Does Texturing of UHMWPE Increase Strength and Toughness?

A Pilot Study

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Abstract

Background Crosslinked UHMWPE as a bearing surface in total joint arthroplasty has higher wear resistance than conventional UHMWPE but lower strength and toughness. To produce crosslinked UHMWPE with improved mechanical properties, the material can be treated before crosslinking by tension to induce molecular alignment (texture).

Questions/purposes We asked how (1) the microstructure of UHMWPE evolves when subjected to tension and (2) whether the new microstructure (texture) increases strength and toughness.

Methods We analyzed microstructure evolution of UHMWPE by small- and wide-angle xray scattering and

scanning electron microscopy. We then developed a method to characterize the local strength and toughness of undeformed and textured UHMWPEs by means of nano-scratch tests along and perpendicular to the specimen axis. In three samples we determined the scratch characteristics in terms of deformation mode, coefficient of friction (μ), and viscoelastic recovery (r).

Results Before the tensile process, the scratch behavior of UHMWPE was characterized by a μ ranging from 0.64 to 0.68, no cracking, and r ranging from 0.58 to 0.60. Microfibrillar morphologic features resulted from the tensile process. The new microstructure had an increased strength ($r = 0.78$) and decreased toughness (cracking + $\mu = 0.77$) perpendicular to the fibril axis and decreased strength ($r = 0.53$) and increased toughness (no cracking + $\mu = 0.55$) parallel to the fibril axis.

Conclusions Textured UHMWPE behaves like a fiber composite with high strength and toughness in well-defined directions. However, the effect of crosslinking on these specific properties is unknown and therefore it is important to verify that the properties are retained. If wear resistance of crosslinked-textured UHMWPE is at least as high as that of crosslinked UHMWPE, novel medical devices made of crosslinked-textured UHMWPE could be developed and clinically tested.

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Each author certifies that his or her institution either has waived or does not require approval for the human protocol for this investigation and that all investigations were conducted in conformity with ethical principles of research.

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Introduction

UHMWPE is used as a bearing surface in THAs and TKAs against a metallic or ceramic part. The friction between these two parts leads to gradual failure of the plastic component either through delamination from fatigue failure (occurring primarily in TKAs) or surface wear that releases polymer debris (occurring in THAs and TKAs)

[5, 10]. The debris produced in the periprosthetic tissue stimulates osteolysis [9] that causes loosening of the implant and eventually leads to its failure [4, 9, 19]. Which of these two wear mechanisms dominates depends on the mechanical properties of the contacting bodies, counterface roughness, lubricant composition, and loading configuration. Crosslinking decreases surface wear of conventional UHMWPE. However, the crosslinking procedure causes a decrease of strength, ie, the ability of the material to withstand stress and toughness, ie, the ability of the material to show plastic deformation and resistance to an impact load without failure [14, 24]. Fracture toughness and ultimate stress of crosslinked UHMWPE are reportedly 30% and 42% lower than those of an untreated product, respectively [14]. Based on wear prediction equations derived from the mechanical properties of UHMWPE, decreasing toughness could increase the intensity of fatigue failure mechanisms in the case of TKA [27]. However, it is unclear whether the reduction properties have any clinical relevance or are merely a laboratory curiosity. Developing crosslinked UHMWPE retaining the strength and toughness of conventional UHMWPE would be desirable if the reductions have clinical relevance.

To produce crosslinked UHMWPE with such properties, the material could be treated before crosslinking to increase strength and toughness. This can be achieved by modifying the microstructure of conventional UHMWPE by a solid-state deformation process that induces a molecular alignment (texture) [11, 18]. This simple treatment does not substantially alter polymer chemistry and does not require any posttreatment. Numerous techniques can be used to produce a textured material such as, for example, cold extrusion, channel die, tension, compression, and rolling at the solid state. Moreover, combining texturing with crosslinking reportedly increases wear resistance in comparison with conventional UHMWPE [20, 22]. In one study wear based on a multidirectional pin-on-disk test of a crosslinked roll-drawn material was 82% lower than that of the conventional product [20]. However, before combining these two processes, it is fundamentally important to identify the texturing mechanisms of UHMWPE and to characterize the effect of texture on strength and toughness.

We therefore asked how (1) the microstructure of UHMWPE evolves when subjected to tension and (2) whether the new microstructure state would increase surface strength and toughness of UHMWPE.

