APPLIED SCIENCE

The International Journal of Applied Sciences.
Vol. 2, No. 1, 2026, pp. 62-88
ISSN 3104-8935

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In Situ WAXD Investigation of Temperature-Driven Microstructural Evolution in Metallocene Polyethylene Films under Stress Relaxation and Cyclic Fatigue

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DOI: https://doi.org/10.64440/IJAS/IJAS1002

ARTICLE INFO

Article history
Received Jan 24, 2026
Revised Jan 26, 2026
Accepted Apr 11, 2026

Keywords
Metallocene polyethylene films;
In situ wide-angle X-ray diffraction;
Stress relaxation;
Cyclic fatigue;
Crystalline-domain coupling;
Microstructural evolution;
Molecular orientation;
Crystallinity.

ABSTRACT

The microstructural evolution of metallocene polyethylene (mPE) films during stress relaxation and cyclic fatigue was investigated using in situ wide-angle X-ray diffraction (WAXD), with particular emphasis on the relationship between deformation history, crystalline-domain coupling, and structural reorganization. Two pre-strain levels, 0.15 and 0.91, were considered, corresponding respectively to a deformation below and slightly above the yield strain of approximately 0.8. The WAXD observations reveal distinct structural responses depending on the magnitude of the imposed pre-strain. At the higher pre-strain, extensive sliding between crystalline domains occurs during deformation, resulting in weakened inter-domain coupling and consequently lower energy dissipation during subsequent cyclic fatigue. In contrast, the lower pre-strain preserves stronger interactions between neighboring crystalline domains, requiring greater energy expenditure during fatigue to overcome the associated inter-domain constraints.

Cyclic fatigue progressively reduces the coupling between crystalline domains through repeated loading and unloading, thereby producing a lower stress state than that developed during stress relaxation. Regardless of the initial pre-strain, the crystallinity and molecular orientation of the films remain higher during cyclic fatigue than during stress relaxation. This behavior is attributed to the structural rearrangement induced by repeated mechanical loading, which promotes decoupling of crystalline domains and increases the mobility of molecular chains. Repeated stretching and recovery facilitate preferential alignment of polymer chains along the loading direction and promote the formation of ordered regions that can act as nuclei for further crystallization. Simultaneously, the reduced constraints between crystalline domains facilitate their preferential alignment along the applied stress field, resulting in enhanced structural orientation.The contribution of cyclic fatigue to orientation development becomes less pronounced at the higher pre-strain. This behavior is attributed to the relatively weak crystalline-domain coupling already established during the initial deformation, such that subsequent cyclic loading requires less energy to induce further decoupling. Consequently, the orientation content and degree of orientation attained during fatigue become increasingly comparable to those observed during stress relaxation. These findings demonstrate that the deformation history strongly governs the microstructural response of mPE films and provide a structural basis for understanding the coupled effects of crystalline-domain interactions, chain mobility, crystallization, and orientation during mechanical relaxation and fatigue.

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ISSN 3104-8935

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INTRODUCTION

Metallocene polyethylene (mPE) is a high-performance polyolefin produced using metallocene-based catalytic systems and has attracted considerable interest because of its precisely controlled molecular architecture and favorable combination of mechanical and functional properties [1–3]. Compared with polyethylene produced using conventional Ziegler–Natta catalysts, metallocene polyethylene generally exhibits a more uniform distribution of short-chain branches and a more regular sequence structure. This degree of molecular control can provide improved mechanical strength, optical transparency, processability, and heat-sealing performance [4–7]. These characteristics have contributed to the extensive use of mPE films in flexible food packaging, protective films for electronic components, and medical sterilization materials [8,9].

The performance of polyethylene films in these applications is determined not only by their initial macroscopic mechanical properties but also by their ability to withstand repeated deformation during service. Packaging films, protective membranes, and other thin polymeric materials are frequently subjected to sustained deformation, intermittent loading, and repeated stretching–recovery cycles. Such mechanical histories can produce stress relaxation, fatigue damage, and progressive changes in the internal crystalline structure, ultimately affecting dimensional stability, strength, barrier performance, and functional integrity [13,14].

The microstructure of mPE films is strongly influenced by processing conditions. Variations in film-blowing parameters, including draw ratio and cooling rate, can produce substantial differences in the initial crystallinity and molecular orientation of the resulting films [10,11]. Crystallinity contributes significantly to stiffness and load-bearing capability, whereas molecular orientation plays an important role in tensile strength and deformation behavior [11]. The anisotropic nature of blown polymer films is particularly relevant to practical applications because mechanical properties along the machine direction and transverse direction can differ considerably [12]. Consequently, understanding how the crystalline and oriented structures evolve under service-related deformation is essential for establishing reliable structure–property relationships.

At the microscopic level, polyethylene consists of crystalline regions embedded within a less ordered molecular environment. These crystalline regions are organized into higher-order structures and interact through inter-domain constraints and molecular connections. Mechanical deformation can modify the relative arrangement, orientation, and coupling of these crystalline domains. During stress relaxation, the polymer chains and crystalline domains progressively rearrange under a sustained deformation, resulting in a reduction of the macroscopic stress. Under cyclic fatigue, however, repeated loading and unloading provide a fundamentally different mechanical environment in which crystalline domains can progressively decouple, molecular chains can rearrange, and oriented structures can develop.

Previous investigations of mPE films have largely focused on processing optimization, static mechanical characterization, and structural characterization during film formation [10]. In situ small-angle and wide-angle X-ray scattering/diffraction techniques have provided valuable information about structural development during film processing and deformation [13]. However, the dynamic relationship between crystalline-domain rearrangement, molecular orientation, crystallization, and macroscopic mechanical response under repeated loading remains insufficiently understood [15,16].

An additional factor that can strongly influence polymer-chain mobility and crystalline-domain stability is **temperature**. Temperature modifies segmental mobility, intermolecular interactions, crystalline–amorphous phase dynamics, and the ability of molecular chains to rearrange under an

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applied mechanical field. Consequently, the microstructural response observed during stress relaxation or cyclic fatigue at one temperature cannot necessarily be assumed to represent the behavior of the material under different thermal conditions. Introducing temperature as an experimental variable therefore provides an opportunity to distinguish deformation-induced structural changes from thermally activated molecular rearrangement.

In particular, temperature may influence the balance between crystalline-domain coupling and decoupling. At relatively lower temperatures, restricted molecular mobility may preserve stronger inter-domain constraints and limit structural rearrangement. As temperature increases, enhanced molecular mobility may facilitate chain relaxation, crystalline-domain rearrangement, and the development or loss of preferential orientation. The resulting changes can subsequently affect the stress carried by the material during relaxation and the energy dissipated during cyclic fatigue.

Wide-angle X-ray diffraction (WAXD) is particularly suitable for investigating these processes because it provides direct information about the crystalline lattice, crystallinity, and molecular orientation of semicrystalline polymers. When coupled with an in situ mechanical testing system, WAXD enables the evolution of the crystalline structure to be monitored during mechanical deformation rather than only before and after testing. This approach makes it possible to establish a direct relationship between changes in the microscopic crystalline structure and the macroscopic mechanical response.

In the present study, in situ WAXD is employed to investigate the temperature-dependent evolution of the microstructure of metallocene polyethylene films during stress relaxation and cyclic fatigue. Particular attention is given to the effects of deformation history and pre-strain level on crystalline-domain coupling, crystallinity, and molecular orientation. Two initial pre-strain conditions, 0.15 and 0.91, representing deformation below and slightly above the yield strain of approximately 0.8, are considered. The introduction of temperature as an additional variable is intended to clarify how thermal activation modifies the structural response of the polymer under sustained and cyclic mechanical loading.

