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THERMAL JETTING EFFECTS OF SHS REACTED TITANIUM DIBORIDE

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Thermal jets consisting of rapidly evolving gaseous and particulate materials are formed during the highly exothermic, well contained TiB_2 SHS reactions. The relative magnitudes of the effects of ignition location, precursor powder weight, boron type, titanium purity and nozzle orifice dimension on the jetting action have been measured. The thermal effects of the jets on surrounding materials are found to be strongly correlated with the amount of product material ejected from the reaction vessel. In particular, the most active parameters have been determined to be the nozzle orifice size and the type of precursor powder. This study has also yielded a method for controlling the deposition of jet materials onto nearby surfaces.

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1. INTRODUCTION

The self-propagating high-temperature synthesis (SHS) process is receiving increased attention as an efficient technique for producing high-technology ceramic materials.[1] However, many of the early applications of SHS focussed on utilizing the considerable heat energy liberated during the synthesis reaction rather than the final product material. Examples of these applications include the use of SHS as the source of heat for thermal batteries, aerosol dispersal and TOW missile infrared beacons.

The aim of this study was to investigate the feasibility of utilizing the heat energy and volatile gases produced during the titanium diboride SHS reaction to develop a directed heat energy source or thermal jet. It was hoped that this goal could be achieved by reacting titanium and boron powders in a containment vessel with a nozzle orifice. Although SHS reactions are sometimes referred to as 'gasless reactions', significant volumes of water vapor and hydrogen are known to be released from hygroscopic titanium and boron powder mixtures during synthesis.[2] Early tests of this configuration using steel target plates revealed the tendency for reacted material, ejected through the nozzle, to accumulate on the piece to be heated. This coating of ejected material served as a thermal insulator which had the undesirable effect of isolating the target piece from the majority of the directed heat energy. Thus, the first step in achieving the overall goal was to develop a method for avoiding the deposition of ejected material onto the target piece. Once a system is developed for avoiding these depositions, the feasibility of utilizing the titanium diboride SHS reaction as a heat source for directed thermal energy applications can be evaluated and the system optimized.

There exists an abundance of variables for this system which can potentially affect the performance of the thermal jet including: precursor powder mixing, precursor powder density, precursor powder purity, target plate stand-off distance, precursor powder ignition location, nozzle orifice diameter, quantity of precursor powder and precursor powder type. For a system with so many conceivably important parameters a logical first step is to ascertain which variables do indeed play an active role in determining the thermal jet effectiveness and which variables are inert with respect to this system. One method for identifying active variables is to perform a designed two-level factorial experiment. The details of this method are outlined in Appendix A. Of the system variables already listed, five were chosen as being the most likely candidates for investigation; precursor powder ignition location, nozzle orifice diameter, boron powder type (amorphous versus crystalline), titanium powder purity and quartity of precursor powder. The chosen variable values and designations are listed below:

A_+: precursor powder ignited in region closest to the nozzle orifice.
A_-: precursor powder ignited in region farthest from the nozzle orifice.
B_+: 300 grams of precursor powder mixture.
B_-: 150 grams of precursor powder mixture.
C_+: amorphous boron used in precursor powder mixture.
C_-: crystalline boron used in precursor powder mixture.
D_+: high purity titanium used in precursor powder mixture.
D_-: low purity titanium used in precursor powder mixture.
E_+: 0.56 centimeter diameter nozzle orifice.
E_-: 0.32 centimeter diameter nozzle orifice.

The "noise" effect of the other potential variables was minimized by holding these parameters constant.

2. EXPERIMENTAL PROCEDURE

2.1 Reaction Vessel

In order to reduce machining costs, turn-around time and testing variability, a reusable reaction vessel was developed. Preliminary testing indicated that vessels fabricated from common metals, such as steel or stainless steel, could not repeatedly withstand the high temperatures of the titanium diboride SHS reaction. A graphite reaction vessel was developed which was used as the reaction vessel for all the tests presented in this report. The details of this container are illustrated in Figure 1. The body of this vessel was an 11.0 centimeter long graphite tube with an outer diameter of 11.1 centimeters and an inner diameter of 6.4 centimeters. A back plate and nozzle plate were also machined from graphite with recessed surfaces which pressure-fit the inner body wall. Pressed graphite sheet gaskets were employed to eliminate or minimize the amount of gas blow-by at the pressure plate seals. A coaxial 0.56 centimeter diameter hole was drilled through the nozzle plate. For tests requiring a smaller nozzle diameter, a nozzle insert was press fit into this hole.

