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AUGUST 1981

Process Development
Endeavor No. 208

Lawrence Livermore National Laboratory
Livermore, California
SANL 905-002

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ABSTRACT

Six production lots of low-chlorine TATB powder have been evaluated. Five of the lots were made by a wet-amination process and the other was made by an emulsion-amination process. LX-17 physical properties specimens made from the five wet-aminated TATB lots were exceptionally strong and comparable to dry-aminated TATB, while the emulsion-aminated lot of TATB had lower strength.

INTRODUCTION

TATB with a low chlorine content (0.1% or less) is advantageous from a long-term compatibility standpoint. In the past, the small batches of low chlorine TATB powders made by both wet-amination and emulsion-amination processes produced much weaker plastic-bonded explosives than dry-aminated TATB powders which have a higher chlorine content. In this study, low chloride TATB from Hercules, Inc. production size batches were evaluated in LX-17, 92.5% TATB/7.5% Kel-F.

SYNTHESIS

A total of six low chlorine TATB batches were synthesized by Hercules, Inc. using a wet-amination process. In this process, a quantity of water is added to the toluene amination solvent to dissolve ammonium chloride, a by-product of the amination reaction. In the emulsion-amination process, an emulsifier is also added to the amination solvent. A summary of the synthesis parameters is included in Table I. The first batch, identified as BX-1, was made with no emulsifier added at a five volume percent water level. Batches BX-2, BX-4, BX-5, and BX-6, were made at the ten volume percent water level with no emulsifier added. The third batch, BX-3, was also made at ten volume percent water, but with emulsifier, Atlox 50/50, added. All batches were made at a constant ammonia flow rate of 6.05 kg $NH_3/(m^2)(hr)$. Agitator speed and vessel temperature were held constant at 125 rpm and 125 C, respectively, for all six batches. BX-1, BX-2, BX-3, and BX-4 were made at a TCTNB concentration of 0.091 kg TCTNB/litre toluene, while BX-5 and BX-6 were made at a TCTNB concentration of 0.15 kg TCTNB/litre toluene. Yields were not as high as expected for BX-1, BX-3, and BX-5, while they were slightly higher than expected for BX-2, BX-4 and BX-6.

Table I. Synthesis Parameters for Hercules Wet-Aminated TATB[a].

TATB Designation	Hercules TATB Lot No. 12-11-80-	Volume Water in Toluene (%)	TCTNB Concentration kg TCTNB / ℓ Toluene	Crude TCTNB[b] Used (kg)	TATB Yield (kg)
BX-1	0930-042	5	0.091	226.2	125.2
BX-2	0926-041	10	0.091	212.0	142.9
BX-3	1001-043	10[c]	0.091	212.0	107.5
BX-4	1215-070	10	0.091	217.4	142.4
BX-5	1216-071	10	0.150	358.3	219.1
BX-6	1219-073	10	0.150	358.3	236.3

[a]*Produced at a constant agitator speed of 125 rpm, a constant temperature of 125 C, and a constant ammonia flow rate of 6.05 kg* $NH_3/(m^2)(hr)$.

[b]*Crude TCTNB purity ∿ 85% by weight.*

[c]*Emulsifier, Atlox 50/50, 0.286% by volume in amination solvent.*

The TATB batches identified as BX-4, BX-5, and BX-6 were determined to have too high a chlorine content as they originally came into the plant so they were rewashed before formulation into LX-17. This process consisted of slurrying the TATB in 80 C water for one hour, filtering, and steaming in the filter press for a minimum of thirty minutes. After rewashing, the TATB batches were less than 0.1% in total chlorine content. Each of the six TATB batches were analyzed for moisture content, particle size, and purity before formulation into LX-17. The results of these analyses are listed in Table II.

Table II. Analysis of Hercules Wet-Aminated TATB Powder

Designation	Water in Amination Process (%)	% Cl⁻ Total	% Cl⁻ Inorganic	DMSO Solubles (Wt. %)	Ash (%)	Purity (%)	Fe (ppm)	Karl Fischer Water (%)	Volatiles (%)	BET Surface Area (m^2/g)	Zeiss Mean Length (μm)	Sieve Analysis (Wt. %) <44 μm	<20 μm	<10 μm	Median (μm)
BX-1	5	0.16	0.08	0.25	0.001	98.79	31	0.150	0.042	0.528	45.95	58	21	6	40
BX-2	10	0.07	0.02	0.33	0.001	98.92	52	0.234	0.050	0.710	40.85	65	23	14	36
BX-3[a]	10	0.14	0.02	0.32	0.005	98.33	35	0.120	0.059	0.328	86.65	18	8	6	74
BX-4	10	0.07	0.03	0.11	0.036	99.00+[c]	--	0.079	0.006	0.687	--	64	34	10	36
BX-5	10	0.07	0.02	0.38	0.008	99.00+[c]	--	0.066	0.036	0.692	--	69	38	16	34
BX-6	10	0.09	0.04	0.22	0.006	99.00+[c]	--	0.093	0.054	0.662	--	51	34	15	43
B-34[b]	0	0.53	0.30	0.34	0.002	99.29	37	0.040	0.007	0.492	--	42	18	6	50

[a] *Emulsifier added*
[b] *Blended dry-aminated TATB*
[c] *Preliminary results*

FORMULATION

Each TATB batch was formulated into LX-17 by a modified straight slurry process. The TATB/water slurry was adjusted to a pH of 9.0 or greater by adding small quantities of concentrated ammonium hydroxide (NH_4OH) to the slurry. Preliminary results from another study indicate that superior physical properties are obtained on formulating LX-17 in a basic pH slurry. LX-17 batches 1023-145-01 and 1028-145-01, made from Hercules TATB batches BX-4 and BX-5, respectively, required rework to raise the binder content to the required level. These batches also required additional solvent in the formulation process to produce granules. The reworked batches were designated 1037-145-01 (original batch No. 1023-145-01) and 1041-145-01 (original batch No. 1028-145-01). The LX-17 batches were subjected to gap testing, drop hammer impact sensitivity testing, the Chemical Reactivity Test (CRT), Thermal Gravimetric Analysis (TGA), Henkin time-to-explosion, Differential Thermal Analysis (DTA), and analyzed for moisture content, composition, and particle size by sieve analysis. Physical properties specimens were fabricated from each LX-17 batch and subjected to the various physical properties tests for evaluation.

DISCUSSION

The results of the gap test, drop hammer impact sensitivity test, and CRT are listed in Table III. The moisture analysis, compositional analysis, and sieve analysis results are listed in Table IV. The physical properties test results for each LX-17 batch are summarized in Table V. Comparable results for Lot 0340-145-01, made from LLNL Blend B-34, Hercules-produced dry-aminated TATB, are included in each of these tables for comparison.

Sieve analysis results of the TATB before and after formulation into LX-17 are presented graphically in Figs. 1 through 5 for the five wet-aminated TATB batches. The sieve analysis results for the emulsion-aminated TATB before and after formulation into LX-17 are presented in Fig. 6.

The physical properties test results are presented graphically in Figs. 7 through 12. The diametric disc test curves for the LX-17 batches made from BX-1, BX-2, and BX-3 are compared in Fig. 7, while the LX-17 batches made from BX-4, BX-5, and BX-6 are compared in Fig. 8. The tensile test curves for the LX-17 made from BX-1, BX-2, and BX-3 are shown in Fig. 9, while those of the LX-17 made from BX-4, BX-5, and BX-6 are shown in Fig. 10. The compression test curves for the LX-17 made from BX-1, BX-2, and BX-3 are compared in Fig. 11 and those for the LX-17 made from BX-4, BX-5, and BX-6 are given in Fig. 12. Curves for LX-17 made from LLNL B-34, which was dry-aminated TATB, are included in Figs. 7, 9, and 11 for comparison.

Typical Henkin test results for the first three LX-17 batches are shown in Fig. 13. Typical TGA test results are compared in Fig. 14. A typical DTA thermogram for the LX-17 made from the wet-aminated TATB is presented in Fig. 15. The DTA thermogram for the LX-17 made from the emulsion-aminated TATB batch is presented in Fig. 16.

