Parameters optimization for manufacturing advanced self-reinforced composites based on ultra-high molecular weight polyethylene

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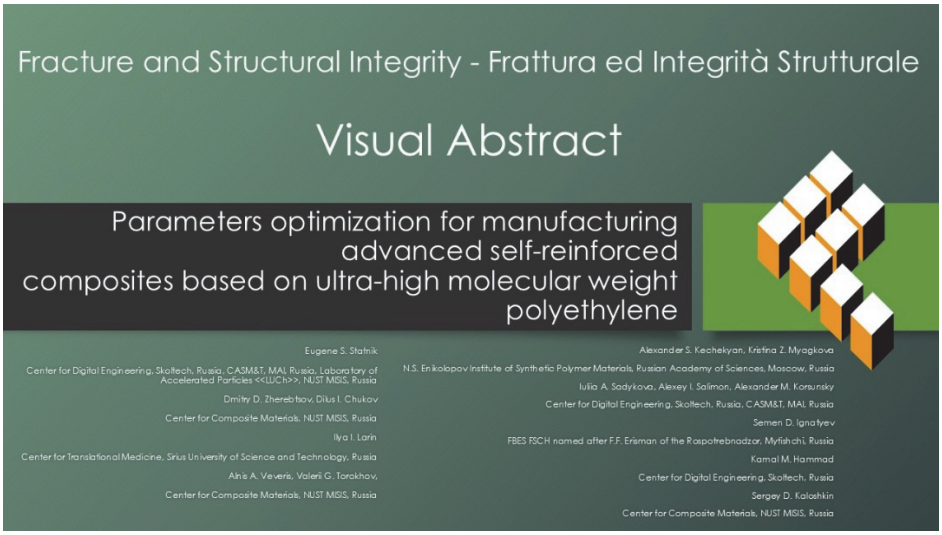

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INTRODUCTION

Composite materials (CMs) are traditionally defined as heterogeneous systems comprising chemically distinct phases, where a reinforcing component is embedded within a matrix. They are distinguished from alloys by insolubility between phases [1]. A unique subset of these materials is self-reinforced composites (SRCs), which are fabricated from structurally distinct components of the same material or polymer class. This earns them designations such as single polymer, monomaterial, or "homogeneous" composites [2].

The single-material composition of such polymers provides significant advantages in sustainability, facilitating straightforward recycling without the need for energy-intensive processes like pyrolysis or the complex separation protocols required for conventional composites [3]. In particular, thermoplastic-based SRCs can be reprocessed via remelting process, thus aligning with the principles of a circular economy.

The mechanical performance of conventional fiber-reinforced composites is often limited by weak interfacial adhesion, particularly when chemically inert components are involved. In contrast, polymeric SRCs achieve stress transfer through hydrogen bonding and macromolecular entanglements, bypassing the adhesion challenges typical of traditional composite materials [4,5]. For example, the interfacial shear strength between ultra-high molecular weight polyethylene (UHMWPE) fibers and a polyethylene matrix is 8.3 MPa, which far exceeds that of UHMWPE-epoxy systems at 1.7 MPa [6]. This highlights the inherent efficiency of single-polymer architecture.

Among thermoplastics, UHMWPE is exceptionally well-suited to SRCs due to its outstanding specific strength in an oriented fiber form [7], which yields the highest mechanical performance of any polymer-based SRC [2]. The unique properties of UHMWPE-based SRCs make them ideal for demanding applications, including ballistic protection, medical implants, and aerospace components. While some studies incorporate additional polyethylene grades to fabricate UHMWPE SRCMs [8], this work focuses exclusively on UHMWPE fibers to simplify processing. Notably, UHMWPE cannot be conventionally melt-processed and requires consolidation via thermal pressing [9].

Although prior studies have examined the structure of UHMWPE-based SRCs [10–12] and proposed molecular-level formation mechanisms [7,13], critical gaps remain. Quantitative analysis of interphase interactions and fracture morphology, as well as the systematic correlation between processing parameters and mechanical properties, remains under-explored.

Recent advances in UHMWPE processing have begun addressing these knowledge gaps, particularly through innovative modifications of fibers and matrices. Several processing parameters significantly impact the microstructure and mechanical

properties of self-reinforced composites produced from ultra-high molecular weight polyethylene. These parameters include the type of reinforcement used, the manufacturing process for the composite, and the treatment of fibers.

Recent work by Fedorenko and Luinstra [14] has advanced *in situ* polymerization techniques for UHMWPE/carbon fiber composites, achieving superior tensile strengths through optimized fiber dispersion and interfacial bonding. Complementary studies by Zhao *et al.* [15] have demonstrated that plasma modification of UHMWPE fibers can enhance impact resistance by approximately 30 %, offering a scalable route to improve energy absorption in SRCs. For harsh environments, Skakov *et al.* [16] have developed acid-resistant UHMWPE/diabase composites, expanding the potential industrial applications of these materials.

In addition, scientists in [17] used single-site catalysts to produce disentangled UHMWPE with reduced molecular entanglements, making it easier to process without compromising its mechanical properties. Industrial-scale production was further refined by Li *et al.* [18], who correlated Ziegler-Natta catalyst structures with polymer crystallinity and tensile performance. For characterization, Saeed *et al.* [19] introduced fractional differential FTIR spectroscopy to quantify the effects of gamma radiation on UHMWPE. This allows for precise monitoring of polymer degradation, which is crucial for medical and aerospace applications.

