Chapter 9

Recycling of Polytetrafluoroethylene (PTFE) Scrap Materials

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Additional information is available at the end of the chapter

http://dx.doi.org/10.5772/59599

1. Introduction

1.1. Plastics

Polymers are formed from *thermoplastic* and *thermosetting plastic* materials. The binding forces between polymer chains in thermoplastics such as polyethylene are the result of van der Walls forces between the molecules and mechanical entanglement between the chains as shown in Fig.1. Most of the thermoplastics can be reused after melting since the bonds between the molecules are easily broken on heating. However, in thermosetting plastics such as Bakelite various polymer chains are held together by strong covalent bond. They are rigid, strong and more brittle. Due to strong covalent bond and cross-link, they are insoluble in almost all organic solvents. They will not become plastic when heated.

1.2. PTFE

Polytetrafluoroethylene (PTFE) was discovered by a research chemist in DuPont in the year 1938. In 1941 it has been patented and got the first brand name as Teflon. It is a fluorinated polymer obtained from tetrafluroethylene (TFE) monomer through free radical vinyl polymerization. Tetra means four carbon atoms are covalently bonded to carbon atoms. Fluro means bonded atoms are fluorine. Ethylene means carbon atoms are joined by a double bond as in the case of ethylene.

In PTFE, carbon to carbon atom double bond becomes a single bond and a linear chain of carbon atoms are formed with two fluorine atoms covalently bonded to each carbon atom. These fluorine atoms shield the carbon atoms and hence no solvent can attack the carbon atoms. As a result, PTFE exhibits extraordinary chemical resistance to acids and alkalis. Carbon to fluorine bonds have high dissociation energy. Due to the high electronegativity of fluorine,

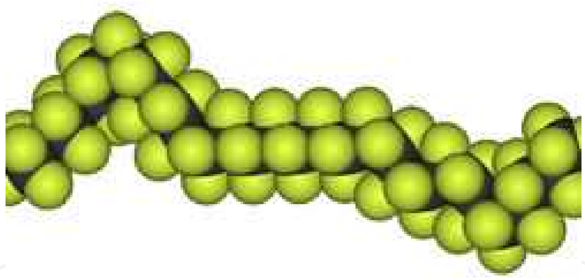

Figure 1. Typical interwinded PTFE polymer. Due to strong intermolecular forces, the polymer chains are tangled. Due to its chemical inertness, PTFE cannot be cross-linked.

