Chapter 3

Cavitation Behavior of Semi-Crystalline Polymers during Uniaxial Stretching Studied by Synchrotron Small-Angle X-Ray Scattering

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http://dx.doi.org/10.5772/intechopen.74224

Abstract

Cavitation appears in many semi-crystalline polymers when they are subjected to uniaxial stretching above their glass transition temperatures. Generally, the formation of voids is influenced by the morphology of semi-crystalline polymers, including their lamellae thickness, lamellae orientation, as well as the arrangement of the amorphous phase. Upon stretching, the size of the voids changes as a function of the local strain. Synchrotron small-angle X-ray scattering (SAXS) can be used as a powerful method to in-situ monitor the evolution of voids with high time and spatial resolution. In this chapter, recent reports about the cavitation behavior of semi-crystalline polymers studied by SAXS are reviewed. Afterwards, the theoretical background related to the SAXS technique is introduced. Lastly, some exemplary results about the cavitation behavior of microinjection-molded isotactic-polypropylene, studied by synchrotron SAXS measurements, are presented.

Keywords: cavitation, synchrotron, small-angle X-ray scattering, semi-crystalline polymer, stretching

1. Introduction

1.1. Cavitation behavior of semi-crystalline polymers

Cavitation behavior has been found in many semi-crystalline polymers, including isotactic-polypropylene (iPP) [1, 2], polyethylene (PE) [3, 4], poly(1-butene) (P1B) [5], and so on, when these polymers are stretched above their glass transition temperature. Stress whitening can be
regarded as the scattering of visible light by voids detected by naked eye. As the size of the voids exceeds 0.5 μm, most of the visible light shed on the sample will be scattered, transforming the sample from a transparent one to an opaque one. In the pioneering work of Peterlin [6] randomly distributed cracks were found in the neck region of PP specimen. In addition, the formation of small cracks was reported to arise earlier than macroscopic stress whitening [7, 8].

Cavitation behavior can be influenced by many factors, which can be divided into two groups. One of them is attributed to experimental factors such as stretching temperature and stretching speed. Generally, a lower stretching temperature or a larger stretching speed favors voids formation. Another group is attributed to the microstructure of polymers, for instance the crystal form, the thickness of lamellae, as well as the state of the amorphous phase. By polymer processing, various microstructures and morphologies can be created in the product depending on the individual processing conditions they used [9–11]. Therefore, understanding the influence of microstructure on the void formation will be helpful for the establishment of a structure-properties relationship.

1.2. Role of crystal form

iPP is a kind of polymorphic polymer owning four crystal forms: monoclinic α-iPP, hexagonal β-iPP, triclinic γ-iPP, and smectic form [12]. By applying shear flow or adding special nucleating agents, the crystal form of iPP could be adjusted. Aboulfaraj et al. [13] found that under tensile deformation, α-iPP spherulites exhibited a brittle failure. The cavitation appeared at boundaries of spherulites or at their equatorial regions perpendicular to the tensile direction. However, no cavitation could be observed in the sample comprising β-iPP spherulites. The β-iPP spherulites were deformed plastically up to large deformation. Chu [14] prepared iPP films containing more than 90% β-iPP. These samples crystallized under either isothermal or nonisothermal conditions. The porosity of the stretched films, which is caused by the existence of voids, increased with the drawing ratio. The voids observed by scanning electron microscopy (SEM) were elongated along the stretching direction and confined by the fibrillar structures. The formation of numerous voids was proposed to be caused by the volume contraction of the film. β-iPP belongs to a metastable phase, so a β-α phase transformation was induced during deformation. The density of α-iPP is higher than that of β-iPP. Therefore, a volume contraction can lead to a formation of voids in the sample. The more pronounced stress whitening behavior in β-iPP rich iPP samples was also confirmed by Pawlak [2].

1.3. Role of lamellae arrangement

In α-iPP, the lamellae are arranged in a unique “cross-hatched” structure, where daughter lamellae grow 80° inclined to the mother lamellae [15]. Nitta et al. [16] observed that cavitation appears earlier if there were more tangential lamellae in a single spherulite. Otherwise, Pawlak found that the reduction of tangential daughter lamellae would advance the formation of voids [17].
1.4. Role of crystallinity

By annealing, Na et al. prepared PP samples with crystallinity ranging from 48 to 56%. The cavitation behavior of the sample was investigated by measuring the volume increase. The results show that in annealed samples, the cavitation behavior was significantly intensified due to the increased stress concentration sites [18]. Boger et al. [19] examined the cavitation behavior of metallocene PP with crystallinity ranging from 0 to 62%. For the sample with crystallinity higher than 36%, the scattering signal originated from a fibrillary structure showed up on the SAXS pattern as the elongation ratio is larger than 3. In case of β-iPP, Bai et al. [20] found that annealing could advance the appearance of cavitation.