Thermal processing remains central to UHMWPE performance. Mao *et al.* [20] showed that higher crystallinity in injection-molded UHMWPE/HDPE blends reduces wear rates by 40 %, highlighting the trade-offs between crystallinity and mechanical properties. Computational insights from Wang *et al.* [21], using molecular dynamics, revealed how pressure optimizes interfacial bonding in hard-soft composites, directly supporting our findings on thermal pressing parameters.

In this study, we present a comprehensive investigation of UHMWPE-based unidirectional SRCs, combining mechanical testing (bending, tensile, and impact) with advanced structural characterization of both as-fabricated and post-fracture samples. By quantifying interfacial interactions and elucidating failure mechanisms, this study establishes a processing-structure-property framework for high-performance single-polymer composites.

MATERIALS AND METHODS

Self-reinforced composite fabrication

In this study, gel-spun ultra-high molecular weight polyethylene fibers Dyneema® SK75 (Dyneema, Netherlands) were used to create UHMWPE-based SRCs. The fibers had a linear density of 440 dtex with an average diameter of 17 μm and exhibited exceptional mechanical properties, including a tensile modulus of 129 GPa, a tensile strength of 3.6 GPa, and a failure strain of 3.5 % [22].

Unidirectional self-reinforced composites were fabricated through a carefully controlled thermal pressing process. Fiber preforms were first prepared by precisely winding continuous filaments between parallel guides to create aligned assemblies matching the mold dimensions (80 × 10.5 mm). The fiber orientation was kept along the mold's longitudinal axis. The number of winding turns was adjusted to achieve the required specimen thickness for subsequent mechanical testing.

The consolidation process involved transferring the oriented preforms into the mold under controlled conditions to preserve fiber alignment. A constant pressure of either 25 MPa or 50 MPa was applied prior to initiating the thermal cycle. The temperature protocol consisted of heating to the target temperature (investigated target points 145, 155, 165, 170, 175, 180 °C) over 50 minutes, followed by a 10-minute isothermal hold at the target temperature. This temperature range was selected to enable the controlled surface melting of the UHMWPE fibers while maintaining their core crystalline structure. To prevent the formation of detrimental transcrystalline layers [10] and minimize residual stresses, samples underwent slow cooling to 40 °C under ambient conditions before pressure release and mold disassembly.

Microstructure characterization

The structural features of UHMWPE-based SRCs were investigated using a Carl Zeiss Crossbeam 550 field-emission scanning electron microscope operating in secondary electron detection mode at an accelerating voltage of 1 kV. This low-voltage approach enabled high-resolution surface imaging without the need for conductive coating.

For cross-sectional analysis, the samples were first sectioned perpendicular to the fiber direction using a diamond saw with a continuous coolant flow to prevent thermal damage. The cut surfaces then underwent a grinding and polishing process using silicon carbide paper with grit sizes ranging from P400 to P1500, followed by final polishing with a 60 nm alumina suspension on a felt pad. To reveal the crystalline microstructure, polished samples were chemically etched for 4 hours at room temperature using an oxidative mixture consisting of a 2:1 (vol./vol.) ratio of concentrated sulfuric acid (98 %) and orthophosphoric acid (85 %), to which 2 wt. % of potassium permanganate was added. Prior to use, the etching solution was homogenized by orbital mixing for 1 hour. This selective etching protocol preferentially removes amorphous

UHMWPE regions while preserving crystalline domains [23], creating distinct contrast between the isotropic matrix and the highly oriented fibers.
Fracture surfaces for failure analysis were prepared by inducing controlled longitudinal delamination along the fiber-matrix interface through manual cleavage. These fracture surfaces were examined in their native state without additional polishing or etching to preserve the authentic failure morphology.
All SEM images were acquired under consistent operating conditions to ensure comparative analysis of microstructural features across different processing parameters.

Interlayer shear testing

The interfacial properties of UHMWPE-based SRCs were assessed through interlayer shear testing using the short-beam shear method according to ISO 14130 [24]. The experiment was conducted on a Zwick Z020 universal testing machine with a permanent crosshead speed of 1 mm/min. The test configuration consisted of loading roller with a 5 mm radius and support rollers of 1 mm radius, positioned 10 mm apart.
The fabricated samples (Fig. 1) with longitudinal fibers orientation and initial dimensions of 80 × 10 × 2 mm (length × width × thickness) were cut into 4 parts to reach a size of 20 × 10 × 2 mm, respectively. This geometry ensured failure primarily through interlayer shear rather than flexural modes.

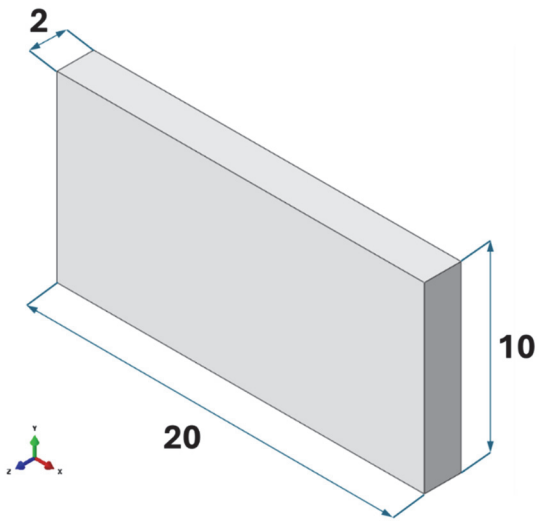


Figure 1: Sketch of samples for interlayer shear testing

The interlayer shear strength was calculated using the standard equation:

$$\tau = \frac{3F}{4bh} \tag{1}$$

where τ is interlayer shear strength (MPa), F is maximum load at failure (N), b is specimen width (mm) and h is specimen thickness (mm).