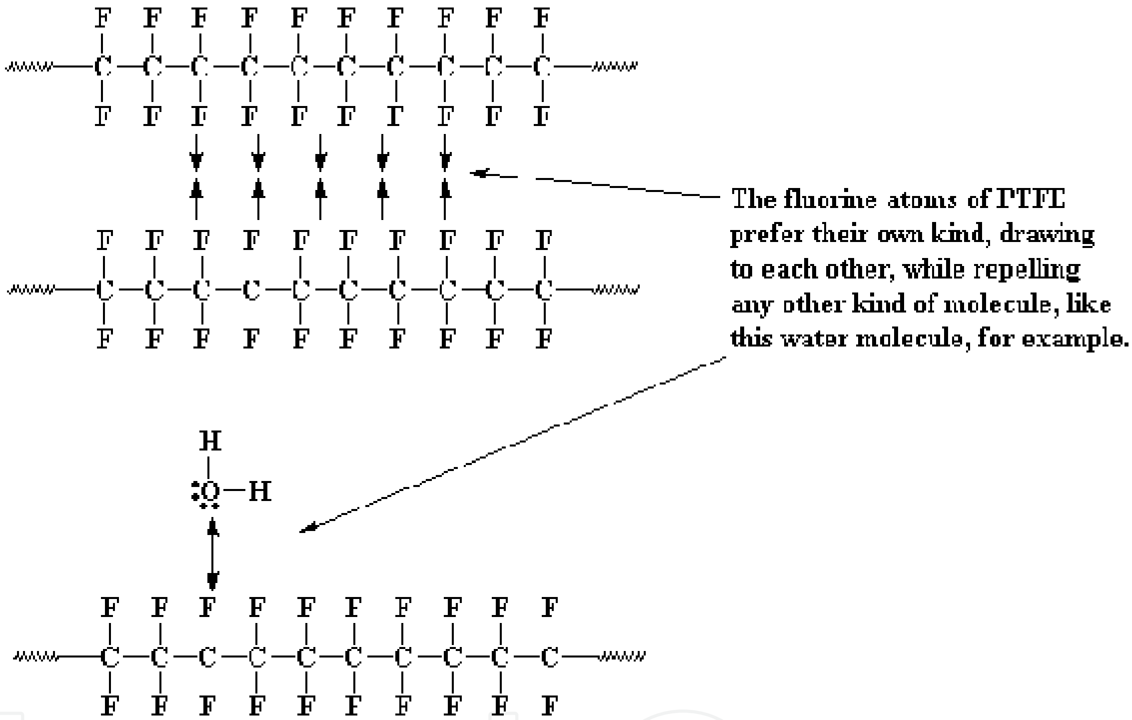

Figure 2. The molecular structure of PTFE

PTFE repels everything and hence no molecules can stick to the PTFE surface which makes it slippery (Fig.2). Ice is the only material that is slicker than PTFE. A thin PTFE coating over metal cooking pans makes them nonsticky with food items. PTFE can withstand a wide range of temperature (-184°C to 260°C) and is used in cold as well hot environments. It is hydrophobic (water repellant) and hence is resistant to weathering. It has fantastic chemical resistance and superb electrical insulation properties. It is the only plastic which can withstand temperatures up to 300°C. On heating to temperatures above 400°C, PTFE disintegrates with the production of carbon. Above 500°C, when heated in air, PTFE disappears altogether due to the production and escape of carbon and fluorine in the form of CO_2 and fluorine gases into the atmosphere [1]. PTFE is insoluble in common solvents and is resistant to nearly all acidic and alkaline

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materials. It has a high dielectric strength and low dielectric loss. Due to high melt viscosity, injection or blow molding is not possible with PTFE. Only hot sintering or ram extrusion manufacturing processes which are relatively expensive are being followed for making PTFE products. In rapidly growing economies like China, the demand for PTFE has grown 5 times over the past 5 years.

2. Filler grade PTFE

PTFE undergoes creep (deformation under loading) which can be reduced with the help of high shear modulus fillers such as glass. Fillers hinder the relative movement of the PTFE molecules past one another and in this way reduce creep or deformation of the parts, reduce the wear rate of parts used in dynamic applications, and reduce the coefficient of thermal expansion. Other popular fillers used along with PTFE include carbon (improved thermal conductivity and low deformation under load), graphite (improved lubrication), bronze or stainless steel (excellent wear resistance) etc. Since PTFE powder is hydrophobic (it floats in water as seen in Fig.3) and does not flow freely, mixing it will free flowing fillers is a major task. One has to use a cryogenic medium such as liquid nitrogen to remove the electrostatic forces that hold the PTFE powder together. This technique is being used to manufacture thermoluminescent material filled PTFE discs (1:3 weight ratio) which are used for personnel radiation monitoring in India and elsewhere [2]. After radiation exposures, these discs are usually heated to 300°C during luminescence measurements and PTFE is the suitable binder for such applications. Organic liquids such as ethanol can also be used to mix free flowing fillers with non free flow PTFE since they wet PTFE powder unlike water medium. Alternately, one could use mechanical shearing force to separate the PTFE particles. The last choice is industrially viable and hence was adopted by us for manufacturing filler grade PTFE powders (Figs.4 and 5). An overview of different fillers used along with end use can be had from the brochures supplied by Dupont and other PTFE manufacturers.

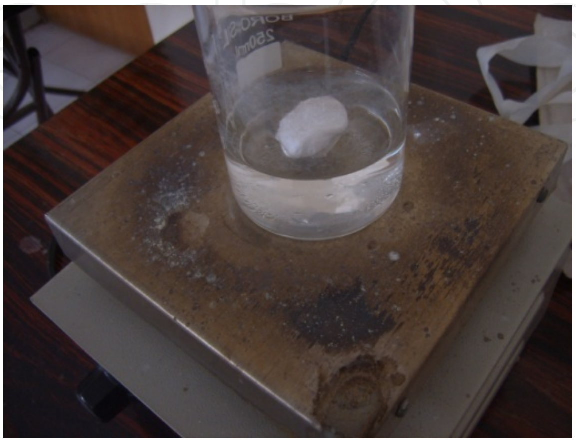

Figure 3. PTFE is hydrophobic – water repellant

Figure 4. A blue pigment (3%) mixed non free flow PTFE powder.