1.5. Role of the thickness of lamellae

Generally, thinner lamellae would prevent the formation of voids. The reason responsible for that was proposed by Seguela et al. [21]: a thinner lamella bears larger tie chain density, which transfers the load to lamellae in a better way and leads to the plastic deformation of lamellae instead to cavitation in the amorphous phase.

1.6. Role of lamellae orientation

The cavitation behavior of oriented β-iPP was investigated by Bao et al. [22]. The samples were cut from extruded sheets, and the deformation direction was parallel to the orientation of β-iPP lamellae. Their results proved that at temperatures lower than 110°C, the orientation of β-iPP remains almost unchanged during deformation, and void forms before fragmentation and reorientation of β-iPP. As the deformation temperature risen to 130 and 140°C, β-iPP reorients gradually upon stretching, and the size of voids decreases because at high stretching temperature less β crystal fragmentation takes place.

1.7. Role of the state of amorphous phase

Pawlak and Galeski [23] compared the cavitation behavior of PP with similar crystallinity and crystal thickness but different molecular masses of 400 and 250 kg/mol. They found that the samples having lower molecular weight showed stronger cavitation as a result of reduced number of entanglements in the amorphous phase. Rozanski and Galeski extracted the additives in the amorphous phase by critical CO2 and also by a mixture of nonsolvents. They found that purified PP exhibited more intense cavitation than pristine PP [24]. The intensified cavitation process in the purified samples was caused by the change in free volume by eliminating low molecular weight fractions and soluble additives in the amorphous phase, indicating that the nucleation of voids is not heterogeneous. In their later work, they proved that only partial filling of the free volume pores of the amorphous phase with low molecular weight modifier leads to a decrease of intensity or complete elimination of the cavitation phenomenon. [25]
2. Synchrotron X-ray scattering

2.1. X-ray and its sources

X-ray, a kind of electromagnetic radiation (see Figure 1), is also named as Röntgen radiation, after Wilhelm Röntgen, who discovered X-rays in 1895 [26]. Since that time, X-rays have been employed in the field of materials science as a nondestructive analytical technique. Traditionally, X-rays are produced by X-ray tubes. In X-ray tubes, the electrons emitted from cathode wire are accelerated by an electric voltage before they hit the target. The wavelength of X-rays produced by X-ray tubes depends on the target material. For instance, the characteristic wavelength of the X-ray produced is 1.54 Å by Cu target and 1.79 Å by Co target.

The main disadvantages of X-ray tubes are its low energy, broad focus (around $2 \times 12$ mm), and long exposure time (around 60 s) [27]. In the mid-1970s, the limitation of X-ray tube was overcome by the establishment of the synchrotron radiation, where electrons orbiting in a magnetic field lose energy continually in the form of electromagnetic radiation. The first synchrotron light source was the Stanford Synchrotron Radiation Laboratory (SSRL) built in 1977 [28]. Nowadays, a few synchrotron radiations have been set up all over the world, and the synchrotron radiation has been developed into the third generation, to name a few, European Synchrotron Radiation Facility (ESRF) in France [3], Deutsches Elektronen-Synchrotron (DESY) in Germany (see Figure 2) [29], Shanghai Synchrotron Radiation Facility (SSRF) [30] in China, and so on. At PETRA III of DESY, the size of the X-ray beam could reach a few micrometers and the exposure time could be milliseconds. The high spatial and temporal resolution of synchrotron X-ray source enables to perform in-situ X-ray scattering measurements combining, for example, thermal/mechanical environment.

