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Mechanism of tensile stress release in the keratin fiber cuticle: I

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Synopsis

During the extension of keratin fibers, their two major morphological components, the cuticula and the cortex, accommodate the stresses imposed on the fiber each in a totally different fashion. While the latter extends by mechanisms that have been discussed extensively and appear to be well understood, the cuticle cells are essentially inextensible and have to move relative to one another. In the multilayer structure of the cuticular sheath of human hair fibers, this relative movement has to be accommodated by the various layers within each cuticle cell and by the bonding layers between cells, and finally causes the lifting of surface scale edges at higher strain levels. It is proposed that extension mainly causes shear stresses between layers of different composition and extensibility within the cuticle cell. This leads to failure in the weak endocuticular layer and results in "delamination" and lifting of the outer layers of the surface cuticle. The damage is irreversible upon release of the fiber and immersion in water, as reflected in the onset of scale lifting at considerably lower strain levels during a second extension. Scale lifting was not observed during the extension of wool fibers, which appears to be a reflection of the higher rigidity of the cuticle cells of wool.

INTRODUCTION

Standard grooming practices, such as shampooing, combing, and brushing, can cause considerable damage to the cuticle of human hair, as summarized recently by C. R. Robbins (1). Apart from the general abrasive loss of cuticle layers, which can be quite severe and can eventually lead to the complete loss of the cuticular sheath and formation of split ends (2–5), there are various processes that damage the cuticular structure in such a way that scale edges become particularly vulnerable to abrasive action. One of the processes of damage introduction is the imposition of stresses on individual hair fibers during combing, especially when encountering a snag. These stresses result in reversible fiber extension as well as in some irreversible processes involving the cuticle.

The two major morphological components of the hair fiber respond quite differently to the stresses introduced into the fiber during extensions beyond the yield point. The cortex is able to release stresses by the unfolding of α helical structures into the pleated sheet arrangement of the β structure. This α–β transformation occurs in the microfibrils that are embedded in and interconnected to the disulfide cross-linked noncrystalline matrix material that deforms along with the unfolding of the α helices. There is thought to be no relative displacement of cortical cells. This mechanism of deformation, which is reflected in the stress-strain curve of the fiber, has been discussed extensively in the literature and appears to be well understood and generally accepted.

The deformation response of the other major morphological component, the cuticular sheath, is much less well understood. In torsional deformation, Wolfram and Albrecht (6) have found that in the wet state the modulus of the cortex is twenty times greater than that of the cuticula, while in the dry state the torsional properties of cortex and cuticula are similar. In extensional deformation, on the other hand, it is thought that the cuticula does not contribute to the stress-strain curve of the fiber. The cuticle cells are much more rigid in the longitudinal direction and do not have the capacity to release stress by transformation on a molecular level like the cortical cells. Since the cuticular sheath has to deform concomitantly with the deformation of the cortex and the whole fiber, it is assumed that the deformational stress that occurs on the cuticular sheath is released by the movement of cuticle cells relative to each other.

The individual cuticle cell is a multilayered structure enveloped by a cellular membrane, the epicuticle, and it adheres to neighboring cuticle cells through the intercellular cement. The outermost cuticle layer is the highly disulfide cross-linked A layer in which 30–35% of the amino acids are half cystine, making it extremely tough and inextensible. The next layer, the exocuticle, has a somewhat lower cross-link density and would be expected to be somewhat more easily deformable. The innermost layer is comprised of the much less cross-linked, easily swellable, and highly extensible endocuticle. During extension the difference in deformability of the various layers would be expected to result in the generation of shear forces that develop between these layers. At higher extension levels these shear forces can lead to stress concentrations and failure in the weakest layer, the endocuticle.

We have studied the response of the surface cuticle of human hair and wool fibers to extension by fluorescence and scanning electron microscopy and will discuss details of that investigation in the following paragraphs. While extension levels used in this investigation are mostly higher than those normally encountered in hair grooming procedures, our results shed light on the deformation processes in the cuticular sheath and its most vulnerable components.

EXPERIMENTAL

MICROFLUOROMETRY

Individual hair fibers are mounted in an extension frame and extended manually under ambient conditions (~50% relative humidity, room temperature) while one observes the fiber in a fluorescence microscope (Leitz MPV 1.1 with Ploemopak attachment). Upon excitation in the ultraviolet range (nonpolarized light, 340–380 nm), the untreated unextended hair fiber tends to glow with a weak and diffuse blue-white autofluorescence (Figure 1, left). This intrinsic fluorescence of proteins upon excitation in the UV is based on the presence of certain aromatic amino acids, i.e. tryptophane, tyrosine, and phenylalanine (7,8). It is known that the disulfide groups of cystine are fluorescence quenchers and that their environmental or oxidative scission leads to a significant increase in fluorescence intensity. During extension the surface cuticle edges become

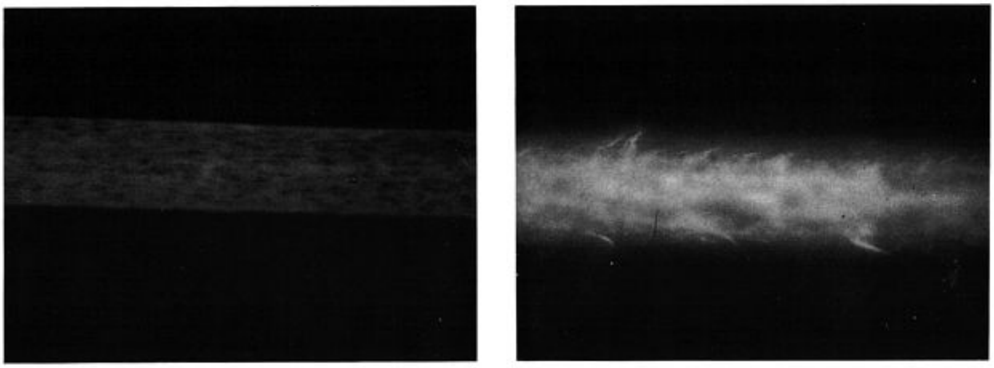

Figure 1. Microfluorometric views of typical hair fiber root sections before (left) and after (right) extension. Excitation at 340–380 nm.

