DEVELOPMENT OF PASSIVATED PYROPHORIC METAL POWDERS (HAFNIUM AND ZIRCONIUM) WITH REDUCED ELECTROSTATIC DISCHARGE (ESD) SENSITIVITY

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ABSTRACT

High-density metals such as hafnium and zirconium are of interest to the propellant, explosive, and pyrotechnic industry due to their high oxidation enthalpies; however, these metal fuels suffer from electrostatic discharge (ESD) complications creating safety concerns. Previous attempts to passivate these powders have failed due to abrasion of the protective coatings. Two novel methods for desensitizing hafnium powder based on technology developed by MACH I, Inc. were successfully demonstrated in a Phase I SBIR project: both approaches negate abrasion complications. First, *Reactive-Milling* (*RM*) of hafnium powder with NH_4OH to 800 °C in a N_2 atmosphere produces a thin protective coating of HfN on hafnium. The olive-gold material exhibited elevated ESD thresholds. The second method, *RM* of hafnium and aluminum powders in an Ar atmosphere to 700° C followed by nitridation at 610°C, generated a protective intermetallic coating (Al_2Hf) on hafnium, which also exhibited excellent passivation levels. These materials were fully characterized by optical, SEM, and XRD analyses.

1. INTRODUCTION

Metal powders are used as fuels for incendiary devices but suffer from pyrophoric and electrostatic discharge (ESD) complications.[1] In the presence of oxidizers, such characteristics are unsafe and clearly unacceptable. Consequently, it is necessary to develop technology that passivates pyrophoricity, reduces ESD sensitivity, and is environmentally safe without compromising metal fuel combustibility.

Previous efforts to reduce ESD sensitivity of metal powders have relied on surface modifications that suffer abrasions from routine wear over time.[2] Such complications are even more alarming due to false confidence in the powder's stability. An alternative approach to passivating metal powders that does not

degrade over time is desired. Several essential features are required, making this a difficult task. On one hand, the pyrophoric nature is desired, yet on the other hand, this pyrophoricity creates ESD complications. Similarly, care must be taken to desensitize the powder sufficiently without rendering its combustibility to be too poor or nonexistent. This delicate balance must be met consistently, since one susceptible particle could set off the entire fuel contents.[3] Although it is difficult to imagine a scenario in which a particle is left uncoated, one expects particles to exhibit a distribution of surface coating depths. Consequently, a higher average coating thickness will be required to compensate for particles with thinner coatings, which results in less energetic performance. However, if received hafnium particles were uniform in shape and size (*e.g.,* spherical), a thinner coating thickness could be utilized, passivating the material adequately within appropriate safety margins.

The scope of this work employs surface coating technology that has been developed to some degree in a previous SBIR grant (N90-20 "Production and Coating of Pure Boron Powders") with some modifications.[4] By successfully altering the hafnium powder surface, ESD sensitivity (or threshold) may be manipulated to safe levels. Sparking threshold measurements on samples are indicative of passivation success. In general, two different types of coatings were employed by MACH I: (1) reaction of surface hafnium with nitrogen (N_2, NH_4Cl, and NH_4OH) and oxygen to form a surface layer of nitride or oxide, and (2) physically depositing a less reactive metal on hafnium and for aluminum coats further passivation by reacting with nitrogen at elevated temperatures.

2. EXPERIMENTAL

2.1 Electrostatic Discharge (ESD) Measurements

ESD sensitivity was tested employing commercial instrumentation at the Indian Head facilities. In general, a 1 g sample was divided amongst ~25 pans. The initial ESD threshold was determined by sacrificing the first pan (*i.e*., continually increasing the capacitance level until a spark was observed). Then 20 successful trials were required to pass a threshold level (a successful trial occurs when no sparks or flashes were observed). If any of the 20 trials fail, the next lower threshold level was attempted, and so on, until either 20 successes were accomplished or an "all-fire" was observed.

2.2 Surface Characterization

Differential-scanning calorimetry (DSC) was performed on a TA Instruments 2010 DSC instrument. Samples were referenced to an empty aluminum pan and measured in an Argon atmosphere. TA Instruments performed the TGA under a N_2 atmosphere while Microtek, Inc performed optical microscopy and particle-size distribution. Micron, Inc. performed the SEM work. XRD analyses were performed using a Sieman's Model-K4 X-ray diffractometer with a 30 Amp current applied through a 30 V potential.

