EFFECTS OF DIFFERENT SOAKING TIME ON WEAR RESISTANCE OF CRYO-TREATED PTFE MATERIAL

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Abstract - *The objective of paper is to study effects of cryogenic treatment by varying soaking time of its cryo-treatment process on wear resistance of virgin PTFE. PTFE sample were cryo-treated at -196°C for different soaking time of 4, 8, 12 and 16 hours. Mechanical properties like hardness and wear resistance were studied. Wear test was performed on Pin-on-disc machine and average wear in terms of weight loss for all samples were considered. X- ray diffraction test performed to find the changes in percentage crystallinity at microstructural level. From the experiment it was found that cryogenic treatment was an effective method to improve the wear resistance of material and increase in hardness and percentage crystallinity of material after cryogenic treatment was responsible factors for improvement in wear resistance of PTFE material.*

***Key Words*: Cryogenic treatment, Crystallinity, Hardness, PTFE material, Soaking time, Wear.**

1. INTRODUCTION

PTFE is widely used fluoropolymer in different engineering applications like coating material, sealing material, tubing, valve body manufacturing, chemical transfer hoses, nozzles manufacturing etc. due to its excellent properties like high chemical resistance to corrosive reagents, self-lubrication, low coefficient of friction, stability at high temperatures etc. but along with this properties PTFE shows poor resistance to wear, easily gets deform and shows dimensional instability of components which leads to early failure or malfunctioning of components during its application. According to R. Thornton, et al. cryogenic treatment is an effective heat treatment process for improving the wear resistance of materials [1]. According to J. Indumathi, et al. increase in hardness and crystallinity plays important role in improvement wear performance of the polymers [2]. In this paper attempt has been made to study effects of cryogenic treatment by varying soaking time during cryogenic process of PTFE material and its wear test was conducted on pin-on-disc machine, changes in hardness value for each sample was observed and find relationship between harness and wear resistance of PTFE material. Microstructural changes were observed by X-ray diffraction to study changes in crystallinity and amorphous nature of PTFE material.

1.1 Cryogenic treatment

The importance of deep cryogenic treatment process is increases from last few decades in various engineering applications. Cryo process is considered as an add-on process over the conventional heat treatment process. Three sub-zero heat treatments processes can be categories according to their temperature ranges as:

1. Cold treatment (0 to -80°C),
2. Shallow cryogenic treatment (-80 to -160°C),
3. Deep cryogenic treatment (160 to -196°C) which is also referred to as DCT [1].

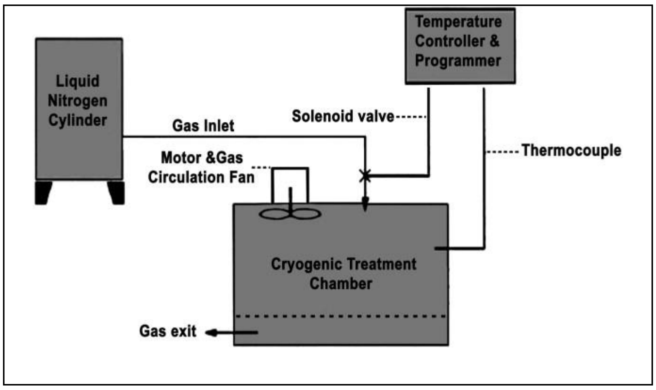


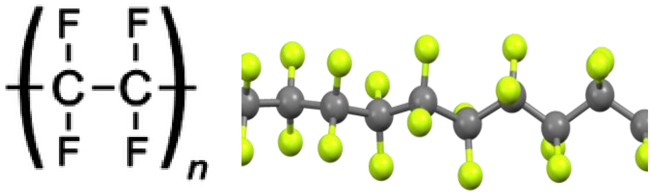
Fig-1: Schematic Diagram of Cryogenic setup

Material which is to be cryotreated was kept inside the cryostat chamber (cryobath) and temperature of cryostat chamber is maintained. This low temperature is maintain by various cryogenic fluid like liquid helium, liquid nitrogen, liquid hydrogen , liquid neon, liquid argon, liquid krypton etc. but liquid nitrogen is most commonly used in engineering applications. In this experimentation also liquid nitrogen is used as cryo fluid. Material is held inside chamber for specific soaking time as per requirement of experiment and then material temperature gradually bring back to room temperature. As Cryogenic processing is a sub-zero heat treatment that affects the entire cross-section of materials or component. Cryogenic treatment process is performed with the objective of improving mechanical properties such as hardness, wear resistance by causing micro structural changes into different stable or meta- stable states of material.

