
      > solved through nozzle shielding and UV off during travel moves
    - Curing of material inside passive nozzle

      > solved by using different extruder configuration and UV off when passive
    - Difficult to define nozzle offset

      > partly solved by using different extruder configuration
    - Air inclusions in syringe after filling

      > solved using special filling tool

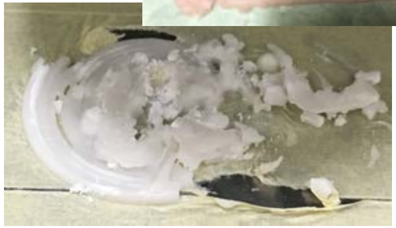




WP2 LAYERED PRINTING

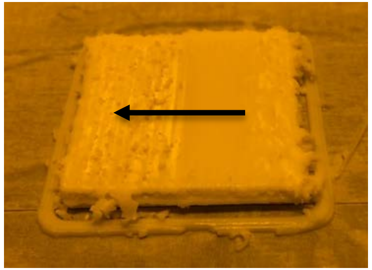
- Print trials
  - Many print trials have been performed

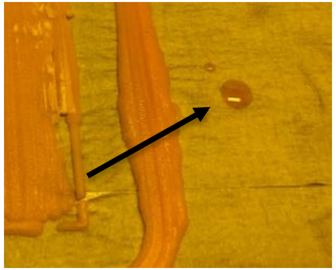



WP2 LAYERED PRINTING

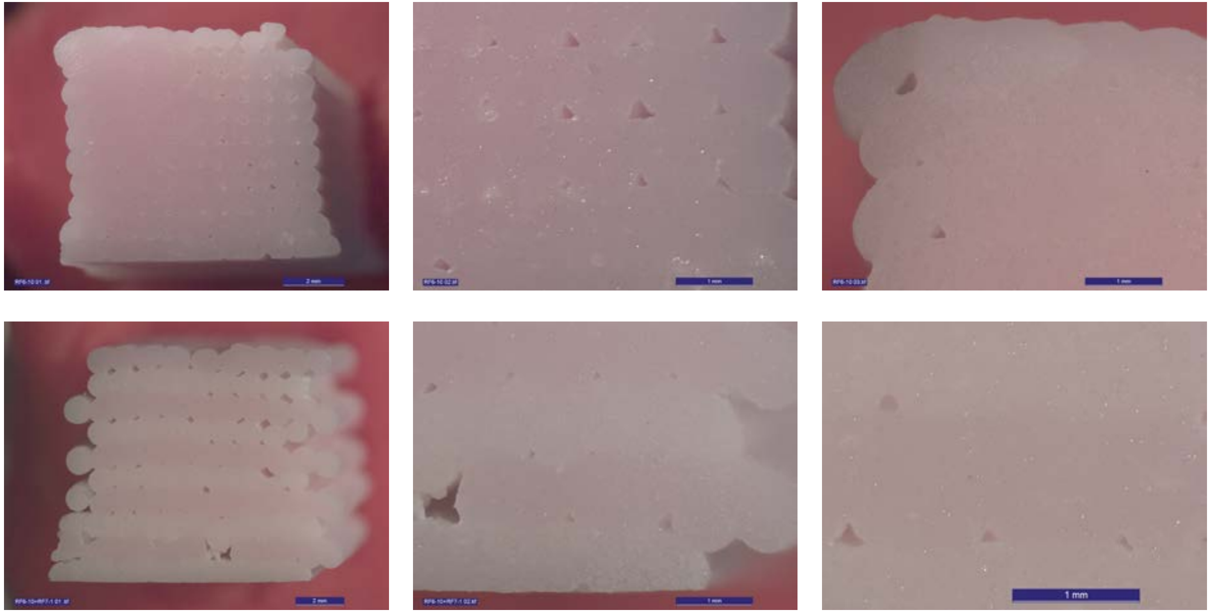
- Scaling up to larger items has introduced new problems
- Problems seem to be related to extruding a larger percentage of the syringe
- Most likely cause is demixing of the composition
- The following solutions are being tried:
  - Changing the particle size distribution (bimodal optimal?)
  - Adding a bonding agent
  - Lowering extrusion pressure by using an elevated syringe temperature (up to 60 °C)



