MASTER

TATB FORMULATION PROCESSES

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DEVELOPMENT DIVISION

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ABSTRACT

The effect of several processing parameters were evaluated. Included were TATB particle size, processing time, batch concentration, binder treatment, granule size and others. Batch concentration and granule size emerged as the most important factors affecting mechanical response.

INTRODUCTION

TATB (1,3,5-triamino-2,4,6-trinitrobenzene) coated with Kel-F 800 elastomer constitutes the plastic bonded explosives (PBX), which varies in Kel-F content from 5 to 10% by weight.

Pantex Plant became involved in the development of formulation processes for the TATB PBX in support of ERDA activities to develop an insensitive explosive. Batches of the PBX have covered a wide range of sizes from a few grams to 122 kg, depending on requirements, and blended lot sizes have approached 500 kg.

Kel-F 800^a (a chlorinated-fluorinated hydrocarbon - a co-polymer of trifluorochloroethylene and vinylidene fluoride) was one among three primary candidates as the binder, the other two being Estane^b (a low density polyurethane), and Viton^c (a co-polymer of perfluoropropylene and vinylidene fluoride). The Kel-F 800 was preferred because the physical properties of the PBX are more consistent throughout the temperature range; Kel-F does not become as brittle at the lower temperatures as Estane and Viton.

PROCEDURE

TATB PBX is produced by varying slurry process parameters, then physical properties data are generated as the evaluation criteria. Certain physical properties are quite responsive to the manner in which the TATB PBX is made.

^a 3M Corp.

^b B. F. Goodrich

^c DuPont

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SLURRY PROCESS

Basically the slurry process is the coating of the TATB powder in a water slurry. A binary solvent system of four parts MIBK (methylisobutyl ketone) and one part NBA (normal butyl acetate) is used to dissolve 20% by weight of Kel-F 800 elastomer to form a lacquer. The lacquer is added to the slurry and as heat is applied and the solvent evaporated, granules suitable for pressing are formed.

PHYSICAL PROPERTIES TESTING

The normal physical properties test consists of 3 to 5 each of tensile and compression specimens. The tensile specimens have the configuration shown in Fig. 1. The compression specimen is a right cylinder, 2.03 cm (0.798 in) diameter by 6.35 cm (2.5 in) high. An Instron Universal Test Instrument is used to test the specimens.

Tensile results are reported via a normal stress/strain curve. Compression results are handled somewhat differently, as shown in Fig. 2. After the initial fracture of the specimen, stress continues to be applied until the ultimate failure of the specimen. The most desirable property for both compression and tensile is the maximum strain. Continuing the stress until complete failure of the specimen on the compression test aids in distinguishing differences between materials.

DISCUSSION

GRANULE SIZE

Very early during the initial experience of making TATB PBX at Pantex, before mechanical properties could be tested, the goal was to produce from the slurry process, large uniform granules of the PBX for pressing. During the physical properties testing, as data began to be generated, a difference began to be apparent in the way the test samples broke. The test samples with the poorest strain, like that shown in Fig. 2, broke along the molding powder granule or bead interfacing, while breakage occurred across granules for the test samples with the higher strain. This seemed to be, at least in part, a characteristic of the granule size; the larger granule material have the lower strain values. This is illustrated in the photographs shown in Fig. 3.

As more testing was done and more physical properties data became available the trend toward decreased strength with increased granule size became clearer (shown in Fig. 4). The weighted mean granule size is plotted against work to rupture, which is defined as the area under the compression strength curves. This was done for the 7.5% and 10% Kel-F 800 materials. As granule size increases the strength decreases. All of the data shown on the 10% Kel-F 800 curve were obtained from recycled materials, that is material which has been processed, pressed, machined and then regranulated by the slurry process. A program is currently under way at Pantex to develop a procedure which will yield acceptable physical properties starting with reclaimed PBX or machine scrap. After the

undesirable effect of large granules on physical properties became obvious the procedures were modified and small uniform granules have been produced for all subsequent batches.

PROCESSING PARAMETER STUDY (30-LITRE REACTOR)

The purpose of this series of experimental studies was to more precisely identify other major contributing factors affecting physical strength. Those factors suspected of influencing physical properties were included for examination. They are; (1) batch concentration, (2) processing time, (3) Kel-F lots, (4) TATB particle size, and (5) a miscellaneous category; results are summarized in Table I.

