Next Generation 3D Printed Propulsion Charges for Guns

Always a Step Ahead

ARDEC

ARMAMENTS

AIChE Northern NJ - March Dinner Meeting **Duncan Park** March 21, 2017

Previously presented in Defense Manufacturing Conference 2016 By Elbert Caravaca

UNPARALLELED COMMITMENT & SOLUTIONS Act like someone's life depends on what we do.



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PARTICIPANTS



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- 🎘 leidos
- AMRDEC





- Dana Kaminsky, Elbert Caravaca, Joseph Laquidara, Carly Occhifinto, Carlton Adam, David Keyser, Robin Crownover, Viral Panchal, John Bolognini, Michael Fair, Daniel Schmidt, Giuseppe Di Benedetto, Joe Quijano, Leon Moy, Paula Cook, Rajen Patel, Jeff Kraft, James Zunino, Tom Carty, David Bird, Sarah Longo, Duncan Park
 - Randy Mrozek, Jason Robinette, Shawn Cole
- Leidos International
 - Keith Eagen
- AMRDEC

ARL

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- Greg Drake
- CAPCO LLC
 - Chris Williams
 - Theodore Spence
 - Alice Dow
 - Jesse Coquoz
 - Rochester Institute of Technology
 - Mark Olles



OUTLINE



- Background
 - Gun Propulsion

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- Potential Benefits: Why 3D print a charge?
- Technical Approach
 - Vat Photo Polymerization Development
 - Material Extrusion Method Development
 - Interior Ballistic Modeling Development
- Technical Conclusions
- Path forward





- Gun propulsion today is conversion of chemical energy into kinetic energy through controlled energy release
- Design variables
 - Geometry
 - Burn Rate
 - Ignition







BACKGROUND - MANUFACTURE



- Conventional Manufacturing provides certain geometries
 - Flake
 - Ball
 - Stick
 - Granular
 - Perforated

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- Slotted
- Driven by cost and ease of loading
- Ingredients also limited the methods of manufacture (Nitrocellulose, Nitroglycerin, etc.)



BACKGROUND - CURRENT PROCESSES



- Solvent-based Processing
 - Can be relatively cheap (often function of cutting step)
 - Multiple facilities
 - Flexibility
 - Mixer type
 - Solvent system
 - Drying Procedure
 - Grain deformation
- Solventless Processing
 - Uses roll mill vs. horizontal mixer

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- More expensive
- Water based
- Consistent geometry
- More processing & formulation restrictions
- Continuous Processing
 - Ball Powder[®]
 - Very cheap
 - Lot to lot variation
 - Twin Screw Extrusion (TSE)





Sigma mixer

Press Extruder



Sweetie Barrel



Slurry Tanks



Rolling/ Milling



Twin-screw Extruder (TSE)



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T'ABLE 1. IBHVG GRAIN GEOMETRIES AND CODES

GRAIN CODE	GEOMETRY	
1 2	7-perforated cylinder 1-perforated cylinder	
5 5	cord rectangular strip sphere	
6 7	slotted tube 37-perforated hexagonal	
8 9	19-perforated hexagonal 19-perforated cylinder	
10	7-perforated hexagonal	



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Degressive Grain Neutral Grain Pressure Progressive Grain Travel

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Ball-regressive





Additive manufacturing allows the rate of energy release from a propellant to be highly optimized by controlling the surface area of the grain.

At the start of the interior ballistic cycle, the available volume is small and a low rate of gas generation is required to maintain pressure. As the projectile moves down-bore, volume increases drastically and a higher rate of gas generation is required to maintain pressure.

使导致的运用,但在这次

Multiperf grain geometries are the state of the art in surface area control, however they offer at best a 2:1 ratio in surface area increase over the life of the grain. Additive mfg. techniques are capable of much higher ratios of surface area increase.





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POTENTIAL BENEFIT – IMPROVED PERFORMANCE (CONT'D)



Better control over gas generation results in a higher Piezometric efficiency, more energy delivered to the projectile, and faster launch velocities for the same peak pressure and acceleration.