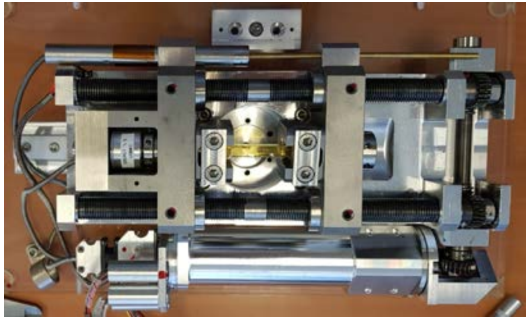


WP2 LAYERED PRINTING

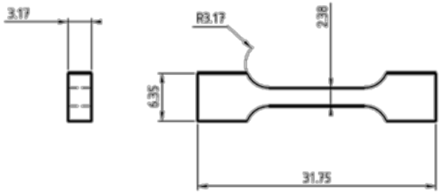
- Microscopic analysis
  - Analysis of inter-filament interfaces
  - Both single and dual material
  - No visual transition between deposited paths
  - Open channels between filaments need to be eliminated

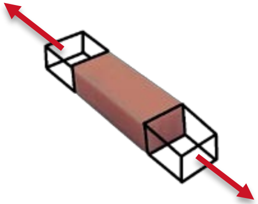


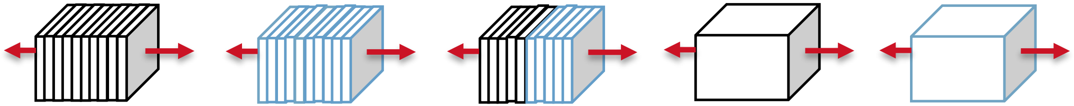
WP2 LAYERED PRINTING



- Tensile testing
  - Specimens will be tested in micro-mechanical tensile testing device
  - ¼ JANNAF dog bone
    - Clamping has been proven problematic with similar samples
    - Alternative: attach aluminum blocks to rectangular sample using adhesive
  - Samples have been partially prepared, but tests need to be performed
  - Different configurations will be tested







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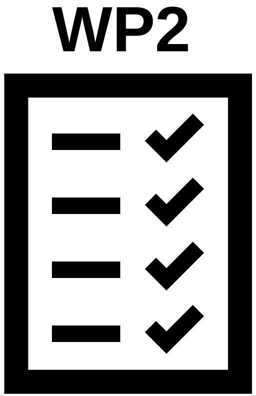
WP2 LAYERED PRINTING

- Chimney burn testing
  - Fast and slow burning compositions
    - 70 wt% AP/RDX
    - 60 wt% AP/RDX
  - Printed and manually prepared samples
  - Tested in N_2 flow under 40 bar
  - Ignition using hot wire

  - Still investigating what causes the unexpected burning behavior
  - Suspected that RDX content needs to be lowered

SUMMARY WP2

- Most teething problems have been dealt with
- Successful small scale print trials have been performed
- Scaling up to larger items looks promising, but some challenges remain
- Microscopic analysis of printed items revealed great adhesion between print layers and paths
- Open spaces need to be eliminated, but this should not be a big issue
- Tensile testing and chimney burn testing in progress



TECHNICAL PROGRESS

WP3 – HARDWARE ADAPTATION

WP3 HARDWARE ADAPTATION

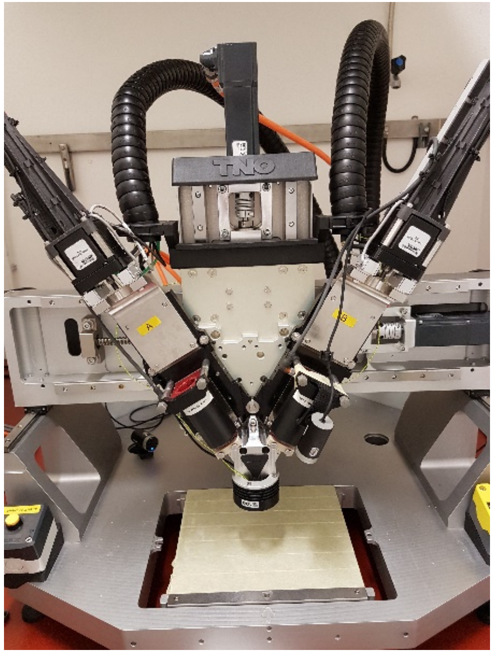
- Original plan was to construct a nozzle assembly for an existing printer
  - No suitable COTS printer was found fulfilling safety and performance requirements

