

Spatially Resolved Cavitation within Tensile Stretched Isotactic Polybutene-1 Spherulite

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Electronic Supplementary Information

Abstract The precise microstructural origin of cavitation in semicrystalline polymers remains a subject of persistent controversial. Consequently, the exact spatial initiation of voids within a single spherulite remains under debate. Resolving this ambiguity has long been hindered by the spatial resolution limits of the conventional characterization techniques. To overcome this limitation, we employ an integrated approach of *in situ* synchrotron microfocus X-ray scattering and ultrasmall-angle X-ray scattering to investigate the cavitation behavior within individual isotactic polybutene-1 spherulite during uniaxial stretching. It turns out that early-stage voiding exhibits a distinct spatial sequence. Structural damage preferentially initiates at the spherulitic center, subsequently emerges in the equatorial region, and ultimately propagates to polar regions. By reconstructing the three-dimensional lamellar orientation within the undeformed spherulite, we revealed that lamellae oriented parallel to the stretching direction are extensively distributed across all spherulitic regions. Based on these findings, we propose a micro-mechanical cavitation model. In this framework, parallel lamellae undergo direct mechanical fragmentation under stress, with microvoids nucleating within the interstitial gaps between adjacent crystalline blocks.

Keywords Polybutene-1; Spherulite; Cavitation; Microfocus X-ray scattering

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INTRODUCTION

The macroscopic mechanical behavior of semicrystalline polymers is intrinsically governed by their hierarchical structural evolution under applied stress. During uniaxial stretching, these materials accommodate macroscopic strain through a complex interplay of localized microscopic mechanisms, predominantly including fine and coarse slips within crystalline lamellae, melting-recrystallization, and cavitation.^[1–3] Specifically, small-strain cavitation emerging in the vicinity of the macroscopic yield point is of paramount interest.^[4,5] This early-stage voiding not only signifies the onset of irreversible material failure but also functions as a crucial pathway for energy dissipation, thereby fundamentally dictating the macroscopic mechanical properties and ultimate toughness of the polymeric system.^[6–9]

Despite its critical role in polymer failure, the precise microstructural origin of cavitation within semicrystalline matrix remains a subject of persistent controversy. Currently, two distinct models have been proposed in literature. One perspective proposes that cavities appearing around the yield

point are initiated in the amorphous phase between adjacent crystalline lamellae.^[10–13] On the other hand, we have previously proposed that cavitation around the yield point during the stretching of a semicrystalline polymer should be initiated first within those crystalline lamellae with their normal perpendicular to the stretching direction.^[14,15] While both models have found certain experimental evidence supporting aspects of their respective views, they yield fundamentally divergent predictions regarding the spatial distribution of early-stage voids within a single spherulite.^[16] If cavitation is primarily an amorphous-phase phenomenon, it is anticipated to initiate preferentially in the equatorial sectors of the spherulite, where inter-lamellar separation is maximized. In contrast, according to our crystal-destruction model, the onset of voiding should localize in the polar sectors, where lamellae oriented parallel to the loading axis experience maximal normal stress, leading to fragmentation.^[17,18]

Validating these spatially divergent predictions has long been hindered by the inherent resolution limits of the conventional characterization techniques. Standard small-angle and wide-angle X-ray scattering (SAXS/WAXD) methodologies typically employ beam sizes that are substantially larger than the dimensions of individual spherulites, producing macroscopically averaged structural information.^[19,20] Such spatial averaging inevitably obscures the intricate sector-

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dependent deformation processes, making it experimentally impossible to distinguish whether the initial cavitation localizes in the equatorial or polar regions.^[21] To resolve this fundamental ambiguity, a spatially resolved approach capable of mapping the real-space distribution of reciprocal-space structural parameters is required. Fortunately, synchrotron microfocus X-ray scattering provides opportunity to overcome this problem.^[22–24] By utilizing micrometer-scale beam to conduct high-precision mapping across a single spherulite, it is now feasible to pinpoint the exact initiation sites of cavitation and test controversial models.

To systematically investigate this fundamental issue and test our proposed model, isotactic polybutene-1 (*i*PB-1) serves as an optimal model system.^[25,26] Its stable crystal modification is characterized by a "crystal-fix" nature, which kinetically hinders dislocation glide, thereby inherently favoring rigid-body lamellar fragmentation and cavitation over plastic slip.^[27,28] In this study, we present a comprehensive and spatially resolved investigation of the structural evolution within individual *i*PB-1 spherulites during uniaxial stretching. Employing *in situ* synchrotron microfocus SAXS/WAXD in conjunction with ultra-small-angle X-ray scattering (USAXS) and polarized optical microscopy, we systematically map the localized deformation pathways across the entire spherulitic architecture. This integrated approach allows us to visualize the damage initiation with precision. It turns out that early-stage cavitation preferentially emerges in the equatorial sectors under current condition. Crucially, however, we revealed that this equatorial voiding is not driven by amorphous-phase dilation as conventionally assumed, but is intrinsically coupled with the mechanical fragmentation of crystalline lamellae. These findings reconcile the macroscopic spatial distribution of voids with their underlying microscopic origin, providing experimental confirmation for our crystal-destruction model and offering physical insights into how local stress anisotropy dictates the ultimate failure pathways in semi-crystalline polymers.

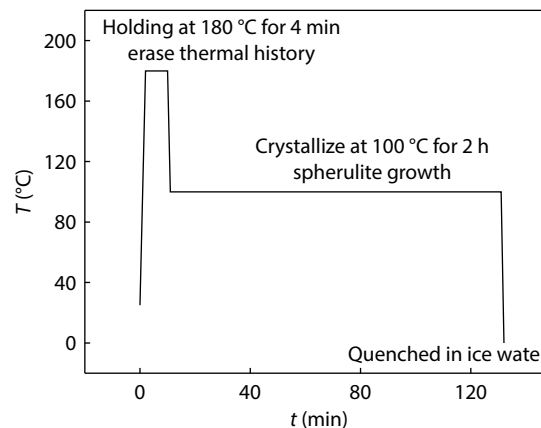
EXPERIMENTAL

Materials

The *i*PB-1 used in this study was a commercial homopolymer (trade name: PB0110) produced by LyondellBasell (The Netherlands). The polymer had a weight-average molecular weight (M_w) of 711 kg/mol and number-average molecular weight (M_n) of 203 kg/mol, resulting in a polydispersity index (M_w/M_n) of 3.5. The melt flow index (MFI) was reported as 0.4 g/10min (190 °C, 2.16 kg). The as-received pellets were used without further purification.

Sample Preparation

To eliminate the prior thermal history, the *i*PB-1 pellets were first compression molded into thin films (approximately 100 μ m in thickness) at 180 °C for 4 min under a pressure of 10 MPa. Subsequently, the films were transferred to a preheated oven for isothermal crystallization at 100 °C for 120 min, allowing for the growth of large spherulites with diameters of several hundred micrometers. After the isothermal step, the films were rapidly quenched in an ice-water bath (0 °C) to immediately arrest the spherulitic growth. Scheme 1 provides a schematic representa-



Scheme 1 Schematic illustration of preparation of the *i*PB-1 spherulites.

tion of the sample-preparation procedure. The as-prepared films, containing metastable form II crystals, were then aged at ambient temperature (about 25 °C) for 30 days to ensure complete transformation to thermodynamically stable form I.

Characterization Methods

In situ polarized optical microscopy (POM)

The morphological evolution of *i*PB-1 spherulites during uniaxial stretching was observed *in situ* using a polarized optical microscope (Axio Imager A2m, Carl Zeiss, Germany). The microscope was equipped with a compact tensile stage (TST350, Linkam, UK). The stretching experiments were performed at a constant tensile speed of 10 μ m/s at room temperature (25 °C). A 10 $\times$ objective lens was used for all observations.

In situ synchrotron small- and wide-angle X-ray scattering (SAXS/WAXD)

In situ microfocus SAXS/WAXD measurements were performed at beamline BL10U1 (Shanghai Synchrotron Radiation Facility, SSRF). Two Dectris Pilatus 1M detectors were employed to simultaneously collect 2D WAXD and SAXS patterns. The sample-to-detector distances were calibrated to be 131.2 mm for the WAXD and 3244 mm for the SAXS. The X-ray wavelength was 0.124 nm (corresponding to an energy of 10 keV). To achieve a high spatial resolution, the micro-focused X-ray beam size was set to 10 μ m $\times$ 8 μ m (Full Width at Half Maximum, FWHM).