The study is designed to establish a structure–mechanics framework linking **temperature, pre-strain, crystalline-domain coupling, crystallinity, molecular orientation, and macroscopic stress response**. Particular emphasis is placed on determining whether temperature modifies the extent to which cyclic fatigue promotes crystalline-domain decoupling and molecular alignment and whether these structural changes differ from those occurring during stress relaxation. Such information is expected to provide a more comprehensive understanding of the service behavior of mPE films and to support the optimization of their mechanical reliability under combined thermal and cyclic mechanical conditions.

2. Experimental

2.1. Materials and instrumentation

Metallocene linear low-density polyethylene (mMLLDPE) film was selected as the model semicrystalline polymer. The material had a number-average molecular weight ((M$_n$)) of (1.78 \times 10^5) and a weight-average molecular weight ((M$_w$)) of (5.23 \times 10^5), with 4.2 mol% hexene

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incorporated as the comonomer. The material was processed into thin films using a conventional blown-film process.

Small-angle X-ray scattering (SAXS) measurements were performed using a Xeuss 2.0 system equipped with a multilayer-focused Cu Kα X-ray source (GeniX3D Cu ULD) and a Pilatus 100K detector. In situ wide-angle X-ray diffraction measurements were conducted using a microfocus X-ray system coupled to a miniature in situ mechanical testing platform. The mechanical deformation and X-ray measurements were synchronized to enable structural monitoring during stress relaxation and cyclic fatigue.

2.2. Film preparation

The mMLLDPE films were prepared by the blown-film process using a blow-up ratio of approximately 2.5–3.0, a die diameter greater than 12 cm, a processing temperature of 180–190 °C, and a take-up speed of 14–15 m min^{-1}. The resulting film thickness was approximately 30 μm.

All tensile deformation experiments were conducted along the machine direction (MD), corresponding to the principal draw direction of the blown film. Dog-bone specimens were prepared from the film using a punching die. The effective gauge dimensions of the specimens were approximately 10 mm × 5 mm × 0.03 mm.

The use of the machine direction as the loading direction was maintained throughout the study to ensure consistency between the initial processing-induced molecular orientation and the applied mechanical field.

2.3. Initial microstructural characterization

Prior to mechanical testing, the initial hierarchical structure of the mMLLDPE film was characterized using SAXS and WAXD.

For SAXS measurements, the X-ray wavelength was 0.154 nm and the sample-to-detector distance was 1046 mm. Two-dimensional SAXS patterns were collected with an exposure time of 120 s. The SAXS measurements were used to characterize the organization and orientation of the higher-order lamellar structure before mechanical deformation.

WAXD measurements were subsequently used to characterize the crystalline lattice and molecular orientation of the untreated film. The WAXD measurements employed an X-ray wavelength of 0.154 nm, with an X-ray beam size at the sample of approximately 0.04 mm × 0.06 mm. The sample-to-detector distance was 59.3 mm, providing an effective diffraction-angle range of approximately (2\theta = 8–28^\circ). Each WAXD pattern was collected using a 400 s exposure with a Pilatus 100K detector.

Background contributions were subtracted from all WAXD patterns before quantitative analysis.

The initial SAXS pattern showed concentration of scattering intensity along the machine direction, indicating a pronounced orientation of the lamellar structure. In contrast, the characteristic (110) and (200) reflections observed in the initial WAXD pattern exhibited an approximately uniform azimuthal distribution, indicating that preferential orientation at the unit-cell/crystalline-lattice level was comparatively weak before mechanical loading.

2.4. In situ WAXD measurements during mechanical deformation

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The WAXD system was coupled directly to the miniature mechanical testing platform to monitor structural changes during mechanical loading. This configuration enabled successive WAXD patterns to be collected while the polymer film underwent stress relaxation or cyclic fatigue.

The two principal pre-strain conditions used in the original deformation protocol were 0.15 and 0.91. These values represent deformation below and slightly above the yield strain, respectively, with the yield strain being approximately 0.8. The comparison between these two deformation states allows the influence of deformation history and crystalline-domain coupling on subsequent structural evolution to be evaluated.

During stress-relaxation experiments, the specimen was rapidly deformed to the prescribed pre-strain and subsequently maintained at the specified deformation while the evolution of the macroscopic stress and WAXD pattern was monitored.

During cyclic-fatigue experiments, the specimen was subjected to repeated loading and unloading around the prescribed deformation condition. Sequential WAXD measurements were used to monitor changes in crystalline structure and orientation during the repeated deformation cycles.

2.5. Temperature-dependent experimental design

Temperature was introduced as the principal additional experimental variable in the developed study. Mechanical and WAXD measurements are to be conducted under controlled thermal conditions while maintaining the same specimen geometry, pre-strain level, loading protocol, and X-ray acquisition parameters.

For each selected temperature, stress-relaxation and cyclic-fatigue experiments should be performed independently at the two pre-strain levels of 0.15 and 0.91. This factorial arrangement allows the individual and combined effects of temperature and deformation history to be distinguished.

The temperature-dependent measurements should be performed after sufficient thermal equilibration of the specimen to ensure that the polymer reaches a stable and uniform test temperature before mechanical loading. The selected temperature levels should be reported explicitly with the corresponding experimental data because the original study does not provide numerical temperature conditions beyond the film-processing temperature.

The principal variables obtained from the temperature-dependent WAXD measurements should include:

1. **Crystallinity or relative crystalline content**, determined from the integrated crystalline and amorphous contributions of the diffraction profile.
2. **Crystalline orientation**, determined from the azimuthal distribution of the characteristic diffraction reflections.
3. **Orientation degree**, quantified from the azimuthal intensity distribution using an appropriate orientation parameter.
4. **Diffraction peak position and width**, used to evaluate changes in crystalline lattice characteristics and structural disorder.

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5. **Macroscopic stress evolution**, recorded simultaneously during stress relaxation and cyclic fatigue.
6. **Energy dissipation during cyclic loading**, calculated from the mechanical hysteresis response where complete loading–unloading data are available.

This experimental framework enables the structural consequences of temperature to be separated from those produced solely by mechanical pre-strain.

2.6. Analysis of WAXD patterns

The WAXD patterns were analyzed after background subtraction to determine changes in crystalline structure and orientation during mechanical deformation. The characteristic diffraction reflections of the polyethylene crystalline phase were used to follow structural changes throughout the relaxation and fatigue processes.

The azimuthal distribution of the diffraction intensity was used to quantify molecular/crystalline orientation relative to the machine direction. Changes in the integrated intensity and azimuthal distribution of the diffraction peaks were compared between stress-relaxation and cyclic-fatigue conditions.

The structural parameters were subsequently correlated with the simultaneously measured macroscopic mechanical response. This correlation was used to determine whether reductions in stress and changes in energy dissipation were accompanied by crystalline-domain decoupling, increased chain mobility, enhanced crystallization, or preferential molecular alignment.

2.7. Experimental strategy

The developed experimental strategy can therefore be summarized as:

Temperature → molecular mobility → crystalline-domain coupling/decoupling → crystallinity and molecular orientation → macroscopic stress relaxation and fatigue response.

The two pre-strain levels provide the deformation-history dimension of the study, while temperature provides the newly introduced thermal dimension. In situ WAXD serves as the structural probe connecting these variables to the evolution of the semicrystalline morphology.

This design makes it possible to determine whether the structural mechanisms responsible for stress relaxation and cyclic fatigue remain consistent across thermal conditions or whether increasing temperature changes the balance between crystalline-domain constraints, molecular mobility, crystallization, and orientation.

3. Mechanical Characterization and Temperature-Dependent Structural Analysis

3.1. Mechanical testing protocol

Uniaxial tensile deformation, stress-relaxation, and cyclic-fatigue experiments were performed using a miniature in situ mechanical testing system coupled with the WAXD measurement platform. In the original experimental configuration, the measurements were conducted at room temperature. In the developed experimental framework, temperature is introduced as an additional controlled variable to investigate its influence on the mechanical and microstructural response of the mMLLDPE films.

