Effects of thermal treatments with a curling iron on hair fiber

S. B. RUETSCH and Y. K. KAMATH, *TRI/Princeton, Princeton, NJ 08542.*

Synopsis

The effect of curling hair with a curling iron has been investigated. Possibilities of thermal damage with repeated curling according to, and in violation of, the manufacturer's specifications have been studied. The propensity of hair surface to damage depends on the moisture content of the hair, and these experiments have been conducted in both wet and dry conditions, with and without application of tension, and with short or prolonged times. Scanning electron microscopic (SEM) examination revealed that fibers treated under the dry condition (50% RH) show radial and axial cracking along with scale edge fusion. Similar thermal treatment on wet hair resulted in severe damage of the type described above, as well as bubbling and buckling of the cuticle due to the formation and escaping of steam from the fiber. Fibers subjected to repeated curling in the dry condition show slight increases in tensile mechanical properties, characteristic of a crosslinked fiber. Fibers treated with conditioners show an improvement in characteristic life, especially in the case of low-molecular-weight conditioners, such as CETAB, which can penetrate into the hair fiber (shown by TOF-SIMS analysis).

INTRODUCTION

Temporarily curling hair by application of heat is one of the grooming practices used for hair styling. This work examines the effects of such thermal treatments with a curling iron on the cuticular structure of the hair fiber. Manufacturers of curling irons claim that no damage is inflicted upon the hair fiber if the curling iron is used according to the specified treatment conditions, such as dry hair, short curling times, and normal tension (no applied load).

The process of curling, by its very nature, has the potential for hair damage if the specifications are ignored. Therefore, we carried out a multifaceted study involving the recommended conditions, as well as "severe" conditions that ignored the specifications of the manufacturer of the curling iron. The protocols we investigated were the effects of:

1. *Repeated, cyclical short-term* curling/cooling of *dry* hair under *moderate tension* as specified by the manufacturer of the curling iron
2. *Prolonged* curling of dry hair under *increased tension*
3. Repeated, cyclical *short-term* curling/cooling of *wet* hair

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4. *Prolonged* curling of *wet* hair under *increased tension* with no regard to the specifications.

Obviously, repeated curling was adopted to understand the cumulative effect of this grooming procedure on hair damage. The other purpose of this study was to understand the combined effect of hygrothermal and mechanical stress on the structure of the hair fiber.

EXPERIMENTAL

MATERIALS

Hair. Root sections of untreated (not chemically treated) dark brown European hair (14″ long) was obtained from DeMeo Brothers, New York.

Curling iron. We used Philips Styling Comb Model HP-4480 (Courtesy of Philips) under various conditions. The temperature gradient was measured along the length of the curling iron with a thermocouple. Avoiding the top and the bottom, the middle region of the curling iron used in this study ranged in temperature from 110° to 120°C.

METHODS

Individual dry or wet hair fibers were used in this study. Each fiber had direct contact with the hot metal surface, and the temperature of the curling iron was not affected by the single fiber, as in the case of a hair tress. It is assumed that the single hair fiber will be much more severely affected by the hot metal surface of the curling iron than a hair tress, which tends to absorb and dissipate the heat at the fiber/metal interface, thereby reducing the temperature of the hot metal surface.

Four separate experiments were carried out, each to establish the nature of damage to the hair fiber at the exact location where the hair fiber surface and hot metal are in contact with each other. In each of the experiments, individual fibers were mounted in parallel to assure direct contact of specific lengths and areas of fibers with the hot metal. Four curling protocols adopted in this work are detailed below.

(1) Repeated cyclic curling/cooling of dry hair fibers. Individual fibers were mounted in parallel and subjected up to 50, 100, 150, and 200 times to ten-second intervals of thermal treatment with a curling iron interrupted each time by cooling (20 fibers for each category). This experiment simulates curling of hair fibers approximately every two, three, and seven days during a given year. The fibers were held at the root ends and the middle/tip sections were curled, simulating the way the hair would be curled during normal styling practices. At least ten fibers were examined in the SEM for each curling frequency. After the thermal treatment, the side of the curled hair fiber, which had been in direct contact with the heated metal, was examined.

(2) High-load and long-time curling of dry hair fibers. In the first experiment, individual fibers were taped together at the root and tip sections and then strung over the heated curling iron for ten minutes under various levels of tension. While the control fibers were allowed to hang freely without an applied load, other fibers were individually suspended under an increasing load of 10 g, 20 g, and 30 g (20 fibers for each category). At least ten hair fibers were examined for each category. The increasing loads were

chosen to simulate various tensions that may be applied to hair fibers during styling with a curling iron. After curling for ten minutes and cooling, the hair fiber was gently uncurled and the hair/metal contact zone was examined.

(3) Repeated, cyclical short-term curling/cooling of wet hair. For this experiment, individual hair fibers were immersed in water for 15 minutes, blotted between paper towels, and then subjected to 20, 50, and 100 cycles of curling with the curling iron. Each cycle consisted of ten seconds of thermal treatment with the hot curling iron and 30 seconds of immersion in water, followed by blotting. The fibers were held at the root ends, and the middle/tip sections were curled, simulating the way the hair (attached to the head) would be curled during normal styling practices. After the respective number of cycles of wetting and thermal exposure, the fibers were allowed to cool. Each hair fiber was gently uncurled, and the fiber/metal (f/m) contact zone was examined for the nature of cuticular damage.

(4) Prolonged, high-load curling of wet hair. Individual hair fibers were shaped into large loops by taping together the root and tip ends. The looped fibers were immersed in lukewarm water for 15 minutes, blotted between paper towels, and strung over the heated curling iron for ten minutes under loads of 0 g, 10 g, 20 g, and 30 g. After cooling, each hair fiber was gently uncurled and the cuticula in the heated zone were examined for damage.

FIBER SURFACE EXAMINATION

The f/m contact zone was examined by mounting the inside of the curl "face-up" on double-sided tape and coating with 100 Å of platinum. We used a Hitachi S-4500 digital cold field emission scanning electron microscope (FESEM) for this study.