visible as they start to move, and brilliant white lines develop at the scale edges as random scale lifting starts in various locations along the hair fiber. It is conceivable that the increased fluorescence intensity at the scale edges during initial fiber extension is indicative of the onset of relative scale movement, revealing endocuticular material that is no longer protected by the quenching effect of the exocuticle with the particularly high disulfide content in its A-layer. Upon further extension and scale lifting, more endocuticular and intercuticular material is exposed and fluorescence intensity increases further. The possibility of fluorescent light scattering upon separation of the scale edge

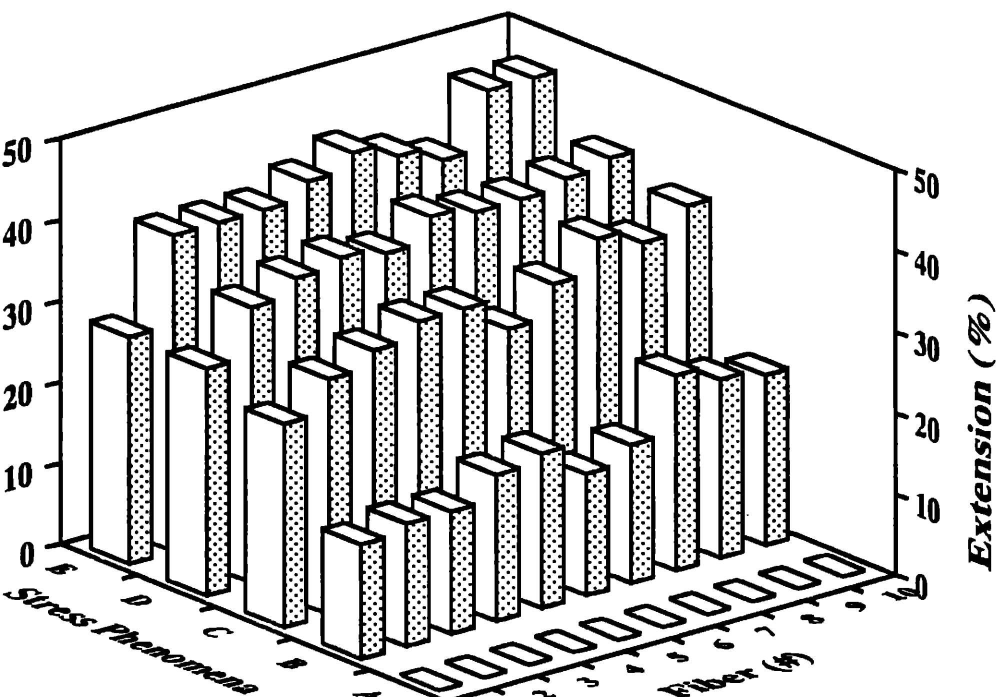

Figure 2. Extension of near root section of untreated, brown European hair fibers. A: Unextended hair fiber. B: Random scale lifting. C: Common scale lifting. D: Extreme scale lifting. E: Hair fiber breakage.

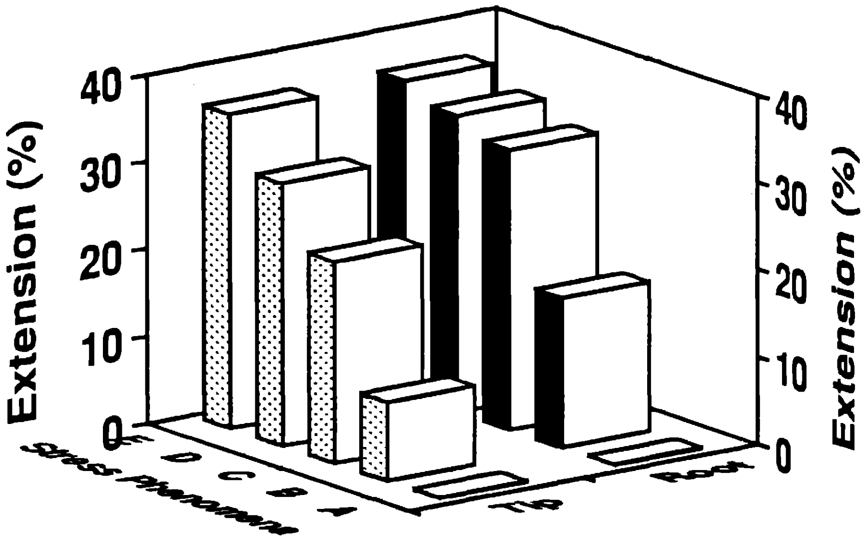

Figure 3. Comparison of scale lifting phenomena in root and tip sections of hair fibers. A: Unextended hair fiber. B: Random scale lifting. C: Common scale lifting. D: Extreme scale lifting. E: Hair fiber breakage.

from the scale face below and the formation of stress concentrations at the scale edges should also be considered as a source of increased fluorescence intensity. The random scale lifting and brilliant scale lines become more common upon further extension, and finally scale lifting becomes extensive in frequency and angle (Figure 1, right), followed shortly thereafter by breaking of the hair fiber. The extension levels at which specific scale lifting phenomena occur were recorded.

SCANNING ELECTRON MICROSCOPY

The extended fibers were mounted on metal stubs and were studied in a JEOL-JSM-12 and more recently in a Hitachi S-4500 field emission scanning electron microscope.

REVERSIBILITY OF EXTENSION

Fibers were extended to 30–35% under ambient conditions, held in the extended state for ~5 min, and then released and transferred into water at room temperature. After 20 hours the fibers were dried and conditioned (65% RH, 21°C) for at least 24 hours. These fibers were then re-extended under the microscope, and the scale lifting phenomena were re-examined. The mechanical properties of fibers with and without prior 30% extension were determined in water by using an Instron tensile tester.

