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Influence of cryogenic treatment on the friction co-efficient of nylon 6 and caprolactam - graphite composite

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ABSTRACT

Cryogenic treatment is an effective treatment process to enhance the hardness and wear resistance of some metal parts under rubbing conditions. Earlier studies have revealed the reduction in friction as an after effect of cryogenic treatment in metals. Although friction and wear resistance enhancement in tool steels is important for tribological applications, never the less, some polymers are found to be very suitable under certain tribological environments. Thus it is very important to enhance the tribological properties of certain useful polymers and polymer composites. In addition, some of the polymers and polymer composites are self lubricating. Thus any technique or process involved in enhancing the wear resistance and reducing friction is welcome. The present study is intended to investigate the effect of cryogenic treatment on the friction of polymer and polymer composite material parts.

Keywords: Cryogenic treatment, co-efficient of friction, nylon6, caprolactam-graphite composite, Pin-on-Disc

1. INTRODUCTION

Zamud [10] investigated the methods of improving service life of tools made of steels by cold treatment which was first proposed in USSR in the year 1937. In contrast to the usual treatment of steel with cold, which must be conducted from room temperature directly after quenching, shock cooling can be used both in the process of heat treatment and after finishing of the tool. Cold treatment can be conducted with the tools both at room temperature and high temperature.

In the recent few decades interest in low temperature effects have been demonstrated particularly during heat treatment cycles of tool steels. The initial studies conducted by Barron [1] on the different kinds of tool steels showed remarkable improvement in the tool life and wear resistance. Barron [1] investigated the effect of cryogenic treatment for several materials including the M2 high speed steel at -84°C (maintaining it at this temperature for 24h) observed a significant improvement on the wear resistance in sliding abrasion tests when compared to conventionally heat treated steel (quenched and tempered). When the temperature of the cryogenic treatment is reduced further to -189 °C, the wear resistance increased even more. The literature review indicates that the lives of tools and other steel components may increase significantly after being submitted to subzero (below 0°C) temperatures. The results are surprisingly good, depending on the application. Reports of 92% to 817% increase in tool performance after they have been treated at –189 °C are found.

The work of Chen [2] is the investigation of the effect of cryogenic treatment on the micro-structure and properties of twelve kinds of commercial Al alloys. The study extends the scope of the cryogenic treatment to the materials other than steel. The Al alloys are cryo-treated at –193 °C for 24 h for the investigation. The results show that cryogenic treatment can improve the room temperature strength of some Al alloys but reduce the ductility of these alloys.

The work of Brostow [9] provides good foundations for the tribology of materials science and engineering with the emphasis on polymer science and engineering. The investigations propose a methodology for the characterization and determination of friction with simple mechanical testing machines. The study provides an example of friction resulting in a polymer system of the thermo-set and thermoplastic blend type.

A kind of PTFE based multilayer self lubricating composite materials with steel-backing was fabricated by Zhao [11]. The friction and wear properties as well as limiting PV are studied by sliding the material against steel. The friction co-efficient is determined by measuring friction torque during the testing. Wear is determined by weight loss method. The experimental results show improvement in friction and wear behavior of PTFE composite.

Towards the fabrication of self-lubricating materials the work of Shin [8] is significant. A low-friction coefficient material with its color based on proper selection of polar conditions of pigment and liquid lubricant was developed. An excellent self-lubricating material of frictional coefficient less than 0.02 is fabricated which is superior to the most of the commercial products claiming a low friction coefficient μ = 0.05 to 0.06.

Indumathi [5] and others studied the wear of cryo-treated engineered polymers and composites. In the study the wear tests are conducted on a series of thermoplastic polymers such as PolyImide, PTFE, PolyUrethane, PolyCorbonate, PolyEtherImide ad their Co-Polymers. The cryogenic treatment for the samples is conducted for 24 hours under liquid nitrogen temperature. The abrasive wear performance is evaluated using a pin-on-disc tribometer. The counter-face for the polymer pins is chosen as silicon carbide abrasive paper. The studies are also conducted for the thermoplastics with glass fiber fillers. The results are analyzed and compared for untreated and cryo-treated samples. The effect of cryogenic treatment is found to be fruitful in case of some polymers. The studies conducted on polymer composites reveal the dependence of cryogenic treatment on the wear resistance which in terms depends on the matrix, filler and the fabrication of the composite.