Materials and Methods

For the first question, three undeformed and three textured samples of UHMWPE were produced and characterized by

scanning electron microscopy (SEM) and small- and wide-angle xray scattering (SAXS/WAXS). For the second question, we developed a method to characterize the surface strength and toughness of textured UHMWPE by means of a nanoscratch tester. One scratch test was performed on each tensile specimen; three scratch tests therefore were performed per deformation state. Each experimental value was presented as an average value and range, and each image or each curve represented three experiments.

We used GUR 1020 ($M_w = 5.0 \times 10^6$ g/mol) from Ticona GmbH (Oberhausen, Germany). Four-millimeter-thick plates were compression-molded from initial powder during 30 minutes at 240°C and under 33 bars using a Carver laboratory press (Carver Inc, Wabash, IN, USA) equipped with heating plates. The cooling step was performed under 50 bars using room temperature water circulation in the press plates. To remove the internal stresses that can appear during this processing method [21], plates were annealed under vacuum at 130°C during 24 hours.

The texture state of UHMWPE was generated by a uniaxial tensile test using an MTS 810 testing machine (MTS Systems, Minneapolis, MN, USA) equipped with the optical extensometer VidéoTraction (Iris Techno, Moncel-lès-Lunéville, France) [1, 2]. This apparatus provided real-time mechanical behavior (true axial stress, σ_{33} , versus true axial strain, ϵ_{33}) of a thin material section located at the center of the tensile specimen called representative volume element (RVE). Therefore, necking, which generally occurs in the specimen center, was taken into account in the measured mechanical behavior. A CCD camera recorded displacement of markers previously printed in the form of a cross on the front face of the tensile specimen [1, 2]. The speed of the servohydraulic actuator was controlled so the instantaneous true axial strain rate measured in the RVE, $d\epsilon_{33}/dt$, remained constant during the experiment. Six rectangular tensile specimens were machined from one plate to conform to the dog-bone geometry used for mechanical tests conducted with a VidéoTraction extensometer [1, 2]. The overall dimensions of these specimens were $50 \times 12 \times 4$ mm³, whereas the volume of the calibrated part was $24 \times 6 \times 4$ mm³. A geometric defect was milled in the center of the specimen with a curvature radius of 30 mm until the smallest cross-section was equal to 5.8×4 mm². Among these six specimens, three specimens were deformed, whereas the three other specimens were not deformed (reference samples). Tensile tests were performed at a temperature of 30°C and at a strain rate of 1×10^{-3} s⁻¹. The deformation state of interest was obtained after drawing the specimen up to the axial strain $\epsilon_{33} = 1.35$ and unloading it to zero stress (Fig. 1). During

Fig. 1 UHMWPE specimens were deformed in tension at 30°C and $1 \times 10^{-3} \text{ s}^{-1}$ to create a textured material. Each specimen was drawn up to the true axial strain 1.35 and was unloaded to zero stress. During the unloading stage, axial strain gradually decreased from 1.35 to 1.13.

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To assess structural features of undeformed and deformed specimens, we performed SAXS/WAXS experiments with a Panalytical X'Pert Pro MPD device (Panalytical BV, Almelo, The Netherlands). With this technique, we measured the long period L_p and the index of crystallinity X_{cw} of the samples. The radiation used, Cu $K\alpha$, was generated by an xray tube operating at 40 kV and 45 mA. A focus mirror and the PIXcel detector were used with specific slits (divergence slit, antiscatter slit, receiving slit, and soller slit). The heads of tensile specimens were first carefully cut. We positioned the central part of the specimens on the sample stage in such a way that the xray hit the RVE with the tensile axis first perpendicular to the detection direction (detection along the azimuthal direction $\phi = 90^\circ$) (Fig. 2) and then parallel to the detection direction (detection along the azimuthal direction $\phi = 0^\circ$). A last spatial direction, $\phi = 45^\circ$, was tested only by SAXS. X_{cw} was evaluated from WAXS diagrams as the ratio of total crystalline peaks area to total scattering area (including total crystalline peaks area and amorphous bump area). The long period, L_p , was calculated from the first-order scattering peak position q_p ($L_p = 2\pi/q_p$) revealed on SAXS plots of Lorentz-corrected intensity Iq^2 versus scattering vector q .