RESULTS AND DISCUSSION

In the initial studies, root sections of 18-in-long brown European hair (from DeMeo Brothers, New York) were used, in order to eliminate some of the damage that is inflicted on the cuticular region by standard grooming practices and to ensure the presence of multiple cuticle layers in the cuticular sheath. In our efforts to quantify the phenomena observed during extension, we decided to establish the extension level at which characteristic scale lifting phenomena are observed. We differentiate among (A) the unextended hair, (B) the first randomly distributed lifting of individual scales, (C) common scale lifting along the whole length of the fiber, (D) extreme scale lifting both in frequency and angle, and finally, (E) breakage of the fiber. The results for the extension of the root section of ten (10) hair fibers are shown in Figure 2.

When the studies were extended to the near tip section of 18-in-long hair, we observed that the onset of scale lifting occurred at significantly lower extension levels, with the other stress phenomena shifted similarly to lower extension levels (Figure 3). Only the final breakage of tip and root sections occurred at approximately the same level of extension. This shift to lower extension levels clearly indicates that grooming and exposure to stresses experienced during the longer lifetime of the tip section have caused a loosening of scale edges, possibly involving damage to the endocuticular layer as well as damage to and conceivably loss of the intercellular domains, which is definitively irreversible in nature.

In an effort to confirm the cuticular damage observed in autofluorescence and to explore details of failure at the scale edge resulting in the scale lifting as well as any other potential events occurring on the surface cuticle during extension, the extended fibers were transferred to the scanning electron microscope. Highlights of the SEM study displaying the various damage phenomena at specific extension levels are seen in the micrographs shown in Figures 4a–l.

The most dramatic damage phenomenon is the scale edge lifting, which increases in frequency and angle with increasing levels of extension. The frequent appearance of granular material underneath lifted, chipped away, or broken-off surface cuticles, which has been associated with endocuticular debris by Swift (9), suggests that the endocuticular layer is indeed a region of weakness within the cuticle cell. Based on these observations, we can conclude that endocuticular failure precedes scale edge lifting. As pointed out above, the difference in amino acid composition of the different layers of the cuticle cell leads to large differences in swelling and, with it, deformability. During extension of the fiber, this difference in deformability sets up shear forces between these layers, as indicated schematically in Figure 5.

At higher extension levels, this shearing action can lead to stress concentrations and finally failure at the edge of the endocuticle. This edge failure results in a partial "delamination" within the scale structure, and under the influence of the shear forces the upper layers of the surface scale are lifted up, starting at the scale edge. J. A. Swift (9) observed the phenomenon of endocuticular failure during wet combing, where failure of the swollen endocuticular layer within the cuticle cell resulted in severe scale fracturing.

It is well known that the extension of keratin fibers, at least within the yield region up to extensions of 25–30%, is totally reversible upon release of the fiber and its immersion in water. Similarly, extensive and, for some properties, complete recovery of mechanical properties has been observed. In our study, we have observed a total length recovery after release and water immersion and a partial recovery of the extension-induced cuticular scale lifting described above. This recovery was similar in tip and root sections, with most lifted cuticles returning to their original apparently tightly stacked configuration (Figure 6). However, occasional scales that had been lifted to an extreme angle remained