As described in the literature review the cryogenic treatment is an effective process that brings out toughness in some materials. As per the works of Barron [1] and Chen [2] the cryogenic treatment enhances wear resistance properties in steels and few other metals. In most of the materials friction decreases after cryogenic treatment. Even the mechanisms of the improvements are well under stood in some materials. The cryogenic treatment technique is also applied to some of the polymers and the changes in certain tribo-mechanical properties of interest are studied. But the literature survey indicates the deficiency in the cryogenic treatment for other materials like polymers and composites. The effect of cryogenic treatment on the friction of nylon 6 and caprolactam (nylon 6)- graphite composite is studied in the present work.

2. NYLON 6 AND CAPROLACTAM - GRAPHITE COMPOSITE

2.1 Caprolactam

Caprolactam is the precursor of polymer nylon 6. Caprolactam was first prepared by the cyclization of ε-aminocorpic acid. The most common method of synthesizing caprolactam is from cyclohexanone which is converted to oxime using ammonium hydroxide. When oxime is treated with sulphuric acid caprolactam is produced. Caprolactam is a solid which is white in color. The chemical structure of caprolactam is as shown in Fig.1.

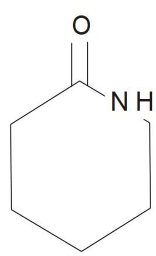


Figure 1: Chemical Structure of Caprolactam

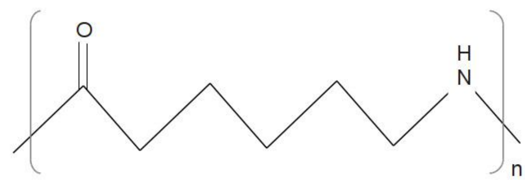


Figure 2: Chemical Structure of Nylon 6

2.2 Nylon 6

Nylon 6 is also called as polycaprolactam. The IUPAC name of polycaprolactam is poly(hexano-6-lactam). Nylon 6 is formed from the polymerization of caprolactam.

n(CH2)5C(O)N H → [(CH2)5C(O)N H]n

The synthesis of nylon is through the ring opening polymerization of caprolactam. Nylon can be readily formed into fibers that are strong and long wearing, making them well suited for use in carpeting, upholstery fabric, tire cords, brushes, and turf for athletic fields. Nylon is also formed into rods, bars, and sheets that are easily formed and machined. In this form, The tribological applications of Nylon 6 are in gears, fittings, and bearings, in automotive industry for under-the-hood parts, as a material for power tools housings and for automobile fuel tanks. The chemical structure of Nylon 6 is as shown in Fig. 2.

2.3 Nylon 6 (caprolactam)- Graphite Composite

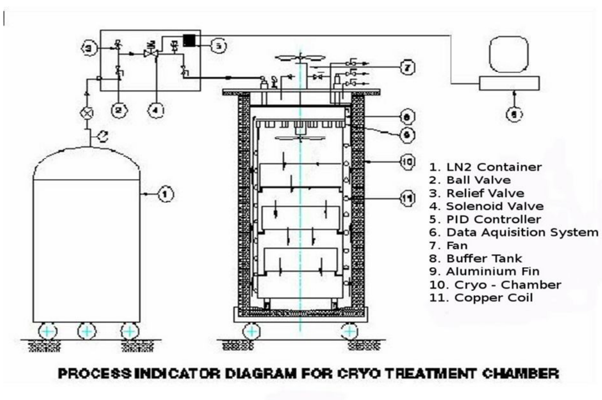
The composite is fabricated with caprolactam matrix and graphite fillers. Preparation of composite systems based on polyamide 6 (PA6) and ex-foliated graphite is attempted by applying a simple procedure, which consists of a preliminary dispersion/ex-foliation of graphite in the monomer, namely, ε-caprolactam, and a subsequent polymerization of the above system [7].