For the uniaxial tensile test, specimens were stretched at a constant displacement rate of 50 μm s^{-1} until fracture. The resulting engineering stress–strain response was used to identify the elastic,

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yielding, strain-softening, and strain-hardening regions and to determine the characteristic yield strain used for subsequent relaxation and fatigue experiments.

For stress-relaxation measurements, the specimen was rapidly deformed to a prescribed initial strain and subsequently held at a constant strain. The load was continuously recorded for 10,000 s. Two initial strain levels, 0.15 and 0.91, were selected to represent deformation below and slightly above the yield strain, respectively.

For cyclic-fatigue measurements, the specimen was first stretched to the prescribed initial strain and then subjected to repeated loading and unloading with a strain amplitude of 0.1 and a frequency of 0.1 Hz. The total duration of each fatigue experiment was 10,000 s.

For the temperature-dependent investigation, the same mechanical protocol should be maintained at each selected temperature. This allows the effects of thermal activation to be separated from those associated with pre-strain and cyclic deformation.

3. Results and Discussion

3.1. Tensile behavior and selection of pre-strain levels

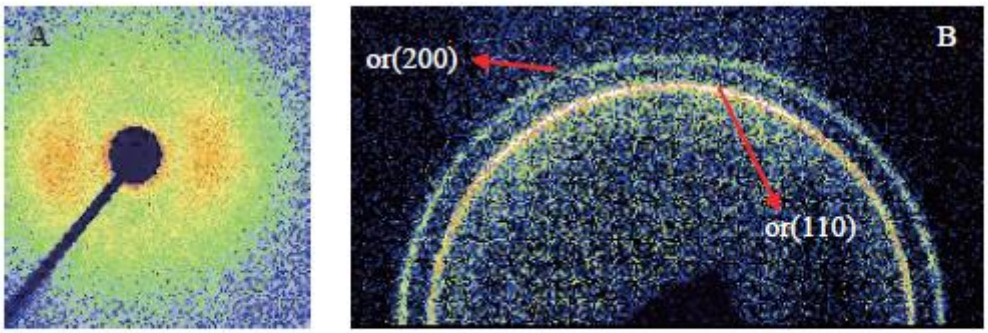

Fig. 1 SAXS (A) and WAXD (B) patterns of the film, machine direction is horizontal

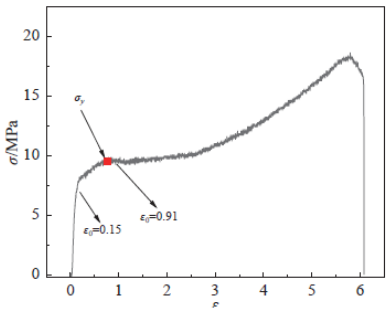

Fig. 2 Engineering stress-strain curve of the sample. The red dot represents the yield point. Two pre-strain values of 0. 15 and 0. 91 were selected to conduct the subsequent stress relaxation and fatigue measurements

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Figure1, Figure 2: presents the engineering stress–strain response of the mMLLDPE film. The deformation process can be divided into several characteristic regimes, including an initial elastic region, yielding, strain softening, and subsequent strain hardening before final fracture. The specimen fractured at a strain of approximately 6, while yielding occurred at a strain of approximately 0.8 with a corresponding yield stress of about 10 MPa.

The yield behavior provides an important reference for interpreting the subsequent structural evolution. Accordingly, two initial pre-strain levels were selected: 0.15, corresponding to deformation well below the yield point, and 0.91, corresponding to deformation slightly above the yield point. These two conditions provide substantially different initial states of crystalline-domain coupling and molecular orientation.

At the lower pre-strain, most of the crystalline-domain network remains mechanically constrained, whereas deformation beyond the yield point is expected to induce substantial structural rearrangement, including crystalline-domain sliding and partial decoupling. The comparison between these two states therefore provides a means of distinguishing reversible molecular relaxation from deformation-induced structural reorganization.

Importantly, the newly introduced temperature variable is expected to modify this response by controlling molecular mobility and the kinetics of structural rearrangement. Thus, the mechanical response at a given pre-strain should be interpreted as the combined consequence of **initial deformation level and thermal activation**.

3.2. Cyclic-fatigue response and energy dissipation

During cyclic fatigue, the viscoelastic nature of the mMLLDPE film produces a pronounced hysteresis between the loading and unloading branches. A representative series of stress–strain hysteresis loops is shown in Figure 3A.

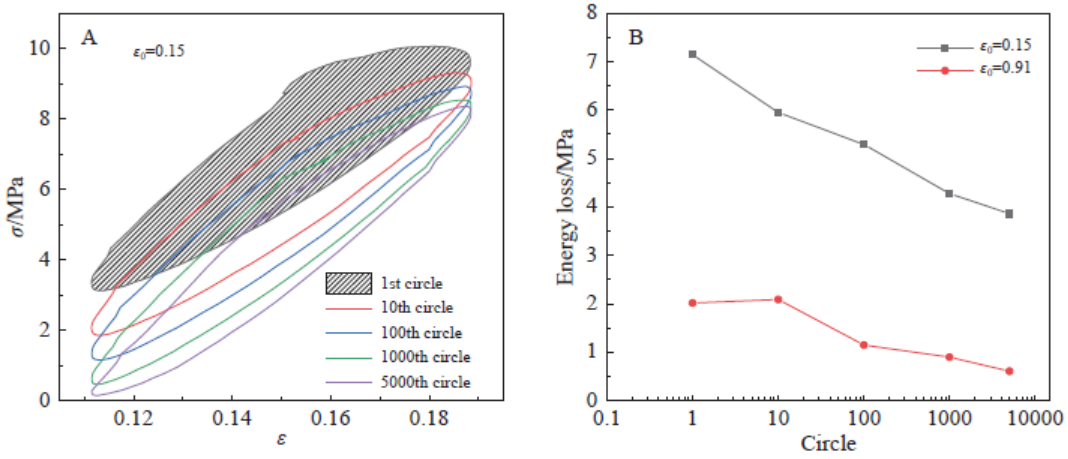

Fig. 3 (A) Stress-strain hysteresis loops of the sample at selected fatigue cycles; (B) Evolution of energy dissipation per unit volume during fatigue testing

Two important features can be identified. First, both the maximum and minimum stresses progressively decrease with increasing fatigue cycles, indicating that stress relaxation occurs

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simultaneously with cyclic loading. Second, the area enclosed by each hysteresis loop decreases progressively during fatigue, demonstrating a reduction in energy dissipation.

The hysteresis-loop area represents the mechanical energy dissipated per unit volume during each loading cycle. The progressive decrease in energy loss indicates that the internal constraints responsible for molecular friction and structural resistance become progressively weaker during repeated deformation.

At the microscopic level, the semicrystalline polymer can be regarded as an interconnected network consisting of crystalline regions and amorphous regions. Adjacent crystalline domains may interact through tie molecules, intermolecular forces, and physical constraints, forming a mechanically coupled crystalline framework. During repeated deformation, these interactions can progressively weaken as crystalline domains undergo relative displacement or sliding.

This structural decoupling increases the mobility of molecular chains and reduces the mechanical work required for subsequent deformation. Consequently, the energy dissipated during each cycle decreases progressively.

A pronounced difference is observed between the two pre-strain conditions. The energy loss at the higher pre-strain is substantially lower than that observed at the lower pre-strain. This behavior suggests that a considerable portion of the crystalline-domain decoupling has already occurred during the initial deformation to 0.91. Consequently, fewer internal constraints remain to be overcome during subsequent cyclic loading.