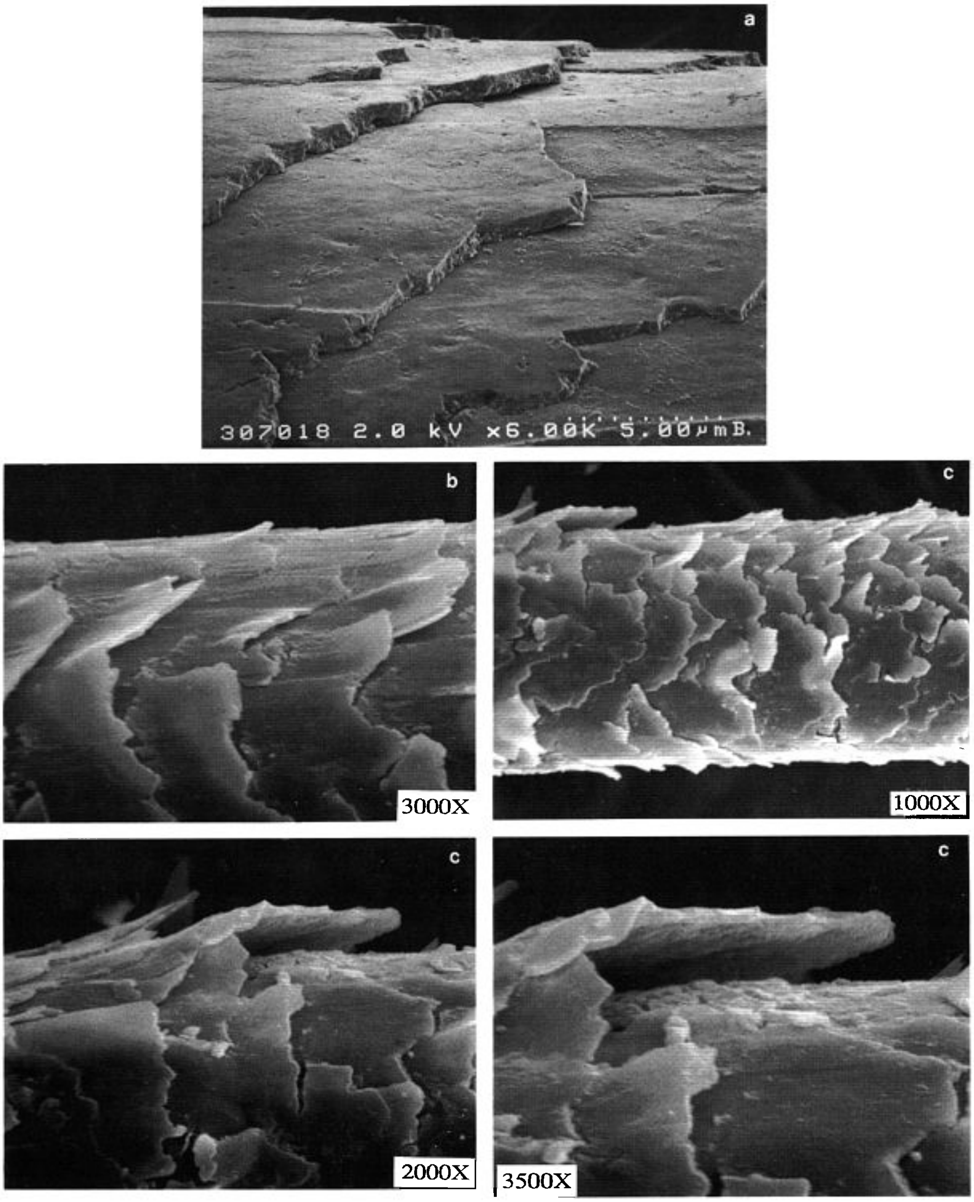

Figure 4. a–c. Highlights of cuticular damage observed in autofluorescence in a light microscope. **a:** Untreated extended hair fiber. **b:** Typical view of an untreated hair fiber extended to ~20%, displaying the start of scale edge lifting. The increasing magnifications of **c** show a high level of scale lifting of surface cuticles, scale buckling, and cracking. The high magnification clearly displays the torn-off endocuticular material underneath the lifted cuticle. Endocuticular failure results in scale edge lifting.

frozen in that position even after relaxation in water. Manifestations of the previously imposed stresses that brought about the scale lifting, buckling, cracking, and in some cases the appearance of exposed endocuticular and intercellular materials can still be seen on the fiber surface. It appears, therefore, that while recovery of the cortex and reversal

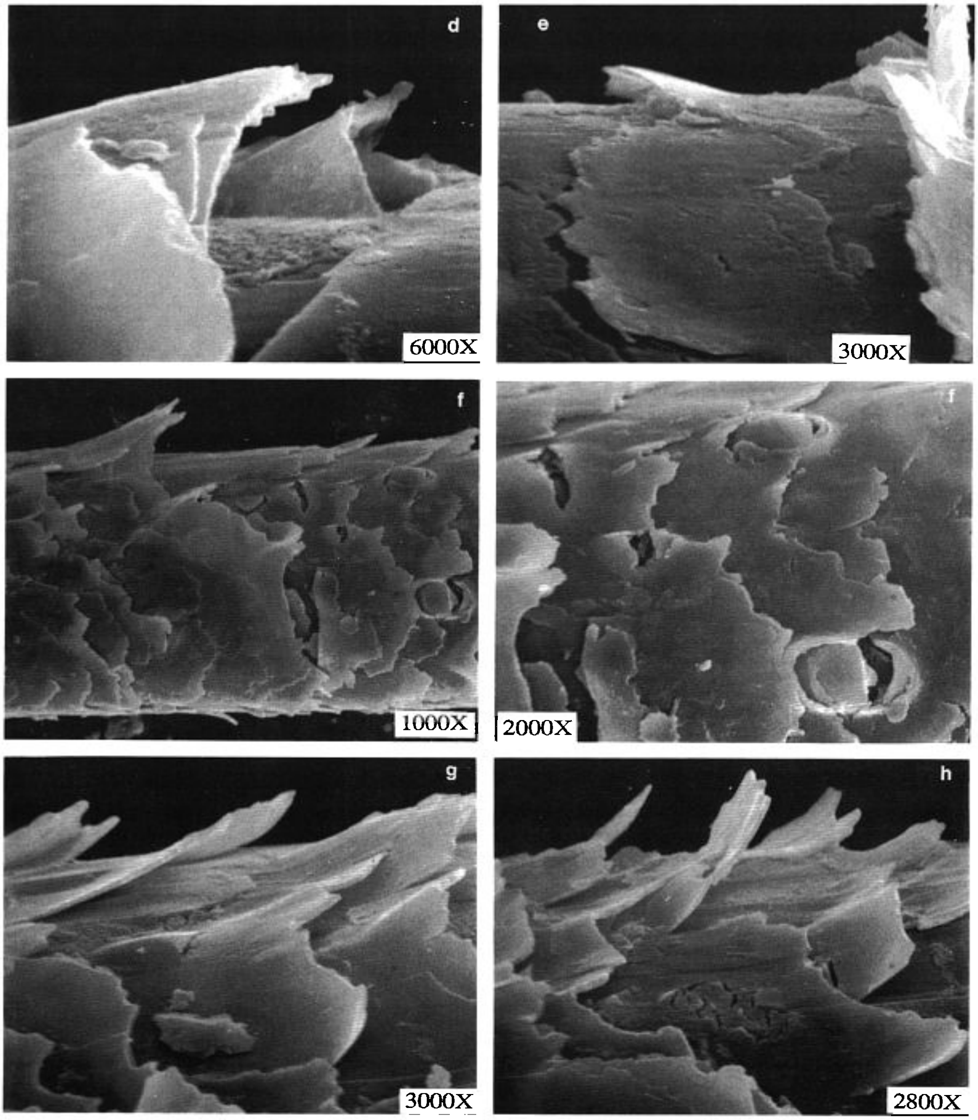

Figure 4. d–h. d: A high level of scale lifting and torn-off endocuticular material underneath the lifted surface cuticle. **e:** Extension-induced, jagged tear lines. **f:** Severe cracking, buckling, and lifting of the surface cuticles and unexplained "plugs," possibly connecting points between cuticle and cortical cells. **g:** Splitting between the surface cuticle's exocuticles A and B, resulting in scale lifting. **h:** The endocuticular layer as surface structure, after epicuticle and exocuticles A and B were worn off.

of the α–β transformation may indeed by complete, the cuticular damage experienced during extension up to 32–36% is not reversible.

