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FABRICATION OF TEFLON
BY HYDROSTATIC PRESSING

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FABRICATION OF TEFLON
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ABSTRACT

Functional parts have been produced by hydrostatic pressing of Teflon. Pressures ranging from 5,000-30,000 psi can be used. Shrinkage of parts from pressing sac cavity dimensions to the sintered form is anisotropic, but falls into patterns related to both size and shape of the product. All commercially available forms or grades of Teflon have been processed satisfactorily. The process is particularly suited to production of shapes difficult or impossible to produce by the more conventional methods described in the literature.

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I. INTRODUCTION

It is the purpose of this paper to describe briefly the successful fabrication of functional parts from Teflon polytetrafluoroethylene by means of hydrostatic pressing. No large number of such parts has been made, nor has any degree of detailed research been done into many of the specific variables potentially involved in the process. Teflon is a product of the Polychemicals Department of E. I. DuPont de Nemours, Wilmington, Delaware. Its properties and fabricating qualities are generally well described in DuPont literature, such as the currently available Bulletin No. X-7d, "Teflon, TFE Fluorocarbon Resin Molding Techniques." The hydrostatic pressing process and its potential application to fabrication of Teflon has not been described in DuPont processing manuals other than as implied in general statements to the effect that Teflon can be fabricated successfully by processes more nearly like those used for powdered metals and ceramics than those used to process conventional thermoplastics.

II. FABRICATION OF FUNCTIONAL PARTS

Dip Tubes

In 1959, there was need for a "dip tube" in oralloy recovery apparatus which was required to operate in hot mixtures of HCl , HNO_3 , H_2SO_4 and other acids necessary to digest waste materials. The environmental requirements clearly indicated that a material with the properties of Kel-F or Teflon should be used to make the part which was specified to be a flanged tube with an overall length of approximately 16 in., a minimum bore of 1.25 in., and a 0.2 in. thick flange with a diameter of 5.0 in. minimum. Wall thickness of the part, for mechanical reasons, was set at 0.15 in. minimum.

Earlier work at LASL involving compression molding of both Teflon and Kel-F had proven the difficulty of fabricating heavy section shapes. The part was considered much too long to be made by compression in the axial direction. The necessity for the flange would have been a complication in design of a mold for Teflon, taking into account the powder bulk factor, and the problems of relative mold part movements necessary to avoid internal shear within the part as it was pressed to shape.

Attempts to make a useful part by a tape-wrapping process using unfused Teflon tape (Scotchplastic #547, a product of the 3M Company) resulted in failure by shrink-cracking of the part during fusion. Changes in wrap tension and in fusion cycles produced no significant reduction of the magnitude or frequency of the typical defects.

Attempts to "flare-form" heavy wall Teflon tubing in a manner since used successfully to produce commercial Teflon lined pipe were also one hundred percent failures, largely attributed to difficulties in temperature control coupled with the well-known tendency of Teflon to fail in internal shear when heated above its transition temperature and subjected to forming pressure.

It was decided that there might be a reasonable possibility of success by using the hydrostatic pressing process. A chamber of adequate size with capability of reaching 30,000 psi was available, and the technology for producing cast vinyl pressing sacs had been developed locally to a high degree.

Initial experiments to produce cylindrical slugs of Teflon 7X at 10, 15, and 30,000 psi resulted in the achievement of unsintered (green) densities of 2.20, 2.21, and 2.22 g/cc respectively. The slugs were fused successfully on a standard cycle used for conventional compression molded Teflon parts. A section of thin wall tube (approximately 0.8 in. bore x 1.03 O.D. x 3.1 in. long) was pressed in the green state, and fused without trouble. A steel mandrel or core was used to maintain the bore dimension, and was pressed out of the green part before sintering as is usually done with metal powder pressings.

A sac system was then designed for production of a full-scale dip tube with generous allowance made for the Teflon bulk factor and for shrinkage of the part during fusion. The first part made was satisfactory in all respects except one: the body of the tube was warped. Subsequent parts were fused while supported by an internal dural tube of a size slightly less than the anticipated bore of the Teflon. Sufficiently true tubes were made by this expedient to satisfy the original requirement. Occasionally a tube shrank onto the dural core, in which case the core was dissolved.