The compressive force required to hold the nozzle and back plates to the body tube was provided by two steel pressure plates connected by threaded rods. The pressure plate on the nozzle end of the assembly also served as a holder for the target plates. Stand-off distance between the nozzle and target plates was determined by supporting blocks located between the nozzle plate and associated pressure plate.

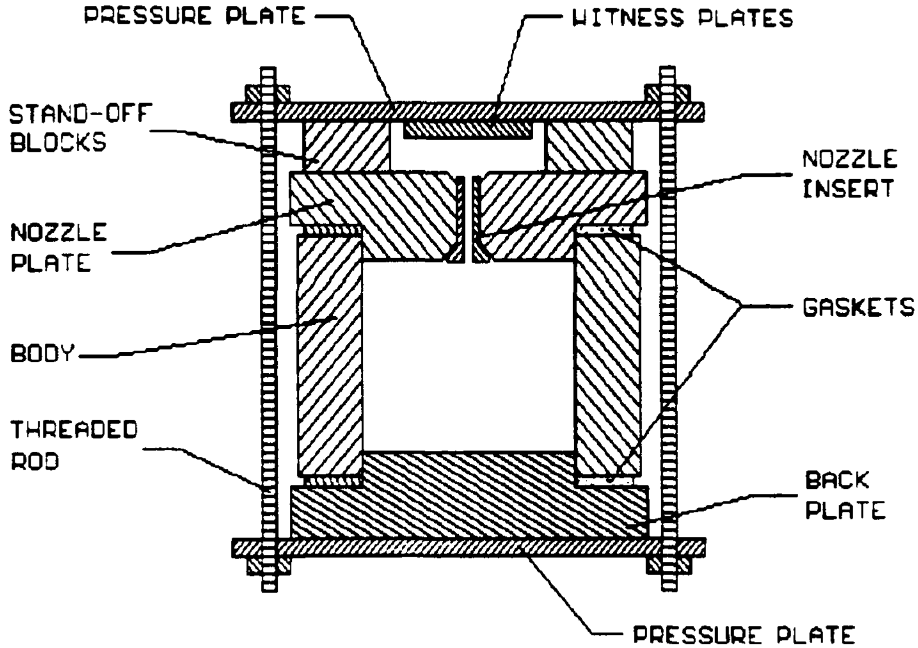

Figure 1. Reusable graphite reaction vessel assembly.

2.2 Precursor Powders

Four different batches of precursor powders were prepared from the possible combinations of two different lots each of boron and titanium powder. These powders and their designations are enumerated below:

C_+: Boron, amorphous 5 micron Consolidated Astronautics* lot# 1112, stated purity 96.5%.
C_-: Boron, crystalline -325 mesh Consolidated Astronautics, lot# 202, stated purity 99.5%.
D_+: Titanium, high purity -325 mesh Micron Metals& specification Ti-020.
D_-: Titanium, low purity Atlantic Equipment Engineers% specification Ti270, stated purity 99.7% but latter determined to contain 3% iron.

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*Consolidated Astronautics, Smithtown, NY 11787

&Micron Metals, Inc., Salt Lake City, Utah 84120

%Atlantic Equipment Engineers, Bergenfield, NJ 07621

The four powder mixtures were designated C_+D_+, C_+D_-, C_-D_+ and C_-D_- according to the specific titanium and boron powders used. For each batch of mixed powders, 689 grams of titanium were mixed with 311 grams of boron to produce a mixture with a titanium:boron atomic ratio of 1:2. All powders were mixed in a 2 liter ceramic container using the following procedure:

1) mixed 1 hour without ceramic mixing balls.
2) mixed 1 hour with 6 ceramic mixing balls.
3) inside of ceramic container scraped as required.
4) mixed 1 hour with 6 ceramic mixing balls.

Step 3 was included because mixtures which included amorphous boron (C_+) tended to cake on the inside of the mixing container when the ceramic mixing balls were included.

2.3 Testing Procedure

With the exception of the nozzle diameter variation, the ordering of the individual tests was randomized to avoid potential biasing of certain variables due to unintentional variations in procedure with time. All the small nozzle diameter tests were conducted first followed by the large nozzle diameter tests to avoid the complications associated with repeatedly inserting the pressed-fit nozzle insert.