We have explored the recovery of the mechanical properties of human hair root and tip sections from 30% extension followed by release and immersion in water for 20 hours. The data shown in Table 1 show very little difference in the mechanical properties between root and tip section, and indicate extensive, although not total, recovery of the

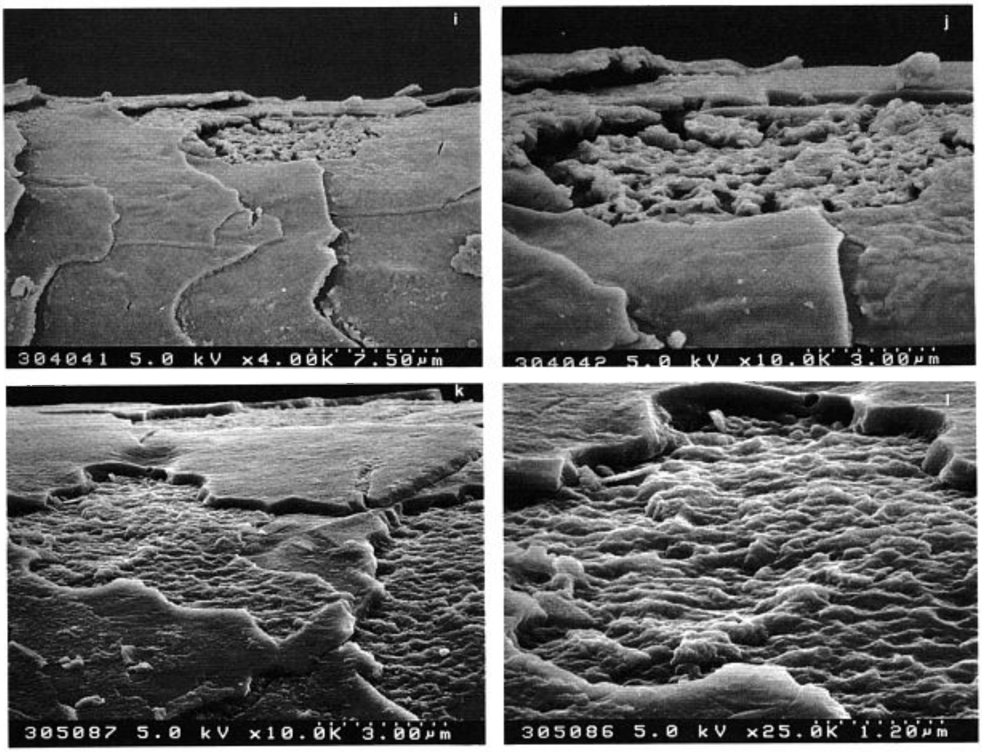

Figure 4. i–l. Extension-induced failure in the endocuticle facilitates scale lifting and chipping away, thus exposing the endocuticle as surface structure.

properties after 30% extension. The tip section shows a slightly lower recovery than the root section, especially in properties such as initial modulus and yield stress, although the differences here are small and not statistically significant.

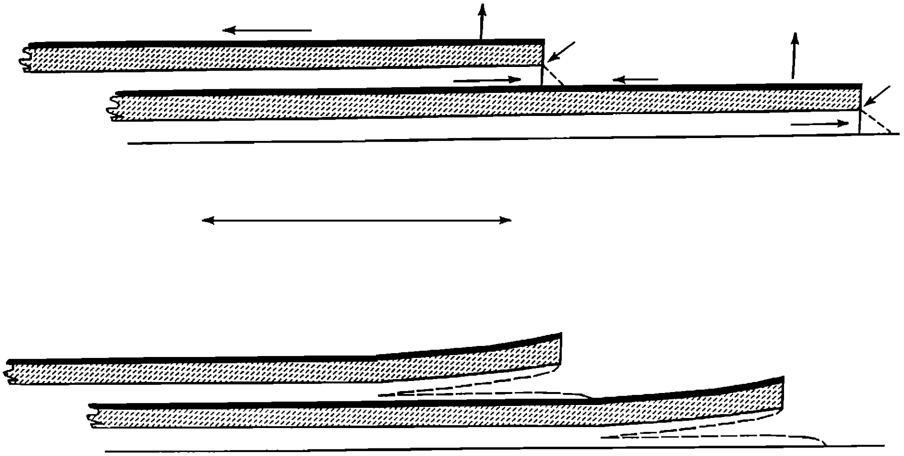

Figure 5. Development of shear forces between cuticular layers during fiber extension and failure in endocuticle leading to surface scale lifting.

