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Research Article

CHARACTERISATION OF POLYTETRAFLUOROETHYLENE (PTFE) BASED COMPOSITES BY STUDYING THEIR PHYSICAL PROPERTIES

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Abstract

Polytetrafluoroethylene (PTFE) is a versatile engineering plastic. It has excellent chemical and water resistance, moderate mechanical and thermal properties, superior electrical insulation properties with low coefficient of friction. As virgin PTFE has low wear, creep resistance and low load bearing capacity, different fillers are therefore incorporated into PTFE to improve these properties. In the present work, a systematic study on the effect of different 15 fillers like granite, porcelain, wollastonite, bronze etc; on various physical properties like bulk density, specific gravity and expansion or contraction in height, diametrical shrinkage. The results showed that highest bulk density of all the fillers studied was obtained for bronze powder (4065.2gm.cc). Among PTFE Composites studied, higher specific gravity (3.396) was found in bronze filled PTFE Containing 50% filler content before and after sintering (3.465). Whereas, the highest expansion value (14.23) was found in case of 50% mica filled PTFE and 5% Tixolex-25 filled PTFE has highest value of 5.882 diametrical shrinkage.

Keywords: PTFE, Fillers, Bulk density, Specific gravity, Shrinkage, Hazard.

Introduction

Teflon is the registered trade name of the highly useful plastic material polytetrafluoroethylene (PTFE), PTFE is one of a class of plastics known as fluoropolymers. It has many unique properties, which make it valuable in scores of applications. Teflon has a very high melting point, and is also stable at very low temperatures. It can be dissolved by nothing but hot fluorine gas or certain molten metals, so it is extremely resistant to corrosion. It is also very slick and slippery. This makes it an excellent material for coating machine parts which are subjected to heat, wear, and friction, for laboratory equipment which must resist corrosive chemicals, and as a coating for cookware and utensils (Gong Xue et.al., 1989) PTFE is used to impart stain-resistance to fabrics, carpets, and wall coverings, and as weatherproofing on outdoor signs. PTFE has low electrical conductivity, so it makes a good electrical insulator. It is used to insulate much data communication cable, and it is essential to the manufacture of semi-conductors. PTFE is also found in a variety of medical applications, such as in vascular grafts. A fiberglass fabric with PTFE coating serves to protect the roofs of airports and stadiums. PTFE can even be incorporated into fiber for weaving socks. The low friction of the PTFE makes the socks exceptionally smooth, protecting feet from blisters (Bahadur et.al., 1984). Even though, the virgin PTFE is having low wear creep resistance and low load bearing capacity, the different fillers are incorporated into PTFE to improve these properties.

Structure of PTFE

The PTFE molecule has highly regular structure with a configuration leading to a 166 helix. It is a fully fluorinated polymer with a linear chain of great molecular length (20,000 to 1,00,000 Monomer units) and highly crystalline polymer (90-95%) with a melting point of 327^{o}C. It has a high melt viscosity (10^{11} poise at 380^{o}C). The working temperature of PTFE is –260^{o}C to +260^{o}C. PTFE is insoluble in any of the commonly known solvents. Certain fluorinated oils and molten alkali metals, etc, attack PTFE. Its structure and chemical formula is

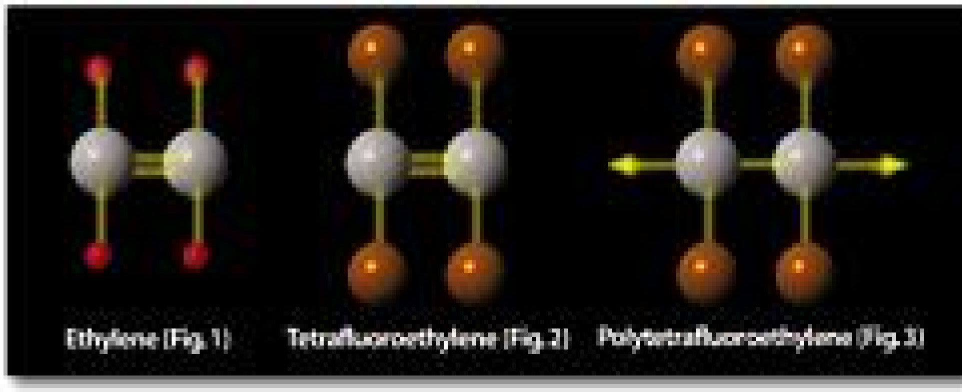

Figure 1-- (- CF2 –CF2-)$_{n}$–PTFE is made of a carbon backbone chain, and each carbon has two fluorine atoms attached to it.

Properties of PTFE:

(M/s Hindustan Fluoro Carbons Ltd., Hyd., Telangana., India.)

A. Physical and chemical properties:

Specific gravity	2.14to2.18ASTM-D1457/87
Weather resistance	Excellent
Chemical Resistance	Excellent
Water absorption	Nil

B. Thermal properties:

1. Melting point	^{o}C	327
2. Working temperature	^{o}C	-260 to +260
3. Heat of combustion	mJ/kg	5.0

Manufacture of PTFE:

Polymerisation is carried out by suspension and emulsion methods using peroxide / red oxide initiators All granular grades of PTFE for compression moulding are made from suspension polymerization method; emulsion grades of PTFE and coagulated fine powder are made from Emulsion Polymerization Method.

Processing of PTFE:

Basic processing techniques used for PTFE are fundamentally different from those commonly used with other thermoplastics. The melt-viscosity of PTFE is very high and it does not flow even at temperature above the melting point. PTFE is processed similar to the techniques followed in powder metallurgy and ceramics; granular PTFE powders are processed by one of the following methods: a)Compression moulding: Compression moulding process is usually done in a mould to produce semi-finished shapes, rods, tubes, blocks, sheets, slabs which are later machined after sintering to form a variety of end products. In addition to virgin (PTFE) grades, a wide variety (PTFE) grades, a wide variety of filled grades are also available for compression moulding. Incorporation of fillers in PTFE gives the better compression strength, lower thermal expansion, better wear resistance (Masaki et.al., 1986). b)Sintering: Sintering is done in an electrically heated oven having forced air circulation arrangement. It must be capable of continuous operation with a programmed sintered cycle from 0 to 375^{o}C $\pm$ 5^{o}C. PTFE will retain its moulded shape even at sintering temperature (Kobayashi et.al., 1967). Sintering cycle varies depending upon the dimensions of component being sintered. After sintering the cooling cycle is very important to give desired crystallinity. Slow cooling gives maximum crystallinity while rapid cooling gives low crystallinity (Mateus et.al., 2004) .

Importance of filled PTFE

Virgin PTFE is inadequate for a number of more demanding engineer uses. In particular, its cold flow or creep kept PTFE out of mechanical applications (Oshima et.al., 2001). In the sixties, it was discovered that the physical properties of PTFE could be improved by the addition of fillers (Ganz et.al., 1966) and a range of filled compounds were found to be highly suitable for gaskets, valve sheets, piston rings, high voltage switches, bearings, pipe lining, etc. It turned out that through a proper combination of basic resin and one or more fillers, compounds could be tailor- made for many useful applications\

Practically any material that can withstand the sintering temperature of PTFE (upto 400^{o}C) can be used as filler depending upon the application (Herts et.al., 1978). Important characteristics of the filler are Physical properties, Particle shape, size and surface finish, bulk behavior, Chemical nature. Amount of filler added to PTFE may be expressed as a percentage by weight or as a percentage by volume. The former has gained favour as the means of describing materials, but it is the latter that is the more important factor in determining the properties optimum fabricating conditions and ultimate performance of the product. It is generally accepted that the maximum useful filler content is about 40% by volume (Bahadur et.al., 1984).

