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Progressive hair straightening using an automated flat iron: Function of silicones

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Synopsis

An automated hair iron was built with which the hair temperature, contact force of the iron against the hair tress, and gliding speed were controlled. The changes in keratin were characterized by several techniques including differential scanning calorimetry, birefringence measurements, and wet tensile tests. Undamaged curly hair was ironed for several iron cycles at temperatures ranging from 120°C to 175°C and washed between each iron cycle. Irreversible straightening of curly hair was observed and depended on the temperature and the number of cycles. The birefringence data suggested that the straightening was related to a gradual decrease of the microfilament organization. Silicone treatment did not significantly affect the course of microfilament denaturation, but it improved the quality of straightening. It enhanced the fiber alignment under the gliding action of the iron.

Progressive thermal straightening may be a promising method to achieve permanent smoothing of curly hair without chemical treatment. Ironing at the onset temperature (~154°C), before substantial disulfide bond scission occurred, seemed to be a good compromise between process speed, straightening performance, and hair integrity (i.e., reduced loss of cross-linking).

INTRODUCTION

Thermal treatments for hair styling are becoming increasingly popular with consumers both at home and in hair salons. High-temperature irons with maximum temperatures of 250°C seem to provide better straightening permanency. However, since these ironing treatments are often combined with chemical treatments, it remains unclear how the chemical and heat contributions to straightening decouple. Controlled studies to address the process of thermal straightening are still needed.

The mechanisms involved in setting treatments of keratin fibers have been discussed extensively in the literature, especially for wool. With chemical treatments (alkaline or reducing agents), it is usually accepted that straightening involves breaking chemical bonds, followed by reforming new bonds while the keratin fibers are held straight (1,2). The permanent setting of wool can occur in the absence of chemical treatments when wool is held extended in boiling water (3). Feughelman showed by x-ray and birefringence measurement that it was

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related to an irreversible physical modification of the microfilament keratin conformation induced by the coupled action of heat, strain, and water (4).

More recently, differential scanning calorimetry (DSC) analyses of human hair have shown that thermal denaturation of α-keratin occurred between 140°C and 200°C depending on the water content of the samples (5–8). The denaturation is characterized by an endotherm corresponding to a transition from a crystalline to a more amorphous state. Although hair can reach those high temperatures during ironing, there are significant differences between the heat transfer processes in an iron versus a hermetic DSC pan. The hair iron involves multiple heating and cooling cycles, and the heating rate in the iron is usually much faster than in a DSC experiment. In addition, in an iron, the moisture content around the heated portion of hair decreases with time and hair fibers undergo deformation and stress.

Here the objective was to develop a controlled iron experiment to study the effect of multiple iron cycles on the straightening permanency of curly hair. Since holding the fibers straight is key to straightening, another aim of this study was to elucidate the contribution of silicones to the process. Silicones are often used in hair thermal treatment because of their thermal stability (9). Because of their low surface tension, silicones form films reducing iron gliding force; they condition the hair and produce increased fiber–fiber interaction with an anti-frizz effect (10).

An automated iron was built with which the hair temperature, contact force of the iron against the hair tress, and gliding speed were controlled. The stretching force was recorded by a load cell. Fiber alignment could be monitored by a camera mounted on a stereomicroscope. The changes in keratin were characterized by several techniques including DSC, birefringence measurements, and wet tensile tests. The initial moisture of the hair tress was controlled. All tests were performed without the use of chemical relaxers or cross-linking agents.

EXPERIMENTAL

MATERIALS

We purchased 2 g undamaged naturally curly hair tresses and ethnic Afro hair swatches from International Hair Importers & Products (Glendale, NY).

The silicone emulsion used in the study was made by Momentive (Columbus, OH). The silicone was an alkyl-modified amodimethicone from Momentive, trade named Silsoft* AX® conditioning agent with International Nomenclature of Cosmetic Ingredients (INCI) name Bis-cetearyl amodimethicone. The silicone was emulsified with nonionic emulsifiers to form a stable emulsion with a particle size of around 350 nm.

