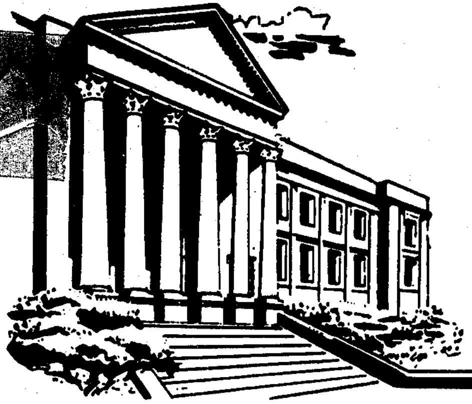

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Final Report **F-A2266**

FEASIBILITY STUDY OF THE MANUFACTURE OF 7.62 mm CARTRIDGE CASES FROM PLASTIC MATERIALS

by

Stephen G. Seger, Jr.
Edmund Thelen

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1. INTRODUCTION

1.1 Purpose

The objectives of this project as stated in the contract, are as follows:

a. Investigate and determine the feasibility of making 7.62 mm cartridge cases from plastic materials which will meet both the performance and mass production requirements of metallic cases.

b. Produce several lots of plastic cases using the most promising materials and processes.

c. Deliver batches of the best resulting cases and a summary report.

1.2 Summary

a) All available plastic materials which from literature searches and our own experience were deemed promising were screened in the Laboratory with particular emphasis being placed on tests that would attempt to define the behavior of the plastic compositions under the most stringent conditions pertinent to their performances in cartridge case. These tests consisted of:

1. Heat deformation of 400°F for 15 minutes.
2. Degradation of 165°F for 3 days.
3. Stress-strain relationship at:
   a) -65°F
   b) room conditions (approximately 75°F)
   c) +165 °F
4. Impact resistance at:
   a) -65°F
   b) room conditions (approximately 75°F)
5. Shock resistance

Procedures for these tests are described in Appendix A.

b) From the screening test results (Section 2) it was concluded that a 30% glass filled 6/10 nylon, heat stabilized and a 30% glass filled phenolic, Fiberite 4030-190 were the best materials for further study.

c) An injection mold was built by Curley-Nelson Company, Inc. for the manufacture of Fiberfil G-2 cartridge cases, (Section 3.1) and a transfer mold for the manufacture of Fiberite cases was constructed by Penn Plastics Company (Section 3.2).

The assembly and firing behaviors of the glass filled phenolic cases are discussed in Section 4.1, while the behavior of the Fiberfil G-2 cartridge cases is treated in Section 4.2.

d) Successful firing was accomplished when the butt sections were composed of brass. No plastic butt sections were satisfactory in the present cartridge design.

e) With brass butts adhesively bonded to phenolic cylinders, projectile velocities were about 2633 ft./second. However, the cylinders were damaged, showing radial cracks above the butt section and severe cracking of the neck.

f) The nylon/brass butt combination gave projectile velocities of about 2630 ft./sec. with virtually no damage to the fired cartridge case.

g) A lot of 500 of these cases, (nylon/brass) molded under somewhat different conditions was then tested at the Arsenal, for velocity, water proofness, and pressure at various temperatures. The results of these tests are discussed in Section 4.2 of this report. The values for the above tests were in line with those for brass cases. However, due to the difference in molding conditions, most of these cases, unlike those previously tested (f)above) showed a longitudinal crack.

h) The high temperature requirements for plastic cases is discussed in Section 5. The study suggested that thermoplastic materials other then those that passed the bending beam test at 400°F could conceivably be feasible for plastic cartridge cases provided they retained adequate strengths at 350°F. Two such materials, Lexan and Nylatron G. S. were injection molded by Curley-Nelson Co., and assembled and fired at the Arsenal. The performance of these materials could not be proved because the butts failed. Results from these studies also appear in Section 5.

i) From the present state of the art it can be concluded that a cartridge case composed of a brass butt section and a glass filled nylon cylindrical wall is nearly successful in performance and can be developed readily to be satisfactory.

j) Cost and weight of these cartridge cases are about forty percent less than for all-brass cases.

k) The further cost and weight advantages of an all-plastic case might be realized through design changes in the cartridge. See Section 6 for recommendation.

2. SCREENING OF PLASTIC COMPOSITIONS

2.1 Screening Tests

A minimum number of screening tests were devised to guide us in the selection of compositions worthy of trial in firing tests. It was realized that it was important that these screening tests define the behavior of the plastic compositions under the most stringent conditions and with this realization it was decided to use the following tests:

1. Heat Deformation at 400°F for 15 minutes (Bending Beam Test)
2. Degradation at 165°F for 3 days
3. Stress-strain Performance (Tensile strength, elongation, Young's Modulus) at:

   a) -65°F
   b) room conditions (approx. 75°F)
   c) +165°F

4. Impact Resistance at:
   a) -65°F
   b) room conditions (approx. 75°F)
5. Compatibility Tests with propellant
6. Moisture Absorption

The procedures for these tests are described in Appendix (A).

Rather than subject every plastic composition to the entire screening program it was decided at the start that each material would first be tested in the area of its own most probable weakness, therefore, the thermoplastics were in most cases initially subjected to the Bending Beam Test at 400°F and the thermosets were tested for impact strength. Further screening was undertaken only if the materials passed their respective initial test.

2.2 Materials Selected

Materials chosen for original screening included some with outstandingly high heat distortion temperatures, and for comparison purposes, some with lower values. Other recorded test values were also taken into consideration such as the values for tensile strength, elongation, resistance to heat (continuous), and impact (Izod). Table 1 lists the basic materials that were selected from handbook data for screening, along with the above-mentioned pertinent information. Table 2 lists the exact compositions that were screened including some for which handbook data were not available. This latter table includes trade designations and suppliers.

Table 1

SELECTED MATERIALS(1)

Material	Filler	Resistance to Heat (Continuous) °F	Heat Distortion °F (ASTM-D-648)	Impact ft lb/in. of notch	Tensile psi	Elongation %
Phenol Formaldehyde	Glass	350-450	600	10-15	5000-10000	0.2
Silicone	Glass	480	500-900	3-5	4000-5000	-
Polyester	Glass	300-350	-	5-30	15000-30000	0.5-5
Cast Epoxy	-	300	130-365	0.45-17	9000-12000	-
Nylon	Glass	300-400	-	Variable	18000-20000	6.5-8.5
Teflon	-	500	250 (66 psi)	3.0	1500-3000	100-200
Genetron VK(2)	-	-	350	-	-	-
Polysulfide Cast Epoxy	-	250-300	150-250	2-10	3000-10000	0-100
Diallyl Phthalate	Glass	300-350	-	5-30	15000-30000	0.5-5

(1) All of the values, except those for Genetron, VK, were taken from the Plastic Property Chart - Modern Plastics Encylopedia Issue, 1956. The Heat Distortion value for Genetron VK was taken from the book, Fluorocarbons, Rudner, Meritt Allen, Reinhold, New York: 1958.

(2) Genetron VK is a modified trifluorochloroethylene Polymer.

Table 2

MATERIALS SCREENED

Basic Type	Filter	Trade designation	Supplier
Nylon	Unfilled	Zytel 101 (6/6 nylon)	Dupont
	Unfilled	Zytel 31 (6/10 nylon)	Dupont
	Unfilled	Zytel 33 (6/10 nylon -heat resistant)	Dupont
	30% Glass	Fiberfil G2 (Zytel 31)	Fiberfil Corporation
	30% Glass	Fiberfil G2 (Zytel 33)	Fiberfil Corporation
	Molybdenum Disulfide	Nylatron GS (6/6 nylon)	Polymer Processes, Inc.
Diallyl Phthalate	Orlon	Durez 16694	Durez
Vinylidene Chloride modified	Unfilled	Genetron VK	Allied Chemical Corporation
polychlorotrifluoroethylene	15% Glass	Genetron VK	Allied Chemical Corporation
Phenol-Formaldehyde	**Woodflour**	BMG-5000 BK	Bakelite
	Mineral	RX 460	Rogers Corporation
Polyester	Asbestos	RA 4100	Rogers Corporation
	Glass (30%)	4030-190	Rogers Corporation
	Nylon Flock	Durez 14475	Durez
	Nitrite rubber	Durez 16840	Durez
	10% Glass	Alkyd PM4	Allied Chemical
Polycarbonate	30% Glass	Alkyd 446	Allied Chemical
	Unfilled	Lexan	General Electric
Teflon	**Unfilled**	**Teflon pwd. No. 5**	Dupont
	10% Asbestos	Teflon (filler added in Lab.)	Dupont
	20% Glass	**Teflon** (filler added in Lab)	
Cross linked Polyethylene	Carbon Black		----------
Polyvinyl chloride plastisol	Unfilled	No. 2012	Quelcor Inc.
Polysulfide modified	Unfilled	Epon 828 & Thiokol LP3	Shell Chemical, Thiokol
	Unfilled	Epon 824 & Thiokol LP3	Shell Chemical, Thiokol
Epoxy	25% Glass	Epon 828 & Thiokol LP3 (filler)	Shell Chemical, Thiokol
	25% Glass	Epon 824 & Thiokol LP3	Shell Chemical, Thiokol
Modified Epoxies	Unfilled	Stycast 2651	Emerson & Cumming, Inc.
	15% Glass	Stycast 2651 & Thiokol LP3	Emerson & Cumming, Inc.-Thiokol
	Unfilled	Stycast 2741	Emerson & Cumming, Inc.-Thiokol
	10% Glass	Stycast 2741	Emerson & Cumming, Inc.-Thiokol
	Unfilled	Stycast 2340 M	Emerson & Cumming, Inc.-Thiokol

2.3 Thermoplastic Materials

The thermoplastic materials investigated during this study are as follows:

a) Nylon
b) Teflon
c) Polycarbonate
d) Polyethylene (Cross-Linked)
e) Polyvinyl Chloride Plastisol
f) Modified Polychlorotrifluoroethylene

These materials were screened either without filler content or filled as noted in Table 2 of the previous section. The important properties of these materials, with respect to their use in cartridge cases, are discussed below.

a) Nylon-Base Products

The original screening of nylon was carried out on molded, unfilled nylon Zytel 101 which is the poly condensation product of the inter-reaction between adipic acid and hexamethylenediamine. This polyamide is commonly referred to as a 6/6 nylon. The screening test results for this material are shown in column 1 of Table 3.

The data obtained from these tests indicated that the material was worthy of further consideration for use in cartridges, particularly if its coefficient of linear expansion and moisture absorption could be reduced, as we expected, by incorporating the proper filler, or using a different type of nylon, such as nylon 6/10 in place of the 6/6. The following experimentally determined characteristics of the 6/6 nylon are worthy of note:

(1) In the temperature range of -65°F to 400°F, the rigidity of the nylon changes by a factor of 5 or less and over the entire temperature range the material remains tough and resilient.

Table 3

SCREENING DATA ON VARIOUS NYLONS

Temperature	Tests	Unfilled Zytel 101	Unfilled Zytel 31	Unfilled Zytel 33	30% Glass Zytel 31	30% Glass Zytel 33	Molybdenum Disulfide
-65°F	Drop Ball Impact (notched)	30.3 in lbs			12.7	11.1	62.7
	(unnotched)	41.6 in lbs					
	Rigidity Modulus, dyn/cm^2	8.86 x 10^9					1.91 x 10^{10}
	Tensile strengths, psi	> 11,444					>17,118
	Elongation at break, %	9.0(1)					21.6(1)
Room	Rigidity Modulus, dyn/cm^2	7.7 x 10^9	2.1 x 10^9	2.7 x 10^9	6.9 x 10^9		6.9 x 10^9
	Tensile strength, psi	8950	5600	6000	5000		9764
	Elongation break, %	28.2	300	222	8.3		77.6
	Drop Ball Impact (notched)	>117			25.1	25.0	>140
	(unnotched)	83.8					
400°F	Moisture Absorption	5.8	2.87	3.14	2.24	0.00	
	Impact (Charpy)	11			53.4	59.6	124
	Rigidity Modulus, dyn/cm^2	3.2 x 10^9					2.84 x 10^9
	Tensile strength, psi	5663					7740
	Elongation at break, %	54					158.9
	Rigidity Modulus, (Beam) dyn/cm^2	2.9 x 10^9	failed	failed	2.66 x 10^9	2.04 x 10^9	3.14 x 10^9
	Permanent Deformation (Beam) %	53.2					0.7
	Rigidity Modulus (Tension) dyn/cm^2						3.21 x 10^9
	Permanent Deformation (Tension) %	-1.3					-0.5
	Coefficient of Linear Expansion, cm/cm/c°c	11.0 x 10^{-5}	5.9 x 10^{-5}	6.0 x 10^{-5}	2.8 x 10^{-5}	2.14 x 10^6	6.4 x 10^{-5}

(1) Elongation at max stress

(2) Nylon exhibits a second order transition point at about 117°F, and in heating through this temperature the rigidity and tensile strength abruptly decrease by a factor between 1.5 and 2. Below and above this temperature, these properties are surprisingly independent of temperature.

