*Available online at*www.scholarsresearchlibrary.com

Scholars Research Library

Archives of Applied Science Research, 2015, 7 (7):48-58
(http://scholarsresearchlibrary.com/archive.html)

ISSN 0975-508X
CODEN (USA) AASRC9

Effect of fillers on mechanical properties of PTFE based composites

Gogu Venkateswarlu[1], Ravirala Sharada[2] and Mamidi Bhagvanth Rao[3]

[1]*Bharat Heavy Electricals Limited, R. C. Puram, Hyderabad, Telangana*
[2]*TSWRS Junior College (Girls), Mallapur, Dist. Karimnagar, Telangana*
[3]*University College of Technology, Osmania University, Hyderabad, Telangana*

ABSTRACT

In the present work, a systematic study on the effect of different fillers namely; glass, granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, sand, bronze, wollastonite, porcelain, china clay and tixolex – 25 on mechanical properties like hardness, tensile strength and % elongation of virgin PTFE and different filled PTFE composites (filler content 5 – 50%) were made in order to achieve maximum properties. Results indicated that, highest hardness value based composites were found to be 15,20 and 25% in case of garnet filled PTFE, 50% in case of marble filled PTFE and 40% in porcelain filled PTFE, 5% bronze filled PTFE shows highest tensile strength and % elongation values.

Key words: PTFE, fillers, hardness, tensile strength, % elongation.

INTRODUCTION

Polytetrafluoro ethylene (PTFE) is a crystalline polymer. It has excellent properties like chemical inertness, low co – efficient of friction, non –toxic, non – flammable, negligible water absorption, non – adhesive, anti – stick, high thermal stability, low dielectric constant, moderate mechanical properties that make it suitable for many applications (Brydson et. Al., 1989). The influence of fillers on physical and mechanical properties of composites evaluated by Jing Cai et. Al., (2007).

Mechanical properties of PTFE are: They are used in stuffing boxes on rotating or alternating shafts or pumps, compressors, auto-claves, valves etc. Packing in pure PTFE or in other materials impregnated.

- In the printing and textile trade, for coating of rollers to make the surfaces antistic.
- The electrical insulation properties of PTFE and their small variation are used over a wide range of temperature and humidity, and also in corrosion environments and this makes the material preferable in electrical and electronic industries.
- PTFE is generally used more and more for the purposes of aviation, electronic computers, radar, etc.
- PTFE is also very generally used in making connection components co-axial plugs, supports for electronic tubes and aerials, cross bars for high tension conductors, terminal plates insulation components, etc.
- For all this uses, pure or ceramic (glass, porcelain, alumina, etc) filled PTFE can be used. Di-electric properties of PTFE can be used for designing and manufacturing condensers using either skived tapes or films made from dispersion.

- Metallurgy, ceramics and glass industry have created many materials which in the majority of cases and if carefully chosen can solve the practical problems in the design of equipment intended for handling chemical products.
- Maintaining of tight joints between the various units making up a chemical assembly is a pressing problem and the transmission of movements inside a completely sealed enclosure seals and joints are now one of the most popular fields of application of PTFE in the chemical industry. Apart from this use, PTFE is equally effective for pipelines, valves, pumps etc.
- Static and dynamic seals can be made from,
- Flat static seals made from sheet PTFE and used for normal flat flanges or flange with single or double groove
- Seals of special section which fit into a particular joint profile.
- Composite seals compressing a core of klingerite or other elastomer covered with PTFE
- PTFE is widely used in valve gear either in association with other materials (fillers) or occasionally as the soul material. For example, the valve seats, flaps bellows seals, packing rings, diaphragm seals etc of many types of metallic (Bronze, Antimony trisulphide) or glass valves are made in PTFE. These components permit the optimum use of the properties of main unit.

Though PTFE (Virgin) has excellent properties as mentioned, but it suffers from some draw backs like; low wear resistance, low thermal conductivity, low load bearing capacity, high elongation and low resistance to compressive deformation. Hence, different fillers are incorporated into PTFE to improve those properties. The choice and quantity of fillers to be incorporated into PTFE depends upon specific end applications.

In the present work, an attempt is made to study the effect of filler and filler content on mechanical properties like hardness, tensile strength and % elongation.