Table III. Gap, Impact Sensitivity and CRT Results for LX-17 Made from Hercules Wet-Aminated TATB

LX-17 Batch No.	TATB Powder	Gap Sensitivity Density (Mg/m³)	Gap Sensitivity G_{50} (mm)	Drop Hammer Impact Sensitivity (cm)	CRT N_2	CRT O_2	CRT CO	CRT NO	CRT CO_2	CRT N_2O	CRT Total[a]
0312-145-01	BX-1	1.917	1.24	3 No Go's @ 200	0.007	0.001	-	-	0.001	-	0.009
0311-145-01	BX-2	1.914	1.35	3 No Go's @ 200	0.014	0.001	-	-	0.004	T	0.019
0315-145-01	BX-3[b]	1.923	0.89	3 No Go's @ 200	0.010	0.001	-	-	0.003	T	0.014
1037-145-01	BX-4	1.907	1.96	3 No Go's @ 200	-	-	-	-	-	-	-
1041-145-01	BX-5	1.906	1.83	3 No Go's @ 200	-	-	-	-	-	-	-
1029-145-01	BX-6	1.906	1.96	3 No Go's @ 200	-	-	-	-	-	-	-
0340-145-01	B-34[c]	1.897	1.92	3 No Go's @ 200	-	-	-	-	-	-	-

[a] *mℓ gas evolved from 0.25-g sample after 22 hours at 120 C.*

[b] *Emulsifier added.*

[c] *Dry aminated.*

Table IV. Analysis of LX-17 Lots Made From Hercules Wet-Aminated TATB

TATB Powder	LX-17 Lot No.	Karl Fischer Water Wt. %	Volatiles (%)	FLAG @ 120 C (% H_2O)	Compositional Analysis Wt. % Kel-F 800	Sieve Analysis (Wt. %)			
						<44 μm	<20 μm	<10 μm	Median (μm)
BX-1	0312-145-01	0.149	0.074	0.003	7.41 ± 0.23	72	31	9	30
BX-2	0311-145-01	0.150	0.050	0.005	7.19 ± 0.08	80	35	24	32
BX-3[a]	0315-145-01	0.138	0.056	0.003	7.08 ± 0.04	21	11	7	68
BX-4	1037-145-01	0.119	0.02	0.012	7.49 ± 0.07	84	55	31	18
BX-5	1041-145-01	0.102	0.02	0.011	7.61 ± 0.10	81	48	32	23
BX-6	1029-145-01	0.064	0.01	0.003	7.27 ± 0.13	81	57	34	17
B-34[b]	0340-145-01	0.130	0.010	-	7.43 ± 0.11	41	18	6	-

[a]*Emulsifier added*
[b]*Dry aminated*

Table V. Physical Properties of LX-17 Made from Hercules Wet-Aminated TATB

LX-17 Batch No.	TATB Powder	Diametric Disc Ultimate Stress (MPa)	Diametric Disc Ultimate Strain (%)	Tensile Ultimate Stress (MPa)	Tensile Ultimate Strain (%)	Compression Ultimate Stress (MPa)	Compression Ultimate Strain (%)	Compression Termination[a] Strain (%)	Compression Specimen Density (Mg/m^3)
0312-145-01	BX-1	4.15	0.39	9.11	0.38	21.57	1.84	7.87	1.910
0311-145-01	BX-2	4.32	0.36	10.73	0.38	23.42	2.24	7.44	1.908
0315-145-01	BX-3[b]	2.61	0.26	5.96	0.28	15.39	1.76	8.77	1.918
1037-145-01	BX-4	4.80	0.44	10.29	0.41	24.29	2.92	6.83	1.907
1041-145-01	BX-5	4.48	0.43	9.42	0.39	23.73	2.73	5.30	1.909
1029-145-01	BX-6	4.78	0.37	10.48	0.38	24.75	2.31	7.64	1.904
0340-145-01	B-34[c]	4.57	0.33	9.14	0.40	22.09	1.69	5.43	1.896

