Viscoelastic characterization of leathers properties: a novel nondestructive method using micro-indentation technique

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ABSTRACT

Collagen, the main constituent of natural skins and tanned leathers, is responsible for the viscoelastic nature exhibited by these materials. Currently, the most used analytical technique for studying these properties is Dynamic Mechanical Analysis (DMA). However, the instrumentation required for this type of analysis is not only very expensive but also needs the preparation of standardized test samples, which must be appropriately cut from a whole leather. As a result, this characterization methodology is destructive, rendering the initial leather matrix unusable and leading to material waste in an industry that already has an inherent environmental impact.
For these reasons, having access to an alternative analytical technique capable of assessing viscoelastic behavior, its temporal variations, and any modifications due to thermal or mechanical treatments should be highly advantageous. In this context, an innovative device designed by VESevo, startup at Federico II University of Naples comes into play. Originating from the world of Formula 1, the device has been designed for non-destructive evaluation of the behavior of high-performance vehicle tires using micro-indentation principles. Due to its non-invasive nature, this technology is particularly well-suited for applications where assessing the viscoelastic properties of the finished product is highly complex or, in some cases, even impossible.
One such application is in the leather industry, for the characterization of the change in the viscoelastic properties of leathers changing chemicals or after thermal or mechanical treatments. Thanks to a high-performance displacement sensor, the device defines the mechanical behavior of leather by analyzing rebound curves obtained by dropping a metal rod with a semi-spherical indenter onto the surface of the sample under analysis. A sophisticated processing algorithm takes these curves as input and outputs the viscoelastic properties in terms of Storage Modulus and Loss Factor. Additionally, a thermal sensor allows for the evaluation of these properties at different temperatures, providing a more detailed understanding of the temperature-dependent behavior of leather. In the present study a test method for the assessment of the viscoelastic behaviour of leather is proposed by means of micro-indentation principles.

Keywords: Viscoelasticity, Leather, Micro-indentation, Non-destructive technique

1. Introduction

Leather is a complex natural material primarily constituted of collagen fibers arranged in hierarchical architecture. This structural organization is not only responsible for its distinctive mechanical characteristics such as high tensile strength and flexibility which are fundamentally linked to its viscoelastic behavior. The fibrous network of collagen directly governs the time-dependent mechanical response of leather, making viscoelasticity an intrinsic consequence of its fibrous structure (Umberto Sammarco, 2007). These properties are critical to the material's performance across a variety of industrial applications, ranging from fashion to automotive sectors.

Despite its importance, the accurate assessment of viscoelastic properties in leather remains challenging due to the intrinsic heterogeneity of the material and the inherent limitations of conventional testing methodologies. Dynamic Mechanical Analysis (DMA) is widely considered the standard technique for viscoelastic characterization of polymers, composites and elastomers, enabling the quantification of parameters such as storage modulus, loss modulus and damping factor typical of a viscoelastic material. The viscoelastic properties of leather and their potential variations using DMA have been extensively investigated to assess several aspects, including the effect of tanning agent concentration, exposure to radiation, and the use of alternative tanning methods. Several studies have explored the impact of chrome tanning on the dynamic mechanical properties of bovine hide. Some have demonstrated that chrome tanning significantly increases the storage modulus (E'), enhances thermal stability, and reduces both the loss modulus (E'') and the damping factor ($\tan \delta$), particularly at higher frequencies (Nalyanya et al., 2016b). These results highlight the stiffening effect of chrome crosslinking on the collagen network, contributing to improved resistance under dynamic loading conditions. In addition to tanning chemistry, environmental factors such as UV radiation have been shown to alter the viscoelastic behavior of leather. In a separate study, the effects of UV254 exposure on leather treated with different tanning agents have been analyzed (Nalyanya et al., 2016a). Their DMA results revealed a complex response: while UV exposure led to increased stiffness and viscosity in non-tanned (pickled) hides, the effect was largely detrimental for chrome-tanned leathers, suggesting photodegradation of collagen crosslinks. Further extending the investigation of environmental stressors, the effects of gamma irradiation on tanned leather have been studied too (Department of Physics, Egerton University, Egerton, Nakuru, Kenya et al., 2024). Their work showed that low to moderate radiation doses could enhance the mechanical stability of leather, particularly in vegetable-tanned samples, due to new molecular interactions. However, at higher doses, degradation effects became dominant, especially in chrome-tanned samples. The choice of tanning agent itself has also been addressed in a comparative thermal study where the thermal degradation of leathers processed with different tanning agents, including "wet-white" (chrome-free) alternatives was evaluated (Rosu et al., 2018). They found that these alternatives influenced the thermal behavior and viscoelastic response differently from conventional chrome tanning, suggesting opportunities for sustainable leather production with tailored mechanical performance. In the end, Dynamic Mechanical Analysis was used to investigate the effect of moisture content on the viscoelastic behavior of leather, revealing that water plays a critical role in modulating its stiffness and damping response. Some studies demonstrated that the storage modulus (E') and damping factor ($\tan \delta$) of leather are highly sensitive to moisture levels throughout the drying process. Their work showed that water acts as a plasticizer within the collagen matrix, and its removal leads to a progressive increase in stiffness. The study further highlighted that humidity control is more influential than temperature in determining the mechanical performance of the dried material (Jeypalina S. et al., 2007).