Charpy Impact Testing

The impact toughness of UHMWPE-based SRCs was characterized through Charpy impact testing in accordance with GOST 4647-2015 [25]. Due to the high impact resistance characteristic of UHMWPE fibers, specifically designed unnotched samples of type 2/179-1 with longitudinal fibers orientation were tested. Each specimen had the following dimensions: 75 mm in length, 10 mm in width, and 3 mm in thickness (Fig. 2).
The test configuration consisted of a 60 mm support span with the pendulum set to a 4 J potential energy level. For statistical reliability, 4 specimens were evaluated at each critical processing temperature, representing the transition range from partial to complete fiber consolidation.

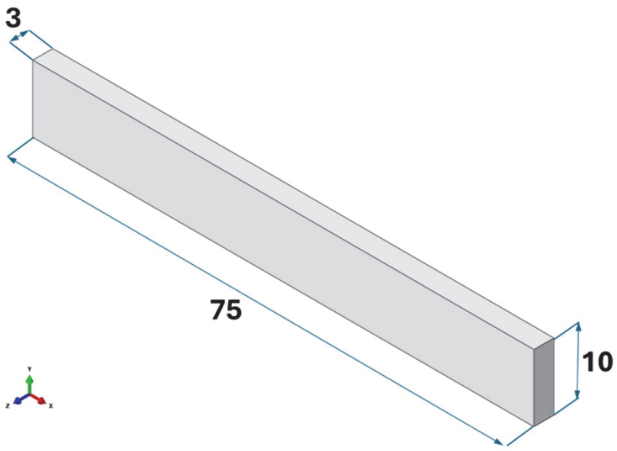


Figure 2: Sketch of samples for Charpy impact testing

Bending testing

The flexural properties of UHMWPE-based SRCs were evaluated through three-point bending test conducted in accordance with GOST R 56810-2013 [26]. Testing was performed on a Zwick Z020 universal testing machine under controlled displacement conditions at a constant crosshead speed of 10 mm/min. The test configuration consisted of a 10 mm diameter of loading roller and 1 mm diameters support rollers spaced 32 mm apart.

Rectangular specimens measuring 80 mm (length) × 10 mm (width) × 2 mm (thickness) were prepared with fiber orientation aligned parallel to the specimen length (Fig. 3).

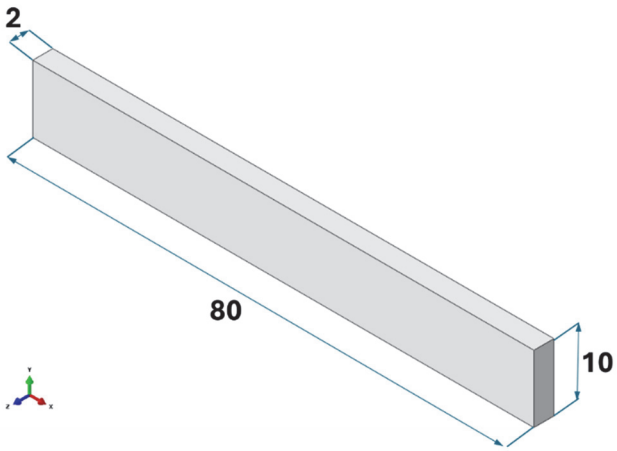


Figure 3: Sketch of samples for bending testing

Tensile testing

Tensile characterization of UHMWPE-based SRCs was performed in accordance with ASTM D882-10 [27] using a Zwick Z020 universal testing machine. Specimens with dimensions of 80 mm (length), 5 mm (width) and varying thicknesses (200–300 μm, 1 mm, or 2 mm) were tested at a constant crosshead speed of 10 mm/min.

Initial testing revealed significant challenges in achieving proper grip adhesion and inducing failure within the gauge section. Multiple approaches were systematically evaluated to optimize specimen preparation: (1) various grip enhancement methods including adhesive bonding (cyanoacrylate, epoxy) and surface treatments (sandpaper abrasion) and (2) stress concentration features such as controlled notching. Through iterative testing, an optimized specimen configuration was developed that successfully produced valid tensile testing. It requires:

- Ultra-thin specimens (200–300 μm thickness × 5 mm width) to reduce absolute load requirements (Fig. 4).
- Precision notches up to 50 % of specimen width to localize failure initiation.
- Sandpaper-treated grip regions (30 mm length) for enhanced surface adhesion.