The reaction vessel was prepared by fitting the graphite body tube onto the back plate, including a sheet graphite gasket, and loading the specified amount and type of precursor powder into the graphite container. During the loading of the vessel, care was taken to avoid unnecessary powder compaction or tamping. A commercially available electric ignitor[a] was located in the portion of the green compact where the titanium diboride reaction was to be initiated. The leads from the electric ignitor were routed out through the nozzle and the nozzle plate was fit onto the body including another graphite sheet gasket.

The targets for all tests were eight stacked layers of 1 millimeter thick 6061-T4 solution heat treated sheet aluminum. Pretest weight measurements were recorded for the target assemblies. The target was mounted to the nozzle end pressure plate and spaced 6.8 millimeters from the nozzle plate by stand-off blocks. The entire reaction vessel assembly was then securely sealed by tightening the threaded stock between the two pressure plates.

Following the reaction event, post-test measurements were recorded for the weight of the product material remaining in the reaction vessel, the weight of the target assembly and the number

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[a]Estes Industries, Penrose, CO 81240

of aluminum plates penetrated by the thermal jet. Examination of the sheet graphite gaskets during disassembly of the reaction vessel indicated whether the hot gases evolved during the SHS reaction had blown past the nozzle or back plate seals. Selected samples of ejected product material were collected for analysis by scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDS).

3. RESULTS AND DISCUSSION

Experimental parameters and results for the thermal jet tests are listed below in Table 1.

TABLE 1
TEST PARAMETERS AND RESULTS

seq #	run #	A, ign. loc.	B, pow. qty.	C, Bor typ.	D, Ti typ.	E, noz. dia.	target weight loss (gm)	% eject.	num. plate penet.	blow by ?
12	1	-	-	-	-	+	21.85	36.6	8	no
4	2	+	-	-	-	-	5.46	8.1	8	no
7	3	-	+	-	-	-	4.60	10.7	8	yes
10	4	+	+	-	-	+	28.83	18.8	8	no
5	5	-	-	+	-	-	1.91	4.0	7	no
13	6	+	-	+	-	+	9.26	11.1	8	no
11	7	-	+	+	-	+	4.80	17.2	8	yes
8	8	+	+	+	-	-	3.86	3.9	7	yes
3	9	-	-	-	+	-	1.81	1.8	8	no
15	10	+	-	-	+	+	8.43	10.4	8	no
16	11	-	+	-	+	+	7.41	11.3	8	no
2	12	+	+	-	+	-	0.52	0.7	3	no
14	13	-	-	+	+	+	8.08	13.6	8	no
1	14	+	-	+	+	-	0.00	1.4	1	no
6	15	-	+	+	+	-	1.39	1.9	8	yes
9	16	+	+	+	+	+	9.04	5.9	8	no

The sequence number is the order in which the tests were performed. Run numbers were defined to serve as test designations. The pluses and minuses in columns 3 through 7 refer to which of the two possible levels was used for each parameter as defined earlier. Thus in run #4, which was the tenth test to be performed, the parameter values of $A_+B_+C_-D_-E_+$ indicate that the precursor powder charge of 300 grams of crystalline boron and low purity titanium was ignited in the portion of the powder closest to the 0.56 centimeter diameter nozzle. The target weight loss column lists how much material was removed from the target plates due to the effect of the thermal jet and thus represents a figure of merit for the jet performance. The % ejected column lists the percentage of product material that was lost from the reaction vessel during the titanium diboride SHS reaction. Plates penetrated indicates how many of the 8 available target plates were penetrated by the jet. The column labelled blow-by indicates whether there was any sign of leakage past the gasketed surfaces of the reaction vessel following the reaction.

The variable with the largest effect on the performance of the thermal jet was the nozzle orifice diameter, (E). As shown in Figure 2, the tests with the larger nozzle diameter had a statistically significant increase in the amount of target plate material removed by the thermal jet.