A pressure-controlled SEM, FEI FEG Quanta 200 (FEI Europe BV, Eindhoven, The Netherlands), was used to investigate the microstructure of UHMWPE in undeformed and deformed states. The morphologic features were first revealed by etching the UHMWPE samples for 18 hours in a reagent made of 65.8 wt% H_2SO_4 , 32.9 wt% H_3PO_4 , and 1.3 wt% KMnO_4 [23]. Observations of

Fig. 2 The principles of small- and wide-angle x-ray scattering analysis (SAXS/WAXS) are shown. The incident beam (0.6 mm thick and 4 mm width) penetrated through the specimen and generated scattered xray in directions depending on spatial orientation of the scattering dipoles (crystalline planes and amorphous chains). The detection direction of the equipment was horizontal. The sample was first positioned with the tensile axis perpendicular to the detection direction (detection along the azimuthal direction $\phi = 90^\circ$). Then, the sample was positioned with the tensile axis parallel to the detection direction (detection along the azimuthal direction $\phi = 0^\circ$). At low 2θ – angle (below $2\theta = 2.5^\circ$), the equipment was configured to perform SAXS measurement (optimized resolution at low 2θ – angle). Otherwise, the equipment was configured to perform WAXS measurement (optimized resolution at high 2θ – angle). For SAXS tests, the scattering intensity was plotted as a function of scattering vector $q = 4\pi \sin(\theta)/\lambda$.

the etched specimens subsequently were done at a water pressure of 150 Pa and at the accelerating voltage of 6 kV. For the deformed specimens, observations were made in the RVE where mechanical variables were assessed.

A NST nanoscratch tester from CSM Instruments (CSM Instruments SA, Peseux, Switzerland) was used to characterize surface mechanics of UHMWPE at two states of bulk deformation and along two directions (parallel and perpendicular to the tensile direction). We selected a diamond-like carbon indenter with a 2- μm tip radius (conical tip with an angle of 90°) to generate 300- μm -length scratches with a constant normal load of 100 mN and a speed of 17 $\mu\text{m}/\text{second}$ (loading and unloading rate of 20 mN/second). The normal load and speed were selected in such a way that the material did not exhibit a purely elastic response. Scratch tests were performed in the center of the tensile specimens and therefore in the RVE of textured specimens. We examined (1) the surface deformation regime (viscoelastic, viscoelastoplastic, viscoelastoplastic + cracking, brittle fracture, etc); and (2) the coefficient of friction $\mu = F_T/F_N$ (F_T and F_N being the tangential and the normal forces, respectively). We considered that the obtained deformation regime

and coefficient of friction provide information regarding the material surface toughness. In this case, this characteristic is defined as the material ability to exhibit plastic deformation without failure during the scratch deformation. In particular, the appearance of cracking during the scratch process reflects a low toughness. This increases the work done during the deformation and therefore induces a high coefficient of friction [8]. We also measured the dynamic penetration depth P_d to have information regarding the vertical deformation of the surface, ie, the full vertical viscoelastoplastic response. A final parameter, the residual depth R_d , was recorded by a second pass of the tip at a slight normal force of 1 mN just after the first scratching. R_d corresponds to the viscoplastic part of the vertical deformation assuming the viscoelastic recovery along the scratch axis is negligible. We assumed the intensity of the viscoelastic recovery process reflected the material strength. Accordingly, the surface strength was presumed proportional to the intensity of the recovery process [11] and quantified by means of the parameter $r = 1 - R_d/P_d$, called recovery parameter. The scratch groove was observed in three dimensions using the following protocol: (1) topographic information was first collected by means of a three-dimensional white-light interferometer (Wyko NT 3300; Veeco Metrology Inc, Tucson, AZ, USA); and (2) the topographic data were treated using the software Mountains Map from Digital Surf (Digital Surf Company, Besançon, France) to reconstruct the surface.