(3) On heating the samples for 3 days at 165°F immersed in propellant, there is a loss of 0.5% to 0.6% in weight as water is lost. Since water plasticizes nylon, the loss of it causes the material to increase in rigidity and tensile strength, and decrease in elongation. That the loss in weight is due to a loss of water is confirmed by the fact that a loss (0.2% in 2 days) also occurs on desiccating the sample, at room temperature and a greater loss (0.68% in 2 days) occurs on heating at 165°F in circulating air.

(4) The nylon, in 3 days at 165°F, acquired a yellow stain from the contact with powder, which masked the normal fluorescence of nylon in ultraviolet light. This stain penetrated an appreciable depth into the nylon.

However it did not alter the contact angle of water drops on nylon, which suggests that the stain material is not chemically far different from nylon. The formation of stain did not measurably affect the mechanical properties of the nylon; some hardening was observed but more likely was due to loss of plasticizing water.

(5) The coefficient of linear expansion, determined experimentally from the elongation of specimens on heating them to 400°F, closely checks handbook values.

(6) After the sample was heated to 400°F and cooled, it was found to be permanently reduced in length by 1 to 2%, and also to be considerably increased in brittleness. These changes are characteristic of an increase in crystallinity which is known to occur in nylon subjected to heating for appreciable time intervals.

(7) No measurable elongation of a column of nylon under 192 psi tensile load at 400°F could be noted. Rigidity of the material (Bending Beam) was calculated and found to be in line with expectations based on rigidity measurements at other temperatures.

We concluded that the mechanical properties of the nylon over the entire temperature range were favorable to its use in cartridge cases. Furthermore, while the nature of the stain due to contact with propellant has not been defined closely, present evidence is that it does not indicate a degradation of nylon by propellant.

Since the properties of molded nylon compositions are dependent upon molding conditions, any attempt to compare the various types of nylon with each other with any degree of certainty requires that the molding conditions be adequately controlled. Therefore an injection mold was constructed by Curley Nelson Company, Inc., with the approval of the Arsenal, for the molding of square sheets 5 inches on a side with their thickness being reduced in steps from .0625 in. to .025 in. and then to .015 in.

Utilizing this mold various nylon compositions were injection molded for processing through our screening procedures, so that we could choose the composition possessing the best mechanical and thermal properties, as well as low moisture absorption, for molding into plastic cartridge cases. As was mentioned previously in the injection molding of nylon, the molding conditions greatly influenced the final physical properties of the molded piece. In our particular case, we were interested in a final product possessing a high degree of toughness. This toughness can be achieved by the rapid cooling of the molded piece. Therefore, the mold temperature and the temperatures of the molten nylon, which is injected into the mold under pressure, should be balanced to give the fastest cooling time possible, and still fill the mold without creating internal stresses by rapid cooling.

Since the mold for making the square sheets was far larger in cavity than one that would be designed for the molding of cartridge cases, the cooling rate, would out of necessity, be longer for the sheet mold. Thus the toughness of the flat sheets would be inferior when compared against the toughness of cartridge cases. To circumvent this problem it was decided not to fill the sheet mold completely. This permitted faster cooling of the molded product thereby increasing its toughness. Thus the final molded sheet was approximately 1/2-inch short in the 0.015" section, however, enough of the thin material was available for testing.

Table 4, based on data from Curley-Nelson Company, Inc. shows the temperatures, pressures, and cycles used for the injection molding of the various nylon compositions.

Table 4

INJECTION MOLDING CONDITIONS

	Temperatures, °F				
	Cylinder				
Material	Front	Back	Mold	Pressure psi	Cycle sec.
Nylatron GS	520	550	150	20,000	15/5
Zytel 33	480	480	150	17,000	15/5
Zytel 31	450	500	130	17,000	15/5
30% Glass, Zytel 31	530	520	150	18,000	15/5
30% Glass, Zytel 33	530	520	150	18,000	15/5

The screening data for these materials can be found in Table 3. It will be noted from this table that the entire series of screening tests for the Zytel 31 and 33, either unfilled or containing 30% glass fibers, was not carried out. Mainly those tests that covered the area in which either the unfilled Zytel 101 or the molybdenum disulfide filled nylon showed weaknesses were investigated, specifically the coefficient of linear expansion and the moisture absorption. However, the impact strengths at a -65°F and at room temperature as well as the rigidity modulus (Beam) at 400°F were also determined to assure that these materials behave satisfactorily under these conditions.

Zytel 31 and Zytel 33 are both 6/10 nylons (poly condensation products of the reaction between hexamethylenediamine and sebacic acid) however Zytel 33 contains an additive that decreases its temperature susceptibility. On cooling after being subjected to 400°F for 15 minutes, Zytel 31 is extremely brittle, while Zytel 33 still retains its flexibility. This holds true for the glass filled nylons as well.

The shock resistance was then run on the various nylons (see Table 5) and the behaviors of these nylon compositions were compared against Cycolac LTH 3003, a high impact polystyrene that was successfully used by Naval Ordnance Laboratory for their 105 mm cartridge case. Table 6 lists the B_p impact values of the various nylons. Although the 30% glass filled Zytel 33 was not tested for shock resistance, there is no reason to believe that its behavior would be any different than that of Fiberfil G2, a 30% glass filled Zytel 31.

Considering the shock resistance results as well as the screening data, Table 3, 30% glass filled nylon Zytel 33, appeared most worthy of test firing.

Up until this time the impact resistance of polymeric compositions under sub-zero conditions had been determined only at -65°F. In all cases the B_b impact values at this temperature were much lower than those obtained at room temperature. However, the temperature choice of -65°F was purely arbitrary, being chosen to allow a wide margin of safety to assure the proper behavior at -40°F. According to government specifications the cartridges should be capable of being fired successfully

Table 5

SHOCK RESISTANCE OF NYLON COMPOSITIONS

Material	Thickness of Specimen Tested (inches)	Results
Fiberfil G-2	0.025	med. hole, no radiating cracks
	0.0625	med. hole, no radiating cracks
Nylatron GS	0.025	med. hole, fine radiating cracks
	0.0625	large hole, long cracks (3 samples)
Zytel 33	0.025	med. hole, fine radiating cracks
	0.0625	3 out of 3 samples shattered
Zytel 31	0.025	med. hole, fine radiating cracks
	0.0625	2 out of 3 samples shattered
Zytel 101	0.025	med. hole, fine radiating cracks
	0.0625	1 out of 3 samples shattered
Cycolac LTH 3003	0.0625	small punctured-dimple, no radiating cracks

Table 6

AVERAGE IMPACT (B_p) VALUES OF NYLON COMPOSITIONS

Material	Thickness of Specimen (in.)	Average B_p Value* (in. lbs.)
Fiberfil G-2	.015	9.8
	.025	8.5
	.0625	53.4
Nylatron GS	.015	245
	.025	12.6
	.0625	48.3
Zytel 33	.015	168
	.025	105.1
	.0625	66.9
Zytel 31	.015	138.4
	.025	92.7
	.0625	39.7
Zytel 101	.015	297
	.025	147
	.0625	44.2

*Impact specimens 0.015" thick were un-notched at -40°F. Realizing that glass filled nylon could possibly exhibit a transition point at a temperature lower than -40°F, the drop ball impact test was run on 0.025" thick specimens of 30% glass filled Zytel 33 at temperatures of -35°F and -40°F. Values at room temperature and at -65°F had already been obtained.

Table 7 lists the B_b impact values on the 30% glass filled Zytel 33 for the temperatures mentioned.

Table 7

IMPACT VALUES OF 30% GLASS FILLED ZYTEL 33 AT SUB-ZERO TEMPERATURES

Temperature	B_b Impact Values (in. lbs)
room temperature	14.4
-35°F	13.1
-40°F	10.0
-65°F	3.4

From these data it appears that a transition temperature does exist somewhere between -40°F and -65°F, and at this point the impact resistance of the material decreases markedly. Much to our benefit the impact resistance is only slightly influenced by the sub-zero conditions encountered above this transition temperature.

b) Teflon

The Teflon test specimens were cut from a 6" x 6" x 1/16" sheet that was molded by pre-forming the sheet in a compression mold at room temperature under a pressure of 2000 psi. The compressed sheets so formed were removed from the mold and sintered at 700°F for 30 minutes. The sintered products were then cooled at two different rates. In one case the cooling time was approximately 20 minutes; in the second case the cooling time was greater than five hours. A slow rate of cooling is necessary to accurately control dimensional tolerances, however for ultimate strength the sintered product should be rapidly cooled. Stress-strain curves were run on these samples as well as on specimens die-cut from a sheet of commercially molded Teflon. These values appear in Table 8.

Table 8

EFFECT OF RATE OF COOLING ON TEFLON MOLDINGS

Cooling Time	20 min	5 hrs	Commercial Sample Unknown
Rigidity Modulus (dyn/cm^2)	1.18×10^9	1.70×10^9	1.50×10^9
Tensile Strength (psi)	2030	1980	3500
Elongation (%)	126	91	56

All these samples flowed from the test holder at 400°F, under a 16.5 gm load which shows that their rigidity at this temperature is much too low. 10% asbestos filler, Grade 7TF2 from Canadian Johns-Manville Co. Ltd., was dry blended with the Teflon powder. This composition was molded and sintered according to the procedure outlined above and the molded piece was cooled in the oven. The final product was extremely weak. Even when the sintering time was increased from 30 min to 1 hr and finally to 2 hrs, the same weakness was apparent in the molded specimen. Using 20% by wt of glass fiber, (1/4 in. long chopped glass, water treated from Owens Corning) in place of the asbestos, produced a final sheet with the same weakness that was noted for the asbesto filled Teflon. According to a paper presented by R. R. Marshall and P. E. Pitt of Melpar, Inc. at the 1957 Biennial Electronic Materials Symposium, entitled, "High Temperature Properties of Filled Teflon", a 20% glass filler should increase the heat distortion temperature of Teflon under a load of 264 psi to 270°F, and a 10% by weight of asbestos fiber should increase this temperature to 275°F. (Unfilled Teflon has a heat distortion temperature under a load of 66 psi of 250°F). This paper however, made no mention of the final strength of the molded piece. From our experiments it was concluded that the final products were too weak to be considered for any structural purpose.

c) Polycarbonate

Attempts to determine the rigidity modulus at 400°F on a molded specimen of unfilled polycarbonate (General Electric's Lexan), failed due to the fact that the sample flowed from the test holder under its own weight at elevated temperature. Thus the rigidity modulus of this material at 400°F is much lower then the allowable 3.08×10^8 dyn/cm^2 (See Appendix B), and therefore this material is unsatisfactory for use in the manufacture of plastic cartridge cases.

d) and e) Polyethylene (Cross Linked) and Polyvinyl Chloride Plastisol

Neither a carbon black-filled cross-linked polyethylene nor a plastisol (Quelcor Inc.'s No. 2012), a polyvinyl chloride dispersed in a completely polymerizable plasticizer, succeeded in passing the Bending Beam Test at 400°F. These compositions had a rigidity moduli at 400°F lower than the calculated allowable 3.08×10^8 dyn/cm^2.

f) Modified Polychlorotrifluoroethylene

Genetron V.K. is vinylidene chloride modified polychlorotrifluroethylene. It was molded according to the procedure outlined by the manufacture, Allied Chemical Corporation, the mold temperature being 450°F. A hundred and ten grams of powder were placed into the cavity of the hot mold and molded for 2 minutes at 300 psi. The pressure was then increased to 833 psi and held for another 2 minutes. The sample was then removed from the mold and cooled rapidly by quenching in cold water. According to the manufacturer, this rapid cooling increases the strength of the material.