As X-rays interact with an object, they can be absorbed or scattered. For the scattering of X-rays by a single free electron, assuming elastic scattering the wavelength of the scattered wave is the same with that of the incident one. The relation between the scattered wave $E_2$ and incident wave $E_1$ is given as follows:

$$E_2 = E_1 \frac{\varepsilon_0^2}{m_0 c^2 r} \exp(-iqr)$$

(1)
where \( \frac{e_0^2}{m_0 c^2} = 3.54 \times 10^{-4} \ \text{Å} \) [32]. \( r \) is the position of the electron and \( q \) is the scattering vector, the magnitude of the scattering vector is

\[
q = \frac{4\pi}{\lambda} \sin \theta
\]  

(2)

\( \theta \) is the scattering angle. In addition to \( q \), another scattering vector \( s \) is used in the field of scattering,

\[
q = 2\pi s
\]  

(3)

The interference of all the scattered X-ray waves gives the final scattering signal. The efficiency of the scattering process could be described by the differential scattering cross-section \( \frac{d\sigma}{d\Omega} \) [32], which is given by

\[
\left( \frac{d\sigma}{d\Omega} \right) = \frac{I_m}{\Phi_0 \Delta \Omega} = \frac{|E_2|^2}{|E_1|^2} R^2
\]  

(4)

\( \Phi_0 \) defines the strength of the incident beam, \( I_m \) is measured scattering intensity, that is, the number of scattered photons recorded per second by the detector, \( \Delta \Omega \) is the solid angle, and the distance between the object and the detector is \( R \).

Depending on the distance between the object and the detector, the scattering experiments can be divided into four subareas, which are wide-angle X-ray scattering (WAXS) containing the classical X-ray diffraction, middle angle X-ray scattering (MAXS) covering the characteristic scattering of liquid-crystalline structure and rigid-rod polymers, small-angle X-ray scattering (SAXS) comprising the typical nanostructure in semi-crystalline polymers and thermoplastic elastomers, and ultra-small-angle X-ray scattering (USAXS) extending the detection range to micrometer scale [27]. Considering the scope of this chapter, SAXS will be emphasized especially. SAXS comprises the scattering angle range \( 2\theta < \frac{1}{2} \). With the help of SAXS, structures with the size of 1–500 nm can be detected, covering the size of lamellae and small voids.
2.2. Small-angle X-ray scattering

In SAXS measurement, X-rays detect the difference in electron density $\Delta r$ and the scattering intensity ($I_m$) is

$$I_m(s) = \Delta \rho^2 V_p^2 |F(s)|^2$$

(5)

$F(s)$ is the form factor and $V_p$ is the volume fraction of particles (for instance, the lamella in semi-crystalline polymers, the voids during deformation, and “shish” structure during shear induced crystallization). $R_g$ is the radius of gyration of the particle. In the extremely small scattering angle range, $sR_g \to 0$,

$$F(s) \approx 1 - \frac{s^2 R_g^2}{10}$$

(6)

The detailed deduction of Eq. (6) can be found elsewhere [33]. The initial intensity decay is approximated by Guinier approximation

$$I_m(s) \approx \Delta \rho^2 V_p^2 \left[ 1 - \frac{s^2 R_g^2}{10} \right]^2 \approx \Delta \rho^2 V_p^2 \left[ 1 - \frac{s^2 R_g^2}{5} \right] \approx I_m(0) \exp \left( -4\pi^2 R_g^2 s^2 \right)$$

(7)

The absolute scattering intensity $Q$ which is independent of the shape of the scatters,

$$Q = \int_{-\infty}^{\infty} I(s) ds \propto \Delta \rho^2 V_p (1 - V_p)$$

(8)

The pattern of SAXS measurement depends on the microstructure of the material. For the material with the periodically stacked structure (lamellae in semi-crystalline polymers) inside, the pattern exhibits a homogeneous ring or “two-spots” depending on the orientation of lamellae. The long period $L_p$, which comprise a layer of crystalline phase and a layer of amorphous phase in the two-phase model, can be evaluated by Bragg’s law,

$$L_p = \frac{2\pi}{q_{\text{max}}}$$

(9)

$q_{\text{max}}$ represents the position of the scattering ring or spots on the pattern in the reciprocal space. For a material with an oriented elongated structure, for instance an extended chain structure (shishs induced by flow) as well as voids, a streak scattering will show up in the pattern. Considering a perfect orientation of the elongated structures, Ruland [34] described the integral breadth $B_{\text{obs}}(s_{12})$ of the elongated structure, measured as a function of $s_{12}$, as follows:

$$B_{\text{obs}}(s_{12}) = \int_{-\infty}^{\infty} I(s_{12}, s_3) ds_3 / I(s_{12}, 0)$$

(10)

