Spray Coating of Binders

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14. ABSTRACT

This project sought to demonstrate that fluidizing Mg (magnesium) powder with liquid spraying could coat Mg powder particles with a binder. Successful operation would greatly diminish or exclude the emission of VOCs (volatile organic compound) into the atmosphere while providing powder that would meet flare requirements when incorporated into a flare composition. The binder would either be dissolved in a VOC with the latter being recycled or would be a melt. Verification of the process would be achieved through the capability to stably fluidize and coat Mg powder, physical examination and analysis of the powder to determine the extent of the coating and handling safety, and radiometric performance of flare pellets. The latter would contain compositions of the coated Mg blended with PTFE (polytetrafluorethylene) powder. The project's scope was in response to the SON's (statement of need) overall requirement to reduce environmental and health impacts of energetics manufacturing and specifically to develop solventless processing.

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Table of Contents

List of Tables

List of Figures

List of Acronyms

CM ≡ Countermeasure
EPA ≡ Environmental Protection Agency
ESD ≡ Electrostatic Discharge
EVA ≡ Ethylene-Vinyl Acetate Copolymer
FPP ≡ Frontier Performance Polymers Corp.
g ≡ Gram
h ≡ Hour
HAP ≡ Hazardous Air Pollutant
Hi ≡ High
in ≡ Inch
Lo ≡ Low
MEK ≡ Methyl Ethyl Ketone
Mg ≡ Magnesium
mg ≡ Milligram
Mid ≡ Middle
min ≡ minute
mL ≡ Milliliter
mm ≡ Millimeter
N/A ≡ Not Applicable
No ≡ Number
PA ≡ Polyacrylate Resin
psi ≡ Pounds Force per Square Inch
PTD ≡ Pyrotechnics Technology Division
PTFE ≡ Polytetrafluorethylene
s ≡ Second
SEM ≡ Scanning Electron Microscope
SMCA ≡ Single Manager for Conventional Ammunition
SON ≡ Statement of Need
TPE ≡ Thermoplastic Elastomer
VOC ≡ Volatile Organic Compound
W/sr ≡ Watt per Steradian
wt ≡ Weight
µm ≡ Micrometers

Keywords

Binder
Flare
Fluidized Bed
Magnesium
Polyacrylate Resin
Polytetrafluoroethylene
Pyrotechnic
Solvent
Spraying
Thermoplastic Elastomer
Volatile Organic Compounds

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Abstract

This project sought to demonstrate that fluidizing Mg (magnesium) powder with liquid spraying could coat Mg powder particles with a binder. Successful operation would greatly diminish or exclude the emission of VOCs (volatile organic compound) into the atmosphere while providing powder that would meet flare requirements when incorporated into a flare composition. The binder would either be dissolved in a VOC with the latter being recycled or would be a melt. Verification of the process would be achieved through the capability to stably fluidize and coat Mg powder, physical examination and analysis of the powder to determine the extent of the coating and handling safety, and radiometric performance of flare pellets. The latter would contain compositions of the coated Mg blended with PTFE (polytetrafluorethylene) powder. The project's scope was in response to the SON's (statement of need) overall requirement to reduce environmental and health impacts of energetics manufacturing and specifically to develop solventless processing.

Numerous trials incorporating modifications to the fluidized bed and liquid spraying system were executed to achieve a unit that was capable of producing coated magnesium in the quantities necessary for evaluation. The system included a bench top fluidized bed system with 5liter steel and glass tubs. The former was used initially to enable viewing of the states of fluidization and spraying. A borescope camera system was installed within the metal tub to be able to view inside it. Other key components included hardware for the atomization and spraying of the liquid component, pressurized tank for the storage and flow of the liquid, inlet filter for fluidizing air and outlet filter. The inlet filter was a 'sandwich' of two different mesh sizes with a Wurster inlet configuration. Sieving of the magnesium powder was found necessary to reduce the particle size range of the powder. The wideness of the range made it essentially impossible to set fluidization conditions that could fluidize all of the loaded powder simultaneously.

Soxhlet extraction was selected for determining the amount of coating on the powder. Morphology on the coated powder to include SEM (scanning electron microscope) and instrumental particle size had been planned. After mixing the coated powders with PTFE further tasks were to be ESD (electrostatic discharge) sensitivity determination and pressing the composition into pellets. These pellets would be intentionally ignited and burn times and radiometric outputs measured. Composition made through the standard process would also be evaluated to provide a baseline.

Due to the actual effort being greater than originally planned there were insufficient funds to install solvent recycle into the fluidized bed and spray coating system, to spray EVA (ethylene-vinyl acetate copolymer) melt, and to perform morphology and ESD testing.

After optimization the system was able to produce approximately 413 g (gram) of coated Mg powder with close to 2 wt (weight) % TPE (thermoplastic elastomer) binder for making flare composition. The material was blended with PTFE and pressed into pellets at three different percentages of Mg content. Pellets were burned in a flare tunnel and burn time and radiometric intensity in the 3μm (micrometers) – 5μm region measured. Pellets made from flare mix prepared per the current method ('uncoated' Mg) were also tested. The limited data demonstrated the pellets with the coated Mg burned approximately twice as fast as the uncoated samples, and the integrated radiant intensities, a measure of total energy, were approximately the same.

This implied that the fluidized system delivered an equal product and could be a viable, environmentally superior technology. Further work would be needed to optimize the process and equipment. Immediate needs would be the inclusion of solvent recovery, increasing the amount of coating on the Mg powder, a refinement of the Soxhlet extraction technique or replacement for it, fabricating and testing flare pellets closer to the size actually used and conducting morphological analysis and ESD testing. Further on the spraying of EVA melt and the simultaneous spray coating of Mg and PTFE would be attempted.

Objective

The project intended to demonstrate that a fluidized bed could be used to greatly reduce or eliminate the emission of VOC into the atmosphere during flare manufacture. This would be accomplished by spraying a VOC with dissolved binder onto fine Mg powder. The VOC would flash off leaving the binder as a coating on the powder particles. The VOC would be reclaimed in the apparatus and recycled for reuse as the dissolving agent for the binder. This process would greatly reduce the release of the VOC into the atmosphere. A variant on this would be spraying a binder at or above its softening temperature. The temperature would be just below the maximum safe temperature for processing of energetic materials. The figures below are top level displays of the concept:

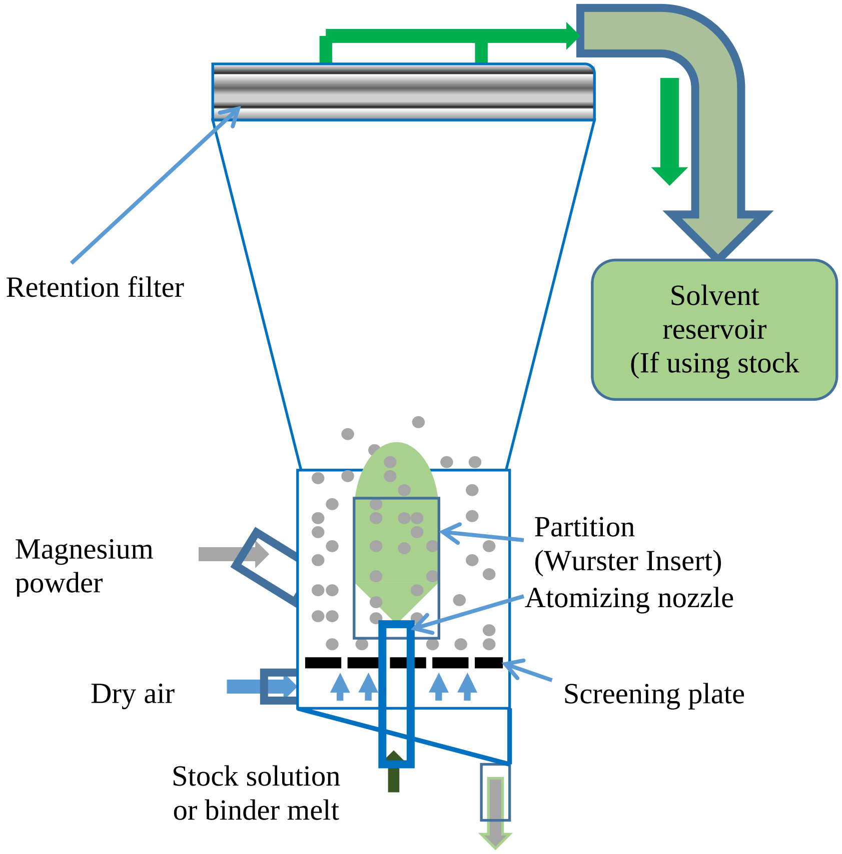

Figure 1. Generic Fluidized Bed Concept for Coating Magnesium Powder

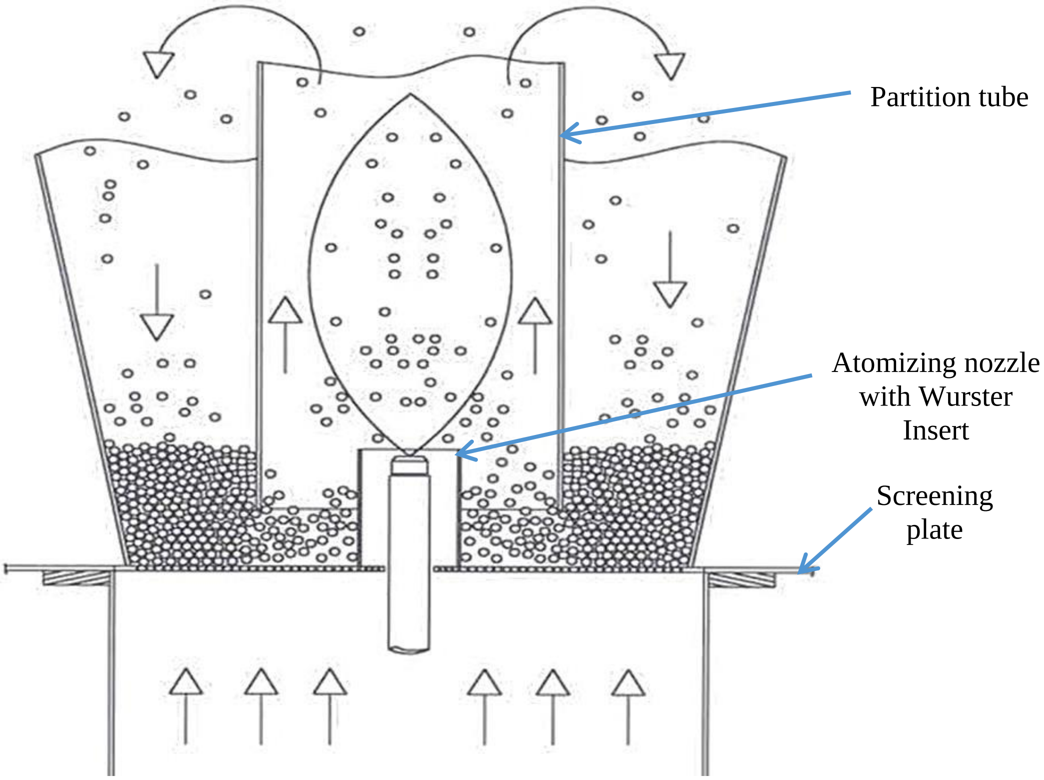

Figure 2. Generic Details of Fluidized Bed

Coated Mg powder from the fluidized process would be combined with PTFE. The resulting mix would be pressed into small flare pellets. These pellets would then be subjected to radiometric and burn time testing. Additional characterization would be particle morphology, ESD sensitivity of the mix and pellet mechanical strength. To give a basis of comparison identical formulations would be made but with Mg and PTFE coated by open evaporation of the solvent containing the binder material and subjected to the same tests. This is the current method. Success for the project was defined by the ability to efficiently and stably fluidize and coat the Mg and that test results on the coated powder and pellets were equal or more favorable to material produced by the current process.

This project met the overall objective of the SON to which it was submitted. That objective is to reduce environmental and health impacts of energetics manufacturing and to develop a technology of interest specifically mentioned in the SON; solventless processing techniques. This cost-effective and scalable technology can greatly improve environmental impact, industrial hygiene and safety without undue economic impact. Since it makes use of a unit operation common in industry materials, processes and equipment can be readily transferred to the production base but with novel modifications to achieve reduced or no solvent amount. This would eliminate exposure of workers to solvent.

Background

VOCs are used throughout the manufacture of various pyrotechnic items and commodities but by far the greatest users of VOC are manufacturers of Mg-PTFE-TPE based flares. The general manufacturing sequence for this composition is dissolving the TPE in acetone and introducing it into a high dispersion mixer. Then Mg and PTFE powders are added to the mixer and the materials blended together. Deposition of the TPE on the Mg and PTFE particles is accomplished in one of two ways. One, decanting and flashing of the acetone until there is no evident liquid phase. Two, introduction of n-hexane which causes the TPE, none of which are miscible in n-hexane, to precipitate and coat the Mg and PTFE. The leftover mixed acetone and n-hexane are decanted and recycled or sent out as hazardous waste. Invariably there are releases of the VOC into the atmosphere for this process. The finished compositions are formed into pellets either through consolidation on hydraulic presses or ram extrusion through dies. After drying at elevated temperatures to drive off any residual volatiles the initiation compositions are applied to the pellets. These compositions contain Mg-PTFE-TPE and use acetone to dissolve the TPE and form a slurry that is applied to the pellet. Again the pellet is dried at elevated temperatures. Once dried the pellet is wrapped with aluminum tape and then inserted into a case. The cases are packaged and the process is complete.

