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Low friction poly(amide-imide) coatings with silicones as tethered liquids

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ABSTRACT

In the production of enamelled copper wires, coated with the high-performance polymer PAI (poly(amideimide)), friction between multiple polymer surfaces as well as between the polymer and the slot substrate are significant issues. These effects drastically reduce the achievable packing density of the wires, which directly correlates to efficiency of the products. Conventional methods of lubrication involve use of leeching lubricants or addition of wax layers, which in return produces other problems. Moreover, the most potent lubricant PDMS cannot be used due to incompatibilities with the PAI matrix.

A desirable solution for these issues is the covalent attachment of lubricating substances, ideally PDMS, to the PAI matrix. To achieve this, block copolymers from PAI and PDMS of different shapes and block length distributions were synthesized and cured as coatings. It was found that, depending on the specific type of block copolymer, friction can be drastically reduced by up to one order of magnitude. This effect was observed in lab scale experiments as well as in industrially produced enamelled copper wires.

AFM investigations furthermore revealed a surface structure with distinct PDMS domains, which show liquidlike behavior as well as strong covalent binding to the matrix. Several parameters of the domains can be finetuned by variation of block copolymer parameters.

1. Introduction

Friction behavior and lubrication of polymeric surfaces are highly relevant aspects in coatings technology, as this is necessary to understand and consequently control wear and sliding resistance. A wide range of slip additives are used as state of the art, to minimize friction and consequently wear. Typically, liquid lubricants like poly(dimethyl siloxane) (PDMS) and poly(dimethyl siloxane)-b-polyether block copolymers are used. Another option is usage of solid additives like polytetrafluoroethylene (PTFE), molybdenum disulfide and graphite.

Despite their use for decades, underlying fundamental principles for thin film polymeric lubricating layers are scarcely understood. The most common lubrication mechanisms, used to explain friction phenomena of lubricated surfaces, are hydrodynamic, elasto-hydrodynamic, and boundary lubrication. Frictional resistance is typically increasing from hydrodynamic to boundary lubrication, while lubricating layer thickness is decreasing (Fig. 1) [1].

For hydrodynamic lubrication the lubricant film thickness has to be up to 100 times thicker than the height of surface asperities. Here, friction characteristics are dominated by the bulk viscosity of the lubricant [2,3]. Typical coefficients of friction (CoF) are 0.001 to 0.01 [2]. In case of elasto-hydrodynamic lubrication, the lubricant film

thickness is about 3-10 times thicker than the height of surface asperities. The solid substrates are elastically deformed preventing direct asperity contact. The length of the contact region is often more than 1000 times higher than the lubricating elasto-hydrodynamic film. The viscosity increases dramatically to even solid like consistency, due to the Hertzian pressure in micro contacts and the lubricant cannot be squeezed out [4]. Typical CoF are 0.01 to 0.1 [2]. For both, hydrodynamic and elasto-hydrodynamic lubrication, friction increases with increasing thickness of the lubricating layer [2,4]. Boundary lubrication is a typical behavior of monomolecular or polymolecular physical or chemical adsorption layers. The layer thickness is typically much lower than the asperity thickness. Thus, there are considerable asperity interactions. Friction is dominated by shear strength of the lubricating layer or the bulk material in case of welded asperity junctions [2–5]. Typical CoF are 0.03 to 0.2 [2]. In contrast to the other types of lubrication, friction decreases with increasing layer thickness [2,4].

One application with especially high demand for minimized friction are enamelled metallic wires, coated with the high-performance polymer poly(amide-imide) (PAI), for use in transformers, generators, or electrical motors [6]. Those copper or aluminum wires are mostly coated with a PAI multilayer of $1-3 \,\mu\text{m}$ each and $10-20 \,\mu\text{m}$ overall as insulating and protective coating. As the efficiency for electrical motors

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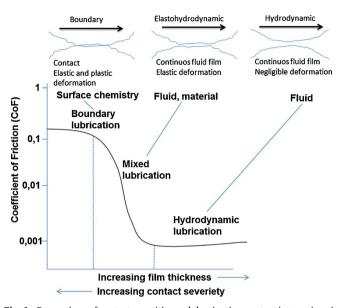


Fig. 1. Comparison of contact severities and dominating contact interactions in the different contact regimes (after Hsu and Gates [1]).

made of enamelled copper wire depends on the wire packing density, the coating has to be as thin as possible [6]. More importantly though, the maximum achievable packing density depends on the surface friction of the coating. If it is too high, optimum wire coil packaging is prevented. Relevant friction aspects are coating against itself and coating against the slot substrate, most often steel or polymer substrate.