[a] *Terminated at different percents of ultimate stress.*

[b] *Emulsifier added.*

[c] *Dry aminated.*

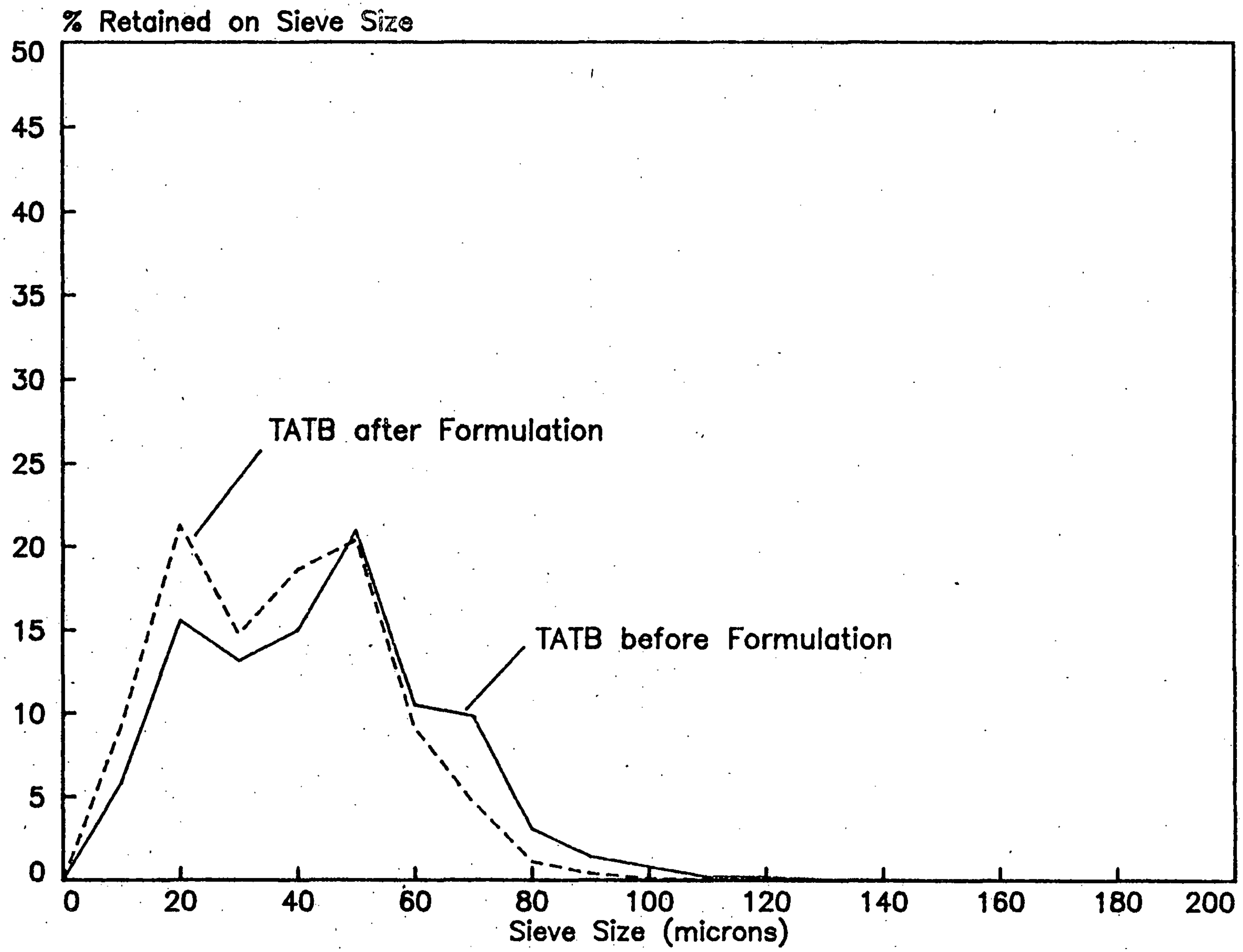

Fig. 1. TATB, BX−1 Distributions Before and After Formulation of LX−17

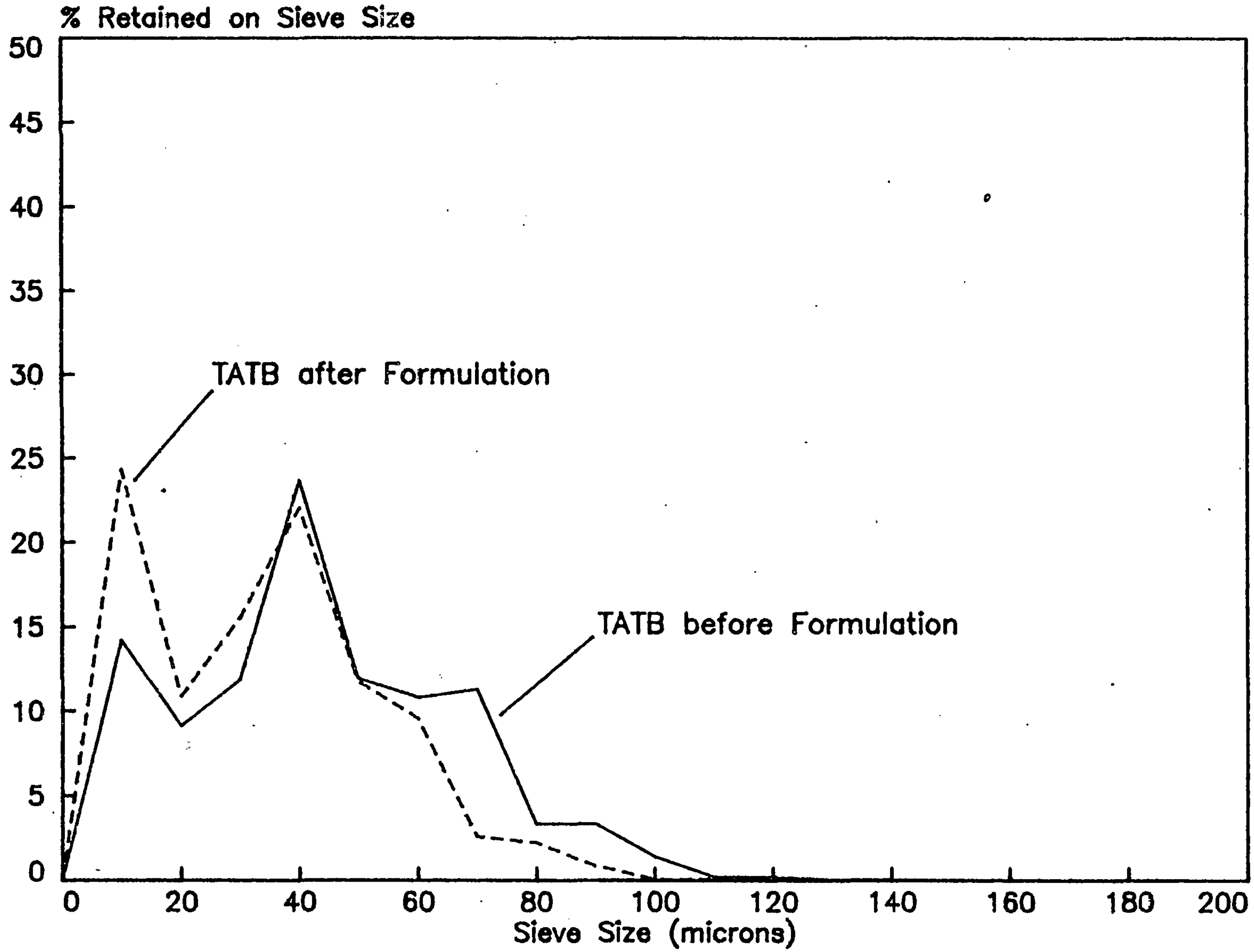

Fig. 2. TATB, BX−2 Distributions Before and After Formulation of LX−17

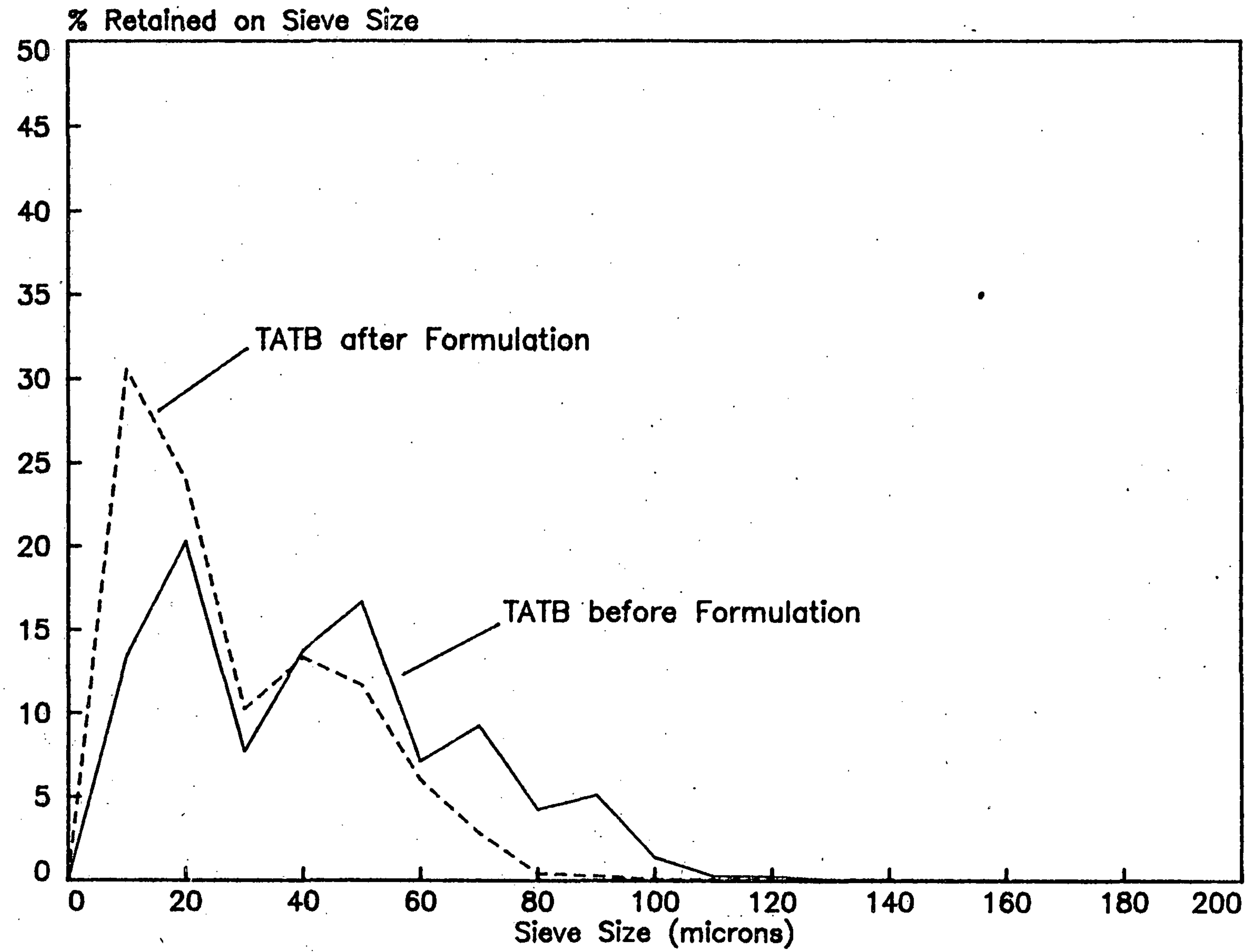

Fig. 3. TATB, BX-4 Distributions Before and After Formulation of LX-17

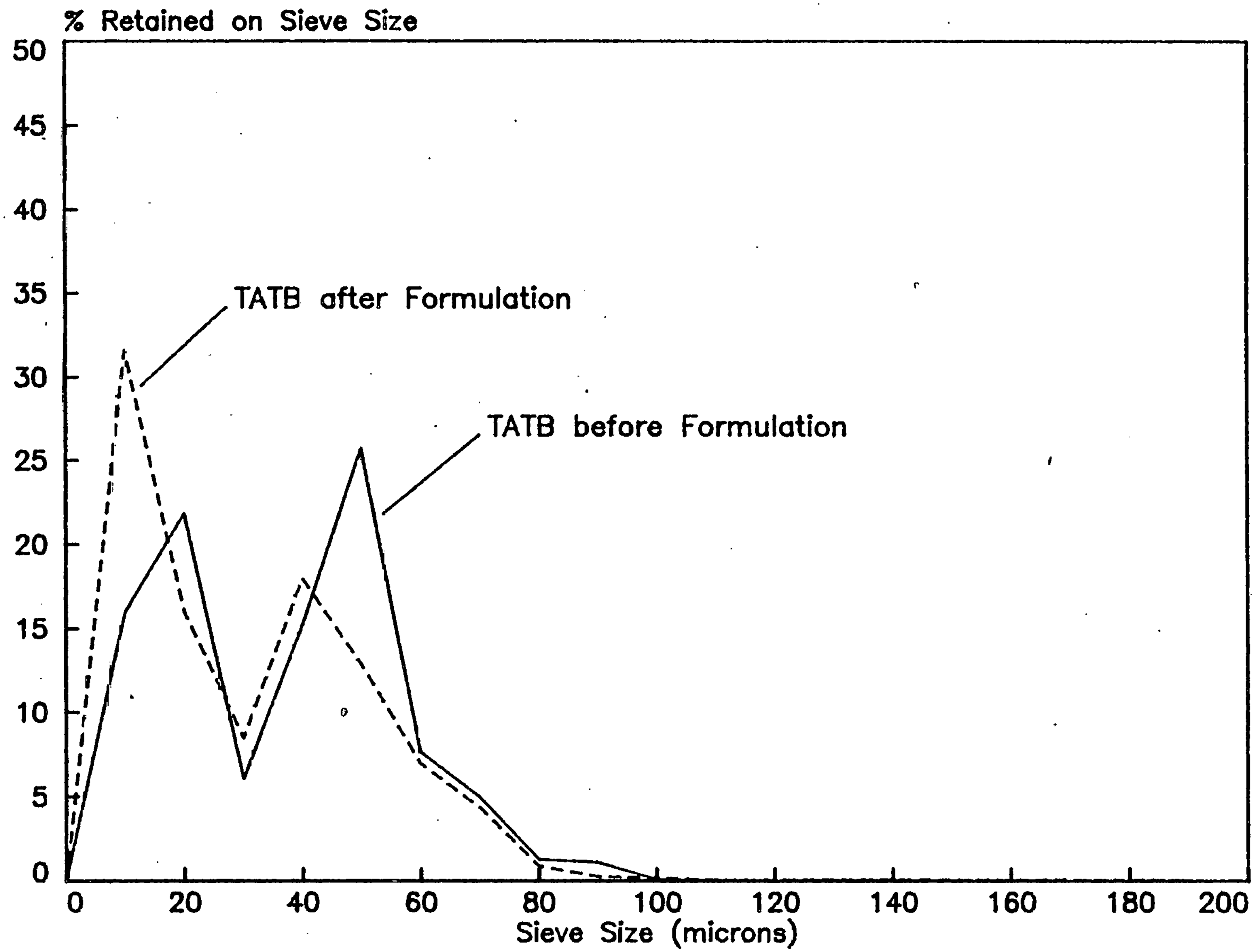

Fig. 4. TATB, BX–5 Distributions Before and After Formulation of LX–17

