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TATB FORMULATION STUDY

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MASTER

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ABSTRACT

This report describes the small-scale production and mechanical properties of TATB/Kel-F 800 (92.5/7.5), a useful plastic bonded explosive.

TATB particles are slurried in water while adding a solvent solution of Kel-F. As the solvent is removed from the slurry at 75-80°C under vacuum, the fine particles of TATB become coated with Kel-F and clump together to form granules. After drying at 80°C the granules are pressed at 120°C and 138 MPa (20,000 psi) to form needed shapes.

Both the TATB particle size distribution and the conditions used in the slurry process must be controlled if high strength and toughness are to be achieved when the granules are molded. Important factors in the slurry process are: agitation speed, which controls the granule size; rate of solvent removal, which affects the granule surface character and slurry temperature. Also, TATB batches having a sharp bimodal particle size distribution can be molded into much tougher specimens that show less growth and creep than specimens made from TATB that has a unimodal distribution.

Preferred granules that produce the toughest moldings have diameters in the 1-3 mm range and a dull surface luster. Shiny granules or those with diameters larger than 3 mm yield pressings of inferior properties. Granules smaller than 1 mm in size are dusty and difficult to handle.

The observed tensile and compressive stress-strain properties of TATB/Kel-F molded specimens differed, depending on the testing location. Part of the observed variation stems from differences in testing conditions at LLL, Pantex and Site 300, but other factors may be important.

Other polymers tried as binders for TATB were not as suitable as Kel-F 800. Formulations with Teflon 7C were brittle and those with Estane, Viton A and Viton AHV had low strength. An Estane/Viton A/Kel-F mixture with TATB that was expected to impart the advantages of all three polymers also had low strength. LG-160 fluorolube plasticizer did not improve the strength of a TATB/Kel-F formulation.

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GLOSSARY

TATB	1,3,5 triamino-2,4,6-trinitrobenzene
Kel-F 800	Poly(trifluorochloroethylene)/vinylidene fluoride 3:1
Viton A	Hexafluoropropylene/vinylidene fluoride 1:2
Estane	A polyester polyurethane
PBX	Plastic-bonded explosive
X-0219	A PBX containing 90 wt% TATB and 10 wt% Kel-F 800
RX-03-BB	A PBX containing 92.5 wt% TATB and 7.5 wt% Kel-F 800
RX-03-AU	A PBX containing 95 wt% TATB and 5 wt% Kel-F 800
LASL	Los Alamos Scientific Laboratory, Los Alamos, New Mexico
LLL	Lawrence Livermore Laboratory, Livermore, California
Pantex	Mason and Hanger, Silas Mason Company, Inc., Pantex Plant, Amarillo, Texas

INTRODUCTION

TATB is a yellow crystalline explosive first synthesized in 1888. Interest in TATB has revived in recent years because of its high melting point of 450°C and its extreme insensitivity; these properties place it high on the list of thermally stable, safe explosives.

Initial efforts to use this explosive were frustrated by its fine particle size, which made it difficult to press into tough, high density moldings. Then a new method for TATB synthesis developed at LASL¹ provided larger diameter particles that, after formulation with a Kel-F binder (LASL product code X-2019), could be pressed into parts of satisfactory toughness.²

After TATB was chosen as the explosive for several devices at LLL, intensive work began to learn how to formulate TATB into a high quality plastic-bonded explosive and scale up its manufacture. Based on explosive energetics and sensitivity, the decision to concentrate on the composition TATB/Kel-F 800 (92.5/7.5) had already been made before this investigation began.

DEVELOPMENT OF A TATB/KEL-F 800 (92.5/7.5) MOLDING POWDER

Development of the Slurry Coating Process

TATB/Kel-F 800 molding powder is made by slurry coating particles of TATB with a solution of Kel-F 800 and recovering the dry, spherical granules.

A 20 wt% solution of Kel-F in 4/1 MIBK/n-butyl acetate is added at 50°C to an agitated 20% slurry of fine TATB particles in water containing Elvanol wetting agent. As solvent is removed at 75-80°C with continued agitation by

a vacuum air sweeping technique, the fine TATB particles are coated with Kel-F and aggregate into spherical granules. After cooling, filtering, washing and drying in a forced air oven at 80°C, the granules are ready for pressing into special shapes as needed.

Typically, PBX molding powders should be granular, free-flowing and dustless for safety and ease of handling. Initial large scale (30 kg) preparations were made in the 378-liter slurry kettle at Site 300 with slow agitation rates (50-100 rpm), and yielded an unsatisfactory fine, dusty powder. Consequently, smaller batches were made to study the variables in the process.

