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PREPARATION OF CONDUCTING SOLID MIXTURES

Technical Report

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ABSTRACT

The application of conducting plastic mixtures to the fundamental problem of radiation dosimetry is briefly reviewed. A particular approach to achieving formulations with the necessary characteristics is described. A number of successful mixtures are defined for a number of different specific dosimetry situations. To obtain high quality stable materials requires intense blending and working of the materials at elevated temperatures. One machine that succeeds in this task is the Shonka plastics mixer-extruder. The Shonka mixer is described in complete detail. The procedures used in preparing representative formulations with this device are presented. A number of properties of successful conducting mixtures so prepared are summarized. The conditions required for molding such material are given. Several special welding methods for specific application with these formulations have been devised and are described.

I. INTRODUCTION

The most reliable and absolute means of measuring absorbed dose (ICRU,1971) in diverse radiation fields relates the charge released in a gas-filled cavity to the energy deposited in the walls which surround the cavity. The accurate measurement of the charge released requires the walls to possess adequate electrical conductivity. The determination of absorbed dose in solid conducting materials is accordingly a relatively straight forward matter. Devices which measure radiation intensity in terms of ionization produced in a gas are known as ionization chambers.

The ionization method, though it is relatively direct, fails when the substance of interest is either an insulator or is not solid under normal conditions. In such a case one must rely on less direct methods. The ionization method may yet be used in this case by substituting a conducting solid for the cavity walls which is "equivalent" to the material of interest. Thus the problem reduces to finding or devising a conducting solid which will absorb energy when placed in a given radiation field in the same measure as the material of interest. The use of such equivalent materials is a widely followed strategy in the field of radiation dosimetry. The same approach is equally valid for determining exposure (ICRU,1971) as well as absorbed dose. Of course, in the case of exposure, the need is specifically for a conducting solid which absorbs photons in the same measure as air, an "air-equivalent" material.

The explanation of and the principles behind the operation of ionization chambers are covered by cavity-chamber theory. This theory has been re-

cently reviewed (Burlin, 1968). Numerous types of ionization chambers and the many practical considerations in their implementation have been discussed by Boag (1966).

The importance of special conducting solids matched to a particular substance and used as the walls of an ionization chamber cannot be over emphasized. The need is particularly urgent in the field of diagnostic and therapeutic radiology involving human subjects. Here the consequences of erroneous measurements can be very grave.

This report presents the detailed preparation methods for a number of conducting solid mixtures which allow the direct measurement of the absorbed dose and exposure. The same general methods of preparation can be applied to a wide variety of other mixtures having a broad range of composition and physical properties. The highly successful procedures presently in use represent a culmination of the efforts of a number of people extending over a number of years.

An early tissue-equivalent (muscle) formulation for use in ionization chambers was devised by Rossi and Failla (1956). Their mixture was in the form of a gel and was approximately described by the formula $C_5 H_{40} O_{18} N$. The atomic composition of this substance, being very close to that of soft tissue, guarantees close equivalence with respect to both photons and neutrons of a wide range of energies. However, the physical state of the material presented practical difficulties when incorporated in ionization chambers. Evaporation requires such chambers to be sealed and the humidity inside approaches 100 %. Under conditions of extreme moisture it is difficult to maintain high electrical in-

sulation between the chamber electrodes.

The Federal Telecommunications Laboratories, Inc. (Keel et al., 1953) pursued the development of a tissue-equivalent conducting plastic under contract from the U.S. Atomic Energy Commission. In this work the goal was to achieve a material from which practical ionization chambers could be fabricated. The material was to contain atomic proportions within the following limits:

Hydrogen	9.5 to 10.5 %
Nitrogen	0.5 to 4.0 %
Carbon & Oxygen	balance up to 100 %
Qualified Trace Elements	up to 1 %

The general approach involved a base resin consisting of 75 % polyethylene and 25 % acetylene black for electrical conductivity. Nitrogen-containing and other additives would be introduced at the expense of polyethylene. The final mixture was designated CP-703 and contained 56 % polyethylene, 24 % acetylene black, and 20 % hexadecylmelamine. This material showed distinct promise in terms of general approach but exhibited several weaknesses including: brittleness, uncertain and unstable conductivity, and inadequate homogeneity.

Shonka (1956) initiated some effort directed towards the preparation of conducting plastic mixtures in 1956. In the following year he greatly intensified his efforts on conducting plastic mixtures (Shonka, 1957). A conducting polystyrene-equivalent was achieved consisting of 10 % carbon, 78.33 % polystyrene, and 11.67 % polyethylene. The Federal Telecommunication Laboratories type TE plastic was successfully duplicated

by a mixture in which the necessary nitrogen concentration was obtained by using a nylon 6/6 in place of hexadecylmelamine. The final TE mixture contained 19.64 % carbon, 46.46 % polyethylene, and 33.9 % nylon. Higher-Z elements were added in order to improve the low energy photon response. This led to a mixture containing 12 % carbon, 26.2 % nylon, 13.2 % silicon rubber (Silastic 2007), and 48.6 % polyethylene. The mechanical properties of this mixture left much to be desired.

It became very clear to Shonka that the actual method of mixing, the particular tools employed and the manner in which such are used, played an essential role in fixing the quality of the final product. Screw-type mixers were found to yield results distinctly superior to roller-types such as the Bandbury which had been employed by Federal Telecommunication Laboratories and by Shonka in his initial efforts. Accordingly, Shonka devised two different screw-type plastics mixers (Shonka, 1958).

Both of these mixers involved two parallel screws. In the one the screws were along side each other in separate bores which were joined by passages at each end. The screws rotate simultaneously driving the material to one end in the one bore and to the other end in the adjacent bore. The inter-connecting passages allow transfer of material between the two bores leading to a continuous circulation of the material being mixed.

The second design involved concentric screws. The outer screw fitted closely in a bore and itself was bored to accept the smaller screw. The smaller inner screw of reverse pitch was held stationary and only the larger hollow screw was rotated. By means of radial holes at each end of the hollow screw, a continuous circulation was maintained. Material is driven

to one end on the outer screw and into the inner bore where it is propelled to the opposite end and again onto the larger outer screw.

Both of these designs feature continuous circulation of the substance being mixed. Thus any desired degree of mixing can be achieved merely by adjusting the duration for which a given charge is mixed. Both designs provide efficient, intense, and thorough mixing. The latter design is somewhat simpler in that it has a single rotating part. However both designs suffer from being batch mixers wherein only discrete amounts can be prepared. There is also the question whether each element of a given loading has received the same degree of working. There inevitably are corners where material is immobilized to some degree.

The screw-type mixers led to immediate and decided improvement of all the mixtures that were attempted. The degree of dispersion, uniformity, and stability were manifestly superior to what had been realized up to this time. Having solved the problem of mixing, Shonka addressed the matter of optimizing the composition of different formulations for specific applications. In this the new mixers allowed much greater flexibility in the choice of constituents. Excellent results were obtained in combining materials which previously were believed to be incompatible such as polyethylene and nylon. These results were reported at the Second International conference on the Peaceful Uses of Atomic Energy (Shonka et al., 1958) as well as in the annual report under U.S. Atomic Energy Commission Contract No. AT (11-1)-323 (Shonka, 1958).

Driven by the desire to improve the quality of the mixtures to the ultimate attainable, Shonka (1959) conceived and built a mixer incorpora-

ting a new arrangement. The new mixer is a reciprocating screw-type extrusion machine which is designed to operate continuously. Ingredients in form of powders are fed steadily into the mixer at one end and the mixed plastic is extruded thru a nozzle at the other end. Thus any desired amount of material may be prepared by merely running for a suitable length of time. The degree of dispersion and uniformity obtained with this mixer actually exceeded significantly that which was obtained with the previous devices and required appreciably less effort and time to operate.

There have been no further fundamental advancements in mixing equipment of this type. Actually, it is impossible to imagine in what manner one might effect any substantial improvements. The many successful mixtures which have been produced are largely a result of the Shonka mixer. The same mixer has been employed from its beginning to the present time for essentially all mixtures prepared in the Physical Sciences Laboratory of Illinois Benedictine College. The Shonka mixer and the detailed procedures in using it to prepare a number of conducting plastic mixtures are the main subjects of this report.

This report is concerned with the preparation of mixtures which are primarily intended for use as the walls of ionization chambers. Clearly the same materials may be used with equal effectiveness in the construction of proportional counters. Likewise, it may be convenient and advantageous to employ the same material in the construction of phantoms for depth-dose studies. Although there is no need for electrical conductivity of the phantom material, constructing the phantom and probe of identical

materials would completely avoid any possible interface effects.

The details of preparing a number of distinct mixtures are given in the next section. Materials included are: two tissue-equivalent muscle mixtures, A150 and A180; two tissue-equivalent bone mixtures, B100 and B110; an air-equivalent mixture, C552; and several test mixtures. The physical characterization of these and other mixtures is addressed in the concluding section. Information is presented there on composition, equivalence, and density. Some information is also presented on the molding properties of different mixtures and on special welding methods.