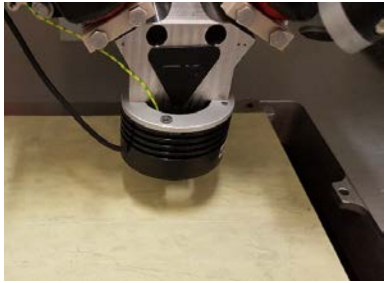
- Instead, a full printer was built, largely financed by Dutch MOD and TNO
  - Based on existing TNO design, but heavily modified for EM safety

- Software and firmware development also turned out to be a considerably larger effort than foreseen
  - Extra safety features included in firmware/UI
  - Existing open source software package heavily modified to allow for the inclusion of continuous functional gradients

WP3 HARDWARE ADAPTATION

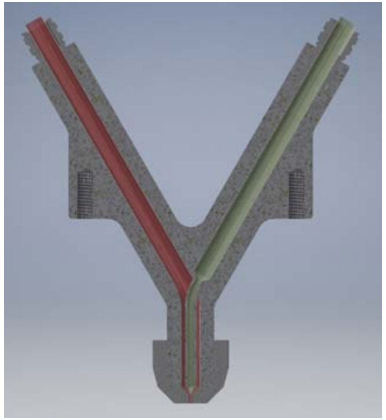
- Large build volume (200 x 200 x 100 mm)
- Dual material feed
- Modular extruder configurations
  - Single, double parallel, double V
- Modular print head
  - Parallel, Y, co-extruded
- UV curing mechanism integrated in print head(s)
- EM safety features include
  - Hard and soft limits on temperature and pressure
  - All parts anti-static or grounded
  - Potential leakage/contamination kept away from moving parts

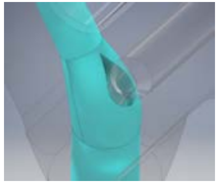


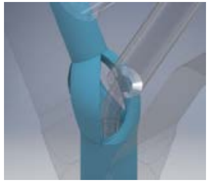



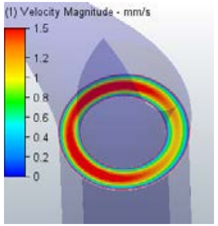
WP3 HARDWARE ADAPTATION

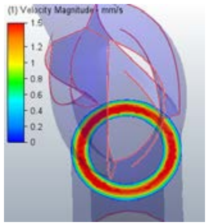
- Coaxial print head
  - Combines two material feeds into core/shell filament
  - Merging region optimized for maximum concentricity and minimum pressure drop
  - Final product 3D printed from stainless steel, with minimal post -processing