Work with small 50-g batches in the 600-ml slurry apparatus and 7-kg batches in the 30-liter slurry kettle at Site 300 soon demonstrated that satisfactory sized granules are formed if the initial agitation rate is 600-700 rpm. However, soon after the granules form, agitation rate should be reduced to 200-300 rpm to avoid breaking the granules into smaller pieces. Granule size was conveniently controlled at this stage; faster rates reduce granule size and slower rates increase size.

Solvent removal times must be controlled. Times in excess of 30 minutes tend to form granules that have a hard, shiny surface and do not compact uniformly when pressed. Short solvent removal times (20-30 minutes) yield dull granules that mold better and show a completely different kind of fracture when broken in compression (see Figure 1). While pressings from the shiny and dull granules have the same tensile properties, in compression the pressings molded from shiny granules break at dramatically lower strain. Table 1 contains tensile and compressive stress-strain data of six batches, identical in composition, but differing in surface appearance. Each group was made with the same TATB particle size distribution. Although we do not understand why the dull granules are more satisfactory (desirable), it is evident that surface appearance is an important property of optimum molding powders.

RX-03-BK
SHINY GRANULES

RX-03-BW
DULL GRANULES

Figure 1. Comparison of broken pressings made with dull and shiny granules.

Table 1. Comparison of compression and tensile properties of formulations with dull and shiny granules. Tests were run at Site 300 facilities.

TATB Group	Formulation RX-03-	Density-gm/cm ³	Granule Surface	Compression		Tensile	
				Max Stress-MPa	Strain at Failure-%	Max Stress-MPa	Strain at Failure-%
1	BG	1.919	shiny	21.4	2.5	9.3	0.23
1	BV	1.909	dull	20.7	5.8	8.6	0.21
2	BE	1.916	shiny	20.0	1.9	8.6	0.20
2	BU	1.912	dull	16.5	5.6	7.6	0.23
3	BK	1.914	shiny	17.9	3.8	7.9	0.21
3	BW	1.908	dull	16.5	6.3	7.2	0.20

We used the new slurry technique to rework some fine, powdery granules and also a molding powder consisting of large, disc-shaped shiny granules. In these runs the granules were reslurried in water, solvent added and the process then run as usual. Table 2 summarizes the results. Both runs produced more acceptable size granules with a corresponding improvement in compressive toughness of pressed specimens made from them. It would appear that inferior formulations can be upgraded successfully by this technique.

Table 2. Compression properties of original and reworked formulations. Tests run at LLL with short specimens.

Batch Identity-RX-03-	Granule type	Batch Size	Max Stress-MPa	Strain at Failure-%	Density-gm/cm ³
BB original	large, shiny discs	227 kg	16.9	4.0	1.908
BB reworked	small, dull spheres	50 gm	18.3	6.2	1.907
AU original	tiny, dull powdery	30 kg	15.9	3.8	1.909
AU reworked	small, dull spheres	7 kg	16.6	5.0	1.908

Table 3 is a tabulation of the stress-strain properties of molded RX-03-BB granules made in three different size batches. The new slurry technique worked when scaling up from a 7 kg to a 32 kg batch size, but molded product from a 328 kg batch was inferior, particularly in tensile properties. Granules from the 328 kg size batch were quite large (3-10 mm diameter) because the agitation rate during granule formation could not be satisfactorily controlled. Poorer properties of these 3-10 mm granules indicate strongly that high agitation rate is imperative when using the slurry technique at any batch size.

Table 3. Compression and tensile properties of different size batches of RX-03-BB. Tests were run at Site 300.

Batch Size-kg	Kettle Volume-liters	Maximum Agitation-rpm	Granule Diameter-mm	Density gm/cm ³	Compression		Tensile	
					Max Stress-MPa	Strain at Failure-%	Max Stress-MPa	Strain at Failure-%
7	30	750	1-3	1.911	16.6	3.8	7.8	0.28
32	378	360	1-3	1.903	16.7	4.5	6.9	0.20
328	1135	240	3-10	1.910	16.0	2.7	5.8	0.14

INFLUENCE OF TATB PARTICLE SIZE DISTRIBUTION ON MECHANICAL PROPERTIES

The particle size distribution of the TATB used to make TATB/Kel-F granules has a definite influence on the properties of specimens molded from those granules. Though correlation with specific percentages of particles in each of several size ranges is not yet possible, we can say that certain distribution patterns are more favorable than others.