Its tensile strength at room temperature was found to be 3012 psi, its elongation widely scattered, ranging from 25.3 to 73.3 percent, and its rigidity modulus 3.7×10^9 dyn/cm^2. The B_p values (Charpy Impact) were also widely scattered, and in all cases high, running from 392 to 635 in lbs.

Its rigidity modulus (Beam) at 400°F, however, was well below the calculated allowable value and the extreme sensitivity to temperature makes the material unsatisfactory for cartridge cases.

Samples of Genetron VK containing 15% by weight of glass fibers, also failed to pass the Bending Beam test at 400°F. These glass filled samples were received in molded form, being molded by the manufacturer.

2.4 Thermoset Materials

As was mentioned in the previous section on thermoplastic materials, the screening of the thermosetting materials initially only involved measurement of the impact strength of these materials at room temperature because this is the property of thermosets which is most suspect in thin sections. The compositions behaving most favorably in this test were then checked for impact at -65°F and for shock resistance at room temperature. It was felt that screening beyond the tests mentioned above would not be necessary since thermosetting materials, by their very nature, have low coefficients of linear expansion, high rigidity at 400°F, and low moisture absorption. This approach was taken for all of the thermosets except the modified epoxys. The necessity of modifying an epoxy with polysulfides in order to increase its impact resistance, reduces its ability to withstand high temperatures. Therefore the epoxy-polysulfide materials were subjected to tensile testing at 165°F prior to investigating their impact strength.

a) Glass Filled Polyesters

A 10% glass filled polyester, Allied Chemical's Plaskon Alkyd PM 4, was molded at a mold temperature of 270°F, and under a pressure of 600 psi. The curing time was 60 seconds. The Charpy impact test was run at room temperature on the molded piece and the calculated average B_p value was 28.3 in. lbs.

The 30% glass filled Polyester, Allied Chemical's Alkyd 446, was molded at a mold temperature of 270°F, and a pressure of 1,666 psi. The curing time was 60 seconds. In the Charpy impact test, the specimen shattered, with a total length of crack of 1-3/8 inches. The impact energy, taking this length of crack into account, was 38 inch lbs.

The coefficient of linear expansion and the rigidity modulus (beam) at 400°F were determined on the 30% glass filled Polyester to give us an insight into the behavior of a thermosetting material at 400°F. Its coefficient of linear expansion was 3.5×10^{-5} cm/cm/°C and its rigidity modulus was 3.4×10^{10} dyn/cm^2. Its average modulus at room temperature was 8.8×10^{9} dyn/cm^2. The higher modulus at 400°F probably means that during the test, the sample cured further.

b) Polysulfide Modified Epoxy

Laboratory formulations were made containing Shell epoxies 828 and 834 modified with a polysulfide Thiokol LP3. These systems were cured for 45 minutes at 165°F using 8 parts of DMP 30 per 100 pts. of epoxy.

The epoxies are too brittle to warrant individual attention; hence the polysulfide was added as a flexibilizer. 1:1 and 1:2 ratios of LP3 to the epoxies were used; at the former ratio, the product was too soft. The material with the 1:2 ratio showed a reasonable rigidity at room temperature, 5.6×10^{9} dyn/cm^2, with a tensile strength of 6480 psi and a percent elongation of 13.9. This formulation, however, was extremely weak at 165°F. Attempts to increase this strength by adding glass fibre failed. Even with the addition of 25% by weight of glass fibre 801 no increase in strength at 165°F was noted. At room temperature, however, its tensile strength was increased to 8640 psi. The inability of these formulations to possess relatively high tensile strengths at elevated temperature was then attributed to the low molecular weight of the epoxies used.

Rather than devote valuable time to a set of experiments to determine the best epoxy material, the specific quantity of polysulfide, and the type and amount of curing agent to use, it appeared feasible to contact a firm familiar with the formulation of heat resistant epoxies. A technical data sheet was therefore requested from Emerson and Cummings, Inc., manufacturers of epoxy formulations. From the materials listed in this sheet, Stycast 2651 was chosen mainly because of its reported resistance to a continuous environment of 400°F, with the ability to withstand 500°F for intermediate periods. And unlike many of the other heat resistant epoxies reported upon in this technical brochure, Stycast 2651 cures in a fairly short length of time making it possible to be used in rapid production.

c) Epoxy Formulations

This material was molded and tested for impact strength at room temperature. The test specimens shattered during this test and gave a low average B_p value of 11.7 in. lb. The addition of 15% by weight of glass fiber 801 failed to increase this value, and the filled specimens also shattered under impact. Modifying the formulation so that it contained a 2.1 to 1 ratio of the Stycast 2651 and Thiokol LP3, and 15% by weight of glass fiber based on total resin content, gave a final cured product with an average B_p value of 93 in. lb. This composition, however, when broken by hand cracks easily with a "soggy" feel, indicating that the above high B_p value is misleading. Under this condition of weakness the formulation as such does not warrant further consideration, so a few experiments were run to determine the possible cause or causes of this "soggy" feel at break. These main lines of thought were investigated:

1. Poor molding resulting in a large amount of air entrapped with the specimen.
2. Poor wettability of glass fiber by the resins.
3. Weakness as a feature inherent to the addition of polysulfide rubber.

The actual specific gravity of molded sheet was determined and compared with the calculated specific gravity. The values were, for all practical proposes identical, showing that structure of the composition was free of voids.

The glass fibers were visually examined after mixing with the Stycast 2651, the Thiokol LP3, and a 2:1 ratio of the epoxy and the LP3; and in all cases the fibers appeared to be poorly wetted by the resins. Therefore each resin-to-glass interface is a possible point of weakness.

A composition composed of 2.1 pts of Stycast 2651 and 1 pt of LP3 was cured and tested for impact strength at room temperature, the resulting average B_p value being approximately 46 in. lb. This composition however, when broken by hand, gives the same "soggy" type feel that was mentioned previously.

It was therefore concluded that the weakness of the glass filled Epon-Thiokol formulation was an attribute of the mixture of these two polymers. An additional cause of weakness was the poor wettability of the glass by the resin.

In order to satisfactorily modify the Stycast 2651, it would be necessary to find a high temperature-resistant flexibilizer that would not lower the cohesive strength of the final product by a critical amount. Also attempts would have to be made to treat the glass fibers so they would be easily wetted by the composition. We found a commercial source for a flexible epoxy composition for which was claimed high impact strength at room temperature and stability at 400°F. The rigidity of this composition can be increased by adding glass fiber, if its "as received" flexibility is too great. Two such compositions were ordered from Emerson and Company, Inc. namely, Stycast 2340M and Stycast 2741.

The degree of flexibility of the Stycast 2741 could be varied by varying the quantity of catalyst added - the higher the catalyst content the greater the flexibility of the cured product. Table 9 shows the effect of catalyst content on the B_p impact values of the cured Stycast 2741.

Table 9

EFFECT OF CATALYST CONTENT ON THE B_p VALUES OF CURED STYCAST 2741

Formulation Number	Stycast 2741	Catalyst 15	B_p values
1	1 pt.	1 pt.	75 in. lbs. (too flexible)
2	2 pts.	1.5 pts.	42 in. lbs.
3	2 pts.	2.0 pts.	10 in. lbs. (too flexible)

The Bending Beam test at 400°F was run on the formulation containing 2 parts of Stycast 2741 to 1.5 parts of catalyst 15. This formulation fell from the holder under its own weight at this temperature and therefore did not meet our screening requirement. The addition of 10% by weight of glass fiber to this formulation did not enable the formulation to pass the Bending Beam test at 400°F. Both formulations were extremely flexible and weak at this elevated temperature. Formulations numbers 1 and 2 were then tested at 400°F and both failed for the same reasons noted above.

The Stycast 2340M, was cast, molded and cured overnight at 150°F as directed by the manufacturers brochure. The cured product was then subjected to the bending beam test at 400°F, where the material failed by falling from the holder under its own weight. Thus the Stycast 2340M lacked the required structural stability at 400°F and could not be considered for use in the manufacture of plastic cartridge cases according to the present specifications.

d) Filled Phenolics

Realizing from experience that unfilled Phenolics would be too brittle in thin sections to warrant attention in our screening procedures, no such material was tested. However, the following filled phenol formaldehydes were checked for impact strength at room temperature.

1. Woodflour filled (Bakelite's BMG-5000 BK)
2. Mineral filled (Rogers Corp., RX460)
3. Asbestos filled (Rogers Corp., RA4100)
4. Glass filled (Fiberite Corp. No. 4030-190)

The Woodflour, glass, and the mineral filled phenol formaldehydes were laboratory molded. The molding procedures that were followed were those that were outlined by the respective manufacturers, and they were identical in all cases. The mold was preheated to 325°F, filled with 30 grams of the molding powder, and pressed to a pressure of 3000 psi. The curing time was 1 minute.

The asbestos filled phenolic was received from the manufacturer in molded form.

The impact strengths of these materials are shown in Table 10.

e) Diallyl Phthalate (Orlon Filled)

The orlon filled diallyl phthalate was (Durez 16694) molded according to the molding procedure suggested by the maker. Molding was done at a mold temperature of 320°F under a pressure of 2000 psi for 2 minutes. The average Charpy impact value was found to be 2.7 in. lbs.

2.5 Impact and Shock Resistances of Thermosets

The B_p Impact values were determined for the thermosetting compositions, and are tabulated in Table 10.

The shock resistance test was then run on representative compositions, as shown in Table 11.

2.6 Additional Screening of Glass and Asbestos Filled Phenolics

The materials that behaved most favorably under the Charpy impact and the Shock Resistance Test, namely Fiberite 4030-190 and Roger's RX-490, were tested for impact resistance at a -65°F, using the drop

Table 10

B_p VALUES FOR FILLED THERMOSETS AT ROOM TEMPERATURE

(Nominal 1/16 inch Thick)

Material	Type	B_p Value
Alkyd PM4	10% glass filled polyester	28.3
Alkyd 446	30% glass filled polyester	38.0
RX-490	asbestos-phenolic	19.0
16840	nitrite rubber phenolic	10.6
14475	nylon flock phenolic	7.0
16694	orlon-diallyl phthalate	2.7
4030-190	glass-phenolic	23.2
BMG-5000 BK	Wood flourphenolic	11.6
RX 460	Mineral-phenolic	4.3
RA 4100	asbestos-phenolic	14.0

Table 11

SHOCK RESISTANCE OF THERMOSETS (1/16" THICK)

Material	Type	Sizes of hole (mm) Front	Back	Comments
4030-190	glass-phenolic	6	6	no cracks, hole extremely ragged
RX-490	asbestos-phenolic	6	9	no cracks, hole ragged
16840	nitrite rubber-phenolic	6	10	single radiating crack, hole sharp and clean
14475	nylon flock-phenolic	8	12	single radiating crack, hole sharp and clean
16694	orlon-diallyl phthalate	9	14	sample cracked in half

ball impact test as described in Appendix A. The B_b impact values of these compositions were also determined at room temperature thereby enabling us to note any decrease in impact resistance when compared to room temperature values. (See Table 12) In all cases the test specimens were a nominal 1/16" thick.

Table 12

ADDITIONAL PROPERTIES OF FIBERITE 4030-190 AND RX 490

Property	Fiberite 4030-190	RX 490
B_b impact value at room temp. (in. lbs)	10.0-13.4	10.0-13.4
B_b impact value at -65°F (in. lbs)	3.4-6.7	3.4-6.7
Coefficient of Linear Expansion (cm/cm/°C)	1.39×10^{-5}	1.00×10^{-5}
Rigidity Modulus at 400°F (dynes/cm^2)	4.5×10^{9}	6.5×10^{9}
Moisture Absorption	1.29%	1.99%

Although thermosetting materials generally exhibit low moisture absorption values, low coefficients of thermal expansion and high heat distortion temperatures, tests were run on the glass filled and asbestos filled phenolic to verify the behavior of these materials with respect to these properties.

The values of the coefficients of linear expansion, moisture absorption and the rigidity moduli at 400°F of these materials also appear in Table 11. These tests concluded the screening of the various thermosetting compositions under consideration and indications are that the glass filled phenolic Fiberite 4030-190 possesses the best physical properties for cartridge cases and therefore this composition was molded into cartridge cases.