II. METHODS OF PREPARATION

1. General Considerations

The requirements for a material which is suitable for the walls and electrodes of an ionization chamber which is intended for measurements of absorbed dose were given by G. Fialla (1958). These include:

1. tissue equivalence, within certain limits of accuracy, over a specified energy range and for a particular type radiation;
2. homogeneity over dimensions as small as the range of the ionizing particles involved;
3. good electrical conductivity;
4. mechanical properties which would allow the fabrication of chamber parts; and
5. stability, low vapor pressure and low absorption and adsorption of and interaction with gases to be used.

The first item above dictates the atomic composition. Since biological tissues of interest consist almost entirely of elements of low atomic

number and most photon processes exhibit marked dependencies on atomic number, the substitutes themselves must necessarily consist of elements of low atomic number. The general approach is to use one or more thermoplastic polymers as a host in which inert additives are dispersed. The additives are chosen to achieve the desired tissue-equivalence and the necessary electrical conductivity. Providing a host is chosen with adequate stability and low interaction with the gases to be employed, the use of inert additives will guarantee that 5. is satisfied. Choosing additives in the form of extremely fine powders and thoroughly and uniformly distributing such additives throughout the host plastic will achieve the required homogeneity.

The degree of electrical conductivity which is necessary for proper performance of chambers and counters is obtained by the addition of carbon in the form of carbon black. It has been determined that approximately a minimum of 10-18 % carbon black is required to achieve adequate electrical conductivity (Shonka et al., 1958). The actual minimum free carbon concentration will vary among different hosts and will likewise depend on the particular method employed in dispersing the carbon particles in the host plastic.

An ideal equivalent material would be one which has atomic composition identical to that of the substance it simulates and has all of the other physical properties dictated by the application. Unfortunately, in simulating biologically significant tissue it is rarely if ever possible to duplicate precisely the atomic composition of the biological tissue mainly due to the difficulty of achieving the relatively high

concentration of oxygen characteristic of common biological tissues.

In the conducting substitutes, free carbon largely replaces oxygen and provides to the mixture the necessary conductivity. This, to a degree, upsets the photon equivalence as carbon, $Z = 6$, has been substituted for oxygen, $Z = 8$. However, one endeavors to compensate for this mismatch by the addition of elements of slightly higher Z such as fluorine, magnesium, aluminum, and silicon.

The atomic composition of several biological tissues and of water and air are given in Table I. The compositions listed in the table define reference substances which the various mixtures to be described have been designed to simulate. The thermoplastic polymers which are used as host materials are listed in Table II along with certain of their physical properties. The particular nylon 6/6 which has been used at Illinois Benedictine College for nearly twenty years is Zytel 69 supplied by DuPont. Owing to the use of a plasticizer and other chemicals during manufacture, the final composition deviates from the idealized chemical formula shown in the table. In September 1972, DuPont gave the composition of Zytel 69 in terms of percent by mass as: hydrogen - 10.35 %; carbon - 64.85 %; nitrogen - 9.95 %; and oxygen - 14.85 %. In comparison, the idealized nylon 6/6 formula given in Table II yields the following mass concentrations: hydrogen - 9.80 %; carbon - 63.68 %; nitrogen - 12.38 %; and oxygen - 14.14 %.

Additives and some of their important physical properties are listed in Table III. In the preparation as well as in the subsequent processing and use of simulating mixtures one never encounters temperatures much beyond

Table I. Compositions of certain biological reference tissues, water and air.

Element	Percent by Mass				
	ICRU Muscle ^a	ICRU Bone ^a	Woodard Bone ^b	Water	Air
Hydrogen	10.2	6.4	3.39	11.19	-----
Carbon	12.3	27.8	15.51	-----	-----
Nitrogen	3.5	2.7	3.97	-----	75.5
Oxygen	72.89	41.0	44.13	88.81	23.2
Sodium	0.08	-----	0.06	-----	-----
Magnesium	0.02	0.2	0.21	-----	-----
Phosphorous	0.2	7.0	10.21	-----	-----
Sulphur	0.5	0.2	0.31	-----	-----
Chlorine	0.003	-----	-----	-----	-----
Argon	-----	-----	-----	-----	1.3
Potassium	0.3	-----	-----	-----	-----
Calcium	0.007	14.7	22.21	-----	-----

^aNBS Handbook 85, Physical Aspects of Irradiation (ICRU Report 10b), (1964).

^bWoodard, H.Q., Health Phys. 8, 513 (1962).

Table II. Properties of thermoplastic polymers which are used as hosts for conducting substitutes.

Name	Formula	Supplier	Product	Density ^a - g/cm ³	T _m ^b - °F
Polyethylene	(C ₂ H ₄) _m	Phillips	Marlex	.926 - .940	248 - 284
Polymethylpentene	(C ₆ H ₁₂) _m	Mitsui	TPX	.830	464
Polypropylene	(C ₃ H ₆) _m	Hercules	Profax	.902 - .910	349
Polystyrene	(C ₆ H ₈) _m	Monsanto	PIX5	1.04 - 1.09	212 ^c
Polytetrafluoroethylene	(C ₂ F ₄) _m	DuPont	Teflon	2.14 - 2.20	620
Polyvinylidene Fluoride	(C ₂ H ₂ F) _m	Pennwalt	Kynar	1.75 - 1.78	313
Nylon 6/6	(C ₁₂ H ₂₂ N ₂ O ₂) _m	DuPont	Zytel 69	1.13 - 1.15	509
Nylon 11	(C ₁₁ H ₂₁ NO) _m	Rilsan	Rilsan 11	1.03 - 1.05	381

^aModern Plastics Encyclopedia 1977-78, McGraw-Hill (1977).

^bCrystalline melting point.

^cAmorphous melting point.

the melting points of the host plastics. Since the additives have much higher melting points, no interaction involving any of the additives and hosts is anticipated.

Nine different TE mixtures are defined in Table IV in terms of the percent by mass of each component of the mixture. Each mixture consists of four components except two, A153 and B101, both of which have only two components, TFE (polytetrafluoroethylene) and carbon. Carbon is the only component which is common to all mixtures. The compositions of these same mixtures are given in Table V in terms of the mass concentrations of the elements. In computing the concentrations given in Table V, the chemical formulae which are given in Tables II and III were used except for nylon 6/6. For nylon 6/6 the composition which was given by DuPont for their Zytel 69 and which was noted above was used.

It appears that the mixture, A150, was devised and reduced to practice by Francis Shonka in June, 1958 (Spokas, 1975). This formulation was designed to simulate the ICRU muscle composition with respect to photons and neutrons of a broad range of energy. Of all the mixtures prepared, A150 has been one of the most successful and has received the greatest distribution. The next mixture, A153, was intended to simulate the ICRU muscle composition for photons, the same as A150, but specifically to have as little interaction with neutrons as possible. This latter characteristic is achieved by avoiding hydrogen and nitrogen in the mixture as well as any other elements which have substantial neutron cross sections.

The mixture A174 was intended to simulate the ICRU muscle composition for

Table III. Characteristics of some inert powders which have been used as fillers in conducting substitute mixtures.

Name	Formula	Supplier	Product	Density g/cm ³	Melting Point °C	Mean Particle Size μ
Carbon	C	Cabot	Vulcan XC72	1.8 - 2.1	3600	30
Calcium Fluoride	CaF ₂	Kraft Chemical	Fluorspar	3.18	1360	380
Calcium Fluoride	CaF ₂	Fisher Scientific	C-89	3.18	1360	50000
Magnesium Oxide	MgO	Fisher Scientific	M-49	3.58	2800	44000
Silica	SiO ₂	Cabot	Cabosil M5	2.636 - 2.66	1610	18
Silicon Nitride	Si ₃ N ₄	Union Carbide	-----	3.44	1900	2800

Table IV. Defining compositions of various tissue-equivalent formulations.

Component	Percent by Mass								
	A116	A150	A153	A174	A180	B100	B101	B109	B110
Polyethylene	-----	45.14	-----	71.38	-----	30.00	-----	42.77	2.87
Nylon	54.58	35.22	-----	-----	-----	21.60	-----	-----	32.07
TFE	-----	-----	56.75	-----	-----	-----	63.25	-----	-----
TPX	-----	-----	-----	-----	67.6	-----	-----	-----	-----
Carbon	24.02	16.06	43.25	17.94	13.5	14.00	36.75	17.13	13.84
CaF ₂	4.52	3.58	-----	1.93	1.2	34.40	-----	33.20	51.22
MgO	-----	-----	-----	-----	17.7	-----	-----	-----	-----
SiO ₂	16.88	-----	-----	-----	-----	-----	-----	-----	-----
Si ₃ N ₄	-----	-----	-----	8.75	-----	-----	-----	6.90	-----

Table V. Elemental mass concentrations in various tissue-equivalent mixtures.