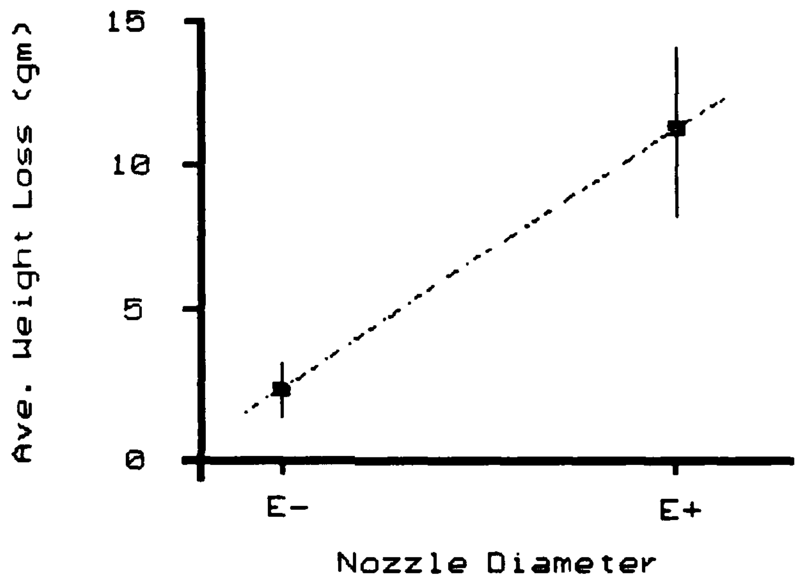

Figure 2. Effect of nozzle orifice diameter on thermal jet performance.

The next three variables with the largest relative effect were the boron type, (C), the titanium type, (D) and the interaction between the boron and titanium types, (CD). These data are best comprehended graphically as shown in Figure 3. The D_+ high purity titanium is shown to be a bad actor in that all tests using this titanium type yield low target plate weight losses. Relatively impure D_- titanium with its 3% Iron impurity yields high target weight loss results only when used in conjunction with the C_- crystalline boron. Thus the types of precursor powders used in these reactions are shown to have a significant effect on the resulting thermal jet performance.

When considered by themselves, the single factors A, ignition location, and B, weight of powder charge, have no significant effect on the performance of the thermal jet as shown in Figures 4 and 5. However, there is an interesting interdependence

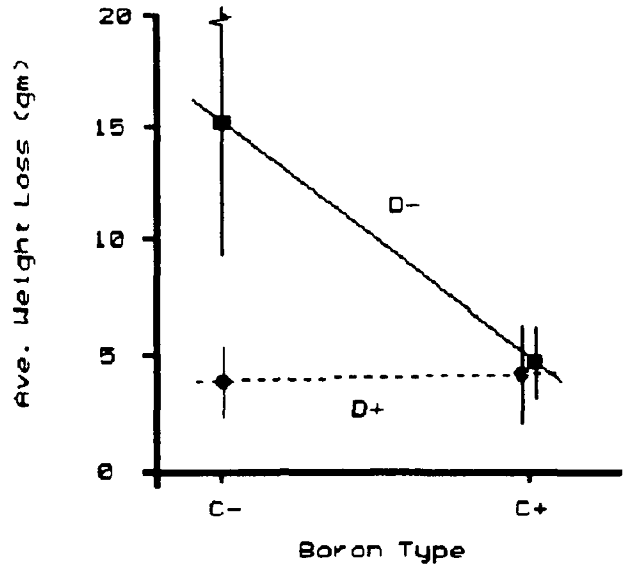

Figure 3. Effect of boron and titanium types on jet performance.

between the ignition location and the quantity of precursor powder as shown in Figure 6. From this figure it appears that increasing the amount of precursor powder charge increases the performance of the thermal jet for the condition of ignition in the region closet to the nozzle but decreases it for the condition of ignition in the region farthest from the nozzle. This apparent interaction between the A and B variables is most probably an artificial situation created by the specifics of the reaction vessel. From the Table of Test Parameters and Results, Table 1, it is seen that 3 of the 4 tests which resulted in gasket blow-by had the test parameter configuration A_-B_+, ignition of a large powder charge at the point farthest from the nozzle. The conditions of large powder charge and ignition far from the nozzle apparently combined to drive the precursor powders towards the nozzle which plugged the orifice and resulted in gasket blow by. Thus the combination of large powder charge and bottom ignition should not be utilized when reaction vessel rupture is a possibility.

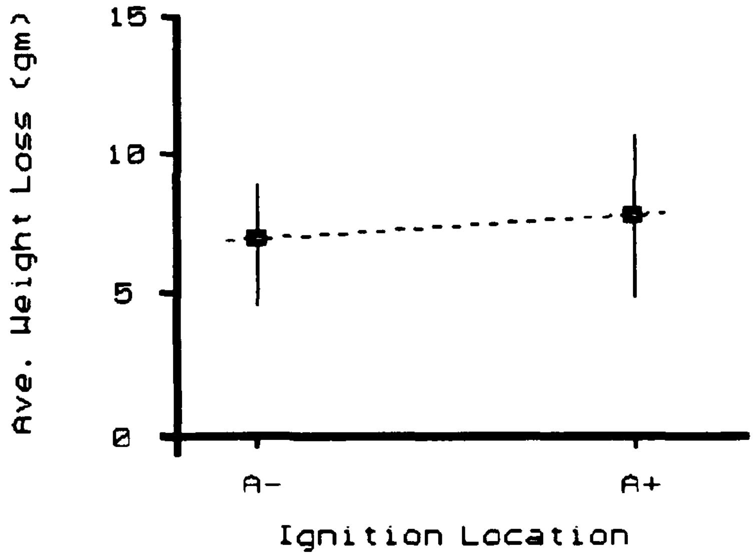

Figure 4. Effect of ignition location on thermal jet performance.