2. EXPERIMENTAL DETAILS

2.1 Material

Polytetrafluoroethylene is fluoropolymer material which is generally known as PTFE material and commercially known as Teflon (named by Du Pont Co.) having chemical formula $-[CF_2-CF_2]-_n$. It is very non-reactive, partly because of the strength of carbon–fluorine bonds.



(a) (b)

Fig-2: (a) Chemical formula and (b) Structure of PTFE

Material was procured in the form of circular rod from the market. Samples are prepared with 12 mm in diameter and 25 mm in length for cryo-treatment and for wear testing.

2.2 Cryogenic treatment process

Prepared samples were placed in cryo chamber for cryogenic treatment at -196˚C. Samples were cool at the rate of 2.5˚C /min and after attending -196˚C; both PTFE and its composite material were held for different Soaking time of 4 hours, 8 hours, 12 hours and 16 hours in cryo chamber. Material was gradually cooled down up to -196˚C by using liquid nitrogen (N_2) as cryo-fluid. Chart-1 shows the temperature vs. time graph for cryogenic treatment cycle.

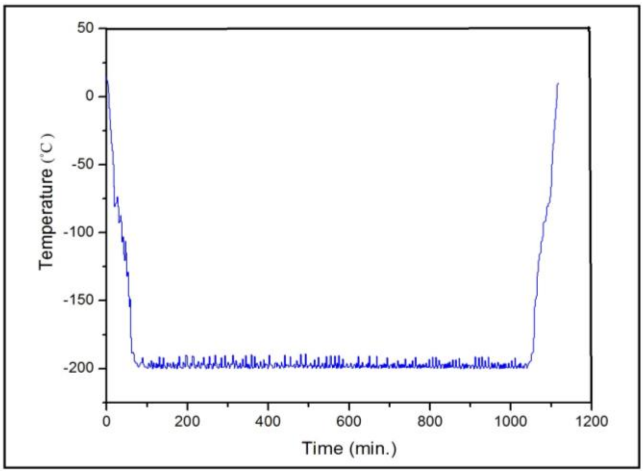


Chart-1: Cryogenic treatment temperature vs. time cycle

Fig-3: Complete setup for Cryogenic treatment

Sample were taken out from cryo chamber as they completed their stipulated time and kept in specially fabricated polystyrene box for gradually bring back to room temperature. Now this cryo-treated and untreated sample of PTFE is consider for mechanical and structural characteristic analysis.

2.3 Wear Test

Wear test is done on pin-on-disc machine of Ducom instruments Pvt Ltd. Company. Material with sample size of ϕ12 mm×25mm is multi pass for adhesive wear test on EN31 disc for 10 minutes with 500 rpm i.e. sliding distance of 1.57 km with sliding velocity of 2.60 m/s by applying load of 2 kg (i.e. 19.6 N) at room temperature.

Fig-4: Wear test on pin-on-disc machine

Experiment was repeated for three times for each reading and average value of wear loss in milligrams (mg) was noted down for each sample.

2.4 Hardness Test

Shore D hardness was measured according to ASTM D2240 for both untreated and cryotreated samples. Shore D hardness was taken at several locations for each sample with indentation load of 40 N for 20 sec.