Element	Percent of element in mixture								
	A116	A150	A153	A174	A180	B100	B101	B109	B110
Hydrogen	5.65	10.13	-----	10.26	9.72	6.55	-----	6.15	3.73
Carbon	59.42	77.55	56.88	79.06	71.38	53.69	51.94	53.75	37.10
Nitrogen	5.43	3.51	-----	3.49	-----	2.15	-----	2.76	3.19
Oxygen	17.09	5.23	-----	-----	7.025	3.21	-----	-----	4.76
Fluorine	2.20	1.74	43.12	0.94	0.58	16.74	48.06	16.16	24.93
Magnesium	-----	-----	-----	-----	10.675	-----	-----	-----	-----
Silicon	7.89	-----	-----	5.26	-----	-----	-----	4.14	-----
Calcium	2.32	1.84	-----	0.99	0.62	17.66	-----	17.04	26.29

both photons and neutrons. It was hoped that A174 would have improved physical properties as well as a more definite and constant atomic composition. The mixture involves a single well-defined plastic component, polyethylene, and so there is no question of compatibility of different polymers. Silicon nitride was introduced into this mixture in order to obtain the necessary nitrogen content which is important to obtain the correct thermal neutron interaction in the simulating mixture. This mixture was prepared for the first time in 1965.

The first mixture given in the table, A116, is an example of a single polymer loaded with several additives. This formulation was designed by Francis Shonka in consultation with Mudundi R. Raju to be a substitute for ICRU muscle with respect to pions. A small quantity of this formulation, probably less than 5 pounds, was prepared in 1968.

The mixture A180 was designed by Lowell Anderson of Memorial Hospital, New York. The intent here was to match prostate tissue with reference to the photon spectrum of Iodine-125. This represents the most recent formulation of which the first batch was prepared in June 1978. Here also is an example of a mixture having a solitary polymer host.

The mixture B100 involves the same four components as A150 but in proportions to match the ICRU bone composition with respect to both photon and neutron interactions. This mixture was described by Shonka, Rose, and Failla in their Geneva Conference paper (Shonka et al., 1958).

The following mixture, B101, had an objective similar to A153. It was designed to have similar interactions for photons as ICRU muscle but to

have minimal interactions with neutrons. Such mixtures as A153 and B101 are very useful in separating the photon and neutron doses in a mixed field.

Ideally, in mixed-field dosimetry, one desires one dosimeter which is entirely unresponsive to neutrons while having the normal response to photons and a second dosimeter which has the normal response for both radiation components. The separate components to the dose can be resolved according to standard procedures (ICRU, 1977). These same procedures can be used with any pair of chambers having different neutron and photon sensitivities. However, the precision with which the neutron and photon contributions to the dose can be resolved is greater the more dissimilar are the neutron sensitivities of the chambers.

Two other important types of conducting plastics have been produced. These are air-equivalent and polystyrene-equivalent mixtures. The former is extremely valuable for the precise measurement of the fundamental unit of radiation exposure, the Roentgen, which is defined in terms of the ability of photons to ionize air (ICRU, 1971). A polystyrene equivalent mixture is of much fundamental significance as it allows the construction of an ionization chamber which is homogeneous to an extremely high degree.

In a chamber there necessarily are insulating as well as conducting parts. For low level measurements the collector electrode requires a high degree of electrical insulation. Polystyrene having perhaps the lowest conductivity of all dielectrics is frequently the material of choice for the collector insulator. A chamber constructed exclusively of polystyrene-

equivalent conducting plastic, pure polystyrene, and filled with acetylene, for example, would have precisely the same atomic composition throughout the entire chamber.

An earlier air-equivalent mixture, C514, consisted of 64.70 % of nylon 6/6, 19.85 % of carbon black, and 15.45 % of silica. This is very similar to a mixture described by Shonka, Rose, Failla in their Geneva Conference paper (Shonka et al., 1958). In 1963 Shonka (Shonka, 1963) designed and prepared a new air-equivalent mixture based upon the then newly developed fluoroplastic, polyvinylidene fluoride. This mixture was designated C552 and has been the standard air-equivalent mixture ever since that time. C552 contains 78.4 % of polyvinylidene fluoride, 20.75 % of carbon black, and 0.85 % of silica.

The addition of 46.13 % of pure carbon to polyethylene results in a mixture having the identical atomic composition of polystyrene. If one combines such a mixture with polystyrene in arbitrary proportions, the atomic composition of the product will also be identical to that of polystyrene. Thus it is possible to prepare polystyrene-equivalent mixtures wherein the free carbon concentration may have any value between 0 and 46.13 %. This is the approach which Shonka adopted to achieve a conducting plastic which is precisely equivalent to polystyrene with reference to any and all radiations. The mixture, D350, which Shonka determined gave the best overall performance contains 71.82 % of polystyrene, 15.18 % of polyethylene, and 13.00 % of carbon black.

2. The Shonka Reciprocating Extrusion Plastics Mixer

The major components of the special Shonka mixer are a cylindrical mixing chamber which contains a closely fitting screw as depicted in Fig. 1. The screw thread is single and rounded in shape actually approximating a sine curve. The pitch is $2 \frac{5}{8}$ inches and the major and the minor diameters are $1 \frac{7}{8}$ and $1 \frac{3}{16}$ inches respectively. The screw is 8 inches in length and is mounted securely axially on a shaft which extends thru the end plugs of the chamber. The inside length of the chamber is 4 inches longer than the screw. The chamber has a uniform bore equal to the major diameter of the screw with just sufficient clearance to allow the screw to rotate freely inside the chamber.

By means of a ball-bearing spline attached to the end of the shaft which extends out the rear end-plug, torque may be applied to rotate the screw.

The chamber is kept essentially full of the correct proportions of host polymer, carbon, and other additives and is maintained at a temperature where the host polymer has a consistency approximating that of bread dough. The mixing and blending action is produced by rotating the screw, for example, clockwise causing it to move forward thru the mixture. Just before the screw reaches the front end-plug, the direction of rotation of the screw is abruptly reversed thus causing the screw to move axially towards the rear. When the screw reaches the rear end-plug, the direction of the rotation is reversed which again causes the screw to move axially forward. Thus, the screw rotates alternately clockwise and counterclockwise simultaneously moving forward and rearward.

Fig. 1 Sectional view of mixing chamber and mixing screw of Shonka plastics mixer.

The ingredients of the particular formulation being prepared are forced into the mixing chamber thru a port in the side of the chamber near the rear end-plug. The forcing is accomplished by means of a screw-type feeder clamped directly to the mixing chamber. The ingredients are in the form of fine powders and are homogenized beforehand. The preliminary mixing of the powders is accomplished by placing discrete amounts of the powdered mixture in sealed round containers and rolling the containers for a fixed length of time on a special treadmill device. The barrel of the feeder has an inside diameter of 1 inch. The optimum shape and pitch of the threads and the length of the feedscrew may differ for each different formulation. Two styles of thread are depicted in Fig. 2. That of Fig.2(a) represents a good basic shape and is the first type used by Shonka.

A small quantity of material is removed from the mixing chamber at the end of each complete cycle. This is accomplished by continuing to rotate the mixing screw in the counterclockwise direction for a preset time after the screw reaches the rear end-plug. The screw is prevented from further rearward axial movement and from contacting the end-plug by a sturdy external ball-bearing thrust bearing. The rotation of the screw while the axial motion is prevented forces the material on the screw forward thereby building up pressure in the front portion of the chamber. A short axial hole in the free end of the shaft intersects a diametrical hole in the shaft. The diametrical hole will be just inside the front end-plug when the screw is at the limit of its rearward motion. The pressure build-up in the chamber by the stationary rotating screw forces

(a)

(b)

Fig. 2 Two styles of feed screws which have been used on the Shonka mixer.

plastic thru the intersecting holes and out the end of the shaft. Nozzles of various orifice sizes may be screwed into the free end of the shaft. Sizes which have been used range from 1/8 to 5/16 inches in diameter. A timer sets the length of time which the screw continues to rotate counterclockwise. This is referred to as the extrude time and can be set to any value in the range 0 - 15 seconds. Typically on the order of 1 % of the material in the chamber is extruded in the course of each complete cycle.

The mixing screw is driven by a gear motor 1 1/2 horsepower. A roller-chain between the motor and ball-bearing spline permits the speed to be readily altered within certain limits by choice of different sprocket combinations. For most mixtures a rotational speed of the mixing screw of 80 rpm is satisfactory.

Two microswitches sense the extreme axial positions of the mixing screw. The rear microswitch is activated when the screw reaches the limit of its rearward travel. The front microswitch is activated when the screw has advanced a certain distance forward. The position of this switch is adjustable and, as will be seen, determines just how far the screw advances in the forward direction.

The mixer operates automatically by means of the control circuit shown in Fig. 3. When the relays, which are wired in parallel, are inactive, the mixing screw rotates counterclockwise thus causing the screw to move rearward. When the screw reaches the limit of this motion, it closes the rear microswitch thus starting the timer cycle. After the

Fig. 3 Mixer control circuit.

timer runs down its contacts close causing the control relay to latch and at the same time reversing the drive motor thru the activation of the reversing relay. The screw then immediately begins to move forward and continues until it activates the front microswitch. This unlatches the control relay and de-energizes the reversing relay. The drive motor is thereby suddenly reversed causing the screw to move now rearward until it activates the rear microswitch again and the same sequence is repeated.

Shonka operated the feed screw intermittently by means of a second timer which was also activated by the rear microswitch. In this mode of operating, the feed would generally operate for a time which was deliberately set longer than the extrude time. A motor equipped with a mechanical disk clutch was used to turn the feed screw. The clutch was operated off the second timer by means of a suitable solenoid.