At 0.15 pre-strain, in contrast, the crystalline domains remain more strongly coupled. Repeated loading therefore requires additional mechanical work to overcome inter-domain interactions and promote molecular rearrangement, resulting in greater energy dissipation.

Temperature-dependent interpretation

Temperature is expected to exert a direct influence on this mechanism. Increasing temperature generally enhances segmental mobility and facilitates molecular rearrangement within the amorphous and interfacial regions. Consequently, the temperature dependence of hysteresis-loop area can provide a direct measure of the extent to which thermal activation assists crystalline-domain decoupling.

Accordingly, the relationship can be expressed conceptually as:

Temperature → molecular mobility → crystalline-domain decoupling → reduced internal constraint → energy dissipation.

The temperature-dependent fatigue experiments should therefore be used to determine whether thermal activation accelerates the decrease in hysteresis-loop area and whether this effect is more pronounced at the lower or higher pre-strain.

3.3. Comparison between stress relaxation and cyclic fatigue

Because cyclic fatigue is accompanied by progressive stress relaxation, the average stress during fatigue,

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can be directly compared with the stress measured during the corresponding static stress-relaxation experiment.

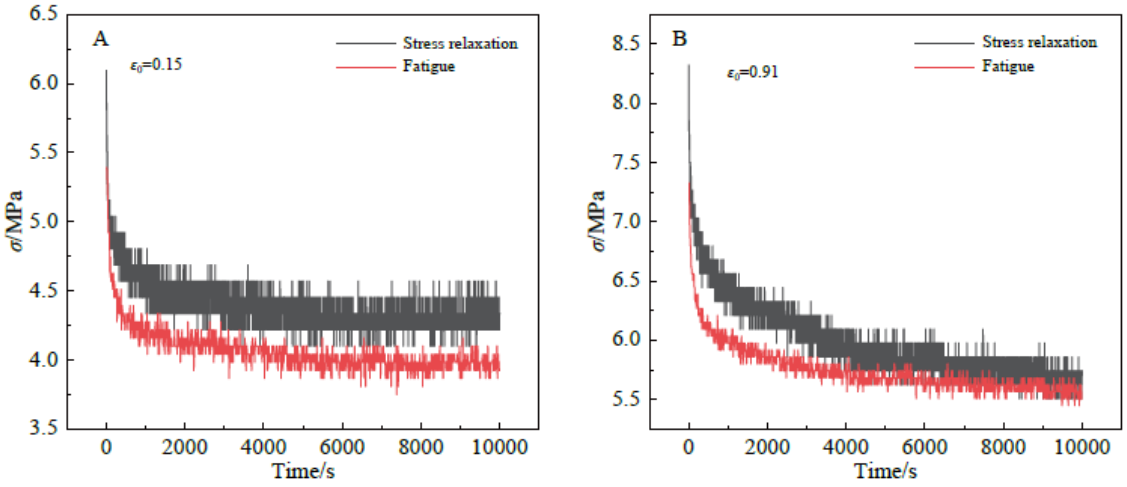

Fig. 4 Stress decay profiles of the sample during stress relaxation and fatigue testing with pre-strain levels of 0. 15 (A) and 0. 91 (B)

Figures 4A and 4B compare the stress-decay behavior at the lower and higher pre-strain levels, respectively.

For both deformation conditions, stress decreases rapidly during the initial stage and subsequently approaches a quasi-stationary state. The rate of stress reduction becomes considerably smaller after approximately 4000 s, indicating that the dominant relaxation processes occur during the early stage of deformation.

The higher pre-strain produces a higher long-time stress than the lower pre-strain. This behavior reflects the substantial structural rearrangement induced by deformation beyond the yield point. Although crystalline-domain coupling is weakened by the larger deformation, the resulting oriented and mechanically interconnected structure can continue to sustain a relatively high stress.

A further important observation is that the average stress during cyclic fatigue remains slightly below the stress observed during static relaxation under comparable pre-strain conditions. This difference can be attributed to the repeated mechanical perturbation imposed during fatigue.

Under small deformation, the crystalline framework contributes substantially to the load-bearing capacity of the semicrystalline polymer. During cyclic loading, repeated movement of this framework can weaken inter-domain coupling and induce limited crystalline-domain sliding. The resulting structural decoupling reduces the effective constraints on the molecular network and consequently lowers the stress required to maintain the deformation.

The difference between fatigue and static relaxation becomes less pronounced at the higher pre-strain. This can be explained by the fact that substantial crystalline-domain sliding and decoupling have already occurred during the initial deformation beyond the yield point. Therefore, cyclic loading has a smaller additional effect on crystalline-domain coupling.

Effect of temperature on stress relaxation

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The introduction of temperature provides an additional mechanism for controlling the relaxation rate. At elevated temperature, increased molecular mobility is expected to facilitate chain rearrangement and relaxation of internal stresses. Consequently, the stress-decay kinetics should be analyzed as a function of both time and temperature.

A temperature-dependent relaxation analysis can therefore be represented as:

where represents temperature and represents the initial pre-strain.

This formulation enables the respective contributions of **thermal activation and deformation history** to be separated.

3.4. In situ WAXD characterization during cyclic fatigue

In situ WAXD was employed to monitor changes in the crystalline lattice during stress relaxation and cyclic fatigue.

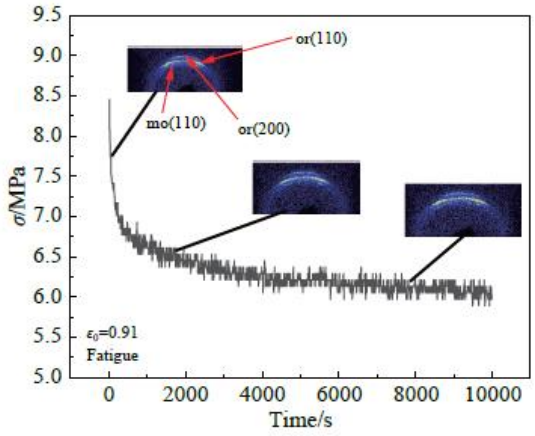

Fig. 5 Stress decay profile of the sample under cyclic fatigue loading and the corresponding two-dimensional WAXD patterns acquired at three selected time intervals. Stretching direction is horizontal

Figure 5 presents representative two-dimensional WAXD patterns obtained during cyclic fatigue at the higher pre-strain. Following deformation, the diffraction intensity becomes concentrated along the meridional direction perpendicular to the loading axis, indicating the development of pronounced crystalline orientation.

The principal diffraction reflections can be assigned to the monoclinic and orthorhombic crystalline structures of polyethylene, including the monoclinic , orthorhombic , and orthorhombic reflections.

The appearance of the monoclinic reflection at the higher pre-strain is particularly significant because this phase is not clearly detected in the undeformed material. Its emergence indicates that deformation beyond the yield point induces a crystalline-phase transformation.

Interestingly, although the macroscopic stress decreases substantially during fatigue, the two-dimensional WAXD patterns exhibit relatively limited changes throughout the subsequent fatigue

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process. This observation indicates that the basic crystalline lattice structure becomes comparatively stable after the initial deformation-induced transformation.

The result suggests that the major structural transformation occurs during the pre-straining stage, whereas subsequent cyclic fatigue primarily modifies the coupling, orientation, and mobility of existing crystalline domains rather than producing substantial additional phase transformation.

3.5. One-dimensional WAXD profiles and crystalline-phase stability

The two-dimensional WAXD patterns were azimuthally integrated to obtain one-dimensional diffraction profiles. Figures 6A and 6B show representative WAXD profiles for the lower and higher pre-strain conditions, respectively.