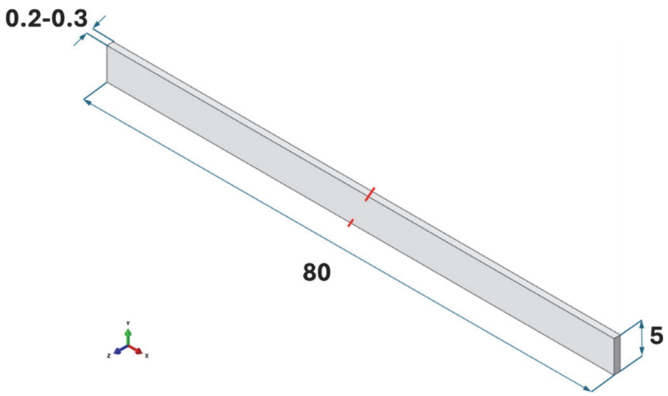


Figure 4: Sketch of samples for tensile testing

RESULTS AND DISCUSSIONS

Microstructural study

The prepared cross-sections of unidirectional self-reinforced composites based on ultra-high-molecular-weight polyethylene fibers were examined using scanning electron microscopy. The distinct contours of individual fibers are shown in Fig. 5. This morphology stems from preferential chemical etching of the less crystalline matrix phase, created by surface melting of UHMWPE fibers during hot pressing.

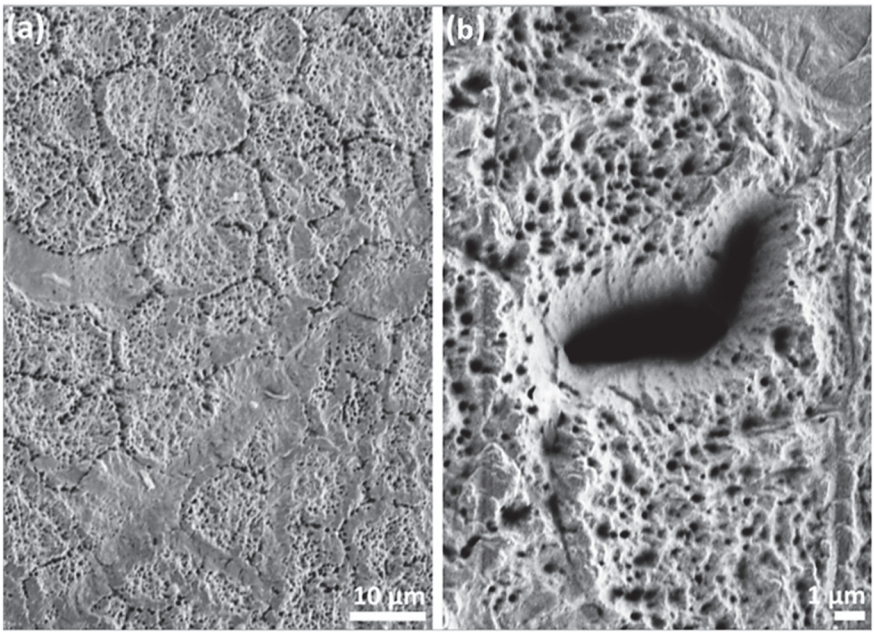


Figure 5: The prepared cross-section of unidirectional SRC based on UHMWPE fibers, fabricated at 165 °C and 25 MPa.

Moreover, the fibers undergo deformation into a hexagonal shape within the composite, corresponding to the densest packing arrangement. This transformation from their initial cylindrical or oval cross-section indicates that thermal pressing generates significant internal stresses, both within the individual fibers and throughout the entire self-reinforced composite. Our previous WAXS analysis of this composite [7] supports this hypothesis, showing deviations from an ideal circular pattern.

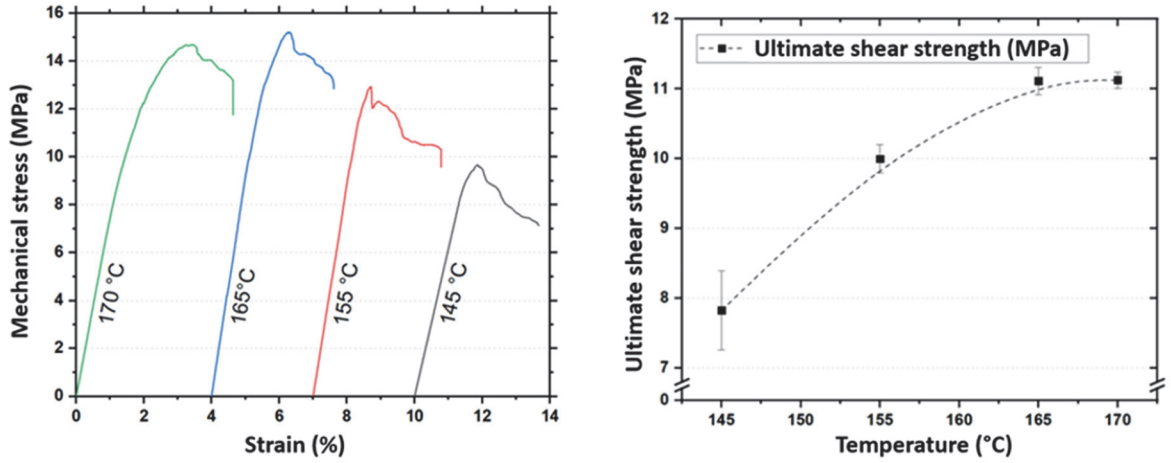
The densest packing arrangement (hexagonal) maximizes fiber contact area, enhancing interfacial bonding via macromolecular interdiffusion. However, different deformation resistance (flow stress) between the fiber core (oriented crystalline phase) and the surface-melted matrix (isotropic phase) generate residual stresses, which play a dual role. On the one hand, they promote interfacial adhesion by increasing fiber-fiber contact pressure, facilitating stress transfer during

loading. This is evidenced by the peak mechanical properties (e.g., 11.1 MPa shear strength, 130 MPa bending strength) achieved at optimal temperatures (165–170 °C) that will be provided below. However, excessive stresses may contribute to void formation as shown in Fig. 5, acting as stress concentrators that initiate failure under load.