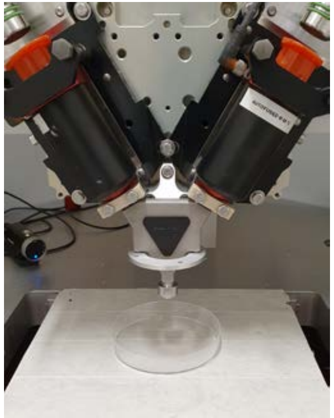






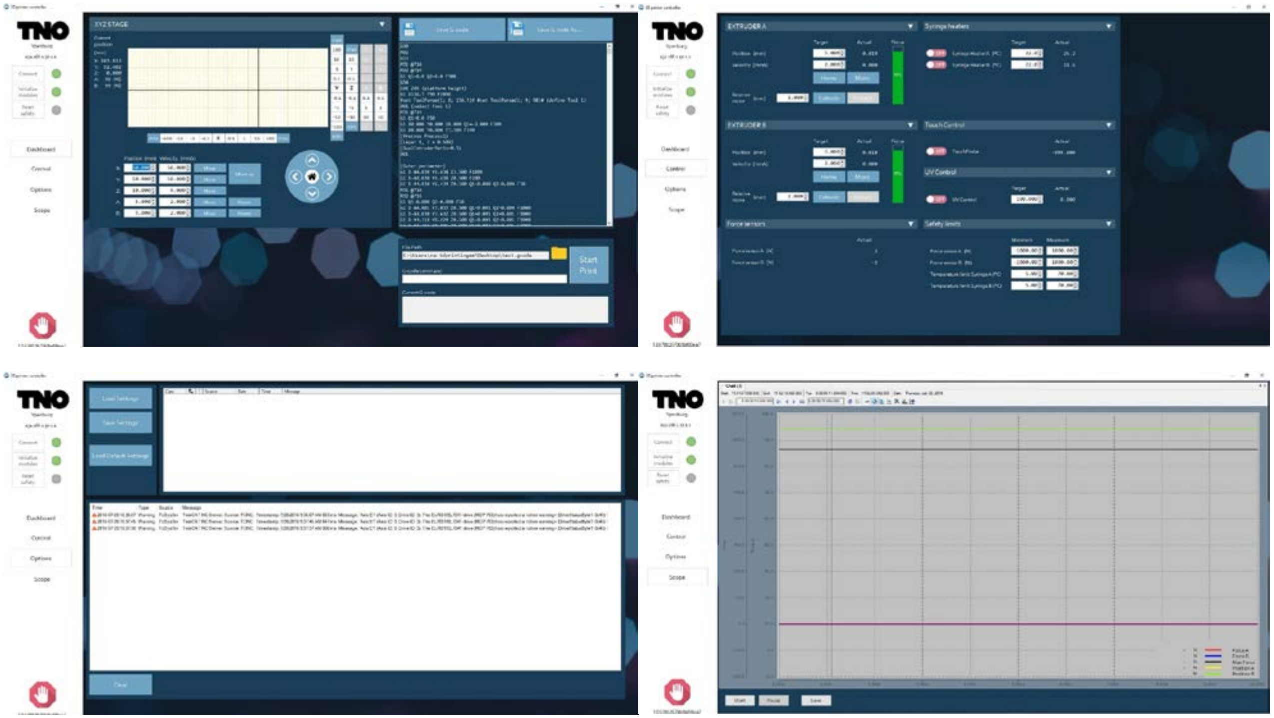






WP3 HARDWARE ADAPTATION

- Firmware & UI
  - PLC controlled using TwinCat PLC Control
  - TNO designed user interface
  - Live axes control
  - G-code editor
  - Live UV and heater control
  - Force and temperature limits
  - Error log
  - Scope