These processes involve considerable use of VOC and the nature of the mixing vessels subject operating personnel to a new exposure of the VOC each time a mix is made. There is also the issue of VOC release to the atmosphere although significant progress has been made to recycle the VOC or least minimize its evaporation. While VOC usage varies yearly since flare production changes annually the average yearly amount is approximately one million pounds for SMCA (Single Manager for Conventional Ammunition) items (ref. 1). Additional usage arises from the production of items for commercial, foreign and non SMCA (e.g. Special Forces Command) usage.

The EPA (Environmental Protection Agency) and the environmental agencies for the states where most CM (countermeasure) production occurs list hexane as a HAP (hazardous air pollutant). Inhalation exposure to n-hexane has been associated with long term neurological impairments in occupationally exposed individuals with initial severity being significant for several months. Other hazards noted for n-hexane include muscle atrophy. Reproductive effects have been observed in rats and this is a suspected hazard for humans. Acetone is not listed as a HAP by either the EPA or state agencies. The EPA lists it as an exempt VOC. However evidence suggests that the effect of inhalation exposure to n-hexane is worsened when acetone is also inhaled simultaneously. The thinking is that acetone enhances the production of 2, 5-hexanedione, the major toxic metabolite produced by intake of n-hexane (ref. 2). As stated these two VOCs are used together in flare manufacture.

The main health hazard associated with acetone is neurological effects although these are temporary (ref. 3).

Both materials are highly flammable with National Fire Protection Agency flammability ratings equal to 3. This rating means that the material can be ignited at almost all ambient temperature conditions.

Previous work to eliminate VOC from the manufacture of CM flares has included twin screw extrusion and use of supercritical fluids. Both of these have limitations that would make their transition into the industrial base difficult if not impossible (ref. 4, ref. 5).

Spray coating in a fluidized bed is a common unit operation in the food and pharmaceutical industries and is relatively simple in function and readily scaled up. However this application of it to the Mg powder and TPE combination has never been attempted. Thus the parameters affecting the coating must be adjusted to provide a uniform coating on the Mg particles at the mass percentage amounts normally used in the flare formulations. The two components of the process that give the coated particles are the fluidization of the Mg powder and the atomization of the TPE stock solution or melt. Each of these presents its own challenges. For the fluidization aspect air flow must be controlled at the correct level to fluidize the Mg powder in a predictable flow pattern that creates a ‘cloud’ of Mg particles that fills the partition chamber and neither drops back down into the powder bed nor be rapidly blown up against the chamber outlet. What potentially complicates this is the wide particle size range of the Mg powder. Spray coating needs to consider the viscosity of the stock solution or polymer melt. Thus nozzle design and placement must be chosen to atomize the droplets to the proper size. For the TPE binder if the droplet is too large it will rise and then fall before the VOC is fully vaporized. If too small the VOC will flash too quickly possibly blocking the atomizing nozzle aperture with binder. And while there is no VOC component with use of the EVA binder melt its high viscosity presents challenges in atomization.

Further both the Mg fluidization and spray coating need to be synchronized. This requires a further optimization of the fluidizing and spray coating parameters. The bulk of uncoated powder and atomized solution or melt need to be in the partition chamber simultaneously to ensure maximum and consistent coating of the Mg powder particles. Ideally, the droplet size is maintained to ensure the droplets are small enough to impinge a single particle only. Yet it must also be large enough that the liquid component is not totally evaporated before reaching the particle. This is a function of the solution flow rate, atomizing air pressure and atomizing cap design. Process airflow is mainly to ensure acceptable fluidization of the particles during coating. The airflow also contributes to the overall drying capacity of the process. Major changes in air flow can impair the process by reducing the speed of particles passing through the coating zone. Airflow changes can also cause agglomeration or rapid VOC evaporation, depending on whether the drying capacity is increased or decreased.

Materials and Methods

Overall Project Plan

To better understand the discussion going forward the below schematic shows the major activities originally planned for the program and indicates their qualitative degree of completion. Achieving the capability to effectively coat the minimal amount of Mg powder for evaluation involved more labor than originally estimated. Consequently the received funding was totally expended before all tasks could be completed. Green represents completed, gray uncompleted and mix partial. Specific items not performed included incorporation of solvent recovery, spraying of EVA melt on Mg particles, morphology characterization of the coated particles, ESD testing and crush strength determination of pellets with flare composition containing the coated particles.

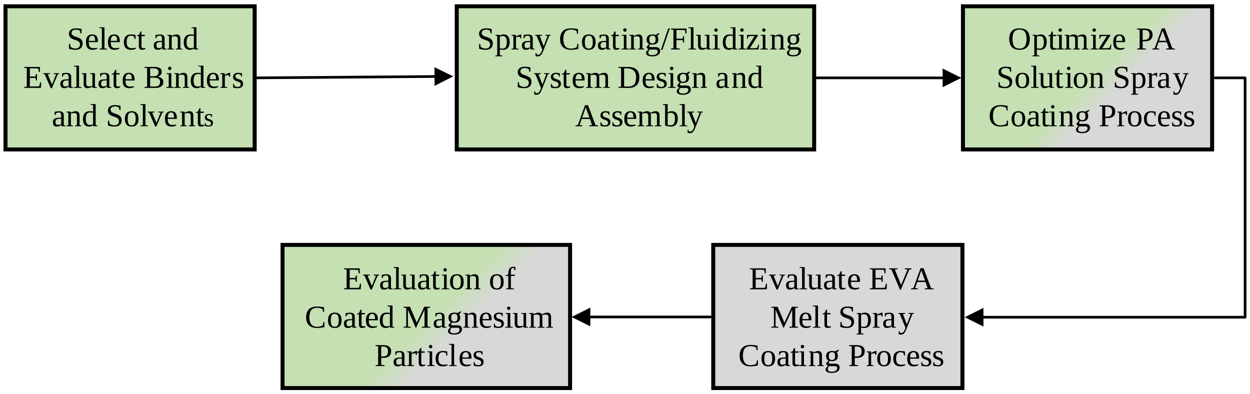

Figure 3. Flow of Major Tasks

Select and Evaluate Binders and Solvent

This was the first major task that FPP (Frontier Performance Polyers Corp.) worked on with assistance from PTD. Since this technology focused on manufacturing methodology and not formulation the binder choices were limited to those in use or shown to perform successfully in flare formulations. Respectively these were PA (Polyacrylate) and EVA based. The former is the binder type currently used for many types of Mg/PTFE/Binder formulations that are pressed into pellets. While EVA is not used in any production items previous work showed that it gave acceptable performance in Mg/PTFE/Binder flares. Solvents for the PA binder would be those able to dissolve the binders. The major supplier of PA binders suitable for the flare formulation are made by Zeon Chemicals Inc. Recommended PA binders manufactured by them considered to be suitable for this effort are shown below with relevant property data (from Zeon Chemicals website) with specific mention of "4451CG, 4051CG and PV04 are recommended for sealant and binder application". The properties listed in Table 1 are relevant to the processing of CM flare mix. Mooney Viscosity and Specific Gravity are considerations for ease of dissolving the binder material into a liquid. Gehman and Volume Swell are measures of the binder's mechanical properties over a temperature range.

PV04 wasn't considered because of its relatively high swell volume. Rather AR71L was evaluated as a possibility because of properties similar to 4051CG and 4451CG. Further work was done with 4051CG, 4451CG and AR71L.

Product	Volume Swell*, %	Mooney Viscosity	Gehman T100 ** ºC	Specific Gravity
HyTemp® 4051	11	46 to 58	-18	1.1
HyTemp® 4051EP	11	35 to 47	-18	1.1
HyTemp® 4051CG	11	25 to 37	-18	1.1
HyTemp® 4451CG	11	25 to 37	-18	1.1
HyTemp® AR71L	11	29 to 41	-18	1.1
HyTemp® PV04	45	25 to 40	-30	1.1
*IRM 903 oil for 70 h (hours) at 150ºC. ** On a nominal 65 Shore A, non-plasticized compound				

Table 1. Properties of PA Binders

PAs are soluble in solvents with a wide range of organic functional groups. However the recommended VOCs are acetone, MEK (methyl ethyl ketone) and toluene. The latter has a much higher vapor pressure than the other two, an unattractive feature for efficient spray coating, and overall possesses more health and environmental risks. Consequently only MEK and acetone were considered.

The final concern was the concentration of binder in solvent. On one hand a high concentration minimizes the amount of solvent that needs to be flashed and thus lessens the burden on the solvent recycle system and the chances of unintentional release to the atmosphere. On the other hand it increases the viscosity such that it would be difficult to form a fine uniform spray. Further there is a solubility limit on the binder in the solvent. This limit is around 20 wt% of binder in solution. Various combinations of the above were evaluated for viscosity using a Brookfield viscometer with data shown in the *Results and Discussion* section.

Spray Coating/Fluidizing System Design and Assembly

Figure 4 gives the overall configuration that was initially selected for producing coated powder. Some components were changed as solution spraying and powder fluidization were attempted individually and then together. These modifications became necessary to produce success in the overall operation. Their rationales are discussed in detail in the *Results and Discussion* section. The configuration that was used to produce the coated Mg powder for evaluation is shown in Figure 5.