3. SAMPLES PREPARATION

Samples of two kinds of polymers are fabricated in the form of pins each with dimensions 5mm Dia and 25 mm length§. The samples are designated as P1 and B1. The pins are cleaned with water and allowed to dry. To ensure full contact during the friction and wear experiment, the pins are initially run-in against SiC paper of grade 400 under a dead load of 5N. The sliding counterpart polished discs used are made of hardened EN32 steel.

4. CRYOGENIC TREATMENT

The test samples are first wrapped with aluminium foils and then with plastic. The wrapped sample pins are placed inside the cryogenic system [Fig.3.]. The system is provided with a container in which the liquid gets filled. Initially the container temperature is maintained at atmospheric temperature. Further the temperature of the system is gradually reduced to the soaking temperature of 98K with a cooling rate of 2.4min/K of temperature drop. This takes about 8 hours to attain 98K temperature.



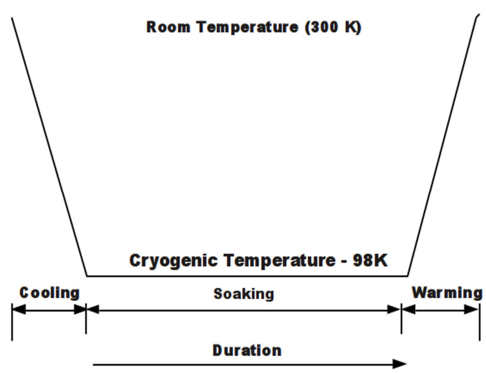


Figure 3: Cryogenic Processor

Figure 4: Cryogenic Treatment Cycle

The cooling is controlled by a data acquisition system which regulates the liquid flow through the solenoid valve. If temperature falls quickly than the preset value, the flow of liquid is stopped so that the temperature drop is maintained at preset-set value (As described in the section Cryogenic Treatment). Once the preset the temperature is attained further the temperature is maintained at the same value for a period of 24 hours (Chen [2], Gopala Krishna [4]. It is a major and important part in the cryogenic treatment. In order to hold this temperature for a period of 24 hours it takes about 450 liters of liquid nitrogen. The beneficial changes of the cryogenic treatment occur during this phase of the treatment. After the 24 hours the system is switched off and allowed to attain the room temperature. The warming is done at the rate of 2.4 min/K and it takes around 8 hours to attain the room temperature. The overall process of cryogenic treatment is as shown in the Fig.4.

5. EXPERIMENTAL

The friction and wear tests for the sample pins are done using a Pin-On-Disc tribometer. The classification of pins is as follows

- Untreated and cryo-treated nylon samples (P1).

- Untreated and cryo-treated caprolactam –graphite composite samples (B1).

The sample pin is fixed in the pin holder of pin-on-disc tribometer and the sliding track radius is selected to obtain the desired linear speed for which the rotational speed of the disc (RPM) is predetermined. The required normal load can be applied by placing weights on the load arm. The RPM can be set in the control unit used to control pin-on-disc tribometer. The data is acquired using a personal computer (PC) with the help of data acquisition card. A software is used to acquire and analyze data. The Data acquisition and analysis software, along with data acquisition, makes real time plots of Co-efficient of friction, Displacement, Normal load, and Frictional force all as function of time. Initially, the experiment is conducted for untreated P1 sample pin keeping the linear speed at 0.5 m/s. The normal load is varied form 20N to 100 N in steps of 20N. The sliding duration for each value normal load is maintained to be 10 min. Full contact is ensured during the run. The above is repeated for other two untreated P1 samples at speeds 1m/s and 1.5 m/s. The repeatability is confirmed by running three samples of the same type for each speed. Same procedure is adopted even for the other samples like cryo-treated P1, Untreated B1 and cryo-treated B1. The same surrounding conditions are ensured for each type of sample. In case of disc heating, the disc is allowed to cool to room temperature and then the experiment is continued with other samples. The results are logged into a data file and stored in the PC with the help of LAB-View software. The experiments are conducted maintaining uniform conditions as mentioned below.