For practical considerations this study was conducted in the 30-litre reactor. Unless otherwise stated, the processing parameters and materials conditions were as follows:

Vessel - 30-litre reactor

TATB Lot 5123-135-01

Kel-F 800 - Lot 552 - precipitated from acetone

Batch Size - 1.8 kg batch, concentration-96 g of HE per litre of water

Processing Time - 1 hr

Test conditions for physical properties measurements were:

Compression - 0.001 mm/sec, constant crosshead speed

Tensile - 0.002 mm/sec, constant crosshead speed

A pressing size of 3.4 kg was chosen because of variations in physical properties observed between specimens made from small 2-7 kg pressings and larger 100 kg pressings. Pressed density, generally speaking, has been on the order of 1.900 Mg/m³. Regardless of the manner in which the PBX was processed, densities are approximately the same. Some pressings, however, were made initially at four specific compaction pressures. The trend toward increased density with increased pressure is shown in Fig. 5. The density range at each pressure is probably caused by variations of preheat temperatures or number of cycles used in pressing the material, or perhaps variations of press loading time.

BATCH CONCENTRATION

Physical strength data for the batch concentration experiments are shown in Figs. 6 and 7. Fig. 8 shows examples of specimen breakage. Notice that the specimen made at the higher batch concentration failed at the granule interface although the granules are small. Maximum stress and strain at complete failure are shown as functions of batch concentration in Fig. 9. Results obtained from material made in the 300-gallon kettle are also included for comparison. Batch concentrations of 100 to 140 grams of TATB per litre of water produced near optimum physical properties.

PROCESSING TIME

The effects of processing time are shown in Figs. 10 and 11 and summarized in Fig. 12. Previous work indicated that the length of time the PBX was in the kettle affected physical properties, i.e., the longer the batch remained in the kettle after the stripping of the solvent began, the poorer the properties. Therefore, this series was run for 1 hour to 3 hours. The compression data show a loss in strain at 3 hours; however, the tensile data do not show a clear difference. It is concluded from these results that a processing time of 1.5 to 2 hours is satisfactory.

COMPARISON OF KEL-F LOTS

The effects of Kel-F purity are given in Figs. 13 and 14. The Kel-F 800 was subjected to a pretreatment for purification. The Kel-F 800 was dissolved in acetone and precipitated into water prior to being made into a lacquer. Removal of the manufacturer's emulsifier by this technique aided in obtaining the exact composition but did not result in improved physical properties compared with Kel-F as received from the manufacturer. This indicates that there is no real physical property advantage in the use of purified Kel-F 800.

TATB PARTICLE SIZE

Traditionally the properties of a PBX are expected to be significantly affected and altered by the particle size distribution of the crystalline explosive. Previous attempts to correlate the TATB particle size to the physical properties of the finished TATB PBX have not been definitive. Therefore, three batches of TATB PBX were made from different TATB particle size distributions and tested for physical strength. Three small lots of TATB, which were categorized as having normal, bi-modal, and fine particle size distributions, were used to make TATB PBX. The physical strength of the PBX did not appear to be significantly altered by the different particle size distributions. Tensile and compression data of all three lots were comparable with the exception of compression data from the batch made with the fine particle size TATB which gave a lower strain-at-rupture. Tensile stress/strain curves are shown in Fig. 15 and compression curves are shown in Fig. 16. The TATB particle size distributions are shown in Fig. 17.

MISCELLANEOUS

A formulation with 10% Kel-F have higher tensile and compression strain values than the 7.5% Kel-F batches. Data for these batches are presented in Figs. 18 and 19. Other variables investigated which appeared not to affect physical strength were (1) treating the TATB crystals with a surface active agent, the polysaccharide MRL 22A, prior to making the TATB into PBX, (2) the use of a non-porous TATB crystal which was synthesized by a water amination process, and (3) the use of acid washed TATB.

SUMMARY

Since the beginning of the production of TATB PBX, there has been a large degree of variability in the physical properties of the pressed material. It became apparent very early that much of the variability in physical properties could be attributed to PBX processing or manufacturing techniques. The first parameter found to significantly affect physical properties was the granule size of the molding powder. This was determined by viewing the manner in which the physical test specimen broke—breakage was along the PBX granule boundaries. Then a series of five processing studies were conducted in the 30-litre reactor to more clearly define the problem areas. Batch concentration emerged as the most important single variable affecting physical properties.

CONCLUSIONS

The experimental studies conducted on a small scale have a high degree of relevance to the large scale production vessels. The findings from the 30-litre reactor studies have been incorporated into the processing procedures used for making the large 122 kg batches in the 300-gallon kettle. Using the Pantex methods, the latest 8 to 10 successive large lots of PBX have been produced with consistently good physical properties.