Applications:

From the sintered billets, simple shapes like bushes, rods, thick sheets, piston rings, etc, can be made (Talat Tevruz 1998).

Materials and Methods

Selection of raw materials (Fillers) and their characterisation:

Polytetrafluoroethylene (PTFE) used in the filled grades studied was HIFLON 71 grade. The properties of HIFLON 71 and fillers used are given below –PTFE (H 71)

Hiflon H 71 is a white fine powder of virgin PTFE grade with an average particle size of ≈50 microns. It is non-flowing and lumpy due to very fine particle size. This grade is generally recommended for making various filled grades.
The properties of this grade are given below.

Name of the property	Units	Value	Standard
Appearance	Irregular white particles		
Bulk density	gm/cc	425 ± 100	ASTM-D 1457/87
Compression ratio	-	4 to 5	-do-
Particle Size		≈50 microns	-do-
Pourability	S	NF	ASTM-D 1895
Apparent Density	gm/cc	325 to 525	-

The properties of sintered virgin PTFE and filled PTFE

Appearance	Units	Value	Standard
Specific gravity		2.142 – 2.18	ASTM-D 1457/87
Shrinkage	%	-3.5 to –4.5	-do-
Tensile strength	kg/cm^2	300 to 350	ASTM-D 1457/87
Elongation	%	350 to 450	ASTM-D 1457/87
Dielectric strength 0.1mm	kV/mm	88 approx	ASTM-D 1457/87
Thermal stability index	-	51 max.	-

Moulding pressure: 150 to 250 kg/cm^2

1. Glass (F) Filler:

Glass fibre is the most widely used filler. It improves the creep resistance of PTFE, both at low and high temperatures. It is chemically stable (except to strong alkalies and hydrofluoric acid – HF). It has little effect on the electrical properties of PTFE and its wear and friction behaviour. A known uncommon problem with glass filled PTFE is discolouration of the unfinished parts, in particular on the inside of the large billets. The glass used in PTFE compound was treated by a proprietary process by certain resin manufactures to reduce this discolouration.

Type: E – Glass Miled fibres, nominal dia, 13 micron or 13 x 10^{-6} m.

Appearance	:	White powder
Nominal length	:	0.8 mm or 8 X 10^{-4} m
Aspect ratio	:	Min. 10
Density	:	2.5 gm/cc
Particle size	:	800 microns
Name of the supplier/source	:	M/s. Owens Corning fibre glass
Corporation, TELEDO, OHIO.		
Screen size	:	794 micron
Moisture, % w/w	:	0.08 maximum

2. Alumina (Al_2O_3) Filler:

Alumina in a highly pure form is obtained by dehydration of high grade bauxite after it is purified from iron oxide. It is an excellent electrical insulator and is used to improve mechanical properties of compounds used in high voltage applications. As it is very hard, machining of the sintered part should be avoided whenever possible. Complicated shapes should be made by isostatic moulding.

Alumina of aluminium oxide is an electrical insulator and is used to improve mechanical properties of compounds used in high voltage applications. As it is very hard, machining of the sintered part should be avoided whenever possible. Complicated shape should be made by isostatic moulding process.

Purity of alumina	:	99.5%
Particle size	:	< 25 microns
Name of supplier	:	Carborandum Universal Ltd.Cochin.

3. Garnet Filler:

Garnet Stones are very hard and whitish in colour with unknown composition.

Source: University College of Technology, Osmania University, Hyderabad

garnet stones are broken into pieces and crushed and ground in a ball mill at the laboratories of College of Technology, OU, Hyderabad

4. Graphite Filler:

Graphite is a crystalline modification of high purity carbon. Graphite filled PTFE has one of the lowest coefficients of friction. It has excellent wear properties, in particular against soft metals, displays high load carrying capability in high speed contact applications and is chemically inert. It is often used in combination with other fillers.

Appearance	:	Black fine powder, irregular particles.
Refuse on sieve	:	44 microns
Percentage w/w	:	1.0 max.
Source	:	Synthetic
Purity	:	>99% C, 1.00 % ash content
Particle size	:	< 7.5 m or < 75 x 10^{-6} m
Density	:	2.26 gm/cc

Name of the supplier : M/s. Industrial Graphite, Uppal, Hyderabad.

5. Tixolex – 25 Filler:

Tixolex-25 is a white fine free flowing, synthetic hydrated sodium silico aluminate obtained by precipitation. The properties of the tixolex-25 are given below –

Sl. no.	Characteristics	Units	Value
01.	Drying loss (2 hrs at 105^{o}C)	%	8 max.
02.	Ignition loss at 900^{o}C	%	14 max.
03.	DOP absorption	100 gm/cc	200 – 270
04.	Specific gravity		2.0 – 2.1
05.	Ultimate particle size	micron	Apprx. 20
06.	Tap density	gm/cc	0.28 – 0.33

6. Wollastonite Filler (Casio$_3$):

It is a natural calcium silicate. It is available in metamorphic rocks.
Properties : Colour white to brown, red, grey, yellow
Mohs hardness : 4.5 – 5
Specific gravity. : 2.8 – 2.9
Occurrence : New York; California

Uses : Fine, medium paint grades, ceramics; paint extender; welding rod coat, rubber filler; silica gels; paper coating; filler in plastics; cements, and wallboard; mineral wool; soil conditioner.
Source : Wolken Limited, Udaypur, Rajasthan.

7. Marble Filler:

It is a metamorphic form of calcium carbonate usually containing admixtures of iron and other minerals, which impart various colour patterns. Marble chips are often used as source of CO_2 in laboratory experiments.

Source: University College of Technology, Osmania University, Hyderabad.
Marble stones are broken into pieces and crushed and ground in a
ball mill at the laboratories of College of Technology, OU, Hyderabad

8. Granite Filler:

Granite is coarse – grained igneous rock consisting essentially of quartz (20 – 40%) alkali feldspar and mica. They have a high proportion of silica (77%) and relatively high soda and potash. It has a low specific gravity (2.7 gm/cm^2) and this fact is of great importance in understanding the nature of continental and oceanic surfaces. Granite and related rocks make up the great bulk of the continental crust. Granite ranges in colour from light gray to medium gray and pink. The colour of feldspar has the greatest influence on the overall colour of the rock.

Source : Khammam Granite (Stone Rock) quarries. Black granite rocks are broken into pieces and crushed and ground in a ball mill at the laboratories of College of Technology, OU, Hyderabad

9. Carbon Filler:

Amorphous carbon is on the most inert fillers, except in oxidizing environments, where glass performs better. Carbon adds to the creep resistance, increases the hardness and raises the thermal conductivity of PTFE. Carbon filled compounds have excellent wear properties, in particular when combined with graphite. The combination of the above properties makes carbon/graphite compounds and the performed material for non-lubricated piston rings. The use of softer carbon has the additional advantage that it lowers tool wear during machining. Thus allowing machining to very close tolerances. Carbon containing compounds have some electrical conductivity and are therefore antistatic.