METHODS

Automated iron. In a manual iron experiment, the heat transfer process controlling the hair alteration is quite variable since it depends on manual factors, including gliding speed of the heating elements, contact pressure, and the amount of hair between heating plates. An automated iron was built to control those variables (Figure 1).

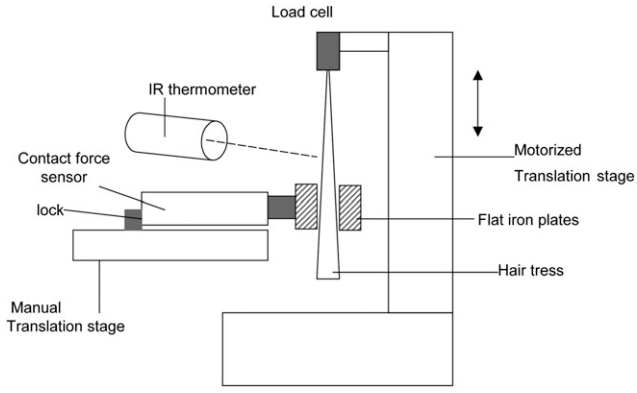

Figure 1. Automated hair iron apparatus.

A 1-inch flat iron was mounted on a fixed holder. Two iron plates were brought into contact by a rigid holder connected to a force sensor (Imada, Northbrook, IL), measuring the contact force on the iron handle at a distance of 2 cm from the heating plates. The hair tress was pulled at a constant speed of 15 mm/s by the vertical motorized stage of a Texture Analyzer (Stable Micro Systems Inc, Godalming, UK). The Texture Analyzer load cell measured the stretching force exerted on the hair tress during ironing. The study was conducted by keeping the contact force constant to achieve a tangential force (stretching force) of approximately 100 g for a 2 g tress. The hair temperature was measured using an infrared thermometer from Raytek (Santa Cruz, CA). The temperature of the iron was varied to achieve hair temperatures in a range of 122°C to 175°C. The hair temperature was measured at a distance of 1 cm above the flat iron heating plates.

Multiple iron cycles protocol. Curly hair tresses were washed and blow-dried. The hair was then dipped in 0.2% silicone dispersion for 1 min. When the hair was removed from the dispersion, excess liquid was squeezed out and the hair tress was blow-dried. Water served as the control treatment. Before ironing, the treated hair tresses were conditioned for 30 min in a 90% relative humidity (RH) chamber. The tresses were then ironed for one cycle of five passes and stored overnight under ambient conditions. The next day, the tresses were washed and blow-dried before being conditioned at 90% RH for 30 min in preparation for the next ironing cycle. This cycle was repeated 2 to 10 times, with the number of cycles being dependent on the temperature of treatment.

Assessment of straightening permanency. Following each ironing cycle, the tresses were stored overnight in the laboratory at room temperature. The straightening permanency was assessed the next day when the hair tresses were washed once with 10% Sodium Laureth Sulfate (SLES) and blow-dried. They were then placed in a humidity chamber for 30 min at 90% RH with forced air flow. Tresses were photographed immediately after being taken out of the high-humidity chamber. Tresses were compared to the nonironed curly tress.

DSC measurement. Hair sample DSC tests were performed with Q1000 TA Differential Calorimeter (New Castle, DE), using high-volume pans. These hermetic pans maintained constant water content throughout the temperature scan (80°C–250°C). Hair samples were cut into 1–2 mm pieces. Then 10 mg sample was placed in a pan and equilibrated for 2 days in a constant humidity chamber at room temperature. RHs ranging from 20% to 97% were obtained with saturated salt solutions (11). A completely dry hair sample (~0% RH) was

obtained by leaving the pan with the hair sample in a dessicator containing Drierite for several days. Before sealing the DSC pan, a 200 cp polydimethylsiloxane oil droplet of 10 µl was added to the pan to act as a thermal medium according to method described by Cao (7,8). Unless specified otherwise, the sample was equilibrated at 58% RH at room temperature. For each hair tress, DSC data was an average of three heat flow curve analyses (three pans). DSC analysis was also performed with ethnic Afro hair, which served as a model experiment; the endothermic peaks of the undamaged peak were sharper and easier to quantify. A typical heat flow curve of undamaged ethnic Afro hair equilibrated at 58% RH is shown in Figure 2. The peak temperature and the area of the peak refer to the keratin denaturation temperature (T_d) and the denaturation endothermic enthalpy (ΔH), respectively.

