Structural anisotropies of PEEK foils revealed by optical dichroism and X-ray scattering methods

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INTRODUCTION: During the last three decades, poly-aryletherketones and especially poly-etheretherketone (PEEK) have been increasingly employed as biomaterials for trauma, orthopaedic, and spinal implants.[1] PEEK is biocompatible, inert and exerts excellent mechanical properties compared to other polymer materials. As its chemical nature suggests structural anisotropy, we investigated PEEK foils using optical and X-ray scattering methods.

METHODS: Commercially available amorphous and semi-crystalline APTIV™ PEEK foils (Series 2000 and 1000, Victrex Europa GmbH, Hofheim, Germany) were marked for direction of rolling. Hot embossing was done with a HEX03 press (JENOPTIK Mikrotechnik GmbH, Germany) at 160 °C and 100 kN. For the optical transmission measurements, the foils were mounted on a 360° rotation table and measured at different wave-lengths with a spectrophotometer (Perkin Elmer, Germany). The small- and wide-angle X-ray scattering (SAXS/WAXS) data were recorded at the cSAXS beamline of the Swiss Light Source (Paul Scherrer Institute, Villigen, Switzerland) in scanning setup.[2] Foils were mounted on an aluminium holder for line scan acquisition.

RESULTS: Absorbance scans parallel and perpendicular to the direction of rolling reveal that semi-crystalline and hot-embossed amorphous PEEK foils are anisotropic contrary to amorphous untreated foils. Around 540 nm, the anisotropy reaches a maximum. Measuring the transmission at 540 nm as a function of rotation angle results in a sine with 3.9% amplitude, suggesting a linear orientation in the foil. The transmission is minimal in rolling direction.

SAXS of the embossed 50 µm-thin PEEK foils also shows the anisotropy revealed in the optical measurements (see Fig. 1). The spot intensity increases with foil thickness from 12 µm via 25 µm to 50 µm. While the semi-crystalline foils also exhibit the anisotropy, the amorphous untreated foils are isotropic. The anisotropy detected in SAXS corresponds to a feature size of about 140 Å. Such a long-range order was already described.[3]

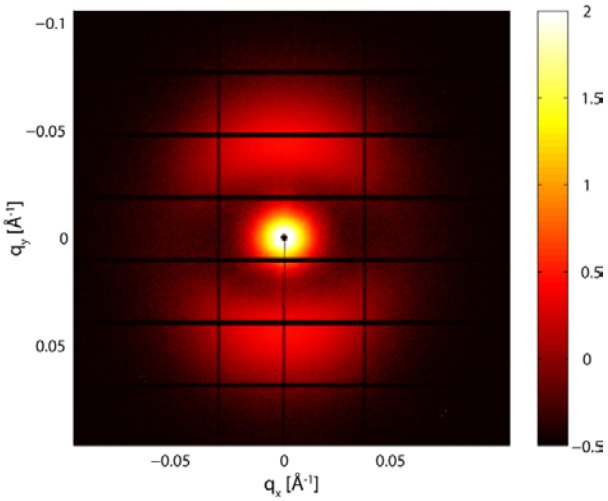

Fig. 1: SAXS pattern of 50 µm-thin embossed PEEK foil, acquired with 11.2 keV photons using 1 s exposure time, averaged over 60 frames.

The unit cell of solution crystallized PEEK is described as orthorhombic with a = 7.75 Å, b = 5.89 Å, and c = 9.88 Å.[4] The WAXS measurements exhibited peaks at q-values of 0.79 Å^{-1} and 1.06 Å^{-1}, corresponding to 7.9 Å and 5.9 Å, respectively, which is in reasonable agreement with the a- and b-values.

DISCUSSION & CONCLUSIONS: Hot embossing of amorphous PEEK foils above glass transition temperature results in anisotropic properties in rolling direction. The direction and the extent of anisotropy in crystalline PEEK foils can be quantified by rather simple optical transmission measurements. SAXS scans identify the equivalent orientation with nanostructures of approximately 14 nm-long units. The data should allow building a structural model to describe this long-range order in PEEK foils.

REFERENCES: [1]S.M. Kurtz and J.N. Devine (2007) *Biomaterials* **28**:4845-69. [2]O. Bunk et al (2009*) New J Phys* **11**:123016. [3]D.J Blundell and B.N. Osborn (1983) *Polymer* **24**: 953-958. [4]A.J. Lovinger and D.D. Davis (1986), *Macromolecules* **19**:1861-1867.