3. RESULTS AND DISCUSSION

3.1 Surface Nitridation/Oxidation of Hafnium Powder

Formation of surface nitride or oxide was readily accomplished by heating hafnium powder under appropriate atmospheric conditions. By producing a surface coating of nitride or oxide, a reduction in ESD sensitivity was expected. Controlling the depth of nitridation or oxidation should fine-tune ESD sensitivity. In effect, nitridation or oxidation protected the material from further reaction with N_2 or O_2. Diffusion through nitrided or oxidized hafnium surface slowed as surface coating depth increased. In general, this occurred as expected, but did suffer from some inherent complications.

First, unless metallic nitride or oxide occupies the same volume as pure hafnium, more stress and strain are added to the system's surface. This phenomenon has been documented and the parameter of import, the Pilling-Bedworth ratio (PBR), is a measure of the surface coating stability.[5] For example, the PBR for TiO_2 is 1.73 compared to 1.09 for TiN. PBR ratios near 1 generate less strain, therefore one predicts (and observes) fissures and cracks to develop in TiO_2 coated titanium particles. This exposes fresh titanium, which leads to rapid oxidation. However, the nitrided material is stable (as predicted), and does not develop fissures or cracks. It is expected and observed that similar results would be obtained for hafnium (HfO_2 and HfN, Figure 1).

Another complication is the amount of N_2 that is absorbed by hafnium. Studies have indicated as much as 5% weights gain of N_2 by hafnium.[6] This is unreacted N_2 generating a solid solution of N_2 dissolved in hafnium. Although this seems innocuous, quantifying the amount of hafnium converted to HfN becomes difficult. In addition, if the presence of N_2 becomes undesirable, removal of the dissolved N_2 becomes an issue.

The first two complications can be addressed within our own technology and process engineering. A third more difficult concern relates to the hafnium powder as it is received. In order to obtain consistent results, uniform feedstock becomes an issue. An optimum source of hafnium would be spherical particles in a limited size range. With uniform particle shape and size, our process would produce a uniform surface coverage. The technology to produce spherical particles is known, and should be explored in further work.[7]

Despite these complications, passivation of hafnium powder by direct nitridation appears to be particularly suited for the production of inexpensive, environmentally benign, large quantities of combustible material. Another feature is the capability to produce the nitrided material at reduced temperatures in the presence of ammonia, NH_3. Previous reports have mentioned the mechanism for nitridation in the presence of NH_3: (1) NH_3 is absorbed onto the hafnium surface, (2) the hafnium facilitates the breakup of NH_3 into three absorbed H atoms and one absorbed N atom, (3) the formation of hafnium hydride (HfH) is kinetically fast, but thermodynamically less stable than hafnium nitride (HfN) formation, and (4) in an atmosphere of N_2, the HfH is converted rapidly to HfN.[6] Our approach was to examine the possibility of substituting ammonium hydroxide (NH_4OH) for NH_3, and to determine if the formation of HfN could be accomplished at lowered temperatures.

3.2 Physical Vapor Deposition of Metals on Hafnium Powder

The formation of magnesium coated metals has been demonstrated by MACH I in earlier studies.[4] By coating hafnium with magnesium using a physical vapor deposition (PVD) process, the ESD sensitivity is expected to be reduced. This technique is amenable to metals with low melting points or that have low decomposition temperatures to give metallic coated materials. In general, coating the extremely sensitive hafnium powder with a less reactive metal should be adequate for ESD desensitization. Moreover, in most instances, controlled nitridation (or oxidation) of the less reactive coating material can be accomplished at lower temperatures to yield exceptionally desensitized hafnium without using up any hafnium.

To avoid premature nitridation (or oxidation) of the metallic coating and hafnium powder, inert atmospheres are necessary. Again, with our reactor this is merely switching gas lines from N_2 or O_2 to Ar to accomplish this task. Another undesirable trait that occurs with the magnesium-coated hafnium is the reactivity of magnesium in aqueous environments. However, changing to aluminum coatings negates this last issue. One additional concern is that the metallic coating be environmentally safe, which is not a problem in the systems above.