RESULTS AND DISCUSSION

For ease of comparison, the typical topography of untreated hair fibers without thermal treatment is shown in Figure 1. The untreated controls show good differentiation of the
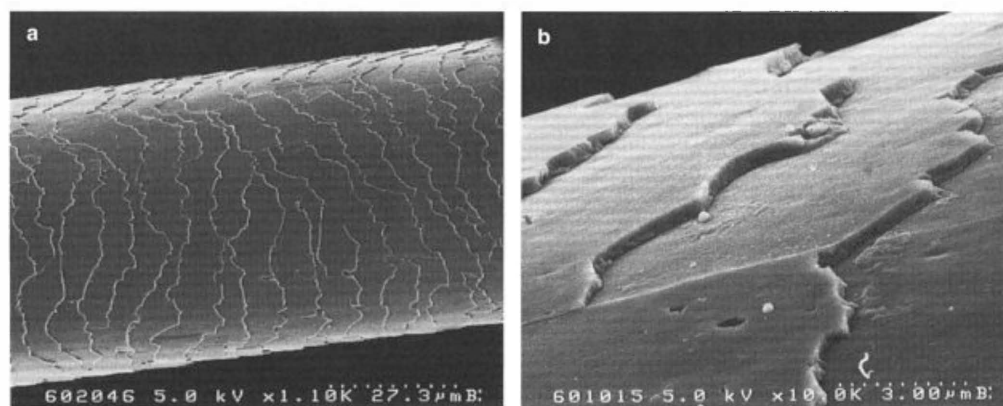

Figure 1. (a, b) Typical topography of untreated hair.

cuticle cell. There may be some debris on the scale faces and jagged scale edges, which is normal and indicative of chipping damage from standard grooming practices.

Twenty hair fibers were prepared for each individual experimental category tested. At least ten of these were examined in the SEM. Damage phenomena or special features observed in more than 75% of the hair specimens of a specific category are described as "typical" or "representative" in this discussion.

EFFECTS OF REPEATED, CYCLICAL CURLING/COOLING OF DRY HAIR

It should be noted that these fibers had been treated individually in a parallel arrangement. Individually treated fibers will be much more affected by the hot metal surface than a hair fiber assembly, which tends to absorb and distribute the heat and moderate temperature within the hair strand.

Hair fibers exposed to 50, 100, 150, and 200 sequential ten-second-long thermal treatments with the curling iron showed an increase in various types of surface damage as a function of progressive thermal exposure. Even though the level of damage increases with the increasing number of cycles of heating/cooling, the same types of damage phenomena are inherent in all exposed fibers. The thermally-induced damage phenomena are shown in Figure 2.

There is increased matting and compacting of the surface cuticle cell. While radial cuticular cracking is still observed, fine axial cracking of the exposed surface cuticle cell has become a frequent damage phenomenon (Figure 2a–d). Increased levels of fusion of the scale edges are displayed (Figure 2e, f). After 200 thermal curling treatments, fusion of the scale edges has become especially severe, as can be seen in Figure 2f. In extreme cases of fusion, pores or holes are frequently seen in the fused scale edges. These pores may possibly be areas where moisture and heat escaped via the CMC and endocuticle during the dry thermal treatment (Figure 2f).

PROLONGED, HIGH-LOAD CURLING OF DRY HAIR

Our SEM observations revealed that prolonged (ten-minute) curling of the "dry" hair fibers under 0 g, 10 g, 20 g, and 30 g of tension resulted in the following types of damage: (a) cuticular compression and moderate disintegration of the cuticula at the fiber/metal interface, (b) fusion at the scale edges, and (c) moderate-to-severe levels of radial cracking of the cuticula. We also showed that the damage became more severe with increasing load during curling.

Topographical examination of the side of the curl of hair fibers that had been in direct contact with the heated metal of the curling iron for ten minutes while freely suspended without tension (0-g load) produced several interesting results that are indicative of moderate levels of cuticular modification: (a) While the inside of some curls showed rather well-maintained surface cuticles and good cell differentiation, other curls displayed matting and compacting of the surface cuticle cell. (b) Most fibers showed various levels of radial cracking of the cuticula during gentle uncurling of the hair fiber in the dry state (Figure 3).