For precise positioning of the microbeam on specific subregions within a spherulite, a custom-built video microscope was integrated into the beam path, allowing for simultaneous optical observation of the sample morphology. *In situ* tensile tests were conducted using a custom-designed miniature stretching stage, which was necessary due to the spatial constraints of the microfocus setup. The stretching speed can be varied from 10 μ m/s to 2000 μ m/s with a displacement precision of 2 μ m. During the *in situ* experiments, the films were uniaxially stretched at a speed of 10 μ m/s at room temperature (25 °C). When the sample was deformed to a predetermined strain, microfocus scanning was performed across a defined rectangular region encompassing an entire spherulite and its immediate surroundings to map the spatial distribution of structural changes. The exposure time was set to 1 s for each pattern. All collected 2D patterns were corrected for background scattering using standard procedures.

Ultrasmall-angle X-ray scattering

To precisely quantify the dimensions of large-scale structures, particularly the micro-voids formed during deformation, ultrasmall-angle X-ray scattering (USAXS) measurements were performed. Experiments were conducted using a customized Xeuss X-ray apparatus (Xenocs SA, France). The system was equipped with a microfocus Cu K α X-ray source ($\lambda=0.154$ nm). A well-collimated beam, with dimensions of $0.2\text{ mm} \times 0.2\text{ mm}$ at the sample position, was defined by two sets of scatterless slit systems located 2.4 m apart from each other. Two-dimensional scattering patterns were collected using a Pilatus 100K detector (DECTRIS, Switzerland), which featured $487\text{ pixels} \times 197\text{ pixels}$ with a pixel size of $172\text{ }\mu\text{m} \times 172\text{ }\mu\text{m}$. A long sample-to-detector distance of 5032 mm was employed to access the ultrasmall-angle region. The exposure time for each static measurement was 30 min .

RESULTS AND DISCUSSION

The mechanical response of the *iPB-1* film specimen was first characterized to establish the relationship between macroscopic deformation and microscopic structural evolution. Fig. 1 shows the true stress-strain ($\sigma - \epsilon_H$) curve obtained by uniaxial stretching.

$$\epsilon_H = \ln \frac{L}{L_0} \quad (1)$$

$$\sigma = \frac{F}{A} = \frac{FL}{A_0 L_0} \quad (2)$$

where L_0 and L are the initial length and instantaneous length of the sample during stretching, respectively, and F , A and A_0 are the instantaneous force, instantaneous cross-sectional area, and initial cross-sectional area of the sample. Despite the heterogeneous microstructure induced by the large spherulites, the specimen exhibited a classical mechanical profile typical of semi-crystalline polymers: (i) an initial elastic region, (ii) a yield re-

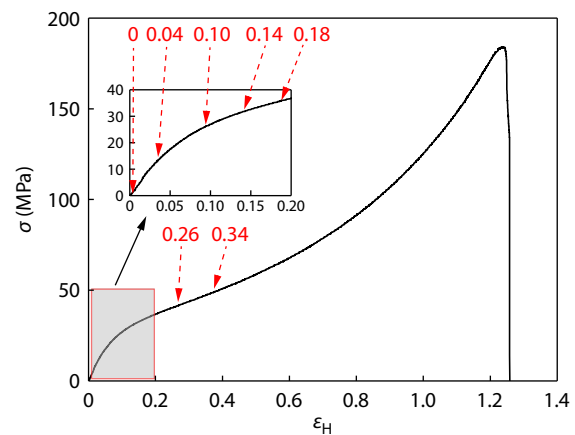


Fig. 1 Representative true stress-strain curve measured for the *iPB-1* sample under uniaxial tension at room temperature. The red arrows indicate the strains adopted in SAXS measurements.

gion ($\epsilon_H = 0.08$), and (iii) a subsequent plastic flow region (strain hardening).

As the purpose of this work is to clarify the cavitation behavior within spherulite around yielding, we limit our investigation below a macroscopic strain of 0.34 . To quantify local strain within spherulite, *in situ* POM observations were performed concurrently with stretching. Fig. 2 presents a series of POM micrographs captured at representative strain levels (as indicated in Fig. 1). Fig. 2 provides a direct visualization of spherulitic deformation corresponding to these mechanical states. In the pristine state ($\epsilon_H = 0$), the spherulite exhibited a distinct Maltese cross pattern, confirming its well-organized, radially oriented lamellar structure. This morphology remained largely unchanged within the elastic region ($\epsilon_H = 0.04$), showing no discernible deformation.

However, as the specimen surpassed the yield point ($\epsilon_H = 0.10$), significant changes were observed in the POM re-

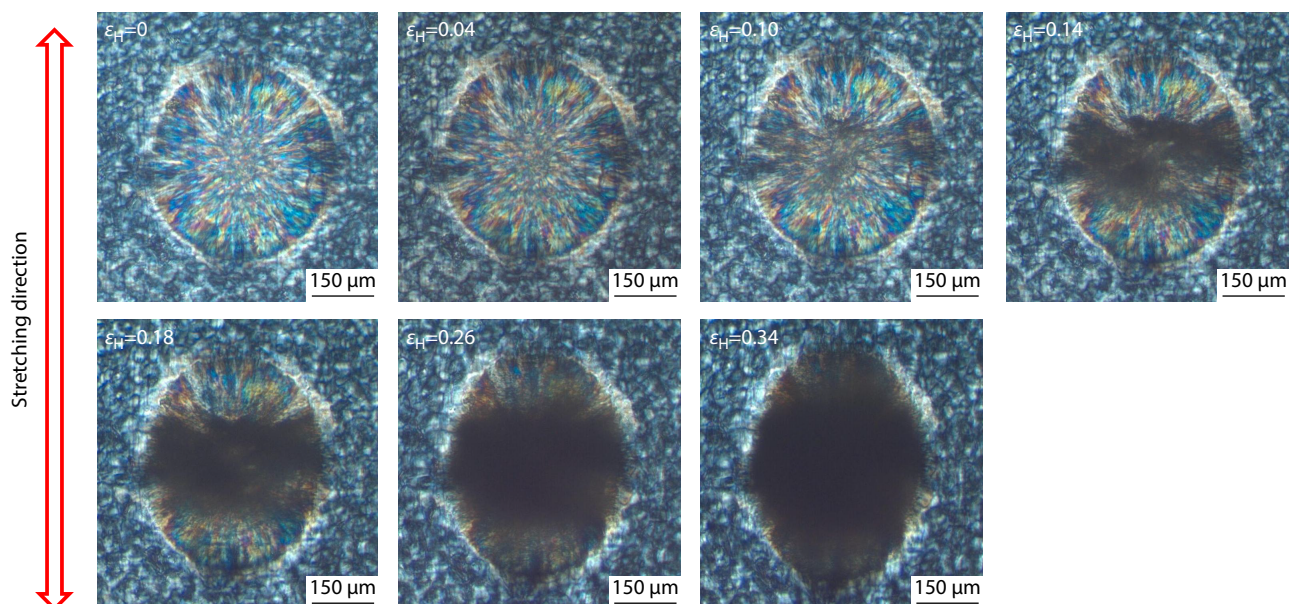


Fig. 2 Morphological evolution of *iPB-1* spherulites observed by POM during *in situ* uniaxial stretching. The arrow indicates the stretching direction.

sults. The spherulite begins to show evident deformation, transforming into an elliptical shape by elongation in the polar region (parallel to the stretching direction) and contraction in the equatorial region (perpendicular to the stretching direction). Concurrently, a subtle but distinct loss of birefringence (*i.e.*, darkening) occurred in the equatorial sectors. This darkening became more pronounced as stretching proceeded into the plastic flow region. At $\varepsilon_H = 0.18$, the dark regions expand and intensify, indicating a progressive disruption of the incident polarized light. At the maximum strain of $\varepsilon_H = 0.34$, almost the entire spherulite has lost its appearance of birefringence.

This progressive "darkening" observed in the POM micrographs is interpreted as a direct optical signature of cavitation also known as "stress-whitening" phenomenon. Although the volume fraction of the cavities during the tensile stretching of an iPB-1 sample is typically only a few percent, strong multiple light scattering occurs because of the strong refractive index difference between the cavities and the polymer matrix. Such multiple scattering of the incident light results in a complete loss of its polarization direction and is thus mostly blocked by the analyzer of the POM. The loss of brightness directly maps the nucleation and growth of these voids.