- The sample pins are slid on hardened steel disc (EN32).
- The discs are initially degreased and allowed to dry. Then the discs are cleaned with ethyl alcohol and the ethyl alcohol is allowed to evaporate under atmospheric air.
- The sliding distance is fixed for the different tracks by adjusting the RPM of the disc.
- Separate sliding tracks are used for each sample.
- Typical practical conditions are maintained throughout the experiments.

6. RESULTS AND DISCUSSION

The variation of co-efficient of friction as a function of Normal load for two kinds of self lubricating polymer composites is as shown Fig. 5 and Fig.6. The plots reveal that the influence of cryogenic treatment on the co-efficient of friction for both Nylon 6 and Caprolactam – Polymer Composite.

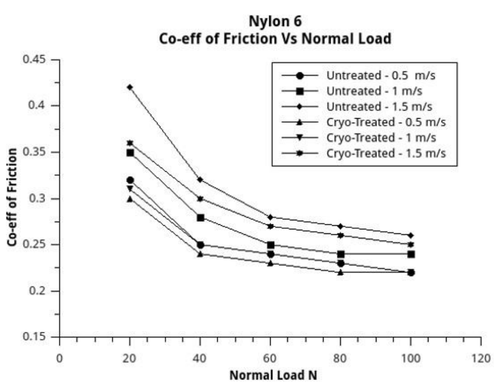


Figure 5: untreated and cryo-treated Nylon 6

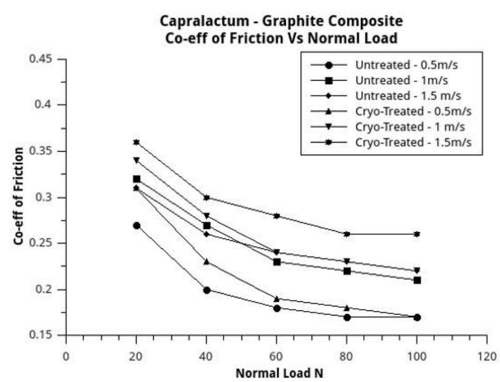


Figure 6: untreated and cryo-treated Composite

6.1 Friction of Nylon 6

The plot in Fig.5. for untreated samples reveal decrease in co-efficient of friction with increase in load for a given speed. The co-efficient of friction for the cryo-treated samples also decreases with increase in load for a given speed but the co-efficient of friction for cryo-treated samples is observed to be lower than the co-efficient of friction of untreated samples. The plot in Fig.6. confirms the increase in friction co-efficient of friction with linear speed at a constant normal load. The co-efficient of friction of nylon 6 is verified in comparison with the work of Gao [3]. The visual observation during the experiment reveals the transfer-film formation of the material from the sample pins made of polymer and polymer composite on to the hardened steel disc [6]. This forms a thin transfer-film lubricating layer of polymeric material on the sliding surface confirming the self lubricating behavior.

6.2 Friction of Caprolactam - Graphite Composite

In case of the untreated samples of the polymer composite the plot in Fig.7. reveals decrease in coefficient of friction with increase in load, for a given speed. The co-efficient of friction for the cryo-treated samples also decreases with increase in load for a given speed but the co-efficient of friction for cryo-treated samples are observed to be higher than the co-efficient of friction of untreated samples. The plot in Fig.8. confirms the increase in friction co-efficient with linear speed at a constant normal load. Formation of transfer-film lubricating layer is very significant when compared to nylon 6. The co-efficient of friction of the composite is smaller than nylon 6, at all loads and speeds, due to the lubricating behavior of graphite and self lubrication of the matrix.