MATERIALS AND METHODS

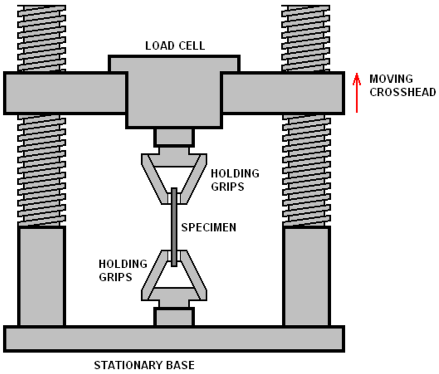
2.0. Experimental methods / Procedure:
The study is on PTFE used in the filled grades HIFLON 71 grade. Fillers used are: glass, granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, sand, bronze, wollastonite, porcelain, china clay and tixolex – 25.

Photograph of virgin PTFE and its filled composite billets after sintering and skived tape

2.1. Test procedure of machines and calculations:
a. Hardness Tester:
Instrument: Durometer hardness tester
Procedure: Both virgin PTFE and its filled composites billets were sintered for 14hours cycle at 375^{o}C and machined to desired surface smoothness for checking durometer hardness and other surface properties. Bottom of the durometer was pressed on the flat surface of the samples so as to have proper contact with the surface of samples. Durometer readings were noted after 30seconds. 10 readings were noted for each sample and average of the readings was taken for each sample and reported in this study.

b. Tensile tester:
The machine is for determining the tensile strength and elongation of various fibrous and generic materials like textiles, rubber, plastic, leather, cardboard, plywood, paper, asbestos, cable, conductors etc.

Description of the machine: The machine consists of base and the vertical column, which supports the load measuring unit. The base houses the drive unit. The drive is affected by electric motor whose stoke is transmitted through the set of pulleys to load the screw. When pull is applied to specimen, the pendulum gets reflected from its vertical position in proportion to pull applied and tensile force is indicated on the dial gauge pointer.



Photograph of Tensile tester

Determination of tensile strength: For the tensile strength and percentage elongation, the specimens were cut according to ASTM-D1457. All filled PTFE billets were skived to same thickness 0.5mm. For tensile strength, the tapes are conditioned at 23^{o}C± 0.2^{o}Cfor atleast 12hours. After conditioning, the tapes are tested for tensile strength and percentage elongation. The samples are held in between grips of the tensile tester to align in the axis of the specimen and grips with imaginary line joining the paths of the attachment of the grips to the machine. The test piece was filled in the clamps of tensile tester while setting the gap to exactly 22.2mm. Tensile tests were carried out for 3 pieces at a speed of 50mm/min until it breaks.

Calculations: For calculating tensile strength (kg/cm^2), divide the observed breaking load in kg by cross-sectional area of the specimen in cm^2, i.e, tensile strength kg/cm^2 = breaking load in kg/ cross-sectional area incm^2.

$$\text{Tensile strength at break} = \frac{\text{load at break (kgs)}}{\text{Area of cross-section}}$$

c. Determination of % elongation:
The extension caused in test specimen during the tensile tests is determined by measuring the change in the distance between gripping devices. For this purpose, extension scale is attached to the bottom grip and pointer is provided on the upper gripping device. After fixing specimen, note the reading of the pointer provided on the upper gripping device. Let this reading be noted by $R_{1.}$ Now note the pointer reading at the time of rupture of the specimen. Denote this by $R_{2.}$ The difference between R_2 and R_1 is extension. The extension scale is graduated in mm.

Calculations: The percentage elongation is obtained by the following formula,

$$\text{Percentage elongation} = \frac{\text{change in length } (R_2 - R_1) \times 100}{\text{Original length } (R_1)\ (22.2\text{mm})}$$

RESULTS AND DISCUSSION

1.0. Effect of filler and its content on hardness:
Hardness of PTFE has increased with addition of filler and its content as shown in fig.1 (a,b,c) and the values are given in table1. In case of antimony trisulphide, sand, granite, porcelian filled PTFE, the hardness increased with increasing filler content. Whereas, in case of glass, graphite garnet, alumina and bronze filled PTFE, the hardness