In fact, aluminum coated hafnium has the desirable features of being environmentally safe, unreactive towards water, and providing additional combustion source as aluminum is also employed as a fuel. Aluminum is passivated at lower temperatures (with either N_2 or O_2) and does not suffer from fissure and

(A)

(B)

(C)

(D)

Figure 1.   SEM of HfO_2 coated Hf (A), HfN coated Hf (B), Al coated Hf (C), and untreated Hf (D). Micrographs were taken at $1000\times$ magnification.

crack formation. Still, care must be exercised to examine the materials that are produced under these conditions. For instance, intermetallic species have been observed in our own trials–not the expected aluminum coated hafnium, but rather Al$_2$Hf on the surface. Formation of intermetallic materials by using ball mills is common (mechanical alloying, MA).[8] The high-energy impacts of the milling balls break-up the starting powders and generates the intermetallic species as intermediates during the production of amorphous alloys. The term *Reactive-Milling* (*RM*) has been adopted to describe this process.

3.3 ESD Measurements

The ESD sensitivity threshold levels are summarized in Table 1. Inspection of the data reveals improved ESD passivation for two of the four technical objectives: (1) direct nitridation of hafnium using NH$_4$OH to form a thin coating of HfN, and (2) metal encapsulation of hafnium powder. Based on these preliminary measurements, modifications for the two successful approaches were investigated.

3.3.1 Thin Coating of HfN on Hafnium Powder via Direct Nitridation in Presence of NH$_4$OH

Examination of the ESD threshold limits as various reactor parameters were adjusted in the direct nitridation process yielded several trends. In comparing runs #4, #20, and #12 (where the T$_{max}$ was increased from 700 to 800 °C), a greater degree of passivation is observed with an increase in T$_{max}$. This is a consequence of increased HfN coating thickness on the hafnium powder. Attempts at mimicking increased reactor times (which are a consequence of heating to higher temperatures) to study thermodynamic aspects of hafnium nitridation (*e.g.*, run #15 vs. #12) were also investigated. The ESD measurements indicate the sensitivity to be more dependent upon temperature than residence time, *i.e.*, the HfN coating thickness is a function of heating to 800 °C, and not just a result of having heated the material at T > 700 °C for an additional 2 hours. Apparently, increases in T$_{max}$ allow for greater diffusion of N$_2$ into the hafnium particle generating a thicker coating of HfN.

3.3.2 Thin Coating of Aluminum on Hafnium Powder via Reactive-Milling (RM)

After initial ESD results indicated that encapsulation of hafnium powder with magnesium showed some promise, it was decided that switching to aluminum would be advantageous. Early work with aluminum yielded unsatisfactory results, but there was indication that higher percentages of aluminum coating performed better. The results of these studies were impressive: (a) a stable intermetallic Al$_2$Hf complex was generated, and (b) this intermetallic showed high levels of passivation. This method of forming a stable intermetallic is unique to our system, and the fact that the intermetallic shows increased passivity is remarkable. Upon successful passivation of hafnium a passivation of zirconium was performed and yielded a high level of passivation.

3.4 Surface Characterization

3.4.1 SEM and Particle-Sizing Analysis

SEM was performed on HfO$_2$ coated hafnium, HfN coated hafnium, aluminum (15%) coated hafnium, and the hafnium powder "as-received", Figure 1. Each type of surface treatment produces a unique surface as shown in the micrographs. These data indicate that "as-received" hafnium powder is very irregular in size and shape, but reacting with O$_2$ generates extremely nodular material with long fissures. The aluminum treated material appears to be "soft" and much larger in size in comparison to the other samples. Whether the surface irregularities that are observed arise from the non-uniform starting hafnium powder or the surface treatment will need to be addressed in further work.

Several samples were examined by optical microscopy for particle-size analysis. The results of these studies are tabulated in Table 2. Microscopy showed the inconsistency in particle shape and size. Interestingly, a particle size of ~6 μm is the most frequent size for all samples (the average particle-size is skewed by the larger particles). In general, the untreated hafnium particles are larger and have a greater

Table 1.

Summary of electrostatic discharge (ESD) measurements.