Wet elasticity measurement. Single-fiber tensile tests were performed using a 100-fiber automated tensile tester (Dia-Stron, Andover, UK). A total of 50 fibers were analyzed per treatment. Tensile tests were performed with the fibers immersed in distilled water in cassette holder. Maximum strain was 10%. The diameter of each fiber was measured using a laser-scanning instrument from Dia-Stron. The fibers were immersed in deionized water for at least 30 min before measurement. Slope of the linear Hookean region was measured to calculate Young's modulus of elasticity.

Birefringence measurement. Because of the anisotropy of its keratin arrangement, hair fibers display clear distinct interference colors in cross-polarized light, which can be analyzed to measure birefringence. Birefringence is a measure of structural organization of the keratin fiber. The single fibers used for tensile test were reused since they were strained in the reversible linear region. They were mounted on microscope slides with Permount medium (Fisher, Pittsburgh, PA) and a cover glass. They were examined on an Eclipse microscope

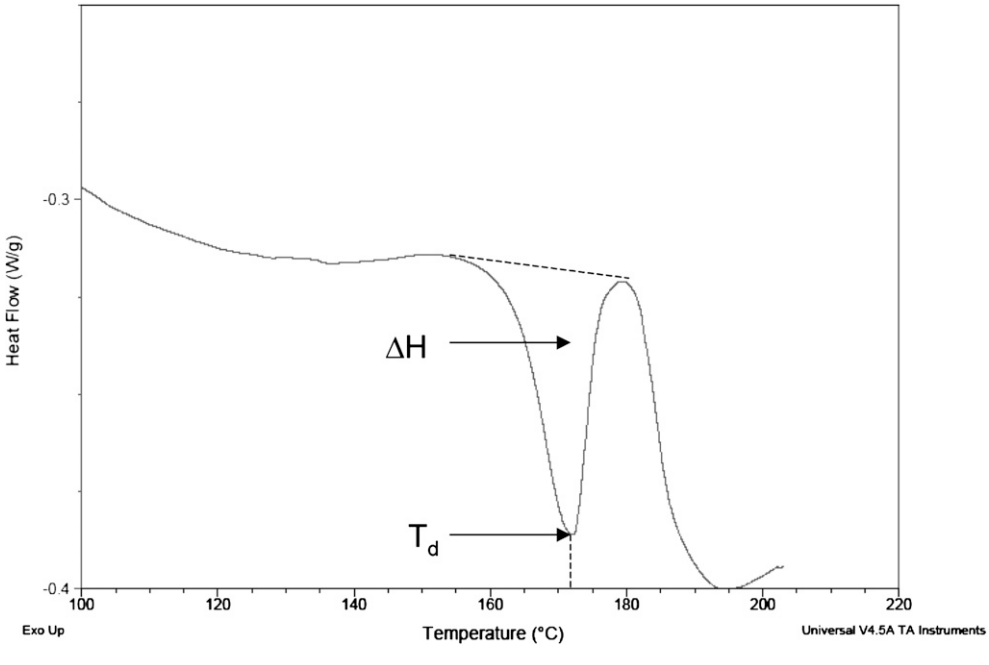

Figure 2. Typical DSC curve for an untreated ethnic afro hair sample equilibrated at 58% RH, prior to sealing of the DSC pan.

(Nikon, Melville, NY) using cross-polarized illumination and the method described by McCrone *et al.* (12). The fiber was oriented 45° to the vibration direction of the crossed polars to obtain the brightest interference color. The main interference colors were observed close to the center axis, which were then compared to a Michel–Levy chart to obtain the value of retardation (optical path difference). The retardation and fiber thickness were related to the birefringence according to the following equation:

$$\lambda = 1000 \times t \times (\Delta n)$$

where λ is the retardation in nm, t is the fiber diameter in micron, and Δn is the birefringence. About 40 to 50 fibers were examined for each treatment. Statistical analysis was performed using Minitab software.