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Additive Manufacturing

• Additive Manufacturing (AM), *n* - process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies (ISO/ASTM 52900:2015(E)).



Source: Short Course Lecture Slides, "From 3D Printing to Factory Floor," Massachusetts Institute of Technology, Cambridge, MA, July 2016.



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TECHNICAL APPROACH



- Investigate using COTS/Novel UV curable resins in a Vat Photo polymerization 3D printers
- Investigate using COTS/Novel materials for extrusion method → FDM 3D printers
- Investigate using COTS/Novel materials for binder jetting method 3D printers (modified)





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Resin Tank

- Form 1+ SLA 3D printer by Formlabs
- <u>https://formlabs.com/3d-printers/form-1-plus/</u>
 - 405 nm UV LED; Proprietary Resins
 - Spare Resin Tanks and Build Platforms



HARDWARE - MATERIALS



Materials

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- RESINS Investigated to date
 - Clear FLGPCL02
 - Black FLGPBK02
 - Castable FLCABL02

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- Cure at 405 nm UV light
- methacrylate based
- Inert solid fillers (simulant)
 - Cornstarch
 - CaO (Lime)
 - Melamine





NH

NH2



	Filler	Density (g/cc)	Resins	Highest Solids % by weight	Yield	Observations
	Dechlorane Plus	~1.8	Clear	0%	0	Would not cure
	Cornstarch	~1.3	Clear, Black	40%	6 out of 10	Viscosity limited
	CaCO3	~2.7	Black	0%	0	Would not mix
	CaO	~3.35	Black, Castable	60%	1 out of 24	Mixed well
7	Melamine	~1.57	Castable	50%	3 out of 10	Requires better mixing and drying of filler



100% Resin Clear

Target shape 12mm x 12mm x 12mm cubes



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SOLIDS LOADING STUDY (CONT)





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50% Resin Clear / 50% CaO (Lime)



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SOLIDS LOADING STUDY (CONT)







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SOLIDS LOADING STUDY (CONT)







SOLIDS LOADING STUDY (CONT)



Quick assessment using RDX and black resin

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- Hardware: 395 nm Lamp UV; variable intensity MAX 4 W

395nm UV lamp Intensity	Exposure Time	Material	Sample	Status/ Observations
Lowest setting (unknown value)	5 seconds	BLACK FLGPBK01 – 40% LIME (CaO) – 60%	~thin	CURED!
Lowest setting (unknown value)	5 seconds	BLACK FLGPBK01 – 40% CLASS V RDX – 60%	~thin	CURED!

- Samples here were much smaller then samples used previously
- Lime (CaO) used for these studies were ~140 micron versus the Class V RDX ~5 – 10 micron size



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SOLIDS LOADING STUDY (CONT)









50% Resin Castable / 50% Melamine

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Evidence of settling



SOLIDS LOADING STUDY (CONT)



Changes to Resin/Filler preparation

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- Initial work was done with hand mixing
- Introduced drawing vacuum prior to printing in order to remove any bubbles introduced with hand mixing
- Moisture impacted both print quality and mix quality
 - Inert fillers are now dried prior to mixing/printing
- Went from hand mixing to using a homogenizer/emulsifier in order to improve homogeneity and get less air entrapment
- Future improvements
 - Addition of additives to improve suspension of filler in resins
 - > 1 hour showed evidence of filler settling
 - Better control of particle size of filler
 - Target size is ~ 5 micron for nitramines for gun propellants
 - Potential to introduce bi-modal or tri-modal particle size distribution for better packing





RDECOM HIGH STRAIN RATE MECHANICAL PROPERTIES



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• Comparing Uniaxial Compression Tests of Propellants at an average strain rate of 100/s







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TECHNICAL CONCLUSIONS



- The Form 1+ printer can print the fillers used in this study to ~50% solids loading
- The printer does exhibit limitations when it comes to viscosity
 - Form 2 printer has a heated tank which could allow for higher viscosity materials
- Typical propellant material development never included investigating light absorption properties of said materials
 - Need to characterize fillers and binder system when it comes to UV light absorption and scattering
- Quality of the initial samples had moderately high variability