Base	: Amorphous petroleum coke.
Purity	: 99%
Particle size	: <75 microns
Density	: 1.8 gm/cc

10. Bronze Filler:

It is an alloy of copper and tin, usually containing from 1 to 10% tin; special types contain from 5 to 10% aluminium (aluminium bronze); fractional percentages of phosphorus (Phosphor bronze) as deoxidizer or low percentages of silicon (silicon bronze). Addition of high percentages of bronze powder to PTFE results in a compound having high thermal conductivity and better creep resistance than most other compounds. Bronze filled PTFE is often used for components in hydraulic systems, but is not suited for electrical applications and it is attacked by certain chemicals. Bronze has a tendency to oxidize, bronze filled compounds should therefore be used fresh and containers should always be kept closed. Some discolouration of the finished part during the sintering cycle is normal and has no impact on its quality.

Appearance	: Brown powder
Flow ability, sec/gm	: 20/30 sec/50gm
Apparent density gm/ml	: 3,200-4.800
Refuse on sieve at 63 microns	: 0.1 max
Refuse on sieve at 40 microns	: 5.0 max
Particle size	: < 60 microns
Density	: 8.95 gm/cc
Copper %, w/w	: 88.2 – 89.8
Tin, % w/w	: 8.5 – 9.5
Zinc, %, w/w	: 1.7 – 2.3
Phosphorous, % w/w	: 0.1 max.
Others, % w/w	: 0.5 max

Hazard	: Powder is flammable
Uses	: Spark-resistant tools; springs; four drinier wire Paint; cosmetics (as powder); electrical hardware, Architecture; fine arts;
Source	: Metal powder Limited, Koyambattur.

11. Mica Filler:

Any of several silicates of varying chemical composition but with similar physical properties and crystal structure. All characteristically cleave into thin sheets, which are flexible and elastic. Synthetic mica is available, it has electrical and mechanical properties superior to those of natural mica; it is also water-free.

Properties: Soft, tanslucent solid, non-combustible; Heat resistant to 600 ^{o}C; colourless to slight red (ruby); brown to greenish yellow (amber).

Specific gravity : 2.6 – 3.2

Refractive index : 1.56 – 1.60
Dielectric constant : 6.5 – 8.7
Hazards (Dust) : Irritant by inhalation; may damage the lungs.

12. China Clay (Kaoline) Filler:

A white – burning aluminium silicate which, due to its great purity, has a high fusion point and is the most refractory of all clays.
Composition : Mainly kaolinite (40% alumina, 55% silica, plus impurities and water).
Properties : White to yellowish or grayish fine powder;
Specific gravity : 1.8 – 2.6
Insoluble in water, dilute acids and alkali hydroxides. Has high lubricity (slipperiness) nontoxic, noncombustible.
Occurrence : Southeastern U S, England, France.

Grades : Technical; NF; also graded on basis of colour and particle size.

Containers : Cartons; paper bags; drums bulk

Uses : Filler and coatings for paper and rubber; refractories; ceramics; cements; fertilizers, chemicals (especially aluminum sulfate) catalyst carrier; anticaking preparations; cosmetics; insecticides; paint; source of alumina (see toth process); adsorbent for clarification of liquids; electrical insulators.

13. Antimony Trisulphide Filler:

(Antimony orange; black antimony; antimony needles : antimonous sulfide; antimony sulfide) Sb_2S_3.

Properties : (a) Black crystals (b) Orange-red crystals. Insoluble in water; soluble in concentrated hydrochloric acid; and sulfide solutions.

Specific gravity : 4.562
Melting point : 546^{o}C.
Derivation : (a) Occurs in nature as black crystalline stibnite

(b) As precipitated from solutions of salts of antimony-trisulphide is an orange-red precipitate, which is filtered, dried and ground.

14. Porcelian Filler:

Potassium aluminum silicate (4 K_2O-Al_2O_3 $3SiO_2$). A mixture of clays, quartz and feldspar, usually contain atleast 25% of alumina. Ball and china clays are ordinarily used. A slip or slurry is formed with water to form a plastic, moldable mass, which is then glazed and fired to a hard, smooth solid.

Properties: High impact strength; impermeable to liquids and gases; resistant to chemicals except hydrofluoric acid and hot, string caustic solutions; usable upto $1093^{o}C$ but subject to heat shock.

Specific gravity	: 2.41
Compression strength	: 100.00 kg/cm^2
Grades	: Chemical and Electrical

Uses : Reaction vessels, spark plugs, electrical resistors; electron tubes; corrosion resistant equipment, ball mills and grinders; food processing equipments, piping, valves, pumps, tower packing, lab ware.

Source: University College of Technology, Osmania University, Hyderabad
Cured Porcelian blocks are crushed and ground in a ball mill at the laboratories of College of Technology, OU, Hyderabad.

15. Sand (Silica/ Silicon Dioxide) Sio_2 Filler: Occurs widely in nature as sand, quartz, flint and diatomite.
Properties : Colourless crystals or white powder ; Odorless and tasteless.

Specific gravity: 2.2 – 2.6

Insoluble in water and acids except hydrofluoric ; soluble in molten alkali when finely divided and amorphous. Combines chemically with most metallic oxides. Noncombustible; melts to a glass with lowest known coefficient of expansion (fused silica). Thermal conductivity about half that of glass.

Melting point : $1710^{0}C$;
Boiling point $2230^{0}C$; High dielectric constant; high heat and shock resistance.
Hazard: Toxic by inhalation; chronic exposure to dust may cause silicosis.

Uses (powder) : Manufacture of glass, water glass, ceramics; abrasives; water filtration;microspheres; component of concrete; source of ferrosilicon and elemental silicon; filler in cosmetics, pharmaceuticals, paper; insecticides; hydrated and precipitated grades as rubber reinforcing agent, including silicone rubber; anticaking agent in foods; flatting agent in paints; thermal insulator.

(Fused): Ablative material in rocket engines, spacecraft, etc, fibres in reinforced plastics; special camera lenses (amorphous) : Silica gel.

Source : Chinnagudem lake (water flow), Nalgonda district .
Washed and dried sand, crushed and ground in a ball mill at the laboratories of College of Technology, OU, Hyderabad.

Figure 2: Virgin PTFE and Fillers

Filler Tests:

A. Moisture Analysis:

5 grams of given powder in a petri dish was taken and kept for heating at 150°C (ASTM 1547) in a drying oven for two hours. After two hours, it was removed from the oven and subjected to cooling at 23°C for 10 minutes.

The values are as below –

Sl no.	Name of the filler	Laboratory finding percentage of moisture
01.	Glass	0.00686
02.	Graphite	0.02998
03.	Granite	0.46090
04.	Garnet	0.34620
05.	Antimony trisulphide	0.69070
06.	Alumina	0.1639344
07.	Bronze	0.39900
08.	Carbon	0.0999
09.	Marble	0.0299
10.	Porcelian	0.099
11.	Sand	0.04398
12.	Mica	0.2813
13.	Wollastonite	0.520842
14.	China clay	0.94340
15.	PF	0.02990
16.	Activated carbon	11.1600

Moisture absorption of virgin PTFE = 0.0014 %.

All fillers used in this study are heat cleaned (at 120°C for 2 hours) to constant weight before use.