The average length $\langle L \rangle$ of the elongated structure is the inverse of the integral breadth:

$$B_{\text{obs}}(s_{12}) = \frac{1}{\langle L \rangle}$$

(11)
If misorientation has to be taken into account, the orientation distribution of the streak \( g(\varphi) \) must be considered, then the apparent azimuthal integral breadth becomes

\[
B_{\text{obs}}(s_{12}) = \frac{1}{I(s, \pi/2)} \int_{-\pi/2}^{\pi/2} I(s, \varphi) d\varphi
\]  

(12)

which depends on the width of the peak in the azimuthal direction. The evolution of \( B_{\text{obs}} \) as a function of \( s \) follows Eq. (13),

\[
B_{\text{obs}}^2(s) = \frac{B_p^2}{s^2} + \frac{1}{s^2 \langle L \rangle^2} + B_g^2
\]  

(13)

if a Gaussian can describe the orientation distribution. \( B_p \) describes the inevitable instrumental broadening and \( B_g \) is the true integral breadth of the orientation distribution. If a Lorentzian fits the orientation distribution [27], one obtains

\[
B_{\text{obs}} = \frac{B_p}{s} + \frac{1}{\langle L \rangle} + sB_g
\]  

(14)

3. **In-situ** synchrotron SAXS investigation about the cavitation behavior of microinjection-molded iPP

3.1. Microinjection molding

Microinjection molding is one of the most efficient methods for the large-scale production of thermoplastic polymer microparts. Depending on the area of interest, the definition of microparts comprises three categories [35]:

- parts processing a weight in the range of few milligrams,
- parts processing features where dimensions are in the micrometer range,
- parts exhibiting dimensional tolerances in the micrometer range but without dimensional limit.

In common injection molding, because of the flow and thermal field gradient, a “skin-core” structure can be found. In the skin layer, lamellae are oriented along the flow direction because of flow-induced crystallization. In the core layer, randomly distributed lamellae could be found due to the weak flow field. As to microinjection molding, the flow field in the cavity is quite strong due to the small cavity size especially for the first two cases. Therefore, a larger fraction of the skin layer is formed compared to common injection molding part [36], which means that a larger fraction of oriented structures exists in the microinjection-molded sample compared to the macroinjection-molded one.

In this chapter, the cavitation behavior of microinjection-molded iPP will be reported, studied by synchrotron SAXS, which serves as an exemplary result for the understanding of the topic in this chapter.
3.2. Experimental part

3.2.1. Materials and sample preparation

iPP used in this study was manufactured by Borealis (Linz, Austria) with the trademark of HD120MO. The weight average and number average molecular weights are 365 kg/mol and 67.6 kg/mol, and the melt flow index is 8 g/10 min (at 230°C and 2.16 kg). NJS (trade mark NJ-StarTR NU100), which is one kind of β-iPP nucleating agent, was kindly provided by Rika International Limited (Oldham, UK). NJS with a weight content of 0.3% was mixed with iPP granules by a twin screw extruder. Then the materials were microinjection molded with the following parameters: the barrel temperature is 280°C, the mold temperature is 25°C, and the injection molding speed is 25 cm³/s.

3.2.2. In-situ synchrotron X-ray scattering

In-situ synchrotron SAXS measurements were carried out at the MiNaXS beamline at Deutsches Elektronen Synchrotron (DESY), Hamburg, Germany. The wavelength of the X-ray was 0.106917 nm. An exposure time of 0.1 s and a time interval for the individual measurements of 0.15 s were used to realize the high time resolution without burning the specimens by X-ray in the meantime. The patterns were recorded by a Pilatus 1 M detector (981 × 1043 pixels, pixel size 172 × 172 µm²) with a detector distance of 4961 mm. Pattern preprocessing including masking and reconstruction of blind areas was performed by self-written subroutines on PV-Wave from Visual Numerics.

The uniaxial stretching was performed on a custom-made miniature tensile machine, as has been described elsewhere, for example, in Ref [37]. During measurements, to keep the X-ray beam at a fixed position on the specimen, both grips were moved simultaneously in opposite directions. The cross-head speed was 0.02 mm/s, and the stretching temperature was 75°C. The displacement during stretching was captured by a camera with a frame rate of 1 Hz. Figure 3 presents a schematic and a photograph of the experiment. The schematic can be also found in Ref. [38]. Hencky strain [39] was used to measure the local strain where the X-ray beam passed the specimen,

\[ \varepsilon_H = \ln \frac{\Delta L + L_0}{L_0} \] (15)

\( L_0 \) and \( \Delta L \) are the initial length and displacement of the grid pattern painted on the specimen during stretching.