Material Extrusion Development

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3D PRINTING AND CHARACTERIZATION



- •Printer: Hyrel System 30M Serial No. 15-00069
- •Extruder: Mk1-250 Serial No. 15-00292
- •Nozzle: 0.5 mm opening

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- •Printed Part: 12mm x 12mm x 12mm Cube
- •Filament: PLA (Green Leaf) supplied by MakerBot



Hyrel S30M Serial Number

Mk1-250 Extruder

Cube



MATERIAL DEVELOPMENT



Energetic Thermoplastic Elastomer (1/2)

- Material Extrusion Method requires thermoplastic materials
- Several Energetic Thermoplastic Elastomers (ETPE) were investigated since the 80s and to 2000s as potential new gun/rocket propellant binders
 - Examples:

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- BAMO-AMMO (poly(bis-3,3-azidomethyl-oxetane-b-3-azidoomethyl-3-methyloxetane))
- BAMO-NMMO (poly(bis-3,3-azidomethyl-oxetane-b-3nitratomethyl-3-methyloxetane))
- These are co-polymers comprised of hard and soft blocks → many possible ways to vary the material properties
- ETPEs were processed with various solid energetic fillers and plasticizers and evaluated
 - Example: co-layered propellant for mortar, tank, and medium caliber gun applications
- Synthesis of BAMO and AMMO monomers are underway at Picatinny (Keith Eagen and Joe Laquidara)



*Source: G.A. Lindsay et al., Energetic Polyoxetane Thermoplastic Elastomers: Synthesis and Characterization, NWC Technical Publication 6945, Naval Weapons Center, China Lake, CA, December 1988.

**Source: M. Kramer et al., "Environmentally Friendly Advanced Gun Propellants: Final Technical Report," SERDP PP-1363, Sep 2004.



MATERIAL DEVELOPMENT



Inert Thermoplastic Elastomer

- ETPE is preferred over inert thermoplastics, however it is difficult to acquire ETPE (i.e. cost and time)
- Commercially available inert thermoplastics can be loaded with energetic solid fills and be 3D printed
- Wide range of materials exist:

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- ABS (Acrylonitrile-butadiene-styrene), PLA (polylactide), HIPS (high impact polystyrene), PE (poly ethylene), PEEK (polyethyl ethyl ketone), PVA (polyvinyl acetate), TPU (thermoplastic polyurethane), etc.
- Wide range in material properties:
 - T_g, mp, viscosity, hardness, elasticity, chemical resistance, solubility, compatibility with energetic propellant ingredients, etc.
 - All these properties collectively affect the processibility and final characteristics of propellant formulation



MATERIAL DEVELOPMENT



Inert Thermoplastic Elastomer

- Several commercial off-the-shelf (COTS) thermoplastics were investigated:
 - Polycaprolactone (Capa[™] 6500, www.perstorp.com) mp = ~60 °C. Pellets could be easily processed without having high temperature processing equipment.
 - ABS (Lustran® 433 ABS, INEOS ABS USA) designed for injection modling
 - PLA (Ingeo[™] Biopolymer 3052D and 3D850, NatureWorks, LLC) designed for injection molding (3052D) and 3D printable filament (3D850)
- Small window of temperature requirement exists
 - HMX will start self-heat to decompose ~272* °C → thermoplastic needs to be processible at 222 °C considering 50 °C safety margin
 - Operational and storage temperature requirement of ammo should be considered
 - High temperature requirement for ballistic firing is set at ~63 °C

*Based on the DSC results (tested at 5 °C/min Ramp rate). Actual self-heat to ignition temperature may be lower.