B. Stability Test:

5 grams powder in a petri dish and kept for 45 minutes at 400°C in a drying oven. Then subjected to cooling for 10 minutes at 23°C. Before and after heating the powder weight taken and calculated the percentage of moisture of filler as follows –

Sl no.	Name of the filler	Laboratory finding percentage of moisture
01.	Glass	0.00686
02.	Graphite	0.250
03.	Granite	0.0258
04.	Garnet	0.00134
05.	Antimony trisulphide	0.69070
06.	Alumina	0.3271
07.	Bronze	0.199
08.	Carbon	0.338
09.	Marble	0.0177
10.	Porcelian	0.2428
11.	Sand	4.08
12.	Mica	1.2116
13.	Wollastonite	0.222
14.	China clay	0.9434

Percentage of moisture in virgin PTFE = 0.00165

This test was carried out at a slightly highest temperature (400°C) above 375°C to doubly ensure suitability of the filler to incorporate into PTFE.

C. Specific Gravity:

When the sold is in powder form, a specific gravity bottle was used to determine specific gravity. A quantity of the filler/powder is added to the bottle and the whole weighed, if the weight of the bottle W_1 and the total weight W. The weight of the added powder is given be $W-W_1$. The bottle is then completely filled with a liquid (methanol) of known specific gravity "d", in which the solid is insoluble and the weight is again taken W_2. Let W_3 be the weight of the bottle filled with demineralised water. Then the specific gravity – D of the powder is given by

$$D = \frac{(W-W_1)\,d}{(W_3-W_1)-(W_2-W)} = \frac{\text{Powder weight x d}}{\text{of water – Wt. of liquid}}$$

Machines and Instruments Used:

I. a) Type of the machine : Hydraulic press, vertical, with 4 columns.
b) Clamping force (capacity) : 150 Tons
c) Opening force : 40 Tons
d) Stroke of the clamping : 1250 mm plates.
e) Height of the working surface : 830 mm
f) Name of the manufacturer : Ets. GUIGNOT S.A.R.L. OYONNAX MODEL FRANCE.
g) Mould size : 25 mm height MS Mould, 94mm dia.

II. a) Type of the machine : Free Air Sintering Oven
b) Voltage : 400 V, Phase 50 Hz.
c) Temperature : 400°C maximum.
d) Name of the manufacturer : J. POARMILLEUX, Autochem, France

e) Model : Forced CONVECTION OVEN No.36255

III. Type of machine : Compression testing machine
Model : ZTM-10 (GDR Make)

IV. a) Sample punching die : As per ASTM 1457
b) Vernier callipers : Mitutoyo JAPAN
c) Thickness gauge : Mitutoyo JAPAN
d) Electronic balance : Mettle AE 160 Made in Switzerland
e) Muffle furnace : Kumar Industries, Bombay, India

Testing and Characterization of the Composites:

a. Mould Filling:

PTFE compression moulds are simple in design. In most of the cases, moulds are cylindrical in shape whose internal surface is smooth and free from scratches and other defects. Moulds are normally made of carbon steel; In case of large scale production, hardchrome plated moulds are recommended.

Billet size – 94 mm x 26 mm. A weighed quantity of filled PTFE powder was taken into the clean and dry stainless steel mould and pressed in between two pusher plates. The lumpy material was sieved with 2 mm aperture sieve.

The lower plate inside the mould rests on the lower platen of the 150 tons capacity hydraulic press, while the upper plate must freely slide within the mould cavity. For pressing, the hydraulically operated press is used. It is a vertical hydraulic type having sufficient day light since PTFE powder has a compression ratio of upto 4:1. The closer speed of the press is also critical to avoid air trapping in the mould. Initially the mould is rested over a support. The material was poured into the mould. Then the support (top pusher) is removed and placed on the mould. After placing the top pusher, the powder was pressed/compacted (as per recommended pressure) in the hydraulic press at a speed of 2 mm/sec.

Spacer bar was removed after applying the 100 kg/cm^2 pressure to the mould for 2 minutes. This material was compressed at 500 kg/cm^2 pressure; pressure rise time is 5 minutes and holding for 5 minutes. The pressure was released within 2 minutes.

The pressed PTFE block called perform (green block) is removed slowly from the mould. The perform is brittle. Hence handling of the perform should be done carefully.

Final stage in the moulding process is the ejection of the preformed part from the mould. Preform ejection from the mould was carried out in one smooth continuous stroke at a speed of 3.5 mm/sec.

Calculations:

$$P_1S_1 = P_2S_2$$

$$P_1 = \frac{P_2 S_2}{S_1}$$

P_1 = Recommended pressure as per grade
P_2 = Pressure to be given
S_1 = Moulding area
S_2= Ram area
S_1= D^2

Billet weight = C/A x Density x Height

b. Sintering of the Billet:

The billet which was taken from mould is placed in free air circulation oven for sintering. During this process, the PTFE melts (soften) and coalescesion and adhesion between PTFE particles and filled particles takes place which makes the billet hard and rigid. After cooling to room temperature the billet was taken out for doing various tests

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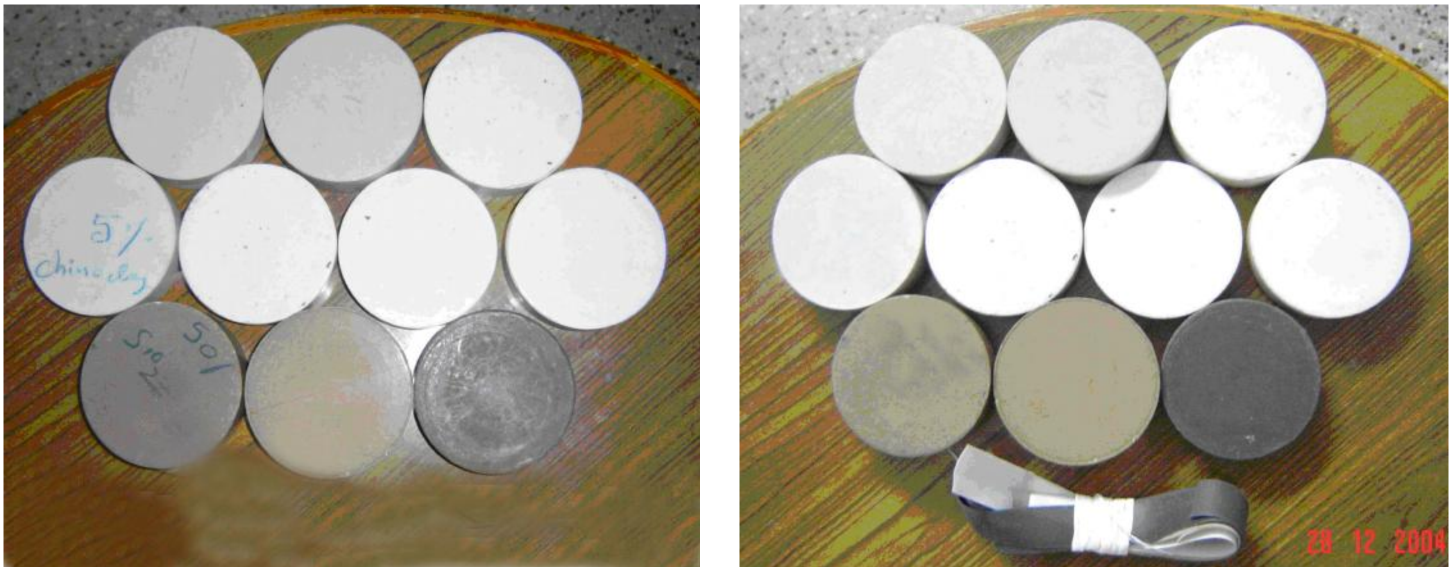

Figure 3 Virgin PTFE and its filled composite billets (before and after sintering including skiving tapes)

c. Skiving of the Billets:

Skiving of the billet was done on the skiving machine. For this purpose, special arrangement was done. This arrangement consists of a low rpm motor with spring loaded drum; on to which tapes will be wound during skiving operation. For the experiment purpose 0.5mm tape thickness was taken and used for tensile and elongation tests, for dielectric strength, 0.1mm thickness tape was made.