The true stress \( \sigma \) can be estimated by

\[ \sigma = \frac{F}{A_0} \frac{\Delta L + L_0}{L_0} \] (16)

\( F \) is the load, and \( A_0 \) is the initial cross-sectional area.
4. Results and discussion

4.1. Microstructure of the microinjection-molded iPP before stretching

The 2D-SAXS and 2D wide-angle X-ray scattering (WAXS) patterns of the microinjection-molded sample are given in Figure 4. In the 2D-SAXS pattern, two scattering spots can be found on the meridian, indicating the orientation of lamellae as a result of shear induced crystallization during the injection molding process. In the 2D-WAXS pattern, from inner to the outer side, a few reflexes could be observed, which are the (110), (300), (040), (130), and (041)/(111)/(−131) crystalline reflexes. Among these reflexes, (300) and (041) belong to β-iPP and the others are originated from α-iPP. The intensity of the reflexes is focused at a specified angle with respect to the flow direction. The azimuthal angle deviation is decided by the lattice plane. The intensity of (110) reflex could be found both on the equator and in the meridian. This is caused by the unique “cross-hatched” structure of α-iPP. In the “cross-hatched” structure, daughter lamellae grow on the mother lamellae with an angle of 80° [15]. Mother lamellae grow directly on the shear-induced nuclei, resulting in the orientation of mother lamellae in the flow direction. Consequently, the daughter lamellae are 80° inclined to the flow direction.
4.2. Representative 2D-SAXS patterns during stretching

The true stress-Hencky strain curves of pure iPP and iPP/NJS composites stretched at room temperature are provided in Figure 5. Obviously, the composite shows a higher true stress at the same Hencky strain. The higher true stress is caused by the nucleation effect of NJS on iPP. Although the crystallinity of pure iPP and iPP/NJS composite is nearly the same, a larger long period of iPP/NJS composite indicates that less imperfect lamellae exist in the nucleated sample.

Some representative 2D-SAXS patterns of pure iPP and iPP/NJS composite during stretching are shown in Figure 6. The stretching direction is vertical in Figure 6. Without deformation, the scattering on the meridian is spherulite-like for iPP/NJS composite and droplike for pure iPP, indicating a more homogeneous distribution of long periods in the composite. Upon stretching, the amorphous phases constrained by the lamellae are stretched along the loading direction, leading to a slight shift of the scattering position on the pattern [40]. In the center
region of the pattern, a streak can be found, which is aligned along the meridian. The appearance of the streak is an indication of the void formation [1]. The direction of the streak with respect to the stretching direction suggests that the elongated voids are perpendicular to the streak direction. As the Hencky strain is further enlarged, the scattering of the streak continues to grow in the vertical direction, which is probably caused by the growth of the void. At an even larger strain ($\varepsilon_H = 0.641$), the scattering of lamellae is covered by the scattering of the void.

In addition, a streak on the equator shows up indicating the formation of the second group of voids. This group of voids is aligned along the stretching direction. The formation of the second group of voids can be triggered by the first group of voids coalesces [41]. With the further increase of Hencky strain, the scattering grows mainly in the meridian, and the scattering intensity on the equator is narrowed.

4.3. The evolution of the scattering invariant

The scattering invariant ($Q$) of pure iPP and the iPP/NJS composite as a function of Hencky strain during stretching is given in Figure 7. As shown in Eq. (8), $Q$ is proportional to $V_v(1 - V_v)$, where $V_v$ is the volume fraction of the void. As $V_v$ is smaller than 0.5, $Q$ increases with $V_v$. However, if $V_v$ is higher than 0.5, $Q$ will decrease. In Figure 7, one can see that at the beginning, $Q$ keeps unchanged until a Hencky strain of 0.1. As the Hencky strain exceeds 0.1, $Q$ increases gradually indicating the formation of voids. As the Hencky strain is larger than 0.2, $Q$ reaches a plateau for pure iPP, but continues to increase for iPP/NJS composite. The larger $Q$ of the composite at the same strain means that more voids are formed in iPP/NJS composite. When the Hencky strain is larger than 0.6, a drastic drop of $Q$ can be found for iPP/NJS composite. Two possible reasons can be proposed to be responsible for the decrease trend: the first one is that the size of the void exceeds the detection range of SAXS, and the second one is that the volume fraction of the voids is larger than 0.5 as discussed above. The first reason seems to be the dominant one.