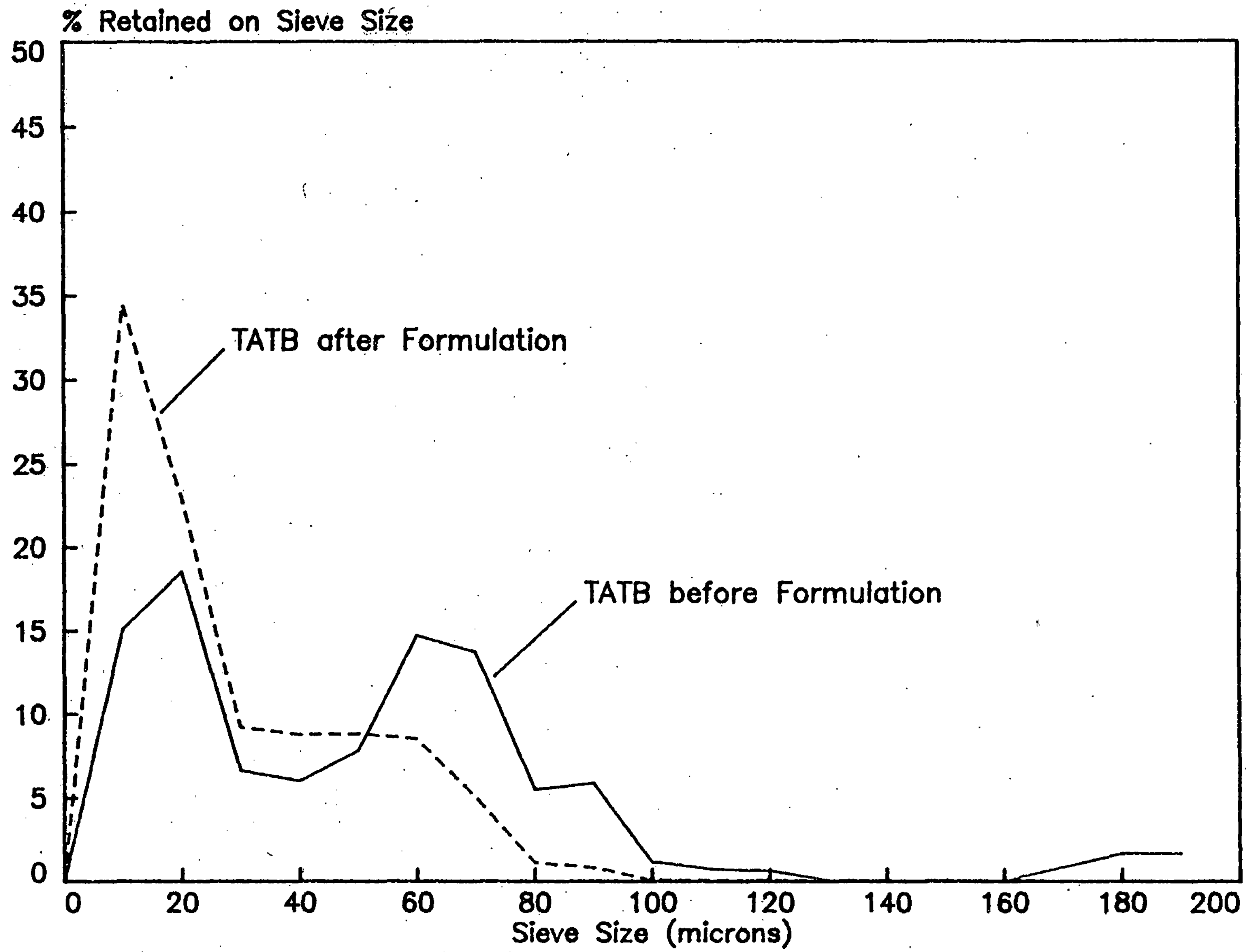

Fig. 5. TATB, BX−6 Distributions Before and After Formulation of LX−17

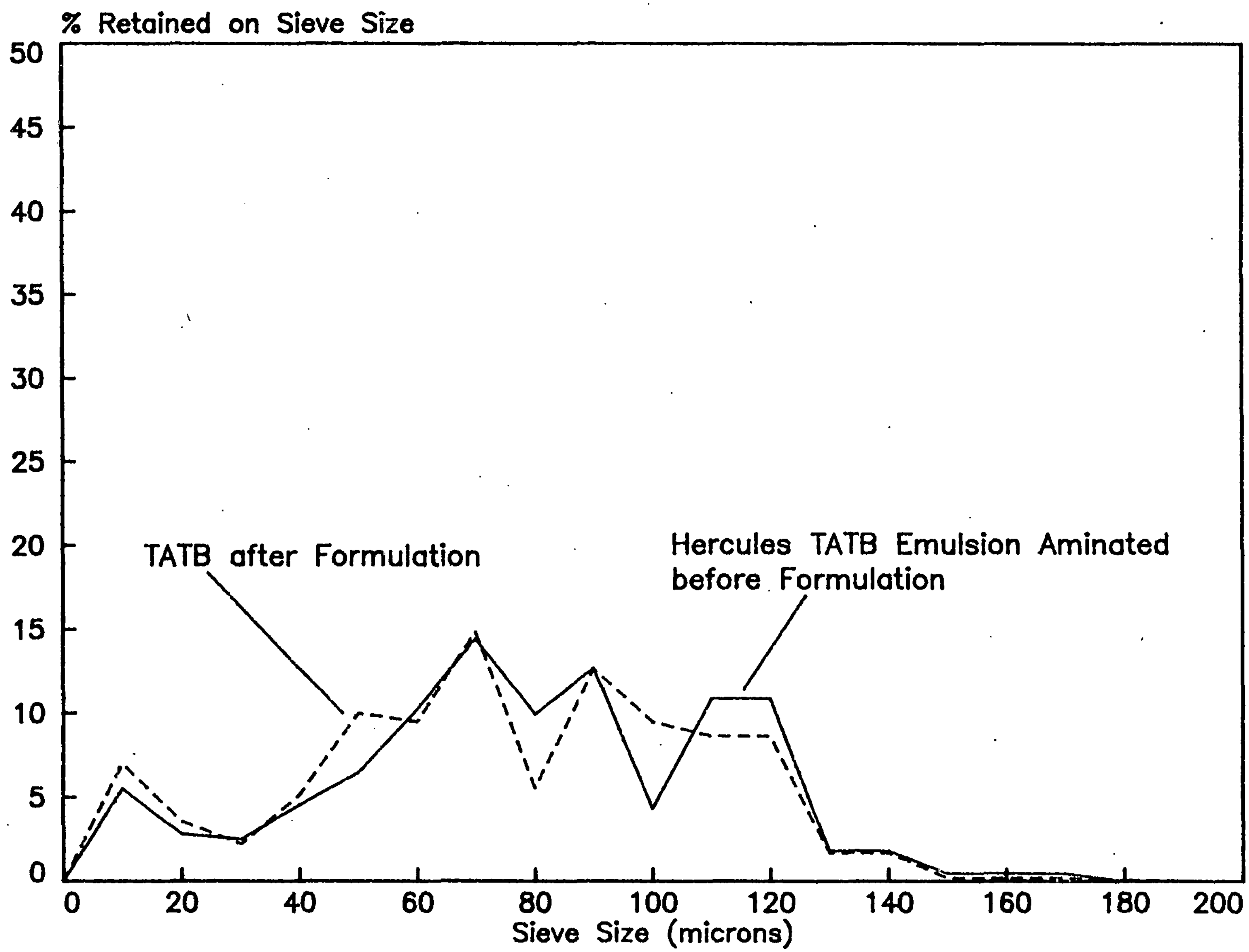

Fig. 6. TATB, BX–3 Distributions Before and After Formulation of LX–17

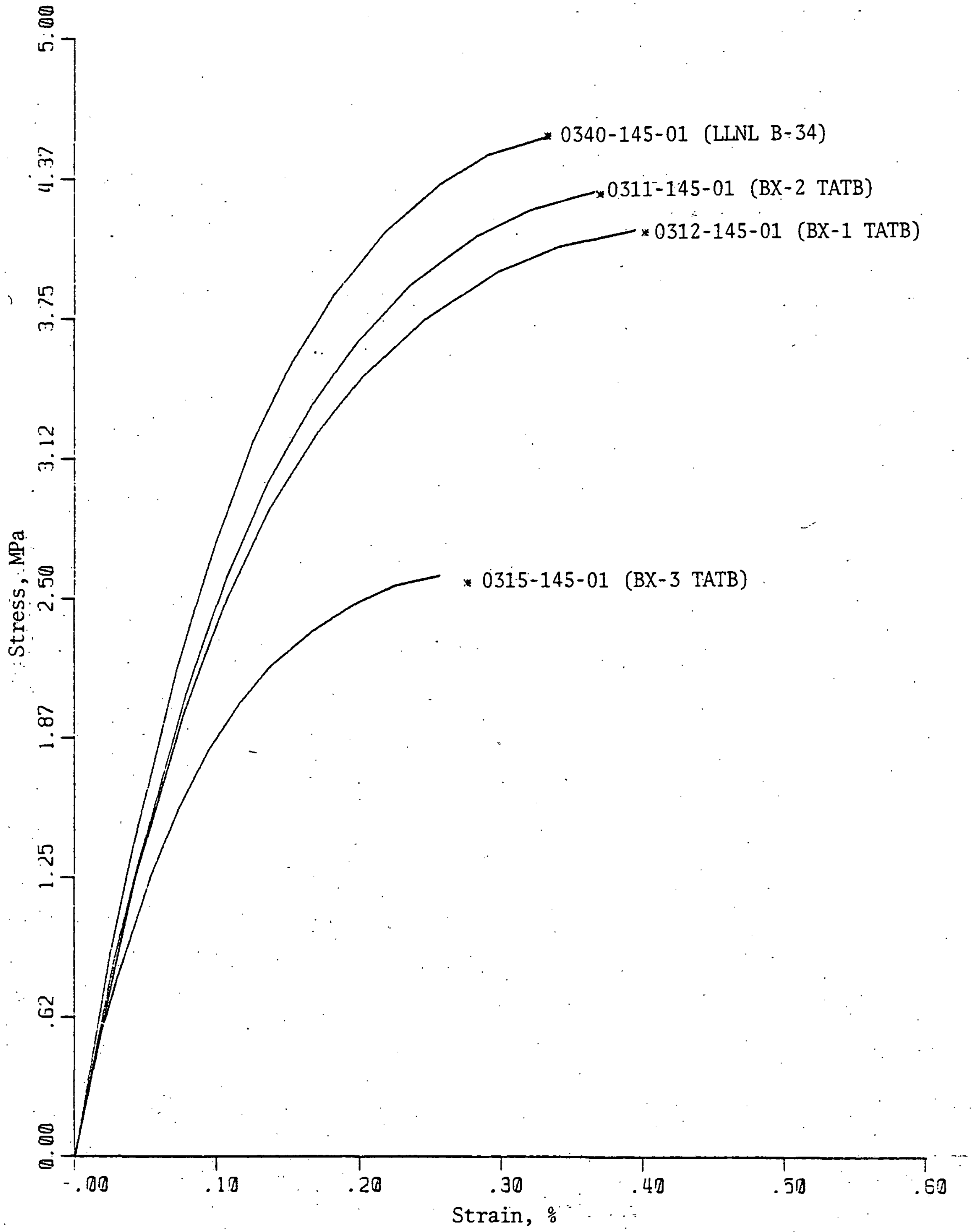

Fig. 7. LX-17 Diametric Disc Mechanical Properties

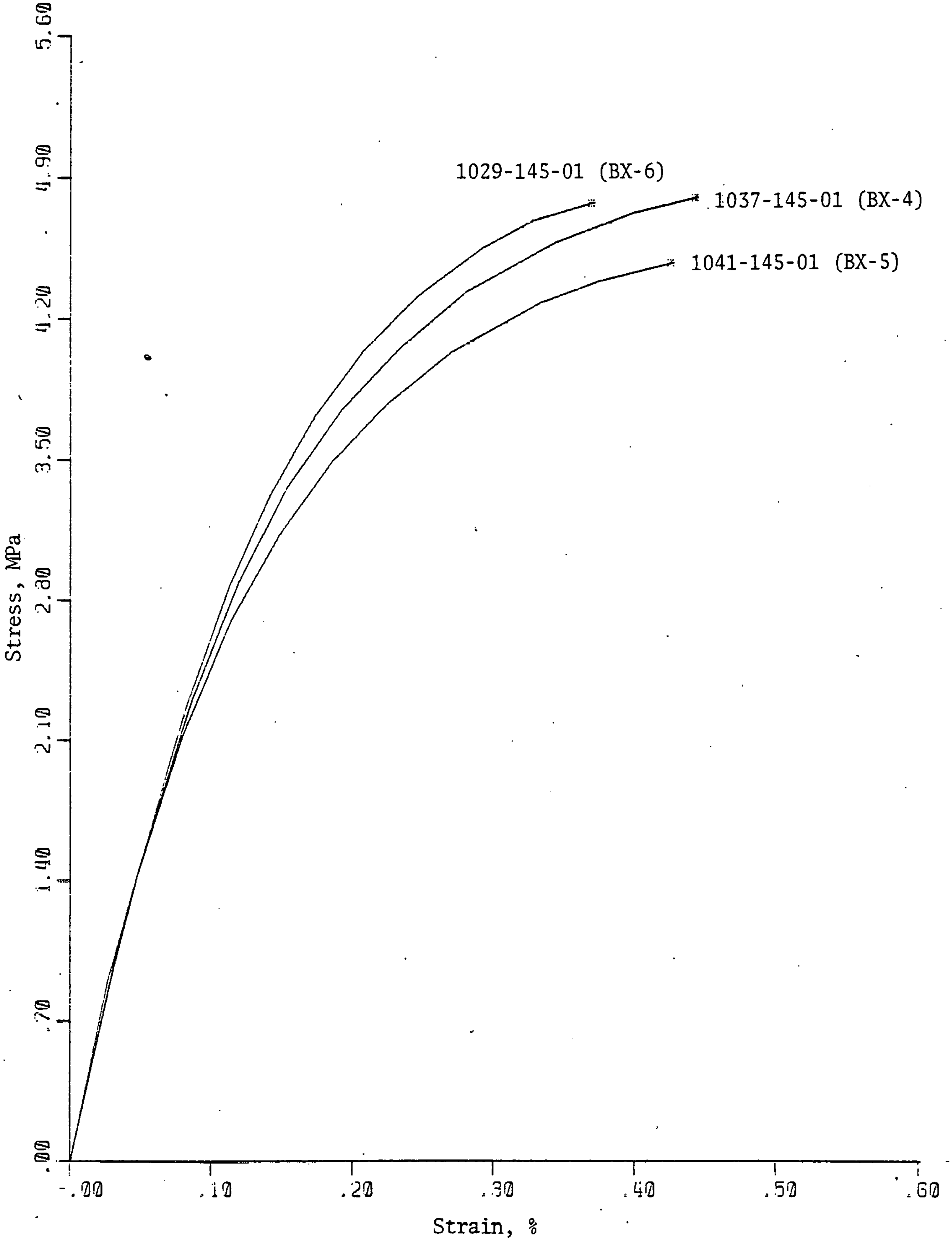

Fig. 8. LX-17 Diametric Disc Strength

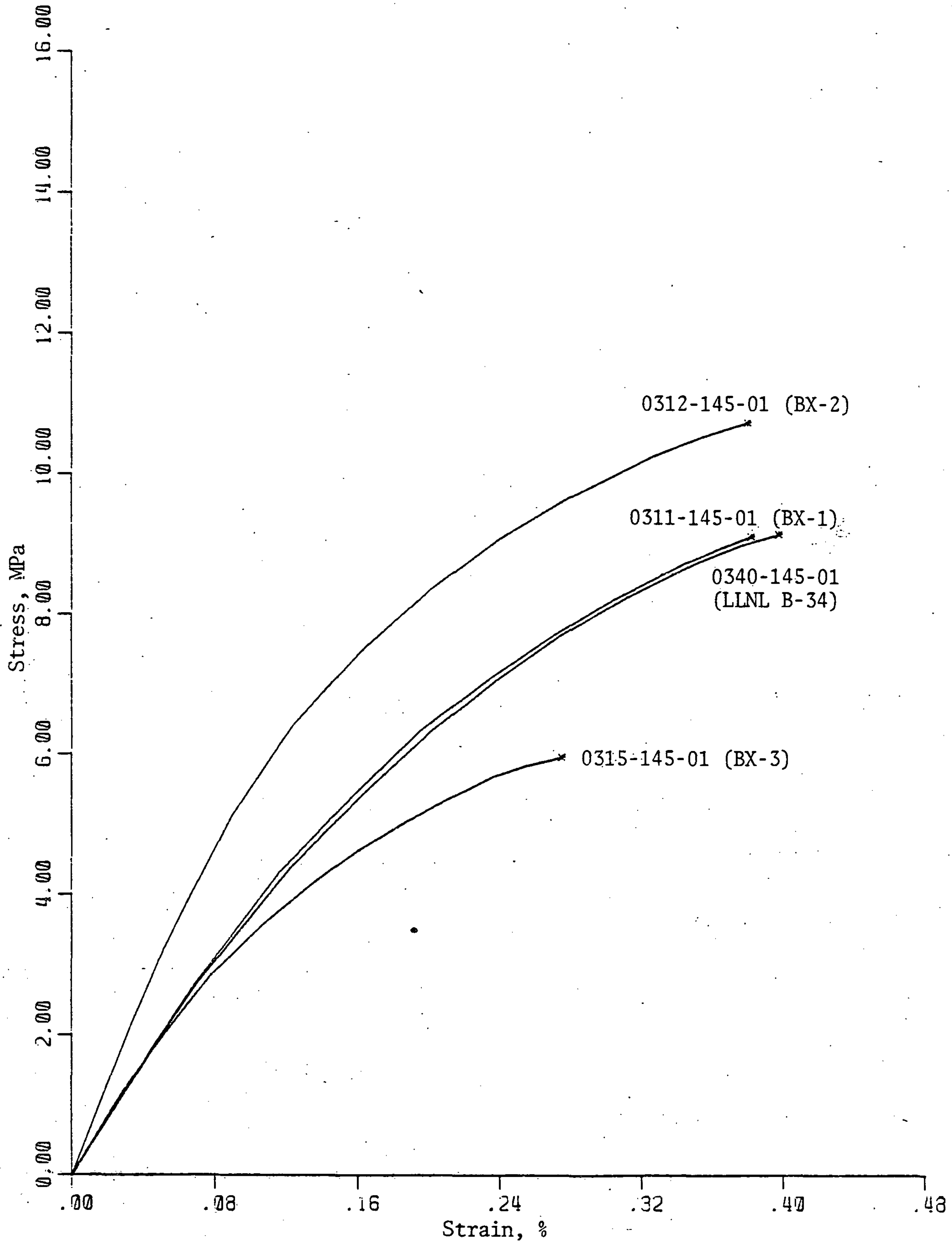

Fig. 9. LX-17 Tensile Strength

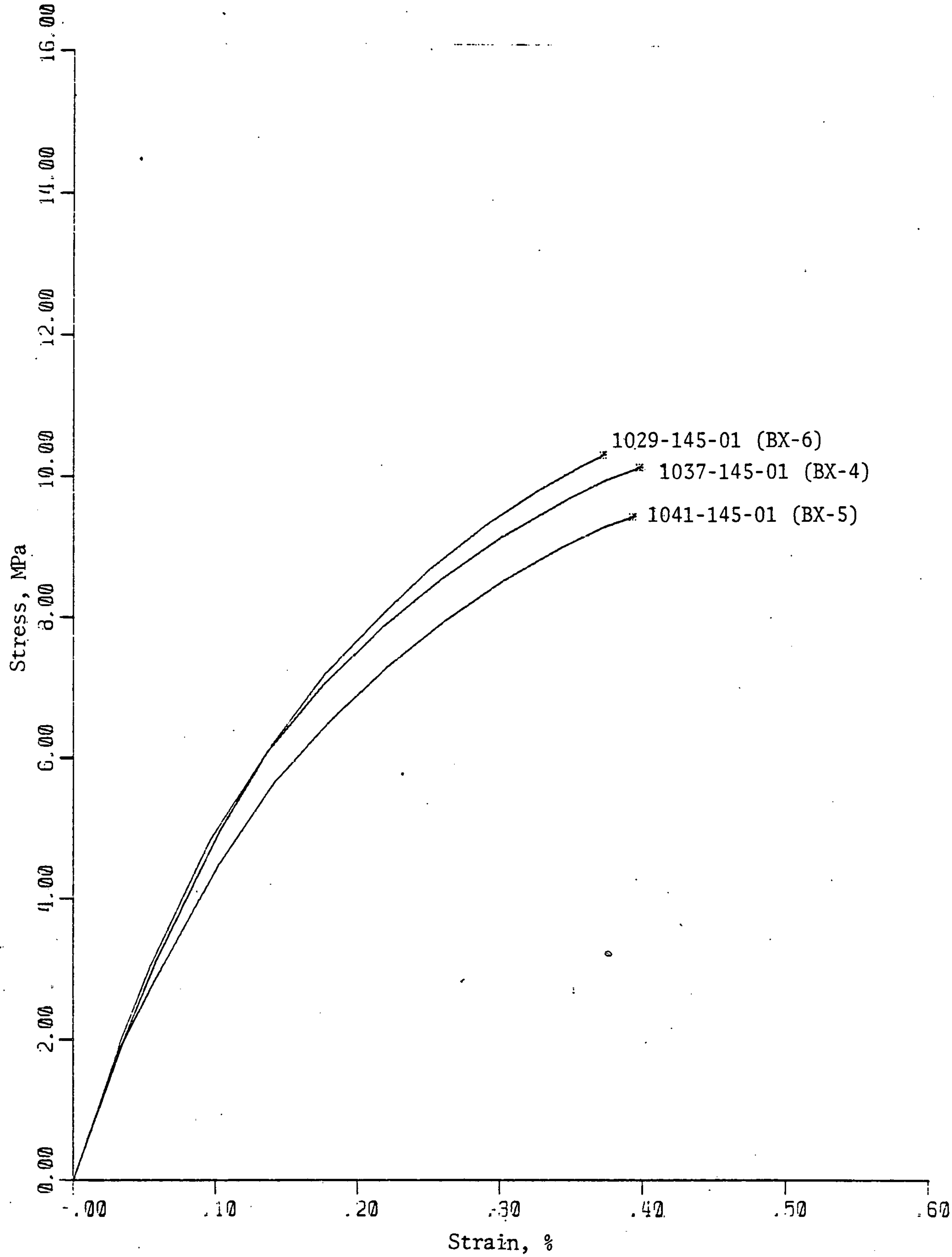

Fig. 10. LX-17 Tensile Strength