Figure 5. Sintered rods made from blue pigmented PTFE, glass mixed PTFE and carbon mixed PTFE

3. Recycling PTFE scrap material

Use of plastics has the capability to cause danger to both environment and human life. PTFE scrap can be neither incinerated nor dumped! In the waste incineration processes which have been usual up to now, highly-corrosive vapors are released which also damage the incineration

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plant itself. Dumping of waste will in future be restricted due to nationwide regulations. In order to avoid this, Environmental Protection Agencies have taken up an action to recycle all the PTFE materials.

Recycling PTFE involves two different processes with two different end results. One involves irradiation of the PTFE scrap to heavy dose of ionizing irradiation which will reduce its molecular weight. On pulverizing, the irradiated PTFE scrap turns into *micro powder* which finds certain applications. The second method involves pulverizing the PTFE scrap without irradiation so that it becomes reusable like virgin PTFE itself.

4. Irradiation stability of PTFE

From Table 1 it is seen that among all plastics, PTFE has the least stability against ionizing radiation. Heavy gamma or electron irradiation (several kilo Gray) has been found to break down carbon-carbon bonds in the polymer chain in the PTFE scrap and reduce its molecular weight which makes it very brittle and the end product is a white, free-flowing PTFE powder which was found to be useful as additives in other materials or systems (see Fig.6). While the turnings of PTFE scrap before irradiation are tough and elastic, those after irradiation in air crumbles into a powdery material. The molecular weight of irradiated PTFE is in the range of a few tens of thousands to a few hundreds of thousands, compared to several million for the unirradiated resins. When irradiated in vacuum or inert atmosphere, the cleavage of the bonds produces highly stable radicals. The recombination of these stable radicals prevents rapid degradation of PTFE, as the molecular weight rebuilds. When irradiation is conducted in air, as is the case in the present experiment, the radicals react with oxygen leading to smaller molecular weight PTFE chains fairly quickly.

Figure 6. PTFE scrap before (left) and after gamma irradiation (right)

Plastic	Ionizing Radiation Stability
ABS	Fair
Amides	
– Aliphatic	Fair
– Aromatic	Excellent
Cellulosics	Fair
Fluoroplastics	
– PTFE	Poor
– Polychlorotrifluoroethylene	Fair
– PVF	Good
– Polyvinylidene fluoride	Good
– Copolymers of ethylene & TFE	Good
– Polycarbonate	Good
Polyesters – aromatic	Good
Polyolefins	
– PE	Good
– PP	Fair
– Polymethylpentene	Good
– Copolymers	Good
Polystyrene	Excellent
Polystyrene acrylonitrile	Good
Polysulfones	Excellent
Polyvinyls	
– PVC	Good
– Copolymers	Fair

Table 1. Radiation Stability of thermoplastic polymers [3]

4.1. PTFE Micropowder

The irradiated and pulverized PTFE scrap differs from PTFE granular resins and fine powders because of the very small particle size, typically in the range of 2 to 20 μm (and hence the word *micropowder*), low molecular weight and the way they are handled and processed. Micro powders were first developed as an outlet for the disposal of scrap resin. The recycled PTFE powder cannot be used for the same applications as PTFE since its properties are quite different; it cannot be molded in the same way and it does not possesses the same plastic properties of virgin PTFE. However, it can be used as an additive/lubricant in other materials (printing ink, thermoplastics, elastomers, coatings and lubricants) and can be used over a wide range of temperatures from -190 to 250°C and depending on the application, may provide non-stick properties, improved lubricity, better wear resistance and reinforcing properties [4]. More of these applications can be seen in the pamphlets published by Dupont. Micro powders down to four micron range used as additives for Inks, Oils, Lubricants, Paints and Coatings, Cosmetics and Thermoplastics show enhanced lubrication and wear resistant properties.