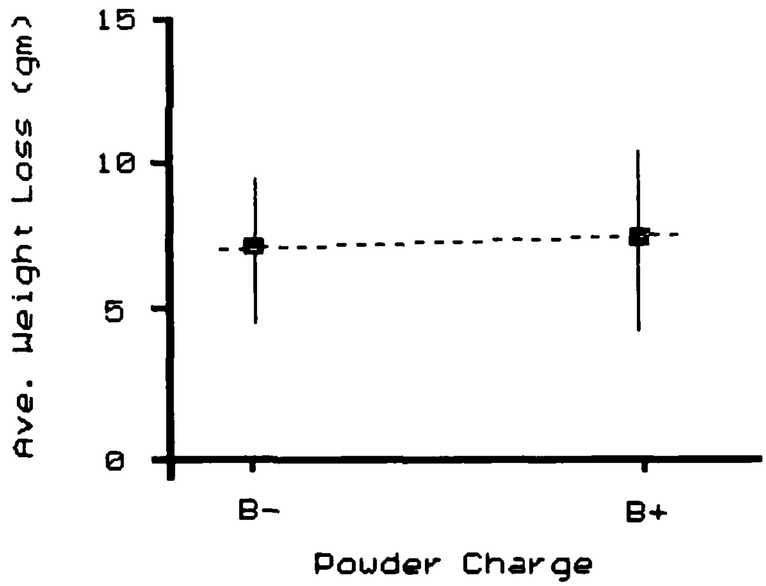

Figure 5. Effect of quantity of precursor powder on thermal jet performance.

Having determined the relative effects and parameter levels which produced the greatest mass loss for aluminum target plates, the question of thermal jet penetration through steel plates was again considered. It was this scenario for which previous tests were hampered by undesirable deposits on the target surface. This

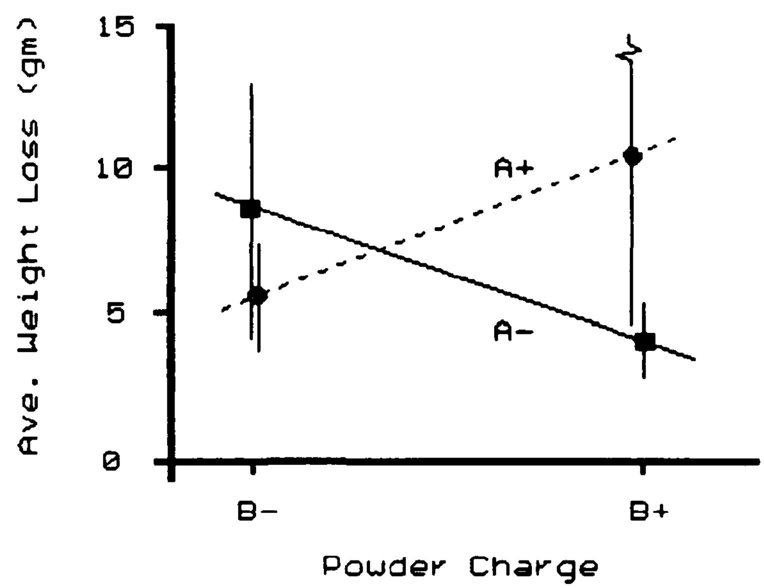

Figure 6. Combined effects of ignition location and quantity of precursor powder on thermal jet performance.

test was conducted using the variable values determined to yield maximum thermal jet performance; ignition near the 0.56 centimeter diameter nozzle of 300 grams of crystalline Con Astro -325 mesh boron and Atlantic Equipment Engineers titanium. The target was a stack of 19 sheet steel plates, each with a thickness of 0.5 millimeter, for a total target thickness of 8.7 millimeters. All other parameters, such as stand-off distance, powder density, etc., were set to the same values used in the parameter study. The thermal jet penetrated 7 of these plates for a total penetration distance of approximately 3.2 millimeters. Perhaps more important, was the fact that there was no indication of a thermally insulating layer as previously observed.