It was found that green parts could be machined to remove excess material before sintering. Machine turnings were pressed hydrostatically in a test sac at 30,000 psi to a density of 2.19 g/cc to produce a short 1.0 in. dia rod which was subsequently fused satisfactorily.

Figure 1 is a photograph of a pair of the dip tubes. The scale is metric. Flanges warp slightly, becoming concave toward the tube body. Slight "bumpiness" or surface irregularities which can not easily be seen in the photograph are characteristic. This phenomenon is variously attributed to minor differences in packing of the powder when the sac is loaded, internal temperature differences during fusion, minor flaws or variations in wall thickness of the pressing sac, variation in degree of crystallinity of the part caused by temperature and cooling rate differentials after sintering, or to some combination of these factors. No work has been done to investigate the effects of any of these variables.

Radiographic inspection of the finished parts showed no major density variations and no voids.

Feed-through Insulators

In late 1963, there was a need for a series of electrical feed-through insulators in the construction of precision counting equipment. The design of the apparatus had been proven with commercial Teflon insulators of a size too small for use in the large unit planned for construction, and no commercial source for sufficiently large parts was known. The first part needed was specified to have a 0.75 in. body diameter, a 4.0 in. length, and contain a #14 copper conductor (0.064 in. dia) located concentric with the part axis. The conductor was to carry 40 KV through the wall of a steel vacuum chamber operating at 2×10^{-7} mm Hg pressure.

FIGURE 1

Hydrostatically Pressed Teflon Dip Tubes

Despite lack of more knowledge of the hydrostatic pressing process as applied to Teflon than has been noted above, and the continued silence of DuPont on the subject, it seemed evident that the best system for production of the required part was hydrostatic pressing. Because other, larger diameter insulators were expected to be needed, the sac cavity diameter was set at 2.0 in. Push-fit end caps for the sac were made with centered holes 10% smaller in diameter than the core wire, and spaced at 6 in. from one another to allow for shrinkage along the part axis. The copper wire was burnished before the mold was loaded and held under slight tension until pressure application was completed. The first part made was pressed at 30,000 psi and fused on the standard cycle with good results. It was machined to the final size and contour required for mounting in a self-gasketed packing gland arrangement. It has functioned satisfactorily for approximately 6 months. Some corrosion of the copper occurred during fusion, as was expected. (This could be avoided by sintering sensitive parts in an inert or reducing atmosphere.)

III. PROCESS DETAILS

Many facets of the general process of hydrostatic pressing as applied to Teflon have not been investigated in any detail. The emphasis has always been placed on production of a functional part in a short time, and no systematic evaluation of variables has been attempted. In the few paragraphs which follow, such information as would be of probable value to one who wished to use the process has been collected.

Sac Materials and Design Features

All work done at LASL has been in mandrel-cast PVC plastisol sacs equipped with slush cast push-fit closures. (There is no valid reason for restricting sacs to this type of material. It happens to be the system most often used by both ceramists and powder metallurgists at LASL and was therefore both convenient and economical.) Usually a 2% interference fit is specified on PVC/PVC joints, and a 2-10% stretch fit over core mandrels where they protrude through a sac wall. PVC/PVC joints are usually best sealed by use of raw plastisol spread over the joint area and fused in a stream of hot gas from a conventional plastics welding gun. The preferred PVC plastisol formulation is one which has been described in detail in other reports.* Sac wall thickness has been of the order of 0.06-0.08 in. in most of the work with Teflon.

Sacs can be re-used in most instances; but, in most of the work done with Teflon at high pressures, new sacs were used for each part.

-
- * (1) J. S. Church and J. K. Donahoe, "Flexible Neutron Shielding Systems Based on Vinyl Plastisols," LAMS-2800, February, 1963 (Table I, p. 14, gives composition, sources of materials, etc.)
 - (2) J. S. Church, "Vinyl Coating of Graphite Plates for Ultrasonic Inspection," LAMS-2211, June, 1958 (This report describes function of ingredients, gives details of preparation, fusion conditions, etc.)

Shrinkage Allowances

Allowances made for compression of the powder and subsequent true shrinkage on sintering have been generous because of lack of good information on which to base accurate calculations. Exact values are logically dependent on: type of Teflon, loading system, molding pressure, sac wall thickness, part and sac design features, presence/absence of core, and sintering conditions. No work has been done to define the actual contribution of any one factor or combination of factors. Improvement in the loading operation is believed to hold the greatest promise for reducing shrinkage below the levels experienced in the work done to date. Experimental values are given in Table I with calculated percentage reduction in size from the cavity to the corresponding sintered part dimension.