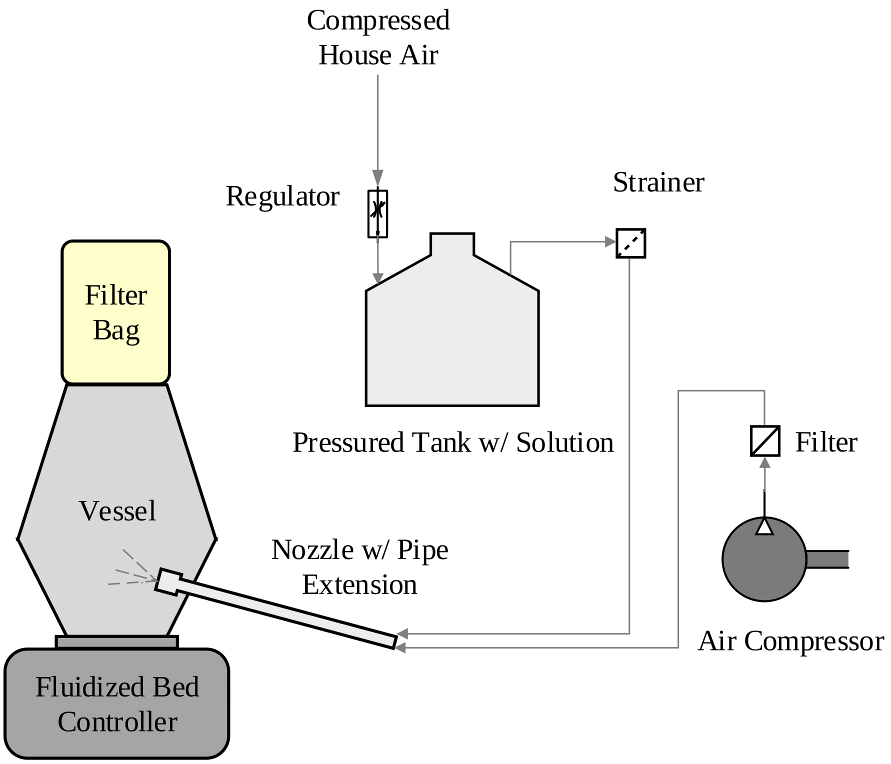

Figure 4. Initial Configuration for Fluidized Bed and Spray Coating System

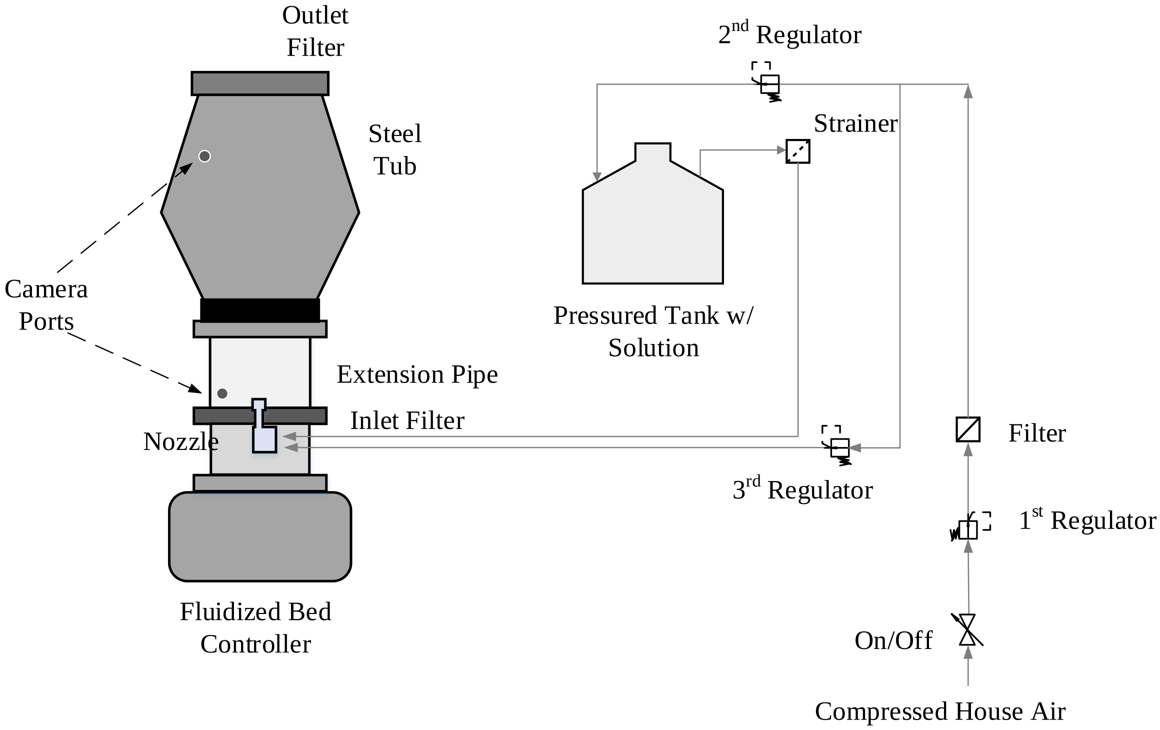

Figure 5. Configuration Used for Fluidizing and Coating Magnesium Powder

Throughout the same fluid bed unit was used. It was manufactured by Sherwood Scientific Ltd.; Model Number M501. Components included stainless steel and glass tubs, terylene outlet filter bag and 25µm mesh stainless steel inlet filter. The glass tub was used to observe initial runs to aid in adjusting process parameters to give effective powder fluidization and spray coating. Tub dimensions and images of the unit follow.

Dimension	Glass	Metal
Bottom diameter	3.95 in (inch)	3.95 in
Middle diameter	5.9 in	9.0 in
Top diameter	5.9 in	5.4 in
Height	12.5 in	10.3 in
Total volume	5L	5L

Table 2. Dimensions for Tubs

Figure 6. Sherwood Fluid Bed with Steel and Glass Tubs

The change in orientation for the spray coating setup was done after initial research indicated that for this design the atomizing spray and Mg powder would interrupt each other causing unstable and non-uniform coating. Adding the extension pipe gave a larger volume for both the fluidized Mg powder and spray to spread out which resulted in more efficient coating of the particles.

Figures 7 and 8 show the details of the set up for atomizing and spraying the PA solutions. All components were manufactured by Spraying Systems Co. Nominal pipe thread was ¼ in, and material of construction was stainless steel. The nozzle's model number was ¼ JAC-SS. The first caps evaluated had orifice diameters of 0.035 in and 0.120 in for the liquid and air respectively (model numbers PF35100-SS and PA120-SS).   However when tested by FPP the coating zone for these was longer than the vendor data showed (7" to 10"). A switch was made to 0.016 in and 0.064 in (model numbers PF1650-SS and PA64-SS) initially based upon vendor data and validation through testing. FPP tested the caps as follows. They set up the spraying hardware under a fume hood next and parallel to a vertical board that had white paper attached to it. PA binder in three different solutions with blue dye added was atomized at various pressures and the spread pattern on the paper analyzed. Adding the camera ports greatly reduced the time to determine the optimum operating conditions within the metal tub.

Figure 7. Fluid and Air Caps

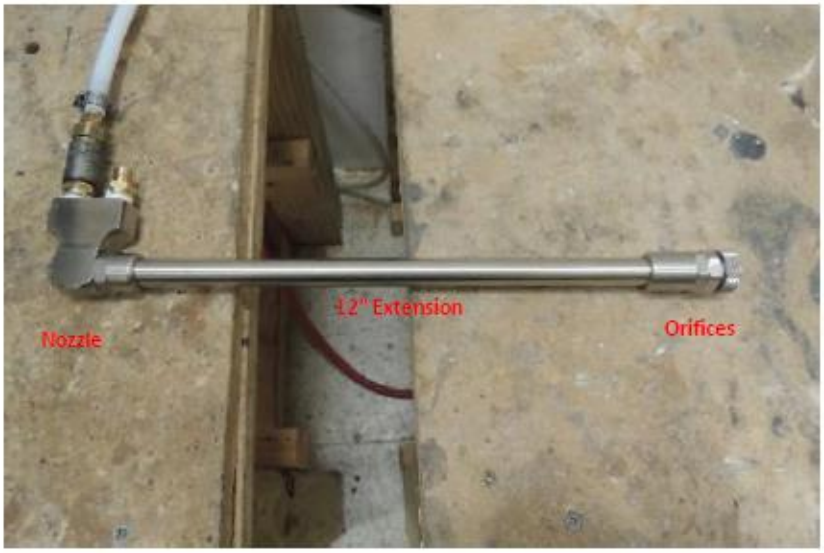

Figure 8. Spray Coating Hardware

Modifying the air supply lines yielded more precise pressure control. This was necessary to obtain a stable spray pattern as small changes in pressures greatly affected the pattern. The added regulators were Bellofram precision air pressure regulators.

Both inlet and outlet filters were changed numerous times to prevent outflow of powder through the top of the tub, to optimize the flow of the fluidizing air and to prevent buildup of powder by the inlet. The final configuration contained a Wurster insert. This configuration (note Figure 1) is routinely used in fluid bed coating processes. The inlet screen was a sandwich of two different meshes as shown below. Early on they were different two mesh sizes but for producing the coated powder both were 10 μm. The opening for the nozzle was 1.75 in. The mesh size of the outlet filter was also changed a number of times but was 30 μm for the final configuration.

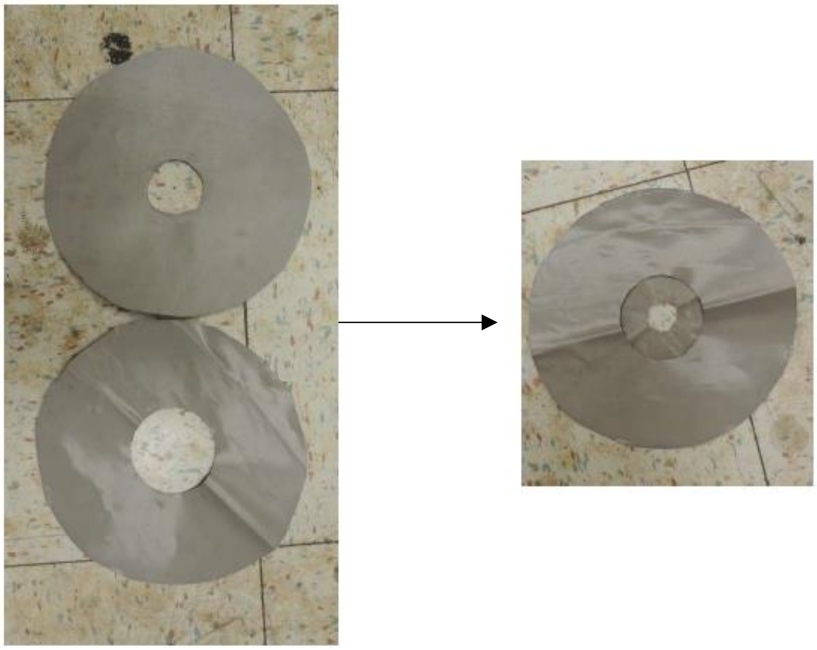

Figure 9. Sandwich Inlet Screen

As fluidization runs were performed the wide particle size range of the Mg powder hindered finding stable fluidization parameters. Thus the particle size range was narrowed. This was accomplished by sieving the powder using No. (Number) 400 and No. 500 sieves. The relevant equipment was a sieve shaker with 8 in sieves. At first the fraction of powder between the two sieves was processed in the fluidized bed unit but it still had enough fines that a considerable amount was passing through the outlet filter. Consequently only material retained on the No. 400 sieve was fluidized. Mg powder was purchased from Hart Metals and conformed to Mil-DTL-14067, Type I, 200/325 mesh.

Evaluation of Coated Mg Particles

Soxhelt extraction was setup for determining the actual amount of binder deposited on the Mg particles. A few runs of the process found that accurate results could be obtained by running one thimble with the coated powder and another as a blank both for 20 h. The thimbles were withdrawn every 6 h to 7 h, vacuum dried, weighed, and returned to the extraction apparatus. Acetone was the extracting solvent.

PTD assessed the efficiency of the coating process by radiometric testing of flare pellets containing the coated Mg powder. The pellet formulations were prepared by blending the coated powder with PTFE powder. The latter's type was DuPont Teflon 7C. Additionally, pellets were made with the current mixing process to serve as a baseline for testing. The following schematic gives the process for both. To eliminate lot to lot variations for Mg powder as an additional variable FPP provided PTD uncoated powder from the fraction retained on the No. 400 sieve.

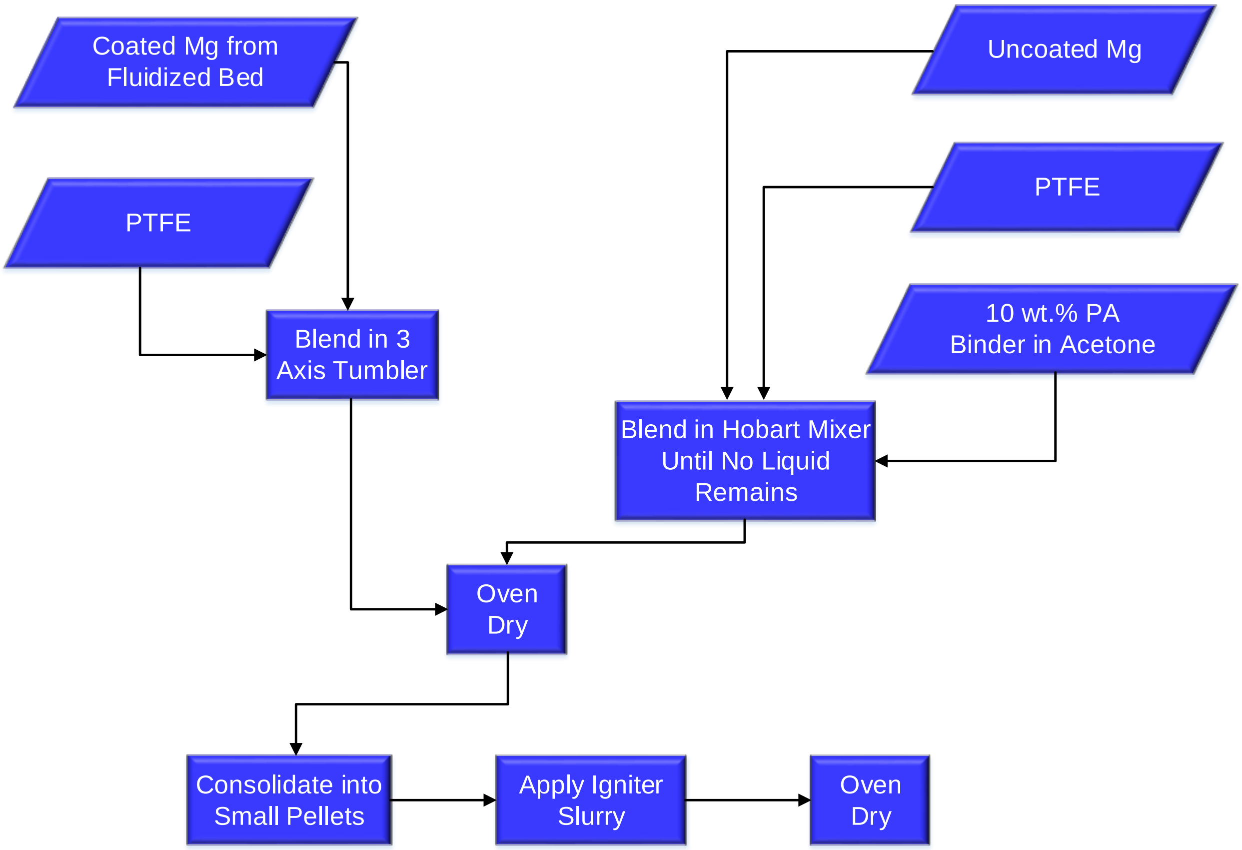

Figure 10. Process Flow for Pellets

Coated mixes were prepared by placing pre weighed portions of coated Mg and PTFE to in a conductive rubber container with the lid taped on. The container was put into a steel over pack container with empty rubber containers and packing materials to secure it during mixing. The over pack container was secured on the tumbler. The tumbler, manufactured by Glen Mills and designated as model Turbula T10B, moves the container in three axes. Operation was for 30 min (minute). At the end increments of the blended materials were weighed to the pellet mass.