2.5 X- ray Diffraction (XRD)

X-ray diffractometer was used to generate X-ray diffraction pattern. Changes in crystal structure for both the untreated and cryotreated PTFE were observed. By using X-ray diffractometer (BRUKER D8 Advance) sample were scan Nickel filtered Cu Ka radiation having wavelength λ = 1.54 A° generated at 45 kV and 40 mA by for 2θ angle with step size 0.02 ° and scanning speed of 2°/minute by using angle range from 5° to 70°.

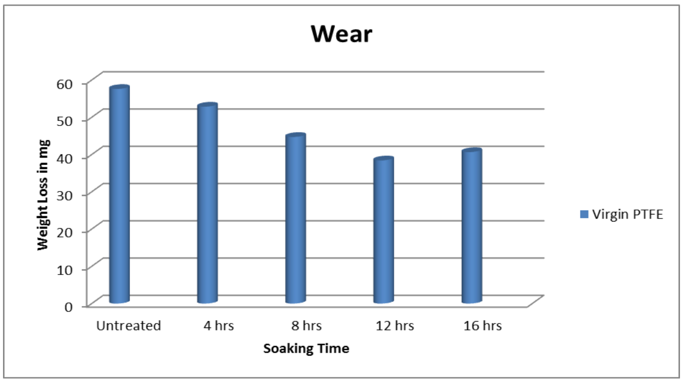
3. RESULTS AND DISCUSSIONS

3.1 Wear test analysis

From the wear test performed on pin-on-disc machine it was found that wear was maximum in case of untreated PTFE and minimum wear for PTFE sample which was cryotreated for 12 hours of soaking time.

Table -1: wear test result in terms of weight loss (mg).

Sr. No.	Soaking time (hours)	Weight loss (mg)	% Improvement in wear resistance (compare to untreated sample)
1	0 (untreated)	57.61	-
2	4	52.85	8.26
3	8	44.72	22.37
4	12	38.43	33.30
5	16	40.66	29.42



From the table it was found that improvement in wear resistance for sample having 12 hours of soaking time was around 33.30 percent which was maximum as compare to all other samples.

3.2 hardness Test analysis

Table -2: Hardness value for all samples

Sr. No.	Sample (Soaking Time)	Hardness Value (Shore D)
1	Untreated	52
2	4 Hours	54
3	8 Hours	55
4	12 hours	57
5	16 hours	56

From the hardness test it was found that hardness value increases as increasing in soaking time of cryogenic treatment process. Hardness value is maximum for 12 hours sample.

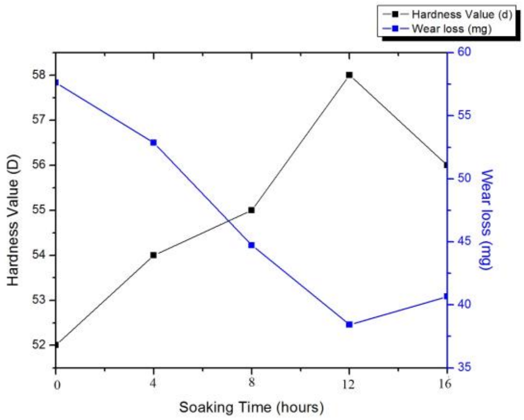


Chart-3: Correlation between hardness and wear

From the graph it was observed that for sample 12 hours cryotreated, value of hardness was maximum and value of weight loss due to wear was minimum. So as the hardness value increases, wear decreases i.e. wear resistance increases. Chart 3 shows correlation between hardness and weight loss due to wear.

3.3 XRD Test analysis

X-Ray Diffraction pattern generated for different soaking time of 4, 8, 12 and 16 hours were compared with the untreated PTFE xrd pattern. Pattern generated by untreated PTFE sample shows two main peaks one small peak at 16.28˚ and another higher peak at 18.01˚ which are correspond to CF_3 and CF_2-CF_2 linkages. Same peaks were observed in Damdhar, et.al. [3]. CF3 peaks shows molecular disorder in PTFE material. Due to cryogenic treatment process CF_3 peak were disappeared. Xrd peaks analysis done on Diffrac.Eva V4.2 software. From the software we had determined the percent crystallinity and percent amorphous nature of material

