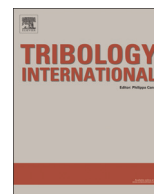




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# The effects of crystallinity on the mechanical properties and the limiting $PV$ (pressure $\times$ velocity) value of PTFE

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## ABSTRACT

In this work, the effects of crystallinity on the LPV (short for the limiting  $PV$ ) value and tribological behaviors were investigated under different surface roughness and velocities. The experiment results revealed that the one which had a higher degree of crystallinity exhibited higher mechanical and thermal properties, lower friction coefficient and higher LPV value. The wear resistance was enhanced with the increase of crystallinity in rubbery state, since larger energy was needed to break the links between crystallites. In the present study, LPV value of PTFE reached to 2.5 Mpa m/s under 2 m/s. Surface roughness of 0.5  $\mu\text{m}$  was the very profile in this work, which exhibited the best performance with the lowest friction coefficient and highest LPV value under 1 m/s.

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## 1. Introduction

Polymers and their composites have attracted tremendous attention and have been extensively investigated in recent years [1–6]. They have replaced the traditional materials in many fields for their unique properties, such as good mechanical properties, lightweight, excellent thermal performance, self-lubricating ability and wear resistance, coupled with outstanding processability and cost-effectiveness. They have been widely used in automotive and aerospace as structural and lubricating materials.

One of the most promising polymers is polytetrafluoroethylene (PTFE). PTFE is a typical semicrystalline polymer; its mechanical properties (including fracture and crack propagation) were largely dependent on subtle changes, such as degree of crystallinity and crystalline phase transformation in the polymer structure. Influences of crystallinity on the mechanical properties depend on which state the amorphous regions of the polymer are in, the glassy or rubbery state. Tsuji Hideto [7] investigated the effects of crystallinity on physical properties and morphologies of poly(L-lactide), and found that the tensile strength increased with increasing crystallinity showing similar behavior to Young's modulus, but decreased when large crystallites or spherulites were formed. Similar results were obtained by Qi Zhou and co-workers [8]. Brown [9–11] investigated the correlation between fracture and crystallinity or crystalline phase transformation. Conclusions

had been made that the crack propagation was largely dependent upon crystalline phase transformation with a brittle-to-ductile transition, and also the fracture toughness and  $\tan\delta$  decreased with the increase of crystallinity.

Crystallinity affects not only the physical characteristics but also the tribological behaviors of polymers. Tanaka [12] found that the wear rate of PTFE was affected by the crystal bandwidth in the fine structure rather than the degree of crystallinity, while the friction coefficient was little influenced by both factors, which was contrary to previous reports [13,14]. Conte et al. [15] explained the relationship between wear mechanism and molecule structure by considering of the contact temperature at the interface induced by frictional heating and studied the links between tribological behavior and degree of crystallinity of PTFE composites [16].

For design purposes, it is important to evaluate the critical operating conditions of lubricating materials, and in particular the dry bearing materials. The  $PV$  factor (the product of the nominal pressure and the linear velocity) is the usual criterion to characterize the thermal and wear resistance of the lubricating materials. A number of research papers dealing with the variety of factors that affect the LPV value (It is short for the limiting  $PV$  value. It is the usual criterion to evaluate the critical operating conditions under which the material fails.) of polymer-based bearings have been published in recent years [1,2,17–20]. The load, velocity and frictional heating have been found to have a significant influence on the LPV value of polymer-based bearings [2,17,18,20–23]. Friedrich and co-workers found that the sliding velocity had a greater influence on  $PV$  limit of PEEK than the contact pressure, and the PEEK with higher molecular weight had

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a higher PV limit than that with lower molecular weight [5]. PTFE is an excellent candidate material for solid lubricant applications because of the low friction coefficient and high melting temperature, which was widely used as bushings and bearings. However, there have been no studies to date elaborating on the effects of crystallinity on the LPV value of PTFE by considerations on the changes of mechanical properties, thermal properties and tribological behaviors.

In this work, we prepared different PTFE materials with different crystallinity by controlling the cooling process. The cold pressing and hot sintering PTFE samples were natural cooled in the furnace, and quenched in ice water, liquid nitrogen and air (25 °C), respectively. Aims of this work are to investigate the effects of crystallinity on the mechanical properties, the limiting PV value and tribological behavior of PTFE under various velocities and surface roughness.

## 2. Experimental

### 2.1. Materials and preparations

PTFE power (M-18 F) was purchased from Daikin Fluorochemicals Co., Ltd. with an average diameter of 75 μm. The PTFE powder was added to a mold after drying at 120 °C for two hours and cold pressed under a pressure of 40 MPa for 20 min. Then, the PTFE samples were taken out from the mold and sintered in an oven at 375 °C for 120 min.

In this work, we prepared different PTFE materials with different crystallinity by controlling the cooling procedure: (1) the cold pressing and hot sintering PTFE samples natural cooled in the furnace, were referenced as TF. (2) Samples of PTFE were cold pressed and hot sintered for 2 h at 375 °C prior to one of three cooling procedures. Samples quenched in the air (25 °C) are referenced as ATTF, in liquid nitrogen referenced as LNTF and the ice water immediately for 30 min referenced as HTF.