Uncoated mix was made per the current practice. A weighed amount of binder was dissolved in acetone overnight to produce a stock solution. The amount of solution required to yield the needed amount of binder by dry weight was weighed out. This portion was then blended with the proper amounts of Mg and PTFE for the formulation. Blending was done with a Hobart mixer using a 5 quart bowl. After blending the composition was removed and dried overnight. After drying increments of the composition were weighed to the pellet mass

Pellets were pressed at 11,000 pounds force on a hydraulic press. Shape was square with lengthwise grooves. Dimensions were nominal 1 in across the longest width and total length 1.30 in to 1.35 in. Pellet weight was 20 g. Pellets were dried overnight after their fabrication. See Figure 11 for pellet configuration (pellet shown is inert; all PTFE). The igniter slurry consisted of Mg powder, PTFE and a stock solution of Viton A dissolved in acetone. It was made the same as the uncoated Mg mixes but prior to use acetone was added to the mix to solubilize the Viton A. The slurry was brushed into the grooves. For both coated and uncoated Mg powders three different compositions were made designated as low Mg, mid Mg and high Mg content. Table 3 has specific data on these. For the coated Mg ten pellets were prepared for each formulation and for the uncoated seven.

Figure 11. Pellet Configuration

	Weight %		
Constituent	*Formula 1*	*Formula 2*	*Formula 3*
Mg	Low	Mid	High
PTFE	Balance	Balance	Balance
HyTemp 4451CG	1.70%	1.86%	2.01%

Table 3. Pellet Formulations

PTD tested the pellets in a flare tunnel per procedures typically used for measuring the infrared output of flares. Pellets were mounted on a stand and ignited remotely by an electric match. Measurements were made of radiant intensity in the 3 μm to 5 μm region and of burn time. Relevant equipment involved a Teledyne Judson series J10D Indium Antimonide detector connected to a DL Instruments preamplifier and CI-Systems blackbody and controller for calibration. An algorithm converted the voltage signal to radiant intensity which was reported as peak intensity and integrated intensity (i.e., area under the intensity-time curve).

Results and Discussion

In this section we will present the results in two different general categories. First, how data measured from the various equipment configurations for the fluidizing and coating of Mg powder were used to design and construct its final configuration. We note that fluidizing of the powder and spraying were initially done as separate operations before being integrated. This sequence was done several times to reach the optimum operating conditions. And the integrated process also went through a number of trials before the final configuration was set. Second, how data taken during the tunnel testing of the pellets containing the coated powder assessed the material for flare performance.

PA Solution Viscosity

As stated in the previous section knowledge of the viscosities of the PA/VOC solutions was vital to select parameters for the solution that would ease its atomization through the spray nozzle assembly. The table below gives the resulting viscosities.

		In Acetone	**In MEK**
Resin	**Wt% Resin**	**Viscosity centipoise**	
HyTemp® 4051CG	5	N/A (not applicable)	N/A
	10	46	56
	15	232	277
	20	823	1030
HyTemp® 4451CG	5	N/A	N/A
	10	51	61
	15	252	346
	20	1071	1128
HyTemp® AR71L	5	N/A	N/A
	10	83	121
	15	708	753
	20	2987	3689

Table 4. Viscosities of PA Binder/Solvent Combinations

Viscosities for the 5 wt% solutions are not reported because the values were outside the spindle's range. The solutions were prepared by combining cut pieces of the binders with the solvents, each appropriately weighed, in glass jars which were then sealed. Dissolution was aided by shaking the jars for 10 s (second) every two hours. After remaining overnight at room temperature the binders were dissolved. The photographs below show the resulting solutions.

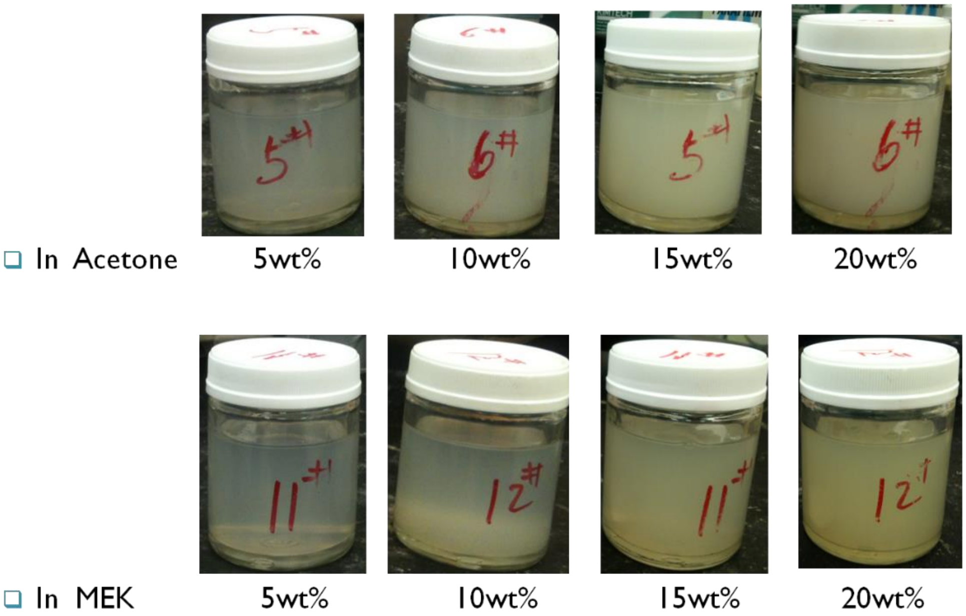

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Figure 12. Appearance of PA Solution

As expected opacity is proportional to viscosity and from some images the binder is not fully dissolved but rather plasticized. Going forward AR71L was not considered because of its significantly higher viscosity and the targeted solution concentration for the HyTemp materials was to be 10 wt% to 15 wt%.

Fluidization and Spray Coating of Mg Powder

The beginning task here was determining the spray pattern. This was accomplished by spraying PA solutions colored with dye against white paper. This was done in two steps. The first was to give a rough idea of the pattern obtained for the various parameters in Table 5. Figure 13 is a descriptive photograph of results from the third trial.

Solution	Air/Liquid Pressure, Psi	Observations
Acetone	30/3.5	Acetone evaporated at 6 in to 7 in from nozzle assembly
2.5 wt% HyTemp® 4451CG in acetone	15/4	Binder was carried another 3 in past where acetone evaporated
1.75 wt% HyTemp® 4451CG in 80 wt% acetone/20 wt% MEK	20/3.5	Acetone/MEK mixture carried binder from atomized state to transition region

Table 5. Initial Spray Pattern Tests Runs

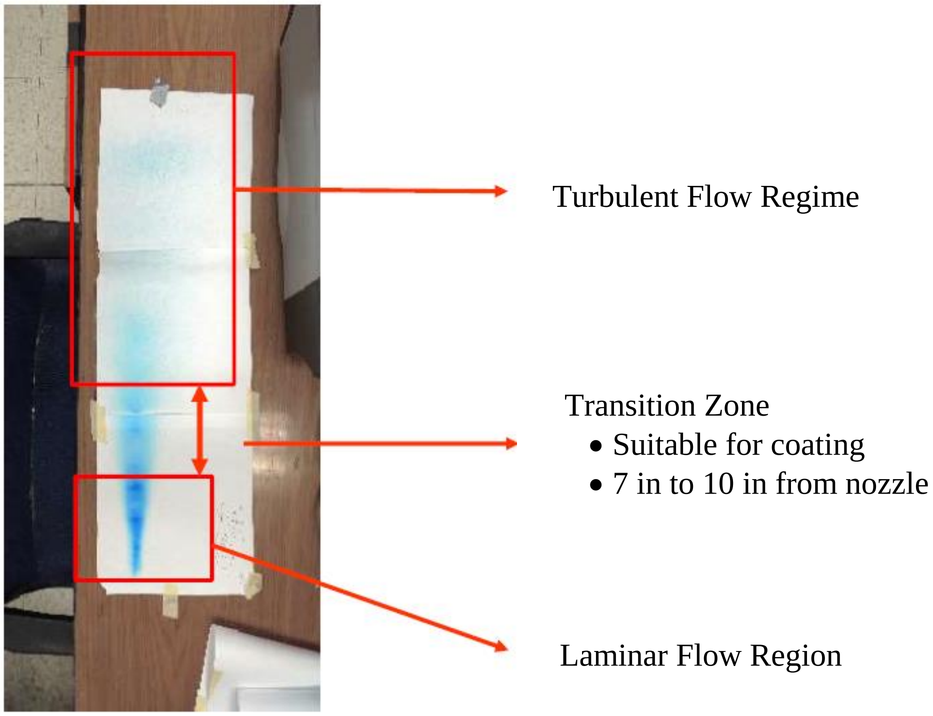

Figure 13. Spray Profile Analysis

Below are the results of all the spray pattern determinations with the 0.016 in and 0.064 in liquid and air orifice diameters.

Solution	Air/Liquid Pressure, psi (pound force per square inch)	Spray Width at Different Spray Distances				
		3 in	6 in	9 in	12 in	15 in
Acetone	10/6	0.5 in	1.0 in	1.5 in	2.0 in	2.5 in
1.75 wt% solution with 80/20 acetone/MEK	30/5	0.5 in	1.0 in	2.5 in	3.5 in	4.5 in
2.5 wt% acetone solution	10/3	0.5 in	1.0 in	2.0 in	2.5 in	4.0 in
	15/5	0.5 in	1.5 in	2.5 in	3.5 in	4.5 in
5 wt% acetone solution	10/6.5	0.5 in	1.5 in	2.0 in	3.0 in	4.5 in
	20/6.5	0.5 in	1.0 in	1.5 in	3.0 in	3.25 in
5 wt% MEK solution	10/10	0.5 in	1.75 in	3.0 in	3.0 in	4.5 in

Table 6. Spray Width at Various Distances from Spray Cap

Having established the spray pattern dimensions the next task was fluidizing the Mg powder. The specification for the powder, Mil-DTL-14067, allows 60% of the powder to pass through a No. 325 sieve. For the drum of powder purchased for this project instrumental particle size analysis showed 100% less than approximately 105 μm, 50% less than approximately 25 μm and 10% less than approximately 12 μm. Consequently this wide range of powder size would present a challenge for finding the optimum conditions for fluidization as described in the discussion that follows. These initial fluidizing runs used the glass tub to enable observation of the powder while being fluidized.

Four initial trials used 50 mg (milligram) to 100 mg of powder and slowly increased the volumetric flow rate to the level were the powder was fluidized and moving through the tub. Problems noted included:

- Leakage of powder through joints and a loosely fitting filter bag.
- Accumulation of powder on sides.
- Inability to sustain stable fluidization. While able to identify a flow rate that fluidized the powder the intensity of the fluidization essentially ceased after a short time.

Sealing the joints and better securing the filter bag fixed the leakages issues. An outlet filter of 10 μm mesh was installed to reduce the leakage for the second and third runs but overly restricted air flow through the tub. A tighter fit for the filter bag was sufficient to reduce loss of powder. At this point the extension pipe shown in Figure 5 was added. Made of steel it was 12 in long and flanged to mate with the other pieces. It gave extra volume for fluidization to better develop and stabilize. Accumulation of powder was mitigated by use of the Wurster insert with the sandwich inlet screens. Details for the screens were 1.5 in diameter center hole for the nozzle assembly and mesh sizes of 5 μm and 10 μm. Further for the last three trials air was passed through the air cap at 15 psi adding to the total air flow through the unit. While these changes aided in minimizing leakage and accumulation of powder they did not assist preventing the drop in fluidization. Observation of the dispersion of the powder indicated that this was likely a function of the powder's wide particle size range

Following the above, the first attempts at coating the Mg powder were made. The same apparatus setup was used except that a 20 μm outlet mesh was inserted to enable air flow. Three runs were done involving 70 mg of Mg powder. The solution was 5 wt% HyTemp® CG4451 in acetone and air and liquid pressures were 10 psi and 6 psi respectively. Mg was fluidized at approximately 30 cubic feet per min to 35 cubic feet per min and then the solution sprayed. Run times were 5 min. In summary there was little or no evaporation of the acetone resulting in wetting of the tub and outlet filter surfaces and deposition of a paste of solution and powder on the inlet mesh and nozzle assembly.