Irradiation time in sec	Dose, Mrad	Average particle size (μm)
2.5	5	11.1
5.0	10	5.3
7.5	15	2.5
10	20	1.5
12.5	25	0.9

Table 2. Effect of electron irradiation dose on PTFE Micropowder Particle Size

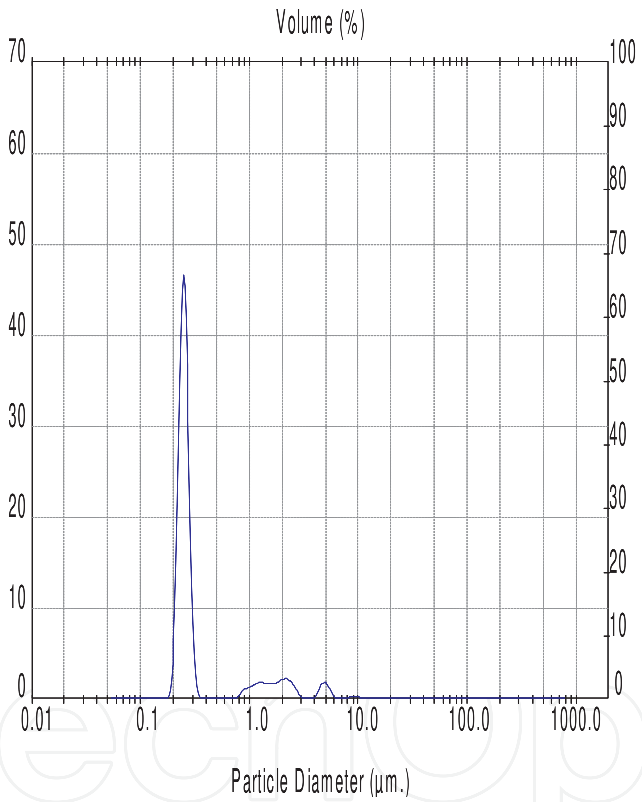

Figure 7. Particle size analysis of micro powder using laser light scattering.

Table 2 shows that with increasing electron irradiation dose in the range 5 – 25 Mrad, the average particle size of the PTFE micropowder decreases from 11.1 to 0.9 μm. The melt flow index goes up as the molecular weight of the powder goes down with increasing dose of irradiation. Electron irradiation is reported to cause cleavage of bonds and generation of gases such as HF acid vapor, which must be removed by means of adequate ventilation from the processing areas. Electron irradiation also increases the temperature of the sample which is held below 121°C by fractionating the irradiation. Both these problems are much less severe in gamma irradiation as the irradiation rate is several orders of magnitude less than that of electron irradiator.

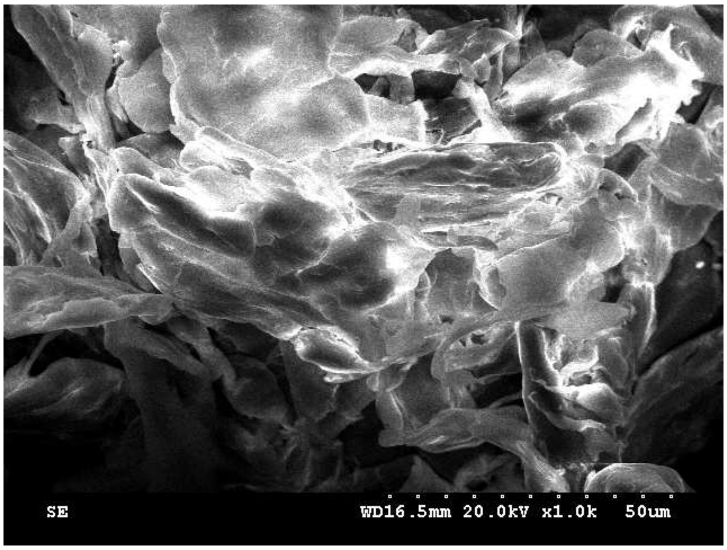

Figure 8. Scanning electron micrograph of PTFE micro powder - irradiated and pulverized

Particle size distribution of the micropowder was carried out using a laser light scattering instrument (Aerosol Dust Monitor Model 1.108 of M/S GRIMM Aerosoltechnik, GmbH, Germany). The scattering angle by a single particle is inversely proportional to the size of the particle. By measuring the forward angle of scattering and intensity of the scattering light, both size distribution and number concentration could be obtained. Fig.7 shows that an average particle size of 0.26 μm was obtained on electron irradiation of the PTFE scrap followed by milling which was achieved by a jet mill or a hammer mill. In the jet mil particles strike against each other, causing them to fracture into smaller particles. The flaky morphology of imported scrap powder seen in Fig.8 offers better lubricant property of the micro powder.

5. Reprocessing of unirradiated PTFE scrap

Unirradiated PTFE scrap can be recycled into many other products and used for rods, tubing and sheets etc by pulverization followed by suitable heat treatments similar to that of virgin PTFE. The recycled PTFE is known technically as “Reprocessed” or “Repro” PTFE or mechanical grade PTFE. Though off-white in color reprocessed PTFE has certain advantages over virgin PTFE namely low creep and better mechanical strength.