The amounts of material ejected from the reaction vessel during each test were also recorded. These values were determined from the difference in weight between the precursor powder charge and the product material recovered from the reaction vessel. A factorial analysis of the dependence of the percentage of material ejected on the five system variables revealed that the nozzle orifice diameter and type of precursor titanium powder were the two factors which had a statistically significant effect. These results are plotted in Figures 7 and 8.

The fact that generally the same system variables control the performance of the thermal jet and the amount of material ejected from the reaction vessel suggests a correlation between these two effects. Figure 9 indicates that there is indeed a strong correlation with increased material ejection yielding increased thermal jet performance. SEM and EDS where utilized to

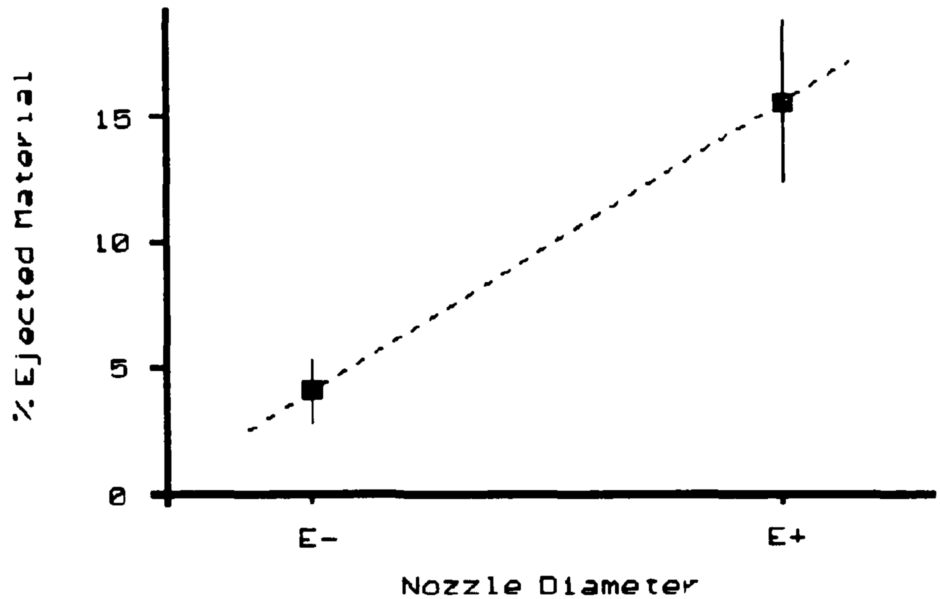

Figure 7. Effect of nozzle orifice diameter on percent of ejected material.

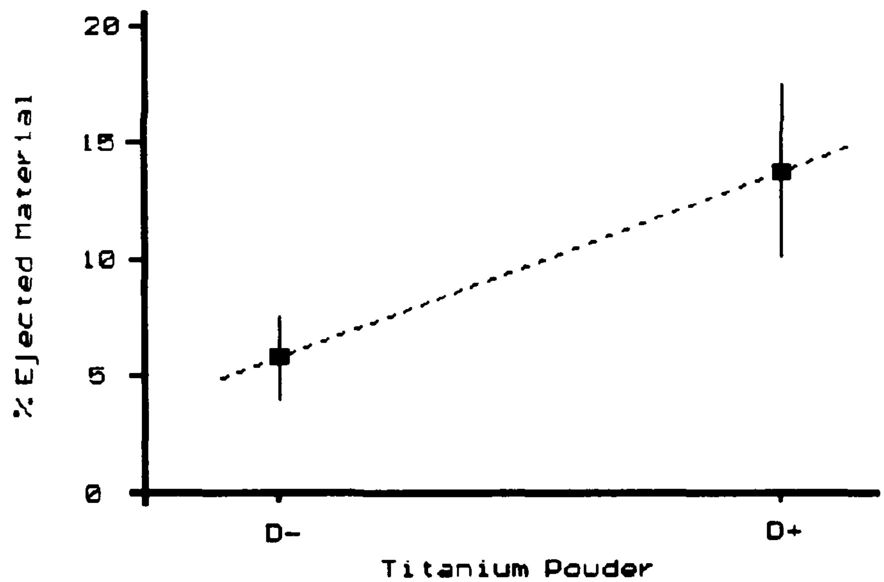

Figure 8. Effect of titanium precursor powder on percent ejected material.

