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Correlation of crystallization behavior and mechanical properties of thermal sprayed PEEK coating

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Abstract

An amorphous PEEK coating was prepared on an Al substrate by a flame spraying process. The amorphous coating was subjected to an annealing treatment under an annealing temperature from 180 to 300 °C and a holding time from 1 to 30 min. The cold crystallization behavior of the as-sprayed coating differential scanning calorimetry (DSC) and wide angle X-ray diffraction (WAXD) measurements. The hardness and tribological behavior of the coatings were investigated. Both DSC and WAXD analysis revealed that the annealed coatings exhibited a semi-crystalline structure. Coexistence of double crystal entities in annealed coatings was deduced. The annealed coatings exhibit higher hardness, lower friction coefficient and wear rate. Both the annealing temperature and holding time can benefit the coating hardness. The annealing condition in the studied range has little influence on the tribological behavior of the coatings. The variances of the coating mechanical properties were correlated with the modifications of the coating structures induced by the annealing treatments.

Keywords: PEEK coating; Flame spray; Annealing; Crystallization; Hardness; Tribological behavior

1. Introduction

An increasing number of products, manufactured from aluminium, are coated with a layer of polymer film. This film protects the metal surface from corrosion, prevents excessive wear and serves decorative purposes [1]. PEEK (poly–ether–ether–ketone) has become one of the most attractive polymer materials and been more and more used in industry due to its excellent thermal stability, friction reduction and wear resistance [2–4]. In order to meet the demand of engineering and design driven by ecological and economical reasons, some researches recently contributed to the preparation of PEEK coating, using thermal spraying technique, on lightweight metallic substrates for friction reduction and anti-wear applications [5,6]. In such cases, PEEK powders were injected into a flame or a plasma jet where they were melted and propelled towards the substrate to produce a coating. The feasibility of coating deposition with different spraying processes and the coating microstructure were especially investigated in the previous studies [7–9].

In this work, a homogeneous amorphous PEEK coating was deposited on an Al substrate using flame spraying. The as-sprayed coating was isothermally treated under different temperatures and durations. The objective of this work was to correlate the coating crystallization behavior caused by annealing treatment with coating mechanical properties. Differential scanning calorimetry (DSC) and wide angle X-ray diffraction (WAXD) analyses were carried out to characterize the coating crystallization behavior. Coating microhardness and tribological behavior were also investigated.

2. Experimental details

2.1. Coating procedure

PEEK coatings were sprayed using a flame gun (Eutectic-Castolin Castodyn 8000 powder flame spray gun) mounted on a robot (ABB 4400). The feedstock was the commercially

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available Vitrex PEEK powder (Vitrex sale Ltd. Lancashire, UK.) having a mean diameter of 25 μm. It has a glass transition temperature (T_g) of 143 °C and a melting temperature (T_m) of 343 °C. Two types of samples were prepared in this work. One was for WAXD and DSC analysis, and also microhardness measurements (referenced as sample *T*). The substrates were Al plates with dimensions of 120×30×1.5 mm. The second type was for friction test (referenced as sample F). The Al discs of 65 mm in diameter and 5 mm in thickness were used as substrates.

The substrates were preheated up to 400 °C using the flame torch. The substrate temperature was measured by a thermocouple fixed behind the specimens. During spraying, the specimens were fixed in front of the torch that was moved with a linear velocity of 150 mm/s. After spraying, the specimens were immediately quenched into the iced water. The thicknesses of the two types of coatings were approximately 300 and 100 μm, respectively. From the comparison of IR spectra of the feedstock powder with the flame-sprayed coating, only very little chemical modification is detected after the spraying process [5].

2.2. Isothermal treatment

The as-sprayed coatings were referenced as T1 and F1, respectively. The samples prepared for DSC and WAXD measurements, and friction test as well were all subjected to the same annealing treatment. Table 1 lists the annealing conditions. After the annealing treatment, the samples were then quickly immersed into the iced water. The corresponding samples were referenced as T2 to T7 and F2 to F7.

2.3. DSC measurement

The measurement was carried out in a TA Instruments 2010 DSC (Newscastle, DE) with an inert nitrogen atmosphere purging the sample cell. The sample size was around 5 mg. The sample was loaded into the DSC cell at room temperature after being sealed in an aluminium pan. It was firstly equilibrated at 20 °C and then heated to 380 °C at a scanning rate of 10 °C/min.

2.4. WAXD measurement

WAXD was performed to examine the possible differences in the crystal perfection of the as-sprayed and treated coatings. WAXD was carried out in a Simens D5000 automatic diffractometer with a Cobalt anticathode and a wavelength of 1.79 Å. The 2θ angle region ranged from 10° to 40° with a scanning rate of 5°/min.

Table 1
Annealing conditions of samples T2–T7 and F2–F7

Sample code	Annealing temperature (°C)	Annealing time (min)
T2, F2	180	30
T3, F3	220	30
T4, F4	260	30
T5, F5	300	30
T6, F6	260	1
T7, F7	260	10

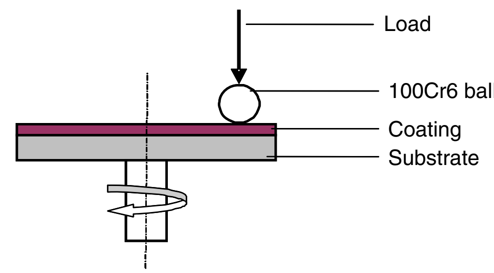

Fig. 1. Schematic of ball-on-disc tribometer.

2.5. Microhardness test

Vickers hardness measurement was performed on the as-polished cross-sections using a LEICA VMHT 30A automatic microhardness tester with a load of 1 N and a dwell time of 15 s.

2.6. Friction test

Friction test was performed with a ball-on-disc arrangement. The schematic of the tribometer is shown in Fig. 1. The wear track diameter was 3 cm. The counterbody was a 12.5 mm diameter 100Cr6 steel ball with a mirror finished surface (R_a: 0.02 μm). The hardness of the 100Cr6 ball was 62 HRC. The friction force was measured with a sensor and dynamically recorded into a computer. The applied load and sliding velocity were 15 N and 0.2 m/s, respectively. The test was carried out under an ambient temperature of ~20 °C and a humidity of ~70%.

The wear rate was defined as the worn volume per unit of applied load and sliding distance. The cross-section of the worn tracks was obtained with a Taylor-Hobson Surtronic 3P profilometer (Rank Tayloe Hobson Ltd., UK) after 500 m relative sliding. The cross-section area of the wear tracks timing the sliding distance permitted to obtain the total worn volume.

The worn surfaces of the coatings were analyzed with scanning electron microscopy (SEM) (JSW-5800LV, JOEL) after gold sputtering.

3. Results and discussion

3.1. DSC results

Fig. 2a shows the DSC scanning trace corresponding to the as-sprayed coating (T1). A glass transition is apparently at about 143 °C which is followed by a marked crystallization exothermic peak. Both features imply an amorphous structure of the as-sprayed coating. Fig. 2b and c show the DSC thermograms of T2–T5 and T6–T7 samples, respectively. The annealed coatings do not exhibit the crystallization exothermic peak any more above T_g. The absence of exothermic peak indicates that no further crystallization occurred during DSC