![Figure 6](image-url) Representative 2D-SAXS of pure iPP and iPP/NJS composite during stretching. The stretching direction is vertical.
4.4. The evolution of the void size

As introduced in the first part, the size of the void can be evaluated by Ruland’s streak method, if the shape of the void is regarded as cylindrical [27, 42]. To avoid the burning of the detector during the measurement, the center region of the beam is covered by a beamstop. To get information about the void size, the 2D-SAXS patterns in Figure 6 are fitted by a sum of two 2D-Gaussian functions

\[
l(s) = p_{v0} \exp \left( -p_{v1}s^2 - p_{v2}\cos (2\phi) s^2 \right) + p_{m0} \exp \left( -p_{m1}s^2 - p_{m2}\cos (2\phi) s^2 \right)
\]

\[
\phi = \arctan \frac{i}{j}
\]

\(i\) and \(j\) define the position of the pixel in the pattern. The voids are described by the first Gaussian curve by parameters \(p_{v0}\), \(p_{v1}\), and \(p_{v2}\). The scattering of the matrix is described by the second Gaussian curve. The fitting process is carried out by a self-written subroutine. A representative application of the fitting procedure is provided in Figure 8.

After the center fitting procedure, the void scattering can be extracted. The voids length along the stretching direction \((L_h)\) and the voids length perpendicular to the stretching direction \((L_v)\) can be calculated as

\[
L_h = \sqrt{\frac{p_{v1} + p_{v2}}{\pi}}
\]

\[
L_v = \sqrt{\frac{p_{v1} - p_{v2}}{\pi}}
\]

The evolution of the void size is presented in Figure 9. As the Hencky strain is smaller than 0.1, no voids can be detected. At Hencky strains beyond 0.1, voids show up as evidenced in
Figures 6 and 9. $L_h$ is around 110 nm and $L_v$ is 200 nm for pure iPP, indicating that the longitude of the void is perpendicular to the stretching direction. In addition, in this Hencky strain range, the void length is similar for pure iPP and iPP/NJS composite. This suggests that at the beginning of void formation, the addition of NJS has a negligible influence on the void size. As the Hencky strain is increased to 0.6, it can be found that $L_v$ decreases gradually and $L_h$ starts to increase, implying that the direction of the void is changed. Finally, the longitude direction of the voids changes from transversal to parallel direction with respect to the stretching direction. The change of the voids direction is caused by coalescence of small voids as well as the affine deformation of the voids. In this Hencky strain range, $L_h$ of iPP/NJS composite reaches 550 nm, finally. This is much larger than that of pure iPP, which is only 200 nm. It should be pointed out that due to the growth as well as the direction change of the voids, no obvious streak signal can be found on the SAXS pattern in the Hencky strain of 0.3–0.6. So the voids size in this strain range is not provided.

Figure 8. Center fitting and modeling of the 2D-SAXS pattern by the sum of the void scattering and the matrix scattering according to Guinier approximation. The stretching direction is along $s_3$.

Figure 9. The plots of void length as a function of Hencky strain. $L_h$ is the void size along the stretching direction and $L_v$ is the void size perpendicular to the stretching direction.
5. Conclusions

In this chapter, recent reports about cavitation behavior in iPP during deformation are summarized. The influence of morphological factors including crystal form, crystallinity, lamellae thickness, state of the amorphous phase, and lamellae orientation is emphasized. In addition, the background related to the synchrotron SAXS is introduced. Finally, as exemplary results, the cavitation behavior of microinjection-molded iPP is presented. By center fitting, the blank region on the SAXS pattern is fitted by extrapolation. The size of the void is calculated by an approximation according to Guinier law. The result shows that upon stretching, the longitude of the void is aligned perpendicular to the stretching direction in the early stages of deformation and then transfers to the stretching direction. In the early stages, the addition of NJS has a negligible influence on the void size. The size of the voids is 210 nm perpendicular to the stretching direction and 110 nm along the stretching direction for both pure iPP and the iPP/NJS composites. However, in the late stages, the size of the voids along the stretching direction increases to 550 nm for the iPP/NJS composites. This is much larger than that of pure iPP, which is only 200 nm.

Acknowledgements

Baobao Chang would like to thank the grant by China Scholarship Council (CSC). The authors are grateful to DESY for beamtime within project I20140478. The authors acknowledge Prof. Stribeck at University of Hamburg for helpful discussions about the Ruland’s streak method and Matthieu Fischer (IPF Dresden) for the preparation of microinjection-molded samples.

Conflict of interest

The authors declare no conflict of interest.

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