In recent years it has been found expedient to operate the feed screw continuously. Thus the second timer and the entire clutch mechanism has been eliminated. By adjusting the speed of rotation of the continuously rotating feed screw it is possible to exercise more than sufficient control over the mixer cycle. Depending on the particular mixture and style of screw, the feed screw runs at speeds between 50 and 250 rpm.

For best results it is necessary to hold the temperature of the mixing chamber within fairly narrow limits. The requisite control is achieved by a combination of band heaters fitted over the chamber and blowers directed at the chamber. The band heaters are regulated by two temperature controllers which sense the temperature of the chamber at a point near the

center and a point near the front end. For many formulations, the action of the mixing screw on the plastic generates more than sufficient heat to maintain the temperature without the aid of the band heaters. In such a circumstance it is necessary to furnish some degree of forced cooling otherwise the chamber temperature would rise excessively.

The air-equivalent formulation, C552, is an excellent example of a mixture which exhibits considerable internal friction. For such, the band heaters are only needed to initially establish the operating temperature. When the mixing commences, the band heaters may be disconnected and blowers must be turned on to provide forced air-cooling.

An early photograph of the only Shonka reciprocating plastic extrusion mixer ever built is shown in Fig. 4. The mixing chamber is partially exploded revealing the screw proper (B), end-plugs (D), extrusion port (F), and a removable extrusion nozzle (G). The spline fitting rides on a pair of radial ball-bearings in the housing (E). The sprocket and chain which drive the spline fitting can just be seen at the extreme left of the photograph. The drive motor in the center of the photograph is bolted to the bottom side of the plate to which the mixing chamber and spline housing are attached. The control circuits are located in the box attached to the front side of the mixer frame.

The important operating parameters of the Shonka mixer are: rotational speed of mixing screw, stroke which is the axial movement of the mixing screw, temperature of the chamber, size of extrusion orifice, extrusion time, and rotational speed of the feed screw. Perhaps the most important

dependent variable is the average mixing rate, that is, the average throughput rate of material thru the mixer. It is possible to exercise considerable control over the mixing rate by means of the operating parameters. Somewhat surprisingly, for a number of mixtures the extrusion time and orifice size have practically no effect on the mixing rate. For a given speed of the mixing screw, it is primarily the speed of the feed screw which sets the mixing rate with nearly a direct proportion obtaining between the two.

3. General Mixing Procedures

The ingredients for a mixture are to be in the form of finely divided powders. Discrete amounts of each component are accurately weighed out and placed in containers. Typically 500 g of powder containing precisely the correct proportion of each ingredient are placed in the containers. One gallon glass and polyethylene jars have been used for this purpose as well as 3-lb coffee cans. Each 500 g amount of powdered mixture is thoroughly homogenized prior to feeding into the Shonka plastics mixer.

This preliminary homogenization is readily accomplished by revolving the containers about a horizontal axis for 20-60 minutes. Plastic mixing blades attached to the covers of the jars greatly accelerate the preliminary homogenization process. A considerably more effective technique is to place a pair of free rollers in the containers during rolling. One roller might be shaped like a drill with deep spiral flutes. Such a roller causes an axial movement of the powder in the course of rolling. The

Fig. 4 An early photograph of the Shonka plastics extrusion mixer.

second roller then would be either a round or hexagonal rod. The loose rollers in the containers do an excellent job of breaking the lumps which frequently occur in most of the ingredients thereby allowing a significantly greater degree of homogeneity.

Starting up the mixer is an easy matter in the case of a mixture which has been mixed previously. In this case, granules of the mixed material of approximately a size corresponding to 3/16 mesh are fed into the mixer. When the chamber is full and the material begins to appear at the extrusion nozzle, the feeding of granules is stopped and the feeding of unmixed but homogenized powder is started. If no previously mixed material is on hand as in the case of a formulation being prepared for the first time, the mixing must be started using the powdered ingredients directly. In this case the first several pounds of material which emerges from the mixer are ground to an appropriate particle size and sent thru the mixer again. Thus, ultimately all portions of the final product receive the same degree of working, kneading, and blending.

In some situations, particularly when the polymer particle size is extremely small, the feed mechanism is unable to generate adequate force on the ingredients in the feed barrel. In such a case, the mixing rate would drop off significantly. The mixing of coarse granules of previously mixed material with the powdered ingredients promotes the feeding of the powder and restores the throughput rate to acceptable levels.

Gases and volatile elements in certain plastics frequently create problems in getting the ingredients in to the mixing chamber. The pressure

build-up of gases in the feed barrel must escape thru the open end of the barrel. This acts on the powder contrary to the feed screw. The forward movement of powder is halted until the gas is relieved one way or another. One solution to this problem is to add a vent hole at an appropriate position in the feed barrel to enable the gases and vapors coming off the powder to escape. A temperature gradient develops along the feed barrel. The vent hole is ideally located at the position where the majority of gas is released. Some powder inevitably is blown out the vent along with the gas and means must be provided for collecting this discharge. The powder collected is subsequently fed back into the mixer. Gassy powders generally will feed better with a feed screw of minimal length. In some cases it may be possible to use such a short feed screw as to obviate the need for a vent hole.

The mixture A150 illustrates a special two-step procedure that may be used in the case of a dual polymer host one of which is not readily available as a finely divided powder. A150 contains 45 % polyethylene and 35 % nylon 6/6. The latter is only available in the form of pellets whereas the former can be obtained as a reasonably fine powder. Because of the physical property of nylon, special methods must be used in order to grind the pellets into a powder. Accordingly, a premium would have to be paid to get the job accomplished.

To avoid the need of grinding the nylon pellets, a preliminary mixture containing the same proportions of polyethylene, carbon black, and calcium fluoride as are found in A150 is prepared following the methods outlined above. This preliminary mixture is then granulated to an average par-

ticle size equivalent to that of the nylon pellets. The pre-mix is then combined with the nylon in the correct proportion for A150 and in discrete amounts. The granular mixture is thoroughly homogenized by rolling in suitable containers as with fine powdered ingredients. Finally the homogenized granular mixture is sent thru the Shonka mixer.

4. Conditions Used in Preparing Specific Mixtures

Since 1971 twelve different conducting plastic formulations have been made. The mixing conditions, particularly with reference to temperature, and special procedures which were followed are given below. The mixtures are listed in chronological order according to the initial preparation of the particular mixture.

C552 This is the standard air-equivalent formulation. When the powdered ingredients are well homogenized and contain no lumps, this is the most difficult material to force into the Shonka mixer. Appreciable quantities of gas evolve from the powder as it is heated. Accordingly to obtain adequate feeding rates with a long feed screw requires venting. A screw which is 4 inches in length is workable without venting. It is expedient to add 30-50 % of granular material which has been mixed previously to the homogenized ingredients in order to promote the feeding process. Mixing temperatures have ranged from 460 to 550 °F. The lower temperatures are to be preferred. Temperatures even lower than the lowest indicated may be practicable but this has not been investigated. During mixing, more than sufficient heat is generated in the mixture to maintain the operating temperature. Blowers are usually necessary to prevent an ex-

cessive rise in temperature. Above 550 °F, the mixing rate will drop off and the self-heating will increase forcing the temperature even higher. Such a condition must be avoided as the consequences could be disastrous. The plastic host of this mixture, polyvinylidene fluoride, decomposes and transforms to a brittle non-plastic substance at the higher temperatures. Such could jam the mixing screw and cause mechanical damage to the mixer. Should such a thermal runaway commence, the mixing should be suspended until the temperatures decrease sufficiently. In such a case, greater external cooling may be needed or, perhaps, a more effective feed mechanism may reduce the self-heating to a tolerable level.

E105 and E106 These mixtures consist of 30 and 20 % respectively of carbon black in polypropylene. These mixtures were prepared for evaluation as the barrel material in low cost direct reading personal pocket dosimeters of the chamber-fiber electrometer type. Mixing temperatures of 375 - 400 °F gave satisfactory results. Polypropylene does not exhibit noticeable self-heating and heaters are required in order to maintain a proper working temperature. Here also, lower temperatures than indicated may be satisfactory but have not been investigated.

B100 This is the standard bone-equivalent formulation. As there are two polymer components in this mixture one of which, nylon 6/6, is not available as a powder, mixing is accomplished in two steps. A preliminary mixture containing polyethylene, calcium fluoride, and carbon is first simply extruded. In this operation only superficial mixing and blending occurs. The extruded pre-mix is subsequently granulated, combined with pelletized nylon 6/6, and mixed in the Shonka mixer. Temperatures range

from 350 to 400 °F. Other experience noted below suggests that temperatures as low as 315 °F would work equally well. Appreciable gases evolve from the ingredients of the pre-mix and even more copious amounts from the nylon 6/6. The latter vapors largely represent a volatile plasticizer employed in the manufacture of the polymer. The vapor is rather noxious and adequate ventilation is essential.

E100, E101, E102, E103, and E104 Each of these five mixtures contain 13.5 % carbon black combined with various proportions of polypropylene and nylon 11 ranging from 100 % polypropylene to 100 % nylon 11. Polypropylene and nylon 11 can be obtained as fine powders which is the form used here. These mixtures were prepared for the purpose of assessing the compatibility of polypropylene and nylon 11 and the degree of electrical conductivity which is obtainable. Operating temperatures in the range of 350 to 425 °F were employed. Since no significant self-heating occurs in any of these mixtures, heat must be supplied externally to maintain the temperature of the mixing chamber.