### 2.2. Characterization

X-ray diffraction was used to evaluate the crystallinity of the PTFE samples, the degree of crystallinity ( $W_{c,x}$ ) was calculated by the following expression:

$$W_{c,x} = \frac{I_c}{I_c + K_x I_a} \times 100\% \quad (1)$$

Where  $I_c$  is the scattering integral intensity of crystalline area,  $I_a$  is the scattering integral intensity of amorphous area, and  $K_x$  is the correction factor of 0.66.

Differential scanning calorimetry (DSC) was carried out on a NETZSCH DSC-200F3 thermal analyzer under nitrogen atmosphere with a heating rate of 10 °C/min. And also the percentage of crystallinity was estimated by integrating the melt endotherm (heat of fusion) and calculating the ratio with a theoretical 100% crystalline sample. For our calculations, we used a value of 80 J/g ( $\Delta H_f^0$ ) and calculated the weight fraction crystallinity by the relation  $W_{c,h} = \Delta H_f (\text{sample}) / \Delta H_f^0$  [24,25].

Density measurements were performed by immersion in dihydrogen oxide based on volume displacement and sample mass. The density values were used to estimate the mass fraction crystallinity ( $W_{c,d}$ ) using the following expression,

$$W_{c,d} = \frac{\rho_c(\rho - \rho_a)}{\rho(\rho_c - \rho_a)} \times 100\% \quad (2)$$

where  $\rho$  is the sample density,  $\rho_a$  is the extrapolated density of the pure amorphous phase ( $\approx 2040 \pm 30 \text{ Kg/m}^3$ ) and  $\rho_c$  is the

extrapolated density of the pure crystalline phase ( $\approx 2300 \pm 10 \text{ Kg/m}^3$ ) [26,27].

The dynamic mechanical analysis (DMA) was performed over a temperature range from 20 °C to 250 °C on a DMA 242C analyzer (Netzsch Instruments, Germany) with double-cantilever mode at a heating rate of 3 °C/min and a frequency of 1 Hz. In the tests, three specimens (60 mm × 10 mm × 4 mm) were tested for each sample composition.

Tensile properties, flexural and compression performances were studied on an Electron Omnipotence Experiment Machine SANS-CMT5105 (ShenzhenSANS Testing Machine Co., Ltd., China), according to GB/T1040.2/1A-2006 (ISO527-2/1A:1993), GB/T1041-2008 (ISO604:2002) and GB/T9341-2008 (ISO178:2001) under ambient condition, respectively. The Shore scleroscope hardness was determined by a TH210 Shore durometer (Jinan fangyuan test instrument Co., Ltd, China). In the tests, five specimens were tested for each sample composition. The detailed information on the tested materials is shown in Table 1.

### 2.3. Tribological tests

The tribological tests were performed on an Electro-hydraulic servo PV friction testing machine (Jinan Time Shijin Testing Machine Co. Ltd. Shandong, China), shown in Fig. 1A, at room temperature (25 °C) according to the standard GB7948-1987. The specimens were cut from the cold pressing-hot sintering molded sample rings with a dimension of 3 mm × 10 mm ×  $\Phi_{in}35.15$  ( $\Phi_{out}41.15$ ) mm, shown in Fig. 1B, and surface roughness  $R_a$  of 0.2–0.3 μm. In the limiting PV tests, the specimens were sliding under a constant velocity (In this study, velocities of 1 m/s, 1.5 m/s and 2 m/s were chose, respectively. ), the initial load was 0.5 MPa and increased 0.25 MPa every 10 min until the specimen showed a failure. The counterpart was an ASTM1045 steel ring with 35 mm in diameter, surface roughness of 0.3 μm (achieved by the grinding process) and the hardness of HRC 50. To study the effect of surface roughness on the LPV value of PTFE, experiments were performed under 1 m/s, surface roughness of 0.3 μm, 0.5 μm and 0.8 μm, respectively. During the wear process, a thermocouple was used to estimate for the apparent specimen temperature. In this study, at least three repetitive tests were performed to calculate the average wear rate. The time-related depth wear rate ( $W_t$ ) was calculated using the following expression:

$$W_t = \frac{\Delta h}{t} = \frac{\Delta m}{\rho l d t} \quad (3)$$

where  $\rho$  is the density of the specimen,  $l$  is the width of the specimen,  $d$  is the inner diameter of the specimen,  $t$  is the total sliding time and  $\Delta m$  is the wear mass loss of specimen. The worn surfaces of the specimens were observed with a JSM-5600LV scanning electron microscope (Japan).

**Table 1**  
Mechanical strength properties of selected PTFE.

Properties	TF	LNTF	HTF
Tensile elongation (%)	280 ± 4	320 ± 4	370 ± 4
Tensile strength (MPa)	20.3 ± 0.16	22.7 ± 0.16	26.1 ± 0.25
Compressive modulus (GPa)	0.336 ± 0.002	0.269 ± 0.002	0.231 ± 0.002
Compressive strength (MPa)	42 ± 0.82	34 ± 0.82	22 ± 0.82
Flexural modulus (GPa)	0.475 ± 0.001	0.359 ± 0.001	0.338 ± 0.001
Flexural strength (MPa)	12 ± 0.4	10.6 ± 0.4	9.5 ± 0.4
Shore hardness	62 ± 0.25	58 ± 0.25	56 ± 0.3

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