Effort now returned to fluidization of the powder alone. Since particle size range was identified as a factor blocking steady fluidization sieving was performed to reduce it. The process used 8 in No. 400 (38 mm [millimeter]) and No. 500 (25 mm) sieves with a bottom pan. These were mounted on a Gilson sieve shaker and shaking done for five min for both 100 g and 200 g amounts. Considering both amounts together 60 wt% to 65 wt% of the powder was retained on the No. 400 sieve with the balance on the No. 500. Negligible amounts passed through the No. 500 sieve. This distribution didn't match that reported by instrumental analysis. This was expected since sieving of powder gives a low separation efficiency when particles are finer than 38 μm (No. 400 sieve)

The fraction between the two sieves was fluidized in both the glass and steel tubs, one run each. Operating conditions were 35 g of powder loaded, air cap pressure of 10 psi, 15 μm for the inlet mesh and 25 μm for the outlet mesh. These parameters were changed from previous runs to avoid deposition of a paste of solution and powder on the inlet mesh and spraying system assembly and to increase airflow. In the glass tub most of the material stuck on the outlet filter and for the metal tub went through the filter. FPP attributed this to the air pressure being too high and/or the mesh sizes being too large. To mitigate this the setup of the sandwich inlet filter was varied. The thinking was that high porosity toward the circumference and low porosity toward the nozzle assembly would prevent powder buildup around the nozzle and aid in fluidization. Also, the outlet filter was sealed tighter against the top edge of the glass tub. Air pressure of 10 psi and outlet filter of 25 μm were retained for the tests involving the changes in the inlet filter. Results of these tests, all performed using the glass tub, follow.

Sandwich Filter *Bottom Mesh Opening (μm)/Top Mesh Opening (μm)/Diameter of Inside Cutout in Top Mesh (in)*	**Observations** *Numbers without units refer to blower settings*
15/15/1.75	Minimum for fluidization was 5. Able to fluidize within 6 to 10 but not sustained due to powder loss. Over 14 totally blew powder out through outlet screen.
10/15/1.75	No fluidization at 10 and below. Consistent fluidization observed at 15 for 6 min to 8 min then disappeared. Above 15 powder loss through outlet screen.
10/10/1.75	Test #1: No fluidization 10 and below. Stable fluidization at 14 for 3 min to 15 min then ceased. At 18 and above powder is blown through outlet screen. Test #2: Within 2 to 13 gradual fluidization noted. Observable fluidization at 14 and powder loss through outlet screen above 14.
10/10/1.5	Gradual fluidization at settings between 2 and 15 with obvious achieved at 16 and then ceasing. Above 16 powder lost through outlet screen.
10/10/2	Observable fluidization began at 6. But was unstable until 12. Above 12 powder lost through outlet screen.

Table 7. Powder Fluidization Results with Sandwich Inlet Filter

From the above data the 10/10/1.75 design gave the best chance of stable fluidization. There remained the problem of powder exiting. At this point better control of air pressure both for the air and liquid caps was installed to facilitate the fluidization and coating of the particles based upon preceding experiences.

Attention now shifted back to the sieving of the Mg powder. The objectives were finding the minimum time needed to achieve the maximum amount of separation between the sieves and the maximum amount of powder that could be sieved. Twenty five min was identified and the weight was limited to 100 g. Both the fractions retained on the No. 400 sieve and between the No. 400 and No. 500 sieves were set aside for the testing described in the next two paragraphs. Approximately 19% of the powder was retained on the No. 400 sieve, 79% on the No. 500 and the remaining 2% through the No. 500.

This stage of fluidizing powder attempted fluidization of both size fractions. Component details were sandwich inlet filter sizing 10 μm/10 μm/1.75 in, 20 μm mesh outlet filter and pressure through the air cap was kept below 5 psi. The glass tub was used. Since the powder's particle size distribution was narrowed by sieving the extension pipe was removed. Four runs were done with results described below. In each case fluidization went on for 15 min to 20 min.

	Powder retained on No. 400 sieve		**Powder through No. 400 sieve, retained on No. 500 sieve**	
	Trial		*Trial*	
Parameter	*#1*	*#2*	*#1*	*#2*
Starting weight of powder	40 g	30 g	30 g	40 g
Blower setting for start of fluidization	10	12	14	15
Fluidization description	Stable and 3 in above the orifice	Stable	Powder cloud noted and then disappeared. Not stable fluidization ~5 in above the orifice	Stable
Ending weight of powder	About 40 g after 15 min	29 g after 20 min	17.4 g after 15 min	22.1 g after 15 min

Table 8. Fluidization Trials with Sandwich Filter; Second Set of Tests

Evidently powder retained on the No. 500 sieve still contained powder finer than the mesh opening (25 mm) since it went through the outlet filter evidenced by powder on top of the filter. For the coarser fraction of powder stable fluidization was achieved indicating that lower pressure in the air line and a finer mesh outlet filter diminished the loss of powder.

Another set of experiments spraying the PA solution on the Mg powder was conducted again with the glass tub. Five runs were performed with the sandwich inlet filter sizing 10 μm/10 μm/1.75 in, 20 μm mesh outlet filter and pressures through the air and liquid caps at 4.5 psi and 1.5 psi to 2.0 psi respectively. The essential process for all of these runs was.

- Fluidize powder to a minimal level → Introduce air into the spray coating hardware → Increase fluidization air to achieve stable fluidization → Introduce liquid or solution into the spray coating hardware → Continue until liquid or solution is used up → Keep air flowing for five min afterwards

The finer fraction of Mg powder was used in the first run. This run was not successful. Liquid component was blue dyed isopropyl alcohol with no dissolved TPE. Flooding from the solvent occurred within 10 s of it being introduced into the apparatus. Of the 30 g of powder initially loaded only 13 g was recovered. The lost powder went out through the outlet filter. To prevent this loss the remaining runs fluidized the coarser fraction of powder. Further these subsequent runs cycled the spraying of solvent or solution. Liquid would be atomized for 5 s and then spraying ceased until powder fluidization recovered to the level prior to spraying. This strategy enabled both successful fluidization and coating of the powder particles. Details of these runs follow. Like the first run blue dye was added to the liquid.

	Value			
	Trial			
Parameter	*#1*	*#2*	*#3*	*#4*
Starting weight of powder	35 g	35 g	35 g	35 g
Solution	100% isopropyl alcohol	100% acetone	400 mL (milliliter) 0.8 wt% HyTemp® 4451CG in acetone	400 mL 1.0 wt% HyTemp® 4451CG in acetone
Blower setting for start of fluidization	12	11	11	11
Number of spraying on-off cycles	10	15	10	10
Ending weight of powder	32 g	33.2 g	Not measured	Not measured

Table 9. Fluidization and Spray Coating Trials with Sandwich Filter for Coarse Powder Fraction

Trials #1 to #2 gave successful fluidization and wetting of the powder and Trial #3 resulted in successful coating. For Trial #4 the slightly higher concentration of resin in the solution resulted in the outlet pores being blocked with resin. Subsequently air flow was blocked with no further fluidization and the powder agglomerating.

To more efficiently obtain powder for future fluidization and spraying operations the powder sieving process was reexamined. Nominal weights of 25, 50 and 100 g were sieved each for 30 min, 35 min and 65 min respectively with sieve and pan fractions weighed every 5 min. Results were similar to those obtained in the prior sieving operation however approximately 60 min of run time was necessary. To increase the amount of material per unit time three nests of one No. 400 sieve and a bottom collection pan were shaken simultaneously. Seventy five grams of powder was introduced into the top of each pan and all nests shaken for 75 min. This was done numerous times to produced enough powder for the remaining program requirements. Separation efficiency was not as good as prior sieving. Approximately 4 wt% of the powder was retained on the No. 400 sieve. This fraction would be used in all work going forward because of the inability to fully separate material retained on the No. 500 sieve and that passing through it. Previous fluidizing runs demonstrated that significant amounts of material between the 400 and 500 sieves passed through the outlet filter screen implying that it was less than 20 µm.

Mg fluidization in the metal tub, briefly evaluated before, was again examined since the inlet filter design and Mg granulation were now fixed. These runs were done with no extension tube, and for the first two no outlet filter and filter bag. The inlet sandwich filter configuration remained at 10 µm/10 µm/1.75 in. Relevant data are shown as follows.

	Value					
	Trial					
Parameter	*#1*	*#2*	*#3*	*#4*	*#5*	*#6*
Starting weight of powder	27 g	29.7 g	20.3 g	22.7 g	21.6 g	35.1 g
Blower setting to begin fluidization	10	16	14	18	20	18
Filter bag in place?/Outlet filter mesh size	No/No filter	No/No filter	Yes/ 20 µm	Yes/ 20 µm	Yes/20 µm	Yes/20 µm
Air pressure through air cap	No air	4.5 psi	4.5 psi	4.5 psi	4.5 psi	4.5 psi
Fluidization stable?	Yes	No	Yes	Yes	Yes	Yes
Ending weight of powder after 15 min fluidization	21 g	0 g	19.7 g	21.6 g	20.3 g	34.3 g

Table 10. Results of Powder Fluidization in Metal Tub

Addition of the outlet filter enabled stable fluidization of the powder with minimal loss through the top of the apparatus.

Spray coating of the powder was now attempted in the metal tub. Values of operating parameters that resulted in successful fluidization were maintained. For all of the runs in Table 11 the air pressure through the liquid cap was 1.5 psi.

	Value		
	Trial		
Parameter	#1	#2	#3
Starting weight of powder	35.0 g	34.3 g	34.1 g
Blower setting to begin fluidization	18	18	18 to 22
Liquid component	100% acetone	1 wt% HyTemp® 4451CG in acetone	1 wt% HyTemp® 4451CG in acetone
Coating process	Sprayed 450 mL in 1 min	Spray for 10 s every 2 min; 600 mL of solution	Spray for 10 s every 2 min; stopped after 13 cycles
Ending weight of powder	34.3 g	34.1 g	29.8 g
Observations	No agglomeration of powder	No agglomeration of powder	Fluidization and spraying stopped when outlet filter was blocked by buildup of powder coated with resin. Powder on walls also.

Table 11. Fluidization and Spraying Trials in Metal Tub

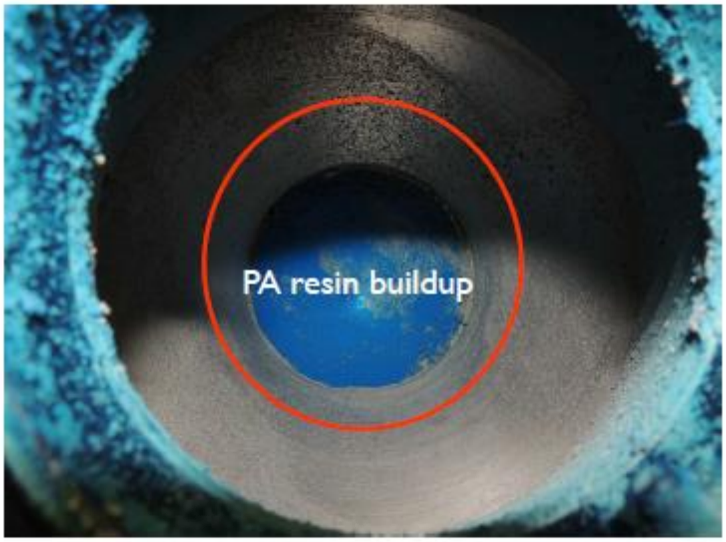

Figure 14. Buildup of Powder with Resin on Outlet Filter

Figure 15. Buildup of Powder with Resin on Inlet Filter Adjacent to Nozzle

To mitigate these problems the extension tube was inserted and the outlet filter was switched to 30 μm mesh size. These modifications would increase the volume and thus residence time for the solution to contact the Mg particles and increase the air velocity through the tub. Apparatus settings remained the same except the on off cycling was spray for five seconds with five minutes no spray intervals. Thus for a 60 min run time there were 12 total sprays. Air was blown for an additional 20 min to drive off residual solvent. This time was verified as being sufficient by placing the coated powder in a vacuum dryer. Weight loss was negligible indicating no remaining solvent. Further the solution was now 0.75 wt% HyTemp® 4451CG in acetone.

	Trial					
Parameter	*#1*	*#2*	*#3*	*#4*	*#5*	*#6*
Starting amount of powder	75 g	-	64.3 g	61.0 g	60.9 g	59.6 g
Blower setting to begin fluidization	22	22	22	24	26	34
Powder on extension tube wall at end	-	28.7 g	6.2 g	3.1 g	2.1 g	1.2 g
Powder on tub wall at end	-		6.8 g	3.0 g	0.7 g	0.3 g
Powder on inlet filter at end	-	35.6 g	48.0 g	54.8 g	56.7 g	57.7 g
Total powder amount	-	64.3 g	61.0 g	60.9 g	59.6 g	59.2 g
Powder lost	-	-	3.3 g	0.1 g	1.3 g	0.4 g
- Means weight was not measured						

Table 12. Fluidization and Spraying Trials in Metal Tub Incorporating Extension Tube and Larger Mesh Outlet Filter

This data demonstrates that coated powder can be produced in the metal tub provided that the extension tube is in place. Reducing the starting weight of powder and increasing the blower setting assisted in lowering the amount of powder on the tub and extension tube walls. Soxhlet extraction yielded a coating amount of 2.39 wt% on a random sample of coated powder from these trials.

Before further work with the fluidized bed system the borescope camera system was installed. It greatly assisted in knowing what was occurring in the metal tub. A run with powder fluidization only was performed to understand its capability. The pictures below depicts what the camera by the nozzle assembly was seeing.