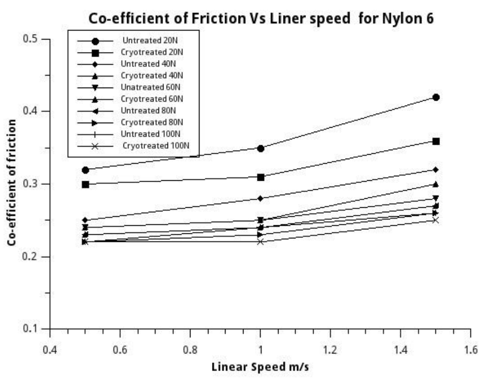


Figure 7: untreated and cryo-treated Nylon 6

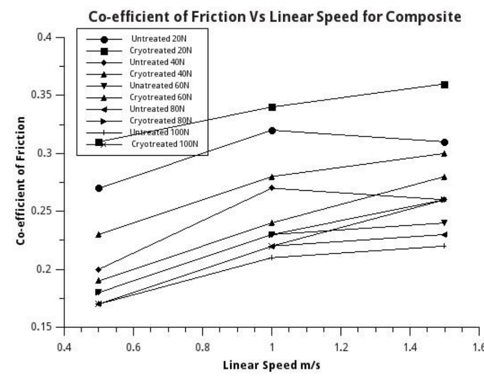


Figure 8: untreated and cryo-treated Composite

The plots in the figures [Fig.7 and Fig.8] reveal the friction characteristics of nylon 6 and composite with reference to three linear speeds. For both Nylon 6 and composite the co-efficient of friction increases with linear speed for a given load.

7. CONCLUSIONS

- The co-efficient of friction is lower for cryo-treated nylon 6 in comparison with the friction co-efficient of untreated nylon 6.
- The co-efficient of friction is higher for caprolactam-graphite composite in comparison with the friction co-efficient of untreated composite.
- The friction co-efficient of caprolactam-graphite composite is less than that of nylon 6.
- The reduction in friction co-efficient of nylon 6 is an after effect of cryo-treatment.
- The cryogenic treatment is not an effective process for reducing friction in caprolactam-graphite composite.

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References

[1] Barron R.F. (1982), 'Cryogenic Treatment of Metals to improve the wear resistance', Cryogenics, pp. 409-413.
[2] Chen Ding, Li Wenxian (2000), 'Cryogenic treatment of Al and Al alloys,The Chinese Journal of Nonferrous Metals (Zhongguo Yousejinshuxuebao Xuebao)', vol 10, issue 6.
[3] Gao Jintang (2000), 'Tribochemical effects in formation of polymer transfer film', Wear, vol 245, pp. 100-106.
[4] Gopal Krishna P. V., Kishore K., Ramadevudu G. & Sikandar Ali (2012), 'Performance evaluation of cryogenic treated tools in turning', Advances in Production Engineering & Management, vol 3, pp. 187-194.
[5] Indumathi, J., Bijwe, J., Ghosh, A. K., Fahim, M. & Krishnaraj, N. (1999), 'Wear of cryo-treated engineering polymers and composites', Wear, vol 225–229, pp. 343–353.

[6] Kinell, P. O., Ranby, B. & Runnstrom-Relo, V. (1973), 'ESR Applications to Polymer Research', Sweden.
[7] Orietta Monticelli, Sergio Bocchini, Alberto Frache, Erika Simona Cozza, Ornella Cavalleri & Luigi Prati (2012), 'Simple Method for the Preparation of Composites Based on PA6 and Partially Ex-foliated Graphite', Journal of Nanomaterials, vol 2012, Article ID :938962.
[8] Shin Jen Shiao, Wei Shung Fu, Chin Lin Tuo & Un I Cheng, (2001), 'Polymer-based self-lubricating material', Journal of Applied Polymer Science, vol 80, issue 9, pp. 1514–1519.
[9] Witold Brostow, Jean-Laurent Deborde, Magdalena Jaklewicz & Przemyslaw Olszynski (2003), 'Tribology with emphasis on polymers; friction, scratch resistance and wear', Journal of Materials Education, vol 24, issue 4-6, pp. 119 - 132.
[10] Zamud, E. (1980), 'Improved tool life After Shock Cooling (Translated from Metallovedenie i termicheskaya Obrabotka Metallov)', vol 10, pp. 3-5.
[11] Zhao-ZhuZhang,Wei-ChangShen,Wei-Min Liu, Qun-JiXue & Tong-Sheng Li, (1996), 'Tribological properties of polytetrafluoroethylene-based composite in different lubricant media', Wear, vol 196, pp. 164-170.

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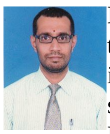

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