

The effect of processing method on dry sliding performance of polyimides at high load/high velocity conditions

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Abstract

Polyimides can be processed by sintering or injection moulding, depending on their molecular structure. Raman spectroscopy shows differences in imide I (C=O stretch), imide II (C–N–C axial vibration) and imide III (C–N–C transverse vibration) bands for both polyimide types, indicating that (i) further imidisation happens during sintering, while being amorphous and (ii) crystallisation happens during melt processing. Thermogravimetric analysis indicates no glass transition or melting temperatures until degradation at 592 °C after sintering, while moulded polyimides show a glass transition temperature at 250 °C. A dehydration reaction at 180 °C will importantly influence the sliding properties of both sintered and injection moulded polyimides. For sintered polyimide, friction and wear in cylinder-on-plate tests is mechanically controlled and mainly determined by normal loads. Overload for sintered polyimides is due to brittleness above 150 N, as also observed in debris morphology. For moulded polyimide, both normal loads and sliding velocity cause a decrease in friction and increase in wear. Overload for moulded polyimide is thermo-mechanically controlled. Sliding stability is controlled by smooth transfer, which is influenced by thermal action, plastic deformation or shear and promoted by elongation at break. Overload conditions are further discussed in relation to deformation of the Hertz line contact, recovery after sliding and the *pv*-limit (contact pressure × sliding velocity) related to frictional heating.

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1. Introduction

Polyimides are polymers with extreme mechanical strength, chemical inertness and thermal stability, having a linear or cyclic imide unit connected by aliphatic [1] or aromatic [2] groups. Polyimide films were introduced under dry sliding for space applications [3] and tested under vacuum [4], in con-

tact with cooling agents [5] or under fretting conditions [6]. High-performance polymers allow for further miniaturisation of sliding parts as they resist high temperatures and/or loads. A breakthrough of polyimide bulk materials in latest decennia is due to better control of the production processes on industrial scale [7]. Flexible shaping allows, e.g., for flanged bearing designs carrying a combination of radial and axial loads. They serve nowadays in niche applications as sealants for textile, food, pharmaceutical, glass and metal manufacturing, electronic

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industries, or as bearings, gears, rollers under severe sliding conditions in aerospace, chemical or nuclear industry.

Conventional polyimide resins undergo chemical cross-linking reactions during processing, resulting in a network structure that cannot be further reformed. For producing polymeric bearings, injection moulding or extrusion cannot be used as polyimides are insoluble and infusible with consequently inferior mouldability. A sintering process can be used, while it remains difficult to obtain complex shapes: products should either be directly formed in the sintering mould or more complex shapes should be machined from a pre-form, inducing high costs and material loss. Similar techniques were used for other polymers with high melt viscosity such as ultra-high molecular weight polyethylene [8]. For melt processing, polyimides are needed with well-defined glass transition or melting temperature and appropriate viscosity [9,10] or better solubility [11]. By varying the monomer composition with e.g. trifluoromethyl [12], an extremely broad range of mechanical or chemical properties and applications can be covered while retaining the thermal stability of the imide structure. Recent investigations focused on the synthesis of melt-processable polyimides, either as condensation-type [13], addition-type [14,15] or hybride-type [16] polymers. Common methods to control the molecular weight introduce endcapping reagent [17]. Kuroki et al. [18] investigated the effect of crosslinkable agent on processability and physical properties. Copolymerisation of imide-structures with ester [19], ether [20], amide [21] or siloxane groups [22] results in good processing, but somewhat lower mechanical properties. They are used as insulating material rather than mechanical performance materials. Polyetherimides are amorphous with a glass transition temperature of 215 °C and have lower thermal stability. Aromatic polyamide-imides are synthesized in a solution of aromatic diamine and tricarboxylic acid anhydride, reacting into a polyamic acid and subsequent imidisation, but they are also amorphous. For sliding applications, polyetherimide and polyamide-imides are mainly used as matrix for reinforced composites [23,24]. Thermoplastic polyimides are semi-crystalline, while the level of crystallinity should be carefully controlled to avoid excessive brittleness and stresses in finished products. Crystallisation is slow and a moulding cycle of 30–60 s produces low crystallinity [25,26]. The extruded product should retain its amorphous condition dur-

ing processing by quenching. An appropriate degree of crystallization is induced by a 10 h annealing process at 260 °C.

Although flexible shapes can industrially be obtained with various processing techniques, the differences in sliding behaviour of sintered and moulded polyimides should be clearly understood. The tribological performance of those materials was previously investigated under mild mechanical conditions and high temperatures [27]. However, due to their strength and thermal stability they are also expected to function under extremely high contact pressures and/or sliding velocities (*pv-value*). Then, the dimensional stability becomes important and failure mechanisms under overload conditions are not well reported in literature. It will be illustrated that the processing conditions strongly influence friction and wear properties of polyimides, controlled by the formation of a polymer transfer film under mild loads and brittleness or plastification at high loads.

2. Experimental

2.1. Test materials

Sintered polyimide (Fig. 1a) is synthesized from a two step method. First, soluble polyamic acid (PAA) is prepared from pyromellitic dianhydride (PMDA) and 4,4'-diamino-diphenyl ether or oxydianiline (ODA). The reaction happens at room temperature in a dry polar aprotic solvent. The reaction mechanism involves the nucleophilic attack of the amino group and the carbonyl carbon of the anhydride, followed by the opening of the anhydride ring. Details on the effects of reactivity of both the diamine and dianhydride parts are given by Dunson [28]. The effect of the solvent on curing of polyimide resins has been investigated by Hsu and Liu [29], because residual solvent causes internal stresses [30]. Second, dehydration is done by *thermal cyclisation*: a typical multi-stage heating schedule includes a 1-h resident time at 100 °C, followed by a relatively rapid temperature rise to 200 and 300 °C, each kept constant for 1 h. Palmese and Gillham demonstrated that a step-wise imidisation scheme aids in the formation of linear species [31]. Further heating at 300 °C or higher does not result in complete conversion because of the so-called 'kinetic interruption' effect [32]. The hydrolytically unstable residual amic acid units occur as defect sizes. Reported side reactions contain a reversible decrease in molecular weight in the early

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