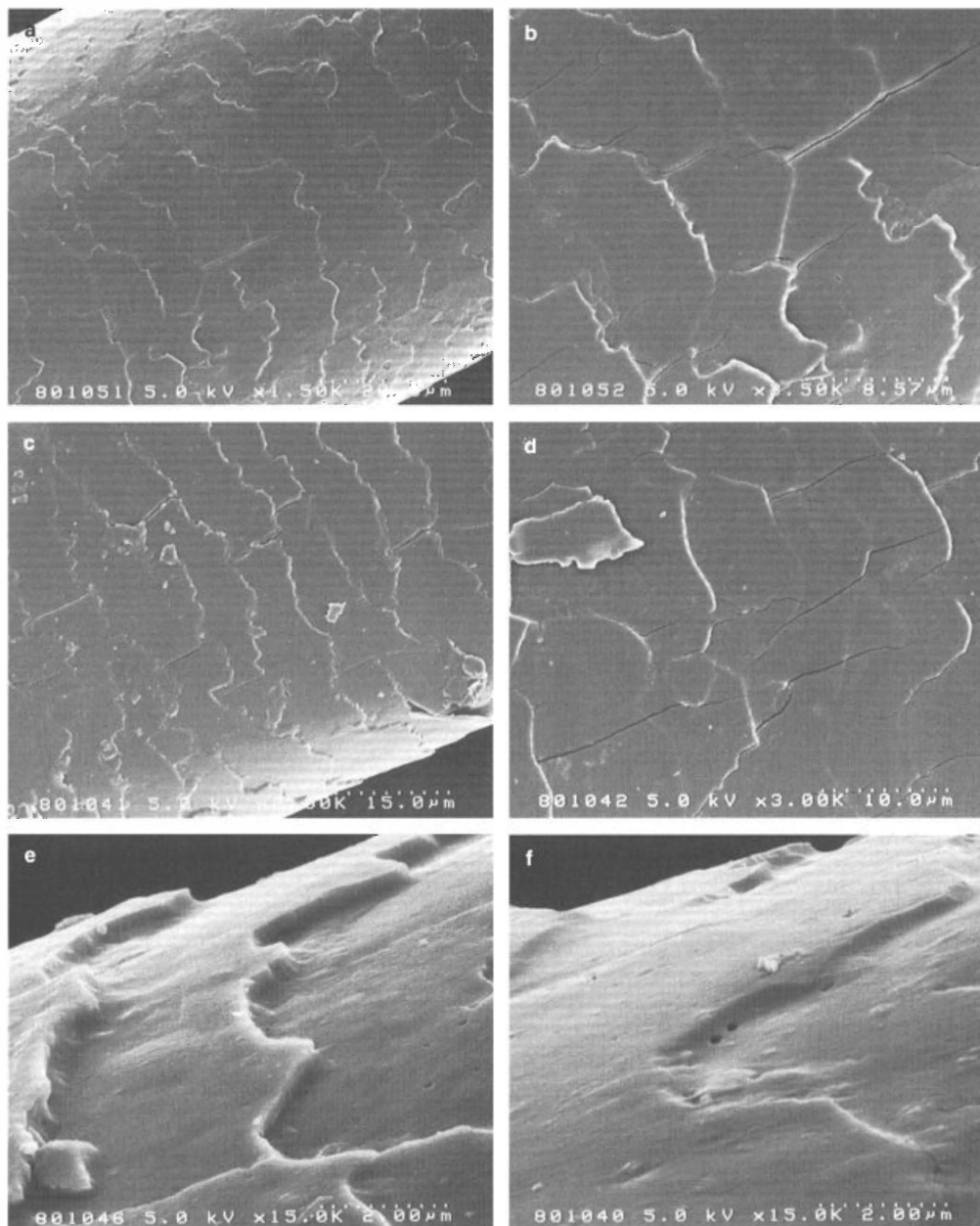The above observations seem to suggest that the contact time of ten minutes for

Figure 2. (a–f) Thermally induced damage phenomena commonly observed in dry hair fibers exposed to 50, 100, 150, and 200 sequential ten-second-long thermal treatments with the curling iron.

individual hair fibers with the heated metal surface of the curling iron is a severe condition. The severe radial cracking of the cuticula during gentle uncurling of the cooled hair fiber in the dry state especially indicates that the long contact time of the fiber with the hot curling iron leads to cuticular damage. Uncurling in the wet or moist state when the fiber is swollen may reduce cuticular cracking.
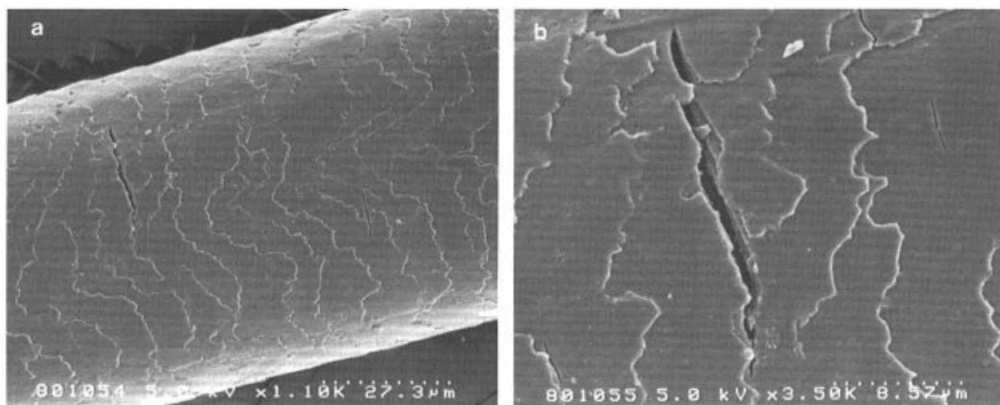
Figure 3. (a,b) Typical topography of dry fibers curled for ten minutes without tension (0-g load).

The f/m contact zone of hair fibers curled for ten minutes under constant loads of 10 g (Figure 4a), 20 g (Figure 4b), and 30 g (Figure 4c, d) produces rather similar topographies, irrespective of the increasing tension during treatment. The inside of many curls displayed matting and compacting, as well as spotwise mutilation of the surface cuticle
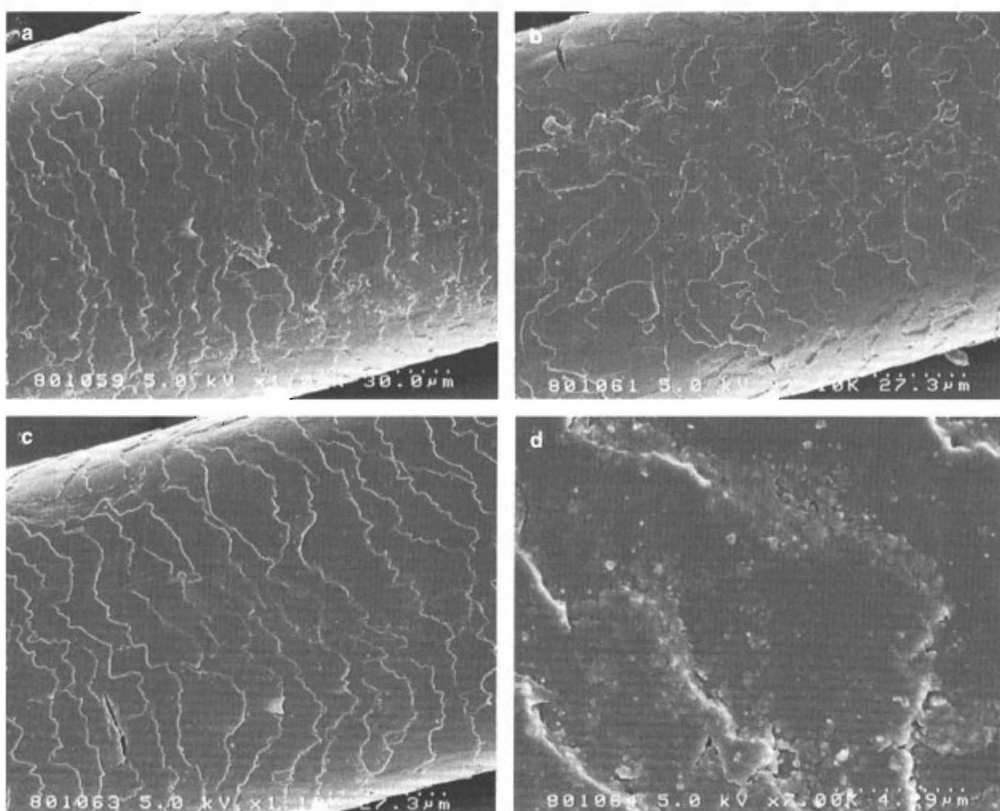