However, DMA necessitates the sampling of specimens from the material, a procedure that is inherently destructive. Moreover, the high cost, substantial energy consumption and complexity of DMA

instrumentation restrict its routine application in quality control or on-site diagnostics. To address these limitations, the present study introduces a novel, micro-indentation-based methodology for the non-destructive evaluation of viscoelastic properties in leather. Originally engineered within the motorsport domain for the characterization of high-performance tires, the testing device developed by VESevo Smart Technologies integrates high-resolution displacement sensing with dynamic mechanical response analysis. The technique involves the controlled impact of a semi-spherical indenter on the leather surface, followed by analysis of the resulting rebound curves. From this, key viscoelastic parameters such as storage modulus and loss factor are extracted. Additionally, an integrated thermal sensor enables characterization of temperature-dependent mechanical behavior, a critical consideration for assessing leather performance under real-world service conditions (Farroni et al., 2024).
Unlike conventional mechanical testing protocols, the proposed technique requires no sample preparation and can be directly applied to leather or leather products. This non-invasive and non-destructive approach preserves the integrity of the material, reduces waste, and facilitates in-line or in-situ process monitoring. Its high sensitivity, repeatability, and portability make it particularly advantageous for detecting process-induced variations resulting from tanning, chemical treatments, thermal exposure, or mechanical conditioning, without disrupting production workflows.
The primary objective of this study is to validate the VESevo-based micro-indentation system as a robust, accurate and versatile alternative to traditional viscoelastic characterization methods using DMA as already verified for elastomers in automotive applications (Farroni et al., 2024). A comprehensive experimental campaign has been conducted on a representative set of leather samples to evaluate the method's reliability, precision, and applicability. The results underscore the system's potential for widespread implementation in industrial diagnostics, quality assurance and advanced material research within the leather sector. In particular, once the suitability of the technique was verified, it was used to measure the change in viscoelastic behaviour of automotive leather after mechanical and thermal treatments.

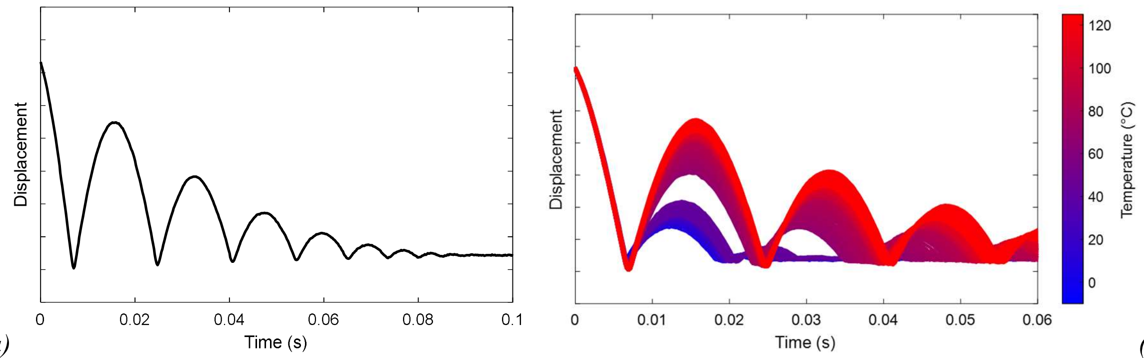
2. Material and Methods

The viscoelastic properties of leather samples were evaluated using an innovative portable device based on the principles of micro-indentation.

