Femtosecond laser machining for the characterisation of dose dependent mechanical properties of polymer foils

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In order to directly measure the directional dependency or degradation of mechanical properties of material samples with limited dimensions it can be necessary to produce very small specimens. Metallic structures can readily be milled by e.g. Ga- or Xe+ ion beams with the currently well-established focused ion beam technique, however the method is rather time consuming for structures with sizes in the order of magnitude of a few tens or even hundreds of micrometres and involves the risk of implanting the respective sputtering gas and damaging the specimen. In contrast, ultrashort laser ablation provides material removal rates orders of magnitude higher, with ideally negligible damage to the surrounding material for virtually any type of material. In this work, a femtosecond laser was used to prepare tensile specimens of VDA coated 25 µm Upilex-S® and 127 µm FEP foils to study the electron irradiation dose dependency of their tensile properties. The feasibility of machining samples consistently with an overall length of about 1.1 mm and a gage width of about 25 µm was shown. Stress-strain curves of the tested specimens were found to be very well agreeing with the values specified by the manufacturer and till the chosen electron irradiation dosage of up to 1 MGy no major change could be detected in the ultimate tensile strength.

For the machining of the tensile specimens the focal plane of the laser beam has been determined by selecting the f-theta objective lens to focus the laser beam for a flat image plane and to ensure an approximately normal incidence of the laser beam onto the sample surface. In order to ensure an approximately normal incidence of the laser beam the sample has been placed in the middle of the scan field. The cutting of the tensile geometry a material sample of roughly 6 x 6 mm² has been fixed into a clamp, which afterwards was used for mounting in the tensile testing device, too. First a rectangular shape is cut, afterwards the dog bone shape is fabricated (see Fig. 1b), where the width of the gage length was adjusted such that it would match the foil material’s thickness. The samples are placed in a distance of 1.4 mm to each other to ensure enough space for the testing gripper. This enables to test a maximum of 4 samples on each mount.

Tensile experiments have been performed on a Kammrath & Weiss fibre tensile module. This device enables to measure forces up to about 2N with a resolution of approximately 10 µN. The displacement is registered with a resolution of about 30nm. The experiments are also recorded using a camera connected to a stereo microscope, recording images every five seconds. Using a moveable stage the sample can be properly aligned with the griper; a 100 µm thick tungsten foil with the negative shape embossed in the head of the testing machine. The experiments were performed under displacement control while the forces were recorded. The testing speed was either 1 or 2 µm/s resulting in strain rates of about 0.3 or 0.6 %/s. The experiments have been conducted at room temperature (22°C) and each sample has been elongated until failure.

The tensile properties of non irradiated materials show a large difference in both, the maximum strength and the maximum elongation (see Fig. 3). Upilex-S VDA shows a high ultimate tensile strength, whereas the FEP VDA foils exhibit a high tensile strain. The resulting stress-strain curves of both materials are comparable to results of macroscopic experiments, although the match is not perfect due to the influences of the laser machining. As mentioned in the introduction, especially with polymers the laser pulse repetition rate needs to be chosen carefully. The excessive energy of each laser pulse, which is not used to ablate material, introduces a large amount of heat. If the repetition rate is too high, the heat accumulation effects can lead to a local melting of the processed materials. Especially for materials with a low heat conduction this is critical. Further, also the ambient atmosphere plays an important role during processing. Using atmospheric conditions the high energy density in the focal spot leads to the formation of a plasma. The plasma intensity increases with increasing laser fluence, finally leading to lower ultimate strain and ultimate strength values for FEP VDA samples (see Fig. 3). This degradation in tensile properties has not been found for the Upilex-S VDA. The samples show a reduction of the ultimate tensile strength of about 40% for electron irradiation doses of 250 kGy. The FEP VDA samples do not show a dose dependency of the mechanical properties for Upilex-S and still match with the values specified by the manufacturer (520 Mpa and 42% at 25°C). In contrast, FEP VDA foils are strongly affected by the electron irradiation. Foils exposed to a higher dose than 250 kGy actually embrittle so much, that the foils fractured due to handling. In case of the pristine samples the ultimate tensile strain was in the range of about 320 to 360 %, whereas the radiation dose of 250 kGy reduced this value to about 20 to 90 %. Furthermore an increase in the variation of the yield strength is observed. The influence of the ambient processing conditions on non-irradiated FEP VDA samples (as shown in Fig. 3) has not been found in irradiated FEP VDA samples. In this case the influence of the electron irradiation dominates the decrease of the ultimate tensile strength and strain.

The evaluation of mechanical properties using femtosecond laser processed micro-tensile samples for polymer foils shows a good agreement with macroscopic data found in literature. For the FEP VDA samples the ambient processing conditions play a critical role, due to the degradation of the material when a plasma is formed near the ablation site. Where the electron irradiation up to 1 Mgy did not influence the tensile properties of Upilex-S VDA foils, FEP VDA foils exhibited a factor of 4 - 5 in reduction of the ultimate tensile strength when irradiating to a dose of 250 kGy. Femtosecond laser processing allows a fast and reproducible fabrication of micro-samples up to multiple hundred micrometers, therefore enabling a high resolution in the investigation of local mechanical properties.