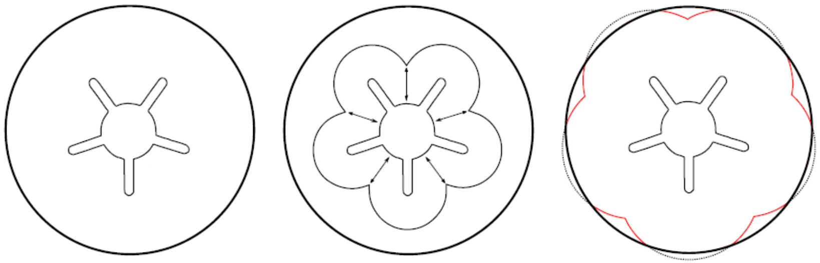


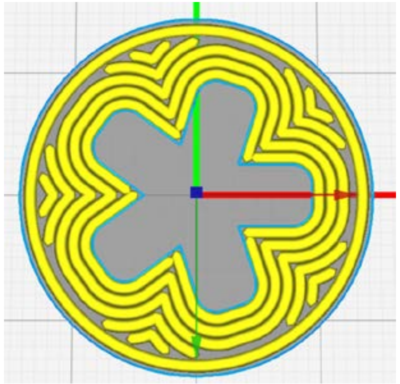
WP3 HARDWARE ADAPTATION

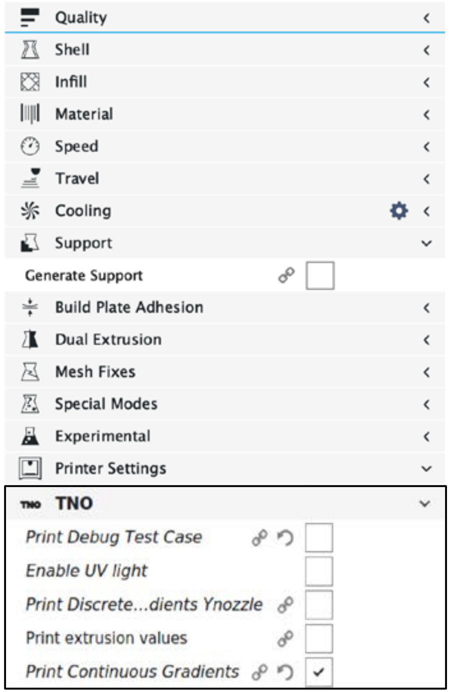
- Slicer software
  - Printer originally came with Simplify 3D software
    - \+ Very capable, with options such as editable layers and customizable supports
    - \- Not open source
    - \- Slow update cycle
  - Switch to Ultimaker Cura software
    - \+ Open source
    - \+ Free
    - \+ Fast update cycle
    - \- Not as capable as Simplify 3D

WP3 HARDWARE ADAPTATION

- Slicer software
  - All standard options available
  - New infill patterns designed specifically for gun and rocket propellants
  - Translation to G-code dialect required by printer PLC



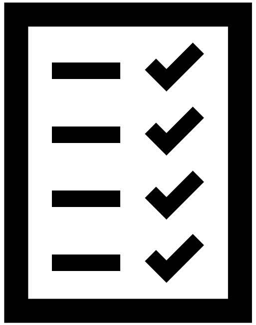




SUMMARY WP3

- A fully modular, multi-material gradient printer was designed and built
- A co-extrusion print head was developed
- Firmware and UI written for printer
- Slicer software adapted for gradient printing