No air flow

Blower setting = 8

Blower setting = 11

Blower setting = 21

Figure 16. Borescope Camera Images of Fluidized Powder in Metal Tub

Using the parameters for fluidization and spraying that gave the results shown in Table 12 further operation of the apparatus sought to optimize the following parameters to coat enough powder for pellet testing

- Starting weight of Mg powder
- Concentration of HyTemp® 4451CG resin in acetone
- Identify maximum amount of resin that could be coated onto the powders with a desired amount of 5 wt% but actual range anticipated 2 wt% to 3 wt%.

The operation was stopped when the camera’s video showed no further fluidization although as above air flow continued for 20 min afterwards to drive off residual solvent. Four trials were conducted. Table 13 gives the operating parameters and end states for these. Observations on each run are in paragraphs that follow.

Parameter	Trial #1	Trial #2	Trial #3	Trial #4
Starting amount of powder	130.0 g	90.9 g	90.1 g	75.0 g
Solution concentration	1.5 wt%	1.5 wt%	1.5 wt%	1.0 wt%
Amount of solution sprayed	286.2 g	114.5 g	206.9 g	99.3 g
Powder remaining after 16 h processing	104.7 g	88.2 g	82.5 g	71.2 g
Powder lost	25.3 g	2.7 g	7.6 g	3.8 g
Nominal coating content*	4.1 wt%	1.9 wt%	3.8 wt%	1.4 wt%
Coating content via Soxhlet extraction	2.00 wt%	Not done	Not done	1.38 wt%
*= Solution concentration x Solution sprayed/Powder remaining				

Table 13. Results of Initial Trials to Produce Coated Mg Powder for Flare Pellets

Trial #1 contained 14 shutdowns to clean the outlet screen and seven to clean around the nozzle assembly and tub wall. The amount of powder loaded at the beginning strained the system's capability to efficiently operate. When loaded it nearly reached the top of orifice on the nozzle assembly and lead to dense packing that interfered with coating effectiveness, increased agglomeration and deposited powder on the tub walls. Reduction of the powder amount improved the situation but agglomeration was still present. For Trial #3 the amount of solution sprayed was increased to ostensibly raise the percentage of coating. Agglomeration occurred again. Consequently both the amount of powder to be coated and the binder content of the solution were reduced. These changes limited agglomeration.

Based upon the above the following configuration details were applied to produce the coated Mg powder for fabrication of pellets. The overall schematic is that shown in Figure 5.

- Inlet filter: Sandwich design with 10 µm mesh over 10 µm mesh with 1.75 in radius inside cutout for top mesh.
- Outlet filter: 30 µm mesh with no filter bag.
- Extension pipe length: 12 in.
- Air pressures for air and liquid caps respectively: 4.5 psi and 1.5 psi
- Mg particle size: Retained on No. 400 sieve ($\geq$ 38 µm).

The operating procedure started with the preparation of the powder by shaking three sieve nests simultaneously and dissolution of binder into solvent through a magnetic stirrer for 8 h to 10 h. After these materials were weighed and loaded into their appropriate spots the process was begun by turning on the fluidized bed blower to achieve fluidization. This was followed by spraying of the solution using the five seconds on/five minutes off cycle. After one hour the system was shut down and the walls and outlet filter were cleaned with a brush. The entire run ended when powder could no longer be fluidized even at the maximum blower setting due to agglomeration and increased particle weights because of the coating. Air was kept blowing through the apparatus to remove excess solvent. Remaining solution was weighed. Powder collected from the inlet filter and tub walls was vacuum dried at 30 degrees Centigrade for six h and then weighed. Samples were collected from each batch produced and analyzed per Soxhlet extraction. Six batches were made with details shown below. The amount of coating was limited by the bed's maximum volumetric air flow. Had this been greater more solution could have been sprayed while maintaining stable fluidization.

	Batch 1	**Batch 2**	**Batch 3**	**Batch 4**	**Batch 5**	**Batch 6**
Starting Amount of Powder (g)	75.0	75.4	75.3	65.6	73.9	72.9
Binder concentration (wt%)	1.5	1.0	1.0	1.0	1.0	1.0
Blower setting to begin fluidization	12	13	11	10	10	15
Solution Sprayed (g)	99.3	83.7	75.3	102.4	79.6	76.8
Coated powder recovered (g)	71.2	66.1	73.0	64.0	69.1	69.9
Nominal coating (wt%)	2.09	1.27	1.03	1.60	1.15	1.10
Coating per extraction (wt%)	1.38	1.62	1.87	2.16	2.13	1.89

Table 14. Data on Batches of Coated Mg Produced for Pellets

In total approximately 413 g of coated Mg powder were prepared for PTD to blend with PTFE and then press the resulting compositions into pellets. The next subsection describes the results of the radiometric testing with these pellets.

Radiometric Evaluation of Pellets

Data is shown in the following table and plots. Figures 18 – 20 are arranged to graphically reflect the values in Tables 14 -16. The control charts, Figures 21 – 23, are included to show point to point spread.

Formulation	Number Data Points	Average, s	Standard Deviation, s	Maximum Value, s	Minimum Value, s
Low Coated Mg	10	2.71	0.39	3.32	2.12
Mid Coated Mg	10	2.51	0.05	2.61	2.47
High Coated Mg	10	1.87	0.07	1.97	1.76
Low Uncoated Mg	6	4.92	0.34	5.37	4.61
Mid Uncoated Mg	6	4.07	0.12	4.30	3.96
High Uncoated Mg	7	3.92	0.16	4.25	3.75

Table 15. Burn Times for Flare Pellets

Formulation	Number Data Points	Average, W/sr (watt per steradian)	Standard Deviation, W/sr	Maximum Value, W/sr	Minimum Value, W/sr
Low Coated Mg	10	2,227	516	3,185	1,463
Mid Coated Mg	10	2,511	165	2,805	2,313
High Coated Mg	10	3,775	289	4,396	3,379
Low Uncoated Mg	6	1,228	50	1,278	1,157
Mid Uncoated Mg	6	1,482	126	1,647	1,274
High Uncoated Mg	7	1,706	67	1,766	1,574

Table 16. Peak Radiant Intensities for Flare Pellets

Formulation	Number Data Points	Average, W/sr	Standard Deviation, W/sr	Maximum Value, W/sr	Minimum Value, W/sr
Low Coated Mg	10	2,794	136	3,005	2,567
Mid Coated Mg	10	2,891	131	3,113	2,718
High Coated Mg	10	2,926	174	3,177	2,716
Low Uncoated Mg	6	2,762	73	2,860	2,668
Mid Uncoated Mg	6	2,920	277	3,146	2,399
High Uncoated Mg	7	3,277	96	3,448	3,147

