Optimized handling of microtome cuts in IR microscopy

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Abstract

IR microscopy provides spatially resolved chemical information on samples and is one of the easiest and most sensitive methods to obtain qualitative as well as quantitative sample information. Thus, IR microscopy is a popular tool to establish structure-property relationships. This technique was also applied in the present case, where the material composition of a polymer with regards to its mechanical properties should be understaff and optimized.

To perform IR transmission analysis microtome cuts are required in many cases. So usually, microtome cuts are prepared of the components to be analyzed, in the present case dog-bone test specimens of a polymer. However, the challenge here is to optimally mount these microtome samples under the microscope, as cracking and twisting during the mounting as well as shifting during the measurements must be avoided. To this end, in a cooperation between Freudenberg and S.T.Japan-Europe optimized clamps for the MicroVice Sample Holder of S.T.Japan were developed and validated during this project.

Keywords

Sample Preparation, FTIR Microscopy, Microtome, Material Development, Structure-Property Relationship, Crystallinity, Polymer

Introduction

To analyze mechanical characteristics of polymers routinely dog-bone test specimens are prepared and tested subsequently.

The batch of dog-bone, investigated in the present application note, revealed surprisingly different mechanical characteristics than expected. The goal was now to understand the reason for these mechanical differences and provide potential approaches to prevent their occurrence.

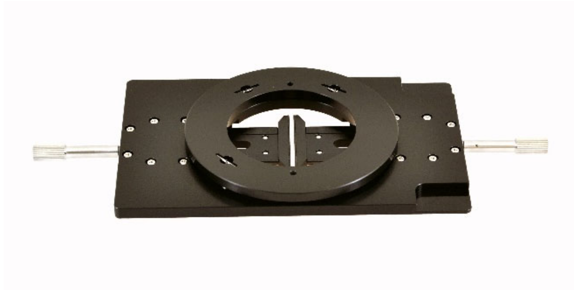
Mechanical characteristics of the test specimens do not only depend on the material composition, but also on other molecular properties like crystallinity (structure-property relationship). Crystallinity and its spatial distribution in the test specimens are highly influenced by the processing conditions during the injection molding process (e.g. temperature profiles). IR microscopy allows for spatially resolved quantification of crystallinity in analyzed polymer sample.

To investigate crystallinity distribution in the sample in this project, IR line scans were performed along the cross-section of the test specimens. The challenge was to optimally mount the samples under the FTIR microscope to be able to perform linescans with best possible results. Therefore, the MicroVice sample holder with the newly developed clamps was applied.

Experimental Setup

Sample Holder MicroVice STJ-0116-A

MicroVice Holder Type A
Front View

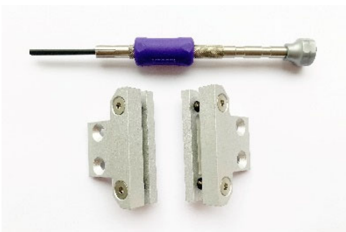


MicroVice Holyder Typ A
Back View

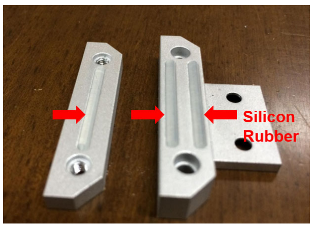
The sample holder was developed based on the MicroVice Holder Type A. To ensure a tight mounting without any cracking or twisting of the sample, new clamps were designed specialized for fixing films and fibers. The clamps are equipped by a set of cylindrical silicone rubber cords. These cords prevent the samples from slipping during the measurement and facilitate an easy assembly.

Furthermore, spring-loading of the clamps ensures a user-friendly and comfortable mounting of specimens, as an easy opening and closing of the clamps is guaranteed. This is especially beneficial if delicate specimens like single fibers or microtome cuts have to be handled. Thus, twisting of samples is avoided and a flat mounting between the clamps is ensured.

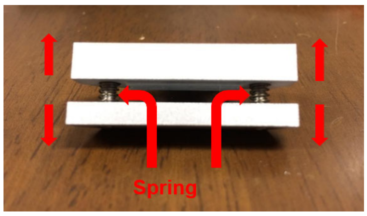
MicroVice Films & Fibers Clamps STJ-0116-A-N-NIC



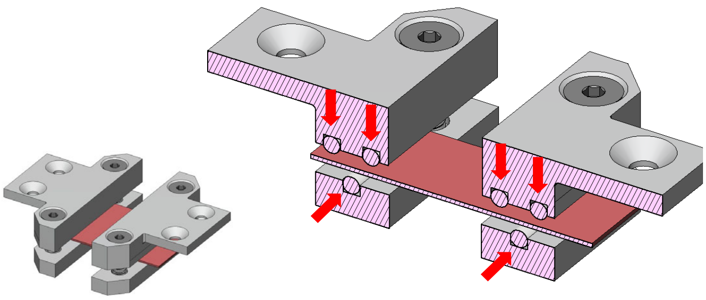
Films & Fibers Clamps

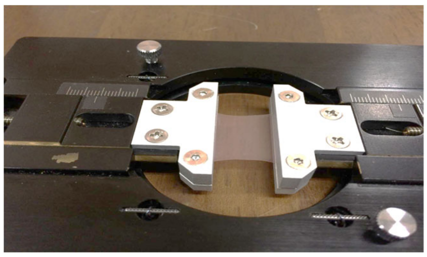


The upper and lower cylindrical silicone rubbers firmly grip the sample and fix it in place without damaging it.