In addition to the observed variations in the optical properties, we quantitatively compared the macroscopic strain of the bulk specimen with localized strains within the equatorial and polar regions of the spherulite. The true strains were calculated using following equations:

$$\varepsilon_{H-Polar} = \ln \left(\frac{r_p}{r_0} \right) \quad (3)$$

$$\varepsilon_{H-Equatorial} = -2 \ln \left(\frac{r_E}{r_0} \right) \quad (4)$$

where r_0 denotes the initial spherulite diameter; r_p and r_E represent the deformed spherulite diameters along the polar and equatorial directions, respectively. The results are presented in Fig. 3. It is evident that the macroscopic strain of the bulk sample is significantly larger than the localized strains within both equatorial and polar regions. This disparity indicates that the deformation occurring outside the spherulite contributes substantially to the total strain compared with the deformation activated within the spherulite. This observation aligns with the fact that the interior of spherulite possesses a higher modulus than its surrounding regions.^[29]

Furthermore, a distinct reversal in the relative magnitudes of the intraspherulitic strain was observed. At macroscopic elongations below 20%, the localized strain in the equatorial region exceeded that in the polar region. However, once the macroscopic elongation exceeded 20%, the polar strain rapidly overtakes the equatorial strain. This specific deformation sequence is governed by local lamellar orientations. Initially, the equatorial region was mechanically softer because of its specific lamellar arrangement, allowing it to deform, the polar region remained largely undeformed. This initial resistance confirms the structural robustness of the polar region. It is only after macroscopic deformation reaches a critical threshold that this robust polar region begins to yield significantly, leading to rapid strain accumulation and subsequent localized cavitation.

To provide direct, spatially resolved evidence for the cavi-

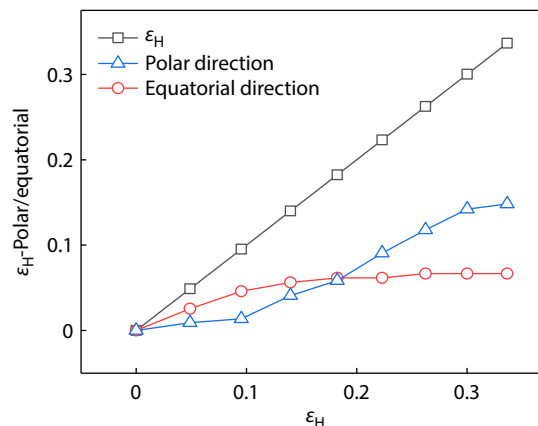


Fig. 3 True strain-extension curves of the whole tensile test bar and the PB-1 spherulite both in the equatorial direction and polar direction.

tion mechanism proposed from the POM analysis and elucidate the lamellar-level structural changes, *in situ* microfocus SAXS/WAXD measurements were conducted as a function of strain. The scanning experiment, which maps the entire spherulite, is schematically illustrated in Fig. S1 (in the electronic supplementary information, ESI).

Fig. 4 presents a composite map of the 2D SAXS patterns collected from a single, representative spherulite in its undeformed state ($\varepsilon_H = 0$). All patterns have been normalized for incident beam intensity variations to allow for quantitative comparison. To maintain consistency, the strain values denoted here and hereafter refer to the macroscopic strain of the bulk specimen, which is identical to the convention used in POM analysis. In this initial state, the 2D SAXS patterns revealed the local orientation of the lamellar stacks, which followed the well-established radial growth model of spherulites.

Due to the reciprocal nature of the scattering technique, the direction of the scattering maxima is parallel to the real-space lamellar normal (*i.e.*, the stacking direction). This anisotropy is clearly demonstrated when comparing distinct spherulitic regions that are defined relative to the future stretching direction. In the equatorial regions, where the spherulite radius is perpendicular to the stretching direction, the SAXS patterns exhibit distinct meridional reflections (scattering maxima parallel to the stretching direction). This indicates that the lamellar normals were oriented parallel to the drawing direction (*i.e.*, perpendicular to the radius). Conversely, in the polar regions, where the spherulite radius is parallel to the extensional direction, the scattering peaks are concentrated at the equator (perpendicular to the drawing direction), indicating that the lamellar normals are oriented perpendicular to the stretching direction. This spatially resolved map confirms a structural model where lamellae are arranged tangentially relative to the radial growth direction.

However, a crucial point must be addressed. The spherulite is a three-dimensional object, whereas two-dimensional SAXS patterns capture only the projections of lamellae whose normals lie in the plane perpendicular to the incident X-ray beam. Thus, it was insensitive to lamellae stacked parallel to

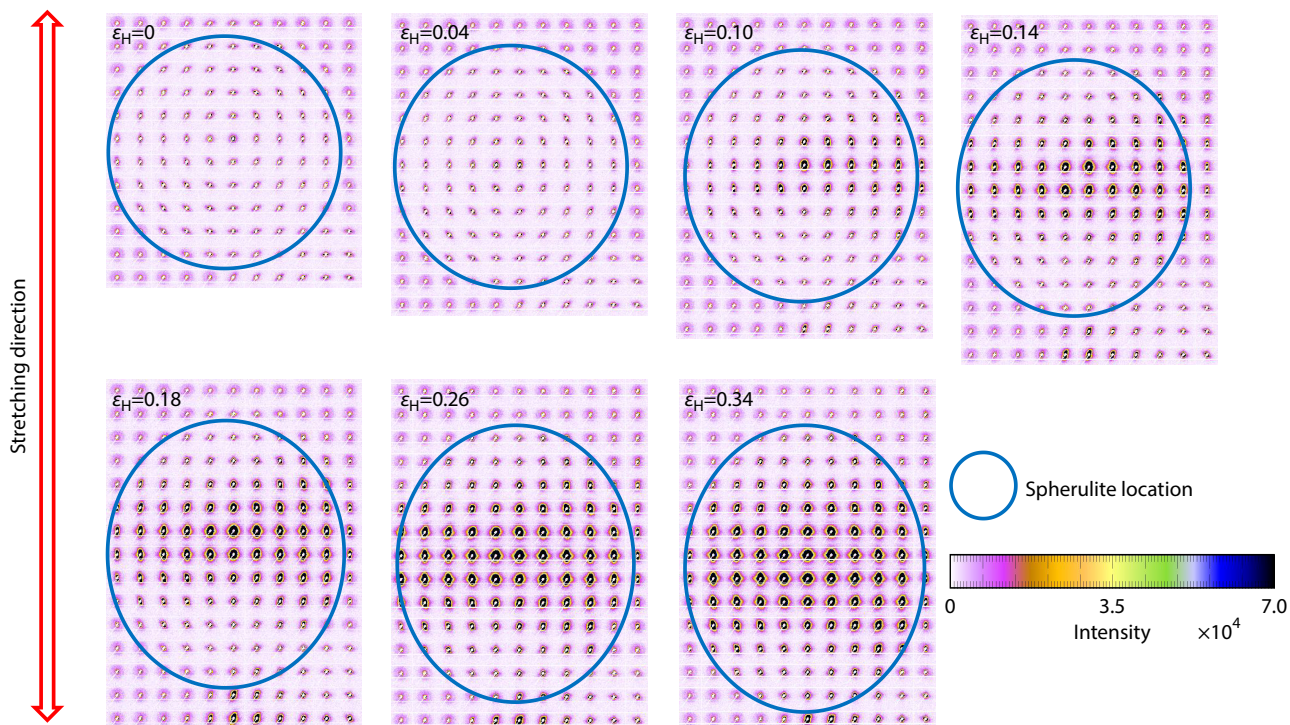


Fig. 4 Spatial distributions of two-dimensional SAXS patterns in the *iPB*-1 spherulite recorded at various strains.

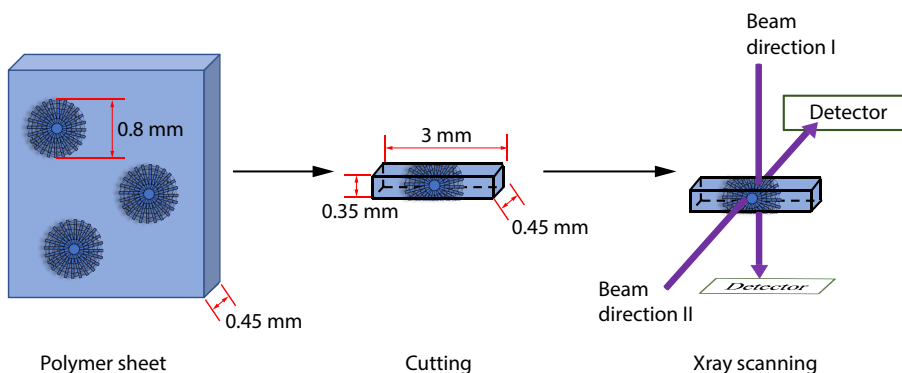


Fig. 5 Schematic diagram of scanning *iPB*-1 spherulites under different X-ray directions.

the X-ray beam. It was well-documented that lamellae in polyolefin spherulites, including *iPB*-1, often grow in a helically twisted manner along the radius.