Results

The tensile deformation process caused transformation of the initial lamellar morphologic features into microfibrillar morphologic features containing lamellae fragments. Moreover, the crystalline and amorphous chains of the microfibrillar morphologic features were oriented in the tensile direction. This was supported by the following findings at the microscale (SEM investigation) and nano/subnanoscale (SAXS/WAXS investigation): At the undeformed state ($\epsilon_{33} = 0$), the material was characterized by randomly oriented and intertwined crystalline lamellae (Fig. 3A). At the textured state ($\epsilon_{33} = 1.13$), lamellae fragments mainly oriented diagonally to tensile direction and forming a chevron were observed (Fig. 3B). At the nanoscale, the lamellae fragments generated a scattering bump along $\phi = 45^\circ$ (Fig. 4A). However, we also observed scattering bump along $\phi = 0^\circ$ (Fig. 4B) and 90° (Fig. 4C) indicating the presence of scattering objects oriented perpendicular and parallel to tensile direction,

Fig. 3A–B The microstructure of UHMWPE was observed by means of scanning electron microscopy (150 Pa, 6 kV, large field detector [LFD]) at the (A) undeformed state and (B) textured state (tensile axis is vertical). At the undeformed state, randomly oriented and intertwined crystalline lamellae were seen. The textured material had microfibrillar morphologic features oriented in the tensile direction and containing lamellae fragments.

respectively. This phenomenon could be induced by the presence of lamellae oriented in these two directions. Whatever the spatial direction, the long period of textured material was lower than that of the undeformed material (nearly 16–22 versus 52 nm) (Table 1). At the subnanometer scale, the orientation of crystalline and amorphous chains in the tensile direction showed reinforcement of the reflection intensity of crystalline planes ($hk0$) and of scattering intensity of the amorphous phase along $\phi = 90^\circ$ (Fig. 5A). At the same time, deformation caused a decrease in the reflection/scattering intensity of the chains along $\phi = 0^\circ$ (Fig. 5B). This orientation phenomenon implied a low crystallinity for $\phi = 0^\circ$ (36.5 wt%) and a high crystallinity for $\phi = 90^\circ$ (57 wt%) that is nearly equal to the initial value (58.5–60 wt%) (Table 1).

Fig. 4A–C UHMWPE was characterized by small-angle xray scattering at two states of deformation ($\epsilon_{33} = 0$ and $\epsilon_{33} = 1.13$) and three azimuthal angles: **(A)** $\phi = 45^\circ$, **(B)** $\phi = 0^\circ$, and **(C)** $\phi = 90^\circ$; tensile axis is parallel to $\phi = 0^\circ$. We observed scattering

phenomena in the three directions resulting from lamellae oriented perpendicular to the tested directions. The scattering peak position was indicated by a black dot (q_p).

Table 1. SAXS/WAXS results of undeformed and deformed UHMWPE*

Axial strain ϵ_{33}	Azimuthal angle ϕ (°)	X_{cw} (%) [†]	L_p (nm) [‡]
0.00 ± 0.00	0	58.5 ± 3.5	52.3 ± 0.9
0.00 ± 0.00	45	–	50.4 ± 0.8
0.00 ± 0.00	90	60.0 ± 2.9	52.5 ± 1.1
1.13 ± 0.02	0	36.5 ± 4.4	21.6 ± 1.0
1.13 ± 0.02	45	–	16.5 ± 0.7
1.13 ± 0.02	90	57.0 ± 3.2	17.9 ± 1.0

* Average values with ranges, obtained at three azimuthal angles ($\phi = 0^\circ$, 45° , and 90°); [†] X_{cw} = the weight index of crystallinity; [‡] L_p is the long period; SAXS/WAXS = small- and wide-angle xray scattering.

The microfibrillar morphologic features had increased strength and decreased toughness perpendicular to the fibril axis and decreased strength and increased toughness

parallel to the fibril axis in comparison to undeformed UHMWPE. The values of the coefficient of friction (Table 2) combined with the microscopic observations of residual scratches (Fig. 6) enabled the tested materials to be classified according to surface toughness. As a result, toughness increased in this order: textured UHMWPE tested perpendicular to fibril axis (viscoelastoplastic and cracking + $\mu = 0.77$) < undeformed UHMWPE (viscoelastoplastic response + $\mu = 0.64$ and 0.68) < textured UHMWPE tested along fibril axis (viscoelastoplastic response + $\mu = 0.55$). The intensity of the viscoelastic recovery (Fig. 7), characterized by the parameter r (Table 2), enabled the tested materials to be classified according to strength. Thus, surface strength increased in this order: textured UHMWPE tested along fibril axis ($r = 0.53$) < undeformed UHMWPE ($r = 0.60$ and 0.58) < textured UHMWPE tested perpendicular to fibril axis ($r = 0.78$).