Figure 4. Typical topography of dry fibers curled for ten minutes under constant load of (a) 10 g, (b) 20 g, and (c, d) 30 g.

cells that had been in direct contact with the hot curling iron. This mutilation or disintegration of the surface cuticle cells becomes especially pronounced at the scale edges of hair fibers suspended under the 30-g load (Figure 4d). However, it may be argued that the debris deposited at the scale edges, seen in Figure 4d, may be a combination of extraneous deposits, scale edge fragments, and endocuticular material squeezed from underneath the cuticle cell during the prolonged thermal treatment under considerable pressure. Severe radial cracking (Figure 4b, c) is observed as well.

This study suggests that the contact time of ten minutes between the hair fiber and the heated metal surface of the curling iron is much too severe a condition for thermal treatments. Such conditions lead to modification of the cuticula (embrittlement), resulting in severe radial cracking of the cuticular sheath during gentle uncurling of the hair fiber in the dry state.

There is an important feature of this experiment that should be pointed out. The thermal treatments (up to 200) of this study were inflicted upon the same set of surface cuticle cells, which is an unlikely scenario in standard grooming practices. During everyday grooming, the hair is shampooed/conditioned, dried, combed, and brushed between thermal treatments with the curling iron. This distributes the damage over many fibers, whereas in our experiment, damage is concentrated in the same cuticle cells.

REPEATED, CYCLIC CURLING OF WET HAIR

Wet hair fibers exposed to 20, 50, and 100 cycles of heating (ten seconds), followed by 30 seconds of immersion in water and blotting, consistently showed the same type of damage, irrespective of the numbers of cycles. This "typical" damage is shown in Figure 5a–d, e–f, and g–j, respectively, for 20, 50, and 100 cycles of wetting/curling.

The cuticular damage observed in the curling of wet hair was characteristically different from that observed in the case of dry hair. This new damage phenomenon is seen in the form of bulges or bumps in the scale faces and ripples or "half-domes" at the scale edges. These cuticle distortions are caused by the hygrothermal "fatiguing" of the wet cuticle. Both the bulges/bumps in the scale faces and the ripples at the scale edges are most likely created by the explosive release of steam trapped in the wet cuticula during contact with the heated metal of the curling iron. The ripples at the scale edges appear like scale lifting; however, they are very localized and restricted to the delamination of very small areas at the scale edge. These bulges/bumps and ripples occur all around the hair fiber and are not confined to the fiber/metal interface. They are often accompanied by axial cracking of the cuticula (Figure 5e, f). The fiber/metal interface is primarily characterized by various levels of cuticular compression and disintegration.

Except for a few deeper radial cracks, axial cuticular cracking phenomena observed in these fibers appear to be confined to the individual surface cuticle cell without propagating into the underlying layers of the cuticular sheath, or spreading axially. We would like to emphasize that an increased number of cycles of wetting/curling slightly increases the level of damage, but not the type of damage.

PROLONGED, HIGH-LOAD CURLING OF WET HAIR

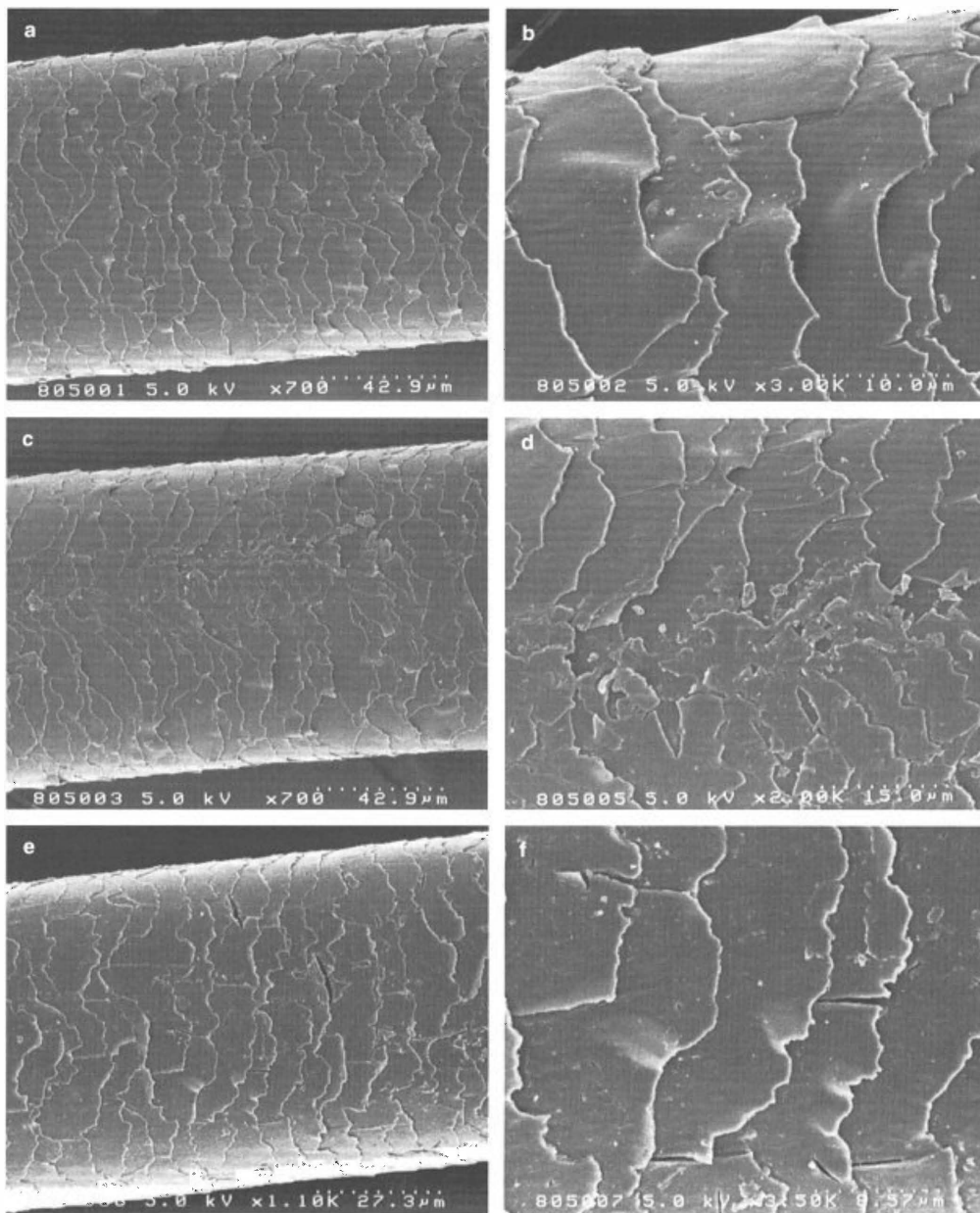Thermal damage from prolonged (ten-minute) curling of the wet fiber under different