In order to confirm this complex three-dimensional arrangement, WAXD patterns were collected from the equatorial region using two mutually perpendicular incident X-ray directions. Only this part of the study was completed at the BL13SSW at SSRF. As illustrated in Fig. 5, a specimen containing a single spherulite with an initial diameter of approximately 800 μm was prepared. Subsequently, the segment encompassing the equatorial region of the spherulite was precisely sectioned into a rectangular strip with dimensions of 0.35 mm $\times$ 0.45 mm $\times$ 3 mm. Subsequently, the specimen was scanned from two mutually orthogonal directions utilizing an X-ray microbeam with a spot size of 10 μm $\times$ 13 μm .

Based on the acquired two-dimensional WAXD patterns, the Hermans orientation factor for the (102) crystal plane was calculated. The orientation factor is defined as:^[30]

$$f = \frac{3 \langle \cos^2 \phi \rangle - 1}{2} \quad (5)$$

where ϕ is the angle between the normal of the diffraction plane and the reference axis (defined as pointing horizontally to the right, as depicted in the left panel of Fig. 6). The average orientation, $\langle \cos^2 \phi \rangle$, is calculated from the azimuthal intensity distribution $I(\phi)$ as:

$$\langle \cos^2 \phi \rangle = \frac{\int_0^{\pi/2} I(\phi) \cos^2 \phi \sin \phi \, d\phi}{\int_0^{\pi/2} I(\phi) \sin \phi \, d\phi} \quad (6)$$

where $\cos \phi$ is derived from the Bragg and azimuthal angles according to the following relation:

$$\cos \phi = \cos \theta_{hkl} \cos u \quad (7)$$

where $2\theta_{hkl}$ and u denote the Bragg diffraction angle of the plane and the azimuthal angle, respectively. The resulting spatial distribution of $f_{(102)}$ is presented in the middle panel of Fig. 6,

whereas the corresponding azimuthal intensity integration profiles for the equatorial region are shown in the right panel of Fig. 6.

Notably, when the incident X-ray direction was altered to be mutually orthogonal, the Hermans orientation factor exhibited an identical spatial distribution. This observation provides compelling evidence that the orientation of the molecular chain *c*-axis is independent of the macroscopic observation. Consequently, this demonstrates that within the equatorial sectors, the *c*-axes underwent a continuous 360° twist around the radial direction (*i.e.*, the spherulitic growth axis).

Furthermore, the normal of the crystalline lamellae—formed *via* polymer chain folding and stacking—exhibit an analogous rotationally symmetric distribution around the radial axis. Given the intrinsic radial symmetry of a static spherulite, this topological rule is not confined to equatorial regions; the lamellar assembly in the polar sectors must inherently possess equivalent three-dimensional geometric characteristics. Accordingly, a realistic three-dimensional spatial arrangement of the lamellae within the entire spherulite is schematically illustrated in Fig. 7.

Unlike classical two-dimensional spherulite models, this

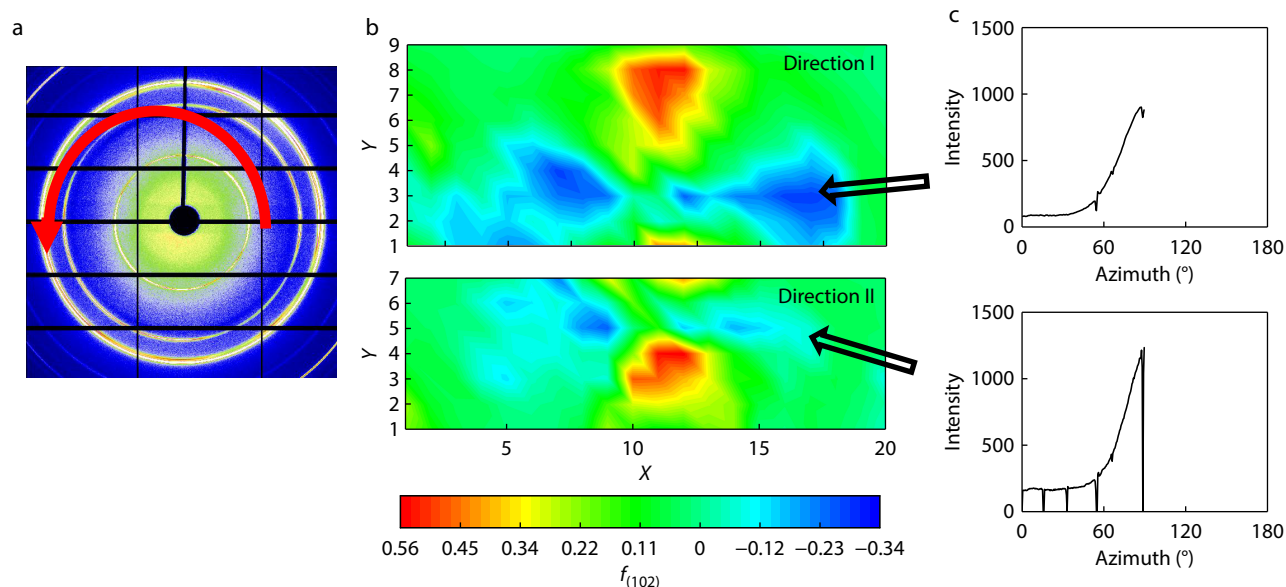


Fig. 6 Three-dimensional orientation characterization of *iPB-1* spherulites under different X-ray incident angles. (a) Azimuthal integration direction of WAXD patterns; (b) Hermans orientation factor of the Form I (102) plane; (c) Azimuthal integration curves at the equatorial region.

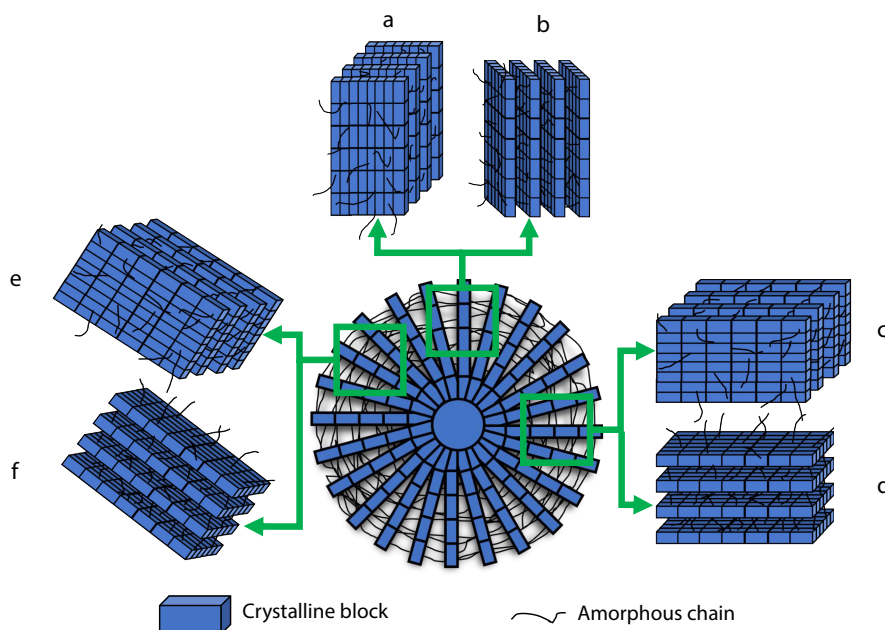


Fig. 7 Three-dimensional orientation model of lamellar in *iPB-1* spherulite: simplified lamellar models with two mutually perpendicular orientations in the polar (a, b), equatorial (c, d), and diagonal (e, f) regions. The lamellae are parallel to the stretching direction in (a, b, c), perpendicular to the stretching direction in (d), and tilted with respect to the stretching direction in (e, f).

three-dimensional framework reveals a more complex topological reality. Assuming a vertical macroscopic stretching direction, lamellae oriented parallel to the stretching direction coexist across all regions. Specifically, in the polar sectors, all the lamellar populations were aligned parallel to the stretching direction. Conversely, in equatorial sectors, only a minimal fraction of lamellae is oriented strictly perpendicular to the stretching direction, fundamentally contradicting the assumptions of simplified two-dimensional models. Such pronounced spatial heterogeneity in both the specific orientation and volumetric proportion of lamellae across different spherulitic sectors directly dictates their distinct local cavitation behaviors during the subsequent stretching process.

Beyond the complex spatial orientation of the lamellae, the long spacing (d_{ac}) of the lamellar stacks within and outside the spherulite before stretching can be obtained by reading the peak position of the Lorentz-corrected one-dimensional SAXS intensity distribution ($I(q) \cdot q^2$ versus q) obtained by integrating the 2D patterns in Fig. 4 at $\varepsilon_H = 0$. Within the spherulite a value of approximately 51 nm for d_{ac} was obtained at different locations. The value of d_{ac} was only 21 nm outside the spherulite because of the much lower crystallization temperature during quenching.