Fig. 5A–B UHMWPE was characterized by wide-angle xray scattering at two states of deformation ($\epsilon_{33} = 0$ and $\epsilon_{33} = 1.13$) and two azimuthal angles: (A) $\phi = 90^\circ$ and (B) $\phi = 0^\circ$; tensile axis is parallel

to $\phi = 0^\circ$. The reinforcement of (1) reflection intensity of (hk0) planes and (2) scattering intensity of amorphous bump along $\phi = 90^\circ$ indicated chains oriented in the tensile direction.

Table 2. Dynamic and residual scratch characteristics (average values with ranges) of undeformed and deformed UHMWPE

Axial strain ϵ_{33}	Direction	Mode	μ	P_d (μm)	R_d (μm)	r
0.00 ± 0.00	A	Viscoelastoplastic	0.64 ± 0.02	21.4 ± 0.5	8.5 ± 0.4	0.60 ± 0.02
0.00 ± 0.00	T	Viscoelastoplastic	0.68 ± 0.02	21.5 ± 0.4	9.0 ± 0.3	0.58 ± 0.01
1.13 ± 0.02	A	Viscoelastoplastic	0.55 ± 0.03	22.1 ± 0.6	10.4 ± 0.5	0.53 ± 0.01
1.13 ± 0.02	T	Viscoelastoplastic + cracking	0.77 ± 0.02	21.6 ± 0.4	4.7 ± 0.5	0.78 ± 0.02

* Evaluated by means of nanoscratch tester along axial and transverse directions of the specimen denoted as A and T, respectively; μ = coefficient of friction; P_d = dynamic penetration depth; R_d = residual depth; $r = 1 - R_d/P_d$ = recovery parameter.

Discussion

To produce crosslinked UHMWPE having acceptable mechanical properties, the crosslinking process could be performed on a material treated to increase strength and toughness. This can be achieved by modifying the microstructure of conventional UHMWPE by a solid-state deformation process that induces texture. However, the effect of texture on strength and toughness is not or is little documented. In this work, we studied these two mechanical properties in the case of UHMWPE textured by a tensile process and asked how (1) the microstructure of the material evolves when subjected to tension; and (2) whether the new microstructure state would increase the surface strength and toughness of UHMWPE.

Our study has some limitations. First is the small number of tests performed for each condition. Only three SAXS/WAXS, SEM, and scratch experiments were performed per deformation state. As a result, this work must be considered a pilot study. Second, we characterized the surface strength and toughness of textured UHMWPE in only two spatial directions: parallel and perpendicular to the fibril axis. It will be important to complement the study

with tests performed in other directions. Nevertheless, the proposed methodology provided the two extreme surface mechanical behaviors of the textured material. Third, we obtained only qualitative information regarding local strength and toughness. Despite the lack of quantitative information, we provided the basis of the methodology and first relevant results in accordance with previous data resulting from macroscopic measurement of strength and toughness [11, 28].

The initial lamellar morphologic features of UHMWPE (Fig. 3A) [25, 26] resulted from the low plastic flow and chain mobility that avoided the formation of spherulites during the crystallization step [15]. The mechanical drawing of the morphologic features successively involved: (1) orientation of amorphous chains between the entanglement points; (2) transmission of the stresses from the amorphous phase to the crystalline phase by means of the tie molecules; (3) rotation of lamellae; (4) slip mechanisms of lamellae; and (5) coarse fragmentation of the lamellae by kinking forming chevrons in response to deformation (Fig. 3B) [6]. This last mechanism (5) generated a scattering bump along $\phi = 45^\circ$ (Fig. 4A). In some parts of the material, the coarse fragmentation was achieved totally

Fig. 6A–C Scratches on the UHMWPE at two states of deformation were observed by optical microscopy: (A) $\varepsilon_{33} = 0$, (B) $\varepsilon_{33} = 1.13$, and (C) $\varepsilon_{33} = 1.13$, with the scratch being generated parallel, as observed on illustrations (A) and (B), and perpendicular, as observed on illustration (C), to the tensile direction (tensile direction is indicated by an arrow). These micrographs showed the scratch mode of UHMWPE that was viscoelastoplastic deformation for all the cases, except for textured UHMWPE tested perpendicular to the fibril direction, the mode being viscoelastoplastic deformation + cracking in this case. We considered that the scratch mode reflected the surface toughness. The lack of focus of the micrograph in illustration (B) was the result of the limited depth of field of the optical microscope (the groove depth was high for the investigated sample).