FORMULATION



ETPE-Based

- Remnants from a previous program were reprocessed to make filament feedstock
 - BN-7 ETPE based (BAMO-NMMO (70:30) co-polymer) synthesized and delivered by Aerojet (now Aerojet Rocketdyne), Sacramento, CA.
 - Contains energetic plasticizer
 - Contains RDX
- In parallel, BAMO and AMMO monomer synthesis is underway as previously mentioned
 - No specific formulation is set
 - Additional co-polymer synthesis and characterization are needed
- In process of acquiring and evaluating various BAMO-AMMO polymeric binders made by Thiokol (now Orbital-ATK), Utah



FORMULATION



Inert TP-Based

- Polycaprolactone: $CaCO_3$ (60:40 by wt) Inert simulant
- ABS pellets were mixed while varying CaCO₃ loading (35-65 wt%)
- Polymeric binder poly lactic acid (PLA)

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- PLA is a widely used material for AM, it combusts and has melt point below the auto ignition temperature of HMX
- IngeoTM Biopolymer 3D850 3D printing monofilament, high heat grade
- IngeoTM Biopolymer 3052D designed for injection molding application
- Filler calcium carbonate, an energetic fill simulant
- Solid loadings were varied from 45 to 55 and 65 wt%
 - All three were successfully processed using Ingeo[™] 3052D
 - Only the formulation with 55 wt% loading was successfully processed using Ingeo[™] 3D850, others were not attempted

Physical Properties	Ingeo™ 3052D	Ingeo™ 3D850	ASTM Method
SG	1.24	1.24	D792
Melt Index (g/10min),210 °C/2.16 kg	10-25	7-9	D1238
Relative viscosity	3.0-3.5	4.0	D5225
Melt Temperature (°C)	150-165	165-180	D3418
Glass Transition Temp. (°C)	55-65	55-60	D3418
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UNCLASSIFIED RDECOM® **PROCESSING AND CHARACTERIZATION** U.S.AR **ETPE-Based*** Young's Modulus Plots (from Uni-axial Compression Test) 13 Aug 2001 13 Aug 2001 Legend 13 Aug 200 AFRO IFT / JA2 PROPELLANTS AEROJET/JA2 PROPELLANTS AEROJET/JA2 PROPELLANTS NAXAL COMPRESSION TEST NIAXIAL COMPRESSION TEST NAXAL COMPRESSION TEST TESTED AT 21 C TESTED AT -32 C TESTED AT 63 C Black: JA2 Yellow Green: JAZ LOT HCLESJO1 4-0 +18.00 EROJET LOT 8194 STRESS STRESS **PAP-8194** STRESS HE LOT HELES 1014-00 (MPo) (MPa) (MP of ACRONET LOT 8194 +52.00 +32.00 A2 LDT HCL93014-00 NET LOT 8194 Y-axis: Stress +18.00 X-axis Strain STRAIN (% $T = 63 \circ C$ (Left), 21 $\circ C$ (Middle), -32 $\circ C$ (Right)



Post-Test Specimens (Left) Pull-Test (Middle)

- Mechanical properties of PAP-8194, under compression, are "good" across temperatures
- Failure modulus of PAP-8194 is comparable to that of JA2
- Shows work hardening
- Bonding b/t layers are "Good"

SEM Image at the Layer-Layer Interface (Right)

*Source: T. Manning et al., Development and Performance of High Energy High Performance Co-Layered ETPE Gun Propellant For Future Large Caliber System, US Army, RDECOM-ARDEC, Picatinny Arsenal, NJ, November 2006.

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- ~1.5 kg of BAMO-AMMO (62.5% BAMO) was located
- Solids loading and processibility studies were planned
 - Increase solids incrementally from 25 wt% to 75 wt%
- Processed using Ross Mixer (vertical blades w/ heating capacity)
- Formation w/ 25wt% solids slumped at room temperature
- Solids loading study was halted



Melting Experiment (Left Section), Mixing (Middle Section), Extruded Filament (Right)

- Polycaprolactone:CaCO₃ (60:40 by wt) was mixed and extruded using a sigmablade batch mixer and heated ram press
- Processing temperatures ranged 68 77 °C
- Filament's OD is 1.75 mm (nominal)