At present, it appears that for parts with appreciable length/diameter ratios of the magnitude 2-5, the sintered part diameter will be 40-50% smaller than the sac cavity, and the sintered length will be decreased to 70-75% of the sac cavity. Corresponding effects should be expected for tubes, except the correction should be applied to the wall thickness.

If the length/diameter ratio is smaller than 2, radial allowance should be made for the order of 20% reduction, and thickness or length allowance should be 50-60%.

If the length/diameter ratio is larger than 5; length reduction of 10-15% should be anticipated in conjunction with a radial reduction of 40-50%.

TABLE I

Typical Dimensional Changes During Processing
of Hydrostatically Pressed Teflon Parts

(dimensions are in inches)

Insulator - 30,000 psi - Teflon 7X

<u>Process Stage</u>	<u>Diameter</u>	<u>Length</u>
sac cavity	2.0	6.0
green compact	1.25 ⁽¹⁾	4.5 ⁽¹⁾
sintered part	1.2	4.4
finished part	0.75 ⁽²⁾	4.0 ⁽²⁾
size reduction cavity/sintered part	40%	27%

Dip Tube - 30,000 psi - Teflon 7X

<u>Process Stage</u>	<u>Tube</u>			<u>Flange</u>	
	<u>I.D.</u>	<u>O.D.</u>	<u>Length</u>	<u>Diameter</u>	<u>Thickness</u>
sac	1.5 ⁽³⁾	3.10	17.2	6.6	0.6
green	1.5	2.49	17.0	5.4 ⁽¹⁾	0.35 ⁽¹⁾
sintered	1.37	2.12	16.3	5.3	0.3
size reduction cavity/sintered part	9%	32% ⁽⁴⁾	5%	20%	50%

- (1) Approximate dimension, as machined to "clean up" contour.
- (2) As finally machined after sintering.
- (3) Steel mandrel.
- (4) The corresponding value based on total wall thickness reduction is 53%.

Form or Grade of Teflon

Parts have been made successfully with Teflon 1, 5 and 7X as well as with turnings machined from green compacts of Teflon 7X. Working pressures can be reduced for the series 7X turnings, Teflon 1, Teflon 5, and Teflon 7X, decreasing in the order listed. Turnings from Teflon 1 and 5 have not been reprocessed, but should be reprocessable. Generally, the comments in DuPont literature relative to the merits of choosing a grade of Teflon for compression molding appear to apply equally well to the hydrostatic pressing process. Slight differences in part density for compacts made at equivalent pressures occur in the order: Teflon 7X, Teflon 5, Teflon 1, 7X turnings. Teflon 7X exhibits the highest density; the range is the order of 0.03 g/cc.

Loading the Mold

Teflon is susceptible in a high degree to stratification and subsequent cracking or fracture during sintering unless great care is exercised in loading the mold. Precautions recommended by DuPont literature for compression molding are quite realistic for hydrostatic pressing. Powder should preferably not be tamped or rammed in increments unless cracks perpendicular to the tamping force can be tolerated in the finished part. The larger parts made successfully at LASL were loaded on a vibrating table. Tamping can be done after the whole charge is in place and if an even force is exerted, for instance to reduce the bulk and allow an end cap to be seated.

Sacs for larger parts should be supported in a wire mesh or expanded metal container during loading to avoid distortion of the sac (and its product). The support can be used for handling the loaded sac and is usually left in place during the pressing operation.

Equipment

Two hydrostatic pressing chambers have been used for pressing Teflon and Nylon at LASL; both chambers were tested at 45,000 psi and are rated for 30,000 psi operation. An 8.00 in. I.D. x 20.0 in. depth chamber manufactured by the High Pressure Equipment Company of Erie, Pennsylvania, was employed for pressing the dip tube, and a 2.38 in. I.D. x 28 in. depth chamber manufactured by the American Instrument Company, Silver Springs, Maryland, was employed for initial feasibility work and for pressing the feed-through insulators. Equipment used to pump fluid into the pressure chambers was manufactured by Sprague Engineering Corporation, Gardena, California.