Table 17. Integrated Radiant Intensities for Flare Pellet

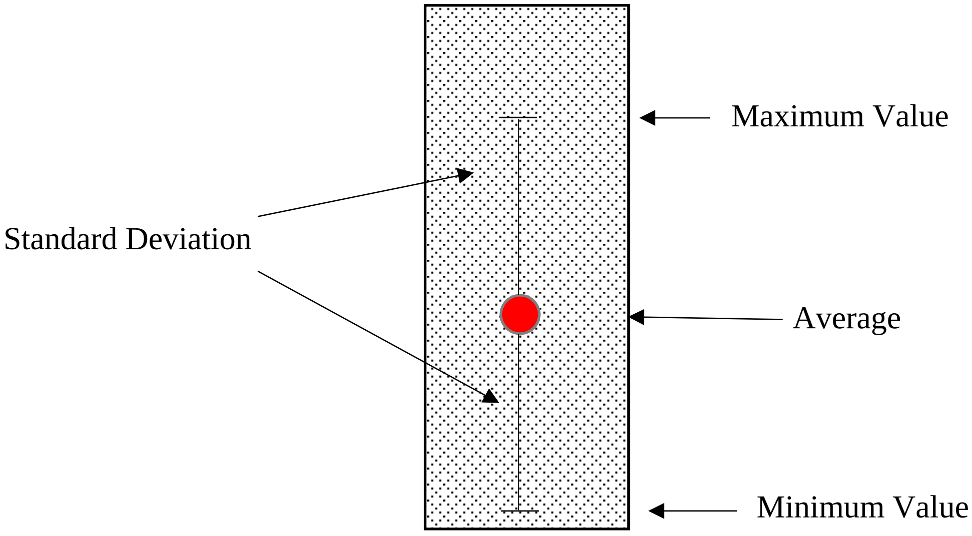

Figure 17. Guide to Comparison Charts Below

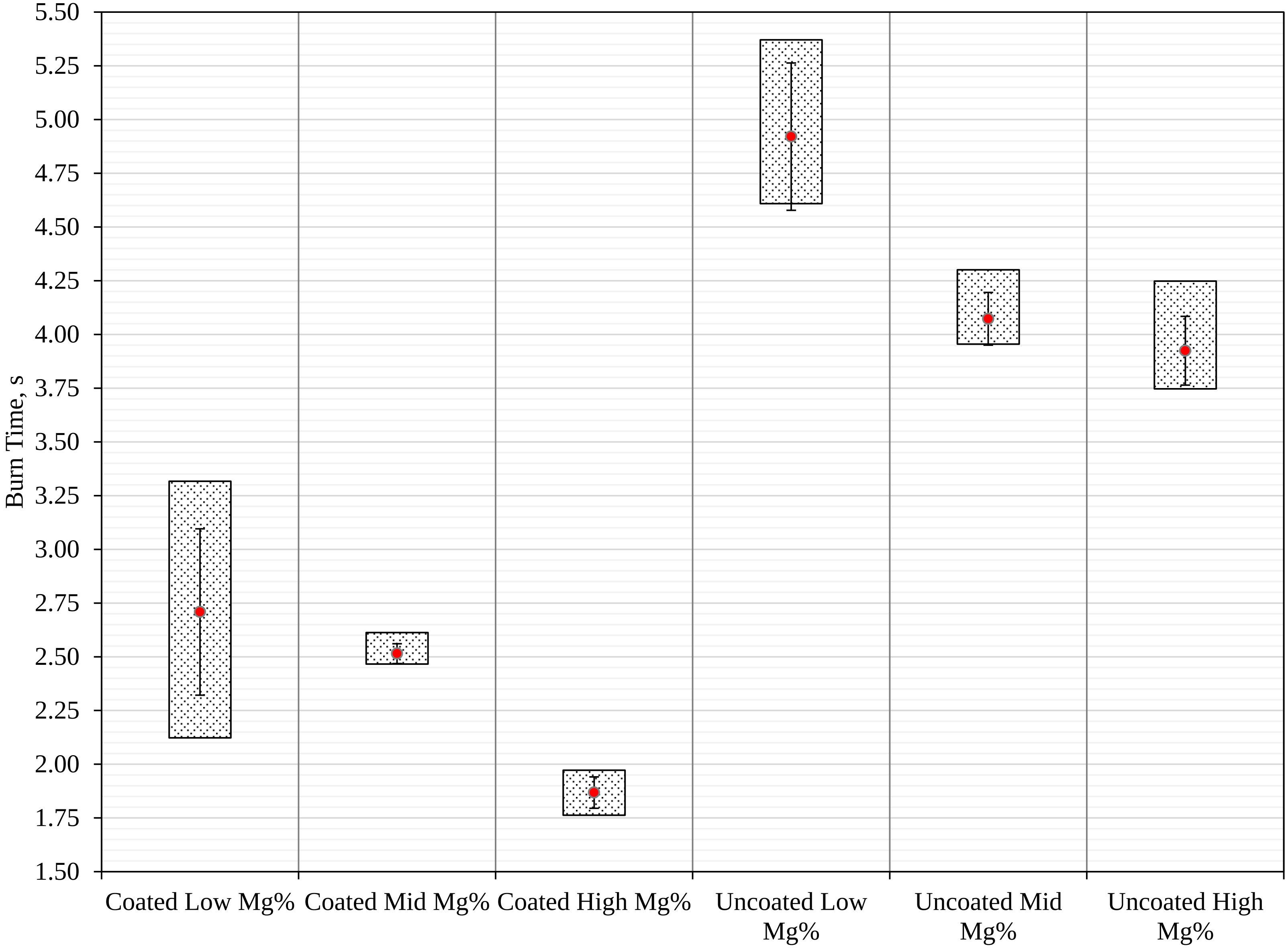

Figure 18. Comparison for Pellet Burn Time

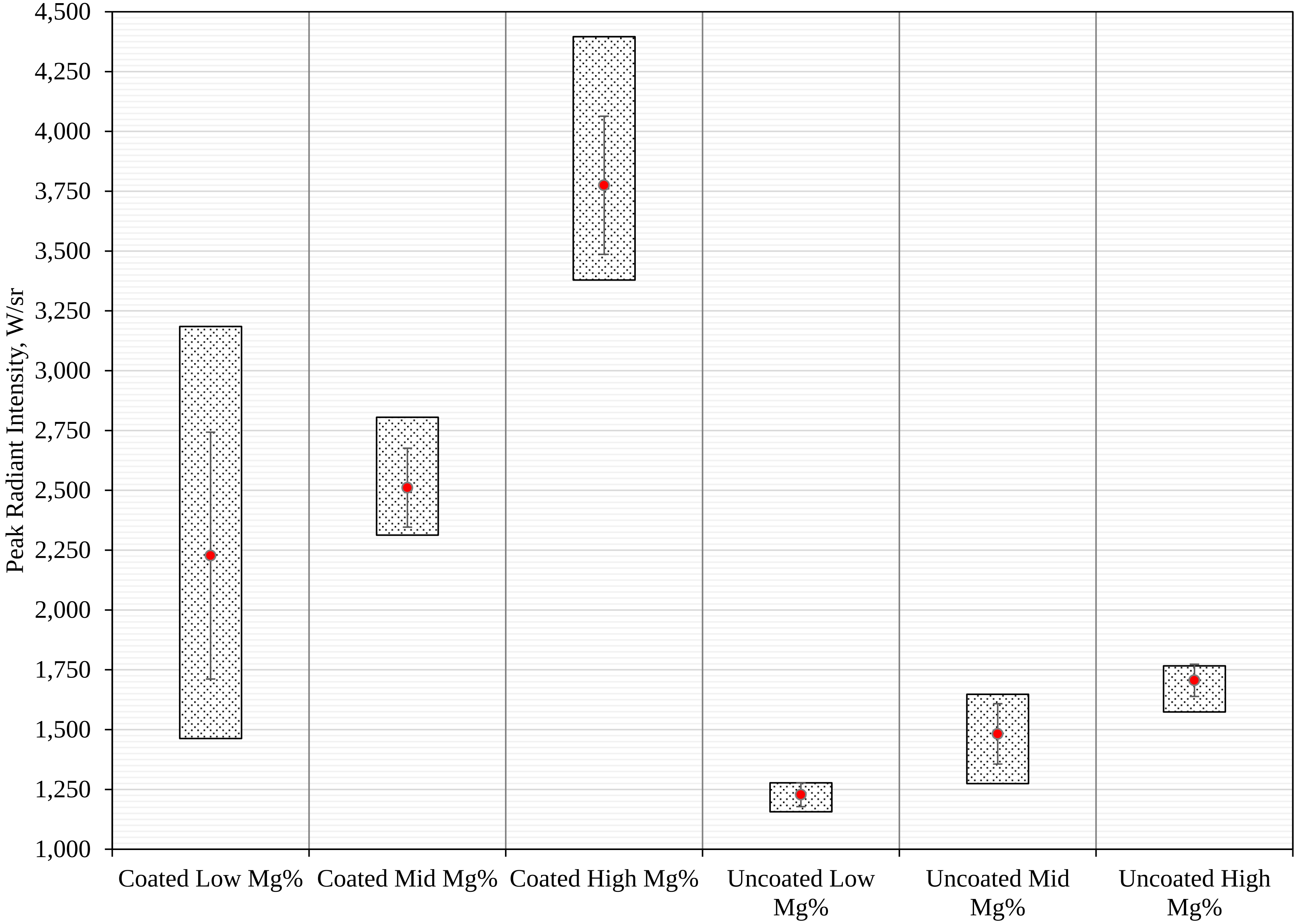

Figure 19. Comparison for Peak Radiant Intensity

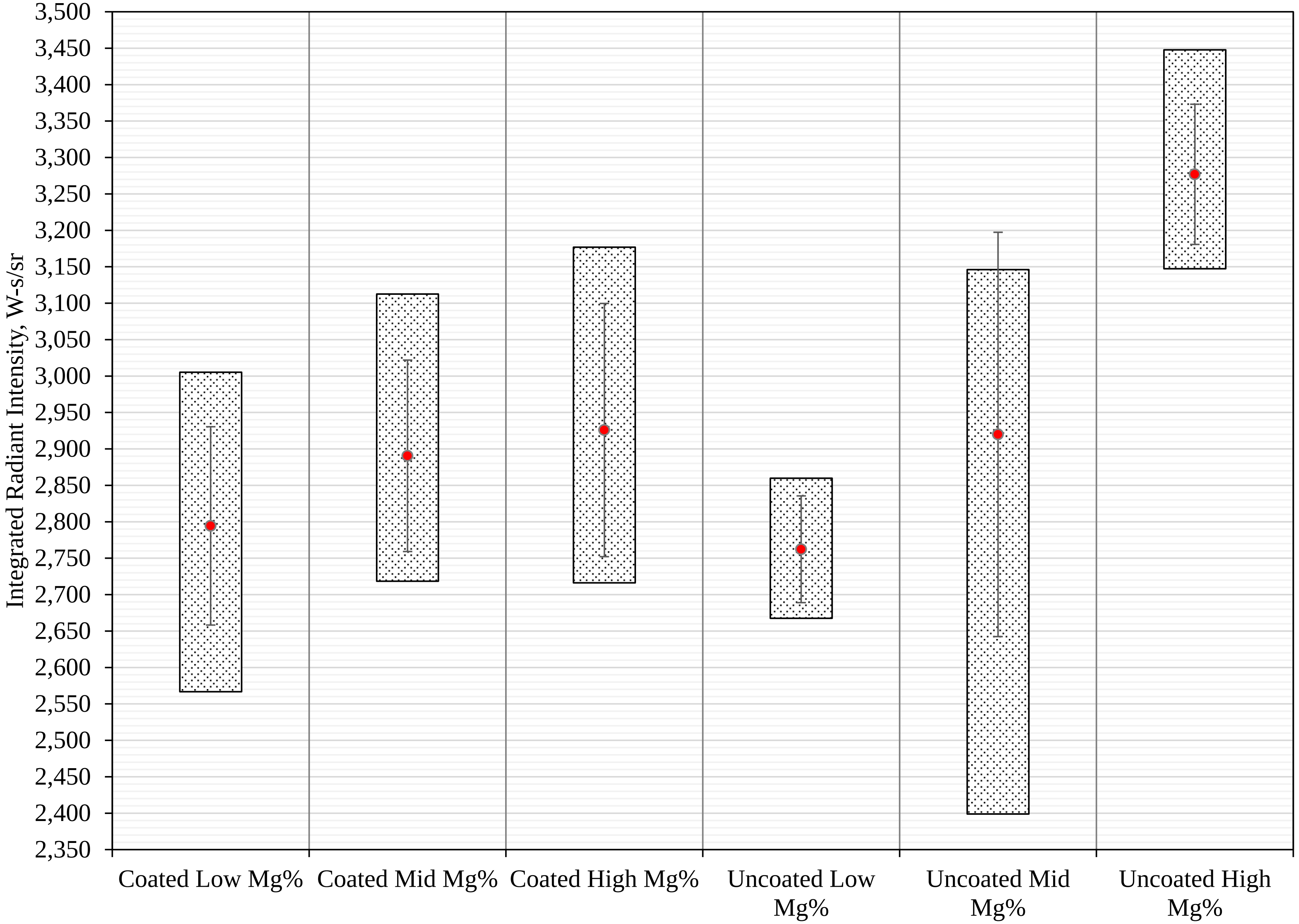

Figure 20. Comparison for Integrated Radiant Intensity

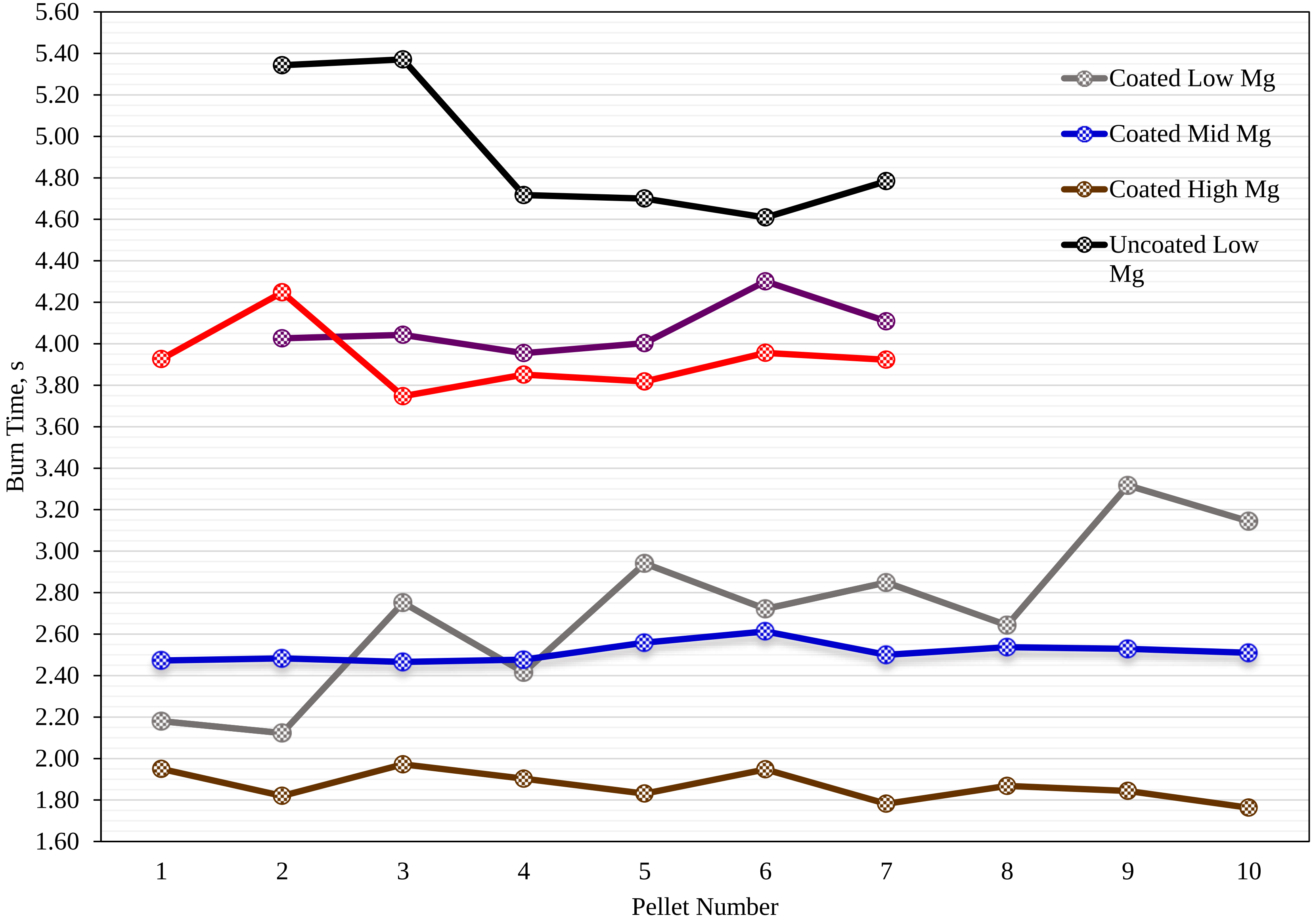

Figure 21. Burn Time Control Chart

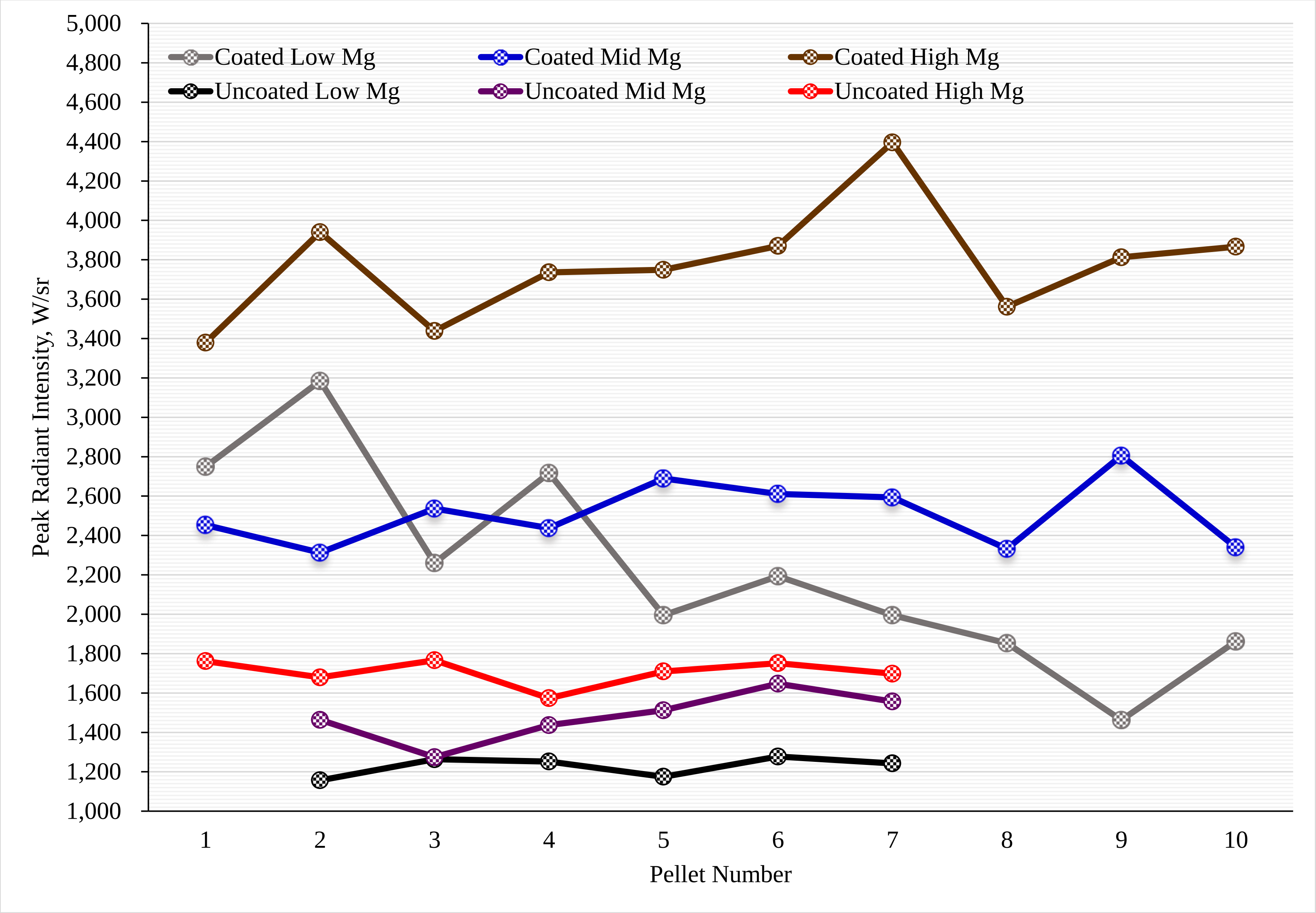

Figure 22. Peak Radiant Intensity Control Chart

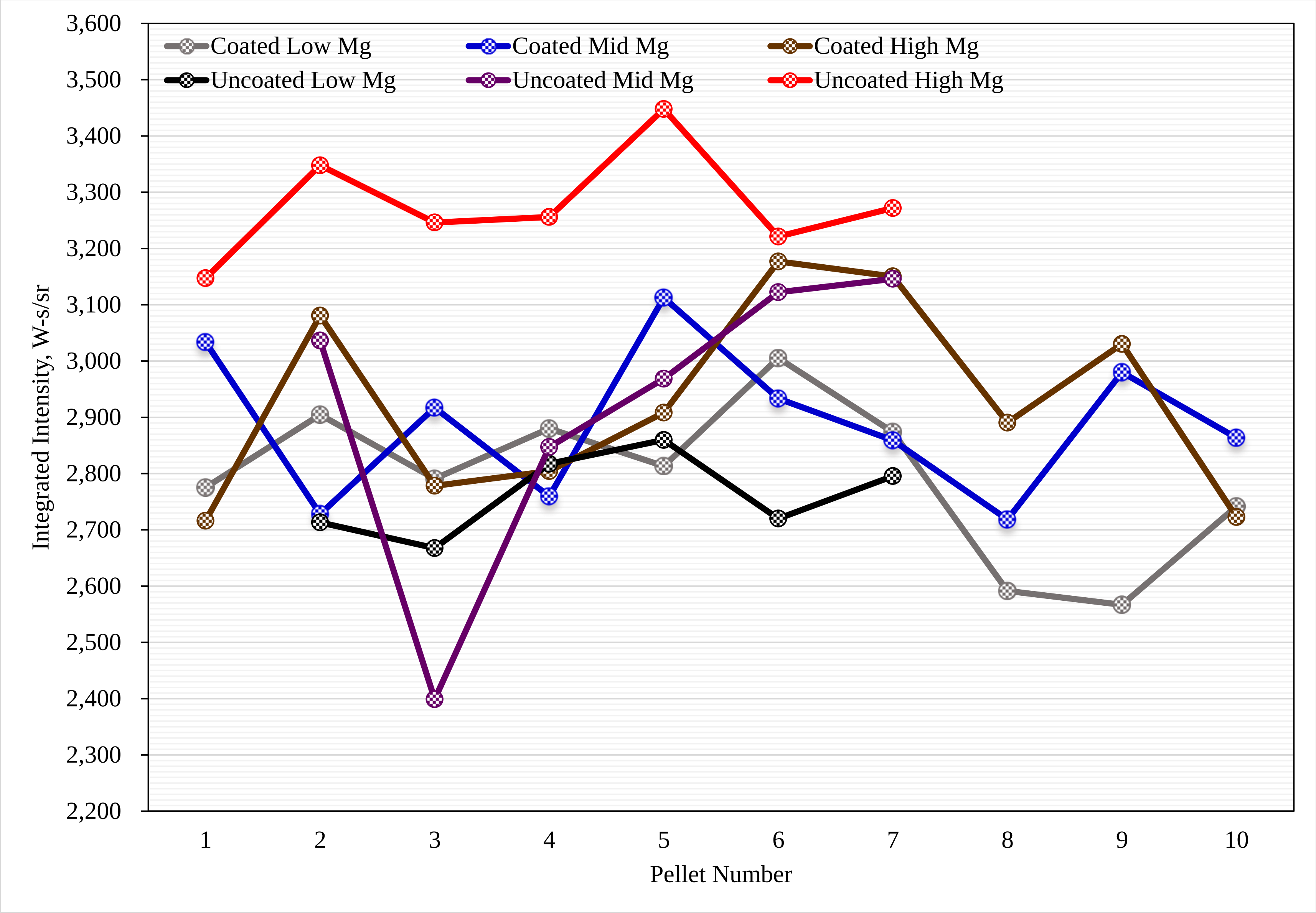

Figure 23. Integrated Radiant Intensity Control Chart

Given the limited amount of pellets tested no quantitative analysis for comparison can be made. Further, the small size of the pellets also had influence upon the spread of data points. Nevertheless qualitative trends can be noted. Burn time for both uncoated and coated formulations increased with decreasing Mg content as would be likely. When comparing coated and uncoated formulations at each Mg content the coated burned approximately twice as fast. Peak radiant intensities for both coated and uncoated formulations the intensities were proportional to the Mg content, also anticipated. And the coated formulations had higher values than the uncoated. This would be an expected outcome since their burn times were faster. The key parameter is integrated radiant intensity. This may be thought as the energy measured for the composition within the 3µm to 5 µm bandwidth. Since each unique formulation has the same constituent amounts the averages for a given formulation should be the same regardless of the coating on the Mg. Indeed this seems to be the case for the low and medium content Mg formulations. However at the high Mg content the uncoated is definitively higher by approximately 12%.

Conclusions and Implications for Future Research

Radiometric testing was the determinant in concluding whether pellets containing Mg coated in the fluidized bed process could yield performance equivalent to those made via the current process. The results obtained showed a rough similarity that would justify further scale up and optimization of the process.

Within the process itself parameters were established that were able to give stable fluidization and spraying of the binder solution on the Mg particles. As discussed achieving this took considerable effort to the extent that there was insufficient funding to complete all of the tasks in the original project plan. Future work, if pursued, would include the following modifications and goals.

- Mg powder requires a narrower particle size range then allowed by Mil-DTL-14067. The vendor can efficiently provide this as they routinely sell powder to many customers who request tailored granulation and particle size ranges. Doing it internally is a waste of labor.
- Inclusion of solvent recovery. This would enable the system to minimize VOC release to the atmosphere and meet the SON’s objectives.
- Increase the amount of coating on the Mg powder. Binder level in the Mg-PTFE-TPE composition is typically 5 wt% to 10 wt%. If for example the formulation of these constituents is 55 wt%/45 wt%/5 wt% then the percentage of binder on the Mg alone would need to be approximately 11 wt%. Sufficient air flow through the apparatus would be necessary to fluidize the increased weight of the particles due to more binder.
- Perform physical characterization of the coated Mg powder to include particle size analysis and SEM imaging. SEM imaging would also be done on the Mg-PTFE-TPE composition. These analyses would assist in determining the extent and consistency of coating on the Mg particles.