3. CARTRIDGE CASE MOLDS

3.1 Injection Mold

With the approval of the Arsenal, Curley-Nelson Company, Inc. was awarded the task of designing and constructing an injection mold as well as the task of molding the required number of glass filled nylon cases, for use in assembly and firing studies. This mold was specifically designed to give a cartridge case with maximum allowable dimensions so that the amount of deformation of the case during firing would be minimized.

Also as a production requirement, the cartridge case had to be molded in two separate sections, the butt end and the cylindrical wall, with the pieces subsequently spinwelded or adhesively bonded. Figure 1 shows the original joint design which had to be modified after the first firing tests to increase the bond area. (See Figure 2.) We fully realized that the new bond design would decrease the volume capacity of the cartridge to some extent, but if after firing tests, the material appeared feasible for plastic cartridge cases a major mold change could be made so that the lap would be contained within the walls of the cylinder and thus the cartridge case would retain its volume capacity (Fig. 3). Latter work, which showed that a plastic butt could **not** withstand firing conditions, lead to a modification of the cylindrical wall so that it could accomodate a brass butt section with a tapered joint as shown in Figure 4.

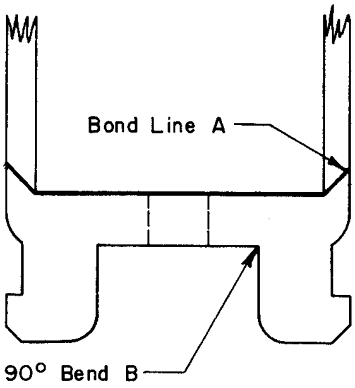


FIG. 1. ORIGINAL BOND AREA

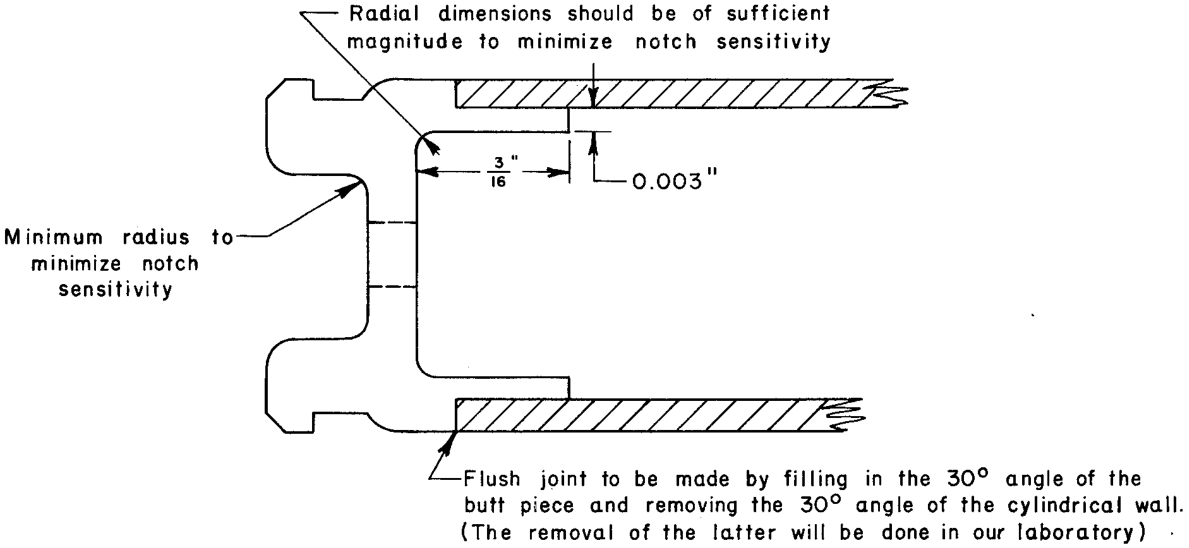


FIG. 2. NEW DESIGN OF BOND AREA

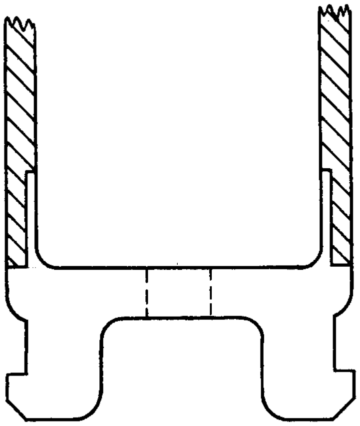


FIG. 3. RECESSED LAP JOINT

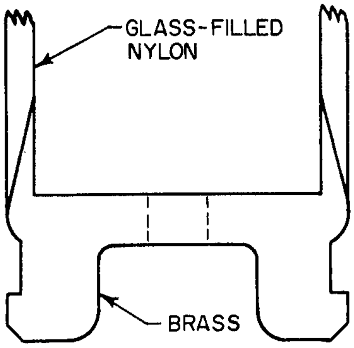


FIG. 4. NYLON-BRASS COMPOSITE ASSEMBLY

3.2 Transfer Mold

On a low bid basis Penn Plastics Company was awarded the task of designing and constructing a transfer mold, as well as the task of molding the required number of glass filled phenolic cartridge cases (Fiberite 4030-190). As with the nylon cartridge cases the phenolic cases were also molded in two sections, but being of a thermosetting nature, the material could not be spinwelded and the adhesive bonding was the only method utilized. Having already had some experience with the firing of glass filled nylon cases, this transfer mold was constructed to give a bond area as shown in Figure 3. As with the thermoplastic cartridge cases, the glass filled phenolic cartridges were molded with maximum allowable dimensions.

4. ASSEMBLY AND FIRING OF PLASTIC CASES

4.1 Glass Filled Phenolic

The Fiberite 4030-190 cases were transfer-molded in two separate sections - the cylindrical wall and the butt end.

Since the phenolic is a thermoset material, spin-welding could not be used to bond the two sections. Bondmaster M620, and epoxy type adhesive, was used for this purpose. During curing, the walls of the cases tended to blister slightly. If the phenolic cases had proven feasible it may have been necessary for maximum joint strength to investigate the curing conditions so that this blistering could be avoided.

The completed phenolic cases were subjected to the assembly procedure used for brass cases at the Frankford Arsenal and it was found that because of the high rigidity of the material a two-step primer insertion operation was required for successful insertion. Furthermore, crimping after the primer is inserted appeared from subsequent firing tests, to be unnecessary. It should be avoided because it will damage the butt.

The high rigidity also ruled out an interference fit between the projectile and the case, because the material will not stretch during the insertion of the projectile. The problem is readily solved by increasing the internal diameter of the neck by 2 to 3 mils over the present specification limits.

A half dozen cases containing the primer alone were fired with successful results - the cases appeared to be completely undamaged by the explosion of the primer. It will be noted that under similar conditions the butt ends of the unfilled and the glass filled nylon cases cracked, the former being damaged to a greater extent than the glass filled. Thus it appears that for successful firing the butt end at least must be made of a material with a high rigidity modulus.

The neck opening of these cases was increased 2 to 3 mils over the present specification limits. The cases were then hand loaded and the projectile adhesively bonded in place using Duco Cement. (A pull of about 25 lbs was required to remove the projectile from the case.)

These cartridges were then fired at the Arsenal. The primer and propellant both functioned although by no means at 100% efficiency and the butt section of the case was completely destroyed, while the cylindrical walls appeared undamaged. Observations on the fired cartridge cases showed that the entire adhesive joint between the butt and cylindrical wall failed and the question that arose was whether or not the butt piece would have been damaged if this bond had withstood the firing conditions. Various adhesive systems were then investigated, as noted below, to see if the adhesive strength of this bond could be increased.

Two adhesive systems for bonding glass-filled phenolic butts and brass butts to Fiberite 4030-190 walls were investigated. These systems were composed of an unmodified epoxy (Epon 828), and a polysulfide (Thiokol LP3) modified epoxy (Epon 828). Both systems were heat-cured using diethylene triamine as a curing agent, as per formulations listed below:

System No. 1

Epon 828	100 parts by weight
Diethylene Triamine	8 parts by weight
Cured 1 hour at 80°C	

System No. 2

Epon 828	50 parts by weight
Thiokol LP3	50 parts by weight
Diethylene Triamine	4 parts by weight
Cured 1 hour at 80°C	

Prior to bonding, the components were pre-cleaned for 5 minutes in an ultrasonic cleaner (Narda Ultrasonic Corp.) at a plate current setting of 50. A solvent combination of 2 parts trichlorethylene, 1 part n-heptane, and 1 part isopropyl alcohol was used as the cleaning solution. (Parts by volume.)

After cleaning, the parts were dried for 1/2 hour at 90°C and then immediately bonded and cured for 1 hour at 80°C.

Regardless of the adhesive used the phenolic butts adhesively bonded to the phenolic walls cracked badly allowing the gases to escape from the back before attaining maximum internal pressure. The projectile did not leave the barrel of the weapon and crack propagation was noted throughout the walls of the case.

Brass butt sections machined from regular brass cartridges cases were then used in lieu of the phenolic butts. These metal sections were bonded to the plastic walls using the same methods that were used for the phenolic to phenolic bond.

The behavior of these cartridge cases under firing conditions was once again independent of the adhesive system used. Projectile velocities of about 2633 ft/second (2603, 2606, 2614) were obtained. However, the cases were damaged, showing radial cracks above the butt section and severe cracking of the neck.

These results plus the behavior of the nylon cases under firing conditions (Section 4.2) indicate that although the butt must be made of a material with a high rigidity for successful firing, cylindrical walls of a thermosetting resin with a relatively high rigidity modulus will be severely damaged during firing. Therefore no further work with the Fiberite 4030-190 was undertaken.

4.2 Glass Filled Nylon Cartridge Cases

The assembly behavior of three experimental glass filled nylon cartridge cases with a joint area as shown in Figure 1, of Section 3.1 was evaluated at the Arsenal with the results showing that this material will lend itself to the primer and projectile insertion and crimping machine procedure presently used for brass cases. However, the welded joints of these cases were extremely weak suggesting that a variety of spinwelding and bonding techniques for joining the cylinders and butts would have to be investigated to determine a bonding method capable of producing maximum strength. Therefore, Curley-Nelson Company, Inc. was authorized to make up another 500 completed cases using the various cleaning and bonding procedures that they felt would significantly improve the strength of the joint. It was also suggested that some of these experimental cases be made with unfilled nylon butts since the glass fibers probably interfere with spinwelding.

Eight different lots - each lot differing in the techniques used to bond the cylinders and butts - were received from Curley-Nelson Company, Inc., and the strengths of these joints were determined in our laboratory. Table 13 lists the various bonding procedures and the corresponding bond strengths using a calculated cross sectional area of 0.041 inch^2.

It should be noted that Procedure No. 2 was the method used for bonding the original three experimental nylon cases mentioned previously. As can be seen from these data, the values for the bond strengths utilizing this procedure were extremely low, as was expected. Procedure No. 4 resulted in superior bond strengths (average 3386 psi) when compared against the other procedures used and therefore, the cases bonded according to this method were subjected to further testing at the Arsenal.

It was found that these cases would lend themselves to the assembly procedure presently used for brass cases with one exception - the primer insertion and subsequent crimping in brass cases is at present a one-stage operation at the Arsenal and under this condition the unfilled nylon butt cracks. However, the unfilled material will withstand a two-step operation, and according to the Arsenal this should present no major problem, since the majority of NATO countries are presently using the two-step operation on brass cases.

The assembled cases were then subjected to firing tests and under these conditions, the welded joints failed. The failure occurred as a result of the internal pressure built up in the ignited primer and the rate of failure was so rapid that the primer failed to ignite the

Table 13

BOND STRENGTHS OF NYLON JOINTS

Procedure Number	Procedure	Bond Strength (psi)
1	Butt Zytel 33, Body Fiberfil As molded, no cleaning Parts spin welded	585 24.4
2	Butt and Body Fiberfil As molded, no cleaning Parts spin welded	244 439 1,390
3	Butt and Body Fiberfil Cleaned in warm water & detergent before spin welding	1,345 854 2,930
4	Butt Zytel 33, Body Fiberfil Cleaned in warm water & detergent before spin welding	3,660 3,660 2,850
5	Butt and Body Fiberfil Cleaned with Tri-Clean before spin welding	1,620 1,463 1,463
6	Butt Zytel 33, Body Fiberfil Cleaned with Tri-Clean before spin welding	1,463 2,340 2,930
7	Butt and Body Fiberfil Cleaned with Tri-Clean before bonding	1,590 293 2,495
8	Butt and Body Fiberfil Cleaned with Tri-Clean before bonding	1,414 1,560 2,780

Note: Zytel is an unfilled Nylon. Fiberfil is a glass-filled Nylon.

main powder charge. Observations made on the cylinders and butts that were subjected to firing, indicated that the following modifications would have to be made:

(1) The butt would have to be made of glass filled nylon thereby increasing its rigidity, and

(2) The geometry of the bonded area would have to be modified to reduce the notch sensitivity of the molded product. As can be seen in Figure 1, the bond line A in the present design is in line with the 90° bend at point B and from observations, it appears as though this is the main location of failure.