Easy-to-use and highly effective springloaded screws for easy opening & closing of the clamps





The design of the clamps ensures that the sample can be fixed flat within the clamps and can be securely and evenly stretched without twisting.

FTIR Microscope:

IR measurements were performed with a Nicolet™ iN™10 IR Microscope from Thermo Scientific™ (Dreieich, Germany). For IR measurements dog-bone specimens were cut by a microtome. Subsequently, IR linescans were performed over the cross-section of the microtome cuts.

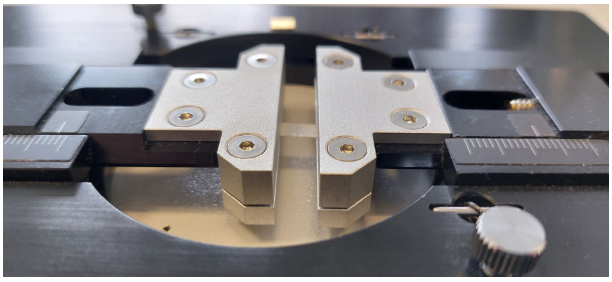
Implementation/ Execution

Step 1. Microtome cuts were prepared from the shoulder of the dog-bone test specimen.

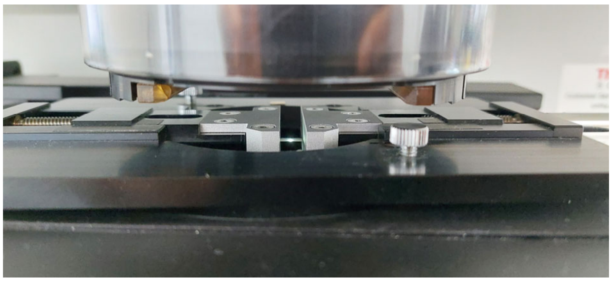
The preparation and handling of microtome cuts require a profound expertise, special patience and a steady hand.

Step 2. Mounting of microtome cuts in IR sample holder.

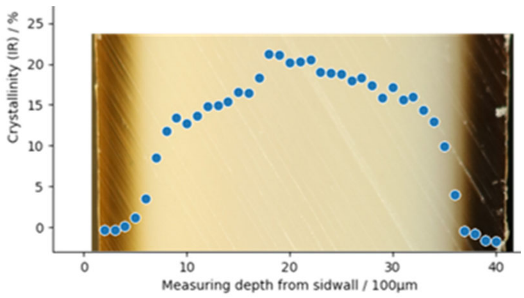
One of the most challenging steps is the fixing of the microtome cuts in a sample holder. The cuts must be mounted thoroughly and carefully to avoid shifting during measurements and also twisting & cracking of the cuts must be prevented. Making sure that the sample is mounted and remains flat during measurements is essential for best results.



Step 3. Setting the MicroVice Holder with the mounted sample onto the stage of the IR microscope



Step 4. IR line scans were performed along the cross-section of the microtome cuts



Step 5. Spectral analysis of the IR line scans was conducted to derive crystallinity profile. Spatially-resolved quantification of crystallinity via IR band evaluation

Results

The crystallinity profile reveals a plateau of higher crystallinity contents in interior part of the cross-section, while at exterior areas a significant drop of crystallinity is observed. This observation could be explained by to low temperatures in the molding tool:

During the injection process of the test specimens the polymer melt is quenched in areas in direct contacts with the molding tool (exterior areas of the later test specimen). As formation of crystallinity is known to be a comparably slow process, the quenching leads to low crystallinity contents.

In contrast to that, temperature hotspots remain in areas of the polymer melt, which are not in direct contact with the molding tool (interior areas of the later test specimens). Thus, the formation of crystallites is favored and the crystallinity is accordingly higher.

Crystallinity profiles as observed in the present case are usually not desirable, since an inhomogeneous distribution can significantly influence the determined mechanical characteristics of the test specimens.

Based on this finding it becomes clear that the processing conditions of the test specimens were not optimal in the present case leading to strong deviations in the mechanical properties.

Conclusions & Summary

The reason for the mechanical differences of the test specimens could be identified by the means of IR microscopy. The microctome cuts could be mounted optimally with the new optimized clamps. Then the MicroVice sample holder could be set directly on the stage of the FITR microscope and the cuts could be perfectly analyzed with FTIR mode-

The sample holder was optimized to enable mounting of microtome cuts and similar samples as follows:

Sealing cord enhances grip of the microtome cuts and thus prevents shifting.

Spring-loaded clamps facilitate the mounting of the microtome cuts

Microtome cuts can be handled more comfortable and precise.The new clamps guarantedd a flat mounting and prevent the twisting of the samples.

The S.T.Japan team in Japan and Germany cordially thanks Dr. Apel and Mr. Toussaint for this interesting and pleasant cooperation.

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