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Qualifying Synthetically Manufactured Alternatives to Natural Materials Using the Calcium Silicide ($CaSi_2$) Project as an Example

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Outline

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- $CaSi_2$ Example
  - Background
  - Requirements
  - Iteration 1
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- Revised Qualification Process

Background

Issue: Current Government TDPs Call Out Natural Materials that:

1) Have Limited Specification Compliant Supplies
2) May Not Consistently Meet the Desired Mil-Spec Requirements
3) May Vary Within Large Lots of Material (Thousands of Pounds)

Desire: Identify Synthetically Manufactured Alternatives that:

1) Provide a Long-Term Source of Supply
2) Guarantee Mil-Spec Compliance (Chemical and Particle Size)
3) Results in a More Consistent Product (Lot-to-Lot and Within Each lot)

Challenges:

1) Identify Key Material Properties
2) Replicate Key Material Properties for the Same End Item Performance

Need to Identify Synthetic Material Alternatives

Qualification Process

1) **Identify Requirements**
   - Specification
   - Manufacturing Process
   - Final Product(s)

2) **Identify Key Material/Manufacturing Process Characteristics**
   - Chemical Composition
   - Particle Size Distribution
   - Material Treatments

3) **Manufacture and Test Material**
   - Design of Experiments

4) **Analyze Manufacturing and Testing Results**

5) **Identify Additional Important Material/Manufacturing Process Characteristics**

6) **Repeat Steps 3 Through 5 as Necessary**

7) **Perform Qualification Testing**
   - Material
   - Final Product(s)

Planning Saves Time and Money Down the Road

$CaSi_2$ Example – Background

Calcium Silicide ($CaSi_2$):

- Fuel for M52A3B1 20mm Primer
- Fuel for Alloy Manufacturing

- Mil-Spec Compliant Material is Almost Impossible to Locate
- Very Few Lots on the Market are Even Close to the Mil-Spec
- ATK Forced to Perform Special 'Out of Spec' Qualification Testing on Last 3 Production Lots

PGU-27A/B TP

PGU-28A/B SAPHEI

Alternate Calcium Silicide Source of Supply is Necessary

Approved for Public Release, PAO 370-12, dated 17 April 2012
Distribution A: Approved for Public Release, Distribution Unlimited, 22 CFR 125.4(b)(13) applicable

$CaSi_2$ Example – Requirements

Specifications:

- MIL-C-324C: Calcium Silicide, Technical
  - Chemical Composition
    - Min 60% Si
    - Min 30% Ca
    - Max 3.8% Fe
  - Particle Size Distribution
    - Retained on 150µm Sieve – 1% Max
    - Retained on 106µm Sieve – 1% Max
    - Retained on 75µm Sieve – 6 to 12%
    - Retained on 45µm Sieve – 25 to 50%
    - Passing 45µm Sieve – 40 to 65%

Manufacturing Process:

- Lake City Primer Manufacturing
  - Minimal Tooling/Process Modification
  - Material Treatment Processes

Final Products:

- M52A3B1 Electric Primer
- 20mm Cartridges

PGU-27 A/B TP | PGU-28 A/B SAPHEI | PGU-30 A/B TP-T | Mk244 APDS | M940 MPT-SD

Large Range of Chemical and Size Requirements

$CaSi_2$ Example – Iteration 1 Plan

Material:

- 10 Different Rotary Atomized Synthetic Samples
  - Varying Chemical Composition
    - % Excess Si (silicon)
    - % Free Fe (iron)
  - Varying Particle Size Distribution
    - Surface Area
  - With and Without Weak Acid Treatment
    - Lab Scale Method
- 3 Control Samples

Testing:

- Lake City Primer Manufacturing
  - Processability
- Primer Performance
  - Resistance
  - Pellet Weight
  - Primer Sensitivity
  - Primer Time
- Cartridge Performance
  - Pressure, Velocity, Action Time (PVAT)
  - Function & Casualty (F&C)
  - Environmental

Assembled a DOE to Identify the Key Properties

CaSi$_2$ Example – Iteration 1 Manufacturing & Testing

Manufacturing:

- 1 Lot of Primer Mix Was Manufactured from each Synthetic CaSi$_2$ Sample
- Charging Operators Rated each Sample During Production
  - Tendency to Cake
    - o Primer Mix Stuck Together
  - Flow Characteristics
    - o Smearing

Control Sample

Representative Synthetic Samples

Testing:

- Mix Impact and Friction Sensitivity
  - Within Normal Control Limits
- Primer Performance
  - Resistance
    - o Lower than Normal Production
    - o 3 Samples Measured No Resistance
  - Pellet Weight
    - o All Samples Measured Heavy
- Cartridge Performance
  - Not Performed

Initial Samples Would Not Work for Production

$CaSi_2$ Example – Iteration 1 Results & Analysis

1) Samples Are Not Ideal for Production:

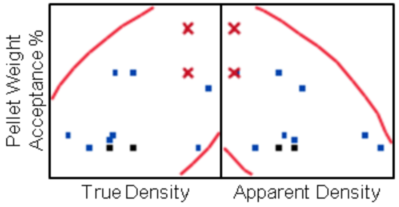
- Primer Mixes Appeared Wetter
- High Primer Pellet Weights
- Low Primer Resistance

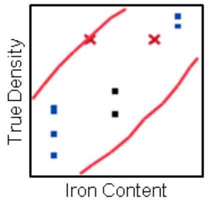
2) Potential Causes

- More Spherical Particle Shapes
  - About Half the Particles Were Not Milled and Still Met the Particle Size Distribution Requirements
- More Fines
  - Full Scale Weak Acid Treatment May Wash Some Fine Particles Away

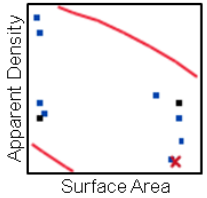
3) Instructed DOE Results:

- Improve Pellet Weight Acceptance

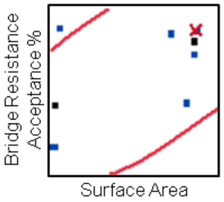




  - High True Density
    - o High Iron Content
  - Low Apparent Density
    - o High Surface Area



- Improve Resistance Acceptance
  - Increasing Surface Area
    - o Jagged, Non-Spherical Particles



DOE Identified Modifications to Improve Manufacturability and Performance

$CaSi_2$ Example – Iteration 2 Plan

Material:

- 3 Different Rotary Atomized Synthetic Samples
  - Constant Chemical Composition
  - Varying Particle Size Distribution
    - o Larger Starting Particle Sizes
    - o Varying Milling Methods
  - With Weak Acid Treatment
    - o Production Method
- Primer were Manufactured from 1 Sample
  - Showed the Best Properties
    - o Densities
    - o Surface Area

Testing:

- Lake City Primer Manufacturing
  - Processability
- Primer Performance
  - Resistance
  - Pellet Weight
  - Primer Sensitivity
  - Primer Time
- Cartridge Performance
  - Pressure, Velocity, Action Time (PVAT)
  - Function & Casualty (F&C)
  - Environmental

Modified Synthetic Material to Improve Acceptance and Processability

$CaSi_2$ Example – Iteration 2 Manufacturing & Testing

ATK

Manufacturing:

- 1 Lot of Primer Mix Was Manufactured from the Synthetic $CaSi_2$ Sample
- Charging Operators Rated each Sample During Production
  - Tendency to Cake
    - No Difference from Production
  - Flow Characteristics
    - No Difference from Production

Testing:

- Primer Performance
  - Resistance
    - Lower than Normal Production
  - Pellet Weight
    - Measured Within Requirements
- Cartridge Performance
  - PVAT
    - Within Specification
  - F&C
    - Within Specification
  - Environmental
    - Acceptable Results

Second Iteration Samples Improved, But Still Would Not Meet All Requirements

$CaSi_2$ Example – Iteration 2 Results & Future Plans

Improved Pellet Weight Acceptance and Processability

Still Not Production Ready:

- Low Resistance

Potential Causes:

- Eliminating the "Other" in the Chemical Composition

Future Plans:

- Increase the Resistance
  - Adding Impurities to Synthetic $CaSi_2$
  - Increase Oxygen Passivation Layer
- Perform a Full Material Qualification

Modifications to Increase Material Resistance are Possible

$CaSi_2$ Example – Lessons Learned

Identify and Understand Key Material Properties Up Front

- Chemical Composition
  - Spec Requirements
    - Ca, Si, and Fe Contents
  - Important “Other” Materials
- Particle Size Distribution
  - Spec Required Size
    - Size Within the Required Range
  - Particle Shape
    - Spherical vs. Jagged
- Potential Material Changes During Product Manufacturing
  - In Process Material Treatments
    - Weak Acid Treatment

Lessons Learned Can Save Money on Future Projects

Revised Qualification Process

1) **Identify Requirements**
   - Specification
   - Manufacturing Process
   - Final Product(s)
2) **Identify Key Material/Manufacturing Process Characteristics**
   - Chemical Composition
     - Required Components
     - Potential Impurities/“Other” Components
   - Particle Size Distribution
     - Size
     - Shape
   - Material Treatments
3) **Manufacture and Test Material**
   - Minimize Deviations from Standard Production Equipment and Processes
   - Design of Experiments
4) **Analyze Manufacturing and Testing Results**
5) **Identify Additional Important Material/Manufacturing Process Characteristics**
6) **Repeat Steps 3 Through 5 as Necessary**
7) **Perform Qualification Testing**
   - Material
   - Final Product(s)

Finalized Qualification Process

References

1) Hafner, Matthew, Randall Busky, and Mark Mansfield. *Thermodynamic Testing Methods in Energetic Material Evaluation.* AIChE Meeting, 2008. Presentation.

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