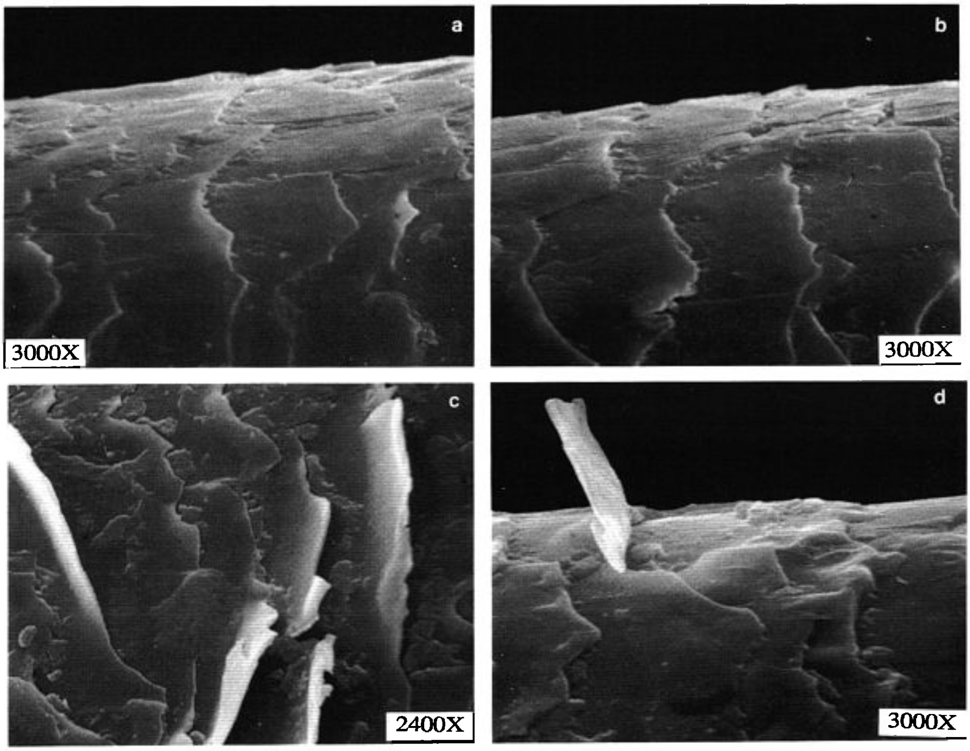

Figure 6. **a**: Root section of untreated hair fiber after 32–36% extension and overnight relaxation in deionized water. **b**: Tip section of untreated hair fiber after 32–36% extension and overnight relaxation in deionized water. **c**, **d**: Cuticles of untreated hair fiber that appear frozen in the lifted position after 32–36% extension and overnight relaxation in deionized water.

In an effort to establish to what extent the visual cuticular damage that still exists after the recovery process affects the various extension phenomena, we carried out a second extension of these fibers. In Figure 7 the damage phenomena in the root section during first and second extension are compared, and it is clearly seen that the damage phenomena occur at much lower extension levels during the second extension. In other words, the first extension has caused irreversible damage, especially in the endocuticular

Table I
Effect of Extension on the Mechanical Properties of Human Hair: Mechanical Properties of Root and Tip

			Break properties		
	Mod.	Yield stress (GN/m^2)	Stress	Energy (MJ/m^2)	Ext. (%)
Root	3.73	9.8×10^{-2}	20.2×10^{-2}	3.01	46.6
Tip	3.90	9.7×10^{-2}	19.3×10^{-2}	2.84	45.2
	Relative recovery (%) of properties after 30% extension				
Root	95	92	95	97	104
Tip	90	90	95	94	102

domain, which is not surprising in view of the extent of damage shown in Figure 4. The much more extensive lack of reversibility after the first extension for the tip section is shown in Figure 8. Here the comparison of root and tip sections shows that the second extension produces the various scale-lifting phenomena at considerably lower extension levels in the tip section, reflecting the effects of several years of grooming on the cohesion within the scale structure.

The almost total reversibility of the mechanical properties of the fiber, and the absence of such reversibility in the properties of the cuticula after extension, release, and immersion in water, indicates that the cuticula does not contribute to the tensile properties of the fibers. These observations support the conclusion of Robbins and Crawford (10) that the cortex is responsible for the tensile properties of human hair and that damage to the cuticula is not reflected in these properties.

CUTICULAR RESPONSE TO EXTENSION OF WOOL FIBERS

We have extended our investigation of the response of the cuticula to keratin fiber

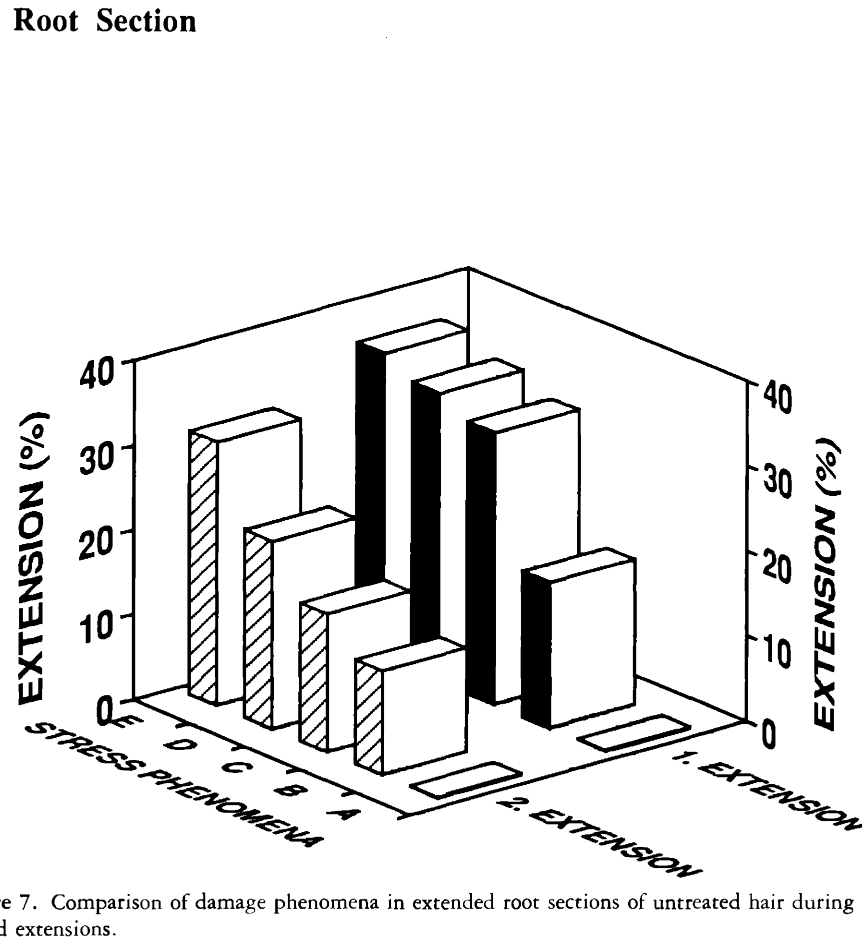

Figure 7. Comparison of damage phenomena in extended root sections of untreated hair during first and second extensions.