The pressure transmission fluid is SAE 10 or 10W oil, spindle oil, or a 10:1 dilution of Cimcool water soluble cutting oil. The last material is a product of the Cincinnati Milling Machine Company and is preferred because it is non-flammable. The general arrangement of the equipment, piping, valves, etc. was not modified in any way from that used routinely for pressing powdered metals and ceramics.

Pressing

Only a small amount of work has been done to investigate the effects of pressure on product quality or properties. Such work as has been done has not been definitive in the sense that finished part density has been used as a criterion of quality, but no concurrent study of degree of crystallinity has been made. One set of data has been plotted as Figure 2. All the parts were sintered on the same cycle, and all were nominal 2 in. dia. cylinders, so comparative values are realistic as plotted. The specific density values shown are in doubt because of the known variation in density of Teflon with degree of crystallinity which results in values ranging from 2.0 to 2.3 g/cc for parts which are considered to be void-free.*

* Gillespie, Sacton, and Chapman, "New Design Data for FEP and TFE, Part I, Strength and Deformation," Machine Design, Jan. 21, 1960.

TEFLON 7X
HYDROSTATICALLY PRESSED AND SINTERED

FIGURE 2
Variation of Sintered Density With
Hydrostatic Pressure

Actual sintering conditions employed were such as to encourage formation of crystalline material.

All of the production parts made at LASL have been pressed at 30,000 psi to maximize density, decrease porosity, and enhance electrical resistivity. It is quite possible that pressures in excess of 10,000 psi are not necessarily of any real benefit. LASL experience with true compression molding has been that best results were achieved in the range 4-6,000 psi, and that pressures above 10,000 psi led to "rebound," stratification or layer slippage, and fracture during sintering. These effects have not been noted with high pressure hydrostatic pressing.

The effects of rate of pressure application and/or pressure release have not been investigated. Based on generalized experience with malleable metal powders, the conservative practice of increasing pressure steadily over an interval of about 30 minutes, maintaining pressure 2-5 minutes (up to 30 minutes for heavy sections), and releasing pressure over a 5-15 minute interval has been adopted as standard. It is quite possible that shorter cycles could be used, but the pumping rate of the equipment available until recently set practical limits, and there has not been any urgent reason to reduce cycle time when only a few parts were to be made.

Fusion or Sintering

Essentially all of the parts which have been pressed hydrostatically have been sintered on the very conservative cycle generally used to avoid fractures during heating. Parts have neither been "coined" nor "quenched." Heating is at the rate of 100°C/hr to 200°C, 50°C/hr to 380°-385°C, followed by a dwell of 3 hours in that temperature range. Cooling is programmed at the same rate as heating, but usually is somewhat slower because of the lag of the oven behind the program, particularly below 275°C. Vented mechanical convection ovens have

generally been used. For parts of unusual contours, severely contrasting sections, or very heavy sections, adjustments in the cycle have been made in the directions recommended by DuPont literature. Obviously, had part functional requirements been such that amorphous material possessed advantages over the highly crystalline form, cooling would have been accelerated by quenching.

IV. CONCLUSIONS

It has been demonstrated that the hydrostatic pressing process can be applied to fabrication of functional parts from Teflon. The technique is by no means recommended for universal adoption, but is strongly endorsed for use in specific instances where other, better-known methods are difficult, expensive, or impossible. These special situations are largely those in which: (1) only a few comparatively large parts are to be made, (2) severe changes in section thicknesses are necessary and the conventional compression mold is therefore impractical, (3) an insert is required, (4) maximum uniform density is desirable for correct function of the product. If more than one of these factors are present, and access to an adequate hydrostatic pressing chamber can be had, the process is well worth investigation. Tooling cost per unit is comparatively low if the number of parts to be made is small, and the method does not seem to be very sensitive to variations in control parameters. If the part need not be pressed and sintered to final dimensions, but can be machined from an oversized blank, it is highly possible that the first piece made will be usable.

Much work could be done to delineate more clearly the real limitations and particular advantages of the hydrostatic process as it can be applied not only to the fabrication of parts from Teflon, but also to certain other thermoplastics. Specifically, the effects of mold loading variables on part density and shrinkage should be investigated in some detail. This portion of the process is believed to be most influential in control of shrinkage.