increased with increasing filler content upto25% and with further increase in filler content, the hardness decreased. In respect of mica and wollastonite filled PTFE, the hardness increased with increasing filler content upto 15% and with further increase in filler content the fall in hardness values was sharp in case of mica filled PTFE. Whereas, in case of marble filled PTFE, the hardness increased upto 15% and decreased upto 30% filled PTFE and thereafter, again it increased with increased filler content. With china clay and tixolex-25 filled PTFE, the hardness decreased with increasing filler content drastically with increase in filler content. In case of carbon filled PTFE, the hardness increased upto 30% filled PTFE and with further increase in filler content the hardness decreased. The increase in hardness (shore-D) values with increasing filler content in case of antimony trisulphide, sand, graphite, porcelian, mica, alumina, bronze, graphite, carbon and alumina filled PTFE might be due to the fact that these fillers were inert and mightnot be reacting with liberated HF from PTFE at the sintering temperature (375^{o}C). As a result, the hardness values were getting increased with increasing filler content in the resin matrix for alumina, granite, bronze, porcelain, graphite, and carbon and antimony trisulphide. In case of china clay, and tixolex-25 filled PTFE, the drastic fall in hardness values might be due to the fact that, china clay and tixolex-25 reacted with liberated HF and other fluorocarbons from PTFE at sintering temperature (375^{0}C) resulting in drastic fall in hardness values. The highest hardness value was 75 shore- D for garnet at 15%, porcelain at 40% respectively. The lowest value was 35 shore- D for mica filled PTFE at 50%.

Table: 1- Hardness (shore-D) of glass (f), granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, china clay, porcelian, sand, bronze and wollastonite filled PTFE forvarious filler content

Sl. No.	% of filler content	Hardness (Shore-D) of						
		Glass(f) filled PTFE	Granite filled PTFE	Graphite filled PTFE	Garnet filled PTFE	Alumina filled PTFE	AntimonyTrisulphide filled PTFE	Carbon filled PTFE
01.	5	64	68	65	70	64	62	63
02	10	66	70	66	74	65	65	66
03	15	67	71	68	75	66	67	67
04	20	67	71	69	75	69	67	68
05	25	68	72	69	75	69	68	69
06	30	62	72	66	72	68	69	70
07	40	62	74	63	70	65	70	66
08	50	54	75	42	64	65	72	62

Sl No.	% of filler content	Marble filled PTFE	Mica filled PTFE	Sand filled PTFE	Bronze filled PTFE	Wollastonite filled PTFE	Porcelain filled PTFE	ChinaClay filled PTFE	Tixolex -25 filled PTFE
01	5	65	60	63	60	65	65	60	66
02	10	66	64	65	61	67	67	56	66
03	15	68	67	67	61	68	68	55	64
04	20	64	60	69	62	68	69	54	63
05	25	65	60	69	65	66	72	-	62
06	30	66	50	69	64	65	73	-	-
07	40	72	45	70	63	61	75	-	-
08	50	75	35	73	62	60	73	-	-