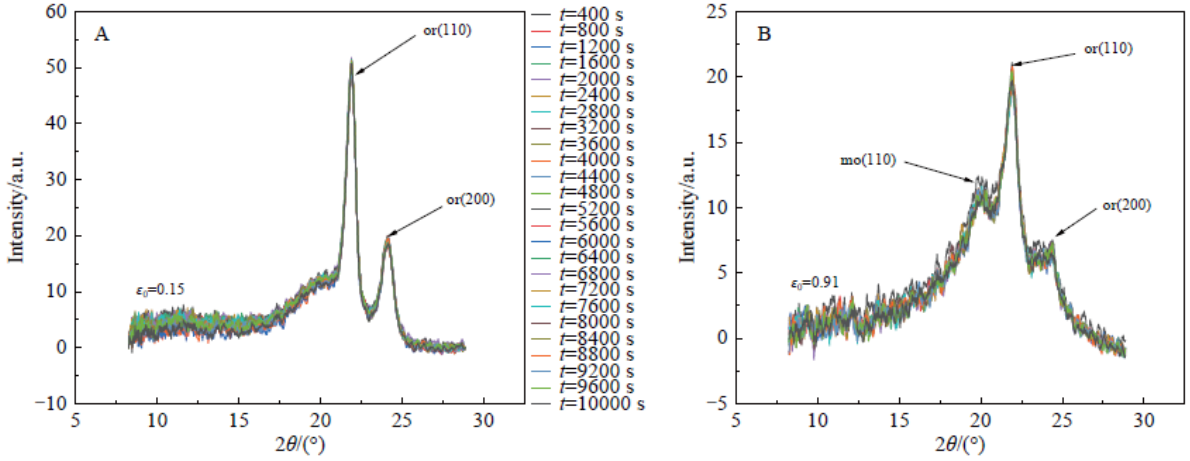

Fig. 6 One-dimensional WAXD curves of the sample during fatigue testing at pre-strains of (A) 0. 15 and (B) 0. 91

The profile obtained at the higher pre-strain contains an additional reflection compared with that obtained at the lower pre-strain. This observation provides further evidence that deformation beyond the yield point promotes a crystalline-phase transformation.

During subsequent cyclic fatigue, neither the diffraction-peak positions nor their relative intensities exhibit substantial changes. Therefore, the crystalline-phase composition remains comparatively stable during fatigue.

The limited amount of monoclinic phase and its formation primarily during the initial pre-straining stage suggest that the monoclinic phase itself is unlikely to be the dominant factor controlling the subsequent fatigue response. Instead, the principal structural mechanisms are more plausibly associated with crystalline-domain coupling, molecular orientation, and chain mobility.

This distinction is important for understanding the newly introduced temperature variable. Temperature may influence molecular mobility and interfacial rearrangement without necessarily inducing a comparable crystalline-phase transformation. Therefore, changes in fatigue behavior with temperature should be interpreted separately from changes associated with phase transformation.

3.6. Evolution of crystallinity during stress relaxation and cyclic fatigue

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The one-dimensional WAXD profiles were fitted to separate crystalline and non-crystalline contributions, and the degree of crystallinity was estimated from the ratio of the integrated crystalline contribution to the total scattering area.

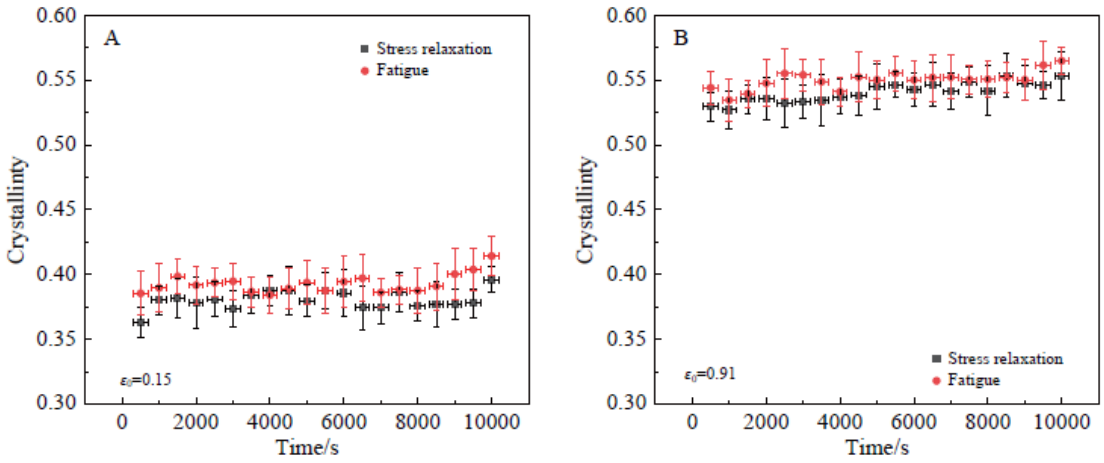

Fig. 7 Evolution of crystallinity of the sample during fatigue and stress relaxation process at pre-strain levels of (A) 0. 15 and (B) 0. 91

Figures 7A and 7B illustrate the evolution of crystallinity under the lower and higher pre-strain conditions, respectively. The experimental uncertainty associated with each point represents the averaging window corresponding to approximately 400 s.

During the initial stage of deformation, the crystallinity remains relatively stable. However, the crystallinity observed during cyclic fatigue is slightly higher than that obtained during static stress relaxation.

This difference can be attributed to repeated stretching and recovery. Cyclic deformation promotes preferential alignment of polymer chains along the loading direction, thereby increasing the probability that locally ordered molecular segments will form additional crystalline nuclei. Consequently, repeated mechanical deformation may facilitate limited strain-induced crystallization.

An important observation is that higher crystallinity does not necessarily correspond to greater energy dissipation. Under the higher pre-strain, the specimen exhibits a higher crystalline content while simultaneously displaying lower hysteretic energy loss.

This apparently counterintuitive behavior indicates that energy dissipation is not governed primarily by the absolute crystallinity of the material. Instead, it is more strongly related to the degree of **crystalline-domain coupling and decoupling**.

When the film is initially stretched beyond the yield point, substantial crystalline-domain sliding occurs. The resulting decoupling reduces the internal constraints that must subsequently be overcome during cyclic loading. Therefore, even though cyclic deformation can maintain or slightly increase crystallinity, the energy required for subsequent structural rearrangement remains relatively low.

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Temperature-driven crystallization response

The addition of temperature provides an important extension of this interpretation. Thermal activation can influence the competition between chain mobility, crystallization, and structural relaxation. Therefore, the temperature-dependent crystallinity should not be interpreted independently of orientation and crystalline-domain coupling.

The key question is not simply whether increasing temperature increases or decreases crystallinity, but rather:

How does temperature modify the relationship between crystallinity and the mechanical energy dissipated during cyclic deformation?

This distinction provides a stronger scientific basis for the proposed temperature-dependent structure–property relationship.

3.7. Suppression of self-heating during cyclic fatigue

Because cyclic mechanical loading can potentially generate heat through viscoelastic dissipation, temperature changes caused by self-heating must be distinguished from the externally controlled temperature variable.

The original fatigue experiments were performed at a relatively low frequency of 0.1 Hz. Under these conditions, substantial heat accumulation was not expected because the generated heat can dissipate rapidly into the surrounding environment.

To verify this assumption, the surface temperature of the specimen was monitored in real time using an infrared thermal imaging camera. The temperature remained essentially stable throughout the fatigue experiment, and no pronounced thermal accumulation was detected.

This observation is particularly important for the temperature-dependent extension of the study because it confirms that externally controlled temperature can be treated as an independent experimental variable rather than being strongly coupled to internally generated fatigue heating.

3.8. Evolution of crystalline orientation

Crystalline orientation represents another key structural parameter governing the mechanical behavior of semicrystalline polyethylene.