Test Procedure of Machines and Calculations:

a. Bulk density:

About 400gms each of virgin PTFE, filler and filled PTFE powders are separately sieved in 1.6mm sieved to eliminate cakes. The sieved material is poured into the volumetric cup placed on stand from a height of 10cm. The cup is filled with this material in such a manner that is forms a mound and overflows. The powder allowed settling for 15 seconds and then the cup and its stand is gently pushed to avoiding agitation while leveling. The same procedure was followed thrice and the average value is calculated.

Calculation of bulk density:

Bulk density (gm/ltr) = weight of the material (powder x 4)

Weight of the material (W) = W_2-W_1

Where W_1 = Weight of the empty cup (250ml)

W_2 = Weight of the material (powder) + weight of the cup

b Specific Gravity:

For this test, standard size (94 x 25 mm) billet of virgin PTFE and its composites (with filler) namely, glass, granite, garnet, graphite, antimony trisulphide, alumina, carbon, marble, mica, porcelian, sand, bronze and wollastonite (with various percentages for fillers) were moulded and sintered. These billets are then weighed in open air with the help of simple balance and weights were noted. Weights of these billets in water are also taken keeping billets hanging in water without touching the side and bottom of vessel containing water.

Calculation:

$$\text{Specific gravity of billet} = \frac{\text{Weight of billet in air}}{\text{Weight of billet in water}}$$

c. Diametrical Shrinkage and Expansion or Contraction in Height.

This test is carried out by taking the measurement of (various filled grade materials (glass, alumina, graphite and tixolex-25) with different percentage of filler) billets before sintering and after sintering. The difference in mm is tabulated.

Dimensions measured dia and height:

$$\% \text{ diametrical shrinkage} = \frac{\text{Dia of before sintering} - \text{Dia of after sintering}}{\text{Dia of before sintering}} \times 100$$

$$\% \text{ expansion/ contaction in height} = \frac{\text{Height after sintering- height before sintering}}{\text{Height before sintering}} \times 100$$

Results and Discussion:

Effect of filler and its content on bulk density:

The variation of Bulk density with respect to filler content is shown in table 1 and the plot of bulk density on filler and content is shown in figure 4 & 5. Bulk density was measured for both PTFE powder, filler respectively on filled PTF composites powder with different filler content.

In case of glass, granite, garnet,alumina, antimony trisulphide, marble, sand, bronze, wollastonite and porcelian filled PTFE powders, there was an increase in bulk density with increase in filler content from 5% to 50%, where as in case of graphite, carbon, mica, china clay and tixolex-25 filled PTFE powders the bulk density decreased with increase in filler content from 5% to 50%.

It was found that bulk density value of virgin PTFE powder is low compared to pure fillers except graphite, china clay and tixolex-25 powders. Bulk density values of graphite, china clay and tixolex-25 were 382 gm / cc, 229.6 gm/cc and 320 gm/cc respectively.

In case of all filled composite powders, bulk density value was found to be higher than pure PTFE powder. The highest value (918.0 gm/cc) was found in case of 50% antimony trisulphide filled PTFE. Bulk density value was found in increasing order with increase in filler content except in case of graphite, mica, carbon, tixolex-25 and china clay filled PTFE composite powders.

In case of all 14 fillers studied the highest bulk density value (4065.2 gm/cc) was obtained for bronze filler and the lowest one is china clay 229.6 gm/cc. Increase in bulk density with increase in filler content may be due to the fact that the pure fillers bulk density values are higher than that of virgin PTFE powders. A decrease in bulk density with increase in filler content was observed in some cases like carbon, graphite, etc, due to their low bulk density.

Sl.No.	% of filler content	Bulk density(gm/cc) of						
		glass (f)	granite	graphite	garnet	alumina	antimony trisulphide	carbon
01.	5	488.40	502.50	425.20	509.35	503.60	517.20	465.20
02.	10	507.60	538.80	421.60	510.40	515.60	533.60	457.20
03.	15	518.50	560.40	387.20	527.20	535.60	579.20	431.20
04.	20	528.00	570.20	386.00	557.20	542.80	585.20	413.20
05.	25	531.60	575.00	356.00	581.20	586.80	679.20	405.60
06.	30	538.00	600.00	346.80	602.40	634.40	687.60	404.40
07.	40	572.40	640.40	343.20	649.60	666.80	740.80	404.00
08.	50	701.60	676.80	340.00	661.20	735.20	918.00	400.00
***		1210.00	1137.20	382.00	1101.20	1289.20	2872.40	***

*** VIRGIN POWDER VALUES

Sl.No.	% of filler content	Bulk density(gm/cc) of							
		marble	mica	sand	bronze	wollastonite	porcelian	Tixolex-25	
01	5	458.00	453.60	512.40	492.00	461.20	450.80	475.00	600.00
02	10	465.60	444.00	528.00	495.60	477.20	462.80	496.00	590.00
03	15	477.20	440.00	528.80	531.60	476.80	468.80	497.00	420.00
04	20	482.40	434.80	552.00	532.40	478.00	482.40	490.00	315.00
05	25	507.60	427.20	554.80	572.00	489.20	482.40	492.00	240.00
06	30	512.40	418.40	558.40	644.00	492.80	506.00	485.00	200.00
07	40	567.20	408.00	570.00	716.40	497.20	523.00	480.00	180.00
08	50	595.60	400.00	614.40	821.20	503.20	566.40	280.00	150.00
***	****	1173.6	358.8	1025.2	4065.2	612.0	963.2	229.6	320.0

*** Virgin powder values

Table1: Bulk density values for filled PTFE composites on different filler content

Virgin PTFE bulk density = 432 gm/cc

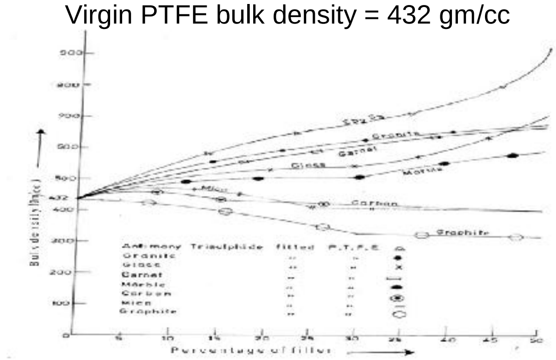

Figure 4: Bulk density of filled PTFE percentage of fillers

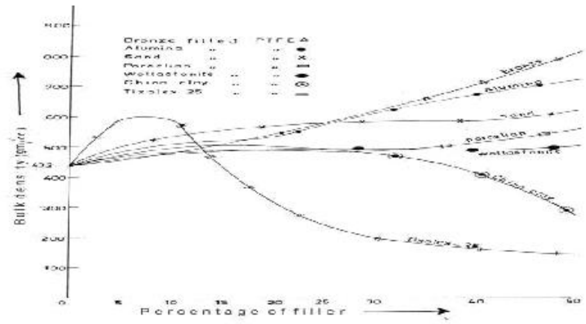

Figure 5: Bulk density of filled PTFE percentage of fillers

Effect of filler and its content on specific gravity:

The variation of Specific gravity with respect to filler content is shown in Tables 2 and 3 and the plots of specific gravity versus filler and filler content are shown in Figures 6, 7, 8 and 9, 10 before and after sintering of virgin PTFE and filled PTFE billets.

n case of glass filled PTFE billet (before and after sintering), there is an increase in specific gravity with increase in filler content ; after reaching certain percentage of filler content, the specific gravity found to be decreasing (before and after sintering). Specific gravity of glass filled PTFE billet (before and after sintering) is in decreasing with filler content from 25-50%.