As can be seen from the data in Table 13, if the cylindrical walls and butt end are both composed of glass filled nylon, the bond strength decreases sharply. To compensate for this the bonding area was increased by slightly modifying the mold so that the bonded joint appears as in Figure 2 of Section 3.1. This feature plus the incorporation of a small radius at the corners of the bottom of the primer housing should reduce the notch sensitivity to a minimum. The strength of spinwelded bond joints using the new design was then determined with the results showing that an average load of 195 lbs was required to destroy the spinwelded bond. This value is approximately three times higher than the load required to break similarly bonded glass filled nylon butts to glass filled cylindrical walls, using the original bond design.

Portions of the remaining components were adhesively bonded using Epoxy type adhesives, namely Eccobond 45 and Eccobond 55 from Emerson and Cuming Inc. The adhesives were applied to the bond area of the cylindrical walls and butts after these components had been either simply degreased in trichloroethylene (TCE) and oven dried (15 minutes at 80°C), or after degreasing, coating with a film of tetra isopropyl titanate (TPT) from a 2% solution in petroleum ether, and subsequently baked for 4 hours at 80°C. The results are shown in Table 14 and it can be seen that Eccobond 45 in conjunction with the surface treatment utilizing TPT resulted in the highest bond strengths.

Table 14

STRENGTHS OF ADHESIVELY BONDED CARTRIDGE CASES

Adhesive	Surface Treatment	Curing Conditions	Pounds load at break
Eccobond 55	Degreased in TCE Oven dried 15 min at 80°C	10% by weight Catalyst No. 9 Cured 64 hours at room temperature	205 165 155
Eccobond 55	Degreased in TCE Oven dried 15 min at 80°C, coated with TPT from 2% solution in petroleum ether Baked 4 hours at 80°C	10% by weight Catalyst No. 9 Cured 64 hours at room temperature	111 180 205
Eccobond 45	Degreased in TCE Oven dried 15 min at 80°C	50% by weight Catalyst No. 15 Cured 64 hours at room temperature	172 221 176
Eccobond 45	Degreased in TCE Oven dried 15 min at 80°C, coated with TPT from 2% solution in petroleum ether Baked 4 hours at 80°C	50% by weight Catalyst No. 15 Cured 64 hours at room temperature	>247 248 >242

Tetra isopropyl titanate is a titanium ester, which when deposited from dilute organic solutions and allowed to hydrolize in air or baked in an oven, adheres tenaciously to any substance containing a few polar groups. This film is capable of improving the adherence of two surfaces which normally adhere poorly. (For further information see DuPont's Technical Information Bulletin No. 3 on Titanium Organics.)

Attempts were made to determine if the adhesion of Eccobond 45 could be further increased (See Table 15) by carefully degreasing the components for 1/2 hour in a ultra-sonic bath of TCE. The effect of a rapid cure (1 hour at 90°C) as versus a slow cure (64 hours at room temperature) was also determined. It was learned that if the system was cured slowly a TPT film increased adhesion from 185 pound load for samples simply degreased in the ultra-sonic bath to 244 pound load for samples similar degreased and then coated with the adhesion promoter. However, if the system is cured rapidly, no significant difference is noted between the two surface treatments: 258 pounds load on the average for those that were only degreased and 243 pounds load on the average for those pre-coated with TPT.

Table 15

EFFECT OF SURFACE TREATMENTS AND CURE TIME ON ADHESIVE STUDY OF ECCOBOND 45

Adhesive	Surface Treatment	Curing Conditions	Pounds load at break
Eccobond 45	1/2 hour TCE-ultra-sonic bath dried 15 min at 90°C coated with TPT from 2% solution in petroleum ether Baked 3 hours at 90°C	50% by weight Catalyst No. 15 Cured 64 hours at room temperature	265 266 200
Eccobond 45	1/2 hour TCE-ultra-sonic bath dried 15 min at 90°C	50% by weight Catalyst No. 15 Cured 1 hour at 90°C	>250 250 275
Eccobond 45	1/2 hour TCE-ultra-sonic bath dried 15 min at 90°C	50% by weight Catalyst No. 15 Cured 64 hours at room temperature	184 185 187
Eccobond 45	1/2 hour TCE-ultra-sonic bath dried 15 min at 90°C coated with TPT from 2% solution in petroleum ether Baked 3 hours at 90°C	50% by weight Catalyst No. 15 Cured 1 hour at 90°C	190 270 270

Although the adhesively bonded joints gave higher bond strengths when compared against the spinwelded cases, it was decided to determine the firing behavior of the latter since even this procedure gave markedly higher bond strengths than the strength of previously fired cartridge cases. This procedure is also, at the present, better suited for quantity production.

Once again it was found that the cases would lend themselves to the assembly procedure presently used for brass cases provided a two step operation was used for the primer insertion and subsequent crimping.

The bullet pull test was run on one of the completed cases and it was found that a pull of less than 1 lb was required to remove the projectile from the case. This value should be in the order of 60 lbs and it was felt that if the internal diameter of the neck of the cartridge case were decreased, values approaching this value should be attainable. Such a solution also would have the favorable aspect of increasing the wall thickness of the neck of the cartridge case.

On firing a dual explosion was noted. The initial explosion apparently was caused by the ignition of the primer and the second explosion was caused by the ignition of the main propellant charge. Large quantities of smoke seeped from the rear of the firing chamber after each explosion and it seemed likely that the pressure from the ignition of the primer is sufficient to damage the case. The projectile only traveled about a 1/2 inch into the barrel.

Inspection of the fired cartridge case showed that the glass-filled butt end shattered, breaking away from the cylindrical insert which was still securely bonded to the walls of the case. This break occurred at the 90° notch shown in Figure 5. It was hypothesized from the behavior of these cases and those fired previously that the radial forces created by the ignited primer produce high hoop stresses in the butt section of the cartridge and the ability to withstand these stresses is greatly reduced at any notch-sensitive area. For this reason the injection mold was once again modified; a radius replaced the notch at A, the magnitude of radius B was increased. Spinwelded cases so modified were assembled in three lots:

1) Primed and with no charge
2) Primed and with low charge (40 grains)
3) Primed and with full charge

These cases were successfully assembled using a one step primer insertion operation in lieu of the two step procedure previously required for successful primer insertion. The use of the special primers originally designed for aluminum cartridge cases was considered because the walls of these primers do not expand as much as usual when fired, hence would cause less stress in the butt section of the cartridge. However, since these primers are oversize, redesign of the butt section would be required in order to accommodate them.

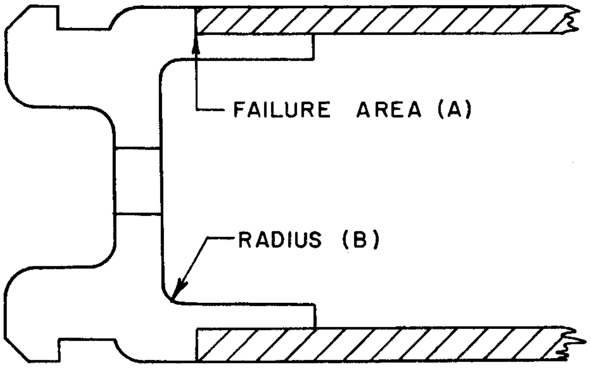


FIG. 5. AREA OF FAILURE

The primed cartridge cases containing no powder charge were fired to determine if the initial damage to the cases was caused by the ignition of the primer alone. Subsequent examination of the fired cases revealed that this was the case. On each occasion a single crack, originating in the butt and propagating into the walls, was observed. Measurements on the radial dimensions of the fired primers indicate that no permanent radial deformation occurs. Therefore, the primer material apparently behaves elastically during firing and it is believed that during its expansion cycle it creates hoop stresses in the butt piece causing failure. Two cases with a low charge (40 grains) were fired and both showed the same crack formation as was noticed in the cases where only the primer was fired and the projectile only traveled about two inches into barrel.

Realizing the need to decrease the susceptibility of the butt section to high hoop stresses it was decided to replace the glass filled nylon butt with ones made of (1) glass filled phenolic, Fiberite 4030-190, (2) brass. These butt sections were bonded to the glass filled nylon walls by degreasing the components in a mixture of n-heptane, isopropyl alcohol and trichlorethylene using the ultra-sonic cleaner. They were oven dried at 180°F, cooled in a dessicator, and then bonded using a 1:1 ratio by weight of Eccobond 45: Catalyst 15. Curing was done at 180°F for 1 hour.

The nylon/brass butt combination gave projectile velocities of about 2484 ft/sec (2400, 2432, 2500, 2512, 2575) with damage to the fired cases being mainly restricted to the area bonded to the brass butt. Although the strength of this bond in tension is greater than the strength of cylindrical wall at its thinnest cross section, the peel strength was markedly lower and the damage to the case was caused by peeling rather than by a failure in tension. Our solution to this problem was to invert our present system, that is, instead of having a brass collar fit <u>into</u> the nylon case, have the nylon case fit into the brass butt as shown in Figure 6. Then as the nylon expands radially the bonded joint is under compression and at the same time the nylon is supported by the outside brass butt.

Brass butts were machined as shown in Figure 7, so that the angle of taper for one lot was 4°15' and for a second lot the angle was 2°50'. The glass filled nylon cases were then machined to mate with these butts; these joints are also shown in Figure 7.

The machined components were then pre-cleaned in the ultrasonic cleaner and bonded using Eccobond 45 and Catalyst 15 in the same manner as mentioned previously.

These cartridge cases successfully passed the automatic deburring, priming and loading operations. No damage was noticeable, either to the nylon cylinders or to the brass butt pieces.

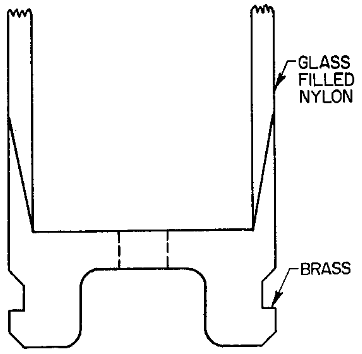
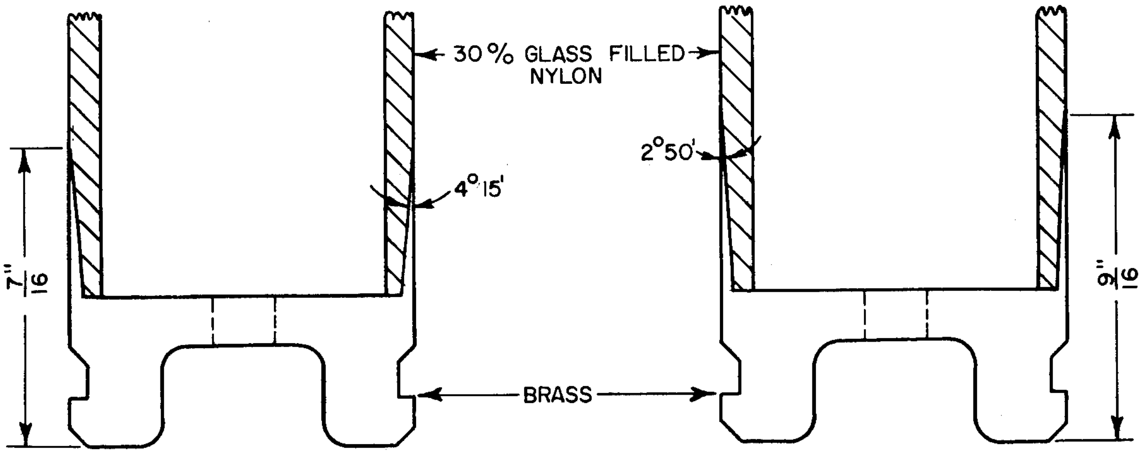


FIG. 6. MODIFIED BUTT JOINT

Table 16 lists the projectile velocities obtained during firing and the condition of the fired cartridge cases. From this table, it can be seen that little, if any difference in projectile velocities or in the condition of the fired cartridge cases exist between the two joint designs. In either case the projectile velocity is around 100 ft/sec lower than specification requirements, however it is felt that if the bullet pulls were increased a subsequent increase in projectile velocity could ensue. This could be accomplished by bonding the projectile in place, possibly with a thicker varnish layer. Either or both of the designs appear satisfactory for successful cartridge cases, but since the design with the 4°15' taper has a shorter brass butt section then the design with the 2°50' taper, the former design was used in the batch of 500 cartridge cases that was run through a series of tests at the Arsenal.