Hardness of virgin PTFE(shore-D) = 63

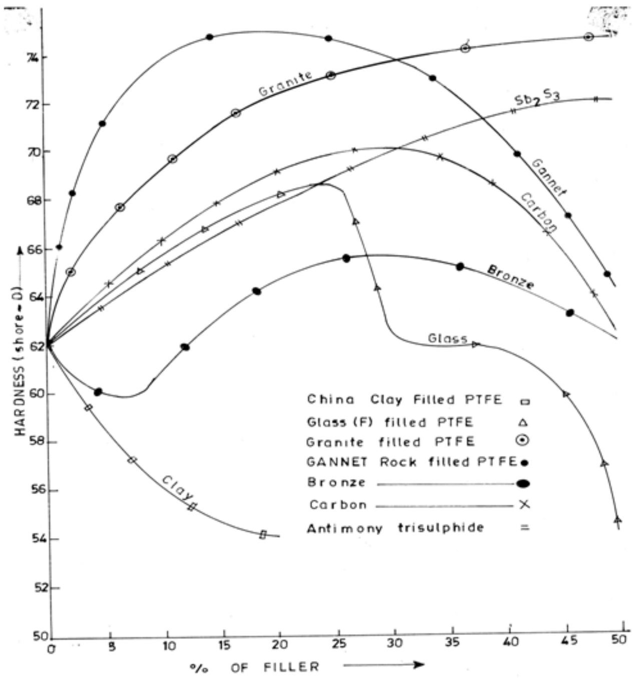


Fig. 1(a) Hardness (shore-D) of Filled PTFE composites vs Percentage of Fillers

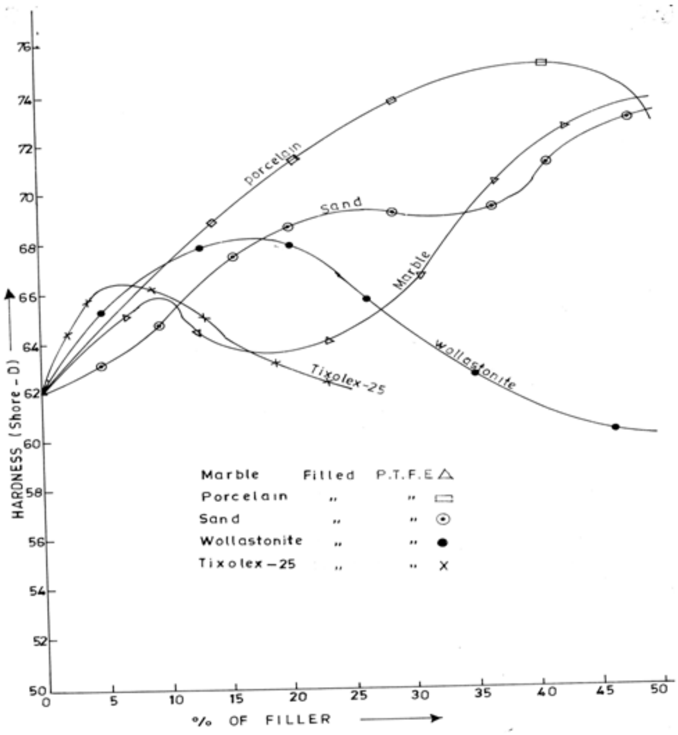


Fig. 1 (b) Hardness (shore-d) of filled PTFE composites vs percentag0e of fillers