Figure 1 – VESevo micro-indenter for the viscoelastic characterization of materials

The device is specifically designed to ensure consistent and reliable contact between a metal indenter and the surface under test. Its operating principle relies on measuring the displacement of a steel rod, equipped with a semi-spherical indenter, that is free to fall and rebound on the material's surface. An internal high-precision mechanical system guarantees that the rod consistently starts its fall from the same height, ensuring repeatability across measurements (Figure 1).
VESevoDAQ was used to acquire the curves, and VESevoLAB was used to process the data. The acquisition software processes and displays both the displacement curve of the rod and the temperature of the tested surface in real time. Typical rebound curves are shown below, both at a single temperature and across a wide temperature range. A single curve shows distinct phases of the rod's motion that include:

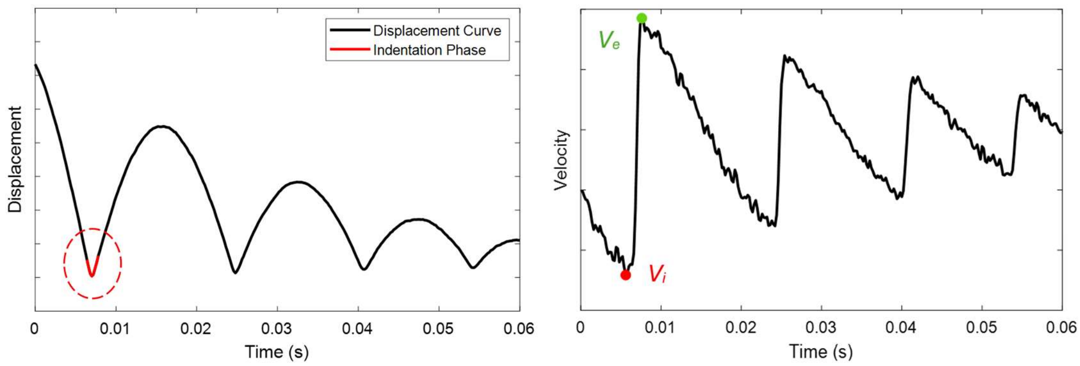
1. The falling phase from the initial release height
2. The indentation phase caused by the penetration of the indenter into the material
3. The transient phase during which the rod exhibits a damped oscillation until a final static contact is established between the indenter and the tested surface, whose displacement is strongly correlated with the inverse of the Storage Modulus.



(a) *(b)*

Figure 2 – Indentation curve (a) including temperature profile of tested surface (b)

The identification of the first indentation phase is crucial for the evaluation of the material's viscoelastic behavior, and it is carried out by deriving the displacement signal with respect to time. In Figure 3 the indentation point was highlighted in red while in Figure 3.b displacement velocity versus time is shown.



(a) *(b)*

Figure 3 - Evaluation of the indentation point: (a) displacement curve and (b) time derivative (velocity)

The resulting rod velocity signal also features characteristic points: the start and end of contact between the indenter and the surface layer under analysis. Once this phase is identified, the quadratic variation between the impact velocity (Vi, red dot) and the rebound velocity (Ve, green dot) of the indenter is proportional to the kinetic energy dissipated in the material, as described by the following equation:

$$\Delta E_k = K\left(V_i^2 - V_e^2\right) \qquad \text{(Eq. 1)}$$

which is found to be proportional to the Loss Factor (Genovese et al., 2022).
The data are processed using an algorithm capable of analyzing each displacement curve at the corresponding temperature and calculating the Storage Modulus (*E'*) and Loss Factor (*tan δ*) according to the following mathematical relationships:

$$E' = f(A_c, T, K_c) \qquad \text{(Eq. 2)}$$

$$\tan\delta = f(A_c, \omega, T, S_c) \qquad \text{(Eq. 3)}$$

where:

- A_c represents the effective contact area between the semi-spherical indenter and the compound,
- ω is the solicitation frequency associated with each test (influenced by both temperature and the material's intrinsic response),
- T is the compound temperature measured by the infrared sensor,
- K_c and S_c denote the equivalent contact stiffness and damping coefficient, respectively, and can be considered representative of the tested material, with the device spring stiffness and rod guide effects being negligible.