B110 This is a new bone formulation designed to simulate cortical bone. B110 contains the same four ingredients as A150 and B100 but in different proportions. The concentration of polymer, 35 %, is the lowest of mixtures which have been blended in the Shonka mixer. Only 6 pounds were prepared of this formulation. Therefore the mixing was accomplished in a single step using some Zytel 69 which had been finely ground beforehand. Temperatures were maintained at 300 - 330 °F. After commencing mixing, blowers are needed to prevent the temperature from rising.

A150 This is the standard muscle-equivalent mixture. It involves the two polymers, polyethylene and nylon 6/6. As the latter is not available in powdered form, mixing is normally accomplished in two steps. The correct proportions of polyethylene, carbon black, and calcium fluoride all in the form of fine powders are combined in amounts totalling 500 grams. Each amount is individually thoroughly homogenized and then worked in the Shonka mixer at temperatures of 315 - 330 °F. It is necessary to furnish external cooling to the mixing chamber as the working of this material by the mixer generates more heat than required to maintain the operating temperature. This preliminary mixture is granulated to a size approximating that of the pellets of Zytel 69, the particular nylon 6/6 employed. Granules of the pre-mix are combined with Zytel 69 in amounts totalling 800 grams in the proper proportions for A150. These discrete amounts are homogenized and then worked in the Shonka mixer at temperatures of 315 - 330 °F. Copious quantities of vapor come off Zytel 69 at these temperatures and good ventilation must be provided. Because of the relatively large particle size, feeding proceeds smoothly and efficiently despite the quantities of vapor which evolve.

A180 This is a new formulation designed to simulate prostate tissue with respect to low energy photons, particularly the spectrum which is characteristic of Iodine-125. The single polymer involved here, polymethylpentene, is only obtainable in the form of pellets. Therefore, the pellets were ground to 325 mesh beforehand. The four components of this mixture are prepared in fixed amounts of 500 grams total in correct proportions. The ingredients are thoroughly homogenized by means of rolling each 500 gram

amount in a container on a special treadmill for about 40 minutes.

Blending and further homogenization is accomplished in the Shonka mixer operating at temperatures of 525 - 550 °F. The ingredients are relatively easy to force into the mixing chamber. Barrel heaters are required to maintain the temperature. Evaluation of the compression molded characteristics of the host polymer and of the final mixture suggests that adequate blending would be achieved at temperatures as low as 450 °F.

III. CHARACTERISTICS OF VARIOUS MIXTURES

1. Compositions

The defining composition and the particular biological tissue simulated have been given in II of this report for the tissue formulations A116, A150, A153, A174, A180, B100, B101, B109, and B110. The ingredients of two different air-equivalent formulations, C514 and C552, have likewise been given earlier. The composition of four different polystyrene-equivalent formulations and five mixtures of polypropylene, nylon 11, and carbon black with different proportions of the two polymers but a constant concentration of carbon black are listed in Table VI.

The Mixture D351 contains the maximum free carbon concentration for mixtures of this type. However the mixture D350 containing 13 % free carbon exhibits adequate electrical conductivity and mechanical properties closely approximating those of pure polystyrene. Accordingly, D350 has been adopted as the standard polystyrene-equivalent mixture.

Table VI. Compositions of several polystyrene-equivalent and nylon-polypropylene-carbon mixtures.

Component	Percent by Mass								
	D350	D351	D352	D353	E100	E101	E102	E103	E104
Polyethylene	15	53.87	21	18.1	----	----	----	----	----
Polystyrene	72	----	61	66.4	----	----	----	----	----
Carbon black	13	46.13	18	15.5	13.6	13.6	13.6	13.6	13.6
Nylon 11	--	----	--	----	21.6	43.2	64.8	86.4	----
Polypropylene	--	----	--	----	64.8	43.2	21.6	----	86.4

The nylon-polypropylene-carbon mixtures were prepared in order to ascertain the miscibility of the three components at temperatures necessary to achieve adequate blending in the Shonka mixer. Nylon 11 has much lower moisture absorption than nylon 6/6 as well as a more precise chemical composition. If adequate mechanical properties and electrical conductivity are achievable in this system, these polymers may be substituted for polyethylene and nylon 6/6 in A150 to obtain a mixture of the same atomic composition as A150. Such a plastic would have less moisture absorption and a more definite atomic composition than A150 and it may have superior mechanical properties as well.

2. Photon Attenuation and Equivalence

Calculations of the total photon attenuation and photoelectric cross sections have been completed for eight of the compositions described above. The calculations cover 24 discrete photon energies extending from 10 keV to 8 MeV and utilize the atomic cross sections compiled in the ENDF/B data file (Simmons & Hubbell, 1973). These cross sections are the most complete and up-to-date which are available and perhaps also are the most reliable. The mass attenuation coefficients for the mixtures were taken as the mass-averaged coefficient averaged over the different elements in the mixture. The expression is

$$(\mu/\rho) = \sum_i \gamma_i (\mu/\rho)_i$$

where (μ/ρ) is the mass attenuation coefficient for the mixture, $(\mu/\rho)_i$ is the corresponding coefficient for the i -th element in the mixture, and γ_i is the mass fraction of the i -th element. The results of this computation

are presented in Table VII. The relative coefficients given in columns 4 and 5 of the table are the percent differences of the coefficients for a mixture relative to the indicated reference substance,

$$\frac{(\mu/\rho) - (\mu/\rho)_r}{(\mu/\rho)_r} \times 100 \%$$

For the mixtures A116, B100, and C514 the atomic composition of the nylon component was taken to be $C_{15} H_{27} N_2 O_2$ (Spokas, 1975). For A150 the atomic composition of the nylon component was taken as that given by DuPont in 1972 (see page 10). In the case of A150, the differences in the mass attenuation coefficients using one or the other nylon composition were found to be only a few tenths of one percent in the case of total attenuation and about one percent and less for the photoelectric effect (Spokas, 1975). The differences in the case of A116, B100, and C514 are expected to be of a comparable magnitude.

It is interesting to compare the calculated coefficients to the measured values. Table VIII lists five measured values for A150 and C552 and corresponding values derived from the calculated data of Table VII using a logarithmic interpolation. The greatest disparity is seen to be nearly 6 % and occurs at 9.87 keV for C552. As might be expected, the calculated values are found to be reasonably reliable.

3. Molding Characteristics

With the exception of A153 and B101, all of the mixtures defined above are readily molded using conventional compression, transfer, and injection molding techniques. These two formulations require special techniques to a-

are presented in Table VII. The relative coefficients given in columns 4 and 5 of the table are the percent differences of the coefficients for a mixture relative to the indicated reference substance,

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3. Molding Characteristics

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Table VII. Total and photoelectric mass attenuation coefficients for several conducting plastic mixtures evaluated from the atomic cross sections of ENDF/B. The percent differences relative to either the muscle, bone, or air composition of Table I are given in columns 4 and 5.

E keV	$(\mu/\rho)_{\text{atten}}$ cm^2/g	$(\mu/\rho)_{\text{photo}}$ cm^2/g	rel. atten. %	rel. photo. %
A116		A116/Muscle		
10	.7506E+01	.7126E+01	45.3624	49.1631
15	.2352E+01	.2052E+01	46.1230	57.1900
20	.1697E+01	.8369E+00	40.4676	62.4755
30	.4550E+00	.2331E+00	24.1549	69.3105
40	.2968E+00	.8361E-01	12.3104	75.7283
50	.2373E+00	.4608E-01	5.6855	76.8374
60	.2202E+00	.2584E-01	2.6622	79.1748
80	.1786E+00	.1040E-01	-1.3170	82.6767
100	.1648E+00	.5159E-02	-2.6506	85.1012
150	.1436E+00	.0464E-02	-3.7313	88.3183
200	.1303E+00	.6085E-03	-4.0370	91.2786
300	.1126E+00	.1817E-03	-4.2143	94.0084
400	.1007E+00	.7910E-04	-4.2587	95.7618
500	.9181E-01	.4220E-04	-4.2763	96.9066
600	.8490E-01	.2557E-04	-4.2960	97.8252
800	.7452E-01	.1103E-04	-4.3020	99.1322
1000	.6700E-01	.6712E-05	-4.3023	100.2354
1500	.5449E-01	.3278E-05	-4.3110	99.3599
2000	.4679E-01	.1972E-05	-4.2875	98.7288
3000	.3762E-01	.1095E-05	-4.2042	98.2570
4000	.3228E-01	.7544E-06	-4.1020	97.5226
5000	.2876E-01	.5648E-06	-4.0155	96.9230
6000	.2620E-01	.4561E-06	-3.9282	96.4701
8000	.2295E-01	.3255E-06	-3.7427	95.8250

Table VII. (continued)