Unlike other thermosetting plastics, on heating to high temperatures PTFE products do not melt but only soften above 327°C. Therefore techniques other than those used for recycling conventional thermosetting plastics such as polyethylene have to be employed for recycling PTFE. One way is to grind PTFE scrap into fine powder and sinter it under pressure after suitable heat treatment and cleaning process. Another way is to decompose PTFE into its raw

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gaseous components (tetrafluoroethene and hexafluoropropene), cleaned and fed back into the production of new PTFE. Dyneon GmbH is building a pilot plant in Burgkirchen, Germany based on the latter technique, to recycle PTFE scrap. It will have capacity to recycle 500 metric tons of PTFE waste annually.

The most common way is to blend the pulverized scrap fine powder with pure PTFE to be used either in compression molding or ram extrusion. Before grinding, the scrap is usually shredded (Figs. 9 and 10) and heated to above its melting point to remove any volatile contaminants. Once ground, it is treated with acid to dissolve inorganics after which it is washed

Figure 9. PTFE scrap being shredded

Figure 10. Shredded scrap

Reprocessed PTFE grade powder is manufactured from pre-sintered PTFE shavings, scrap, etc. It exhibits most of the properties that the virgin grade does but is subject to occasional contamination within the material. This is the grade of choice when cost is a major concern and cleanliness is not an issue. When repro grade is mixed with PTFE the cost comes down. Such mixed grades are used when high purity is not required such as non critical chemical, electrical and mechanical applications [5-8]. However, virgin grade PTFE is the material of choice for use in pharmaceutical, food and beverage, and cosmetics industries or for medical/ electrical applications. Virgin PTFE has better friction characteristics, which may be important in some applications. Reprocessed grade PTFE is produced for thin sheets with a maximum thickness of 0.250", For thicker sheets virgin PTFE is used. However, virgin PTFE is known to undergo creep – deformation under load whereas the compressive strength and deformation under load for reprocessed PTFE are superior to virgin PTFE. Reprocessed grade PTFE also has superior wear resistance than virgin PTFE. Reprocessed grade PTFE rods are available in diameters ranging from 1/8 to 4 inch and lengths of 6 to 12 ft. Reprocessed PTFE is frequently specified for high performance bearings and bushings, particularly in applications that require resistance to corrosive chemicals.

5.1. PTFE has different grades

Grade A: 100% virgin material.

Grade B: 70% virgin material, with 30% recycled material.

Grade C: 50% - 50%

Grade D: 30% virgin, 70% recycled.

Grade E: 100% recycled

High purity reprocessed PTFE is white in color similar to virgin PTFE and is used for applications ranging from extruded billets or molding into tubes, gaskets and ball-valve seats. Lower grade off-white reprocessed PTFE is blended with pre PTFE and is used for packing materials for valve stems and other applications (Table 3).

Repro (%)	**< 5%**	**10-20%**	**>20%**
Visual Slight off white	No notable change		Off white
Component finish	Smooth	Rough	Rough – with powdery burrs
Water absorption	< 1%	> 1%	> 2%
Chemical resistance	No notable change		
Tensile strength	Slightly reduced	Reduced by 10%	Reduced by 20%
Dielectric strength	Slightly reduced	Reduced by 10%	Not suitable
Wear resistance	Slightly reduced	Slightly reduced	Not suitable

Table 3. Properties of different grades of reprocessed PTFE powder.

5.2. Issues to be tackled in manufacturing reprocessed PTFE powder are the following

i. Difficulties in grinding the scrap into a fine powder

ii. Discoloration due to burning of volatiles and organic materials in ground powder

iii. Unlike virgin PTFE scrap filled PTFE or scrap powder could not be sintered at high temperatures (350 -400°C) at atmospheric pressure

iv. Agglomeration and Sintering of the scrap powder during thermal anneal caused cracks in reprocessed PTFE billets during sintering at atmospheric pressure

Efforts needed to solve the above problems are described below.

Figure 11. Charred organic impurities on pre-heating the shredded scrap

Figure 12. Major charred impurities are removed by handpicking

Figure 13. Shredded PTFE scrap after pre-heating treatment

5.3. Grinding PTFE scrap - Kirk-othemer encyclopedia of chemicals

A technique known as Shear Extrusion Pulverisation based on Bridgeman – Anvil was used for this purpose. This technique is also known as

- Double disc mill
- Solid state pulverization (SSP)
- Pressure shear pulverization (PSP).