With alumina filled PTFE, an increase in specific gravity is observed with filler content up to 15%. Thereafter, specific gravity is found decreasing with increase in filler content.

Specific gravity is found decreasing with increase in filler content in case of graphite filled PTFE.

The case is similar in case of tixolex-25 filled PTFE (like graphite and alumina filled PTFE). However, decrease in specific gravity with increase in filler content is drastic compared to other filled PTFE like glass, granite, graphite, garnet, antimony trisulphide, sand, wollaostonite, etc.

In case of granite filled PTFE, the specific gravity is increasing with increase in filler content up to 25%. After that it has shown a decreasing trend with increase in filler content before sintering. Whereas the specific gravity is in increasing trend with increase in filler content up to 40% after sintering.

In case of garnet filled PTFE, the specific gravity increases with increasing filler content before and after sintering upto 30%. After that in both cases it has in decreasing trend.

In case of porcelian filled PTFE, the specific gravity is in increasing manner with increase in filler content upto 20% before sintering. Whereas, increase manner is extended upto 30% after sintering.

The trend is similar, like other fillers, in case of marble filled PTFE. The specific gravity is observed to be increasing before and after sintering with increase in filler content.

The specific gravity is in decreasing trend from 5 to 50% filler content for carbon filled PTFE before and after intering.
In mica filled PTFE the specific gravity decreases with increasing filler content before and after sintering.

In case of antimony trisulphide filled PTFE, the specific gravity increases with increasing filler content before and after sintering from 5 to 50%.

Also the specific gravity is observed in increasing manner in case of bronze filled PTFE as percentage of filler content increases.

In case of sand filled PTFE, the specific gravity increases with increase in filler content upto certain level after that it shows in decreasing manner.

Among the 14 filled PTFE composites studied, highest specific gravity was found in case of bronze filled PTFE containing 50% filler content (3.396) before sintering and (3.465) after sintering. In case of glass, graphite, carbon, mica, china clay, and tixolex-25 filled PTFE, the uniform decrease in specific gravity was found with increase in filler content from 5 to 50%. Whereas in case of garnet, wollastonite and porcelian filled PTFE, the uniform increase in specific gravity was found upto certain percentage with increase in filler content and after that it was found decreasing.

In case of antimony trisulphide, bronze, marble, porcelian and sand filled PTFE uniform increase in specific gravity was found with increase in filler content from 5-50%. This may be due to the fact that antimony trisulphide, bronze, marble, and sand fillers in PTFE resin matrix are behaving mostly as reinforcing fillers. Whereas in case of glass, graphite, garnet, carbon, mica, china clay, tixolex – 25 and wollastonite filled PTFE, the fall in specific gravity with increase in filler content may be due to the fact that these fillers are not behaving as reinforcing fillers. These fillers may be lying embedded in the resin (PTFE) matrix as separate entity providing voids/cracks and similar other defects resulting in low specific gravity value with increasing filler content.

Sl.No.	% of filler content	Specific gravity of						
		glass (f)	granite	graphite	garnet	alumina	antimony trisulphide	carbon
01.	5	2.217	2.199	2.191	2.222	2.222	2.265	2.202
02	10	2.216	2.237	2.150	2.240	2.261	2.313	2.177
03	15	2.214	2.259	2.161	2.250	2.406	2.375	2.143
04	20	2.203	2.263	2.139	2.254	2.347	2.557	2.084
05	25	2.189	2.278	2.106	2.263	2.328	2.616	2.066
06	30	2.170	2.268	2.079	2.161	2.316	2.729	2.013
07	40	2.114	2.262	2.005	2.168	2.395	2.933	1.881
08	50	2.100	2.227	1.878	2.220	2.375	3.150	1.737

Sl.No.	% of filler content	Specific gravity of							
		marble	mica	sand	bronze	wollastonite	porcelian	Tixolex-25	
01	5	2.233	2.176	2.220	2.249	2.245	2.214	2.184	2.145
02	10	2.247	2.181	2.232	2.339	2.330	2.232	2.130	2.045
03	15	2.262	2.171	2.246	2.431	2.430	2.232	1.979	1.928
04	20	2.292	2.133	2.247	2.536	2.530	2.236	2.020	1.787
05	25	2.309	2.129	2.248	2.660	2.018	2.223	2.020	1.657
06	30	2.329	2.060	2.249	2.769	2.108	2.152	-	-
07	40	2.355	2.006	2.250	3.040	2.167	2.159	-	-
08	50	2.365	-	2.165	3.396	2.055	2.204	-	-

Table 2: Specific gravity of glass (f), granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, china clay, porcelian, sand, bronze and wollastonite filled PTFE for various filler content. (before sintering)

Specific gravity of virgin PTFE(before sintering):2.218

Sl.No.	% of filler content	Specific gravity of						
		glass (f)	granite	graphite	garnet	alumina	antimony trisulphide	carbon
01.	5	2.192	2.209	2.171	2.222	2.208	2.233	2.172
02	10	2.206	2.241	2.160	2.236	2.272	2.308	2.163
03	15	2.200	2.304	2.150	2.237	2.305	2.391	2.127
04	20	2.203	2.273	2.140	2.250	2.343	2.489	2.092
05	25	2.202	2.314	2.100	2.263	2.363	2.567	2.079
06	30	2.154	2.346	2.000	2.282	2.368	2.690	2.019
07	40	2.107	2.374	1.900	2.246	2.468	2.869	1.884
08	50	2.000	2.288	1.400	2.075	2.392	3.926	1.725

Sl no.	% of filler cont.	Marble	Mica	Sand	Bronze	Wolla Stonite	Porcelian	China clay	Tixolex-25
01	5	2.193	2.187	2.212	2.281	2.215	2.169	2.193	2.100
02	10	2.224	2.199	2.226	2.368	2.218	2.216	2.066	1.950
03	15	2.247	2.168	2.243	2.438	2.223	2.238	2.054	1.500
04	20	2.248	2.150	2.241	2.567	2.231	2.358	1.964	2.200
05	25	2.254	2.120	2.248	2.698	2.182	2.258	-	1.000
06	30	2.262	1.927	2.248	2.795	2.149	2.259	-	-
07	40	2.339	1.825	2.264	3.090	2.100	2.234	-	-
08	50	2.367	1.694	2.249	3.465	1.895	2.214	-	-

Table 3: Specific gravity of glass (f), granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, china clay, porcelian, sand, bronze and wollastonite filled PTFE for various filler content.(after sintering)
Specific gravity of virgin PTFE(after sintering):2.189