Run	Reactants	Atmosphere	T_{max}	Reaction time	ESD TIL
2	Hf + N$_2$ (g)	100% N$_2$	700 °C	130 min	all fire
3	Hf + NH$_4$Cl (17%)	100% N$_2$	700 °C	95 min	all fire
4	Hf + NH$_4$OH (drops)	100% N$_2$	700 °C	128 min	0.0100 J
5	Hf + Mg (5%)	100% Ar	700 °C	105 min	0.0084 J
6	Hf + O$_2$ (g)	4% O$_2$/96% Ar	700 °C	124 min	all fire
7	Hf + NH$_4$Cl (10%)	100% N$_2$	600 °C	51 min	all fire
8	Hf (baseline)	100% Ar	700 °C	110 min	all fire
9	Hf + Al (5%)	100% Ar^a	700 °C	110 min	all fire
10	Hf + Al (10%)	100% Ar^a	700 °C	167 min	all fire
11	Hf + NH$_4$OH (soaked)	100% N$_2$	700 °C	133 min	all fire
12	Hf + NH$_4$OH (drops)	100% N$_2$	800 °C	214 min	0.0230 J
13	Hf + NH$_4$OH (soaked)	10% N$_2$/90% Ar	700 °C	88 min	0.0084 J
14	Hf + NH$_4$OH (soaked)	10% N$_2$/90% Ar	700 °C	210 min	0.0100 J
15	Hf + NH$_4$OH (drops)	100% N$_2$	700 °C	226 min	0.0100 J
17	Hf + Al (15%)	100% Ar^c	700 °C	168 min	0.0230 J
20	Hf + NH$_4$OH (drops)	100% N$_2$	750 °C	152 min	0.0100 J
21	Hf + NH$_4$OH (drops)	100% N$_2$^{d}	750 °C	155 min	0.0084 J
22	Hf + Al (15%)	100% Ar	700 °C	180 min	> 6 J
23	Zr (baseline)	100% Ar			0.00025 J
24	Zr + Al (15%)	100% Ar	700 °C	180 min	0.036 J

variance, except for run # 17, aluminum (15%) coated hafnium. This corroborates well with ESD results obtained on the aluminum (15%) coated hafnium (run #17). Recall that larger particles have better ESD stability. In fact, the aluminum (15%) coated hafnium particles (as well as all metal encapsulated trials) are expected to be larger due to the actual process involved. Agglomeration of the particles also occurs as evidenced by the large amounts of material left on 325 mesh (> 50 % of the recovered material), with improved processing (run #22) particle agglomeration is reduced (10 % of the recovered material). Whereas the metal encapsulated hafnium particles are predicted to be larger, the ceramic-coated particles (i.e., HfO$_2$ and Hf$_x$N$_y$ thinly coated hafnium) are predicted to be nearly the same size. Examination of HfO$_2$ results in the unexpected observation of decreased particle size and variance (the smallest generated using our reactor). Apparently, hafnium powder reacts with O$_2$, followed by surface volume mismatches that create fissures and cracks to develop, which exposed fresh hafnium. The exposed hafnium reacts rapidly with O$_2$ yielding splintered HfO$_2$ coated hafnium and an overall decrease in particle size.

Table 2.

Optical microscopy and particle-size analysis results
(first row corresponds to total range, second row to a preset range, 0 to 75 μm).

Run	Sample	<diameter>	Standard Deviation (±μm)	Variance	Particle Count	Range, μm
8	Hf (baseline)	16.0 μm	14.8	220.4	1023	2 to 77
		15.9 μm	14.7	216.9	1022	0 to 75
6	HfO_2 coated	12.3 μm	11.0	121.9	1055	3 to 76
		12.2 μm	10.9	118.2	1055	0 to 75
12	HfN coated	15.1 μm	13.2	175.2	1040	3 to 65
		15.1 μm	13.2	175.2	1040	0 to 75
--	HfN (Aldrich)	8.2 μm	6.1	37.2	1030	3 to 59
		8.2 μm	6.1	37.2	1030	0 to 75
16	Al_2Hf (15% Al)	15.2 μm	13.4	180.4	1042	2 to 67
		15.2 μm	13.4	180.4	1042	0 to 75
17	Al_2Hf (15% Al)	17.2 μm	15.0	226.4	1080	2 to 72
		17.2 μm	15.0	226.4	1080	0 to 75

3.4.2 DSC and TGA

Unfortunately for high-refractive systems such as Group IV metals (Ti, Zr, and Hf) and their corresponding nitrides and oxides, the temperature window (T < 650 °C) for our instrument does not extend to regions that allow for characteristic transitions to be observed. Typically, the onset of an exotherm was observed, but no significant differences between samples were detected. These results are not surprising given the refractive nature of HfN and hafnium. Still in future work, DSC might be a viable method for studying the reactivity of small amounts (~100 mg) of hafnium powder in different environments (NH_3, N_2, or O_2), if appropriate safeguards are maintained (*i.e.*, the ability to quench with the rapid influx of Ar) to avoid "run-away" combustion, which might damage the instrument.