WP3

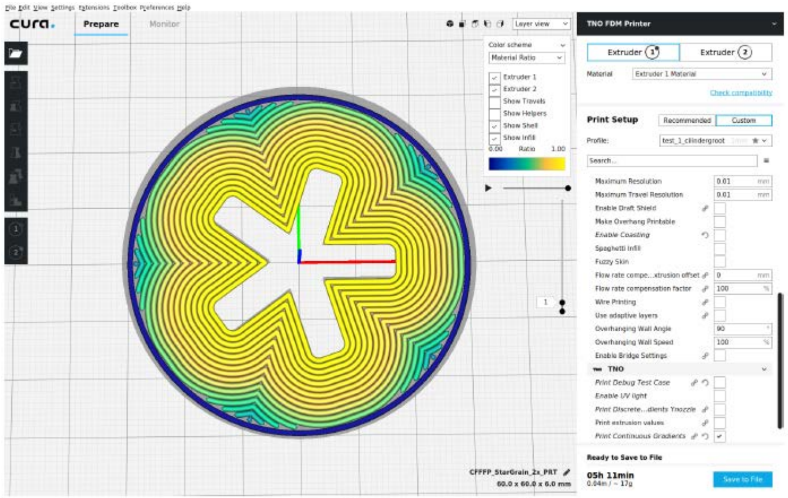


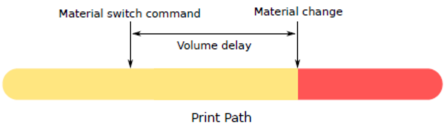
TECHNICAL PROGRESS

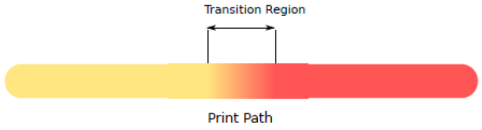
WP4 – GRADIENT PRINTING

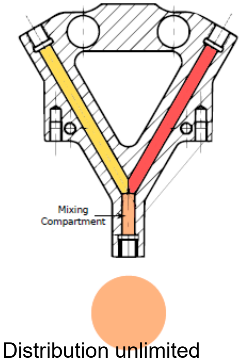
WP4 GRADIENT PRINTING

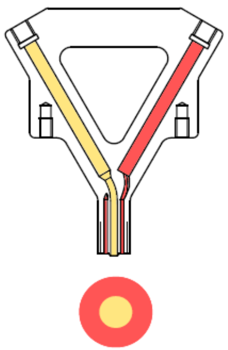
- Further software changes required
  - Cura does not have option for slicing (functionally) graded objects
  - User can now define a gradient function along the burn direction of the propellant (in tabular form)
  - Additional features, such as transition delay and a transition region have been included