RESULTS

EFFECT OF MOISTURE ON THERMAL α-KERATIN DENATURATION

The denaturation temperature and the enthalpy as a function of initial humidity during sample equilibration are shown in Table I for undamaged ethnic Afro hair. The denaturation temperature decreased as moisture increased, which is consistent with the data of Cao and Leroy for Caucasian hair (8). In contrast, the value of denaturation enthalpy did not vary significantly. When the matrix keratin was plasticized by water, the denaturation transition of the crystalline α-keratin microfilaments occurred at lower temperatures. In the next sections, the initial moisture of the hair tresses was controlled by placing them in a 90% RH chamber before ironing.

STRETCHING FORCE

Curly hair was treated as described in the section Multiple Iron Cycles Protocol. Stretching force was recorded as a function of time at each pass. Figures 3A and B represent the stretching force through one ironing cycle for samples treated with water and silicone, respectively. In the water-treated sample, the force was higher in the first pass ($F = 130 \pm 6$ g) and decreased as the hair flattened. In the sample treated with silicone, the stretching

Table I
Denaturation Temperature, T_d and Enthalpy, ΔH as Function of Initial RH of the Hair Sample (Equilibration) for Ethnic Afro Hair Sample Equilibrated at Different RHs

Initial RH (%)	T_d (°C)	ΔH (J/g)
0	213.2 ± 2.2	5.0 ± 1.6
21	186.0 ± 0.1	5.3 ± 0.3
33	181.7 ± 0.3	6.7 ± 0.6
43	180.0 ± 0.3	5.3 ± 0.4
58	177.1 ± 0.4	5.0 ± 0.5
75	167.6 ± 0.4	5.1 ± 0.3
86	166.6 ± 1.8	5.5 ± 0.4
97	163.5 ± 0.4	4.6 ± 0.8

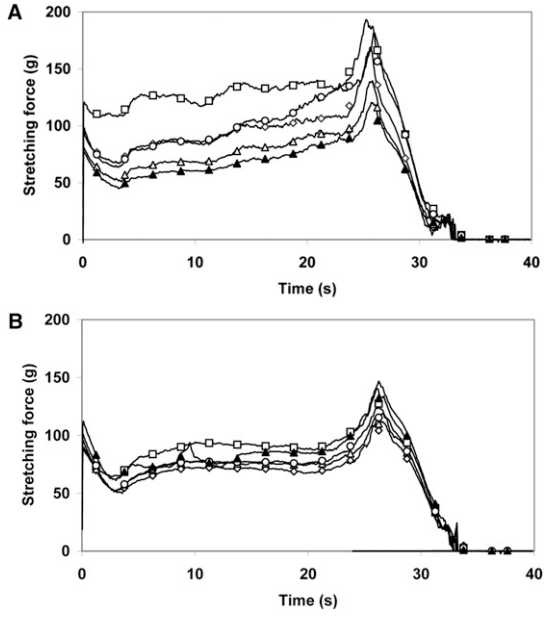

Figure 3. A) Stretching force during ironing as a function of time and pass number for the control tress. Symbols □, ◇, △, ○, ▲ correspond to pass #1, 2, 3, 4, 5 respectively. {Note: Test data. Actual results may vary.}
B) Stretching force during ironing as a function of time and pass number for the silicone treated tress. Symbols □, ◇, △, ○, ▲ correspond to pass #1, 2, 3, 4, 5 respectively. {Note: Test data. Actual results may vary.}

force was lower in the first pass ($F = 90 \pm 2$ g) and stayed low. Figure 4 shows the images taken with a stereomicroscope at 0.67× magnification just after the first and fifth pass of the control tress. In the water-treated tress, the fibers were misaligned and showed a disorganized packing (Figures 4A and B). In contrast, with the silicone-treated tress (Figures 4C and D), regular fiber alignment was observed.