To quantitatively characterize the orientation of the crystalline planes, the Hermans orientation function was employed:

where

Here, represents the angle between the loading direction and the normal to the plane, while represents the azimuthal intensity distribution of the corresponding diffraction reflection.

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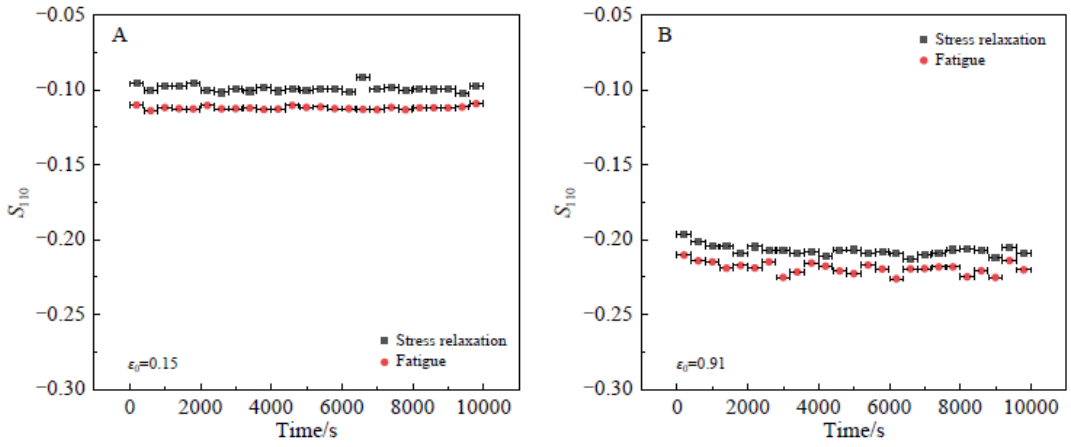

Fig. 8 Evolution of the degree of orientation of the sample during fatigue and stress relaxation process at pre-strain levels of (A) 0. 15 and (B) 0. 91

Figures 8A and 8B present the temporal evolution of at the lower and higher pre-strain levels, respectively.

At the lower pre-strain, the orientation parameter remains comparatively stable throughout the experiment, indicating that the initial crystalline orientation is relatively resistant to subsequent structural rearrangement.

At the higher pre-strain, however, a pronounced change in orientation occurs during the initial stage. The decrease in corresponds to an increase in the preferential orientation of the crystalline domains with respect to the loading direction.

This behavior can be explained by deformation-induced crystalline-domain sliding. When the film is stretched beyond its yield point, partial decoupling between crystalline domains permits previously constrained domains to rearrange more freely. During subsequent stress relaxation and cyclic fatigue, these less-constrained domains can progressively align with the applied stress field.

After approximately 4000 s, the orientation parameter approaches a relatively stable state, indicating that the major orientation rearrangement occurs during the early stage of relaxation.

The orientation observed during cyclic fatigue is consistently greater than that during static stress relaxation. Repeated loading and unloading continuously perturb the crystalline framework, weakening inter-domain coupling and promoting preferential alignment of partially decoupled crystalline domains along the direction of applied force.

3.9. Temperature-dependent orientation mechanism

The temperature variable provides a further explanation for the evolution of orientation.

Molecular orientation is governed by the competition between two processes:

1. **mechanically induced alignment**, which tends to orient polymer chains and crystalline domains along the applied stress direction; and

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2. **thermally activated relaxation**, which promotes molecular rearrangement toward less constrained configurations.

Temperature therefore has the potential to alter the rate at which orientation develops and the extent to which the resulting orientation is retained.

A useful conceptual framework for the temperature-dependent orientation response is:

The temperature-dependent WAXD measurements should consequently be used to determine whether increased thermal mobility accelerates orientation development during cyclic fatigue or instead promotes relaxation of the mechanically induced orientation.

This distinction is particularly important for separating **orientation kinetics** from the final equilibrium orientation state.

3.10. Oriented and unoriented structural fractions

The presence of a measurable unoriented component, even at relatively high deformation, indicates that the microstructural state cannot be described solely by a single orientation parameter.

The overall structural state depends on both:

- the **degree of orientation**, and
- the **fraction of material participating in the oriented population**.

Therefore, the WAXD patterns were further analyzed using the Halo method to separate oriented and unoriented structural contributions.

This analysis provides additional information about the mechanism of structural evolution. A material may exhibit a high orientation parameter while still retaining a considerable unoriented fraction. Conversely, an increase in the oriented fraction can occur without a proportionally large change in the orientation parameter.

The combination of these two parameters is therefore more informative than either parameter alone.

In the context of the developed temperature-dependent study, the oriented fraction can be used to determine whether temperature primarily promotes:

- reorientation of already oriented crystalline domains,
- recruitment of previously unoriented domains into the oriented population,
- relaxation of existing orientation, or
- increased molecular mobility without substantial changes in the crystalline orientation.

3.11. Integrated structure–mechanics mechanism

The combined mechanical and WAXD observations support a hierarchical mechanism for the response of mMLLDPE films.

At low pre-strain, the crystalline domains remain relatively strongly coupled. During cyclic fatigue, repeated loading must therefore overcome these inter-domain constraints, resulting in relatively high energy dissipation.

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At high pre-strain, deformation beyond the yield point induces substantial crystalline-domain sliding and decoupling. Consequently, the internal constraints are substantially weakened before cyclic fatigue begins. The subsequent fatigue process therefore requires less energy and produces lower hysteretic loss.

At the same time, repeated loading promotes molecular alignment and can slightly increase crystallinity. Thus, crystallinity and energy dissipation are not necessarily positively correlated.

The introduction of temperature adds a further level to this mechanism. Thermal activation modifies molecular mobility and therefore changes the competition between crystalline-domain coupling, chain rearrangement, crystallization, orientation, and stress relaxation.

The resulting conceptual model can be summarized as:

Temperature + Pre-strain → Molecular mobility → Crystalline-domain coupling/decoupling → Chain rearrangement → Crystallinity + Orientation → Stress relaxation + Fatigue energy dissipation.

This framework provides a more comprehensive explanation of the mechanical response of mMLLDPE films than a model based solely on crystallinity.

3.12. Quantification of the Oriented Structural Fraction

The two-dimensional WAXD patterns contain contributions from both oriented and unoriented crystalline populations. The oriented component produces enhanced diffraction intensity at specific azimuthal positions, whereas the unoriented component contributes a relatively uniform intensity distribution over the azimuthal angle.

At a given Bragg diffraction angle, the minimum diffraction intensity observed over the full azimuthal range can therefore be used as an estimate of the contribution from the unoriented population. Subtraction of this minimum intensity from the complete two-dimensional diffraction pattern allows the oriented component to be isolated.

Because the WAXD measurements were acquired using a planar Pilatus detector, the experimentally recorded diffraction patterns are subject to geometric distortion associated with the projection of the spherical diffraction wave onto a planar detector. Therefore, Fraser correction was applied to the two-dimensional WAXD patterns before performing the Halo analysis.

The overall procedure consisted of four main steps:

1. acquisition of the two-dimensional WAXD pattern;
2. geometric correction using the Fraser correction;
3. identification and subtraction of the unoriented halo component;
4. integration of the corrected total and oriented diffraction patterns to obtain their corresponding one-dimensional profiles.