characterize the material ejected from the reaction vessel by the titanium diboride SHS reaction. In general, the ejected material consisted of approximately micron sized particles containing a high concentration of the iron, aluminum, magnesium, silicon and potassium impurities present in the precursor powders. Thus it appears that the relatively low melting point impurities in the precursor powders were liquified during the reaction and preferentially ejected through the nozzle orifice. This finding suggests that it may be possible to enhance thermal jet performance by selectively adding impurities to the precursor powders which will melt and be driven out during the titanium diboride reaction. An additional test using steel target plates and the parameters which yielded maximum jet performance was conducted with 20 wt.% -100 mesh iron[$] added to the precursor powders. Rather than enhancing the effect of the jet, the added iron plated out on the steel target plates resulting in reduced plate penetration. Thus the type of impurities which can be added to the precursor powders to enhance jet performance is a critical parameter which requires further investigation.

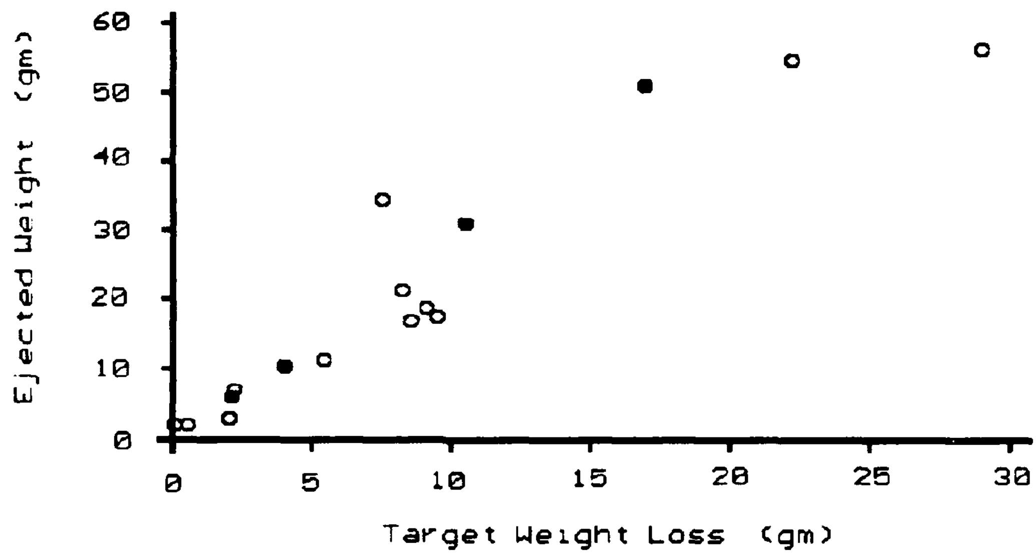

Figure 9. Correlation between ejected material weight and target weight loss. Solid data points represent tests where gases escaped past gasketed surfaces.

4. SUMMARY

A two-level factorial experiment has been conducted to determine which system variables play an active role in determining the penetrating performance of a titanium diboride SHS jet. In particular, a means for preventing the deposition of product material onto steel target plates was sought. The variables considered were the location of SHS reaction ignition in the precursor compact, the quantity of the precursor compact, the type of boron powder (amorphous versus crystalline), the purity

[$] Materials Research Corp., Orangeburg, NY 19062.

of the titanium powder and the diameter of the nozzle orifice. These tests were conducted utilizing a reusable graphite reaction vessel. The variables with the largest effect on the performance of the thermal jet were the nozzle orifice diameter and the types of precursor powders. In particular, crystalline boron powder and low density titanium powder fired through a relatively large 0.56 centimeter diameter nozzle avoided the deposition of product material and maximized the amount of material removed from the target plates. Thus, additional experimentation on this system should be focussed at determining what nozzle dimension and precursor powder characteristics optimize the penetrating performance of the SHS jet. A correlation was observed between thermal jet performance and the amount of material ejected from the reaction vessel. Specifically, the ejection of large amounts of product material yielded enhanced penetration and removal of material from the target plates. Analysis of the ejected product material by scanning electron microscope revealed micron sized particles. Thus the potential exists for developing this system as a particle generator. Additional tests with iron added to the precursor powder mixtures produced poor target penetration results and indicates that added impurities may be a critical parameter which requires further investigation.