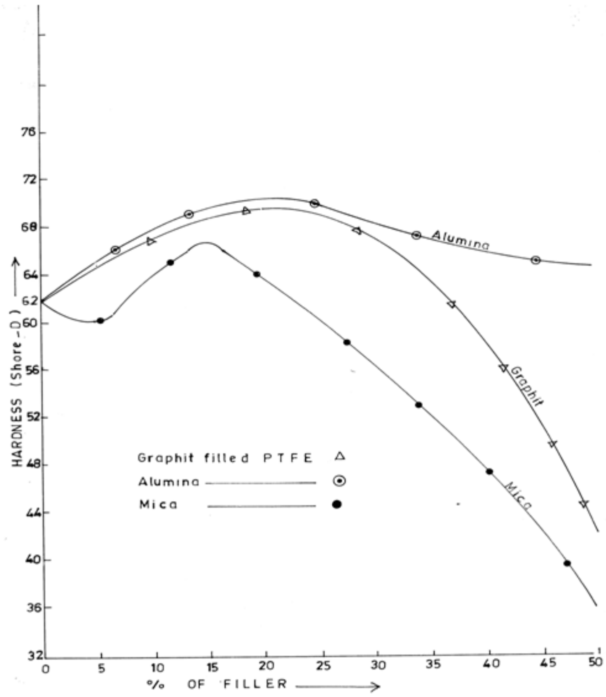


Fig. 1 (c) Hardness (shore-D) of Filled PTFE composites vs Percentage of fillers

3.2 Effect of filler and its content on tensile strength:

The variation (decrease) of tensile strength with increasing filler contents in all the cases studied is shown in figure 2 (a, b) and the results are tabulated in table 2. In all the cases, tensile strength (kg/cm^2) decreased with increasing filler content. In case of mica, carbon, garnet and wollastonite filled PTFE, a roughness was observed for skived tape and it was increasing with increasing filler content for 5 to 15, 25, 30 % filled PTFE. Whereas. For 15% onwards, mica 25% onwards, wollastonite and carbon 30% onwards, sand and garnet also 30% onwards, the tape was very rough and strength was weak. However, a very sharp and drastic decrease in tensile strength was observed in case of tixolex-25, mica and china clay filled PTFE. The percentage decreased in tensile strength with respect to filler content is given in table 2(a). for the same filler content, from left to right, the percentage reduction in tensile strength increased. For the same filler content, in case of tixolex-25, china clay and mica filled PTFE the reduction in tensile strength was very drastic compared to other filled grades studied. In case of glass filled PTFE, percentage reduction in tensile strength was found to be the lowest compared to other filled grades. As percentage of filler increase, same trend in reduction in tensile strength was observed. All ceramic fillers lower the tensile strength of original polymer base in which they were sintered. The same observation is made in this case where the fillers are glass, alumina, graphite, granite, carbon, marble, antimony trisulphide, sand, mica, porecelian, wollastonite, garnet and tixolex-25 added to PTFE. Lowering of tensile strength values with increase in filler content in all the cases might be due to the fact that fillers were just lying embedded in the resin matrix without any chemical bonding resulting defects in skived tapes. In case of mica, wollastonite, carbon, garnet and sand filled PTFE, the lowest value in percentage reduction in tensile strength (in comparison with other filled grades) might be due to the fact that hydro fluoric acid liberated during sintering (375^{o}C) might be reacting with mica, wollastonite, garnet and sand filled PTFE resulting in the lowest values of tensile strength of mica, wollastonite, garnet and sand filled PTFE. In case of other filled grades of PTFE, the reaction of hydro fluoric acid with the fillers might not be effective to that extent as in case of glass filled PTFE. In case of tixolex-25, mica and china clay filled PTFE, as tixolex-25 is a synthetic hydrated sodium silica aluminates the liberated HF and fluorocarbons from PTFE at sintering temperature (375^{o}C) might be reacting with tixolex-25 resulting drastic fall in tensile strength values. Individual filler particle or cluster of fillers may be acting as barrier in between PTFE particle preventing coalescence/ adherence of PTFE particle during compression resulting in decrease in tensile strength values

Table- 2: Tensile strength (kg/cm^2) values for glass (f), granite, graphite, gannet, alumina, antimonytrisulphide, carbon, marble, mica, china clay, porcelain, sand, bronze and wollastonite filled PTFE for various filler content

Sl. No.	% of filler content	Tensile Strength (kg/cm^2) of						
		Glass filled PTFE	Granitefilled PTFE	Graphitefilled PTFE	Garnetfilled PTFE	Aluminafilled PTFE	AntimonyTrisulphidefilled PTFE	Carbonfilled PTFE
01.	5	164	209	264	117	253	220	204
02	10	158	173	155	109	123	173	172
03	15	129	155	124	52	108	190	89
04	20	97	76	108	22	90	197	83
05	25	66	73	68	60	48	87	72
06	30	40	72	37	22	30	89	-
07	40	30	-	-	-	23	-	-
08	50	-	-	-	-	-	-	-

Sl No.	% of filler content	Marble	Mica	Sand	Bronze	Wollastonite	Porcelain	China Clay
01	5	205	91	128	266	172	246	112
02	10	185	62	96	261	142	159	-
03	15	106	45	55	238	91	144	-
04	20	74	-	54	203	71	111	-
05	25	56	-	25	196	45	93	-
06	30	55	-	20	190	-	67	-
07	40	53	-	-	140	-	23	-
08	50	45	-	-	122	-	-	-

Tensile Strength of virgin PTFE – 348 kg/cm^2

Table- 2 (a): Reduction in tensile strength with respective filler content/nature of filler

Sl. No.	% of filler content	Percentage Reduction in Tensile Strength of						
		Glass filled PTFE	Granitefilled PTFE	Graphitefilled PTFE	Garnetfilled PTFE	Aluminafilled PTFE	AntimonyTrisulphidefilled PTFE	Carbonfilled PTFE
01.	5	52.87	39.94	24.13	66.37	27.29	36.78	41.37
02	10	54.59	50.28	55.45	68.67	64.65	50.28	50.57
03	15	62.93	55.45	64.36	85.08	69.96	48.27	74.42
04	20	72.12	78.16	68.96	93.67	74.13	43.38	76.14
05	25	81.03	79.82	80.45	82.75	86.20	75.00	79.31
06	30	88.50	79.30	89.36	93.67	91.37	74.00	-
07	40	91.00	-	-	-	93.39	-	-
08	50	-	-	-	-	-	-	-

Sl No.	% of filler content	Marble Filled PTFE	Mica Filled PTFE	Sand Filled PTFE	Bronze Filled PTFE	Wollastonite Filled PTFE	Porce-lain Filled PTFE	Tixo-lex-25 Filled PTFE	ChinaClay Filled PTFE
01	5	41.09	73.85	63.21	23.56	50.57	29.31	52.82	68.67
02	10	46.83	82.58	72.41	25.00	59.19	54.31	81.74	-
03	15	78.73	87.60	84.19	31.60	73.85	58.62	94.25	-
04	20	69.54	-	84.48	41.65	79.59	61.10	-	-
05	25	84.48	-	92.81	43.67	87.66	73.27	-	-
06	30	87.06	-	94.25	45.40	-	80.74	-	-
07	40	84.19	-	-	59.77	-	93.39	-	-
08	50	83.90	-	-	64.94	-	-	-	-