The TGA results are somewhat similar to DSC studies since the temperature window was too low to observe much activity (T < 800 °C) even in a N_2 atmosphere. Some of the samples display weight losses at lower temperatures, followed by weight gains at T > 400-500 °C. This suggests that hafnium reacts with N_2 at significantly lower temperatures than reported albeit at very slow rates.[1b,c]

3.4.3 XRD Analysis

The preliminary results from XRD analysis provide much insight into the materials generated. The 2θ and intensity values are listed in Table 3 along with 2θ values of authentic Al_2Hf. The sharpness of the peaks in the spectra infers that the samples are not amorphous mixtures (or alloys), rather they are intermetallic in nature. The correspondence between the aluminum (15%) coated products and Al_2Hf is very good. From these data, it appears as though our process is able to generate in a unique way the stable Al_2Hf intermetallic. More work needs to be done in unambiguously determining the surface structure in these materials, and quantifying the amount of unreacted hafnium left in these samples. In addition, these samples need to have their combustibility properties investigated.

Table 3.

Summary of XRD data.

HfN on Hf Run #21		Al flake (15%) on Hf Run #18		Al (15%) on Hf Run #16		Al (10%) on Hf Run #10		Al$_2$Hf (authentic)
2θ	I/I$_{100}$	2θ	I/I$_{100}$	2θ	I/I$_{100}$	2θ	I/I$_{100}$	2θ
		19.57	25					19.5
		20.50	17	20.45	19			20.5
		22.11	16	22.12	13			22.1
		22.80	14	22.83	13	22.72	19	
24.88	9	24.87	10	24.85	15			
				27.15	10	26.9	17	
28.59	6	28.43	22	28.39	17	28.41	16	28.4
				29.17	10			
		31.6	10	31.75	13	31.62	11	
32.10	19	32.19	9	32.19	14	32.21	29	
						33.20	21	
34.35	48	34.22	100	34.39	100	34.17	25	34.3
						35.18	32	
35.60	9					36.1	8	
36.68	100	36.88	81	36.7	51	36.82	100	36.7
37.85	9	37.82	11	37.8	15	37.68	37	
		38.17	24	38.20	29	38.12	21	
39.81	26	39.70	26	39.8	36	39.39	11	39.8
		40.17	84	40.25	56	40.05	29	40.2
		41.11	25	41.12	18	41	6	41.1
		42.13	6					
		45.51	8	45.56	12			45.5
48.05	14	48.55	6			48.5	16	
		51.15	13	51.14	12			51.1
57.20	23	56.8	10					56.9
57.58	21	57.75	18	57.82	27	57.6	25	57.8
		61.21	11	61.23	10			61.3
		62.95	21	63.00	17			63.0
63.67	14							
		65.28	13	65.27	15	64.31	19	65.3
67.19	3							
		68.04	14	68.08	13	67.8	10	68.2
68.83	36	68.84	12	68.9	28	69.35	17	
69.88	10							
72.29	6	72.00	14	72	12			72.2
77.95	5	78.32	11			78.4	14	
		83.73	12	83.79	10			83.8
90.82	6							
91.19	5							
96.57	10							

4. Conclusions

During the course of this study, MACH I successfully reduced ESD threshold levels for hafnium and zirconium powders. Two of our approaches, nitridation by direct reaction to form HfN by using NH_4OH and metal encapsulation via PVD of magnesium, yielded improved passivation results for hafnium (*i.e.*, sensitivity levels better than hafnium powder). In an effort to optimize ESD threshold levels for these samples, modifications based on the successful approaches were systematically studied. Specifically, higher heating temperatures were employed for direct nitridation method and encapsulation of hafnium powder with aluminum instead of magnesium were examined. The results of these studies were impressive: (1) *substantially increased sensitivity performance for nitrided hafnium was obtained at a maximum heating temperature of 800 °C*, and (2) *a novel process for preparing a stable intermetallic species, Al₂Hf, was developed, which more importantly yielded the highest levels of desensitization. Both of these samples performed as well as equivalent size aluminum powder.*

The following conclusions were drawn from our experimental results:

- *Feasibility for passivating hafnium powder* by using direct surface nitridation to form HfN and metal encapsulation by using PVD of magnesium were successfully demonstrated.

- An *increase* in maximum temperature during direct nitridation reactions (with added NH_4OH) resulted in *increased passivation performance*.

- *Reactive-Milling* (RM) of hafnium and aluminum powders, followed by nitridation at moderate temperatures (T = ~600 °C) yielded intermetallic Al_2Hf, which gave the *highest levels of ESD desensitization*.

5. ACKNOWLEDGEMENTS

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