E keV	$(\mu/\rho)_{\text{atten}}$ cm ² /g	$(\mu/\rho)_{\text{photo}}$ cm ² /g	rel. atten. %	rel. phpto. %
A150		A150/Muscle		
10	.4023E+01	.3690E+01	-22.0816	-22.7034
15	.1329E+01	.1050E+01	-17.4056	-19.5171
23	.6766E+00	.4263E+00	-13.4764	-17.2437
30	.3325E+00	.1185E+00	-7.5763	-13.1953
42	.2527E+00	.4765E-01	-4.3792	-11.5772
50	.2182E+00	.2349E-01	-2.7803	-9.8332
60	.2200E+00	.1320E-01	-1.9303	-5.4535
70	.1501E+00	.5328E-02	-1.1355	-6.4481
100	.1686E+00	.2648E-02	-.7978	-4.9948
150	.1485E+00	.7538E-03	-.4718	-2.7183
200	.1353E+00	.3138E-03	-.3670	-1.3656
300	.1172E+00	.9369E-04	-.2865	-.371
400	.1049E+00	.4069E-04	-.2445	-.0665
500	.9569E-01	.2164E-04	-.2276	-.8619
600	.8851E-01	.1306E-04	-.2209	1.0725
800	.7771E-01	.6024E-05	-.2110	-.9478
1000	.6987E-01	.3376E-05	-.1943	-.6954
1500	.5681E-01	.1647E-05	-.2376	-.1797
2000	.4673E-01	.9902E-06	-.3314	-.1867
3000	.3931E-01	.5518E-06	-.6342	-.2442
4000	.3331E-01	.3790E-06	-1.0223	-.7565
5000	.2952E-01	.2836E-06	-1.4705	-1.1265
6000	.2676E-01	.2290E-06	-1.2629	-1.2401
8000	.2320E-01	.1634E-06	-2.6877	-1.6784

Table VII. (continued)

E keV	$(\mu/\rho)_{\text{atten}}$ cm ² /g	$(\mu/\rho)_{\text{photo}}$ cm ² /g	rel. atten. %	rel. photo. %
Al74		Al74/Muscle		
10	.4712E+01	.4367E+01	-3.7428	-9.5248
15	.1521E+01	.1036E+01	-5.5468	-3.3564
20	.7521E+00	.4993E+00	-3.7446	-3.2638
30	.3594E+00	.1369E+00	-1.9720	-.5767
40	.2010E+00	5449E-01	-1.2203	1.4130
50	.1225E+00	.2666E-01	-.8654	2.3215
60	.8226E+00	.1489E-01	-.6585	3.2681
80	.1813E+00	.5955E-02	-.4642	4.5083
100	.1667E+00	.2941E-02	-.3515	5.5100
150	.1489E+00	.8294E-03	-.2109	7.8359
200	.1356E+00	.3437E-03	-.1627	8.7456
300	.1174E+00	.1624E-03	-.1108	9.5476
400	.1051E+00	.4455E-04	-.6888	10.2166
500	.9583E-01	.2378E-04	-.2766	10.9536
600	.8864E-01	.1442E-04	-.2769	11.5679
800	.7782E-01	.6718E-05	-.6681	12.5787
1000	.6997E-01	.3805E-05	-.6546	13.5130
1500	.5689E-01	.1860E-05	-.8949	13.1377
2000	.4860E-01	.1126E-05	-.1774	12.8623
3000	.3968E-01	.6221E-06	-.4344	12.6266
4000	.3340E-01	.4290E-06	-.7871	12.3176
5000	.2962E-01	.3214E-06	-1.1593	12.2651
6000	.2686E-01	.2596E-06	-1.4932	11.7557
7000	.2331E-01	.1855E-06	-2.2076	11.5751

Table VII. (continued)

E	$(\mu/\rho)_{\text{atten}}$	$(\mu/\rho)_{\text{photo}}$	rel. atten.	rel. photo.
keV	cm ² /g	cm ² /g	%	%
	B100		B100/Bone.	
10	.1931E+02	.1884E+02	-2.4850	-2.4102
15	.6101E+01	.5748E+01	-1.6145	-1.4825
20	.2729E+01	.2433E+01	-1.0565	-.9253
30	.9511E+00	.7102E+00	-.4817	.6536
40	.5015E+00	.2938E+00	-.3091	.6474
50	.3461E+00	.1477E+00	-.3088	1.0670
60	.2715E+00	.8411E-01	-.3624	1.3849
80	.2070E+00	.3460E-01	-.4631	1.9371
100	.1781E+00	.1741E-01	-.5234	2.1461
150	.1482E+00	.5046E-02	-.5711	2.5869
200	.1325E+00	.2120E-02	-.5823	2.3019
300	.1134E+00	.6391E-03	-.5759	2.9835
400	.1011E+00	.2787E-03	-.5686	2.9249
500	.9213E-01	.1485E-03	-.5555	2.8311
600	.8515E-01	.8968E-04	-.5615	2.7034
800	.7470E-01	.4132E-04	-.5578	2.3615
1000	.6714E-01	.2311E-04	-.5547	2.2370
1500	.5462E-01	.1124E-04	-.5657	1.9505
2000	.4697E-01	.6742E-05	-.6158	1.8991
3000	.3791E-01	.3746E-05	-.7540	1.9242
4000	.3272E-01	.2570E-05	-.9354	1.8334
5000	.2932E-01	.1918E-05	-1.1345	1.7555
6000	.2685E-01	.1546E-05	-1.3041	1.7233
7120	.2377E-01	.1101E-05	-1.6041	1.6670

Table VII. (continued)

E	$(\mu/\rho)_{\text{atten}}$	$(\mu/\rho)_{\text{photo}}$	rel. atten.	rel. photo.
keV	cm^2/g	cm^2/g	%	%
<hr/>				
	B101		B101/Bone	
10	.5875E+01	.4767E+01	-74.3651	-75.6232
15	.1555E+01	.1272E+01	-74.9195	-76.2028
20	.7417E+00	.4972E+00	-73.1081	-78.7283
30	.3384E+00	.1513E+00	-64.5963	-61.5372
40	.2399E+00	.5093E-01	-52.9173	-62.5510
50	.2020E+00	.2448E-01	-41.8190	-83.2440
60	.1826E+00	.1347E-01	-32.9667	-83.7573
80	.1623E+00	.5282E-02	-21.9334	-84.4543
100	.1506E+00	.2572E-02	-16.3671	-84.9141
150	.1324E+00	.7089E-03	-11.1772	-85.5231
200	.1204E+00	.2896E-03	-9.6296	-85.9637
300	.1042E+00	.8480E-04	-8.6853	-86.3352
400	.9315E-01	.3649E-04	-8.4206	-86.5241
500	.8497E-01	.1932E-04	-8.2833	-86.6182
600	.7857E-01	.1165E-04	-8.2409	-86.6641
800	.6857E-01	.5374E-05	-8.1811	-86.6856
1000	.6200E-01	.3019E-05	-8.1621	-86.6718
1500	.5045E-01	.1483E-05	-8.1627	-86.5517
2000	.4334E-01	.8955E-06	-8.2978	-86.4661
3000	.3490E-01	.4984E-06	-8.6563	-86.4403
4000	.3002E-01	.3451E-06	-9.0927	-86.3255
5000	.2662E-01	.2595E-06	-9.5869	-86.2357
6000	.2448E-01	.2101E-06	-10.0005	-86.1809
8000	.2155E-01	.1565E-06	-10.8508	-86.1001

Table VII. (continued)

E keV	$(\mu/\rho)_{\text{atten}}$ cm ² /g	$(\mu/\rho)_{\text{photo}}$ cm ² /g	rel. atten. %	rel. photo. %
B109		B109/Bone		
10	.1997E+02	.1956E+02	.8619	.9656
15	.6287E+01	.5928E+01	1.3276	1.0124
20	.2893E+01	.2503E+01	1.6245	2.2580
30	.9735E+00	.7285E+00	1.5846	2.6445
40	.5157E+00	.3008E+00	1.1897	3.4237
50	.3497E+00	.1509E+00	.7228	3.3099
60	.2734E+00	.8588E-01	.3211	3.5195
80	.2075E+00	.3528E-01	-.2265	3.8149
100	.1791E+00	.1773E-01	-.4924	4.0113
150	.1479E+00	.5131E-02	-.7431	4.3238
200	.1321E+00	.2154E-02	-.8203	4.4526
300	.1131E+00	.6491E-03	-.8583	4.5934
400	.1008E+00	.2831E-03	-.8653	4.5472
500	.9185E-01	.1509E-03	-.8579	4.4878
600	.8488E-01	.9118E-04	-.8704	4.4236
800	.7447E-01	.4207E-04	-.8691	4.2803
1000	.6692E-01	.2356E-04	-.8698	4.0212
1500	.5445E-01	.1146E-04	-.8700	3.9737
2000	.4683E-01	.6877E-05	-.9185	3.9332
3000	.3703E-01	.3821E-05	-.9812	3.9411
4000	.3067E-01	.2621E-05	-1.0833	3.9265
5000	.2931E-01	.1957E-05	-1.1993	3.8204
6000	.2685E-01	.1570E-05	-1.2923	3.8141
8000	.2361E-01	.1124E-05	-1.5066	3.7083

Table VII. (continued)