Tensile Strength of virgin PTFE - 348 Kg/Cm^2

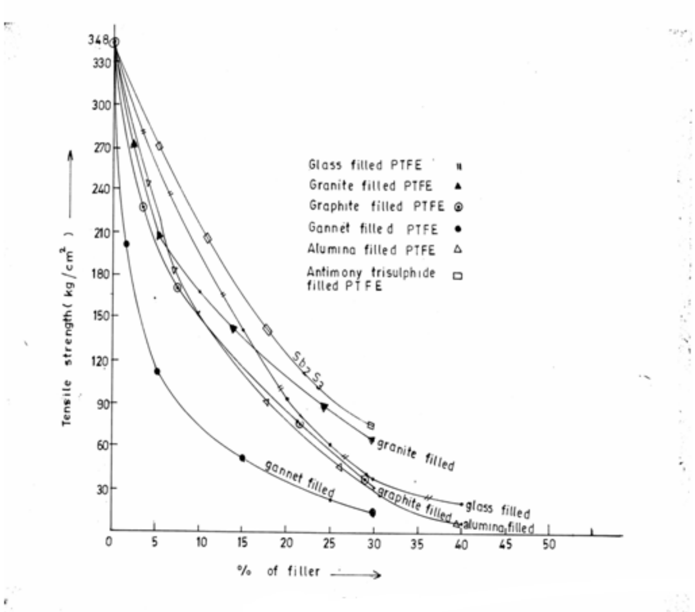


Fig. 2(a) Tensile strength vs percentage of filler and its filled PTFE composites

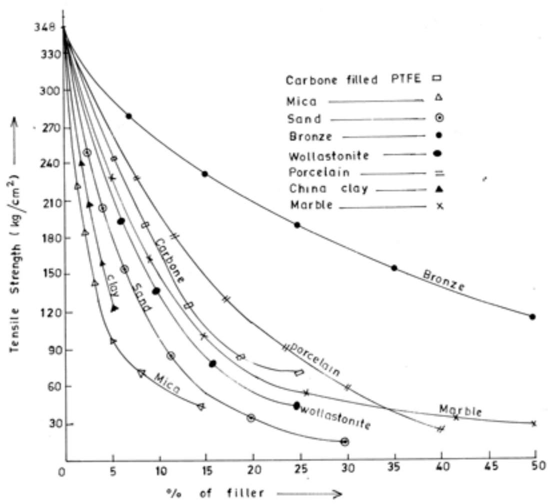


Fig. 2(b) Tensile strength vs percentage of filler and its filled PTFE composites

1.1. Effect of filler and its content on % elongation:

The variation of % elongation with respect to increase in filler content (5-50%) is shown in table 3 and plots of % elongation versus filler and its content are given in fig.3 (a,b) respectively. A decrease in % elongation was observed in all the cases with increasing filler content. However, a very sharp and drastic % elongation was observed in case of mica and trixolex-25 filled PTFE. Reduction in % elongation with increase in filler content for all types of fillers studied is given in table 3(a). For the same filler content, from left to right (3 a), the reduction in % elongation increased. In case of trixolex-25 filled PTFE, the reduction in % elongation was very drastic compared to other filled grade study. With 15% filler content, in case of trixolex-25 and mica filled PTFE reduction in % elongation was about 94.25% and 23%, which might be a peak value as billets could not be made with further increase in filler content. And also for mica filled PTFE, after 15% filler content, billets could not be made with increase in filler content. Whereas, with carbon and wollastonite, after attaining 25% filler content billets got damaged. In these cases also after 25% filler content, we could not proceed further as billets got cracked during sintering. In case of glass filled PTFE, reduction in % elongation was found to be lowest compared to other filled grades. As % of filler increased, a very slight reduction in % elongation was observed in most of the cases. The lowering of % elongation with increase in filler content might be due to the fact that fillers were just lying embedded in the resin matrix without any chemical bonding resulting defects in the billets. In case of glass filled PTFE, the lowest value in the % reduction of % elongation (in comparison with other filled grades) might be due to the fact that HF liberated during sintering (37^{o}C) might be reacting with glass resulting in lowest value in the % of reduction, glass to PTFE. In case of other filled grade PTFE, the reaction of HF with the fillers might not be that extent effective as in the case of glass filled PTFE. In case of tixolex-25 and mica filled PTFE (as mica and tixolex-25 are a synthetic silicates and silica aluminates), the liberated HF and other fluorocarbons from PTFE at sintering temperature might be reacting with these resulting in drastic fall in % elongation values of billets.