In this study, to determine the viscoelastic characteristics of the leathers, four different sample types have been characterized using this technique:

- Garment chrome tanned calf in Crust (Sample 1)
- Footwear bovine chrome tanned leather (Sample 2)
- Automotive glutaraldehyde crust bovine leather (Sample 3)
- By Cast PU finished split leather (Sample 4)

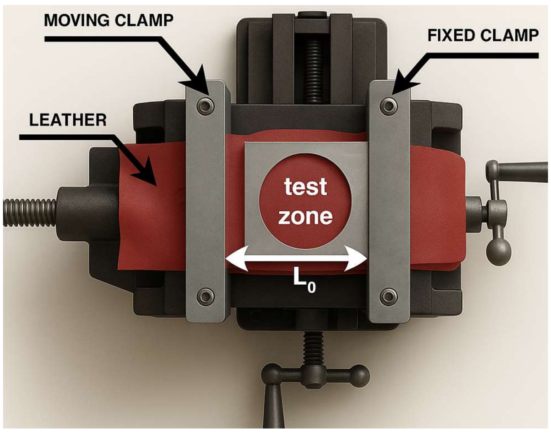


Figure 4 – Test piece leather holder

For each leather sample, specimens to be tested have been cut according to ISO 2418:2023. The device was originally designed for the characterization of elastomeric materials. Since leather is a flexible material for upholstery, its mechanical response is strongly influenced by the degree of tension in the

area where the indenter impacts. Ensuring adequate and consistent stretching in the contact zone is therefore essential for obtaining repeatable and representative viscoelastic measurements. So, for each specimen, storage modulus and loss factor have been investigated at different strain levels, 0% (no strain), 3 %, 5 %, 7 % and 10 %, using the setup reported in figure 4.
It consists of a vice on which two clamps are mounted. One of the clamps is fixed to the vice while the other one is free to move. The moving clamp can be moved by means of a screw that permits setting different strain levels. The strain is calculated by the difference between the final length (after sample is stretched, L_1) and initial length L_0, measured by means of caliper.
For each test, the micro-indenter carries out 20 measurements, providing the standard deviation of the viscoelastic parameters. All tests were carried out at ambient temperature. Dataset has been processed with VESevo processing algorithm in order to remove erroneous data due to incorrect indentation. Then a post-processing has been conducted on them. For each dataset, the mean value and standard deviation of each parameter have been evaluated and outliers beyond two standard deviations were removed.
After the verification of the performances of the test, to assess the potential use of the device for in-process control, measurements were carried out on an automotive crust leather subjected to two distinct treatments: a high-temperature exposure at 120 °C for 4 hours, and a 10 % deformation applied for 1 hour under stress relaxation conditions to simulate the leather nailing process. The tests aimed to evaluate changes in the parameters resulting from configurational modifications of the fibrous structure induced by these thermal and mechanical stresses.

3. Results and Discussion

Figure 5 shows the rebound and damping profiles of Samples 1, 2, and 3, which correspond to materials used in apparel, automotive, and footwear, respectively. It is evident that each material type exhibits a distinct and characteristic response profile, both in terms of amplitude and damping behavior. These differences, typical of their specific viscoelastic properties, are also reflected in the phase shift observed between the curves. Additionally, the final residual height after rebound may provide an indication of the perceived softness of the material and the extinction profile can be attributed to the damping component. However, static softness was not considered in the present study.