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APPENDIX A

One method for identifying whether the variables associated with a system or process are active or inert is to perform a designed two-level factorial experiment.[3] This technique allows a relatively large number of variables to be surveyed with a minimal amount of testing. By assigning only two levels to each variable and selecting parameter levels for each individual test in a premeditated way, each variable level is included the same number of times during the series of experiments resulting in a balanced matrix of test results. This type of testing yields a ranking of these parameters with respect to magnitude of effect and determines which sense of variable level adjustment moves the system toward the desired result. Thus, this technique is useful for quickly sifting through a system's variables to determine which variables should be considered for additional optimization testing.

For the work presented in this report, an L16 experimental series was conducted which for the 5 variables yielded a resolution of 5. This means that single variable effects were confounded by only 4 factor interactions while two factor interactions were confounded by only 3 factor interactions. Valid 3 and especially 4 factor interactions are very rare and so are assumed to be zero. Thus, this experimental design allows the magnitude and sense of two factor interactions to be determined as well as single factor effects. The matrix for the test parameters and results is presented in Table 1 of the text.

In order to give the flavor of how this data is analyzed, consider the effect of titanium type which is variable D. The first 8 run numbers used titanium type D_- (low purity) while the last 8 runs used titanium type D_+ (high purity). Notice also that for the first 8 runs there are an equal number of plus and minus values for the parameters of all the other variables. Thus for these first eight runs, which all use the same type of titanium, the effect of all the other variables averages out to zero due to the balance of the plus and minus values. The same is true for the last eight runs. To determine if titanium type is an active factor with respect to target weight loss, all the weight losses corresponding to a titanium type D_- are summed and compared to the sum of target weight losses for titanium type D_+. If these two sums are approximately equal it is concluded that titanium type is not an active factor, but rather an inert factor with respect to target mass loss. However, if the sums for the D_- and D_+ tests are significantly different, then it can be concluded that titanium type is an active factor and the titanium type which yields the more desirable result can be determined. The process is similar for the other variables with the sums of the + variable value tests being compared to the sums of - variable value tests to determine the relative magnitude of the parameter's effect.

As noted, two factor interactions can also be considered in this experimental design. The analysis is similar using the contrast coefficients tabulated below.

TABLE 2
CONTRAST COEFFICIENTS FOR TWO FACTOR EFFECTS

seq #	run #	AB	AC	AD	BC	BD	CD	DE	CE	BE	AE	(gm) targ wght loss
12	1	+	+	+	+	+	+	-	-	-	-	21.85
4	2	-	-	-	+	+	+	+	+	+	-	5.46
7	3	-	+	+	-	-	+	+	+	-	+	4.60
10	4	+	-	-	-	-	+	-	-	+	+	28.83
5	5	+	-	+	-	+	-	+	-	+	+	1.91
13	6	-	+	-	-	+	-	-	+	-	+	9.26
11	7	-	-	+	+	-	-	-	+	+	-	4.80
8	8	+	+	-	+	-	-	+	-	-	-	3.86
3	9	+	+	-	+	-	-	-	+	+	+	1.81
15	10	-	-	+	+	-	-	+	-	-	+	8.43
16	11	-	+	-	-	+	-	+	-	+	-	7.41
2	12	+	-	+	-	+	-	-	+	-	-	0.52
14	13	+	-	-	-	-	+	+	+	-	-	8.08
1	14	-	+	+	-	-	+	-	-	+	-	0.00
6	15	-	-	-	+	+	+	-	-	-	+	1.39
9	16	+	+	+	+	+	+	+	+	+	+	9.04

The results of the relative magnitude of effect on target weight loss analysis for all the single factor and the most active two factor effects are tabulated below.

TABLE 3
MAGNITUDE OF EFFECT ON TARGET WEIGHT LOSS DATA

variable	Σ+	Σ-	(Σ+)-(Σ-)	rel. rank
A	65.4	51.8	+13.6	9
B	60.4	56.8	+ 3.6	12.5
C	38.4	78.8	-40.4	4
D	36.6	80.6	-44.0	2
E	97.6	19.6	+78.0	1
CD	79.2	38.0	+41.2	3
AB	75.8	41.4	+34.4	5

The $\Sigma+$ column lists the sums of target weight losses for all tests where the variable had a + level and similarly for the $\Sigma-$ column. The $(\Sigma+)-(\Sigma-)$ column lists the difference between the two previous columns where the sign indicates the variable level which yielded the more desirable (larger target weight loss) result. There are 5 single variables which can be combined to yield 10 two variable interactions for a total of 15 single and two variable combinations for which the magnitude of the effect can be determined. The relative rank column lists the rank of the variable with respect to target weight loss effect. Variable B shows a non-integer rank due to a tie in magnitude between the 12th and 13th ranks.

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