To study the influence of TATB size, nine different lots having various particle size distribution patterns were converted to 7.5 kg batches of TATB/Kel-F 800 (92.5/7.5) in a 30 liter kettle under conditions such that the only variable was particle size. Particle size distributions were determined at Pantex using

Buckbee-Mears sieves. The granules from each batch were similar in size (1-3 mm diameter), shape (spherical) and surface texture (dull). Each molding powder was pressed isostatically at 138 MPa (20,000 psi), 120°C, vacuum, with three 5 minute dwells. Specimens for mechanical property tests were machined from a single billet of each batch. All tests were done at Site 300 (see Ref. 3).

Compressive and Tensile Properties

Results of compression and tension tests are summarized in Table 4. To understand the significance of these data, we devised a rating system based on the concept of specimen toughness. Toughness is measured by the amount of energy a specimen can absorb without fracturing. In absorbing energy, work is done on a specimen. Since a stress-strain diagram is actually a force-distance diagram, the area under the stress-strain curve is work done on the specimen and is therefore a measure of its toughness. A toughness rating system such as we devised is reasonable in applications where an explosive is required to survive large shocks without detonation and maintain its original conformation.

The difference between toughness in compression and in tension is large. Figure 2 shows that the area under the compressive stress-strain curve of a typical TATB/Kel-F specimen is nearly 100 times greater than the area under the tensile curve.

Figure 2 . Compression and tensile stress-strain diagrams of RX-03-BW.

Table 4. Compression and tensile properties of formulations made with various TATB particle size distributions. Tests were run at Site 300.

Formulation- RX-03-	TATB particle size distribution	Pressed density- gm/cm ³	Compression		Tensile	
			Max stress- MPa	Strain at failure-%	Max stress- MPa	Strain at failure-%
BV		1.909	20.7	5.8	8.6	0.21
BH		1.908	20.0	4.4	8.8	0.23
BU		1.912	16.5	5.6	7.6	0.23
BF		1.918	17.6	5.9	7.8	0.20
BW		1.908	16.5	6.3	7.2	0.20
BI		1.909	17.2	6.3	7.1	0.17
BD		1.909	15.5	5.8	7.0	0.20
BL		1.908	17.2	4.6	7.4	0.18
BJ		1.908	15.2	4.8	5.2	0.12

In this evaluation we decided that the toughness in both compression and tension are equally important, and that we would assign them the same weight on the rating scale. For compression ratings, RX-03-BV had the highest toughness (103); it was assigned 50 points. We rated the other formulations by multiplying their toughness by the factor 50/103. RX-03-BH had the highest toughness in tension (1.19). It was assigned 50 points and the other formulations were rated by multiplying their toughness in tension by the factor 50/1.19. The sum of the ratings in tension and compression was the overall rating for each formulation. These are listed in Table 5.

TATB exhibiting a sharp bimodal particle size distribution yielded the toughest PBX specimens (RX-03-BV). Formulations with sharply peaked, unimodal distributions (BD, BJ) are at the low end of the scale. The characteristic distributions used in formulations BI and BL are also less desirable. The variety of TATB distributions used in formulations BH, BU, BF and BW have nearly the same ratings. In view of the significantly higher rating of RX-03-BV, the pattern of TATB particle size distribution used in this formulation is the most desirable.

Compressive Creep

Ideally, a PBX should not deform (creep) farther with time after an initial deformation, but all TATB/Kel-F specimens tested so far do creep.

Compressive creep tests (Figures 3 and 4) show that all formulations had low creep rates after large initial deformations. The batch with by far the greatest deformation (BD) was made from TATB having a sharply peaked, unimodal particle size distribution with a high proportion of fines. The formulations with the least deformation and creep (BV, BG) were made with a sharp, bimodal TATB particle size distribution. The curves in Figure 4 also show that the surface texture has no effect on the creep properties; each group was made with the same TATB particle size distribution.

Table 5. Toughness ratings of formulations made with various TATB particle size distributions.

Formulation- RX-03-	TATB particle size distribution	Compression		Tensile		Total rating
		Toughness	Rating	Toughness	Rating	
BV		103	50	1.12	47	97
BH		73	35	1.19	50	85
BU		81	39	1.06	45	84
BF		87	42	0.97	41	83
BW		89	43	0.86	36	79
BI		92	44	0.65	27	71
BD		72	35	0.79	33	68
BL		68	33	0.78	33	66
BJ		61	30	0.36	15	45

Figure 3. Compressive creep of various RX-03 type formulations at 74°C and 0.69 MPa stress. All granules are dull.

Figure 4. Compressive creep of various RX-03 type formulations at 74°C and 0.69 MPa stress.