Figure 5. Hair fibers exposed to 20 cycles (a–d), 50 cycles (e, f), and 100 cycles (g–j) of wetting (30 seconds) and thermal treatment (10 seconds).

loads produced damage significantly different from that observed in the case of the dry fiber.

In addition to compression, disintegration, radial cuticular cracking, and scale edge fusion of the surface cuticle cell observed earlier at the f/m interface (Figure 6a, b), we also observed fine-line cracking scalloped around the fused scale edges (Figure 6c). This
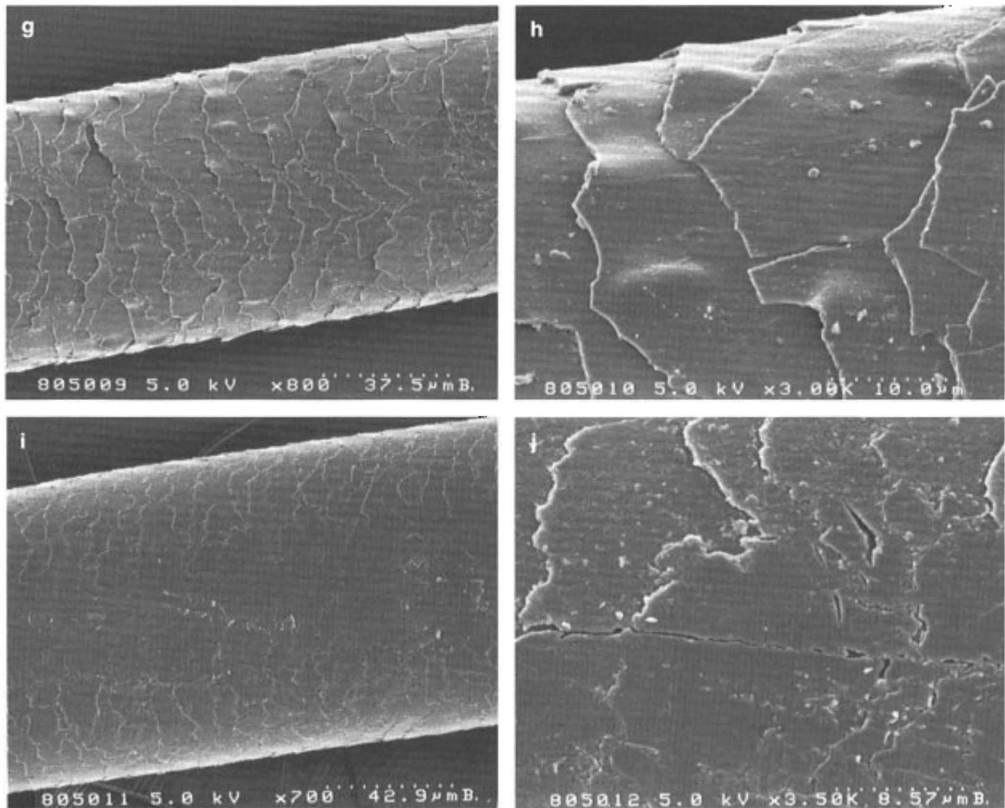
Figure 5. Continued.

was already observed during ten minutes of curling of wet hair under normal load (no added tension).

As expected, cuticular damage increases with increasing load during the curling of wet hair fibers for a longer time. As the tension increases from 10 g to 30 g during thermal exposure, there is increased compression and complete disintegration of the surface cuticle cell at the fiber/metal interface, fine-line cracking as well as severe radial cuticular cracking, and various levels of scale edge fusion. These "typical" damage phenomena are shown in Figure 6d–i. Extremely severe disintegration of the surface cuticle cell into an unrecognizable, fused entity was observed at the f/m interface. This fusion is probably caused by the high temperature flow of the cell proteins plasticized by water at the f/m interface under the influence of the applied load (10–30 g).

These tests have shown that adhering to the recommended guidelines for curling (dry hair, normal tension, short-term curling) results in minimal damage to the cuticula. However, disregarding these guidelines increases the potential for severe surface damage, which culminates in the disintegration of the cuticula, especially when the hair is in the wet state.