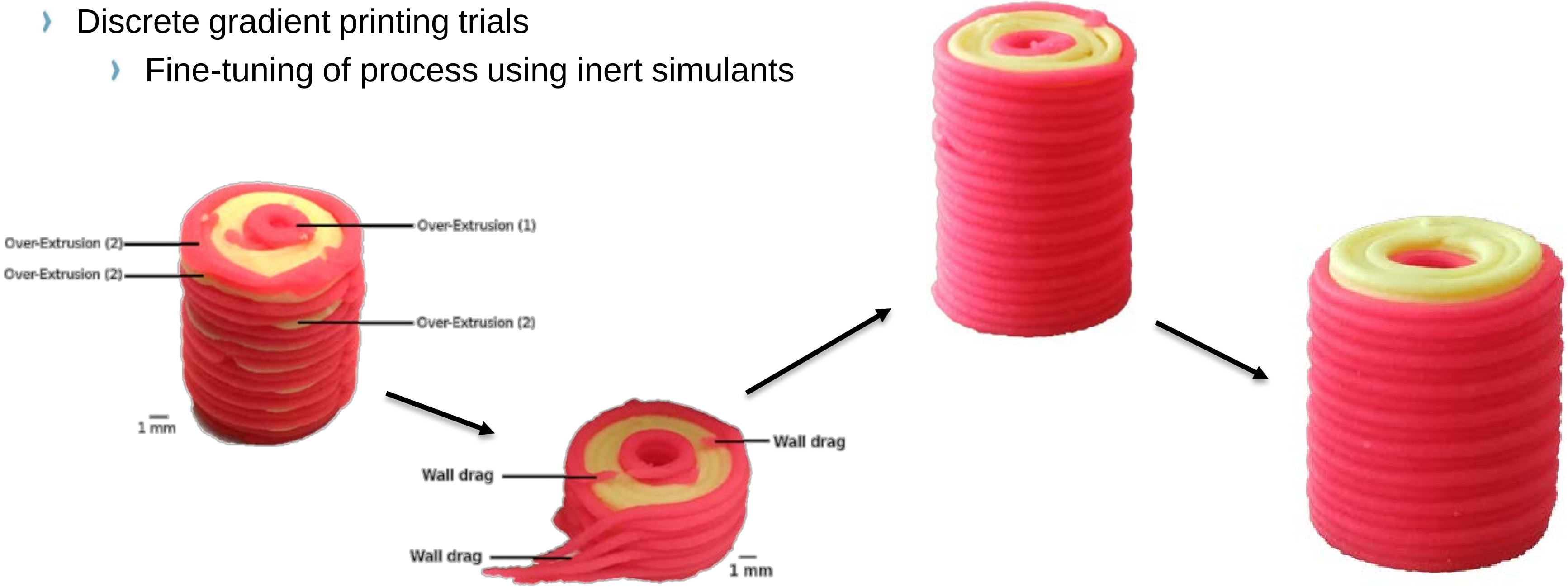






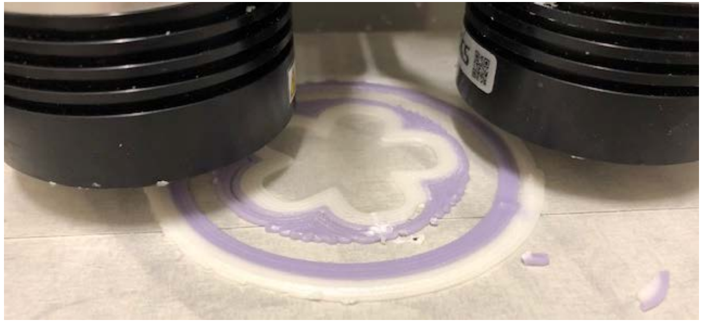
WP4 GRADIENT PRINTING

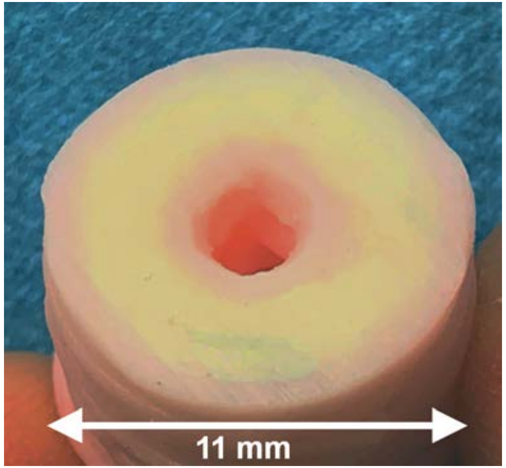
- Discrete gradient printing trials
  - Fine-tuning of process using inert simulants

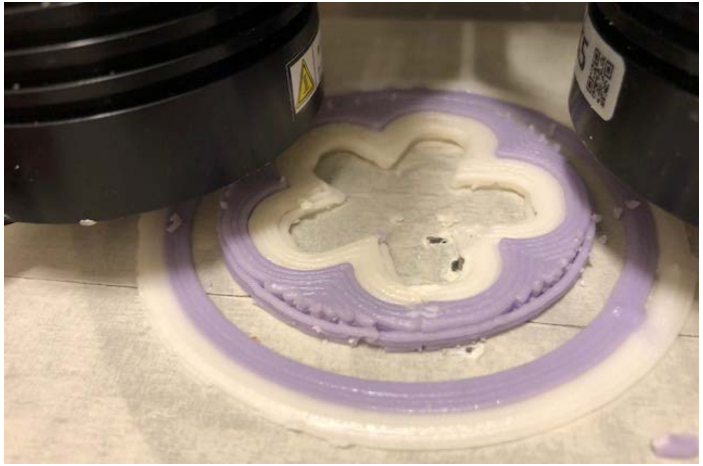


WP4 GRADIENT PRINTING

- Discrete gradient printing trials
  - Switch to propellant compositions







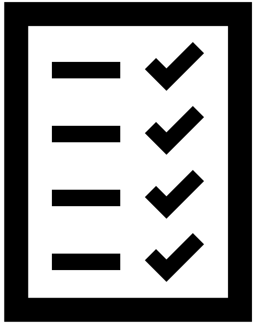
WP4 GRADIENT PRINTING

- Continuous gradient print trials
  - Using co-extrusion print head
  - Using inert simulants

SUMMARY WP4

- Discrete gradient print trials successfully completed with inert and energetic materials
- First continuous gradient print trials successfully complete with inert simulants
- System and materials ready for continuous gradient trials with energetic materials

WP4



HOW TO PROCEED?

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- Short term (remainder of 2019)
  - Recalibrate material composition based on what has been learned
    - 3 new compositions will be defined, with the following changes compared to the current material composition:
      - Bimodal AP
      - Lower RDX fraction
      - Different plasticizer (potentially non-energetic)
      - Added melamine to keep solid load constant
    - Print sample geometries
  - Perform study into demixing of filled resins
    - Influence of solid load, particle distribution, plasticizer

HOW TO PROCEED?

- Longer term (2020)
  - To finish the original project plan:
    - Continuous, reliable operation (without demixing, clogging nozzles, etc.)
    - Eliminate open spaces between print paths
    - Determine burn characteristics (Chimney burner tests)
    - Design demonstrator with continuous gradient
    - Print demonstrator (using co-extrusion nozzle)
    - Perform demonstration (L* test)

THANK YOU FOR YOUR ATTENTION

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