The structural evolution during uniaxial stretching was tracked by *in situ* microfocus SAXS, with representative 2D pattern maps shown in Fig. 4 at various strains. Initially, in the elastic deformation regime ($\varepsilon_H = 0.04$), the 2D SAXS patterns across the entire spherulite showed no significant change compared with the undeformed state. Characteristic scattering from the lamellar stacks in both the polar and equatorial regions remained stable. This confirms that the initial deformation was predominantly elastic and reversible, without inducing significant plastic events.

A significant transformation occurred when the specimen surpassed the yield point. At $\varepsilon_H = 0.10$, a new exceptionally strong scattering signal emerged. This signal first appeared at the spherulite center and was anisotropic, manifesting as an intense streak along the meridional direction (parallel to the stretching direction). This new scattering is direct evidence of cavitation within the spherulite.

The anisotropy of this new signal was critical. Based on scattering principles, strong scattering along the meridian (with scattering vector q parallel to the stretching direction) must originate from real-space structures with interfaces oriented perpendicular to the stretching direction. This observation confirms the formation of highly anisotropic plate-like voids, whose main interfaces are oriented perpendicular to the applied stress. The specific physical mechanism responsible for the nucleation of these voids is complex and will be analyzed in a dedicated discussion later. At this stage, we identify this new meridional signal as the signature of "void scattering."

Furthermore, at the initial stages of voiding (approximately $\varepsilon_H = 0.14$), the spatial distribution of the newly formed voids exhibited a noticeable lack of ideal symmetry. This observation was attributed to the inherent structural heterogeneity within real spherulites. First, the non-crystallographic branching that initiates from the primary sheaf is a stochastic process that leads to localized morphological irregularities and

density fluctuations near the spherulitic core, and its subsequent growth further affects the structure of the entire spherulite. Second, the exceptionally large dimensions of these spherulites (reaching approximately 500 μm) increase the probability of incorporating impurities or structural defects during the crystallization process. During stretching, these factors collectively induce a non-uniform localized stress field, thereby leading to the observed asymmetric distribution of the initial voids.

As the strain further increases ($\varepsilon_H = 0.18$ – 0.34), this intense meridional "void scattering" progressively expands. The cavitation process, which nucleated at the center, propagated radially outward toward the spherulite periphery. At $\varepsilon_H = 0.34$, the majority of the spherulitic area is dominated by this strong void scattering.

It should be noted that at higher strains ($\varepsilon_H \geq 0.14$), the scattering enhancement observed outside the blue-marked region originated from a secondary, morphologically poorly defined spherulite located immediately adjacent to the primary spherulite. Specific micrographs of this spherulite are shown in Fig. S2 (in ESI). Under tensile stress, the imperfect spherulites also underwent localized deformation and voiding. Because its imperfect morphology makes it unsuitable for rigorous quantitative analysis of spatial evolution, it was excluded from our discussion. Furthermore, the absence of voiding signals in the quenched matrix region outside the upper part of the primary spherulite confirms that under the current experimental conditions, the matrix itself did not undergo voiding.

This spatially resolved SAXS evolution aligns perfectly with *in situ* POM observations. The progressive loss of birefringence (darkening) observed in Fig. 2 is now identified as widespread nucleation and growth of these highly anisotropic voids, which disrupt the original birefringence of the lamellar structure.

To quantify the spatial evolution of the cavitation process observed in Fig. 4, the apparent scattering invariant (Q') was calculated for each pixel of the mapped area. Q' is proportional to the invariant Q and is defined as:^[31]

$$Q \propto Q' = \int_{Q_{\min}}^{Q_{\max}} \int_{q_{\min}}^{q_{\max}} I(q_x, q_y) dq_x dq_y \quad (8)$$

This quantity is proportional to the mean square electron density fluctuation, $\Delta\rho^2$ of the system. In the initial two-phase (crystalline/amorphous) state, Q is proportional to $\phi_c(1 - \phi_c)(\rho_c - \rho_a)^2$. However, upon the formation of voids (a third phase with $\rho_v \approx 0$), the system becomes dominated by a new, much larger electron density contrast. The invariant can then be approximated as $Q \approx \phi_v(1 - \phi_v)\rho_p^2$, where ρ_p is the average polymer density. Therefore, a significant increase in Q can be attributed to an increase in the local void volume fraction (ϕ_v).

The spatial maps of Q' for the representative strains are presented in Fig. 8. In the undeformed and elastically deformed states ($\varepsilon_H \leq 0.04$), the Q' map revealed distinct variations linked to the initial microstructure. Specifically, Q' is highest at the spherulite center, followed by the rest of the spherulite body, and lowest in the quenched matrix outside the spherulite.

A dramatic transformation of the Q' map occurred immedi-

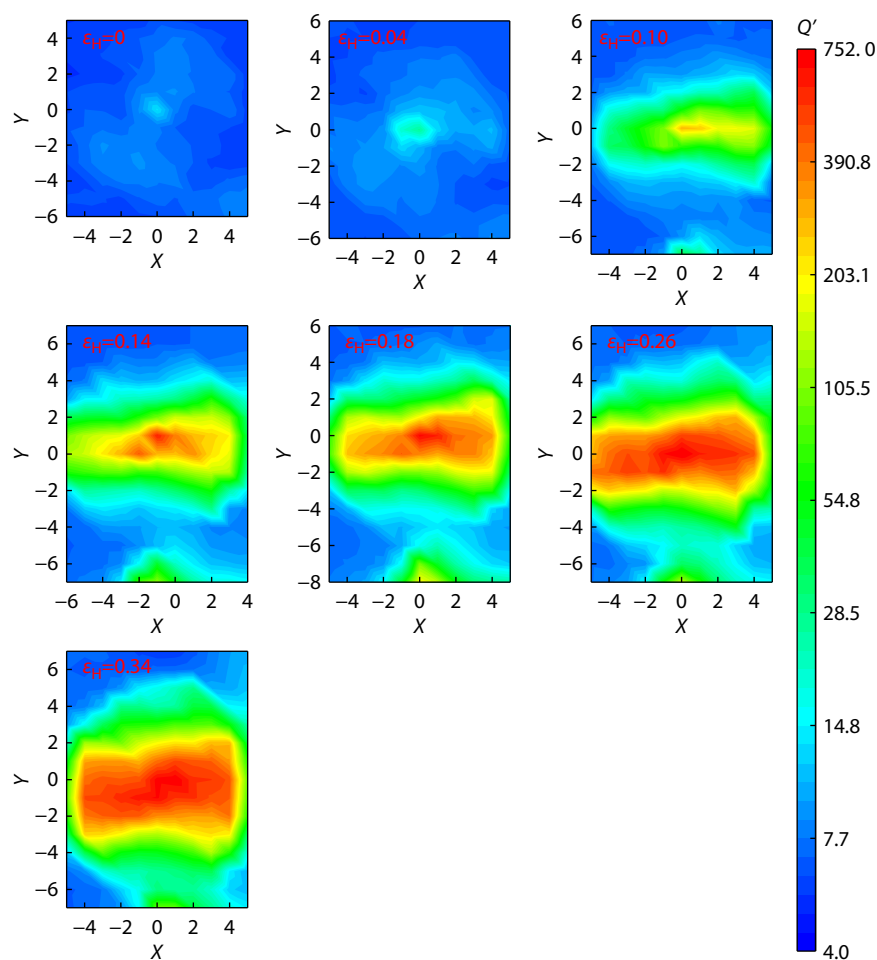


Fig. 8 Spatial distributions of the apparent scattering invariant (Q') in the spherulitic region measured at different strains. X and Y on the graphs indicate the scanning steps of 50 μm per step.

ately after yielding. At $\varepsilon_H = 0.10$, the Q' value at the center of the spherulite increases significantly, confirming that cavitation nucleates preferentially in this specific region. This behavior can be attributed to the confluence of structural and mechanical factors. Structurally, the spherulite center is not a continuation of the radial lamellar, but is often a sheaf-like bundle that acts as the growth origin. In continuum mechanics, such geometric discontinuity, which must transmit the applied load to the entire lamellar structure, inevitably becomes a point of stress concentration. Thus, the center experienced the highest local stress, triggering premature structural failure (cavitation). Concurrently, a notable, albeit small, increase in Q' was observed in the equatorial regions surrounding the center. While previous studies have noted a relatively lower degree of crystallinity at the spherulite center, our quantitative WAXD analysis of identically prepared samples revealed that this structural variance is marginal (approximately 58% crystallinity at the center versus 61% in the surrounding regions).^[32] Because the absolute crystallinity at the core remains sufficiently high to restrict large-scale ductile yielding, the severe stress concentration at the central sheaf-like discontinuity dominates this subtle crystallinity difference, acting as the primary driving force for void nucleation.