EFFECT OF HEAT CURLING ON TENSILE MECHANICAL PROPERTIES OF DRY HAIR

Specimens from the same tress of dark brown European hair were used to examine the
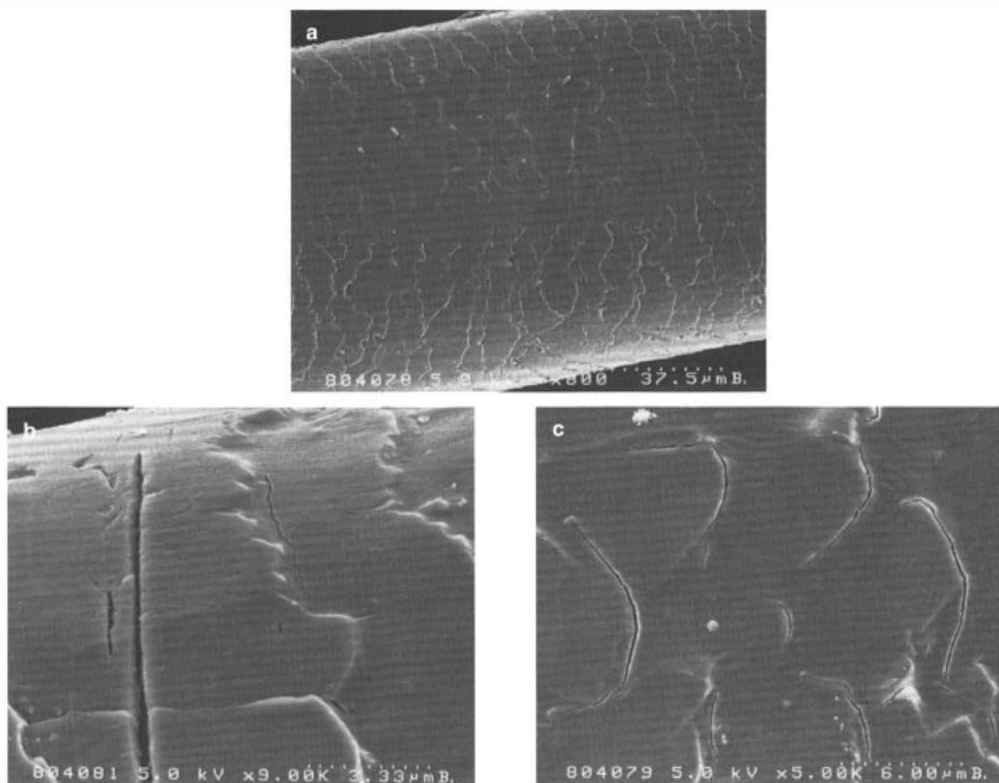
Figure 6. Typical topography of wet hair fibers curled for ten minutes under 0 g tension (a–c) and 10, 20, and 30 **g** constant tension (d–i).

effects of "repeated, cyclical short-term curling/cooling under normal tension" on the mechanical properties of dry hair. The untreated hair fibers served as control. For the curled sample, a small bundle of hair fibers was curled 200 times for ten seconds each time, using the Phillips HP 4480 curling iron, which subjects the hair to a temperature of 110°C. The fibers were extended to break on a Diastron machine (at 65% RH, 21°C). Table I shows the results. In examining the data in Table I, we find a trend toward slight increases in breaking strength, work to 20% with no statistical significance. A slight reduction in extension to break suggests rigidification of the fiber structure. This can be due to the introduction of crosslinks by dehydration reactions at a higher temperature. This is especially true of a large increase in the post-yield modulus. The post-yield modulus, which reflects the interference of crosslinks in the matrix with the $\alpha \rightarrow \beta$ transition of helical structures, shows a significant increase after the hair has been exposed to repeated, cyclical short-term curling/cooling. Therefore, one might hypothesize that this may be due to thermally activated crosslinking in the matrix during curling, and as a result of this crosslinking, the transformation of the polypeptide chains from the α-helical configuration to the β-pleated sheet arrangement may be inhibited.

EFFECT OF HEAT CURLING ON TENSILE FATIGUE BEHAVIOR

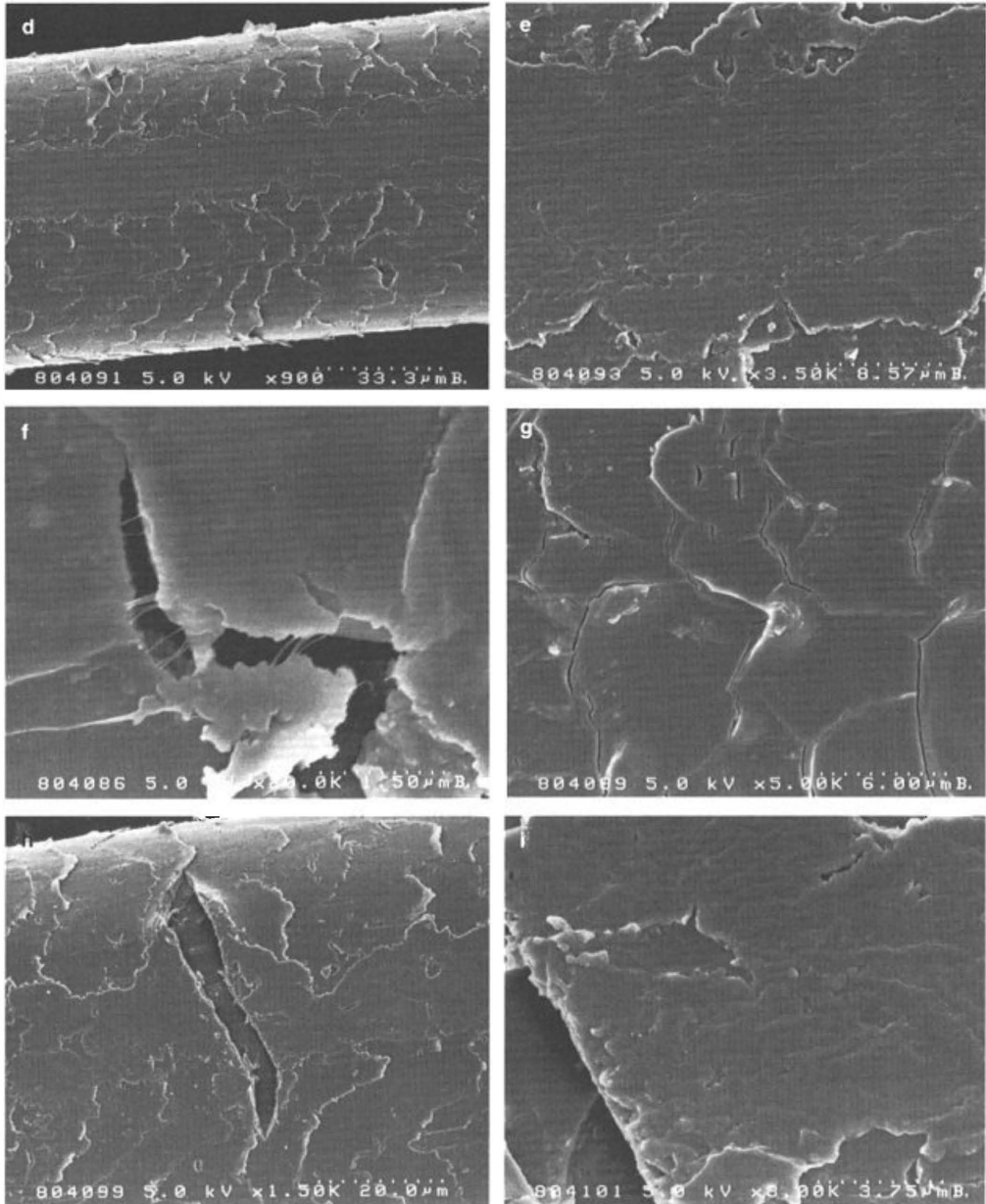An extensive study investigated the effects of thermal treatment under normal tension