Growth

Growth rates (progressive increase in size) were determined by thermal cycling TATB/Kel-F molded specimens five times according to the programmed temperature cycle shown in Figure 5, and measuring the axial lengths at 20°C after each cycle.

The data are plotted in Figure 6. The formulations with the slowest growth rates (BV, BG) contained TATB with a sharp bimodal distribution. Growth rates of the other formulations were only slightly faster. Growth rates did not correlate with the granule surface texture (dull or shiny); each group was made with the same TATB particle size distribution.

Figure 5 . Programmed temperature cycle.

Figure 6. Axial growth of various RX-03 type formulations.

Figure 7 . Linear coefficient of thermal expansion of various RX-03 type formulations.

Coefficient of Thermal Expansion

Coefficients of thermal expansion (CTE) were obtained by measuring the change in the axis length of each growth rate specimen during the final programmed temperature cycle. The results plotted in Figure 7 show a linear relationship between CTE and temperature, with a change in the slope occurring between -10° and 0°C . Neither the TATB particle size nor the surface texture of the granules correlate with differences in CTE; each group was made with the same TATB particle size distribution.

SELECTION OF A BINDER FOR TATB

Formulations with Other Binders

In the early stages of this investigation, we made a cursory study of TATB formulations with various binders and combinations of binders. Toughness in compression was the criterion of success.

Table 6 summarizes the results obtained with the various binders. Formulations RX-03-AY and AZ were 7 kg batches prepared at Pantex, but the other formulations were 50 g samples prepared at LLL.

Judged by their compressive toughness, none of these formulations was as satisfactory as TATB/Kel-F 800 (formulation RX-03-BB). Teflon 7C dry mixed with TATB for several hours (BN) was not homogeneous; pressings were very brittle like pressings of pure TATB. Adding Kel-F 800 plasticized with fluorolube did not improve toughness (compare formulations RX-03-BB with BR and BS). Also, no improvement occurred when the binder was a mixture of Kel-F 800 and Viton A (B0). However, high viscosity Viton A (BQ) did yield a tougher material than standard Viton A (AZ).

Estane binder produced a tough, but fairly soft, PBX (AY). A mixture of Estane, Viton A and Kel-F 800 was tried with the idea of combining the best features of all three binders, but the resulting product had low strength.

Table 6. Compression properties of formulations with various binders.
(Tests at LLL using short specimens.)

Batch Identity- RX-03-	Binder Description	wt% Binder	Pressed Density- gm/cm ³	Max Stress MPa	Strain at Failure-%
--	NONE	0	1.870	12-19	1-2
BN	Teflon 7C	7.5	1.905	19.9	1.4
BB	Kel-F 800	7.5	1.902	20.0	5.0
BR	Kel-F 800 with 6.7% MO-10 fluorolube plasticizer	7.5	1.917	17.9	5.0
BS	Kel-F 800 with 6.7% LG-160 fluorolube plasticizer	7.5	1.917	17.3	5.0
BO	50/50 Kel-F 800/Viton A	7.5	1.897	12.0	6.0
AZ	Viton A	9.0	1.904	7.6	5.6
BQ	Viton AHV (high viscosity)	9.0	1.894	10.3	8.4
AY	Estane 5702 F-1	6.0	1.848	12.7	11.0
BP	1/3 Kel-F 800 1/3 Viton A 1/3 Estane 5702 F-1	7.5	1.875	11.4	8.0

Comparison of Different Lots of Kel-F 800 Binders

Four different lots of Kel-F 800 were formulated with two different lots of TATB in 50 gm batches. The test results from Table 7 show no differences in compressive strength between any of the binders when formulated with the same TATB.

Table 7. Compression test results of TATB formulations using different lots of Kel-F 800 binder. Tests were run at LLL with short specimens.

TATB/Kel-F 800- wt%	Kel-F 800 Lot No.	TATB Lot No.	Pressed Density- gm/cm ³	Max Stress- MPa	Strain at Failure-%
90/10	547	B 209	1.908	22.4	5.2
90/10	552	B 209	1.907	22.4	5.4
92.5/7.5	545	B 216	1.901	20.0	5.0
92.5/7.5	548	B 216	1.901	20.3	5.0

Table 8. Comparison of testing methods at various facilities.