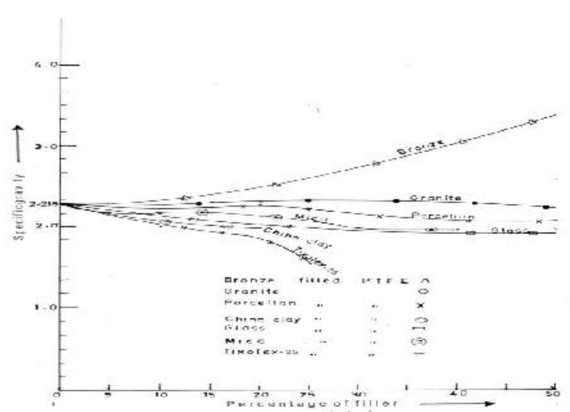

Figure 6: Specific gravity (before sintering) of filled PTFE composites
on the percentage of fillers

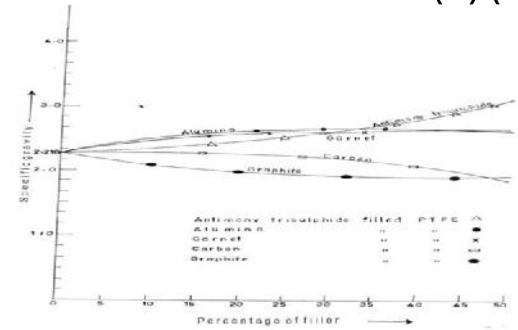

Figure 7: Specific gravity (before sintering) of filled PTFE composites on the percentage of fillers

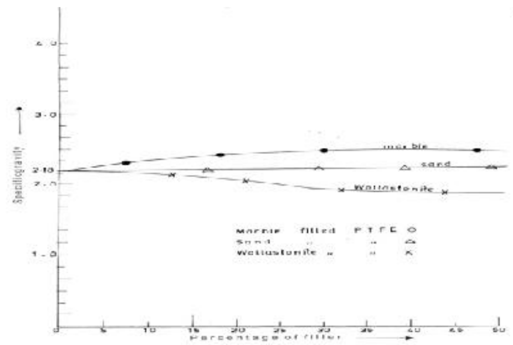

Figure 8: Specific gravity (before sintering)of filled PTFE composites on the percentage of fillers

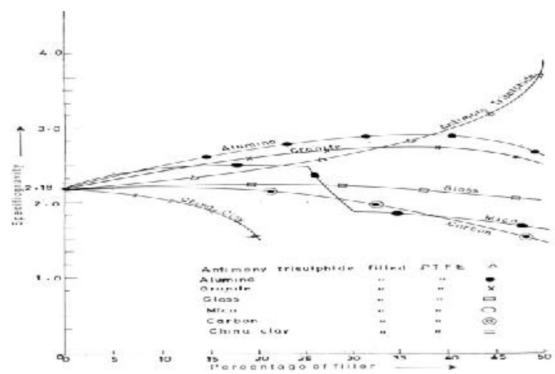

Figure 9: Specific gravity (after sintering) of filled PTFE composites on the percentage of fillers

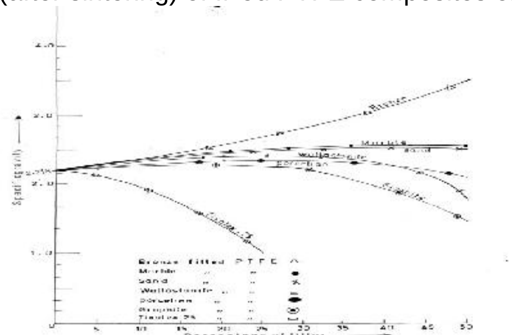

Figure 10: Specific gravity (after sintering) of filled PTFE composites on the percentage of fillers

3.3.Effect of filler and its content on expansion or contraction in height and in diametrical shrinkage:

Figure 11,12 shows the percentage, expansion / contraction in height versus filler and its content and figure 13,14 shows the percentage shrinkage in dia. table 4 shows the effect of filler and its content on percentage expansion / contraction in height; and table 5 shows the diametrical shrinkage.

It was noticed that unfilled PTFE after sintering gets increased in height (% expansion) to the extent of 7.4 in the direction of pressure applied during compaction; shrinkage in dia was found to be in the perpendicular direction.

It is observed that percentage expansion in height in case of glass, granite, graphite, garnet, antimony trisulphide, alumina, carbon, marble, sand, bronze, wollastonite, porcelian and tixolex-25 filled PTFE has reached maximum value in case of 5% filler content, and subsequently decreased with increasing filler content. Whereas in case of mica filled PTFE has reached maximum value at 50% filler content. In these filled PTFE the expansion in height is increasing from 5-50% of filler content.

In case of glass, graphite, antimony trisulphide, carbon, marble, sand, bronze, wollastonite, porcelian and tixolex-25 filled PTFE contraction in height is observed as filler content increases from 5 to 50% filler content. All values of percentage expansion in height in case of bronze filled PTFE were found to be lower than mica filled PTFE irrespective of filler content. The maximum value of percentage expansion in height was found to be 14.23 in case of 50% mica filled PTFE and the contraction value of the same was found to be (-31.818) in case of 40% bronze filled PTFE.

In case of glass, graphite, carbon, marble, mica, sand, bronze, wollastonite and porcelian filled PTFE, the percentage shrinkage in dia was found to be falling with increase in individual filler content, whereas in case of granite, garnet, alumina, and antimony trisulphide filled PTFE there is an increase in percentage diametrical shrinkage with increase in filler content from 5-50%. But in case of tixolex-25 filled PTFE there is an initial increase in percentage of diametrical shrinkage value from that of unfilled PTFE and after attaining peak (at 5% filler content in case of tixolex-25 filled PTFE only), there is fall in percentage diametrical shrinkage values. In both the cases with increase in filler content; the fall in diametrical shrinkage is sharp than other filled PTFE composites.

In case of glass, graphite, antimony trisulphide, carbon, marble, sand, bronze, wollastonite and porcelian filled PTFE fall in percentage increase in height as well as in percentage diametrical shrinkage with increasing filler content may be due to the fact that in both the cases the residual stress stored in the billet resulted in both the fall in percentage increase in height and diametrical shrinkage of billets during sintering. It is to be noted that all the fillers studied (without any PTFE content in it) are not showing any stress relaxation effect like PTFE at the sintering temperature. The pressure given to billets during compaction, in all the cases studied, may be partially stored in the billets depending upon the quantity and the nature of fillers used, as residual stress which gets released during sintering resulting increase in height and proportionate decrease in dia of billets.