Figure 6. Continued.

with a curling iron on the fatigue behavior of untreated and conditioner-treated hair fibers. Untreated hair fibers and fibers treated with 0.5% aqueous solutions of Polyquaternium 10 (PQ10) at 40°C and cetyl trimethylammoniumbromide (CETAB) at 50°C for ten minutes, followed by five one-minute rinses in deionized water, were allowed to condition at 21°C and 65% RH. The hair was held under normal tension (no load applied) for five minutes over a preheated Philips curling iron (~110°C) without overlapping the hair, before being gently unwound and allowed to recondition. Single

Table I
Values for the Mechanical Properties of Thermally Treated Hair

	Initial modulus (GN/m^2)	Extension to break (%)	Load at break (GN/m^2)	Work to 20% (J/m^2)	Total work (J/m^2)	Post-yield modulus (GN/m^2)
Untreated hair	3.09	50.00	0.191	607163	1979180	0.301
Curled hair	3.07	47.34	0.203	621560	1931227	0.386

hair fibers were mounted for the fatigue experiment in TRI's hair fatigue tester, in which the cycling speed was set at the rate of 2.3 Hz with a load of 40 g.

The fatigue failure data, treated according to the Weibull statistics (1), yield a hazard parameter known as characteristic life. Mathematically it represents the number of cycles necessary to break ~63% of the test specimens. Damage results in a reduction of characteristic life.

Figure 7 compares the characteristic life (θ) of dry untreated and conditioner-treated hair fibers before and after curling for five minutes with a curling iron. Surprisingly, an effect of thermal treatment could be established. Hair fibers clearly showed a higher characteristic life (θ) after thermal treatment with the curling iron, indicating improved fatigue resistance. It is possible that this is due to some crosslinking in the interior of the hair fiber as a result of dehydration at high temperature. The presence of the conditioning compounds appears to enhance this thermally induced crosslinking in the form of salt linkages and hydrophobic bonding, leading to a significant increase in fatigue resistance (characteristic life [θ]).

The improvements in fatigue properties of an ionomeric polymer by the introduction of
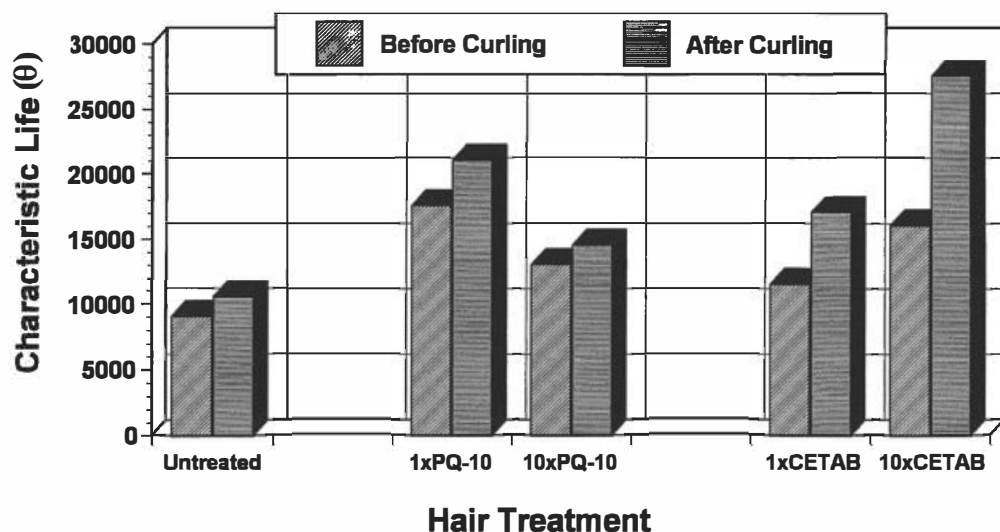

Figure 7. Comparing characteristic life (θ) of dry untreated and conditioner-treated hair fibers before and after prolonged curling for five minutes under normal tension with a curling iron.

crosslinks in the form of salt linkages is quite well known (2). Even though we have not examined the heat-curled/conditioner-treated hair in the SEM, relevant work of others (3) suggests that the cuticle-cracking tendency is significantly reduced by conditioning agents. Recently published work (4) on the effect of conditioning compounds on the scale-lifting behavior of the cuticle supports this concept further.