As the strain further increases ($\varepsilon_H = 0.18$ – 0.34), the Q' values continue to rise, and the region of high Q' (i.e., high void content) expands outward from the center. This propagation exhibits distinct spatial kinetics: while the voided zone extends simultaneously towards both the equatorial and polar region, it pervades the equatorial region significantly earlier. The polar regions exhibited a delayed response, remaining partially intact until they finally became nearly fully cavitated at a strain $\varepsilon_H = 0.34$.

To further elucidate the morphological characteristics, specifically the orientation of the voids, the apparent scattering invariant is decomposed into directional components. For each 2D pattern, apparent partial invariants were calculated by integrating the intensity along specific sectors: the apparent vertical partial invariant (Q'_V), representing scattering along the meridional direction (parallel to the stretching direction), and the apparent horizontal partial invariant (Q'_H), representing scattering along the equatorial direction (perpendicular to the stretching direction). The integration scheme is illustrated in Fig. 9.

The evolution of these parameters (Q'_V , Q'_H) and their ratio (Q'_V/Q'_H) for the equatorial and polar regions are presented in Fig. 10. The ratio Q'_V/Q'_H serves as a sensitive probe for struc-

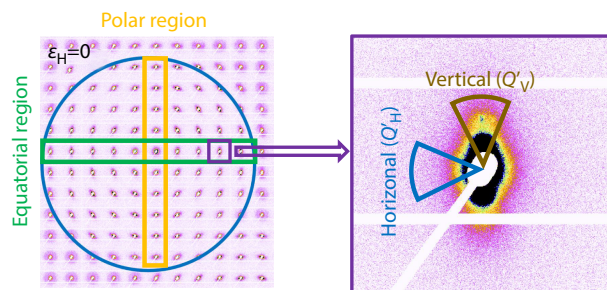


Fig. 9 Schematic diagram for the calculation of apparent scattering invariants in different directions.

tural anisotropy. Owing to the reciprocal nature of scattering, a ratio $Q'_V/Q'_H > 1$ indicates that the scattering signal is concentrated along the meridian. In real space, this implies that the dominant scattering interfaces (whether lamellae or voids) are oriented perpendicularly to the stretching direction. Conversely, the magnitude of this ratio reflects the degree of orientation, with a higher value corresponding to a higher degree of alignment. Crucially, the physical origin of the scattering contrast shifts during deformation: in the elastic region ($\epsilon_H \leq 0.04$), the ratio reflects the orientation of the crystalline lamellae, whereas after yielding ($\epsilon_H \geq 0.10$), due to the immense electron density contrast of voids, the ratio becomes a proxy for the orientation of the voids.

Focusing on the equatorial region (Fig. 10c), the analysis of the initial state ($\epsilon_H \leq 0.04$) revealed a significant radial dependence of the lamellar orientation. The Q'_V/Q'_H ratio, and thus the perfection of the tangential lamellar orientation, is relatively low at the spherulite center and increases progressively toward the periphery. This spatial heterogeneity can be rationalized by two distinct crystal growth mechanisms that operate at different stages of spherulite development.

First, the lower orientation at the center is a consequence of the geometric constraints associated with the nucleation stage. Spherulite growth was initiated in a primary sheaf. To transform from a point-like nucleus into a volume-filling three-dimensional spherical object, the dominant lamellae must undergo frequent noncrystallographic branching or splaying. While this splaying is a geometric necessity for space filling, the branching process is inherently stochastic, causing the daughter lamellae to deviate angularly from the orientation of the mother lamellae. Consequently, the central region is characterized by a high density of branching points and a broader distribution of lamellar normals, resulting in the observed lower orientational order.

Second, as the crystallization front propagates outward beyond the central core, the kinetic selection mechanism becomes dominant. In polymer crystallization, the crystals possess a specific crystallographic axis with the fastest growth. At the growth front, lamellae, whose fastest growth direction happens to coincide with the radial temperature gradient (the radius), possess a competitive advantage. These "favorably oriented" lamellae grow rapidly and occupy the available free space, while lamellae with divergent orientations grow slower and are eventually impinged upon or buried. This competitive growth process effectively filters out misaligned crystals. As a result, the further the growth front advances from the center, the more highly unified the lamellar

orientation becomes, leading to the observed increase in the Q'_V/Q'_H ratio toward the spherulite periphery.

As deformation proceeds into the plastic regime ($\epsilon_H \geq 0.10$), the Q'_V/Q'_H ratio in the equatorial region undergoes a shift in physical meaning, which predominantly reflects the orientation of the newly formed voids.

Two distinct trends were observed in these strains. First, regarding the spatial distribution, the void orientation Q'_V/Q'_H generally exceeds that of the precursor lamellae, yet it retains the same radial dependence: it is lower at the center and increases toward the periphery. This phenomenon indicated a strong structural memory effect. The cavitation process is templated by the underlying lamellar structure. In regions where the initial lamellar stacking is highly regular (the periphery of spherulite), the resulting lamellar failure produces voids that are equally well-aligned. Conversely, in the more disordered center, the chaotic arrangement of the lamellae leads to a more randomized void distribution. Thus, the degree of void ordering is intrinsically linked to, and is limited by the orientation of the precursor crystalline texture.

Second, regarding the temporal evolution, a monotonic decrease in the Q'_V/Q'_H ratio was observed with increasing strain across the equatorial radius. This suggests a progressive reduction in the anisotropy of the void population, which likely arises from the interplay of one mechanism, void geometry evolution. Initially, voids likely nucleate as high-aspect-ratio crack-like plates between fragmented crystal blocks. These sharp, plate-like defects possess extremely high orientation. However, as the plastic flow continues, the material between the voids deforms, causing the voids to open up in the transverse direction (blunting the tips) and evolve from plate-like to more equiaxed ellipsoids. This geometric change reduces scattering anisotropy.

Finally, the behavior in the polar regions (Fig. 10f) merits a distinct explanation. While the initial state showed a radial gradient similar to that of the equatorial region, the post-yield Q'_V/Q'_H values were notably lower than their equatorial counterparts. This quantitative difference is attributed to the superposition of the competing scattering signals. In the equatorial region, both the original lamellae and newly formed voids scatter along the meridian; thus, their signals reinforce each other, yielding a high ratio. In contrast, in the polar region, the original lamellae are stacked radially and scattered along the equator, while stress-induced voids still tend to form perpendicular to the stress and scatter along the meridian. Furthermore, the significant surge in Q'_H within the polar region after yielding indicates the formation of new structural interfaces parallel to the stretching direction. This phenomenon is likely attributed to the fibrils connecting both surfaces of the craze-like cavities (see Fig. 6 in Ref. [27]). Consequently, in the polar region, this enhanced equatorial scattering, combined with the residual scattering from the original lamellae (Q'_H), counteracts the void scattering (Q'_V), resulting in a suppressed Q'_V/Q'_H ratio compared to the equatorial region where the signals are additive.

In addition to orientation analysis, estimating the dimensions of these voids is crucial for understanding cavitation mechanisms. The scattering patterns in Fig. 4 exhibit a "streak-like" feature along the meridian, but with a finite