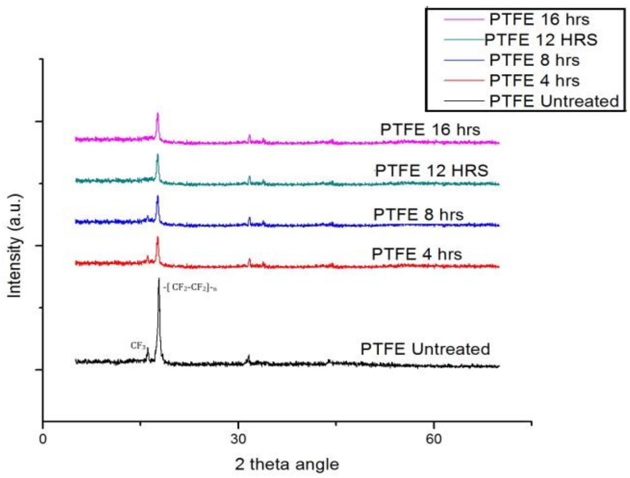


Chart-4: X-ray diffraction patterns for untreated and cryo-treated samples

Theoretically we can find out percent crystallinity by using formula, % Crystallinity = (total area of crystalline peaks) / (total area of all peaks).Where total area of all peaks includes area of crystalline peaks + area of amorphous peaks.

Table -3: percent crystallinity and percent amorphous of untreated and cryotreated samples

Sr. No.	Sample (Soaking Time)	% crystallinity	% amorphous
1	Untreated	36.81	63.19
2	4 Hours	41.92	58.08
3	8 Hours	43.16	56.84
4	12 hours	45.80	54.20
5	16 hours	44.31	55.69

From the table it was observed that % crystallinity shows increasing trend with increasing soaking time up to 12 hours and later it was decreases, similarly wear loss also decreases up to 12 hours and then increases. Chart 5 shows the interrelationship between wear rate, hardness and % crystallinity. From the testing it was clear that 12 hours cryotreated sample shows maximum % crystallinity and minimum wear loss at the same time.

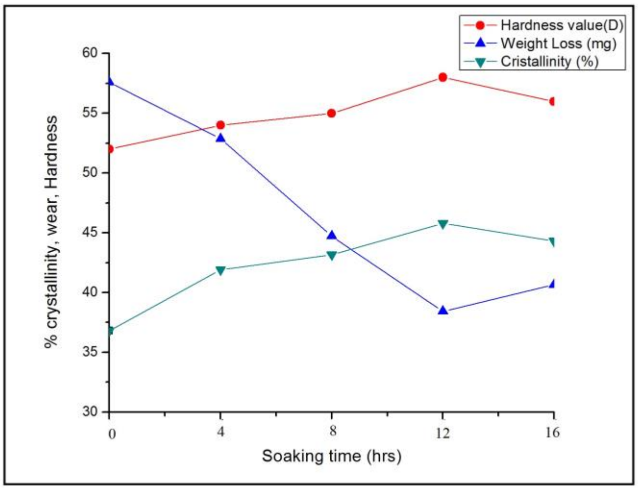


Chart-5: Correlation between % crystallinity, hardness and wear loss

4. CONCLUSIONS

From the experimentation it was found that cryogenic treatment process is an effective method for improving the wear resistance of PTFE material. Following conclusions can be made from the experimentation.

- Increase in wear resistance is found to be maximum for 12 hours of soaking time sample. Around 33.03 % improvement of wear resistance is observed for 12 hours cryotreated PTFE sample.
- Increase in hardness of PTFE material plays an important role in improvement in wear resistance of material. As Hardness of material increases, Wear resistance of material also increases up to 12 hours of soaking time.
- Soaking time of 12 hour is considered as an optimum time for cryogenic treatment for PTFE material.
- Material having higher value of percent crystallinity is one of the factors responsible for improvement in wear resistance of material as uniformity and stability of material after cryo-treatment of is increases.

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