Within our current study we did not directly measure residual stresses, although we acknowledge their significance. However, we also appreciate the complexity of the task of evaluating the residual stresses at the microscale inside or between fiber-fiber interface. Considering the methods for this task, it appears that future the FIB-DIC method [28,29] or Raman spectroscopy [30] could be suitable for quantifying these stresses. However, to the best of our knowledge neither has been applied successfully to the system of our interest, indicating that significant challenges will need to be overcome to extract the desired results.

Interlayer shear testing

To evaluate the influence of manufacturing parameters on interfacial interactions within the composite, interlayer shear strength was measured via the short beam bending method. The resulting stress-strain curves of samples processed at different temperatures but under a constant pressure of 25 MPa are shown in Fig. 6a. The calculated temperature-dependent shear strength is illustrated in Fig. 6b.



(a) (b)

Figure 6: Mechanical properties of unidirectional UHMWPE-based SRCs consolidated at 25 MPa. (a) Representative stress-strain curves from tensile testing, showing the influence of consolidation temperature (indicated by color in the original figure). (b) Short-beam shear strength as a function of temperature, highlighting interfacial bonding efficiency.

Shear strength increased continuously with processing temperature, signaling improved interfacial adhesion driven by enhanced macromolecular diffusion. All specimens failed via single shear between the supports, with measured shear strengths of 7.80±0.56 MPa (145 °C), 10.00±0.20 MPa (155 °C), 11.10± .19 MPa (165 °C), and 11.10±0.12 MPa (170 °C). At 180 °C, however, specimens exhibited plastic shear deformation without interlayer failure corresponding to a behavior of isotropic polymers. This indicates complete remelting of the oriented fiber phase at elevated temperatures, resulting in bulk isotropic or weakly oriented UHMWPE. Bending tests confirm this interpretation: SRCs fabricated at 180 °C displayed mechanical properties similar to isotropic UHMWPE.

In contrast, the elastic modulus followed a non-monotonic trend: 4.10±0.71 GPa (145 °C), 6.50±0.04 GPa (155 °C), 6.50±0.23 GPa (165 °C), and 5.60±0.52 GPa (170 °C), respectively. Insufficient fiber surface melting at lower temperatures causes poor consolidation, reducing both strength and modulus. Conversely, higher temperatures improve consolidation (maximizing shear strength) but simultaneously reduce the oriented phase fraction, ultimately decreasing stiffness.

Failure mechanisms investigation

To elucidate SRC failure mechanisms, we analyzed delaminated samples via SEM as shown in Fig. 7. Fibrils are distinctly visible along fiber surfaces [16], with increasing fibrillar bridge density at higher hot-pressing temperatures. These structures typically form during polymer fracture through molecular entanglements – characteristic of fiber-forming crystalline polymers like UHMWPE.

While prior studies confirm delamination occurs exclusively along fiber boundaries [25], our observed fibrillar structures and inter-fiber separation suggests interfacial bonding occurs via macromolecular interdiffusion. Higher processing

temperatures enhance crystalline phase melting, increasing chain mobility and promoting deeper mutual penetration of UHMWPE chains between fibers. This mechanism agrees with established models [26] where macromolecular migration through a metastable hexagonal phase governs SRC formation.

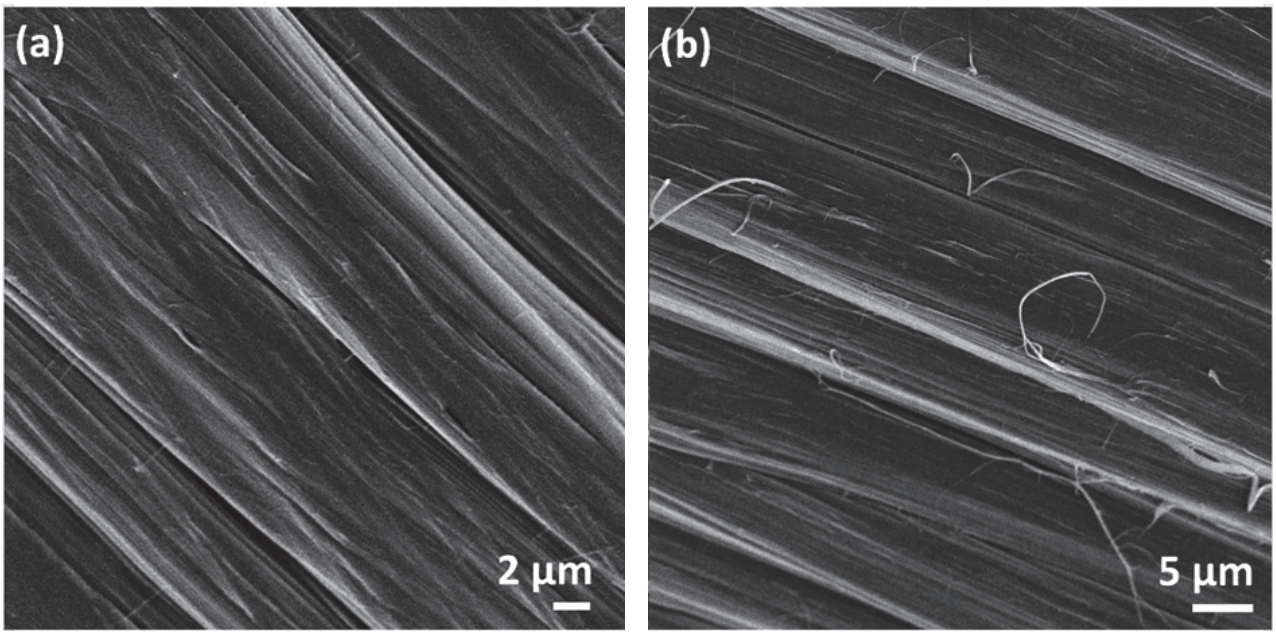
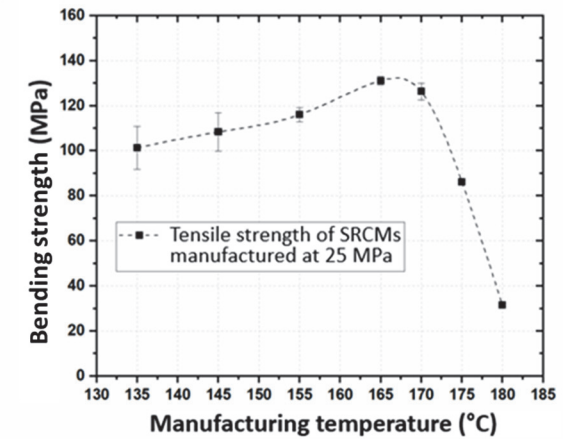