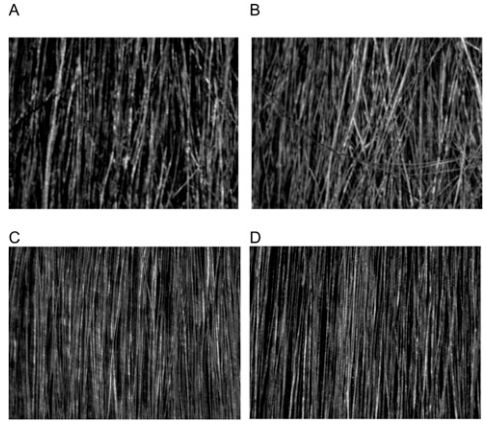

Figure 4. Fiber alignment pictures after ironing pass #1 and after ironing pass #5 for the control tresses (A, B) and for the silicone treated tresses (C, D) respectively. {Note: Test data. Actual results may vary.}

STRAIGHTENING PERMANENCY

In Figure 5, the hair tress photographs are shown at different cycles and different temperatures. In each square picture, the control tress with no silicone was on the left and the tress treated with silicone was on the right. A progressive straightening was observed as temperature and number of cycles increased. The ironing experiment was stopped when permanent straightening was achieved. On the left upper corner of the picture display, hair tresses treated for one cycle and two cycles at 122°C and 144°C, respectively, were curly and puffy. In contrast, at 175°C, a clear decrease in the tress volume and increased straightening was observed at one cycle and two cycles. A similar level of straightening was obtained at 154°C and 144°C with 5 cycles and 10 cycles, respectively. In contrast, after 10 cycles at 122°C, the hair was still curly. In general, at all temperatures and cycles, the silicone treatment provided a better fiber alignment and reduction of tress volume.

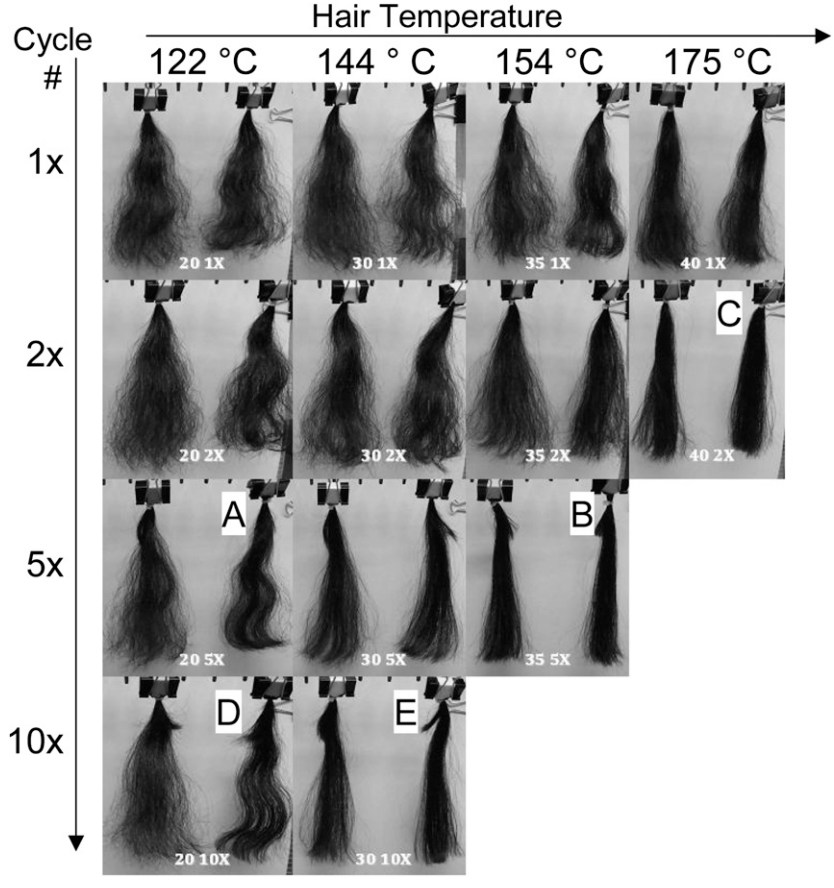

Figure 5. Tress visual after the iron cycles, wash, and storage for 30 min in 90% RH chamber as a function of the hair temperature. On the left, cycle numbers. In each square picture, the control tress is on the left, and the silicone treated tress is on the right. {Note: Test data. Actual results may vary.}

DSC DATA

Multiple iron cycles. DSC analyses were performed for the samples shown in Figure 5. Examples of DSC flow curves corresponding to tresses A, B, and C are shown in Figure 6. Curly sample A treated at 122°C displayed a sharp peak similar to untreated hair, whereas the straight samples (B and C) had much shallower endotherm peaks, indicating a decrease of α-keratin microfilament crystallinity.