Test	Test Facility	Specimen size, Length x Diam - cm	Temp- °C	Test Method
Compression	LLL (s)	2.54 x 2.54	22	Constant crosshead speed of 0.02 cm/min
	LLL (l)	5.08 x 2.02	22	Constant crosshead speed of 0.02 cm/min
	Site 300	5.08 x 2.02	20	Constant strain rate of 0.1%/min ^(a)
	Pantex	6.35 x 2.02	21	Constant crosshead speed of 0.005 cm/min
Tension	LLL	7.62 x 1.27	22	Constant crosshead speed of 0.005 cm/min
	Site 300	12.70 x 2.54	20	Constant strain rate of 0.1%/min ^(a)
	Pantex	12.70 x 2.02	21	Constant crosshead speed of 0.0127 cm/min

(s) short specimen

(l) long specimen

(a) equivalent to 0.0025 cm/min

COMPARISON OF STRESS-STRAIN TESTING AND ANALYTICAL TECHNIQUES AT LLL, PANTEX AND SITE 300

Stress-Strain Testing

A variety of testing techniques is currently in use at LLL, Pantex and Site 300 to determine the stress-strain properties of RX-03-type formulations. Specimen size and test method, or both, are different at the three locations. (See Table 8.)

Despite these differences, tensile stress-strain data from the three locations agreed rather well. (See Table 9.) Compressive stress values for the same batch of PBX were independent of the test method (see Table 10), but compressive strain data (and therefore the apparent toughness of the specimens) varied widely, depending on the laboratory. The constant strain rate method used at Site 300 gives significantly lower results than the methods that use constant crosshead speed. At present there is no way of selecting any one test method as right or wrong.

Table 9. Comparison of tension test results at various facilities.

RX-03-BB Batch Identity, Size	Test Facility	Density-gm/cm ³	Maximum Stress-MPa	Strain at Failure-%
Site 300 No. 75-106, 32 kg	LLL	1.903	6.0	0.27
	Site 300	1.903	6.9	0.20
Pantex No. 5239-145-01, 7 kg	LLL	1.911	6.9	0.27
	Site 300	1.911	7.8	0.28
	Pantex	1.906	7.1	0.22
Pantex No. 5240-145-01, 328 kg	LLL	1.910	4.7	0.11
	Site 300	1.910	5.8	0.14
	Pantex	1.903	5.0	0.09

Table 10. Comparison of compression test results at various facilities.

RX-03-BB Batch Identity, Size	Test Facility	Density-gm/cm ³	Maximum Stress-MPa	Strain at Failure-%
Site 300 No. 75-106, 32 kg	LLL (s)	1.902	18.6	6.0
	LLL (1)	1.903	17.7	8.8
	Site 300	1.903	16.7	4.5
Pantex No. 5239-145-01, 7 kg	LLL (s)	1.914	18.6	5.6
	LLL (1)	1.911	17.9	8.0
	Site 300	1.911	16.6	3.8
	Pantex	1.906	17.7	7.1
Pantex No. 5240-145-01, 328 kg	LLL (s)	1.914	18.6	6.4
	LLL (1)	1.910	17.6	7.0
	Site 300	1.910	16.0	2.7
	Pantex	1.903	17.2	5.5

Comparison of Analytical Techniques at LLL and Pantex

Pantex determines the composition of TATB/Kel-F 800 formulations by extracting the Kel-F binder with a solvent. At LLL, the technique involves digesting the TATB with nitric acid. The results of both methods agree, as shown in Table 11.

Table 11. Comparison of RX-03-BB analysis at LLL and Pantex.

Batch Identity, Size	Facility	TATB-wt%	Kel-F 800-wt%	Method
Pantex No. 5239-145-01, 7 kg	Pantex	92.4	7.6	Solvent extraction
	LLL	92.4	7.6	Acid digestion
Pantex No. 5240-145-01	Pantex	92.6	7.4	Solvent extraction
	LLL	92.6	7.4	Acid digestion

MECHANICAL PROPERTIES OF 100% TATB SPECIMENS

The mechanical properties of pure TATB pressings were studied because of the current interest in using pure TATB as initiators. Four TATB lots with different particle size distributions were pressed at three different densities (1.70, 1.80, 1.87 gm/cm³). The compression test results plotted in Figure 8 show that TATB (B-210), composed mostly of fine particles, is weaker than the others. In all cases, higher density pressings are more brittle.

Figure 8. Density versus stress and strain of pure TATB with various particle size distributions. Compression tests were run at LLL with short specimens.

SUGGESTIONS FOR FUTURE WORK

We are conducting a study of the surfaces of dull and shiny granules using the electron spectroscopy for chemical analysis (ESCA) method. A better understanding of the differences in these surfaces is necessary to continue development work on process techniques. Continued studies of particle size distributions are necessary to establish a TATB manufacturing specification that will result in a PBX with consistent and optimum mechanical properties. A standardization of the mechanical properties testing methods at Pantex and LLL facilities would allow direct comparisons of results, and eliminate rerunning tests on the same materials.

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