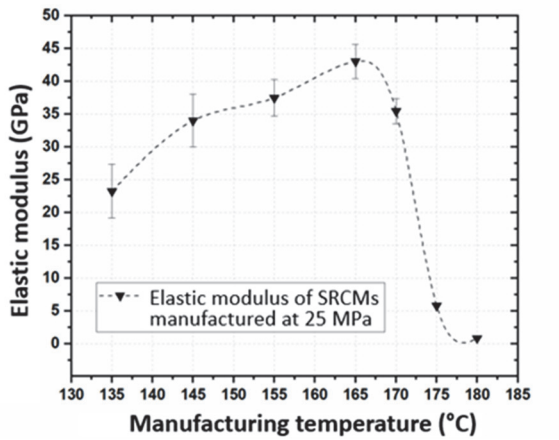
Figure 7: SEM micrographs of fracture surfaces in UHMWPE SRCs fabricated at 25 MPa after delamination. Samples processed at (a) 145 °C and (b) 165 °C.

Bending testing

The mechanical behavior of unidirectional self-reinforced composites based on UHMWPE fibers produced using different processing parameters was investigated via bending tests. Results reveal a pronounced nonlinear processing-performance relationship as shown in Fig. 8.

Both bending strength and elastic modulus exhibit consistent trajectories: initial increases with temperature peak before sharp declines. This trend – persistent across pressures – indicates a fundamental thermal optimum for reinforcement. Maximum values reached ~130 MPa (bending strength) and 40–42 GPa (elastic modulus), remarkably pressure-insensitive. This behavior stems from competing microstructural mechanisms. At first, enhanced matrix development and fiber-matrix adhesion enable efficient stress transfer, boosting strength and stiffness. On the other hand, fiber melting reduces reinforcement fraction, causing modulus collapse. Through interfacial bonding may temporarily sustain strength, complete fiber remelting yields isotropic UHMWPE properties.





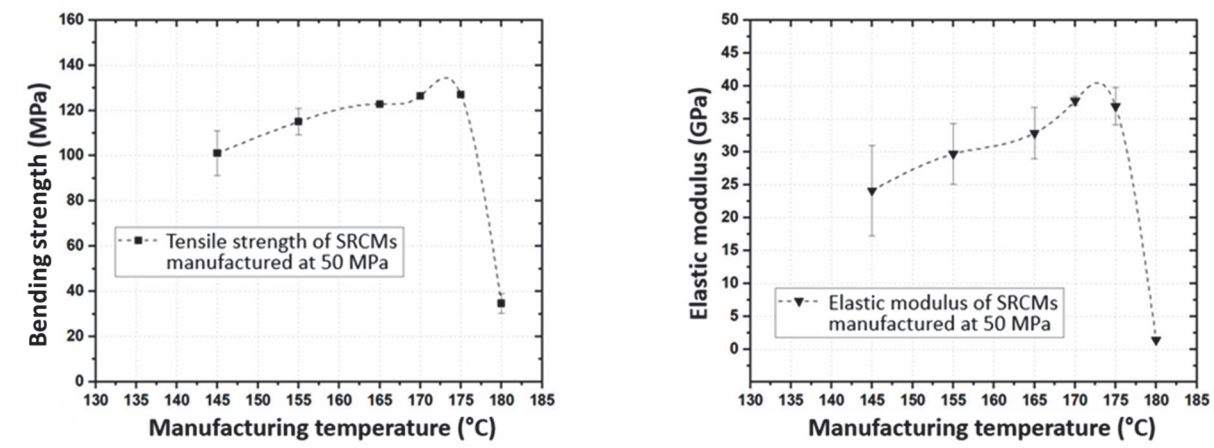


Figure 8: Bending strength and elastic modulus for specimens of unidirectional SRCs based on UHMWPE fibers fabricated at different temperatures and pressures of 25 MPa (top row) and 50 MPa (bottom row).