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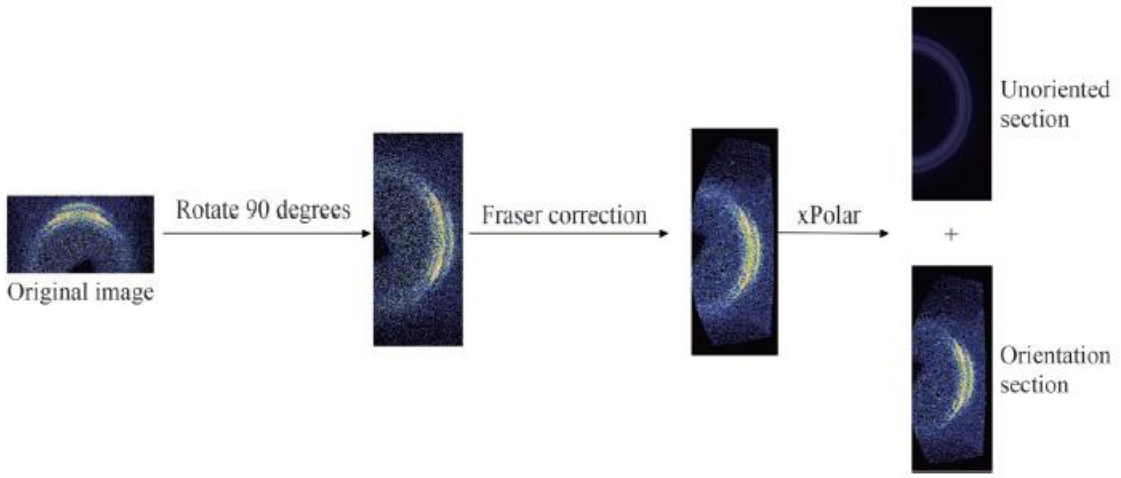

Fig. 9 Separation of a WAXD pattern into oriented and unoriented components using the Halo method

The schematic procedure is presented in Figure 9.

The integrated area of the oriented component was normalized by the integrated area of the complete corrected diffraction profile to determine the fraction of the crystalline structure belonging to the oriented population:

where is the integrated area of the oriented component and represents the integrated area of the complete corrected diffraction profile.

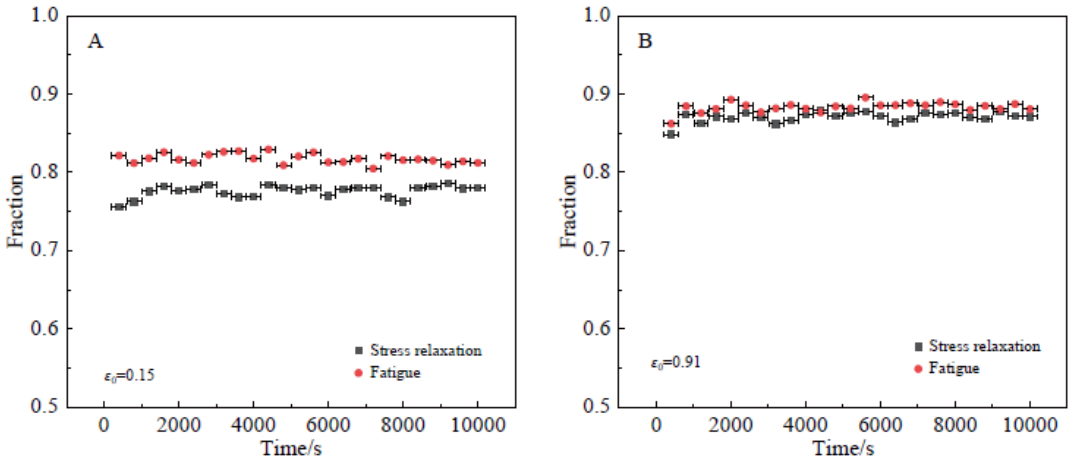

Fig. 10 Fraction of oriented part of the sample during fatigue and stress relaxation measurements at pre-strain levels of (A) 0. 15 and (B) 0. 91

Figures 10A and 10B present the evolution of the oriented fraction at the lower and higher pre-strain levels, respectively.

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The oriented fraction remains relatively stable with time under both deformation conditions. Nevertheless, the fatigue condition produces a slightly higher oriented fraction than the corresponding stress-relaxation condition. This observation indicates that cyclic mechanical loading can transform a fraction of initially unoriented crystalline domains into a preferentially oriented population.

The underlying mechanism can be attributed to repeated mechanical perturbation of the crystalline framework. Cyclic loading weakens the coupling between neighboring crystalline domains and may induce limited relative sliding. Once these constraints are reduced, previously randomly arranged domains can respond more freely to the applied stress field and progressively adopt a preferential orientation along the loading direction.

This mechanism is particularly evident at the lower pre-strain, where a substantial fraction of the original crystalline-domain coupling remains intact before fatigue begins. Repeated loading therefore provides sufficient mechanical work to promote additional domain decoupling and subsequent orientation.

At the higher pre-strain, however, the contribution of fatigue to the oriented fraction becomes considerably less pronounced. The initial deformation beyond the yield point has already induced substantial crystalline-domain sliding and decoupling. Consequently, both the degree of orientation and the oriented fraction are already relatively high before the fatigue stage begins.

This interpretation is consistent with the lower hysteretic energy loss observed at the higher pre-strain. Because a significant portion of the crystalline-domain decoupling has already occurred during the initial deformation, less mechanical energy is subsequently available or required to induce further decoupling during cyclic fatigue.

These observations support the hypothesis that a substantial fraction of the mechanical energy dissipated during fatigue is associated with **crystalline-domain decoupling and the associated molecular rearrangement**, rather than simply with the absolute crystalline content of the polymer.

3.13. Orientation of the Oriented Population

The Hermans orientation function was subsequently applied specifically to the oriented component isolated using the Halo method. This analysis provides an important distinction between the **fraction of the material that is oriented** and the **degree of orientation within that oriented fraction**.

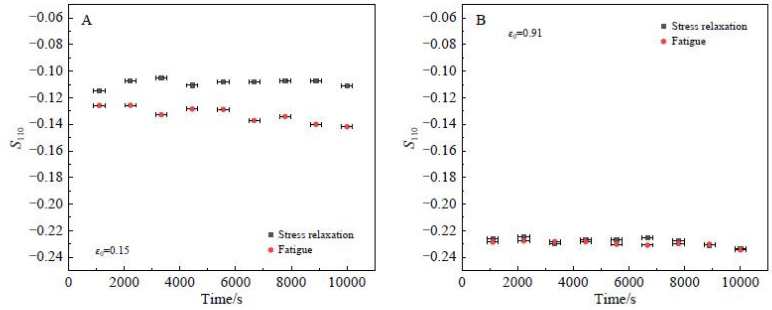

Fig. 11 The degree of orientation for the oriented part of the sample during fatigue and stress relaxation process at pre-strain levels of (A) 0. 15 and (B) 0. 91

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Figures 11A and 11B show the evolution of the orientation parameter for the oriented population under the lower and higher pre-strain conditions, respectively.

At the lower pre-strain, the orientation degree of the oriented population is substantially higher during cyclic fatigue than during static stress relaxation. This result provides direct evidence that repeated mechanical loading promotes further alignment of crystalline domains once the constraints imposed by neighboring domains have been partially weakened.

The mechanism can be understood as a sequence of mechanically induced structural events:

At the higher pre-strain, the orientation degree of the oriented population under fatigue and stress relaxation becomes much more similar. This indicates that the additional contribution of cyclic loading to the orientation of already oriented domains is limited.

The result is consistent with the structural state established during the initial deformation beyond the yield point. Once substantial crystalline-domain sliding has occurred, subsequent cyclic deformation has a reduced ability to produce additional orientation.

An important distinction nevertheless emerges between **orientation degree** and **oriented fraction**. Although the orientation degree of the oriented population is nearly identical between fatigue and stress relaxation at the higher pre-strain, the oriented fraction remains slightly higher under fatigue.

Consequently, the overall orientation of the specimen can still be slightly greater during fatigue. In other words, the increase in overall orientation does not necessarily originate from stronger orientation of already oriented domains; it can instead arise from the conversion of a small additional fraction of previously unoriented domains into the oriented population.