The complete set of DSC data is shown in Table II. In general, enthalpy values decreased as the number of cycles and iron cycle temperature increased. T_d shift was quite small

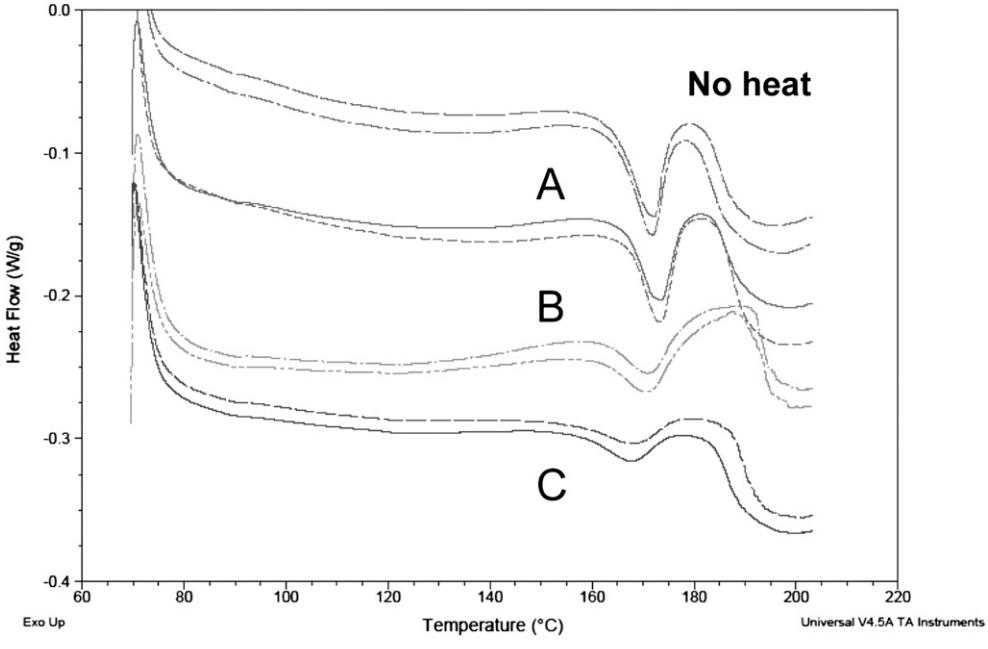

Figure 6. Examples of DSC curves of samples A, B, and C shown in Figure 5 and an untreated hair sample. For each sample two DSC curves (2 pans) are displayed. {Note: Test data. Actual results may vary.}

Table II
Denaturation Temperature, T_d, and Enthalpy, ΔH, for Hair Samples Ironed Multiple Times at Different Temperatures (Hair Temperature was Probed at 1 cm above the Iron)

	Cycle temperature							
	122°C		144°C		154°C		175°C	
Number of cycles	T_d (°C)	ΔH (J/g)	T_d (°C)	ΔH (J/g)	T_d (°C)	ΔH (J/g)	T_d (°C)	ΔH (J/g)
2× water	172.7	5.3	173.7	5.7	173.2	4.7	169.9	4.8
2× silicone	173.5	5.0	173.7	5.7	173.6	5.4	168.0	2.0
5× water	173.7	3.7	172.7	3.4	173.1	2.8		
5× silicone	174.2	4.3	173.0	3.6	172.8	2.6		
10× water	174.4	2.8	172.9	3.6				
10× silicone	174.0	4.3	172.0	3.6				

($\Delta T \sim 1°C$) for hair samples treated at lower temperatures (144°C–154°C). But significant T_d shift ($\Delta T \sim 6°C$) was observed in hair samples ironed at 175°C.