MECHANISMS OF HYGROTHERMAL DAMAGE

The stresses acting on a hair fiber draped on a heated mandrel like the curling iron are shown in Figure 8, where the hair fiber is shown schematically at high magnification. Under dry conditions, the outside of the fiber is under tension and the inside is under compression. Dehydration due to heat makes the f/m contact area rigid and brittle so that an attempt at straightening it can generate radial cracks in the fiber. The cuticle cells are curved. The force of compression tries to flatten them, and brittleness leads to the formation of axial cracks. This is shown in Figure 8, displaying a cross section through the center line (black dashed line).

Wetting of the fiber plasticizes the keratin, and the fiber deforms much more easily and to a greater extent than in the dry condition. Endocuticular material is softened to an
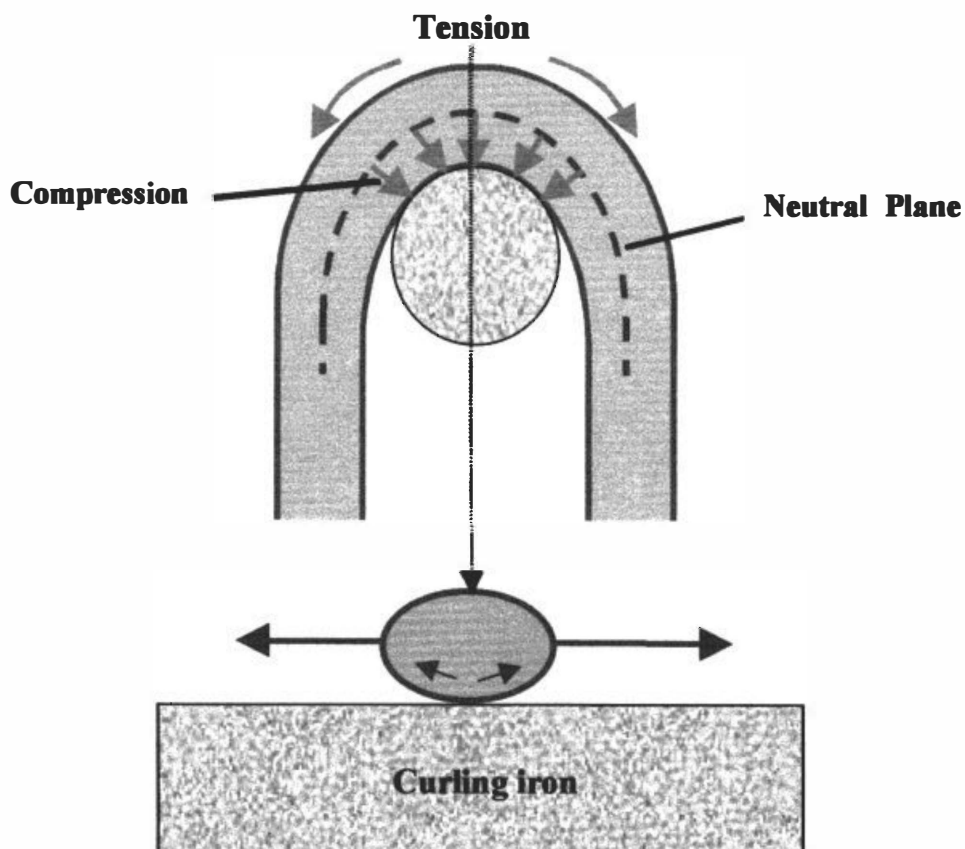

Figure 8. Stresses acting on a hair fiber draped on a heated mandrel like the curling iron.

extent where it can be squeezed out at the cuticle edge, masking the scale structure along the squeeze line. Entire cuticle cells can be displaced and cracked into pieces (Figure 6). It is very likely that this process damages the cuticle sheath parallel to the major axis of the fiber because of the preferred orientation of the fiber with the major axis parallel to the curling iron.

CONCLUSIONS

DRY HAIR

Thermal treatments by use of curling irons according to the manufacturer's specifications (dry hair, short times, and normal tension) result in minimal damage to the "dry" hair fiber. Prolonged (ten-minute) contact times combined with increased tension (10–30 g) lead to compression, disintegration, radial cracking, and scale edge fusion of the surface cuticle cell at the f/m interface.

WET HAIR

Repeated short-term curling and wetting results in less damage to the cuticula, comparable to that observed in the "dry" fiber. Long-term effects lead to distortion of the cuticle cell due to trapped moisture expanding in the form of steam in the fiber, forming bulges or bumps in the scale faces and ripples at the scale edges. This type of damage occurs all around the fiber and is not restricted to the f/m contact zone. With wet hair, prolonged (ten-minute) contact times under increased tension lead to damage similar to that of the cuticula of dry hair, although under these wet conditions the damage is considerably more severe than in the case of the "dry" hair fiber. The high temperature flow of water-plasticized cell proteins creates mutilated and distorted cuticle cells when recommendations by the manufacturer are ignored and abusive practices are carried out.

MECHANICAL PROPERTIES

Thermal treatments by use of a curling iron according to the manufacturer's specifications (dry hair, small skein, short-term curling, and normal tension) showed, quite surprisingly, that repeated, cyclical short-term curling/cooling leads to changes in specific mechanical properties of the hair. The data showed that after repeated short-term curling/cooling, the post-yield modulus of the hair fibers had increased, possibly due to thermally induced crosslinking of components of the cortical domains.

FATIGUE RESISTANCE

Exposure to heat increased characteristic life (θ), indicated by improved fatigue resistance. This behavior is likely to be the result of crosslinking in the interior of the hair fiber as well as in the cuticula due to thermally induced dehydration. The presence of the conditioning compounds enhances this heat-induced crosslinking in the form of salt

linkages and hydrophobic bonding, leading to significant increases in fatigue resistance (increases in characteristic life).

The outcome of this study suggests that curling of hair under moderate conditions specified by the manufacturer of the curling iron is not damaging to hair. On the contrary, it may be beneficial when used in combination with certain conditioning polymers.

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