ABS

- Brabender mixer at PPI, NJIT, NJ, was used
- Processing temperature exceeded 200 °C (higher than desired) work halted



PROCESSING AND CHARACTERIZATION



Inert TP Based Material PLA

Methods

- Co-rotating twin-screw extruder (TSE) was used for mixing and extrusion at ARL
 - ThermoFisher scientific Process 11

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- Barrel bore diameter is 11 mm
- Barrel length to diameter (L/D) ratio is 40
- Single feed port
- Typical output is 20 2,500 g/hr
- Solid fill (calcium carbonate) and thermoplastic binder (PLA) in a pellet form were added manually through a single feed port
 - Not all of calcium carbonate was able to be fed in the first run due to an unwanted and rapid rise in torque
- Extruded filaments were pelletized and fed back into the TSE along with left-over calcium carbonate from the previous feeding operation
- The above step was taken 3-4 times to ensure having a homogeneous and consistent composition throughout the batch

Processing Parameters

 RPM = 50; Torque = ~6.4 Nm; Temp. = 170 – 190 °C for Ingeo[™] 3052D and 200 °C for Ingeo[™] 3D850 <image><image><image><section-header><image><section-header>

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Control Screen

Exudate on Conveyer Belt

TSE – Top View







Filaments

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TGA Results: 55wt% PLA, 45wt% PLA, 35wt% PLA (Left, Middle, and Right)

- TGA results show that the mixing and extrusion process yielded homogenous products
 - Only ~1 wt% difference from the intended formulation
 - All three samples within a lot showed consistent results → mass reduction ranged 34-35 wt% (for 35:65 PLA:CaCO₃ formulation)
- TGA is a suitable method to determine the quality of mix/extrusion



3D PRINTING AND CHARACTERIZATION



- No energetic materials have been printed
- SOP on 3D printing of energetics is being reviewed for approval
- Inert filaments of various binder and filler compositions have been printed
- To better characterize anisotropic nature of printed part, a cube (12mm x 12mm x 12mm) was chosen as the configuration of choice for uniaxial compression test
- Hyrel 3D System 30 Printer was used
 - Up to 4 extrusion heads
 - Various extrusion nozzles available:
 - Thermoplastics
 - Terracotta

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• UV curables



Hyrel 3D System 30 Printer



- Failed to print well
- Print temperature may had been too high
- Filament thickness may had varied more than the desired
- Polycaprolactone has lower than desired mp and was shelved



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3D PRINTING AND CHARACTERIZATION



PLA

	Run 1	Run 2	Run 3	Run 4
Layer Ht (mm)	0.2	0.1	?	?
Seam Position	Aligned	Random	?	?
Fill Density (%)	100	95	95	?
Fill Pattern	Rectlinear	Line	Concentric	?
Top/Bottom Fill Pattern	Rectlinear	Concentric	Concentric	?
Solid Infill every (layers)	0	12	0	?
Fill Angle (degree)	45	0	0	?
Skirt Loops	2	4	4	?
Brim width (mm)	4	24	24	?
Speed (mm/s)	30	20	30	?
Infill Overlap (%)	10	10	10	?
Extruder Temp (°C)	225	220	210	200
Bed Temp (°C)	80	85	45	?





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3D PRINTING AND CHARACTERIZATION



PLA

•12mm x 12mm cubes were attempted to be printed using PLA w/ 4 different slices.

•Slicing parameters were varied slightly.

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•Run 1 was the best print while three other runs printed progressively worse.

•Nozzle temperature setting was set at 225, 220, 210, and 200, for Run 1, Run 2, Run 3, and Run 4, respectively.

•Important parameters appear to be:

•Extruder temperature

- Infill density
- •Seam position
- •Print speed

•Based on the DSC results, it is found that the nozzle temperature setting is not as same as the material temperature.