- Validate that Soxhlet extraction is the best method for analyzing the amount of coating on the powder.

- Determine ESD sensitivities on (Coated) Mg-PTFE-binder composition
- Attempt to coat both Mg and PTFE simultaneously. This is a non-trivial objective as there are differences in particle densities, shapes and other factors. Prior to doing this the SEM analysis on flare composition made the standard way would assist in knowing if the coating adheres equally to both the Mg and PTFE. The current assumption is that it does. If the majority is on the Mg then there would be no advantage in trying to also coat the PTFE.
- Attempt to spray the EVA melt and coat the fluidized Mg with it. Success of this would be dependent upon the ability to spray a very viscous liquid and enable its adherence to Mg particles while still in a liquid state.

Literature Cited

(Ref. 1). Single Manager for Conventional Ammunition Industrial Base Assessment Tool; https://prod.jmc.army.mil/IBAT/splash.asp, US Army Joint Munitions Command, accessed March 2019.

(Ref. 2). Toxicological Profile For n-Hexane, US Department Of Health And Human Services, Washington D.C., July 1999.

(Ref. 3). Toxicological Profile for Acetone, US Department Of Health And Human Services, Washington D.C., May 1994.

(Ref. 4). Campbell, Carol, “Twin Screw Extruder Production of MTTP Decoy Flares”, SERDP WP-1240, Thiokol Propulsion, Brigham City, UT, December 2005.

(Ref. 5). DiNoia, T. P. McHugh, M. A. Cocchiaro, J. E, “Solubility And Phase Behavior Of Pep Binders In Supercritical Carbon Dioxide”, The John Hopkins University, Columbia, MD, November 1996.

Appendix

Formulation	Pellet Number	Burn Time, s	Peak Radiant Intensity, W/sr	Integrated Intensity, W-s/sr
Coated Low Mg	1	2.18	2,749	2,775
Coated Low Mg	2	2.12	3,185	2,905
Coated Low Mg	3	2.75	2,260	2,791
Coated Low Mg	4	2.42	2,717	2,880
Coated Low Mg	5	2.94	1,995	2,813
Coated Low Mg	6	2.72	2,193	3,005
Coated Low Mg	7	2.85	1,995	2,874
Coated Low Mg	8	2.64	1,853	2,591
Coated Low Mg	9	3.32	1,463	2,567
Coated Low Mg	10	3.14	1,862	2,742
Coated Mid Mg	1	2.47	2,453	3,034
Coated Mid Mg	2	2.48	2,313	2,728
Coated Mid Mg	3	2.47	2,537	2,917
Coated Mid Mg	4	2.48	2,437	2,760
Coated Mid Mg	5	2.56	2,689	3,113
Coated Mid Mg	6	2.61	2,611	2,933
Coated Mid Mg	7	2.50	2,593	2,859
Coated Mid Mg	8	2.54	2,332	2,718
Coated Mid Mg	9	2.53	2,805	2,980
Coated Mid Mg	10	2.51	2,340	2,864
Coated Hi Mg	1	1.95	3,379	2,716
Coated Hi Mg	2	1.82	3,940	3,081
Coated Hi Mg	3	1.97	3,438	2,778
Coated Hi Mg	4	1.90	3,736	2,804
Coated Hi Mg	5	1.83	3,749	2,908
Coated Hi Mg	6	1.95	3,870	3,177
Coated Hi Mg	7	1.78	4,396	3,150
Coated Hi Mg	8	1.87	3,561	2,891
Coated Hi Mg	9	1.84	3,813	3,030
Coated Hi Mg	10	1.76	3,866	2,723

Uncoated Low Mg	1	Data Not Recorded		
Uncoated Low Mg	2	5.34	1,157	2,713
Uncoated Low Mg	3	5.37	1,264	2,668
Uncoated Low Mg	4	4.72	1,252	2,817
Uncoated Low Mg	5	4.70	1,175	2,860
Uncoated Low Mg	6	4.61	1,278	2,720
Uncoated Low Mg	7	4.78	1,243	2,796
Uncoated Mid Mg	1	Data Not Recorded		
Uncoated Mid Mg	2	4.03	1,464	3,036
Uncoated Mid Mg	3	4.04	1,274	2,399
Uncoated Mid Mg	4	3.96	1,437	2,847
Uncoated Mid Mg	5	4.00	1,513	2,969
Uncoated Mid Mg	6	4.30	1,647	3,122
Uncoated Mid Mg	7	4.11	1,557	3,146
Uncoated Hi Mg	1	3.93	1,762	3,147
Uncoated Hi Mg	2	4.25	1,679	3,348
Uncoated Hi Mg	3	3.75	1,766	3,246
Uncoated Hi Mg	4	3.85	1,574	3,256
Uncoated Hi Mg	5	3.82	1,710	3,448
Uncoated Hi Mg	6	3.96	1,751	3,221
Uncoated Hi Mg	7	3.92	1,698	3,272

Table 18. Radiometric Test; Values for Individual Pellets