Table-3: Percentage elongation of glass (f), granite, graphite, gannet, alumina, antimony trisulphide, carbon, marble, mica, china clay, porcelian, sand, bronze and wollastonite filled PTFE for various filler percentages

Sl. No.	% of filler content	% Elongation of						
		Glass(f) filled PTFE	Granite filled PTFE	Graphite filled PTFE	Garnet filled PTFE	Alumina filled PTFE	Antimony Trisulphide filled PTFE	Carbon filled PTFE
01.	5	350.00	405.00	407.00	349.00	428.00	372.00	473.00
02	10	330.00	383.00	270.00	293.00	298.00	349.00	338.00
03	15	270.00	338.00	236.00	95.00	190.00	338.00	74.00
04	20	230.00	270.00	118.00	60.00	135.00	315.00	45.00
05	25	200.00	203.00	50.00	56.00	68.00	150.00	45.00
06	30	113.00	150.00	24.00	19.00	56.00	146.00	-
07	40	56.00	146.00	-	-	23.00	-	-
08	50	-	146.00	-	-	-	-	-

Sl No.	% of filler content	Marble filled PTFE	Mica filled PTFE	Sand filled PTFE	Bronze filled PTFE	Wollastonite filled PTFE	Porcelain filled PTFE	Tixolex-25 filled PTFE
01	5	327.00	90.00	293.00	552.00	360.00	350.00	209.459
02	10	304.00	45.00	191.00	518.00	203.00	245.00	81.053
03	15	169.00	23.00	110.00	492.00	135.00	165.00	25.252
04	20	135.00	-	60.00	484.00	56.00	105.00	-
05	25	68.00	-	45.00	417.00	34.00	60.00	-
06	30	45.00	-	23.00	405.00	-	45.00	-
07	40	25.00	-	-	338.00	-	23.00	-
08	50	23.00	-	-	270.00	-	-	-

China Clay 5% of filler content is 162 Percentage of Elongation.
PTFE Percentage elongation of virgin PTFE - 450.00

Table- 3 (a): Percentage reduction in elongation of filled PTFE with respect to filler content

Sl. No.	% of filler content	Percentage Reduction in Elongation of						
		Glass filled PTFE	Granite filled PTFE	Graphite filled PTFE	Garnet filled PTFE	Alumina filled PTFE	Antimony Trisulphide filled PTFE	Carbon filled PTFE
01.	5	22.22	10.00	9.55	22.44	4.88	17.33	-5.11
02	10	26.66	14.88	40.00	34.88	33.70	92.44	24.88
03	15	40.00	24.55	47.55	78.88	57.55	24.88	83.55
04	20	48.00	40.00	73.70	87.55	70.00	30.00	90.00
05	25	55.55	54.88	88.80	78.88	87.55	66.66	90.00
06	30	74.88	66.66	94.60	95.77	69.70	67.55	-
07	40	87.55	67.55	-	-	87.50	-	-
08	50	-	67.55	-	-	-	-	-

Sl No.	% of filler content	Marble	Mica	Sand	Bronze	Wolla-Stonite	Porce-lain	Tixo-lex-25	China Clay
01	5	27.33	80.00	34.88	-22.66	20.00	22.22	53.45	64.00
02	10	32.40	90.00	57.55	-9.33	54.80	45.50	81.98	-
03	15	62.44	94.88	75.55	-7.55	70.88	63.30	94.38	-
04	20	70.00	-	86.68	-15.55	87.50	76.60	-	-
05	25	84.88	-	90.00	10.00	92.40	86.60	-	-
06	30	90.00	-	94.88	7.33	-	90.00	-	-
07	40	94.40	-	-	40.00	-	88.00	-	-
08	50	94.88	-	-	42.25	-	-	-	-