Tip Section

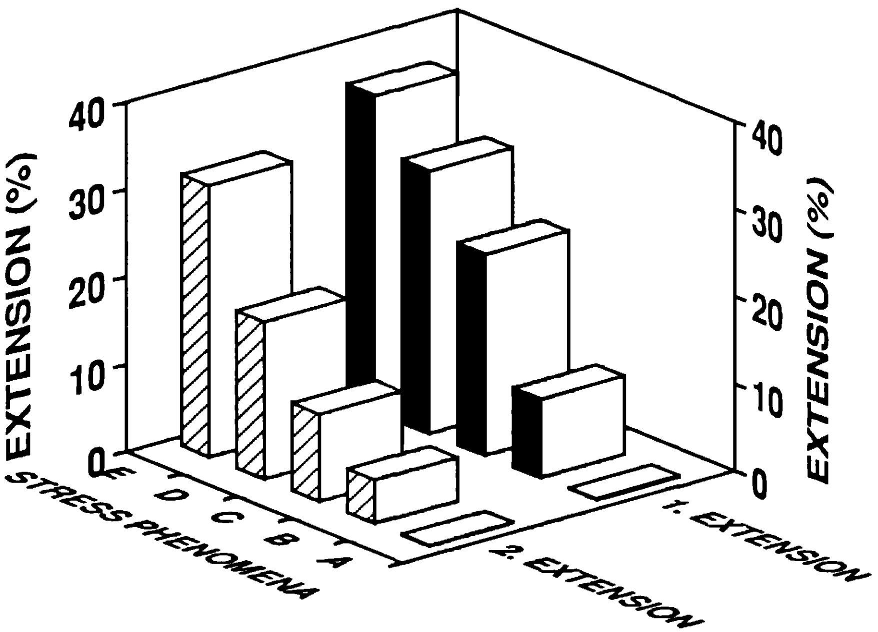

Figure 8. Comparison of damage phenomena in extended tip sections of untreated hair during first and second extensions.

extension to include a preliminary study of wool fibers, which in contrast to the multilayered cuticular structure of the human hair fiber show only a single overlapping layer of cuticle cells. A fine pen-fed Merino wool (~25 μm) and a coarse Coopworth wool (35–40 μm) were used.

Microfluorometry of unextended wool fibers shows diffuse overall autofluorescence when viewed in the UV exitation beam with some highlighting of individual scale edges. In the case of the fine wool, no autofluorescence phenomena suggesting scale lifting could be observed. Only a more pronounced highlighting of scale edges was seen in the form of a fine line. Extension of the coarser wool also failed to produce any scale-lifting phenomena. An increase in fluorescence intensity at the scale edges was observed, but it is impossible to establish whether this increase in brightness is due to scale lifting or simply an indication of stress.

SEM studies of the extended fine and coarse wool fibers confirmed the observations in the fluorescence microscope, i.e., the absence of scale lifting upon extension of the fibers. As seen in Figure 9 a,b and 10 a–c, separation at the scale edges is the common response to fiber extension. Failure seems to occur in the intercellular cement of the cell membrane complex, although there may also be some involvement of the endocuticle.