Notably, higher pressures (50 MPa) shift peak performance temperatures upward – consistent with the Clausius-Clapeyron equation [27] predicting elevated melting points under constrained chain mobility. The pressure-independent maximum values indicate pressure modulates processing window without altering ultimate performance potential.
These findings provide critical fabrication guidelines: optimizing UHMWPE-SRCs requires precise thermal control to balance reinforcement integrity against interfacial development, with pressure mainly adjusting the optimal temperature range.

Charpy impact testing

To evaluate SRC high strain-rate performance, un-notched Charpy impact tests were conducted on specimens fabricated at 25 MPa and various temperatures (145, 155, 165 °C). Results reveal strong temperature dependence in impact resistance with distinctive failure behavior as shown in Fig. 9.

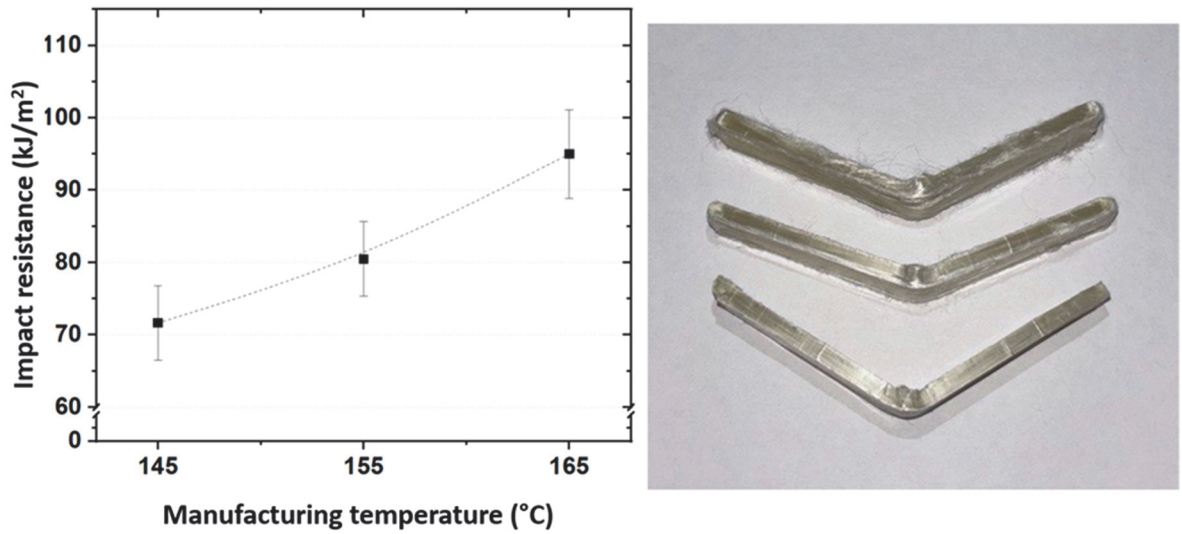


Figure 9: Specific impact resistance for samples of unidirectional SRCs based on UHMWPE fibers fabricated at 25 MPa pressure and different temperatures.

No specimens experienced complete failure. All specimens underwent plastic bending without fiber fracture at impact sites. Ductile response contrasts sharply with brittle failure in epoxy-based UHMWPE composites [20,21]. Specific impact resistance increased progressively with a processing temperature: 72±5 kJ/m² (145 °C), 80±5 kJ/m² (155 °C), 95±6 kJ/m² (165 °C). This enhancement correlates directly with improved macromolecular interdiffusion at elevated processing

temperatures. The absence of fiber rupture and predominant plastic deformation indicate impact energy absorption occurs primarily through matrix deformation and controlled interfacial debonding rather than fiber fracture – notably despite decreasing fiber fractions at higher temperatures.
These findings demonstrate SRCs uniquely combine exceptional impact resistance with avoidance of catastrophic failure modes. This synergy stems from their homogeneous ductile architecture where matrix and reinforcement share similar deformation mechanisms, enabling efficient energy dissipation via controlled plasticity instead of brittle fracture.

Tensile testing

Tensile tests were performed only for SRC samples fabricated under optimal conditions (165 °C, 25 MPa, 10 min) as determined from previous tests. The experimental setup and results are presented in Fig. 10. Testing revealed exceptional performance: tensile strength of 1440 MPa and elastic modulus of 40 GPa. Fracture analysis showed distinctive characteristics: (1) boundary-aligned crack propagation (deviating from brittle material notch paths); (2) complete separation into unconsolidated fiber bundles; (3) fracture surfaces parallel to fiber alignment The measured tensile strength archives 35-50 % of pristine UHMWPE fiber strength, demonstrating efficient stress transfer. This self-reinforced architecture thus maintains exceptional strength while enabling unique ductile failure modes absent in traditional composites.