inducing lamellae fragments oriented parallel to tensile direction and therefore a scattering bump along $\phi = 90^\circ$ (Fig. 4C). In other parts of the material, fine fragmentation mechanism occurred, generating small lamellae fragments aligned in the tensile direction [1]. As a result, SAXS diagrams showed a scattering bump along $\phi = 0^\circ$ (Fig. 4B). All these mechanisms led to microfibrillar morphologic features [12, 17] containing lamellae fragments and amorphous and crystalline chains oriented along the tensile direction. This orientation of the chains was shown by reinforcement of the diffraction intensity of (hk0) planes and the scattering intensity of the amorphous phase in the direction $\phi = 90^\circ$ (Fig. 5A) [6]. The crystallinity was considered as slightly decreasing between the undeformed and the deformed states. The most representative index of crystallinity for the deformed state was that obtained for the direction $\phi = 90^\circ$ (Fig. 5A; Table 1), 57 wt%, considering this direction corresponds to the highest amount of

tested material in terms of mass. The deformation mechanisms described are similar to those occurring in high-density polyethylene [1, 3, 11], except for the kinking mechanism of the lamellae.

During scratching, the tangential load is the sum of (1) a compressive yield component in front of the indenter; and (2) a shear yield component underneath the contacting area [7, 13, 16]. The energy brought about by the tip scratching the materials was dissipated by viscoelastoplastic deformation (Fig. 6A–B) or viscoelastoplastic deformation + cracking (Fig. 6C). The viscoelastoplastic deformation generated by the compressive and shear stresses perpendicular to the fibril axis involved first the interfibrillar zones containing amorphous chains and defects. These regions were compressed transversally and transmitted the stresses through the tie molecules to the intrafibrillar regions. As a result, further extension of the network of entangled amorphous chains and tie molecules and further fragmentation of the lamellae occurred. The further extension of the network of entangled chains and tie molecules required a high level of stress, indicating high strength. However, limited extension of the fibrils occurred [11] as a result of a low level of energy required by slip mechanisms in the chain direction [3]. Cracks consequently were generated to dissipate the stresses once this maximum extension point was reached. During strain recovery, tie molecules and entanglements enabled a marked recovery process of intrafibrillar and interfibrillar regions [6, 11]. When the microfibrillar morphologic features were scratched in the fibril axis, the deformation involved first the interfibrillar regions that were stretched transversally before transmitting the stresses to intrafibrillar regions. Transversal stretching of these intrafibrillar regions engendered fragmentation mechanisms of the lamellae by transverse slip. This slip process required a high level of energy [3], which explains the high toughness along the fibril axis. Moreover, we supposed that the transversal stretching of interfibrillar parts induced the unraveling of tie molecules between adjacent fibrils. This last mechanism was at the origin of low strength and viscoelastic recovery.

The microfibrillar morphologic features of UHMWPE had increased strength and decreased toughness when scratched perpendicular to the fibril axis and decreased strength and increased toughness when scratched along the fibril axis. These specific properties, measured locally, were also representative of the bulk behavior. Classic rupture strength measurements performed on precompressed UHMWPE confirmed texture had opposite strength behaviors at the bulk level [28]. The two opposite toughness behaviors also were reported at the macroscopic scale by measurements of the total energy at break of prerolled high-density polyethylene [11].

Fig. 7A–D Reconstructed scratch grooves of UHMWPE at two states of initial bulk deformation are shown: **(A)** $\epsilon_{33} = 0$, **(B)** $\epsilon_{33} = 1.13$, **(C)** $\epsilon_{33} = 0$, and **(D)** $\epsilon_{33} = 1.13$. The scratch was generated parallel, as observed in illustrations **(A)** and **(B)**, and perpendicular, as observed in illustrations **(C)** and **(D)**, to the tensile

direction (tensile direction is indicated by an arrow). The intensity of the viscoelastic recovery process reflected the material surface strength. As a result, the lower the residual groove depth (R_d), the higher the strength.

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