(a) 4° 15' TAPER

(b) 2° 50' TAPER

FIG. 7. NEW BUTT DESIGNS

Table 16

FIRING DATA

Taper on Brass Butt	Projectile Velocity ft/sec	Condition of Cartridge Case after Firing
2°50'	2632	No damage
2°50'	2623	Longitudinal crack 1/4" in length
2°50'	2632	Slight
2°50'	2623	No damage
2°50'	2630	No damage
2°50'	2635	No damage
2°50'	2636	No damage
2°50'	2634	No damage
4°15'	2637	No damage
4°15'	2624	No damage
4°15'	2634	No damage
4°15'	2635	No damage
4°15'	2632	No damage
4°15'	2639	Butt came off-no damage to cylinder
4°15'	2612	No damage

Although the cylindrical nylon walls with the machined taper withstood the firing conditions, the process of machining decreases the strength properties of the nylon. Therefore to insure maximum properties of the nylon the 500 cartridge cases that were to be tested were prepared by having Curley-Nelson Company, Inc., modify the present injection mold to include the necessary taper. The 4°15' taper on the brass butt section was machined by our shop. These cases were primed and loaded without mishaps and then subjected to tests at the Arsenal to accurately determine velocity, waterproofness, and chamber pressure. Values for these tests appear in Tables 17 and 18, along with notations concerning the behavior of the plastic/brass cartridge cases after firing.

Attempts to automatically fire these cartridge cases in the M-14 rifle failed due to the fact that the butt ends were pulled off during the extraction process. The cases also showed a single longitudinal crack along the plastic wall. This failure along with the failures noted in Tables 17 and 18 are not in line with the firing data obtained in the previous firing tests (Table 16). Modifying the injection mold to include the taper at the back section of the cylindrical wall necessitated modifying the "gate", so that the glass filled material is forced into the mold through a smaller orifice. This change in conditions adversely affected the physical properties of the final molded piece.

From information gained in previous firing tests it is felt that if the molding conditions had remained the same, the above mentioned failures would not have occurred. While further minor development work may be required, it is seen that the glass filled nylon/brass butt combination is feasible for use in the manufacture of 7.62 mm cartridge cases.

It should be noted that one of the above cartridge cases was chambered in a machine gun that had just completed firing 400 rounds of brass ammunition at rapid fire. This case was left in the chamber for 5 minutes. The round did not cook off and no deformation of the case was noted.

5. HIGH TEMPERATURE REQUIREMENTS FOR PLASTIC CARTRIDGE CASES

There are three conditions which must be met by the plastic cartridge case at high temperatures, when following rapid fire, a cartridge is chambered:

a) When the powder grains reach 320°F, they will cook-off, and the case at whatever its temperature then, must withstand the explosion.

Table 17

VELOCITY AND VACUUM

Propellant

Type - W 846
Army Lot - 43045
Charge - 46.5
Primer No. 34 - J 439

Test - Velocity and Vacuum

Object - Durability of Plastic Cases

Ammunition

Type - M80
Caliber - 7.62 mm
Bullet - Ball
Case - Plastic

Rec/Univ No. 28
BRL No. R-89
TRFD 1310

Stored - +70°F
Fired at - +70°F

Shot No.	Reference		Vacuum Test
1	2740	2701 C	O.K.
2	2747	2675 B	O.K.
3	2730-	2688 A	5 bubbles in transit from mouth of case
4	2741	2679	O.K.
5	2737	2683 B	3 bubbles in transit from mouth of case
6	2740	2701+A	O.K.
7	2759+	2685	O.K.
8	2756	2694 C	O.K.
9	2737	2701	O.K.
10	2740	2677 A	3 bubbles in transit from mouth of case
		2667-B	O.K.
		2693	O.K.
		2693	3 bubbles in transit from mouth of case
		2680 C	O.K.
		2683 C	O.K.
		2685 C	2 bubbles in transit from mouth of case
		2690	slow leak at mouth of case
		2692 C	O.K.
		2693 B	O.K.
		2690	O.K.
Total	27427	53750	
Mean	2743	2688	
CF		+7	
Cor'd		2695	
Ex Var	29	34	
Std Dev		15	

Casualties - (A) Base cup separated from case on extraction
(B) Splits in case and case/cup separation-on-extraction
(C) Splits incase

Ref RD Data

Auth - Eptom-7-60
Case Lot - 461
Bullet Lot - 467
Propellant - 42840
Jar - X, Charge - 47.1
Vel Val - 2750

Remarks - Most rds. hard to chamber
Case separation on extraction
Plastic body of cane extracted with ease

Chrono. 4
Range - 8

Proof Technicians - Kaminski (C) - Fickenschey (G)
X.O. - 84654-01
Date - 26 October, 1960

FRANKFORD ARSENAL
Engineering Proof Testing Record

Table 18

PRESSURE (CHAMBER) AT VARIOUS TEMPERATURES

Propellant

Type - W.846
Army Lot - 43045
Charge - 46.5
Primer No. 34 - J 439

Test - Pressure (Chamber) at Various Temperatures
Object - Durability of Plastic Cases

Ammunition

Type - M80
Caliber - 7.62 mm
Bullet - Ball
Case - Plastic

Rec/Univ No. 28 BRL No. G-152 TRFD 450

Stored - +70°F, -40°F, +125°F
Period - 2 hours
Fired at - above

	Reference		Ambient +70° ±2°		-40° + 0 -45°		+125° + 0 +130°	
Shot No.								
1	2731	42000+	2670	36700 A	2579 B	32900	2655 A	35800
2	2727	39200	2688+	39500+C	2618 D	34800	2669 A	40500
3	2723	39200	2677	37700 D	2645 B	36700+	2666	37000
4	2732+	41100	2657	38000 A	2618 B	35100	2680	39200
5	2729	39500	2653	36700	2605 B	32600	2649 A	37400
6	2713	38900	2627-	34800-	2634 B	34200	2658 B	37400
7	2728	40800	2683	38600 C	2630 B	34800	2667 B	39500
8	2729	40500	2655	36100 A	2623 B	34500	2660 A	35400-
9	2720	41100	2669	38600 A	2612 B	34200	2652 A	36700
10	2712-	37000-	2648	36700 B	2573 B	32300	2687 A	39500
			2664	38300 A	2599 B	33300	2664 A	39200
			2670	36700 A	2617 B	34200	2676 B	38300
			2663	37400 B	2595 B	31400-	2690	36700
			2678	39500 B	2640 B	34800	2698+A	41100+
			2676	39500 B	2730+B	33600	2680 A	38000
			2636	35400 A	2570-B	32900	2625-B	37000
			2666	36700	2605 B	33300	2671 A	37400
			2686	38600 D	2637 D	35100	2691	38900
			2655	38600	2625 B	34800	2667 A	37700
			2675	37400	2610 B	33600	2672 A	37400
Total	27244	399300	53296	751500	52365	679100	53377	760100
Mean	2724	39900	2665	37600	2618	34000	2669	38000
CF			-13	-1500	-13	-1500	-13	-1500
Cor'd			2652	36100	2605	32500	2656	36500
Ex Var	20	5000	61	4700	160	5300	73	5700

Casualties - Most cases hard to chamber and extract
(A) Base cup separated from case on extraction
(B) Separation of case and base on extraction and splits
(C) Small crack in case at base cup connection - Chrono. 4, Range - 8
(D) Split in case from drill hole to base cup

Ref RD Data

Auth - Eptom-7-60
Case Lot - 461
Bullet Lot - 467
Propellant - 42840
Jar - X, Charge - 47.1
Vel Val - 2711
CP Val - 38,400 psi

Proof Technicians - Kaminski (C) - Fickenshey (G)
X.O. - 84654-01
Date - 25 October, 1960

FRANKFORD ARSENAL
Engineering Proof Testing Record

b) Immediately thereafter, the case must be withdrawable in one piece under 60 pounds load.

c) The plastic surface in contact with the hot gun chamber walls must not foul or adhere to the chamber walls.

Our specifications in this project assume that the plastic might be exposed to 400°F for 15 minutes, and in this time must not foul the gun or fail to obdurate. The new insight into the problem, contributed by Dr. G. P. Wachtell, Chief of our Heat Transfer and Fluid Mechanics Branch, is that if the cartridge were chambered at 400°F, it would cook-off almost immediately, and the average temperature in the plastic would be well below 400°F at the time of cook-off and obduration. Thus his calculations show that if the cartridge, initially at 130°F, is chambered at 400°F, when the inside temperature of the case reaches 320°F, the temperature half way through the case wall will be 352°F. Similarly, if the initial case temperature were 0°F, the temperature half-way through the wall at cook-off would be 340°F.

Accepting the specification provision that the outside of the cartridge case will be subject to 400°F, and using the figure for cook-off temperature of the powder as 320°F, we can now see that on firing and obduration, half the thickness of the plastic case will be at 352°F or less. Hence, requirement a) and b) above say that at 350°F the strength of 1/2 the wall must be adequate to permit the case to fire and obdurate. Requirement c) is that the plastic must not become gummy or adhere when its outermost surface reaches 400°F. This is a surface problem rather than a strength problem, and could be attacked if necessary by surface treatments such as chemical treatment, varnishing, exposure to radiation, etc.

We believe it has been a proper research direction to use the most heat-resistant plastic available, to provide all possible margin of safety. The present study, however, suggested that thermoplastic materials other then those that passed the bending beam test could conceivably be feasible for plastic cartridge cases provided they retained adequate strengths at 350°F.

Two such materials, Lexan and Nylatron G.S., were injected molded into cartridge cases and successfully assembled at the Arsenal for firing studies. The assembly procedure, however, required the same modifications as were required with phenolic cases, namely, a two step primer insertion operation and by-passing the primer-crimping operation. It was noted that in some cases the bond strength between the cylindrical wall and the butt was weak since some of the cases broke in this area during assembly.

Difficulty was encountered in attempting to fire the above mentioned cases. Apparently the lack of rigidity of these materials allows the primer to "give" under the impact of the firing pin and the primer fails to ignite. In a few cases however ignition of the primer was accomplished and it was found that both materials behaved similarly to the glass filled nylon cases that were fired earlier in the project. The butt piece cracked under the pressure built up by the primer detonation and the main powder charge failed to ignite. These results seem to verify the fact that for successful firing the butt end at least must be made of a material with a high rigidity modulus.

6. RECOMMENDATION AND CONCLUSIONS REGARDING PLASTIC CARTRIDGE CASES

High density polyethylene has been used for .22 caliber pistol cartridge cases and high impact polystyrene has been used for the cylindrical portion of 105 mm cartridge cases. Neither of these materials could meet the temperature requirements of the 7.62 mm NATO cartridge cases, nor was there any experience to show that they could survive the firing conditions and pressure build-up in the NATO rifles and machine guns.

This project has established the feasibility of a cartridge consisting of a brass butt adhesively bonded to a glass-filled, heat-resistant Nylon cylinder. Such a case is practical to manufacture, satisfactory to prime and load, and exhibits velocities and pressures comparable to all-brass cartridges under all firing conditions so far reported. The two points that need further attention are 1) the bullet pull and seal, which can be brought into line by reducing the internal diameter of the case at its neck, and the 2) the bonding and welding at the butt end of the cylinder, both of which can be improved through a limited development program.

The composite case weight 6.2 g as compared with 11.4 g for the all-brass case-a savings of 45.6%. Its cost, figured as shown in Table 19 is $25.06/K, compared with $40.95/u for the all-brass case-a savings of 38.8%.