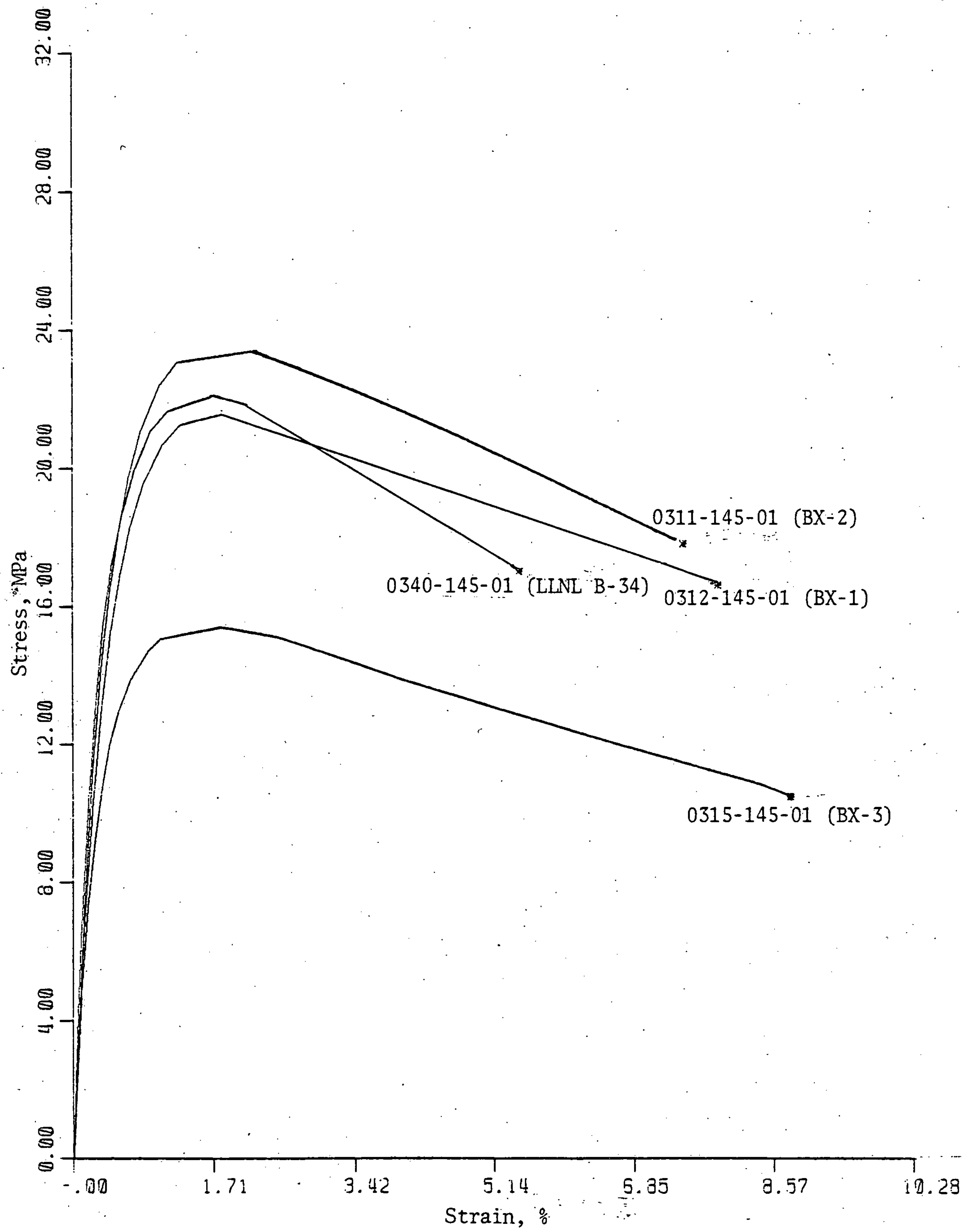

Fig. 11. LX-17 Compression Strength

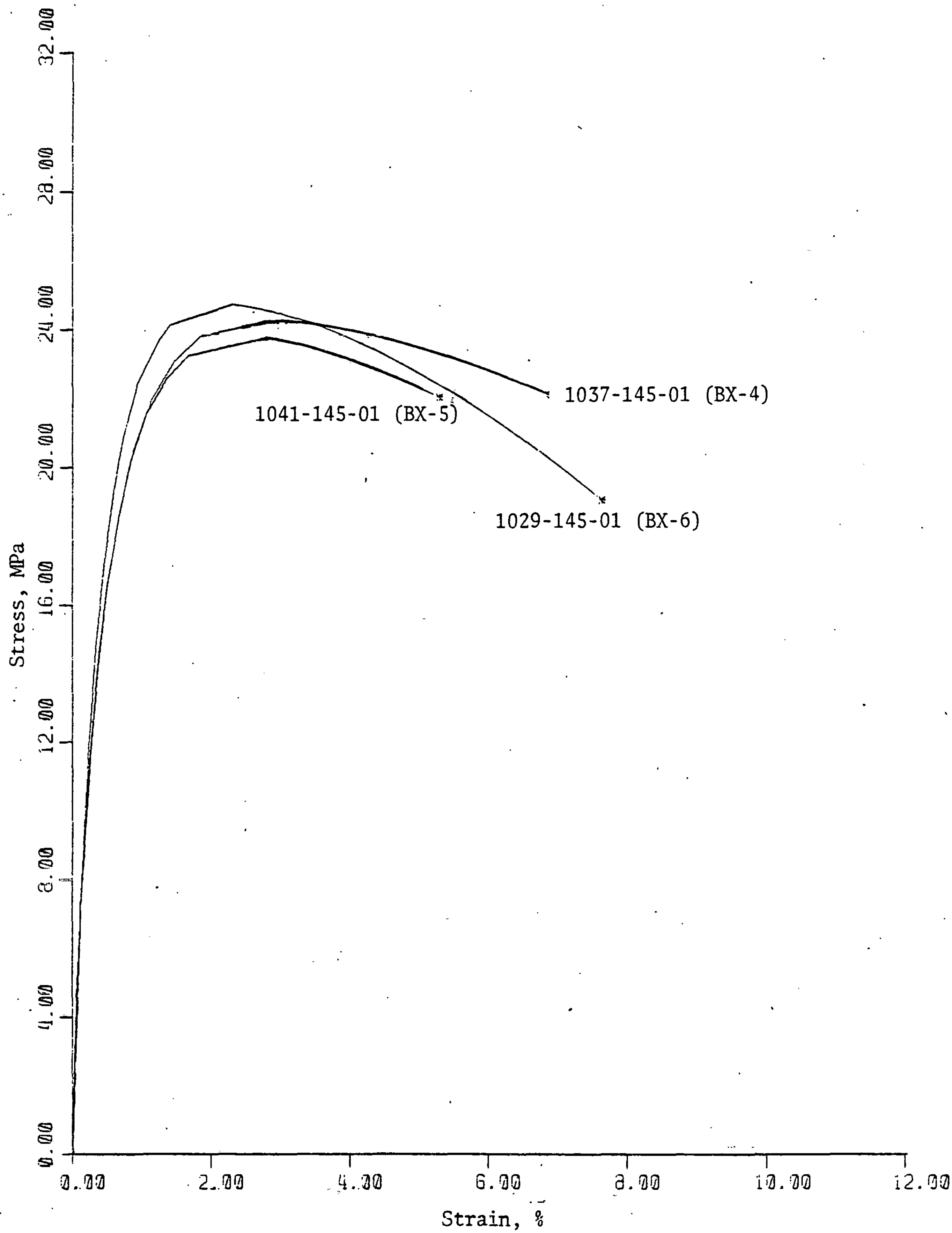

Fig. 12. LX-17 Compression Strength

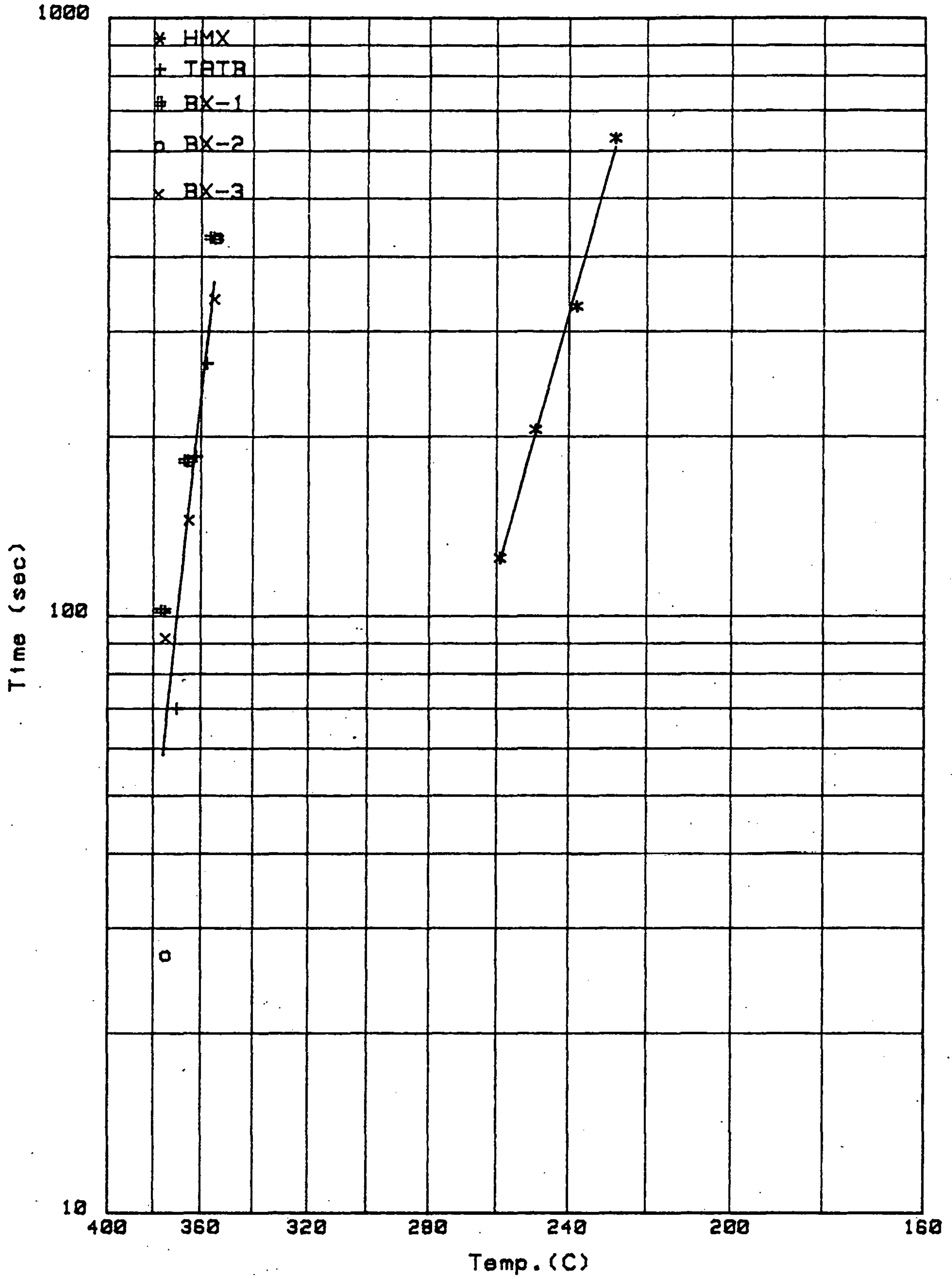

Fig. 13. Henkin Test Results for LX-17 Made from Hercules Wet-Aminated TATB

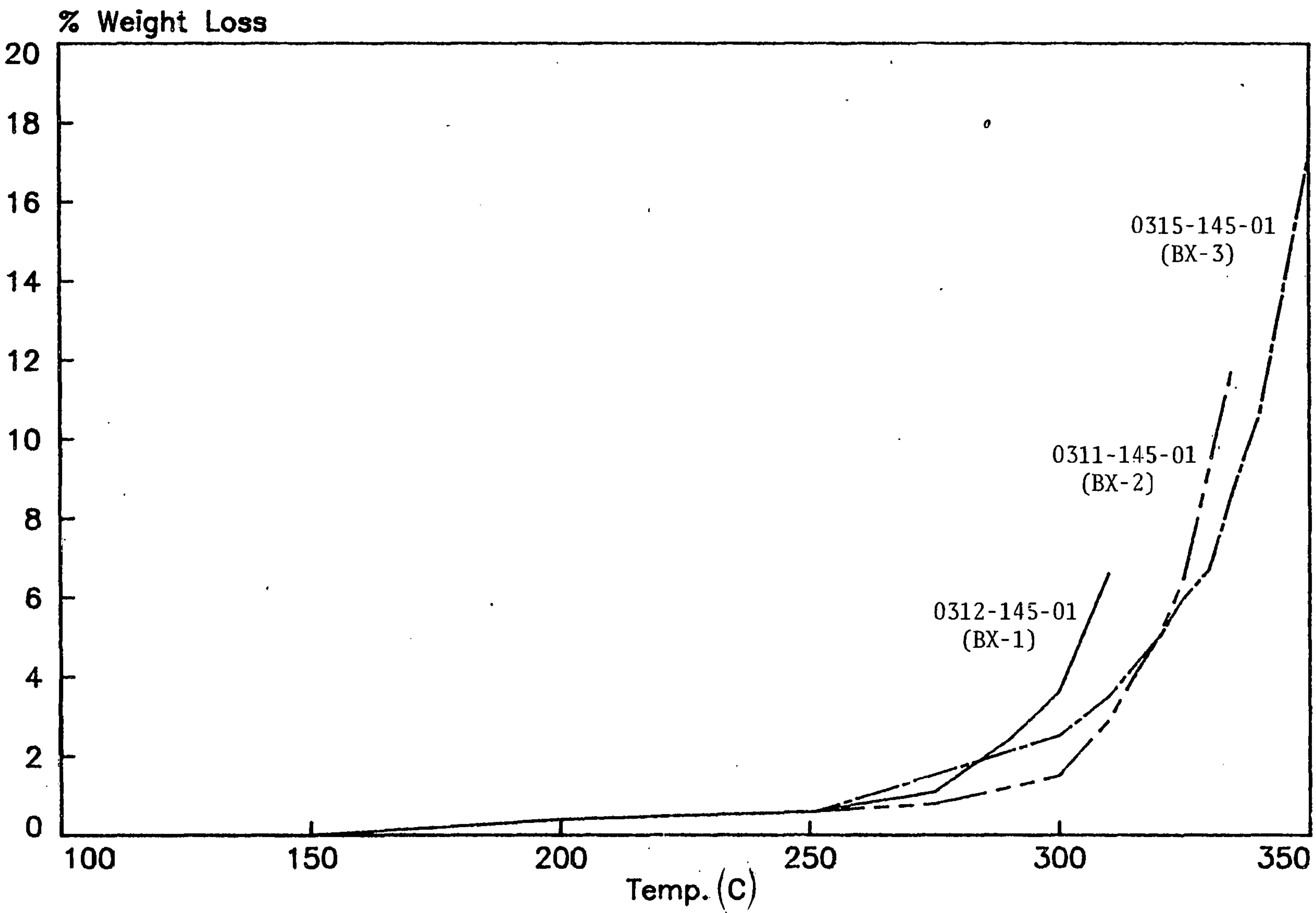

Fig. 14. TGA Test Results for LX-17 Made from Hercules Wet-Aminated TATB

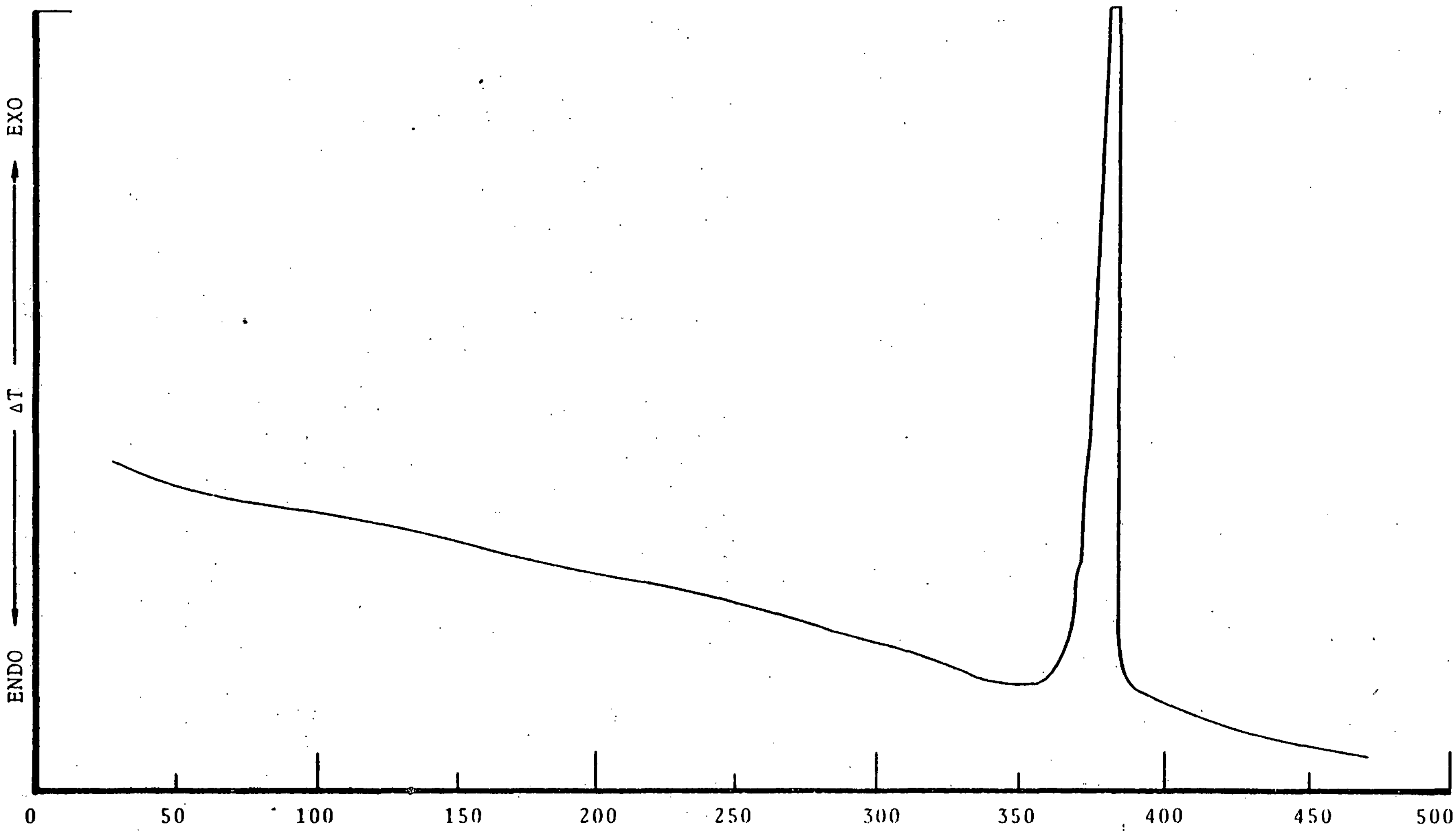

Fig. 15. DTA Thermogram for LX-17 Made from Hercules Wet-Aminated TATB

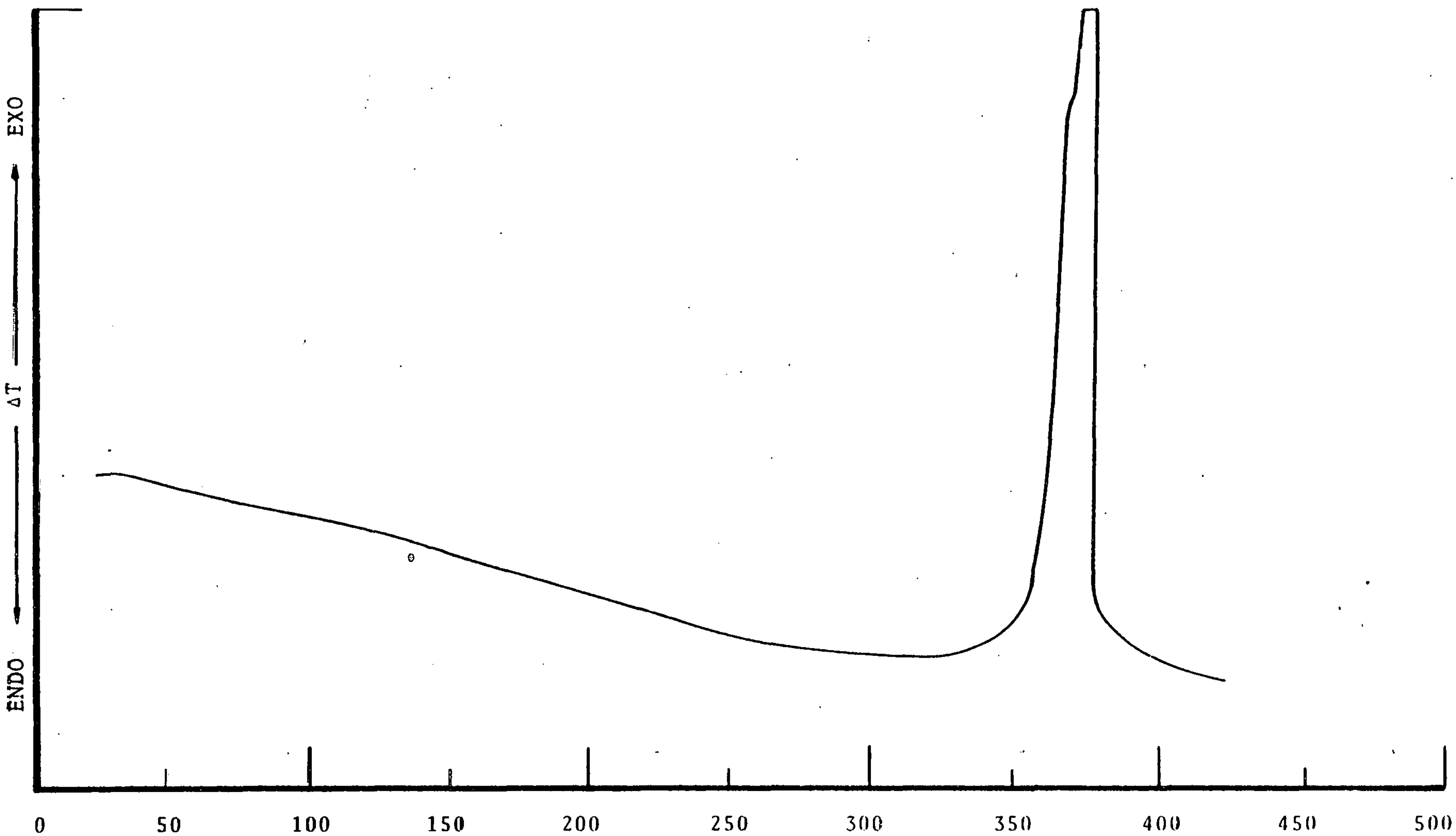