Table I. TATB Physical Properties

Lot No.	Process Description	Compression		Work to Rupture (kJ/m ³)		Tension	
		Stress (MPa)	Strain (%)	Compression	Tensile	Stress (MPa)	Strain (%)
BATCH CONCENTRATION							
5196-03	1.8 kg/96 g/l ^a	18.38	6.2	978.8	12.9	8.45	0.26
5190-03	2.7 kg/132 g/l	18.24	5.8	923.6	18.2	8.76	0.32
5196-04	3.6 kg/180 g/l	18.13	4.9	726.4	9.6	7.38	0.18
5192	5.4 kg/276 g/l	18.61	1.9	270.4	11.3	7.94	0.25
5191	7.3 kg/360 g/l	17.40	2.0	269.8	7.5	7.05	0.19
PROCESSING TIME							
5176-03	1.0 hr ^b	16.70	7.0	996.1	12.3	7.78	0.25
5196-03	1.5 hr ^a	18.30	6.2	1009.1	14.4	8.45	0.26
5197-03	2.0 hr	16.97	7.6	1102.9	18.5	8.73	0.33
5205-03	3.0 hr	18.60	5.2	863.9	15.1	8.73	0.29
COMPARISON OF KEL-F							
5206-05	552 Plain	17.91	7.1	1064.3	17.4	8.07	0.30
5176-03	552 Purified ^b	16.70	7.0	996.1	12.3	7.78	0.25
5210-03	553 Purified	17.73	7.6	1102.3	11.7	8.40	0.22
5210-06	554 Purified	17.04	6.5	923.4	11.9	7.94	0.23
TATB PARTICLE SIZE							
5311-03	Bimodal (5226-16-01) ^c	18.92	5.68	-	-	8.51	0.24
5314-03	Normal (5260-16-01) ^d	19.40	5.89	-	-	8.54	0.25
5316-03	Fine (5191-16-01) ^e	18.99	4.10	-	-	7.95	0.23
MISCELLANEOUS							
5176-03	Control ^b	16.70	7.0	996.1	12.3	7.78	0.25
5211-03	TATB/MRL 22A	17.81	7.6	1120.7	10.8	8.02	0.20
5216-06	Water-Aminated TATB	18.21	6.2	1004.1	11.4	7.85	0.22
5211	10% Kel-F 800	16.38	8.6	1174.5	23.3	8.23	0.40
5233-03	Acid Washed TATB	17.27	6.2	955.6	13.6	7.60	0.27

^aSame batch^bSame batch - control^c(% < 40 μ - 55.74, % < 20 μ - 34.39)^d(% < 40 μ - 47.72, % < 20 μ - 8.44)^e(% < 40 μ - 93.34, % < 20 μ - 35.76)

NOTE: Unless otherwise stated conditions were: 30-litre reactor. LLL B6 TATB: purified Kel-F Lot 552: 1.8 kg (4 lb) batch size. 1 hr processing time. 96 g HE/litre water (0.8 lb HE/gal water), 3.4 kg (7.5 lb) pressing.

FIG. 1. TENSILE SPECIMEN

FIG. 2. COMPRESSION CURVES

COMPRESSION SPECIMEN

*BREAK AT GRANULE INTERFACE
WITH LARGE GRANULES,
STRENGTH POOR*

COMPRESSION SPECIMEN

*BREAK WITH SMALL GRANULES,
STRENGTH GOOD*

WORK TO RUPTURE
MJ/M³

STRENGTH DECREASE WITH LARGER GRANULES

FIG. 4

FIG. 5. PRESSED TATB PBX DENSITY INCREASE WITH PRESSURE

RX-03-BB BATCH CONCENTRATION COMPRESSION TESTS

FIG. 6

RX-03-BB BATCH CONCENTRATION - TENSILE TEST

FIG. 7

No. 5190-03
132g/litre

No. 5191-01
360g/litre

Compression Specimen

Broken Test Specimen
FIG. 8

FIG. 9. RX-03-BB - MAXIMUM STRESS & STRAIN at FAILURE

RX-03-BB PROCESSING TIME - COMPRESSION TEST

FIG. 10

RX-03-BB PROCESSING TIME - TENSILE TEST

FIG. 11

RX-03-BB - MAXIMUM STRESS & STRAIN at FAILURE
FIG. 12

RX-03-BB KEL-F COMPARISON - COMPRESSION TEST

FIG. 13

RX-03-BB KEL-F COMPARISON - TENSILE TEST
FIG. 14

Fig. 15. Tensile Tests - RX-03-BB TATB Particle Size Experiments

Fig. 16. Compression Test - RX-03-BB TATB Particle Size Experiments

Fig. 17. TATB Particle Size Distribution

FIG. 18. COMPRESSION TEST

FIG. 19. TENSILE TESTS