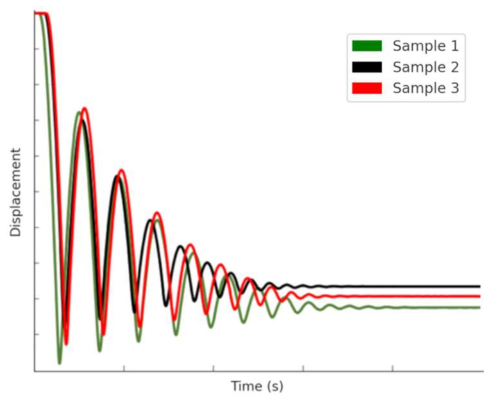


Figure 5 - Rebound and damping profiles of Samples 1, 2, and 3

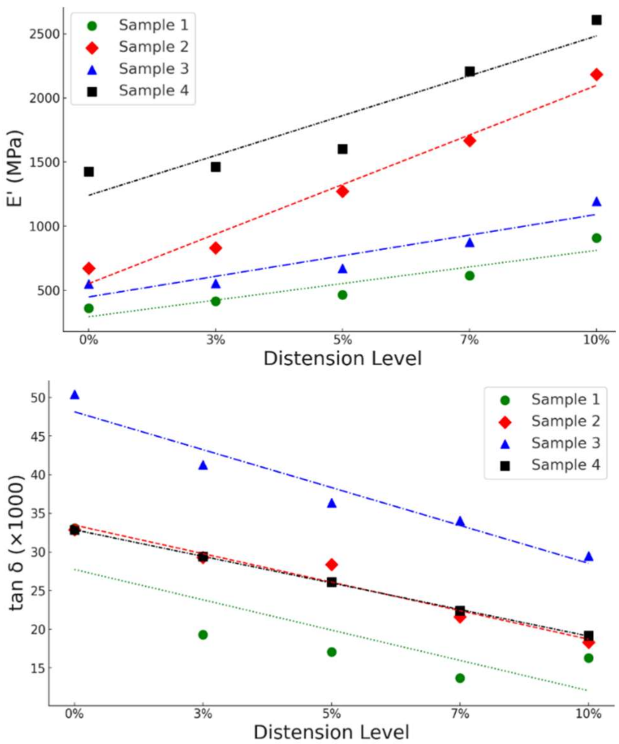
In Table 1 and Table 2, the results of E′ and tan δ obtained for all samples at different initial strain levels are reported. The same results are also illustrated in Figure 6. It is evident that even at 0 % strain, the data are consistent and reproducible, indicating that the application of initial strain may not be strictly necessary for the evaluation of viscoelastic parameters. However, in future comparative analyses, a minimum strain of 3 % will be applied to ensure flat positioning of the leather samples, avoiding wrinkles and folds during testing. The device proved capable of determining viscoelastic parameters and detecting variations in the material related to configurational changes in the fibrous structure. In fact, as strain increases, fiber alignment occurs, leading to a measurable change in mechanical response. For each sample, increasing the initial strain leads to an increase in the storage modulus (E′) and a reduction in the loss tangent (tan δ). This behavior can be explained by the progressive alignment of collagen fibers along the loading direction under higher strain. As the fibers become more oriented, their ability to slide past each other diminishes, resulting in a stiffer structure with reduced internal friction and energy dissipation. Consequently, the material exhibits a more elastic and less viscous response. This strain-dependent behavior of fibrous materials has been widely described in literature, particularly in studies on soft biological tissues and collagen-based materials (Fung, 1993).

DISTENSION	**0%**		**3%**		**5%**		**7%**		**10%**	
SAMPLE N.	**E'[kPa]**	***St.Dev***	**E'[kPa]**	***St.Dev***	**E'[kPa]**	***St.Dev***	**E'[kPa]**	***St.Dev***	**E'[kPa]**	***St.Dev***
Sample 1	362	*36,0*	415	*37,1*	465	*29,4*	615	*46,1*	909	*45,6*
Sample 2	671	*19,8*	831	*33,0*	1273	*23,8*	1667	*13,9*	2184	*43,4*
Sample 3	551	*7,8*	556	*7,3*	673	*15,1*	877	*23,4*	1194	*25,7*
Sample 4	1427	*19,8*	1464	*33,0*	1603	*23,8*	2208	*13,9*	2608	*43,4*

Table 1 – Storage Modulus E' in kPa at different strain level

DISTENSION	**0%**		**3%**		**5%**		**7%**		**10%**	
SAMPLE N.	**tan δ**	***St.Dev***	**tan δ**	***St.Dev***	**tan δ**	***St.Dev***	**tan δ**	***St.Dev***	**tan δ**	***St.Dev***
Sample 1	28,1	*0,92*	25,3	*1,41*	24,7	*0,72*	23,0	*1,12*	20,2	*1,51*
Sample 2	33,1	*2,06*	19,3	*1,02*	17,1	*1,69*	13,7	*0,51*	16,3	*1,30*
Sample 3	32,9	*1,19*	29,3	*1,31*	28,4	*1,25*	21,6	*1,13*	18,3	*0,72*
Sample 4	50,4	*1,13*	41,3	*0,89*	36,4	*0,90*	34,1	*2,23*	29,5	*0,53*

Table 2 – Damping factor tan δ at different strain level



(a)

(b)

Figure 6 – Storage Modulus E' (a) and Damping factor (tan δ) (b) at different strain level

As an example of the application of the micro-indentation technique in a specific sector, the case of automotive leather was considered. Crust leather was subjected to two distinct conditioning treatments commonly used in the automotive sector:

- Mechanical conditioning: a 10 % strain applied to the test specimen for 1 hour under stress-relaxation conditions to simulate the nailing process.
- Thermal conditioning: exposure to 120 °C for 4 hours, a common pre-treatment used in the automotive leather industry to contract the hide and enhance its performance under cyclic hot–dry and cold–humid conditions.