Sl. no.	% of filler content	Expansion / contraction of						
		Glass(f)	Granite	Graphite	Garnet	Alumina	Antimony trisulphide	Carbon
01.	5	4.845	7.694	7.843	1.294	12.340	8.653	8.037
02	10	4.132	7.480	7.031	1.664	10.647	7.868	6.784
03	15	3.843	7.208	6.500	3.349	7.666	7.729	5.914
04	20	3.656	6.180	6.000	1.159	7.130	7.151	5.770
05	25	2.810	5.200	5.660	1.165	7.112	5.686	4.925
06	30	2.766	4.314	5.342	1.542	6.992	4.800	4.827
07	40	2.725	2.090	5.000	1.519	3.638	3.200	4.085
08	50	2.208	0.000	4.200	0.901	1.938	2.060	3.724

Sl no.	% of filler content	Marble	Mica	Sand	Bronze	Wolla-stonite	Porcelian	Tixo –lex-25
01	5	8.538	4.800	10.123	4.700	4.386	7.664	1.736
02	10	7.775	6.425	9.979	4.854	5.523	7.287	0.000
03	15	7.500	9.166	9.090	5.721	6.198	6.827	-2.105
04	20	7.368	6.746	8.263	10.360	6.872	5.352	-3.160
05	25	7.260	7.450	7.468	-1.061	7.550	5.100	-4.065
06	30	7.323	9.960	6.694	-11.874	7.920	4.800	-
07	40	7.054	10.714	5.208	-22.220	6.923	3.200	-
08	50	4.042	14.230	4.440	-31.818	4.340	2.100	-

Table 5: Expansion / contraction in height of glass (f), granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, china clay, porcelian, sand, bronze and wollastonite filled PTFE for various filler content.
Billet dia 94 mm, Height 25 mm
% Expansion / contraction in height of virgin PTFE = 7.4

Sl.No.	% of filler content	Expansion / Contraction of						
		glass (f)	granite	graphite	garnet	alumina	antimony trisulphide	carbon
01	5	2.6510	2.294	2.823	0.172	2.280	2.438	3.172
02	10	2.5172	2.771	2.588	1.635	2.994	2.829	3.115
03	15	2.2000	2.877	2.352	1.771	3.410	3.836	3.087
04	20	2.1650	2.940	2.000	2.452	3.506	3.729	3.019
05	25	1.7420	3.400	1.882	3.729	3.508	3.787	2.668
06	30	1.5000	3.505	1.058	3.904	3.527	3.682	2.651
07	40	1.0000	3.600	1.000	5.905	3.540	3.297	2.060
08	50	0.7320	3.610	0.500	6.150	3.617	2.486	1.425

Sl.No.	% of filler content	Expansion / Contraction of						
		marble	mica	sand	bronze	wollastonite	porcelian	Tixolex-25
01	5	3.058	3.617	3.674	4.055	3.723	3.493	5.882
02	10	2.949	3.617	3.117	3.940	3.404	3.248	5.402
03	15	2.817	3.506	3.069	3.361	3.297	3.230	5.280
04	20	2.776	3.506	3.056	3.915	3.191	3.258	4.865
05	25	2.606	3.446	2.895	3.829	2.787	3.180	4.337
06	30	2.468	1.380	2.542	3.680	2.148	3.100	-
07	40	2.349	0.924	2.448	3.610	1.595	3.000	-
08	50	2.165	0.214	1.817	3.438	0.808	2.500	-

Table 6: Diametrical shrinkage of glass (f), granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, china clay, porcelian, sand, bronze and wollastonite filled PTFE for various filler content.

Billet diameter: 94mm

Height : 25mm

Diametrical shrinkage of origin PTFE : 3.176

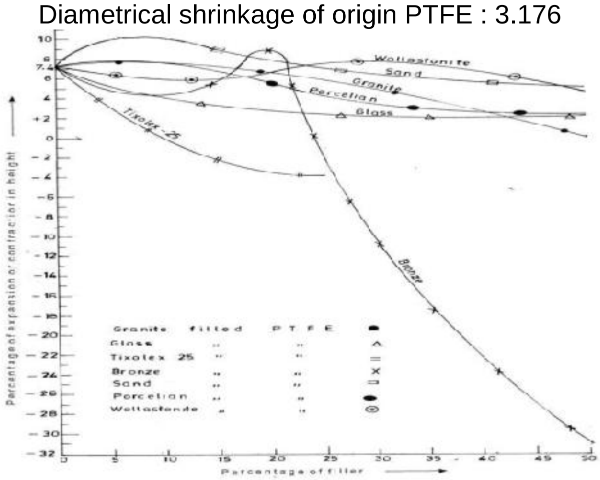

Figure 11: Expansion / contraction in height of filled PTFE composites vs percentage of filler

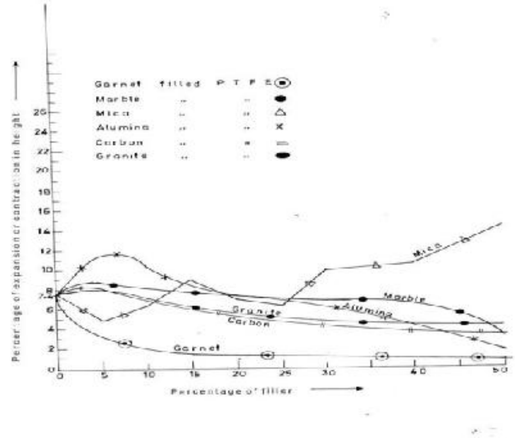

Figure 12: Expansion / contraction in height of filled PTFE composites vs percentage of filler.

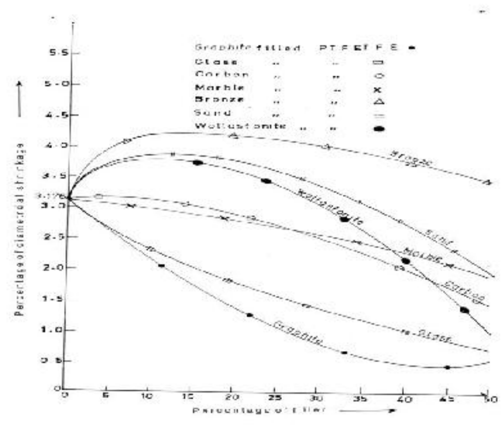

Figure 13: Diametrical shrinkage of filled PTFE composites on the percentage of fillers

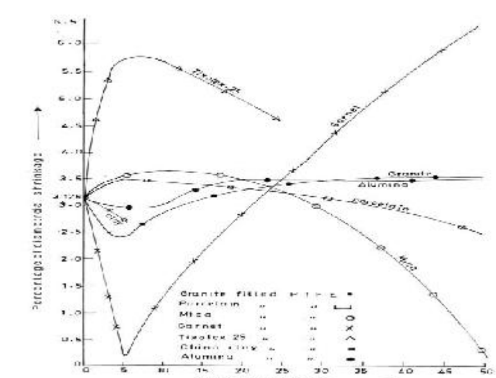

Figure 14: Diametrical shrinkage of filled PTFE composites on the percentage of fillers

Conclusion:

In case of all 14 fillers studied, the highest bulk density value (4065.2 gm/cc) was obtained for bronze powder and lowest value was found in case of china clay (229.6 gm/cc) powder.

It was found that the bulk density of virgin PTFE powder is low compared to all pure fillers except graphite, china clay and tixolex-25 powders (382 gm/cc, 229.6 gm/cc and 320 gm/cc).

Out of all filled PTFE composites studied, the highest specific gravity was found in case of bronze filled PTFE containing 50% filler content before (3.396) and after (3.465) sintering.

It was found, highest expansion value (14.23) in longitudinal direction was found for mica filled PTFE out of all filled PTFE composites (50%).

A highest value of 5.882 diametrical shrinkage was obtained for tixolex-25 filled PTFE out of all composites studied (50%).

Further work on wear properties, thermal properties and chemical resistance properties on each filled grade PTFE may be done keeping in view of the specific applications.

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