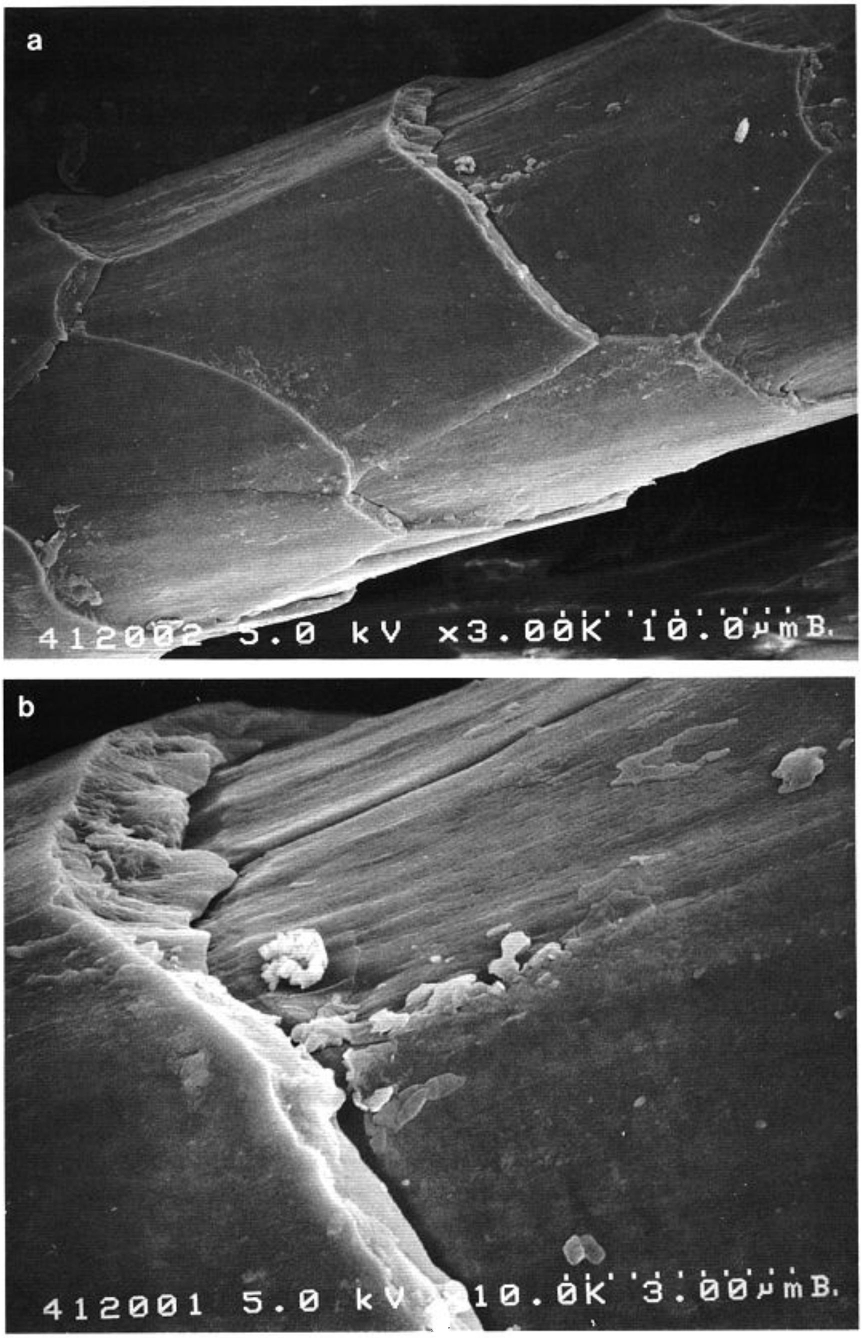

Figure 9. Typical damage to fine wool fiber surface due to extension. **a:** Fiber extended ~25%, magnification ×3000. **b:** Fiber extended ~25%, magnification ×10,000.

It appears that movement of the scales relative to each other—although surprisingly low levels of relative scale displacement in comparison to the strain level are observed—alleviates the stress buildup during extension, similar to the situation in human hair

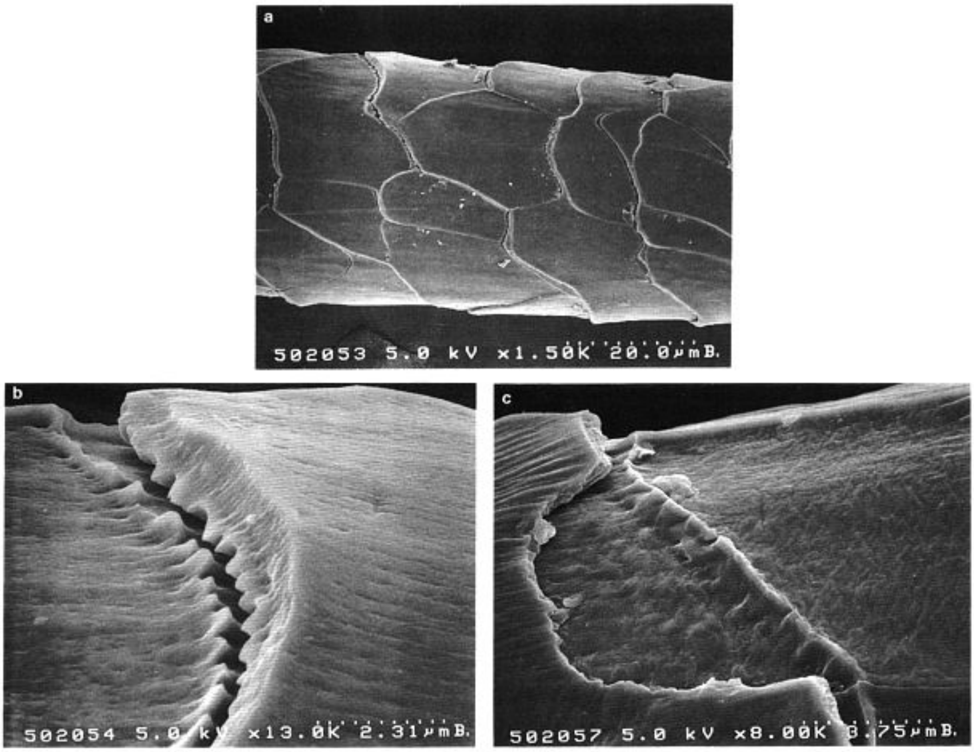

Figure 10. Typical damage to coarse wool fiber surface due to extension. **a:** Fiber extended ~30%, magnification ×1500. **b:** Fiber extended ~30%, magnification ×13,000. **c:** Fiber extended, ~30%, magnification ×8000.

fibers, but without any lifting of the surface scale. The lack of scale lifting may be due to the relatively larger thickness and, with it, rigidity of the cuticle cells in wool fibers. Furthermore, the scale cells on wool fibers are highly curved around this lower-diameter fiber. In fact, for very fine wool fibers, single-cuticle cells may envelop the whole fiber (11). This high curvature gives the cuticle cells additional rigidity and thus may prevent the scale lifting observed for the much less curved cuticle cells in human hair fibers. The need for further work regarding the cuticular response to keratin fiber extension is clearly indicated.

SUMMARY

We have established that surface scale edge lifting occurs during extension of hair fibers under ambient conditions. We used microfluorometry to quantify this loss of adhesion by measuring the extension at which various scale-lifting phenomena occur. We think that scale lifting is due to the development of shear forces during the movement of individual scale cells relative to each other. Failure occurs in the endocuticular domain, and the scale edge begins to lift. The onset of scale lifting occurs at lower extension levels in the tip end of the fiber, where previous grooming damage has been experienced.

When the fiber is released and immersed in water, the fiber length and mechanical properties are largely recovered. However, this is not true for the cuticular layer, i.e., the surface scale. In this domain, irreversible damage has occurred, especially at the edge of the endocuticular layer of the surface scale.

We feel that this irreversible damage to the endocuticle during fiber extension due to grooming may well be responsible for the breaking off of scale fragments during wet combing that was observed by Swift and Brown (4). It is therefore an important part of the scale ablation process in which new surfaces are generated upon loss of the surface cuticle until the whole cuticular sheath has been abraded.

During the extension of fine and coarse wool fibers, no scale lifting was observed. The response of the cuticular structure to the extension of wool fibers appears to be the relative sliding of the scales, with failure in the intercellular cement between cuticle cells. Scale lifting may be prevented by the higher rigidity of the scale cells of wool, which are thicker and more highly curved than those of human hair.

ACKNOWLEDGMENTS

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