E keV	$(\mu/\rho)_{\text{atten}}$ cm ² /g	$(\mu/\rho)_{\text{photo}}$ cm ² /g	rel. atten. %	rel. photo %
C514		C514/Air		
10	.5255E+01	.4096E+01	2.7488	3.4888
15	.1592E+01	.1383E+01	4.2277	4.8265
20	.7716E+00	.5180E+00	4.6901	5.7369
30	.3593E+00	.1385E+00	5.1332	6.6128
40	.2571E+00	.5477E-01	5.5510	7.4252
50	.2178E+00	.2653E-01	5.8346	7.1793
60	.1976E+00	.1469E-01	6.0876	7.3246
80	.1762E+00	.5812E-02	6.2123	7.4361
100	.1637E+00	.2846E-02	6.3119	7.4651
150	.1443E+00	.7929E-03	6.4587	7.5897
200	.1313E+00	.3263E-03	6.5569	7.7456
300	.1137E+00	.9662E-04	6.5177	8.0848
400	.1017E+00	.4195E-04	6.5684	8.5128
500	.9275E-01	.2240E-04	6.5269	8.9947
600	.8579E-01	.1360E-04	6.5653	9.4843
800	.7531E-01	.6358E-05	6.5321	10.4455
1000	.6771E-01	.3617E-05	6.5850	11.4363
1500	.5507E-01	.1773E-05	6.4922	11.4269
2000	.4727E-01	.1069E-05	6.3928	11.4161
3000	.3793E-01	.5938E-06	6.0866	11.1397
4000	.3249E-01	.4105E-06	5.6613	11.2358
5000	.2889E-01	.3082E-06	5.1742	11.2589
6000	.2627E-01	.2492E-06	4.7626	11.1787
8000	.2292E-01	.1783E-06	3.8894	11.1186

Table VII. (continued)

E keV	$(\mu/\rho)_{\text{atten}}$ cm^2/g	$(\mu/\rho)_{\text{photo}}$ cm^2/g	rel. atten. %	rel. photo. %
C552		C552/Air		
10	.5000E+01	.4712E+01	3.2960	3.4215
15	.1563E+01	.1276E+01	2.3080	2.6208
20	.7476E+00	.4994E+00	1.4392	1.5474
30	.3432E+00	.1320E+00	.5342	.5395
40	.2444E+00	.5129E-01	.3215	.1523
50	.2062E+00	.2467E-01	.2435	-.3423
60	.1867E+00	.1356E-01	.2060	-.7006
80	.1600E+00	.5328E-02	.1831	-1.4056
100	.1543E+00	.2595E-02	.1541	-1.9909
150	.1357E+00	.7160E-03	.1519	-2.6501
200	.1235E+00	.2926E-03	.1365	-3.3712
300	.1068E+00	.2575E-04	.1086	-4.0765
400	.9554E-01	.3692E-04	.1150	-4.5074
500	.8710E-01	.1956E-04	.1014	-4.8261
600	.8259E-01	.1179E-04	.1131	-5.0302
800	.7075E-01	.5447E-05	.0770	-5.3827
1000	.6360E-01	.3063E-05	.1067	-5.6461
1500	.5174E-01	.1504E-05	.0647	-5.4746
2000	.4444E-01	.9081E-06	.0229	-5.3470
3000	.3575E-01	.5054E-06	.0061	-5.4008
4000	.3072E-01	.3498E-06	-.0841	-5.2058
5000	.2741E-01	.2630E-06	-.2020	-5.0476
6000	.2500E-01	.2129E-06	-.2917	-5.0006
8000	.2190E-01	.1526E-06	-.4705	-4.9150

Table VIII. Comparison of several measured mass attenuation coefficients with data of Table VII.

E - keV	$(\mu/\rho)_{\text{meas}} - \text{cm}^2/\text{g}$	$(\mu/\rho)_{\text{calc}} - \text{cm}^2/\text{g}$
A150		
9.87	4.30 ^a	4.17
17.43	0.948 ^a	0.934
28	0.370 ^b	0.381
C552		
9.87	5.58 ^a	5.28
17.43	1.07 ^a	1.06

^aD.R. White, private communication.

^bL. Anderson, private communication.

chieve adequate physical properties (Shonka, 1959) and will not be covered in this report. The range of temperature and pressure which are required for compression and injection molding of A150, A180, and C552 are indicated in Table IX. The vast majority of experience involves the three mixtures represented in the table. Furthermore, the mixtures A116, A174,

Table IX. The ranges of temperature and pressure required for molding of A150, A180, and C552 by means of the compression and injection molding methods.

Mixture	Compression Molding		Injection Molding	
	Temp. - °F	Press. - kpsi	Temp. - °F	Press. - kpsi
A150	300 - 400	0.2 - 2	350 - 450	5 - 20
A180	450 - 550	0.5 - 2	500 - 600	3 - 10
C552	400 - 500	0.5 - 20	450 - 550	10 - 50

B109, B110, and C514 would be expected to require substantially the same temperatures and pressures for molding as A150 since the same polymers are involved in the mixtures.

Depending on the particular part, its physical size, shape, and degree of intricacy, conditions at one or the other extreme of the ranges shown in Table IX may be required. Other considerations in determining a suitable molding procedure are the specific physical attributes which are important in the particular molded part and the degree of perfection which is sought. For example, if one is solely concerned about the degree of compaction as revealed by the average mass density, the lowest temperatures and pressures indicated in the table will be sufficient. Even so, one must determine how long the temperature and pressure needs to be maintained and whether the subsequent cooling rate needs to be limited to some maximum value.

The length of time that the forming temperature and pressure are maintained, the dwell time, is governed largely by the thermal conductivity and the molecular diffusion rates of the mixture. Thermal conduction is essentially the only means of getting heat into the interior regions of the molded part. Increasing the heating capacity of the mold proper would be to little avail when the thermal conduction of the plastic is the limiting factor. The dwell time also provides an opportunity for gases in the mixture to diffuse to the outside surfaces of the part and to escape through reliefs in the mold.

The allowable cooling rate is related closely to the thermal conductivity as well as to the amount of thermal expansion which occurs in the molded part. Cooling rates which are excessively rapid will result in distorted parts which do not conform to the shape of the mold and in cracks and fissures which in some cases will not be visible on the outside of the parts.

The minimum conditions which have been determined for molding a block of A150 14 inches in diameter and 3.8 inches in thickness are given here for purposes of illustration. In this case A150 is extruded directly into the mold which is initially closed and heated uniformly to 300 °F. As material flows into the mold, the piston, also called the core pin, is forced so as to disengage itself partially from the mold. This continues until the requisite degree of filling is achieved at which time the feed line to the mold is sealed. As soon as material begins entering the mold, the mold temperature controllers are reset to the value of 275 °F. When the transfer of material to the mold is completed, the transfer line is sealed, the mold controllers are reset to maintain 225 °F, and a force of 120 tons is

applied to the mold piston (this corresponds to a molding pressure of 1.5 kpsi). After 4 hours, the temperature and pressure controllers are turned off and the system is permitted to drift to room temperature overnight. As the temperature decreases, the pressure decreases also owing to the contraction of the plastic mixture.

The same mold has been employed for molding blocks of C552 of maximum thickness equal to 2 1/8 inches. Because of the poor flow properties of C552, it is not possible to extrude this mixture into the mold. Instead, granular C552 is placed in the mold, the mold piston is engaged, and heater power is turned on. A full load of granular C552 will be compressed approximately 50 % in achieving the final molded state. Three temperature controllers are used to maintain the temperatures of the mold base, side walls, and core pin. Initially the temperature controllers are set to 400 °F and the force on the piston is maintained at about 0.5 tons. When all three controllers show 400 °F the force on the piston is increased to and held at 165 tons (molding pressure of 2.2 kpsi). After a dwell of 1 hour, the temperature controllers are reset to 250 °F. Three hours later the temperature and pressure controllers are turned off and the force on the piston is reduced to 25 tons. The system is left to equilibrate with the surroundings from this condition. The following morning the disk may be removed from the mold and the molding of another disk may be started.

4. Density of Solid Conducting Mixtures

It has been noted previously that no interaction is expected between the polymers and additives employed in the mixtures described in this report.

Probably the same is true regarding the polymers themselves in those cases where two polymers are present in the same mixture since the temperatures to which the mixtures are subjected are not really very extreme. Accordingly it should be possible to determine a reliable value for the density of a mixture from densities of the constituents using Vegard's Law,

$$(1/\rho) = \sum_i (\gamma_i / \rho_i),$$

where ρ is the density of the final mixture, ρ_i is the density of the i -th constituent, and γ_i is the mass fraction of the i -th constituent. The results of such calculations are presented in Table X along with the available measured values. The relative differences between the calculated and measured values expressed as percentages are likewise shown.

The densities of the constituent polymers were taken from measurements whenever such have been made. In other cases, the mean of the extreme values given in the Modern Plastics Encyclopedia (McGraw-Hill, October 1977) were used. The particular values used in units of g/cm^3 are:

Nylon 6/6	1.14
Nylon 11	1.046 (meas)
Polyethylene	0.953
Polymethylpentene	0.830 (meas)
Polypropylene	0.905 (meas)
Polytetrafluoroethylene	2.17
Polyvinylidene Fluoride	1.765

The densities of the additives were taken as those given in Table III except for carbon where the value 1.944 g/cm^3 was used. This latter value was derived from analysis of the measured densities of the nylon 11-carbon and

Table X. Calculated densities for a number of conducting plastic mixtures.