It is a physico-chemical process in which cohesive forces within the polymer are broken by means of mechanically induced stress.

The process is based on “Bridgeman phenomenon” and is realized inside a specially deigned pulverizer

In PSP, the polymer is subjected to simultaneous action of axial compression and shear stress between two mirror-like smooth working surfaces in the pulverizer and the pulverized

The process is realized below the melting point of polymers so the technical properties of recycled polymers remain unchanged after polymerization

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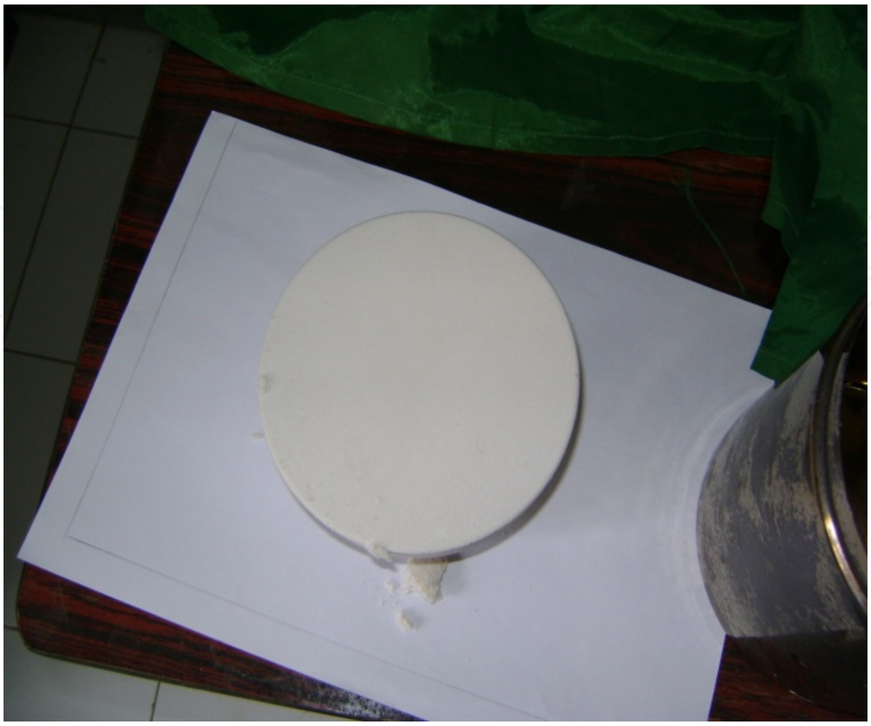

Figure 14. Pre-sintered scrap powder

Figure 15. Ground scrap powder after pre-sintering

Figure 16. Ground PTFE powder being mixed with virgin PTFE powder.

Figure 17. Mixture being cold pressed into pellet.

Figure 18. Fillers like graphite or pigments normally do not melt or agglomerate nor interact with PTFE during sintering at atmospheric pressure but this is not the case with reprocessed PTFE when used alone or used as a filler with virgin PTFE

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Figure 19. Reprocessed PTFE filled (40%) PTFE pellet before (left) and after (right) sintering at atmospheric pressure. The surface became rough on sintering due to agglomeration of reprocessed particles and their migration to surface of the pellet. Reprocessed PTFE is amenable to sintering only under pressure or under ram extrusion.

Figure 20. Sintered rods at atmospheric pressure – note pellets from 100% virgin scrap (second from left) crack and pop out perhaps due to the release of volatile gases whereas the pellet from 100% virgin PTFE (extreme right) are milky white in color and exhibit good integrity on sintering at atmospheric pressure.

6. Compression molding

Compression molding is a method of molding in which the molding material, generally preheated, is first placed in an open, heated mold cavity. The mold is closed with a top force or plug member, pressure is applied to force the material into contact with all mold areas, while heat and pressure are maintained until the molding material has cured. Its advantage lies in its ability to mold large, fairly intricate parts.

Initially it was thought that the die and plunger could be heated during compression in the hydraulic press. A strip heater was wound around the die to heat the die with the help of a heating programmer and a thermocouple was inserted into a hole made specifically for this purpose in the die to measure the temperature during compression, The experimental arrangement is shown below.