Fig. 16. DTA Thermogram for LX-17 Made from Hercules Emulsion Wet-Aminated TATB

CONCLUSIONS

The LX-17 made from the five wet-aminated TATB batches (BX-1, BX-2, BX-4, BX-5, and BX-6) had surprisingly high strength and was comparable to that obtained from normal dry-aminated TATB. Tensile stress values for these batches ranged from 9.11 to 10.73 MPa, with strain values ranging from 0.38 to 0.41%. The LX-17 made from BX-3, the emulsion-aminated lot, had lower strength, with a tensile stress of 5.96 MPa and a strain of 0.28%, which are typical of those values normally obtained in the past with wet-aminated TATB. Dry-aminated TATB produces LX-17 with tensile stress values ranging from 9.9 to 11.4 MPa, and tensile strain values ranging from 0.27 to 0.44%. Densities of all LX-17 pressings from wet-aminated and emulsion-aminated TATB were higher than those of parts made from dry-aminated TATB.

The particle size of the wet-aminated TATB powders (BX-1, BX-2, BX-4, BX-5, and BX-6) was reduced much more by the formulation process than the emulsion-aminated TATB powder, BX-3, as indicated by the sieve analysis results for the powder and LX-17 batches in Tables II and IV and comparing Figs. 1 through 5 with Fig. 6.

Since the overall cost of producing wet-aminated TATB is comparable to the dry-amination process, the strength deficiencies of the wet-aminated TATB appear to have been eliminated, and because of the undeniable advantage of low chlorine content and higher part densities, serious consideration is being given to wet-aminated TATB for LX-17 production.

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