This distinction demonstrates the importance of combining the Hermans orientation parameter with the Halo-based oriented-fraction analysis.

3.14. Temperature-Dependent Interpretation of the Oriented Fraction

The introduction of temperature provides an additional dimension for interpreting the two orientation parameters.

The **oriented fraction** reflects how much of the crystalline population participates in preferential alignment, whereas the **orientation parameter** describes the degree of alignment within that population.

Temperature can potentially influence these two parameters through different mechanisms. Increased thermal activation may enhance molecular mobility and facilitate the conversion of constrained or unoriented domains into an oriented state. At the same time, excessive thermal mobility may promote relaxation of mechanically induced orientation.

Therefore, temperature-dependent WAXD measurements should distinguish between:

and

rather than relying on a single orientation parameter.

This approach allows the developed study to determine whether temperature primarily affects:

- the **number of domains participating in orientation**;

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- the **degree of alignment of those domains**;
- the **rate of orientation development**; or
- the **stability of mechanically induced orientation during relaxation**.

Such differentiation is particularly relevant for understanding the performance of mPE films under simultaneous thermal and mechanical cycling.

3.15. Integrated Mechanism of Stress Relaxation and Fatigue-Induced Structural Evolution

The combined mechanical and WAXD observations provide a hierarchical interpretation of the structural evolution of mMLLDPE films.

The stress-relaxation process of a viscoelastic polymer can be regarded as a progressive transition from a mechanically imposed non-equilibrium state toward a lower-energy configuration. Molecular relaxation units must overcome local energetic barriers to rearrange, while the interactions between crystalline domains constitute an important source of structural constraint.

Under relatively small deformation, the crystalline framework plays a major role in carrying the applied stress. When the film is subjected to cyclic loading, the repeated movement of this crystalline framework progressively weakens the coupling between neighboring crystalline domains.

The resulting decoupling has several consequences:

1. the effective mechanical constraint imposed on molecular chains decreases;
2. molecular mobility increases;
3. the stress carried by the crystalline framework decreases;
4. additional molecular and crystalline rearrangement becomes possible;
5. preferential orientation along the applied stress direction is promoted; and
6. the energy dissipated during subsequent cycles decreases.

Thus, cyclic fatigue should not be interpreted simply as repeated mechanical damage. At the microstructural level, it also represents a process of **progressive structural accommodation**.

The situation is fundamentally different when the initial pre-strain exceeds the yield point. The yield point can be associated with the onset of cooperative crystalline-domain motion and substantial structural rearrangement. At a pre-strain of 0.91, crystalline-domain sliding has already occurred during the initial deformation, and partial decoupling between crystalline domains has therefore been established before the fatigue experiment begins.

As a result, cyclic loading requires substantially less additional energy to promote further domain decoupling. This accounts for the lower hysteretic energy loss observed at the higher pre-strain.

The same structural state explains why the orientation responses during fatigue and stress relaxation become increasingly similar at the higher pre-strain. Since a substantial degree of crystalline-domain

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rearrangement has already occurred during initial deformation, the subsequent fatigue process contributes relatively little additional orientation.

The structural and mechanical observations can therefore be summarized through the following hierarchical pathway:

The newly introduced temperature variable adds a second control axis:

Accordingly, the developed model treats the mechanical response as the result of the coupled effects of **thermal activation, deformation history, crystalline-domain coupling, and molecular rearrangement**.

4. Conclusions

An in situ WAXD approach was employed to investigate the evolution of the crystalline structure of metallocene polyethylene films during stress relaxation and cyclic fatigue. Particular attention was given to the influence of deformation history, crystalline-domain coupling, molecular orientation, and the newly introduced temperature variable.

The principal findings can be summarized as follows:

1. **Crystalline-domain coupling governs energy dissipation during fatigue.** The hysteretic energy loss is substantially higher at the lower pre-strain because a greater fraction of the crystalline-domain interactions remains intact and must be overcome during cyclic deformation. At the higher pre-strain, substantial crystalline-domain sliding and decoupling have already occurred during the initial deformation, resulting in lower subsequent energy dissipation.

2. **Cyclic fatigue produces additional crystalline-domain decoupling.** Repeated loading weakens the interactions between neighboring crystalline domains and consequently produces a lower average stress than that observed during static stress relaxation under comparable deformation conditions.

3. **Fatigue modifies crystallinity and molecular orientation.** The crystallinity under cyclic fatigue is slightly higher than that observed during stress relaxation. Repeated stretching and recovery promote molecular alignment and can generate additional ordered regions that facilitate crystallization.

4. **Orientation development involves both oriented fraction and orientation degree.** At low pre-strain, cyclic fatigue increases both the contribution of the oriented population and its degree of orientation. At high pre-strain, however, the additional effect of fatigue on the orientation degree becomes limited because substantial structural alignment and crystalline-domain decoupling have already occurred during the initial deformation.

5. **Crystalline-domain decoupling, rather than crystallinity alone, controls fatigue energy dissipation.**
   The observation that higher crystallinity can coexist with lower hysteretic energy loss demonstrates that the absolute crystalline content is insufficient to explain the mechanical response. The degree of coupling between crystalline domains and their ability to undergo relative rearrangement are more directly related to energy dissipation.

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6. **Temperature provides an additional mechanism for controlling structural relaxation.** Thermal activation is expected to modify molecular mobility, crystalline-domain rearrangement, orientation kinetics, and the relaxation of mechanically induced structures. The temperature-dependent extension of the in situ WAXD approach therefore provides a means of distinguishing thermally activated molecular relaxation from deformation-induced structural evolution.

7. **A coupled thermo-mechanical structure–property relationship is proposed.** The microstructural response of mMLLDPE films can be described through the interaction of temperature and deformation history, which govern crystalline-domain coupling and molecular mobility and ultimately determine crystallinity, oriented fraction, molecular orientation, stress relaxation, and fatigue energy dissipation.

Overall, the developed framework demonstrates that the mechanical durability of metallocene polyethylene films cannot be adequately described solely in terms of macroscopic stress or crystallinity. Instead, it is governed by the dynamic interplay among **temperature, crystalline-domain coupling, molecular mobility, crystallization, and orientation**. This structure-based understanding provides a useful basis for optimizing the performance of mPE films subjected to combined thermal and cyclic mechanical conditions.

Ethical Considerations

Not applicable. This study did not require ethical approval because it does not include human or animal subjects and does not involve any personal or sensitive data.

List of Abbrevations:

(WAXD): Wide-angle X-ray diffraction;(MD): machine direction; mPE films: Metallocene Polyethylene films;

Acknowledgment:

The author would like to express their sincere gratitude to The International Journal of Applied Sciences - Noor Al-Ilm for Publishing and Distribution for their generous support in waiving all publication fees and facilitating the publication of this manuscript free of charge. Their commitment to promoting scientific research and supporting researchers is highly appreciated.

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The International Journal of Applied Sciences.
Vol. 2, No. 1, 2026, pp. 62-88
ISSN 3104-8935

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Author Contribution:

All authors contributed equally to the main contributor to this paper. All authors read and approved the final paper.

Declaration of generative AI and AI-assisted technologies in the writing process

The authors hereby declare that no generative artificial intelligence or AI-assisted technologies were used at any stage during the preparation of this manuscript, including language editing, proofreading, or content development. The authors take full responsibility for the originality and integrity of the work presented in this publication.

Funding:

This research received no external financial funding. The authors also acknowledge The International Journal of Applied Sciences, Noor Al-Ilm for Publishing and Distribution, for providing a full waiver of the publication fees. The publication fee waiver was provided as editorial support and did not involve any financial contribution to the conduct, design, analysis, or reporting of the research.

Conflicts of Interest:

“The authors declare no conflict of interest.”

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