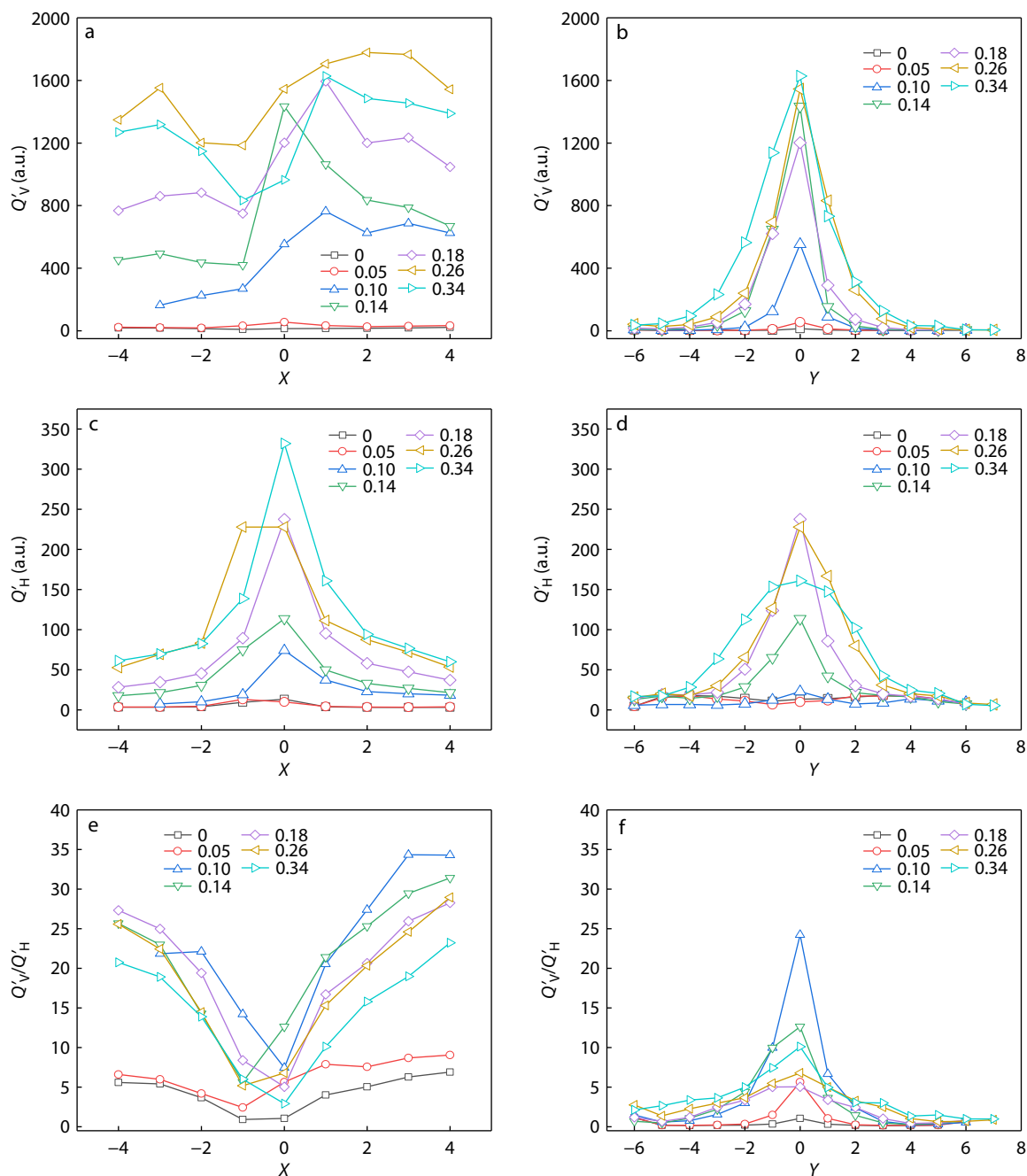


Fig. 10 Analysis of the integrated scattering intensities from the equatorial and polar regions of the *iPB-1* spherulite. (a, d) Integrated scattering intensities from the equatorial and polar regions taken parallel to the stretching direction (Q'_V), respectively. (b, e) Integrated scattering intensities from the equatorial and polar regions taken perpendicular to the stretching direction (Q'_H), respectively. (c, f) Ratio of the parallel to perpendicular integrated scattering intensity for the equatorial and polar regions (Q'_V/Q'_H), respectively.

width, suggesting that the voids can be approximated as oblate spheroids (platelike). In this geometry, the short axis (thickness) is parallel to the stretching direction, whereas the long axis (radius) lies perpendicular to the stretching direction.

Due to the spatial constraints of the microfocus setup, the sample-to-detector distance was fixed at 3244 mm. This configuration limits the minimum accessible scattering vector ($q_{\min}$), making it challenging to fully resolve scattering from large structures. Nevertheless, to obtain a semi-quantitative

estimation of the void thickness, we employed the Guinier approximation on the meridional intensity profiles extracted from the equatorial region which is define as:^[33]

$$I(q) = I(0) \exp\left(-\frac{q^2 R_g^2}{3}\right) \quad (9)$$

where $I(q)$ is the scattering intensity, $I(0)$ is the zero-angle intensity, and R_g is the radius of gyration.

The calculated R_g values for voids in the equatorial region are plotted in Fig. 11(a). It is important to explicitly state a lim-

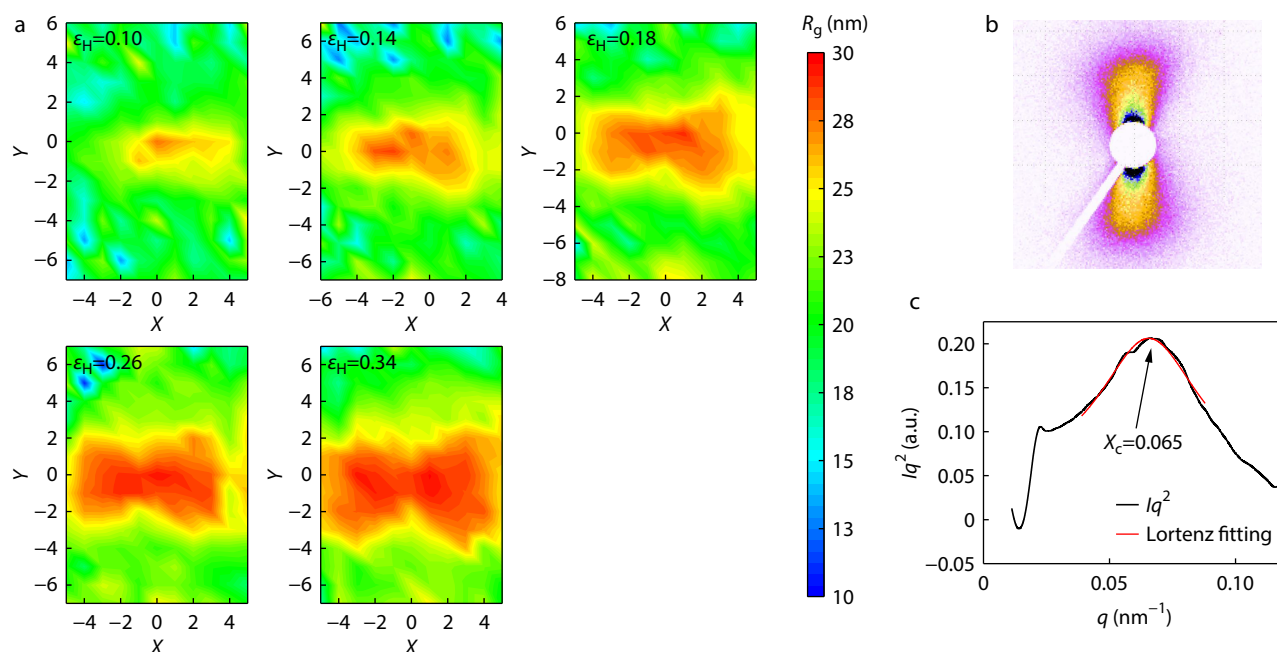


Fig. 11 (a) Spatial distributions of the void dimension obtained parallel to the stretching direction at different strains. (b) Two-dimensional USAXS pattern taken from the spherulite center. (c) Plot of $I(q)q^2$ versus q derived from the USAXS data for determining the void size parallel to the stretching direction.

itation: the calculated structural sizes result in qR_g values that generally exceed the strict validity range of the Guinier approximation ($qR_g < 1.0\text{--}1.3$). Consequently, the absolute values obtained here are likely to underestimate true dimensions. However, they serve as a valuable indicator of the relative scale and stability of the voids. The analysis yielded a consistent R_g of approximately 29 nm for stable voids.

Considering the plate-like geometry of the voids, the radius of gyration R_g along the thickness direction is related to the actual void thickness ($d_{\text{thickness}}$) by the geometric factor^[34]

$$d_{\text{thickness}} = \sqrt{12}R_g \quad (10)$$

By applying this correction, the estimated thickness of the voids is approximately 100 nm. Crucially, this dimension was significantly larger than the long period ($d_{ac} \approx 51$ nm) of the original spherulite. This magnitude implies that cavitation is not merely a separation of adjacent lamellae (which would yield voids comparable to interlamellar amorphous layers) but rather involves a massive disruption that spans multiple lamellar stacks.

It is worth noting that if the initial nucleated cavities possess a substantially different dimension prior to stabilization, distinct scattering signals representing intermediate or transitional void sizes should theoretically be detectable in the boundary zone between the cavitated and non-cavitated regions at lower strains. However, no such transitional signals were observed in our measurements, suggesting that once initiated, the structural disruption rapidly propagated, and the cavities suddenly achieved plate-like dimensions.