•"Railing" is less prominent on the top face \rightarrow perhaps due to heated nozzle tip



3D PRINTING AND CHARACTERIZATION





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Extrusion Temp = 190 °C Speed = 10 mm/s Fill angle = 45° Nozzle Diameter = 0.35mm Fill Density = 80%

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3D PRINTING AND CHARACTERIZATION



PLA Filled with CaCO₃

•Failed to print first time \rightarrow due to the diameter of filament exceeding the nozzle inlet clearance

•Quality of print is less than desirable \rightarrow probably partially due to significant variability in filament diameter

- filament diameter affects the volumetric flow rate
- •Machine assumes that the filament has a constant diameter and uses the linear feed rate to calculate volumetric flow rate
- •Higher quality in filament fabrication is necessary
 - •Will consult polymer processing expert
 - •Perhaps visit a filament factory

•Design of Experiment will be used to evaluate the sensitivity and effects of various independent variables on the quality of print

•Corners of cube will be rounded/chamfered to reduce the cornering-effect



SUMMARY AND CONCLUSIONS



- Material extrusion method has been investigated for producing additively manufactured gun propulsion charge
- Several types of energetic thermoplastic elastomers (ETPE) are being synthesized and evaluated:
 - BN-7 (BAMO-NMMO) based propellant
 - BAMO-AMMO co-polymer

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- Several types of inert thermoplastics are being evaluated as suitable propellant binders and as ETPE simulant
 - Filaments are produced for 3D printing
 - Polycaprolactone, ABS, and PLA have been investigated → viscosity across processing temperature and mp are critical
- Hyrel 3D System 30 printer is being used to 3D print test specimens
 - Processing parameters are being studied for print optimization
 - Printed test specimens will undergo characterization for mechanical properties, density, burning behavior, etc.











Example: Sphere



Surface area:
$$-\frac{dV}{dx} = 4\pi (r - x)^2$$

Burn rate: $\frac{dx}{dt} = \beta p^{\alpha}$

Rate of gas production = $-\frac{dV}{dt} = -\frac{dV}{dx} \cdot \frac{dx}{dt}$

Complex, 3D-printed grains:



- Exposed surfaces are generated abruptly during combustion
- Burning surfaces intersect and change the topography
- Method of decomposition into primitive shapes changes as the grain burns
- Difficult to find a general method for obtaining an analytical solution for $\frac{dV}{dx}$



- Place any arbitrary geometry into an Eulerian mesh using Volume of Fluid method
- Use the phase-field method to regress the grain surface, simulating combustion.
- The grain volume as a function of surface regression is thus calculated numerically.

0.0	0.30	0.50	0.30	0.0
0.30	0.96	1.0	0.96	0.30
0.50	1.0	1.0	1.0	0.50
0.30	0.96	1.0	0.96	0.30
0.0	0.30	0.50	0.30	0.0

Elemental volume fractions of a circular solid in a 5x5 mesh



7-Perf hex grain with bounding box





VALIDATION – SPHERICAL GRAIN





APPLICATION – 3D-PRINTED GRAIN



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SUMMARY AND CONCLUSIONS



• Vat Photo Polymerziation Development

- Solids limit achieved with COTS resins from Form Labs ~50%
- Need to better characterize solids and investigate other resins
- Material Extrusion Method Development
 - Hit limits of ARDEC pilot processing capabilities due to temperatures needed
 - There various parameters that influence quality of print
 - Filament quality also drives print quality
- Interior Ballistic Modeling Development
 - Continue development model(s) of complex geometries





PATH FORWARD



- Vat Photo Polymerization Development
 - Repeat solids loading study with energetic fillers
 - Investigate bi-model or tri-modal particle size distributions for better volumetric packing
 - Look into custom resins that work at different wavelengths depending on UV absorption properties of resins/energetic fillers
 - Look into liquid plasticizers (energetic and inert)
- Material Extrusion Method Development
 - Continue work with inert solids to better understand processing limits
 - Continue development of ETPE materials
 - Further develop design of experiments for critical parameters for higher solids loaded materials
- Interior Ballistic Modeling Development
 - Continue development model(s) of complex geometries