Table 19

ESTIMATED COST FOR 1000 CARTRIDGE CASES

		Composite				
	All-Brass	Butt	Cylinder	Bonding	All Plastic	
Material	18.35^9	7.25^2	5.77^3	$.31^5$	Cylinder	6.27^7
Process	22.60^9	8.91^1	$.50^4$	2.32^6	Butt	4.10^8
					Bonding	2.63^7
	40.95	16.16	6.27	2.63		13.00
			25.06			

Notes

1. Draw operations assumed rather than screw machine.
2. Figured on proportionate weight basis from figures for all-brass case.
3. 3.4 lbs at $1.75 in 10 ton lots of Fiberfil G-2.
4. Injection molded in 16 oz press, at rate of 24000 per hour, based on Curley-Nelson figures.
5. Figuring 0.1 g adhesive per joint, plus 12% waste.
6. Allowing .32/K for ultrasonic cleaning, and $2.00/K for automatic bonding and curing (Design principles for the machine have been visualized).
7. Same as for composite.
8. The plastic cylinder weighs 1.6 g, the butt 1.0.
9. Frankford Arsenal figures.

In figuring the cost of the composite case, we assumed that the butt piece would be made out of brass, with one drawing operation. The shape of the piece, however, lends itself to screw machine or lathe-type of milling, hence the piece probably could be made of steel. Depending on production facilities and volume, a steel piece, so cut, might be cheaper than a drawn brass piece.

If an all-plastic case were able to withstand firing, it would yield a significant improvement in weight and cost over even the composite case. As Table 19 shows, its cost would be 13.00/K and its weight would be 2.7 g/k, representing savings over the all-brass case, of 68.5% and 76% respectively. Similar savings over the composite case would be 48.1 and 56.5%.

An all-plastic case adhering to the same internal and external dimensions as a brass cartridge case, is not feasible using any presently-known plastic materials. There are, however, ways for improving the situation without changing the external dimensions of the cartridge, or its ballistic characteristics. Two of them are as follows:

a) When more powerful propellant becomes available, much more plastic could be incorporated in the butt section to give it the resistance to cracking that it now lacks. With present propellant, the volume required by the additional plastic cannot be made available except by reducing the volume of powder and adversely affecting ballistic properties.

b) If the sidewalls of the primers could be given hoop rigidity so the primer would not expand radially when fired, present destructive stresses in the butt would be minimized and a present butt would have a much better chance to survive. As an alternative to this, a ring insert could be molded into the butt, to contain the primer.

With the feasibility of the composite cartridge case reasonably demonstrated, certain development tasks remain before mass production can be undertaken. These are:

a) Find the best way to gate the injection mold, to obtain best strength in the butt end of the cylinder piece. Other changes in molding conditions (rate of cooling, for instance) should also be optimized.

b) Develop a faster-curing adhesive, to cut production time and temperature. Consideration should be given to roughening or otherwise treating the mating surfaces, to enhance peel strength and shock resistance.

c) Determine the optimum internal radius for the neck of the cylinder, to give the desired bullet pull strength and complete waterproofness.

Edmund Thelen

E. Thelen, Manager
Colloids and Polmer Laboratory

Approved by:

Nicol Smith

Nicol H. Smith
Director of Laboratories

APPENDIX A

SCREENING PROCEDURES

1. Coefficient of Linear Expansion
2. Rigidity Modulus and Permanent Deformation in Tension at 400°F
3. Rigidity Modulus and Permanent Deformation of a Beam at 400°F
4. Charpy Impact Test
5. Drop Ball Impact Test
6. Tensile Strength, Percent Elongation, and Rigidity Modulus under High Load at -65°F, Room Temperature, and 165°F
7. Compatability - Three Days at 165°F
8. Moisture Absorption
9. Shock Resistance

APPENDIX A

SCREENING PROCEDURES

1. Coefficient of Linear Expansion

Three specimens are cut from a molded sheet, approximately 1/16 in. thick, to a length and width of 6 in. by 1/4 in., respectively. Each sample is then marked across its width 3/4 in. from each end so that the distance between the two marks is approximately 4.5 in. A small hole to accomodate a thermocouple is drilled normal to the major axis into the thin side to a depth of approximately 1/8 in. This hole should be located midway between one of the markings and the end of the sample closest to the marking (Fig. 8). The dimensions of the sample, including the distance between the markings are now measured accurately to 0.01 cm. The thermocouple wire is then inserted into position.

The sample is suspended vertically in an oven operating at 400°F (204°C) and having a glass window which allows the amount of thermal expansion between the two marks to be measured with a cathetometer located outside the oven.

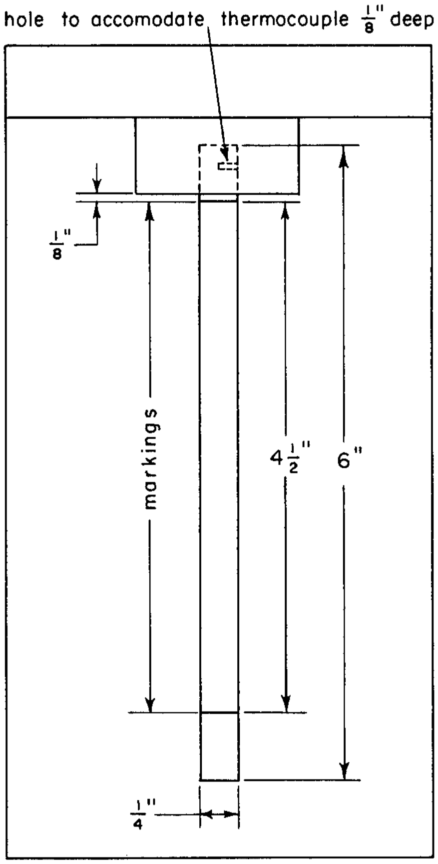


FIG. 8. SPECIMEN FOR COEFFICIENT OF LINEAR EXPANSION
(Cut from Nominal 1/16" Sheet)

The sample is suspended by means of a clamp that is positioned approximately 1/8 in. above the upper marking. The lower end of the specimen is left free. When the specimen reached 400°F, determined by means of the thermocouple in conjunction with a potentiometer, the distance between the markings is measured with a cathetometer to 0.01 cm. (The time required for the specimen to reach 400°F should be noted since this time interval will be of importance in the bending beam test.) The difference between this length and the original length is the amount of extension caused by thermal expansion. From this information, the coefficient of linear expansion can be calculated from Equation (1):

$$C_{LE} = \frac{\Delta L}{L\Delta T} \qquad (1)$$

where

C_{LE} = coefficient of linear equation (cm/cm/°C).
ΔL = change in length (cm).
L = original length between marks (cm).
ΔT = change in temperature (°C).
(204°C minus room temperature).

The thermocouple method for determining the temperature of the specimen can be eliminated after running the first sample, if the length of time required to bring the specimen to temperature is noted, and all subsequent samples are heated for this length of time.

2. Rigidity Modulus and Permanent Deformation in Tension at 400°F

The sample size, preparation, and measurement are identical to those used for determining the coefficient of linear expansion and the upper portion of the sample is clamped in the same manner as depicted in Figure 8. In this test, however, a total potential weight of 3 lb is clamped to the bottom portion of the specimen, but is supported by a wire so that it does not stress the sample; the clamp being positioned approximately 1/8 in. below the bottom mark. When the specimen reached 400°F, determined by means of the thermocouple in conjunction with a potentiometer, the 3-lb load is applied to the specimen by opening the oven door. This permits excellent temperature control. The amount of elongation between the marks under a load of 3 lb and a temperature of 400°F is measured with a cathetometer to 0.01 cm at 3-minute intervals over a 15-minute period. At the end of this time the load is removed and the sample is taken from the oven and cooled at room conditions. The amount of recovery is measured with a cathetometer to 0.01 cm at 3-minute intervals over a 15-minute cooling period. The amount of permanent deformation in percent is calculated from Equation (2).

$$\%\ \text{Permanent Deformation} = \frac{\Delta L}{L} \times 100 \qquad (2)$$

where

ΔL = change in length between marks after cooling (cm).
L = original length between marks (cm).

<u>Note</u>: ΔL can be negative, indicating shrinkage. The Rigidity Modulus is calculated from Equation (3):

$$E = \frac{P x L}{\Delta L} \qquad (3)$$

where

E = Rigidity Modulus (dyn/cm^2).
P = Load (dyn/cm^2).
L = Length between marks (cm).
ΔL = Change in length between marks at 400°F under load for 15 minutes (cm).

3. Rigidity Modulus and Permanent Deformation of a Beam at 400°F

The specimen size for this test is identical to that used to determine the coefficient of linear expansion. In this case, however, no hole is drilled for a thermocouple, and the distance between the markings on the specimen is reduced to 3.5 in., and a third mark is made directly on the center of the specimen.

The sample is positioned horizontally, with its outer marks resting on knife edges (Fig. 9), and this system is placed in an oven set for 400°F. This oven should also contain a window.

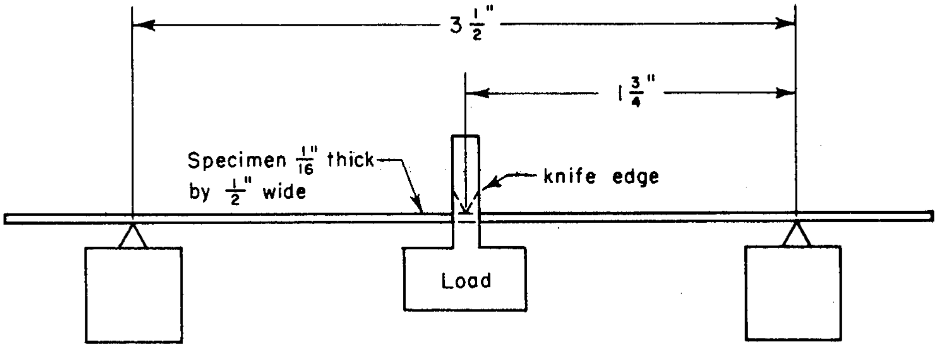


FIG. 9. BENDING BEAM TEST

When the specimen has reached 400°F (determined by measuring the time the specimen is in the oven. The necessary time required for the specimen to attain the desired temperature has been determined previously), a 56.5 g load is saddled over the center mark on the sample. This load has a knife edged rest which bears on the specimen.

The amount of deformation is then measured by means of a cathetometer to 0.01 cm at 3-minute intervals over a 15-minute period. At the end of that time, the load is removed and the sample is taken out of the oven and cooled at room conditions. The amount of recovery is checked with a cathetometer to 0.01 cm at 3-minute intervals over a 15-minute cooling period.

Appendix B shows how limiting values for this test were arrived at.

The Rigidity Modulus, E, is dependent on the moment of inertia, I, which is calculated by Equation (4):

$$I = \frac{bd^3}{12} \qquad (4)$$

where

I = moment of inertia
b = breadth (cm)
d = thickness (cm)

and Equation (5) determines the Rigidity Modulus in dyn/cm^2:

$$E = \frac{kWL^3}{D_m I} \qquad (5)$$

where

$k = 0.0208$
W = load in dynes (980 x g)
L = length between knife edges holding specimens (cm)
D_m = deflection (cm)

The amount of permanent deformation is calculated from Equation (6):

$$\% \text{ Permanent Deformation} = \frac{D_p}{D_L} \times 100 \qquad (6)$$

where

D_p = amount of permanent deformation (cm)
D_L = amount of deformation after 15 minutes under load at 400°F

4. Charpy Impact Test

The actual apparatus used for this test is the Tinius Olsen Impact Tester which conforms to ASTM specification D256-47T. A sample 5-1/4 in. long 1/2 in. high, and 1/16 in. thick is supported horizontally by means of two tight-fitting clamps lined with emery cloth. The length of the sample between the clamps is 3-1/2 in. The specimen is notched directly in the center to a depth of 1/8 in., with an included angle of 45° (Fig. 10).