To overcome the resolution limit of SAXS and obtain a more accurate measurement of void size, USAXS measurements were performed. A larger X-ray beam (0.2 mm $\times$ 0.2 mm) was aimed at the spherulite center (where cavitation is

mostly severe) at a strain of $\epsilon_H = 0.34$. The resulting 2D USAXS pattern is shown in Fig. 11(b). From the Lorentz-corrected 1D profile ($I(q^2)$ versus q) (Fig. 11c), the characteristic void thickness was calculated using the peak position ($q_{\max}$) according to the form factor of the plate-like object:^[14]

$$d_{\text{thickness}} = \frac{3\pi}{q_{\max}} \quad (11)$$

USAXS analysis yielded a void thickness of approximately 140 nm. This value is larger than the SAXS-derived estimate (about 100 nm), confirming that the Guinier analysis of the SAXS data provided a reasonable lower-bound approximation. The agreement in the order of magnitude (100–140 nm) between these two independent techniques establishes that stress-induced voids are macroscopic defects relative to the lamellar scale, confirming the severe localized destruction of the crystalline framework.

To rationalize the aforementioned experimental observations, particularly the spatially dependent size and orientation distribution of the cavities, we propose a micromechanical cavitation model that is applicable to the small-strain regime for single spherulite in semi-crystalline polymers.

As schematically illustrated in Fig. 12, during uniaxial stretching, the initial lamellar system remains largely intact prior to macroscopic yielding. However, once the applied strain surpasses a critical threshold, the intrinsic "crystal-fix" nature of *i*PB-1 severely restricts the intracrystalline chain mobility, exhibiting pre-existing lamellar orientations that are practically unalterable. Consequently, energy dissipation *via* plastic deformation mechanisms such as crystallographic slip is kinetically hindered. Instead, the system resorts to an alternative energy-dissipation pathway. Specifically, the lamellae oriented parallel to the stretching direction, which are ubiquitously located in all regions of the spherulite as previously es-

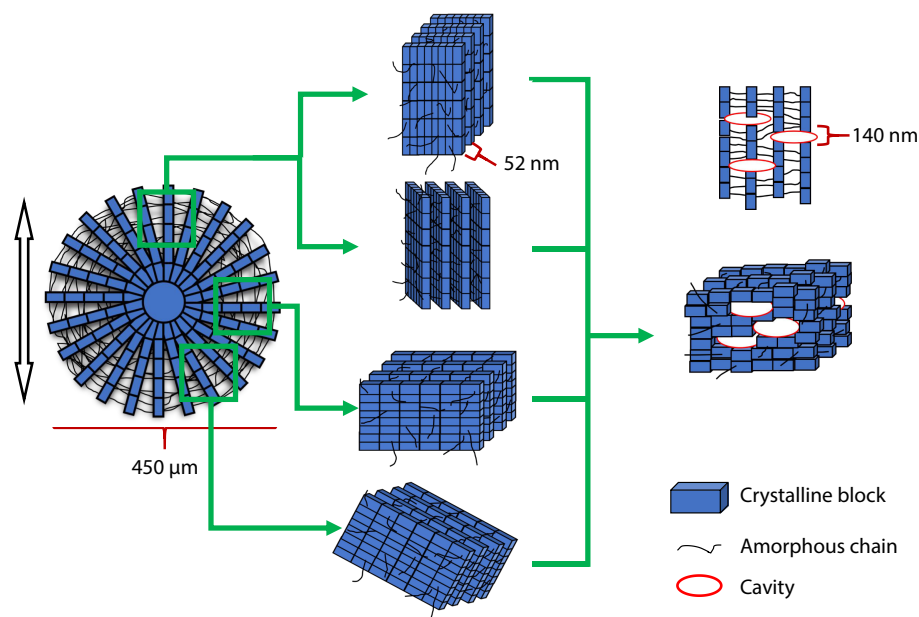


Fig. 12 Physical scenario for cavitation in *iPB*-1 spherulite during stretching of the spherulite (not to scale).

tablished, undergo mechanical fragmentation under the applied stress, with microvoids preferentially nucleating within the interstitial gaps between adjacent crystalline blocks. Upon initiation, these incipient cavities propagate and traverse across several lamellar long periods before structurally stabilizing into the characteristic plate-like morphologies.

To provide a comprehensive understanding of the cavitation mechanism, several crucial spatial sequences regarding the onset of cavitation must be addressed. First, the preferential nucleation of cavities at the center of the spherulite is primarily attributed to the localized stress concentration inherent to the radial structural architecture compounded by a relatively low degree of crystallinity. Furthermore, the phenomenon wherein early-stage cavitation emerges in the equatorial region prior to the polar region can be elucidated from a coupled macroscopic-microscopic perspective. Macroscopically, as evidenced by our strain calculations, the equatorial region of the spherulite inherently experiences maximum localized geometrical strain. Concurrently, at the microstructural level, the lamellae in the polar sectors were aligned parallel to the stretching direction. This highly unified parallel-load-bearing structural configuration effectively distributes the applied force, enabling the polar lamellar network to withstand substantially higher localized stresses without succumbing to immediate structural disintegration.

CONCLUSIONS

In summary, we systematically investigated the localized deformation mechanisms and microstructural origins of cavitation within individual *iPB*-1 spherulite during uniaxial stretching. By employing an integrated approach of *in situ* synchrotron micro-focus X-ray scattering, conventional ultrasmall-angle X-ray scattering, and polarized optical microscopy techniques, we demonstrated that early-stage cavitation exhibits a highly specific spatial sequence. Near the macroscopic yield point, cavitation was preferentially initiated at the exact center of the

spherulite. As the applied strain increased further, cavitation subsequently emerged in the equatorial regions and ultimately propagated to the polar regions. By correlating this distinct spatial failure sequence with the three-dimensional lamellar orientation mapped within the undeformed spherulite, we propose a micromechanical cavitation model. In this framework, the failure pathway is governed by lamellae oriented parallel to the stretching direction, which are extensively distributed across all the spherulitic regions. Rather than yielding *via* ductile slip, these parallel lamellae underwent direct mechanical fragmentation, with microvoids nucleating within the interstitial gaps between adjacent crystalline blocks. Ultimately, this work provides new experimental evidence to resolve the controversy regarding the spatial origin of yield-induced cavities.

Conflict of Interests

The authors declare no interest conflict.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at <http://doi.org/10.1007/s10118-026-3743-1>.

Data Availability Statement

The related data of this paper can be obtained from the author for reasonable reasons.

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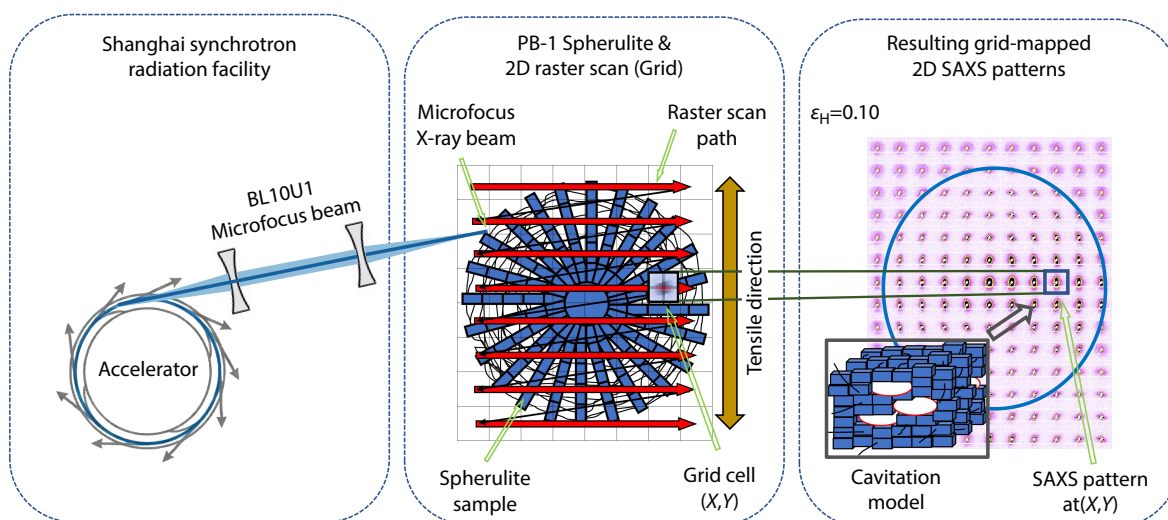
Graphical Abstract

Spatially Resolved Cavitation within Tensile Stretched Isotactic Polybutene-1 Spherulite

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By employing *in situ* synchrotron microfocus X-ray scattering on a single polybutene-1 (PB-1) spherulite, this study provides spatially resolved evidence that stress-induced cavitation occurs in the equatorial region and is primarily governed by the mechanical fragmentation of lamellae parallel to the stretching axis.



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