Tensile Strength of virgin PTFE - 450

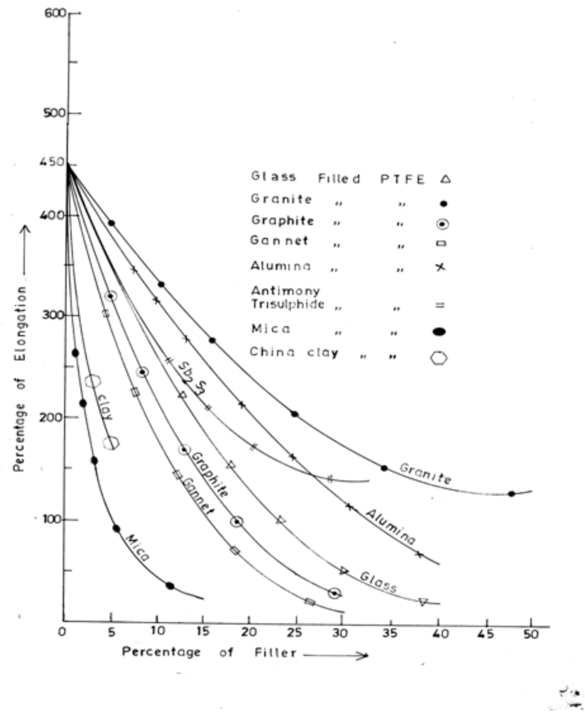


Fig. 3(a) Percentage elongation of filled PTFE composites vs percentage of filler

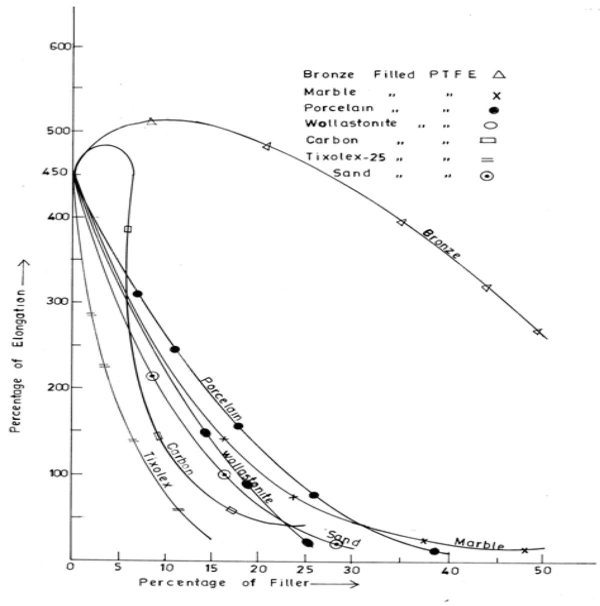


Fig. 3 (b) Percentage elongation of filled PTFE composites vs percentage of filler

CONCLUSION

In the present study, an attempt is made to check mechanical properties of glass, granite, graphite, garnet, alumina, antimony trisulphide, carbon, marble, mica, sand, bronze, wollastonite, porcelian, china clay, tixolex-25 filled PTFE as a detailed study. However, PF resin and activated carbon cannot be used as filler in filled PTFE as they failed in filler stability test (400^{o}C). Highest hardness value based composites were found to be 15, 20 and 25% in case of garnet filled PTFE, 50% in case of marble filled PTFE and 40% in porcelian filled PTFE. 5% bronze filled PTFE shows highest tensile strength and % elongation values.

Addition of ceramic, metallic and non-metallic fillers greatly affects the hardness and tensile strength of PTFE. Among all the filled PTFE studied, tensile strength and % elongation of filled PTFE decrease on increasing filler content (in most cases). Bronze filled PTFE is suitable in applications where optimum tensile strength and elongation properties are preferred.

Future scope: Further work on wear properties, thermal properties and chemical resistance properties of each filled grade PTFE may be done keeping in view of specific applications / end uses.

Acknowledgments
Authors wish to thank Sravani Vidhushi. G & Meghana Shivani. G for their technical support in completion of this project.

REFERENCES

[1] G Venkateswarlu.,Sharada.R.,and Bhagvantha Rao.M., *Journal of chemical and Pharmaceutical Research*, **2014**,6 (10) 508-517.
[2] J.A Brydson, ., Plastics materials.London-Boston: Bulterworths ,**1989**.
[3] Jing Lai., Fengchun Jiang., Kenneth S Vecchio., Marc A. Mayers., Vitali F. Nesterenko., *American institute of Physics,* **2007,** 978.
[4] Wikipedia