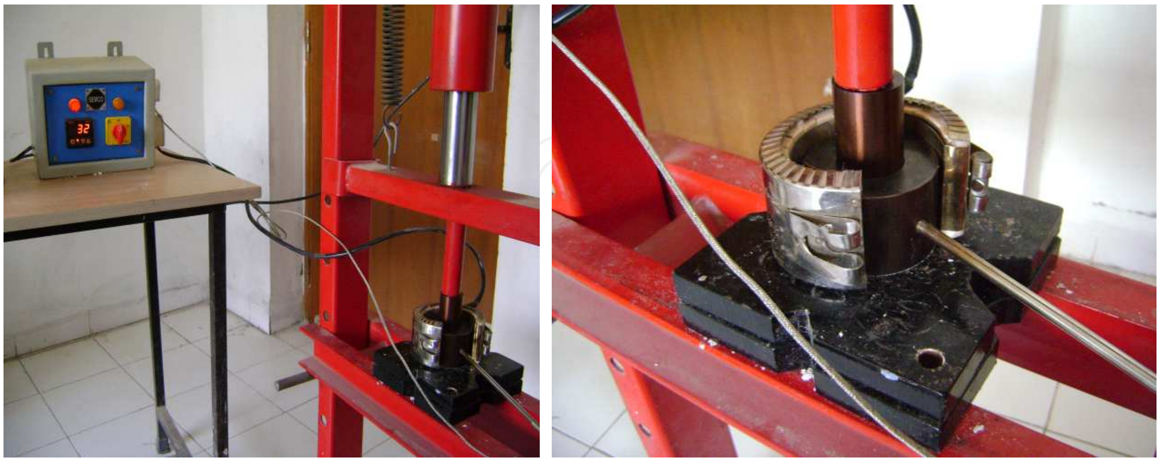

Figure 21. Compression molding

The strip heater wound around the die, however, could not heat it to the desired temperature even after several hours of operation as the heat capacity of the die made out of hardened steel was much high and so this attempt was discontinued. Instead, after cold pressing in the press, the die and plunger itself with the PTFE disc inside it was kept inside the high capacity air over under mechanical pressure.

Figure 22. Cold pressed pellet along with the die and plunger being pressurized with the help of a C-clamp

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Figure 23. Die under C-clamp kept inside the air over for sintering near 400°C

Figure 24. Repro filled PTFE Discs when sintered inside the die and plunger under C-clamp show discoloration which runs through its volume although good surface smoothness is seen and cracks disappeared totally. This was attributed to carbon production on reaction of volatiles with the die material. Even 100% virgin PTFE pellet show slight discoloration but only on its surface (both sides) when sintered under C clamp

Figure 25. 10% mixture, mixee mixed, 300 deg C, 2h pre-heated, 2500 psi, No pressure during sintering at 400 deg C in a furnace. Sample is white in color but surface is rough.

The reason for the discoloration could be due to carbon generation on reaction of volatiles with the stainless steel die under pressure. This can be avoided if a pathway can be provided for the escape of volatile gases during sintering under pressure. This will need fabrication of a new die and plunger with a series of holes. While high pressure (2000 to 3000 psi) is required during cold pressing powder into pellet, a relatively lower pressure (500 to 1000 psi) should suffice during sintering. Further efforts were made by reducing the die pressure to 2500 psi and sintering without pressure.

This showed that mild pressure during sintering is a must. High pressures with clamp not only discolor the pellets but also fuse them with the die. So a compromise in pressure during cold pressing as well as provision to let out the volatile gases are necessary

6.1. Sintering treatment

Sintering temperatures were varied from 350 to 450 deg C and duration from 15 min to 1h. From the points of view of polish, smoothness and strength, the best treatment was found to be 380-400 deg C, 1h which is the same used for sintering virgin PTFE. Lower temperatures resulted in poor strength due to under-sintering while higher temperatures resulted in poor strength as it reduced the polymer strength.

