Characterisation of the fracture behaviour and determination of micro-hardness of medical grade ultra high molecular weight polyethylene (PE-UHMW)

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ABSTRACT

Changes in the crystalline and amorphous regions of implants made of medical grade ultra high molecular weight polyethylene (PE-UHMW) occur during manufacturing, radiation and in vivo use. In this study correlations between molecular and morphological structure and their impact on the mechanical properties are investigated.

INTRODUCTION, EXPERIMENTAL, RESULTS & DISCUSSION

The overall mechanical behaviour of implants made of medical grade ultra high molecular weight polyethylene (PE-UHMW) depends basically on the conditions during consolidation and irradiation procedure. Beside the competition between cross-linking and chain scission during irradiation, the type of post-irradiation thermal treatment has also an influence on the mechanical behaviour of so-called cross-linked materials.

In a first step this PE-UHMW is irradiated in order to produce radicals, which build up cross-links (in absence of oxygen). Afterwards the material is melted or annealed and residual radicals recombine. This treatment enhances the oxidation stability of the implant during shelf aging and in vivo service [1].

The processing conditions associated with the ram extrusion or compression moulding of the resin can alter the polymer morphology or the degree of lamellae stacking or the texture. This can result in substantial differences in the fatigue behaviour [2]. Further, the resulting mechanical properties are affected by the cross-linking that occurs primarily in the amorphous regions. Cross-linking impedes the relative motion of polymer chains, leading to reduced ductility and fracture resistance. The influence of crystallinity on fracture in polyethylene materials is discussed by Brown [3], controversially it is reported, that the fracture toughness of PE-UHMW materials might be expected to decrease with an increase in crosslink density and to increase with an increase in percent crystallinity [4]. However, it is well known that, increasing crystallinity leads to an increase in micro-hardness.

Linear elastic fracture mechanics (LEFM) has been used as a successful tool to describe the fracture properties of brittle polymers. Its applicability is well established for different strain rates (static, quasi-static, dynamic, impact). However, many polymers exist in which significant plastic yielding occurs at the crack tip prior to the onset of crack extension (especially under quasi-static loading and room temperature). Such polymers are for example high impact modified systems (HIPS) and semi-crystalline polymers like PE. With these materials the LEFM concept loses its validity, and the use of a fracture mechanic concept considering the mentioned behaviour is necessary. Among the post yield fracture mechanics concepts the J-integral approach is widely used for materials which exhibit tough fracture behaviour. Typically, in these materials the fracture process is characterized by four phases: crack tip blunting, crack initiation, stable crack growth, and unstable crack growth. The total process can be described by means of the crack resistance curve (R-curve) [5]. The specimen geometry affects the resistance against stable and unstable crack propagation.

Instrumented indentation testing is a powerful set of tools for investigating mechanical properties of materials in small dimensions. In such a test, a hard tip, typically a diamond is pressed into the sample with a known load. The indentation modulus and the hardness of the test material can be calculated according to [6].

The goals of the present study were:

1. to choose an appropriate fracture mechanics concept to describe the fracture behaviour of PE-UHMW (under quasi-static loading and room temperature)
2. to investigate the geometry dependence (different geometries and crack lengths) of the J-integral respective J-R-curve
3. to use the gained knowledge for extending the investigations on an “arc-specimen” geometry, which enables to take samples from acetabular cups
4. to gather data from PE-UHMW in different conditions in order to identify the parameters having an influence on the mechanical fracture behaviour

The different materials investigated in this study were supplied as processed rod stock. They were machined into notched three-point-bending specimens with different geometries. Also “arc-specimens” from cups were machined. Side groves were machined and the specimens were pre-cracked with a razor blade. All tests are performed with a Zwick Z50 materials testing machine at room temperature under climate conditions. The stress intensity factor was investigated at the beginning of the experimental series. The investigations on the J-Integral followed ASTM D6068-96 [7]. The specimens were tested under quasi-static load and three-point-bending modus with defined conditions (temperature, test velocity was adjusted to obtain a constant strain rate of the outer fibre). The resulting stress-strain curve was recorded and the corresponding crack lengths were monitored optically. In the following the J-R-curve is calculated. After the tests, the samples were broken using liquid nitrogen, and the initial crack length and the fracture shape are evaluated from the fracture surface.

On the basis of results obtained from experiments performed currently the influence of the consolidation, sterilisation and cross-linking procedure on the fracture behaviour will be discussed.

The fracture behaviour of PE-UHMW is primary influenced by the amorphous part of the polymer. To optimize the mechanical properties of implant parts, it is also necessary to gain knowledge about the effects of the semi-crystalline morphology, this can be determined by means of Differential Scanning Calorimetry (DSC). One property which is very sensitive to changes of crystallinity is hardness. By means of micro-hardness experiments using e.g., indentation measurements, the micro-hardness is correlated to crystallinity calculated from the DSC.

For these measurements the specimens were embedded in an Araldite resin, water-cooled grinded and polished applying only very low pressure. Indentation measurements with a Nano Indenter XP were carried out depth controlled at a rate of 250 nm/s up to a maximum depth of 5 μm, then hold at maximum load for 30 s followed by unloading. The holding time of 30 s minimizes the effect of creep on the unloading curve.

The micro-hardness results obtained in these investigations for different sterilisation procedures can be correlated with DSC measurements and considerations of the crystallinity changes. The lower hardness of the “cross-linked” sample (Fig. 1) can be explained by the thermal after-treatment (remelting) reduced crystallinity. Changes in the crystallinity correspond to variations in the micro-hardness. We hypothesize that changes in the fracture behaviour are likely to occur. To complete this hypothesis it is also of importance to receive explants of which specimens for a future investigation can be extracted.

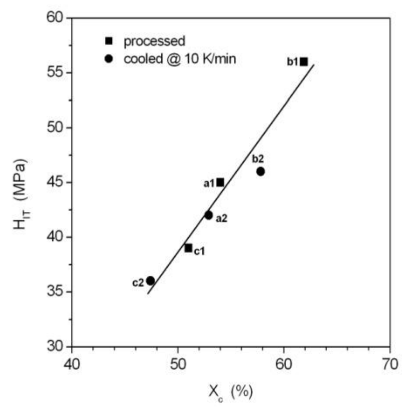

Fig. 1. Hardness in dependence of the degree of crystallinity; a1 and a2 not sterilized, b1 and b2 gamma sterilized, c1 and c2 cross-linked

CONCLUSIONS

On the basis of results obtained so far we conclude that LEFM concept can not be fulfilled under quasi-static loading for the material UHMW-PE neither by varying the notch length nor by different geometry conditions. The J-Integral investigations showed a geometry dependence of the specimens in the results. In order to be able to perform measurements on implant parts a new geometry (arc specimen) is proposed. A linear dependence of micro-hardness on the degree of crystallinity is found. Correlations of micro-hardness results to changes in the fracture behaviour should emerge.

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