Mixture	$\rho_{\text{calc}} - \text{g/cm}^3$	$\rho_{\text{meas}} - \text{g/cm}^3$	Rel. Diff. - %
A116	1.442	1.38	-4.3
A150	1.141	1.125	-1.4
A153	2.066	1.56	-24.5
A174	1.146	----	----
A180	1.067	1.053	-1.3
B100	1.461	1.45	-0.8
B101	2.081	1.90	-8.8
B109	1.512	----	----
B110	1.839	1.77	-3.8
C514	1.351	----	----
C552	1.802	1.76	-2.3
D350	1.110	1.08	-3.1
E100	1.008	1.008	0.0
E101	1.041	1.032	-0.9
E102	1.077	1.078	0.1
E103	1.116	1.116	0.0
E104	0.976	0.976	0.0

polypropylene-carbon mixtures, E103 and E104.

Since the density for carbon black was derived from nylon 11- and polypropylene-carbon mixtures it is perhaps not surprising that the best agreement between the calculated and measured values occurs in the nylon 11-polypropylene-carbon mixtures. The fact that all of the remaining measured values are less than the calculated ones suggests that perhaps the effective density of carbon black is less than the value assumed. Deviations in some of the other assumed densities and other unknown errors may also be contributing to the disparities noted. For example, the large disparities for A152 and B101 are surely due to molding procedures which did not yield complete compaction of the powders.

5. Special Welding Methods

Besides offering a wide latitude in the range of final compositions which are achievable with plastic hosts and an opportunity of molding required parts, parts made of many plastic mixtures can be neatly, simply, and securely joined by various special welding procedures. Many of these procedures are not possible with common metals and insulators. Of course, the ordinary joining and fastening methods such as: using threaded parts; using separate screws, nuts, bolts, rivets, clips, and pins; and using appropriate cements and adhesives may also be used with the conducting plastics. However several special procedures have been developed which take advantage of the unique characteristics of these mixtures. The methods are heat-sealing, friction, capacitor discharge, and ultrasonic welding. When applicable, extremely neat joints and connections can be achieved which are as strong as the materials themselves and are also gas-tight. For purposes of illustration

several specific examples are given below.

The heat-sealing method of joining parts was the first to be developed. There are two distinct versions of this method. The first generally involves flat surfaces. The parts are held in a fixture which holds them in proper relation to each other and which allows them to be readily brought into contact and held under compression. A metal plate of either aluminum, brass, or copper somewhat larger in area than the parts to be joined and of corresponding shape is heated to a temperature of 100 F degrees or more above the softening point of the materials to be joined. The parts are then forced together with the heated plate between them. When sufficient heat has transferred, the plastic parts are momentarily separated, the heated plate quickly removed, and the parts are forced back together and held for several minutes under a compressive force. The equator and neck welds of the 16 liter chamber depicted in Fig. 5 were formerly made by this method. Similar welds had also been used in spherical TE and Air chambers of diameter of 8 inches.

Of the methods described, the heat-sealing method produces the greatest distortion of the parts and generally results in the weakest bond. Accordingly, it has been replaced by another method wherever possible. The collector stem of the chamber depicted in Fig. 5 illustrates a situation where other methods fail so the heat-sealing method must be used. Here two relatively long solid round pieces are joined end-to-end.

The other version of thermal bonding is similar to a molding operation.

The parts are initially molded or machined to match each other and placed in

Fig. 5 A spherical 16 liter TE ionization chamber comprised of two hemi-spherical shells welded at the equator.

a mold which the parts completely fill. The mold is then heated and pressure is applied. Since the material fills the mold, no flow or movement of material will occur despite the elevated temperature and the pressure applied. Thus the individual parts will maintain their shapes as they are fused together. One may wish a cylindrical piece of conducting plastic to have a cylindrical core of an insulating plastic. Here a simple cylindrical mold is required to accomplish the bonding of the two matching parts. The same mold would also allow two or more round disks of different materials to be fused together in a sandwich arrangement.

Friction welding is a possible technique when round hollow sections need to be joined. The equator and neck welds of the chamber shown in Fig. 5 are two examples of friction welds. As mentioned earlier, these welds were formerly made by the heat-sealing method. To accomplish a friction weld the two parts are held in proper alignment in either a drill press or a lathe. The machine spindle should be equipped with a brake to allow quick stopping. While the part held on the spindle rotates at a suitable speed the stationary part is forced against it. The rubbing together generates heat softening the plastic. Just when the plastic begins to wear away the spindle is abruptly stopped. The force is maintained for a minute or so afterwards until the plastic becomes hard.

In this method the force exerted on the parts as they are rotated against each other should be increased in proportion to the area of the weld. A direct inverse relation should be used between the speed of rotation and diameter of weld thus maintaining an approximately constant relative

velocity between contacting elements of the two parts. This is based on the fact that at constant pressure the generation of heat will be proportional to the relative linear velocity.

It should be clear why it is necessary for the parts to be hollow. If the parts were solid at the joint, it would not be possible to have uniform heating conditions throughout the area of the weld. The elements near the axis of rotation would be essentially at rest and would not be heated directly.

Spindle speeds of approximately 500 rpm have been used for the equator weld as well as the neck weld of 8-inch TE and Air chambers similar to the 16 liter chamber shown in Fig. 5. In the case of the Air chambers the neck extension is made of Kynar (polyvinylidene fluoride), the host polymer of the air-equivalent plastic.

A welding technique which is applicable to conducting plastics and which resembles electric spot welding is that of passing an intense current pulse thru the joint to be formed. The current pulse is produced by discharging a bank of capacitors which are connected to the parts being joined. A major fraction of the electrical energy dissipated derives from Joule heating in the circuit resistance. Fortunately in this situation the resistance is largely concentrated at the contacting surfaces. The spherical collector tip of the 16-liter chamber of Fig. 5 and spherical outer electrodes of the chamber shown in Fig. 6 both have equator welds made by this method.

The hemispherical shells to be welded are placed in a closely fitting in-

Fig. 6 A 100 cc Air chamber requiring four separate ultrasonic and one capacitor discharge welds.

ulating sleeve. A tight fit is necessary to insure good alignment when the wall thickness is as small as .010 inches. The sleeve may be made of Teflon or some other convenient material with a thin (.010 - .020 inches) Teflon liner. The hemispheres are forced together with shaped electrodes frequently made of aluminum which make a broad-area contact close to the equator. The common axis of the hemispheres should be approximately parallel to that of the sleeve.

Welding parameters which have been found adequate for several representative cases follow. For an A150 collector shell $3/4$ inches in diameter and .010 inches in thickness such as shown in Fig. 5, 500 microfarads charged to 300 Volts and a clamping force of 7 pounds gave the best results. For similar shells made of C552, 500 microfarads charged to 250 Volts and a clamping force of 7 pounds yields good welds. In the case of the outer electrode of the chamber illustrated in Fig. 6, 4000 microfarads charged to 190 Volts and a clamping force of 42 pounds works satisfactorily.

Where the ultrasonic method is applicable it is to be preferred over the others. This method results in minimal distortion and is perhaps the quickest requiring only .05 - 0.2 seconds for the welding proper to be accomplished. These two facts go hand-in-hand. The sonic energy is delivered immediately and directly to the site where it is needed, the interface between initially separate parts. The method is equally applicable to conducting and insulating materials. One unique feature of this technique is that the energy required enters the part at some distance from the weld interface and therefore must penetrate some depth of material. The flow of sonic energy thru a part proceeds without causing any permanent change in the part and in some cases with little diminution in intensity.

Plastics vary considerably in their responsiveness to the ultrasonic technique. The best materials allow considerable penetration depths. But even the worst materials can be welded if the penetration depth is limited to a few millimeters. This latter situation is referred to as "near zone welding". Kynar and C552 respond similarly and decidedly superior to polyethylene and Al50 with respect to this technique.

Four separate ultrasonic welds are employed in the chamber shown in Fig. 6. These welds are made with a welding horn driven with a maximum power of 200 Watts. Weld 1 is between a C552 part and a Kynar part each 0.31 inches in diameter. The sonic transducer, "horn", is held against the part on the right with a force of 10 pounds. Full power is used for .05 seconds. Weld 2 is between two C552 parts both also having outside diameters of 0.31 inches. Here the horn bares against the part on the right with the force of 10 pounds. Only 60 % power is used for 0.1 seconds to make the weld. Weld 3 involves a Kynar neck extension of outside diameter of approximately 0.5 inches welded to a C552 necked hemisphere. A horn having diameter of 3/4 inches is employed, whereas the first two welds and weld 4 employ a horn of diameter of 1/2 inches. The horn is held against the Kynar part with a force of 10 pounds and a sonic burst lasting 0.2 seconds at 80 % power is used. The fourth weld is between C552 parts of outer diameter 0.25 inches. The force for this case is 8 pounds and a burst of 0.15 seconds duration at 70 % power makes an excellent joint.

The last three sonic welds just described may be accomplished by other methods but doubtlessly neither as efficiently nor with as sturdy a resultant weld. However, the requirements of the first weld would not permit it to be made

with any of the other methods. There are numerous situations where ultrasonic welding allows approaches and designs which may otherwise be impossible.

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