The results are summarized in Figure 7. As expected, mechanical conditioning led to alignment of collagen fibers in the loading direction, resulting in an increase in storage modulus (E′) and a decrease in loss tangent (tan δ). This behavior can be explained by the improved load-bearing capacity of aligned fibers and reduced internal viscous friction, phenomena well-documented in the literature (Fung, 1993). Similarly, thermal conditioning at elevated temperature increased both E′ and tan δ due to the so-called "fiber sticking" effect. Heat promotes the formation of additional cross-links and physical bonds between collagen fibrils, which stiffen the structure but also enhance energy dissipation through internal friction. Studies support this finding, reporting increased stiffness and damping in heat-treated collagen-based materials due to thermally induced interfibrillar bonding (Schrompfer M. and Meyer M., 2011).

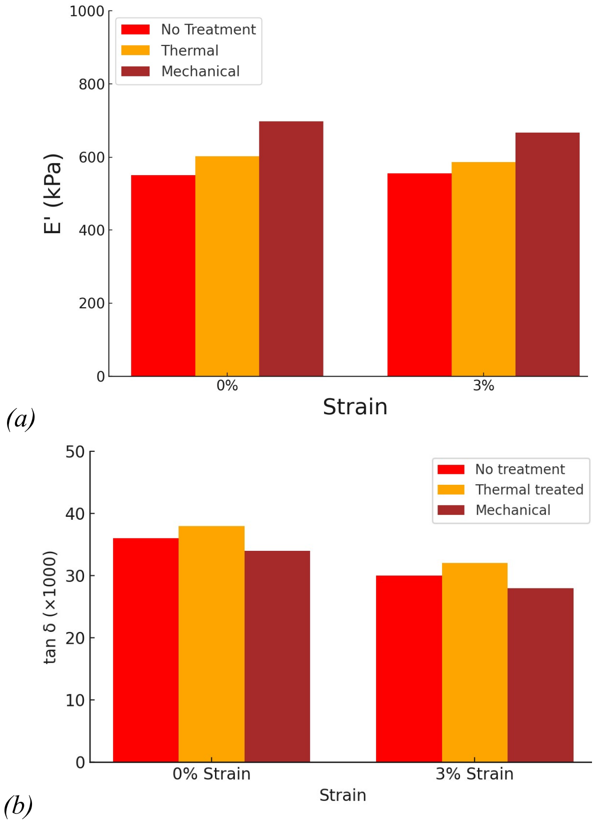


Figure 7 – (a) Storage Modulus and (b) Damping factor for automotive leather after thermal and mechanic treatment

Also in this case, the micro-indenter allowed to measure the changes in viscoelastic behaviour of the material after the above described thermal and mechanical treatment.

4. Conclusions

The VESevo micro-indenter has proven to be a reliable and versatile tool for the characterization of viscoelastic materials through non-destructive testing. The method allows for the evaluation of key mechanical parameters, such as storage modulus (E′) and loss tangent (tan δ), with high sensitivity to both material composition and structural configuration.
The device was also successfully applied to leather, a fibrous and heterogeneous material, confirming its suitability for complex substrates. The results obtained showed that VESevo could detect even subtle changes in viscoelastic behavior resulting from thermal and mechanical conditioning, such as those simulating automotive production and using scenarios.
In particular, the observed increases in E′ and changes in tan δ following stress relaxation and thermal treatment are consistent with known structural transformations of collagen fibers, including fiber alignment and increased intermolecular interactions. This highlights the ability of VESevo micro-indenter to provide insight into the microstructural mechanisms that influence the macroscopic mechanical response of soft and fibrous materials.
Overall, this device represents a promising and efficient approach for the in situ, non-invasive assessment of viscoelastic properties, with potential applications in quality control and material development in sectors such as automotive, footwear, apparel, and biomedical materials.

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