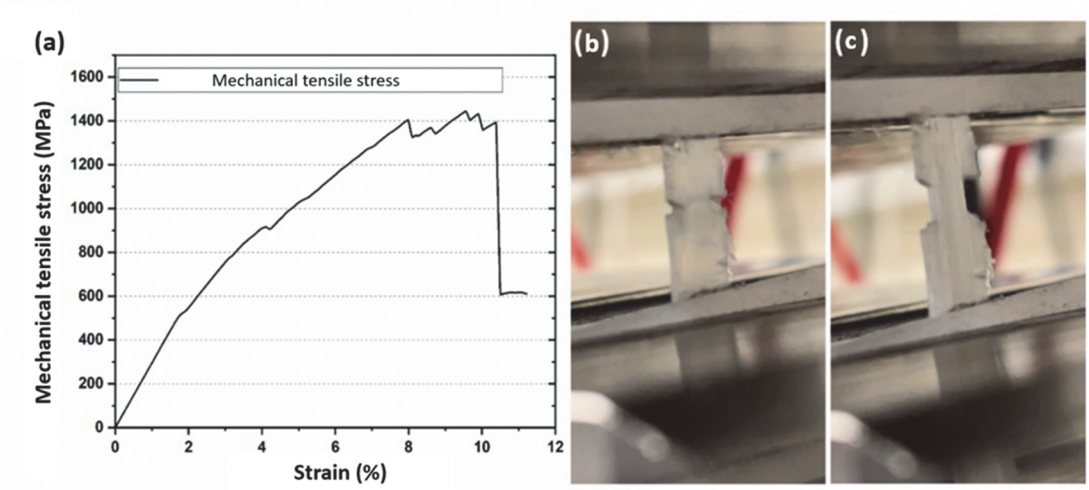


Figure 10. (a) Mechanical stress in tensile test of unidirectional UHMWPE-based SRCs fabricated at 25 MPa and 165 °C, (b) photo of notched SRCM specimen before tensile test, (c) photo of notched SRCM specimen during the tensile test.

The developed SRCs based on UHMWPE have a high tensile strength, but a low bending strength. This behavior is intrinsic to the unique architecture of these materials and stems from the fundamental structure-property relationships. The exceptional tensile strength arises from the continuous, aligned UHMWPE fibers that bear nearly the entire load through their covalently bonded crystalline chains oriented along the fiber axis. The high degree of crystallinity (> 95 %) and molecular alignment enable these fibers to achieve tensile strengths of 3–4 GPa, characteristic of highly oriented UHMWPE structures. In contrast, bending performance is governed by a more complex interplay of mechanisms. During bending, the composite experiences simultaneously tensile and compressive stresses. While the fibers remain effective in the tension regime, the compressive strength of uniaxially aligned composites is known to be limited by fiber buckling and interfacial shear in the partially melted matrix material formed during hot compaction. This matrix, being isotropic and less crystalline exhibits significantly lower compressive strength – typically about an order of magnitude less than the tensile strength of the fibers. This substantial difference is further exacerbated by stress concentrations that develop at fiber-matrix interfaces, particularly in regions where the hexagonal fiber packing deforms under transverse stresses.

CONCLUSIONS

This investigation establishes fundamental processing-structure-property relationships in thermally pressed UHMWPE SRCs.

Microstructural analysis reveals that hexagonal fiber deformation under 25/50 MPa pressure enables dense packing, while persistent boundary voids indicate incomplete consolidation. Delamination studies elucidate primary bonding mechanism – extensive fibrillar bridging, which confirms a three-phase interdiffusion process: (1) partial surface melting of crystalline fibers; (2) interpenetration/entanglement of molten chains; (3) pressure-induced recrystallization into isotropic matrix.
This temperature-dependent process drives mechanical enhancement: interfacial shear strength increases from 7.8 to 11.1 MPa and impact resistance grows up from 72 to 95 kJ/m² during the temperate rise from 145 to 170 °C, respectively. However, while higher temperatures improve interfacial bonding through chain entanglement, they simultaneously reduce oriented-phase content [22]. This creates a narrow processing window where properties peak (bending strength of 130 MPa and elastic modulus of 40–42 GPa) before collapsing at excessive temperatures. Higher pressures (50 MPa) shift this optimum temperature upward per Clausius-Clapeyron kinetics [19], without altering maximum achievable properties.
Mechanical characterization reveals several unique aspects of these SRCs based on UHMWPE. Tensile testing demonstrated exceptional strength (1440 MPa) approaching 35-50 % of pristine fiber values [8], indicating efficient stress transfer. However, the interdiffusion-based interfacial adhesion remains weaker than conventional matrix materials, resulting in distinctive failure modes dominated by fiber pull-out and fibrillar bridging rather than brittle fracture. This behavior, observed across bending, tensile and impact tests, highlights the energy-absorbing capability of self-reinforced architecture.
In summary, UHMWPE-SRCs represent a monolithic polymer system governed by competing mechanisms: interfacial bonding strength improvement with temperature vs loss of reinforcing phase content. This framework enables precision optimization of single-component composites for application-specific performance.

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CREDIT AUTHORSHIP CONTRIBUTION STATEMENT

Eugene S. Statnik: Writing – review and editing. Dmitry D. Zherebtsov: Writing – original draft. Dilus I. Chukov: Investigation. Ilya I. Larin: Formal analysis. Alnis A. Veveris: Investigation. Valerii G. Torkhov: Methodology. Alexander S. Kechekyan: Investigation. Kristina Z. Myagkova: Resources. Iuliia A. Sadykova: Methodology. Alexey I. Salimon: Conceptualization. Alexander M. Korsunsky: Writing – review and editing, Supervision. Semen D. Ignatyev: Validation. Kamal M. Hammad: Writing – review and editing. Sergey D. Kaloshkin: Project administration.

DECLARATION OF COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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