7. Ram extrusion

Ram extrusion enables continuous processing of PTFE [9,10]. The PTFE powder (virgin or reprocessed) is fed into a cylindrical extrusion pipe hydraulically while at the same time compressing it by means of a ram and transported through the pipe, which is heated up to sintering temperature in the range 370 to 400°C (Fig.26). The ram is then withdrawn, the die tube re-charged with powder and the cycle repeated. This way the powder is continuously fed into the heating section of the die tube where it is sintered and then it passes through a cooler section from which the finished products (rods, tubes etc) flow out continuously which are cut into desired lengths. Apart from PTFE materials like ultra high molecular weight polyethylene as well as their compounds can be ram extruded. Uniform distribution of powder into the cavity is essential. The powder should exhibit good flow properties. The extrusion pressure is in the range of 26 to 74 MPa depending on the length of the cold zone above the heated section of the die tube and the extrusion rate is 3 m/h. The heated length of the tube can vary from 44 to 90 cm.

The 50% recycled PTFE ram extruded PTFE rod made in China is shown in Fig.27. It is clear that the discoloration in ram extruded rods (top) made by us was due to the production of carbon (Fig.28). This conclusion was confirmed when the discoloration reduced on subsequent anneal at 400° C for 1h in air oven which led to the escape of carbon into atmosphere in the form of carbon di oxide gas. As a result the extruded rod (bottom) exhibited biscuit color all thorough its volume without any black patches. This is a significant result and confirms that the discoloration is caused by carbon production. The fluorine being a gas produced on disintegration of fluorocarbon perhaps has already escaped during the sintering while carbon in the absence of oxygen has deposited within the rod. Acid treatment before sintering has been found to improve the color further.

Figure 26. A typical ram extruder with 100% virgin extruded PTFE rod.

[Zhejiang, China (Mainland)]

Figure 27. 50% recycled PTFE rod from Deqing VRT Plastic Industrial Co., Ltd.

Figure 28. A 40% repro + 60% virgin PTFE mixture was ram extruded into long rods of diameter 17 mm with the help of a local industry in India. The rods came out fine from the points of polish and strength but was dark in color with patches all through the length and volume due to carbon deposition.

8. Conclusions

Among all plastics, PTFE has the least stability against ionizing radiation, a property which is used to break down carbon-carbon bonds in the polymer chain in the PTFE scrap and reduce its molecular weight which makes it very brittle and the end product is a white, free-flowing PTFE powder which was found to be useful as lubricant additive in other materials or system such as printing ink, thermoplastics, elastomers, coatings and other lubricants. While the turnings of PTFE scrap before irradiation are tough and elastic, those after irradiation in air crumbles into a powdery material. The molecular weight of irradiated PTFE is in the range of a few tens of thousands to a few hundreds of thousands, compared to several million for the unirradiated resins.

Unirradiated PTFE scrap could be successfully ground with the help of a commercial shredder and milling machine. A technique based on Shear Extrusion Pulverization based on Bridgeman – Anvil was successfully used to grind PTFE scrap into a fine powder. Suitable pre-heat treatments were arrived at to remove organic and other volatile impurities. Since normal sintering procedures used for molding virgin PTFE did not work with repro filled PTFE, sintering under pressure and ram extrusion techniques were tried to mold them. Repro filled PTFE Discs up to 40% tried so far when kept inside the die and plunger and pressurized with a C-clamp show discoloration which runs through its volume although good surface smoothness is seen and cracks disappeared totally. This was attributed to carbon production on reaction of volatiles with the die material. The discoloration can, however, be avoided if a pathway can be provided for the escape of volatile gases during sintering under pressure That will need fabrication of a new die and plunger with a series of holes. While high pressure (2000

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to 3000 psi) and hence a solid die and plunger are required during cold pressing powder into pellet, a relatively lower pressure (500 psi) should suffice during sintering.

A 40% mixture was successfully ram extruded by us into long rods of diameter 17 mm. The rods came out satisfactorily from the points of polish and strength but was dark in color with patches all through the length and volume due to carbon deposition. Due to escape of carbon, the discoloration of ram extruded rod considerably reduced on annealing in air at 400°C in atmospheric pressure as the volatile gases found an easy pathway into atmosphere. This is a significant result and confirms our view that the discoloration is caused by carbon production. The fluorine being a gas produced on disintegration of fluorocarbon perhaps has already escaped during the sintering while carbon in the absence of oxygen has deposited within the rod. Discoloration could be further reduced with acid treatment so off-white recycled PTFE rods could be made with ram extrusion.

Acknowledgements

This work was carried out under the TePP-DSIR (New Delhi, India) Techno Entrepreneurship funded project entitled "Development of Filler Grade PTFE powders and Recycling PTFE Scrap Materials". We thank Hindustan Nylons, India for making Ram extruded PTFE rods.

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