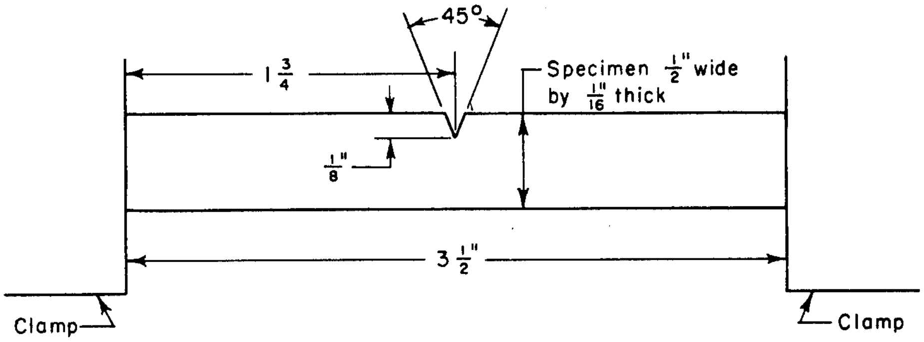


FIG. 10. IMPACT SPECIMEN

From the dimensions of the sample and the scale reading from the Tinius Olsen Tester, the breaking energy, B_p, is calculated from Equation (7). Due to the large size of the test apparatus, this test is run at room temperature only.

$$B_p = \frac{PL}{b} \qquad (7)$$

where

B_p = breaking energy (in. lb)
P = scale reading (in. lb)
L = length of beam between clamps (in.)
b = height of sample from bottom of notch (in.)

In comparing the B_p values of one plastic composition against the values of another, it is important that the specimens possess the same thickness.

5. Drop Ball Impact Test

This test is used, primarily, to determine the impact strength of plastic compositions at -65°F.

A one-pound steel ball is dropped from an electromagnet onto the sample from progressively higher heights until the sample cracks. This is done in an enclosed chamber that has been constructed to permit a temperature control of ± 0.5°F.

The sample is 1/16 in. thick, 1/2 in. wide, and the length of the sample between the clamps is 2 in. The specimen is notched in the same manner as defined for the Charpy Impact Test.

During the test, the specimen is clamped in a horizontal position with its width parallel to the base of the tester. Since it is difficult to visually determine the initial cracking of the specimen, without actually opening the test chamber, the bottom surface of the sample is coated with a thin layer of a silver-filled epoxy (Hysol 6250). With the aid of a Triplet meter it is now possible to determine the initial cracking by observing a marked incrase in the resistance of the circuit.

The breaking energy, B_b, is then calculated from Equation (8):

$$B_b = \frac{Whl}{b} \tag{8}$$

where

B_b = breaking energy (in. lb)
W = weight of ball (lb)
h = height of drop at break (in.)
l = length of specimens between clamps (in.)
b = width of sample from bottom of notch (in.)

Once again, in order to compare the B_b valves of one plastic composition against the values of another, it is important that both specimens possess the same thickness.

6. Tensile Strength, Percent Elongation, and Rigidity Modulus under High Load at -65°F, Room Temperature, and 165°F

The tensile test specimen is the usual dumb-bell shape with a central cross sectional area of approximately 1/64 in.^{2} The length of the sample between the jaws of the tester is 3 in.

The sample is pulled in tension at a loading rate of 1 in./min, until break or until the applied load reaches the maximum range of the tester (300 lb). A stress-strain curve is recorded in conjunction with this process.

This test is run at -65°F, room temperature, and 165°F, with a maximum temperature variation of ±0.5°F.

From the stress-strain curves and the dimensions of the specimen, the following values are calculated:

1. Tensile Strength (psi)
2. Elongation (%)
3. Rigidity Modulus, from Equation (9)

The Rigidity Modulus is actually a measurement of the slope of the stress-strain curve measured along the linear and regular section of the curve.

$$E = \frac{PxL}{\Delta L} \times 68{,}946 \qquad (9)$$

where

E = rigidity modulus of rigidity (dyn/cm^2)
P = Load (psi)
L = original length between clamps (in.)
ΔL = change in length (in.)

7. Compatability - Three Days at 165°F

Three tensile specimens are cleaned by gently rubbing with cheese cloth saturated with a surface active agent such as Trition X-200 (Rohm & Haas). They are then rinsed with hot distilled water, air-dried, and then desiccated for 24 hours.

At the conclusion of this time they are weighed to 1 mg and the contact angle of water on the plastic surface is measured. The specimens are then covered on all sides with the propellant and the system is placed in an oven for three days at 165°F.

At the end of this test cycle, the samples are cooled in a desiccator and then reweighed and their loss in weight calculated as a percent. Any changes in appearance due to the heating cycle are observed and recorded. The contact angle is also measured and any change from the original value is noted. Such a change indicates a difference in the surface energy of the plastic composition which in turn indicates surface degradation. From a concise discussion on the meaning of the contact angles of liquids on solid surfaces, see Gregg, S.J., "The Surface Chemistry of Solids", New York, Reinhold Publishing Corp., pp. 183-204 (1951).

The tensile strength and stress-strain curves are run at room temperature on the treated specimens, and the following values are calculated:

1. Tensile Strength (psi)
2. Elongation (%)
3. Rigidity Modulus, from Equation (9)

8. Moisture Absorption

A plastic specimen nominally 1/16" thick with remaining dimensions of 1/2" x 3" is brought to constant weight in a vacuum oven operating at 50.0°C. The thickness of the sample which has been weighed to 0.001 gram is then measured to a thousandths of an inch and then immersed in distilled water for 72 hours at room temperature. At the end of this time the specimen is blotted dry, re-weighed, and its thickness remeasured. The percent gain in weight and the percent increase in thickness are then calculated. The specimen is then reimmersed in distilled water, at room temperature for additional 72 hour periods until no dimension and weight change is noted.

9. Shock Resistance

A sample nominally 1-1/4 inches in width and 5 inches in height is rested vertically (unclamped) against a wooden frame as shown in Figure 11. A .22 caliber (long) shell is then fired at the specimen from a revolver positioned 7 inches from the test sample. The effect of the high speed projectile on the sample is then noted in terms of size and raggedness of hole, crack patterns and the like.

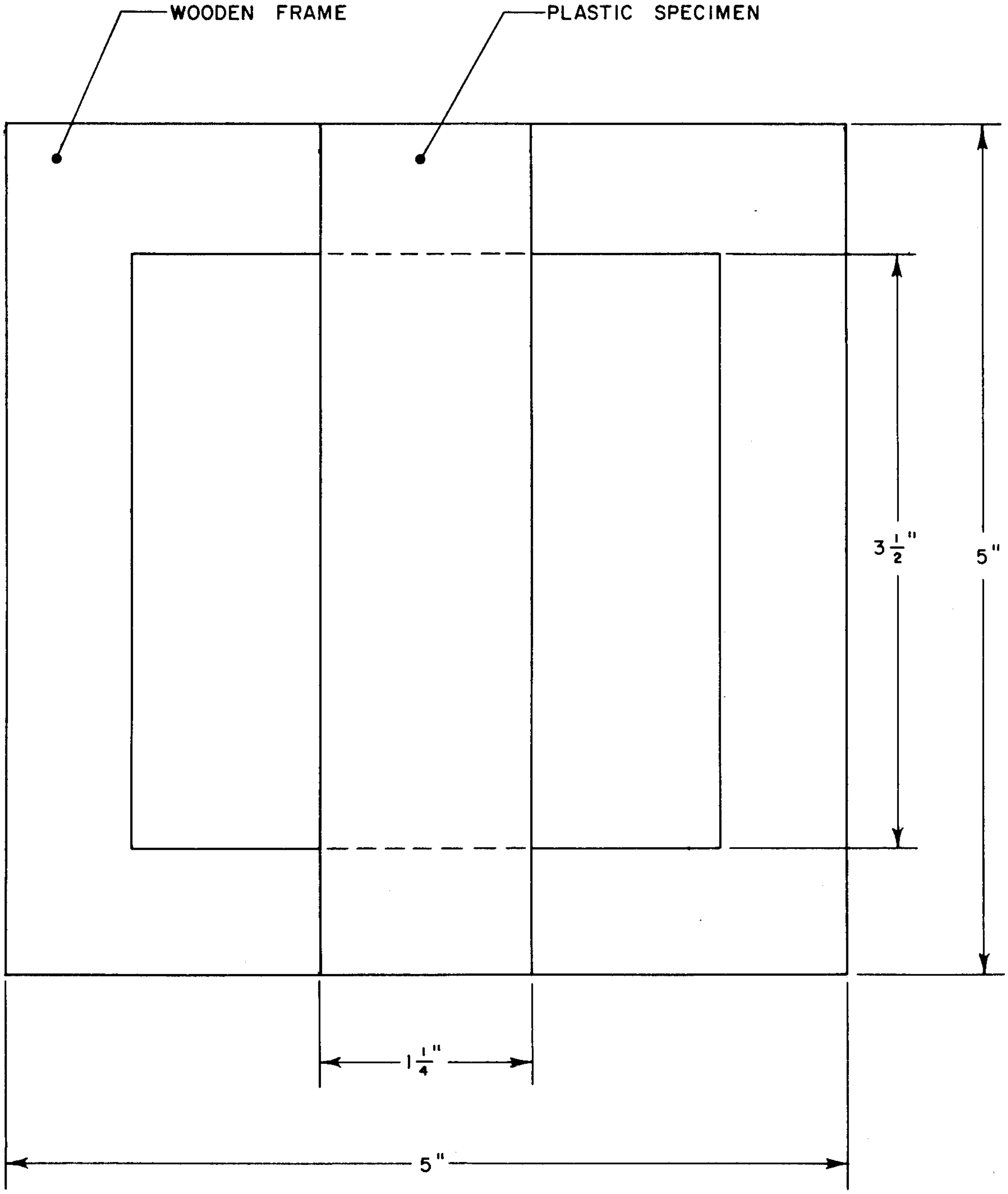


FIG. 11. SPECIMEN HOLDER FOR SHOCK RESISTANCE TEST

APPENDIX B

CONDITIONS FOR THE HIGH TEMPERATURE TEST

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CONDITIONS FOR THE HIGH TEMPERATURE TEST

The cartridge is to withstand 400°F for 15 minutes. Ideally, it should not expand more than 0.001 inch in diameter. We wish to test the corresponding performance of plastic ribbons by a single tension test. What loads shall we use, and what deformation is permissible? Furthermore, what magnitude of complex modulus corresponds to the desired performance?

A. Load

For a thick walled cylinder pressurized inside. Hoop tensile stress

$$S_T = \frac{P_i(r^2_i + r^2_o)}{r^2_o - r^2_i} \tag{1}$$

and the longitudinal stress

$$S_{TL} = \frac{P_i r}{2t} \tag{2}$$

when

P_i = internal pressure, psi

r_i = internal radius

r_o = external radius

t = thickness of wall

r = average radius

Assuming that P_i is due to expansion of gases when the cartridge is heated from 75°F to 400°F,

$$P_i = \frac{400 - 75}{460 + 75} \times 14.7 = 8.9 \text{ psi} \tag{3}$$

For the neck of the cartridge

r_i = 0.151 inches

r_o = 0.168

t = 0.017

Substituting in (1) and (2)

$$S_T = 83.0 \text{ psi}$$

$$S_{TL} = 83.5 \text{ psi}$$

The load to use, then is 83 lbs - cross-section of one square inches.

B. <u>Strain</u>

The limiting strain corresponds to an increase in radius of the neck of the cartridge, of .001 inches.

$$\text{Strain} = \frac{d}{r_o} = \frac{.001}{.168} = .006 \qquad (4)$$

Permissible stretch, then, is .006 x length of ribbon.

C. <u>Modulus</u>

Radial displacement, d, for a metal thick cylinder internally pressurized:

$$d = \frac{r_i p_i}{E}\left(\frac{r_i^2 + r_o^2}{r_o^2 - r_i^2} + \right) = \frac{13.2}{E}$$

when

μ = Poisson's ratio (0.5 for Plastics)

E = Young's Modulus

Using the time dependent complex shear modulus G, for plastics, with G = 1/3 E, and substituting in (5).

$$G = \frac{13.2}{3d} = 4400 \text{ psi or } 3.08 \times 10^8 \text{ dynes/cm}^2 \qquad (6)$$

D. <u>Conclusions</u>

For a ribbon 1/16 in. x 1/4 in. x 5 in., at 400°F for 15 minutes, use

$$\text{Load} = \frac{83}{64} = 1.30 \text{ lbs}$$

Permissible increase in length = .006 x 5 = 0.030 inches.

The required high modulus may be obtainable in thermosetting materials, but not in any commercial, unreinforced thermoplastic.

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