As state of the art, PAI magnet wires are mostly post treated with solvent based wax solutions or wax saturated strings [7]. Another approach is the use of self-lubricating coatings, accessible via incorporation of internal lubricants into the outmost PAI layer [6]. The lubricant separates towards the surface during the curing process. One prerequisite for the lubricant is resistance against the harsh curing conditions (≥ 250 °C for several seconds). Linear poly(dimethyl siloxane) (PDMS) would be an ideal candidate, as it exhibits excellent lubrication characteristics. However, it cannot be utilized within PAI wire enamels in a free form, mainly due to incompatibility and hereof resulting problematic migration characteristics.

To overcome these issues, several approaches have been pursued. For example, PDMS brush model surfaces consisting of covalently bound, linear PDMS have been synthesized, and showed excellent reduction of friction, due to a liquid-like behavior of the PDMS chains [8–11]. This "tethered liquid" behavior was described by (elasto)hydrodynamic lubrication [2,3]. Additionally, PEG-*b*-MDI-*b*-PDMS multiblock copolymers were synthesized. Here, microphase separation resulting in isle-like structures on a length scale of 6–12 nm and wormlike network structure of PDMS phases were observed. It was shown that these separate phases consist of pure PDMS blocks [12].

In this work we present a novel concept of "tethered liquids" via the synthesis of PAI-b-PDMS block copolymers and their application as selflubricating coatings. The effect on the resulting surface structure, due to segregation of PDMS rich phases at the surface of the coating layer are shown. The resulting self-lubricating coatings are characterized in lab scale experiments as well as under technical conditions, applied on enamelled copper wire. Friction characteristics are discussed and compared to literature models of liquid respectively boundary lubrication.

The advantage of this concept is that both relevant components, i.e. PAI and PDMS are bound together without the necessity of another component. Thus, compatibility and adhesion to the main PAI matrix system is easily obtained, and the known issues of free PDMS are circumvented. Additionally, the PDMS is covalently attached to the matrix, preventing leeching and depletion of the lubricant.

2. Material and methods

2.1. Materials

Hydroxyalkyl-terminated PDMS macromers were provided by Gelest. 4,4'-methylenbis(phenyl isocyanate) (MDI), trimellitic anhydride (TMA), n-methyl-pyrrolidone (NMP) and xylene isomere mixture (xylene) are provided by VWR.

2.2. Block copolymer synthesis

The reaction was carried out in a 1 L three neck glass flask with reflux condenser and stirrer, under nitrogen atmosphere. Polymerization temperature was controlled by an oil bath. Xylene and MDI were first charged at 45 °C and hydroxyl-terminated PDMS was added dropwise under stirring into the high excess MDI solution. Reaction temperature was raised to 90 °C for 1 h and then lowered to 45 °C again. After adding NMP completely and TMA in portions, the reaction temperature was held at 50 °C for again 1 h, then slowly raised to 120 °C within 3.5 h. The temperature remained at 120 °C until the viscosity raised above 5 Pa*s to complete the block copolymer synthesis. Reaction progress was monitored via rheology (Rheologica StressTech) and ATR-FTIR (Bruker Alpha). The resulting colloidal polymer solution was diluted with NMP to 30% solid content to get the self-lubricating PAI coating.

2.3. Coating sample preparation

Self-lubricating PAI coatings were applied on ethanol rinsed glass plates and aluminum foil by a 50 μm doctor blade and cured for 10 min at 250 °C. The thickness of the dried layer was 7 \pm 2 μm . Coated samples were rinsed with ethanol to ensure covalent binding of the PDMS macromere.

2.4. Copper wire production

Self-lubricating PAI coatings were tested as topcoat on 0.53 mm commercial polyesterimid (PEI) basecoat, commercial PAI topcoat copper wire. Application conditions were 605 °C oven temperature and 2 m/s production speed at a 5 m oven line. The CoF for steel wire friction was tested according to DIN EN 60851 [13].

2.5. Lab scale friction analytics

Lab scale friction analytics were performed according to DIN EN ISO 8295 [14] using coated glass plates and steel respectively as underlying substrate and coated aluminum foil placed under a 40 cm² 200 g sliding sledge. The friction measurements were performed with a Thwing-Albert-FP-2250 linear Tribometer at 100 cm/min and 8 cm sliding length.

2.6. Atomic force microscopy (AFM)

AFM imaging was performed on Bruker Dimension[®] Icon using normal TappingMode (FESP silicon probe) and the quantitative nanomechanical tapping mode HarmonixMode (HMX silicon probe). AFM samples were prepared by 10×10 mm coated aluminum foil on microscope slides.

2.7. Static contact angle measurements

Static contact angle measurements were conducted using the sessile drop technique and a Krüss G10 goniometer coupled with DSA 1.10 data analysis software. Drops of deionized water (10 μ l) were placed onto the flat coated glass plate and an image of the drop was captured. An average of 5 measurements was taken and all the values were within $\pm 2^{\circ}$.

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