Multiple heat/cool cycles in high-volume DSC pan. A simulation of multiple heating and cooling cycles was conducted in DSC pans. The hair sample was first equilibrated at 58% RH at room temperature. The pan was then sealed and subjected to a number of heat/cool cycles at low temperature before the final temperature scan. Cycle temperature was varied from 120°C to 170°C for either three or six cycles. The experiment was performed with ethnic Afro hair to identify more clearly the changes in the endotherm peak.

In Figure 7, the denaturation peak temperature is plotted as function of cycle temperature for three and six cycles. A nonlinear behavior was observed, indicating an onset of transition at around 140°C. In Figure 8, a similar plot is shown with the DSC data obtained with the ironed tress samples. Surprisingly, although heating and cooling rate were very different in both experiments, a similar nonlinear behavior was observed with T_d, when cycle temperature reached the 150°C temperature range.

WET ELASTICITY

Average wet elasticity of hair samples B, C, D, and E ($N \sim 50$ fibers per sample) is shown in Table III. A significant decrease of wet elasticity was observed as iron cycle temperature increased. On the basis of *t*-test values, the silicone treatment did not affect significantly the heat-induced elasticity change. A plot of wet elasticity versus T_d showed a quite good correlation ($R^2 = 0.76$) (Figure 9).

BIREFRINGENCE

The hair fibers displayed a range of birefringence values. For a 90 microns fiber, blue-green colors were predominant in the samples treated at low temperature. Yellow-brown colors were predominant in the hair samples treated at higher temperatures. Birefringence

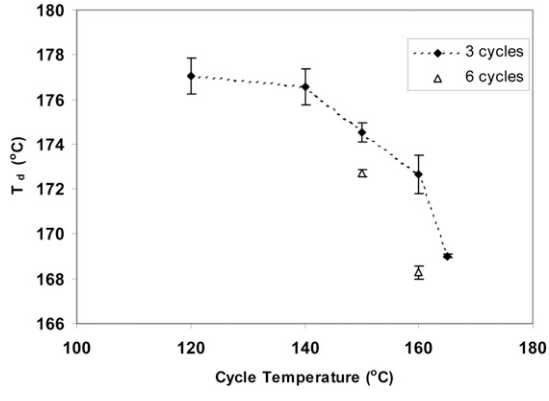

Figure 7. Multiple heat/cool cycles in DSC pans for an untreated ethnic afro hair sample: Denaturation temperature (T_d) as a function of temperature for 3 and 6 cycles. {Note: Test data. Actual results may vary.}

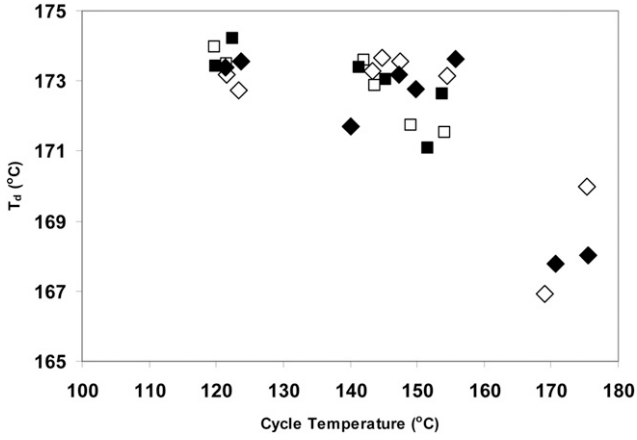

Figure 8. Multiple heat/cool cycles in hair iron: Denaturation temperature (T_d) as a function of cycle temperature (each symbol is an average value of 3 pans). Empty symbols are for the control tresses, bold symbols are for the treated tresses. Diamonds correspond tot he tresses treated for 2 iron cycles. Squares correspond to the tresses treated for 5 iron cycles. {Note: Test data. Actual results may vary.}

value histograms (N = 50) for hair samples treated with silicone (B, C, D, and E) are shown in Figure 10 and average values are shown in Table III. The curly samples (E) showed a broader distribution with a high birefringence average ($\mu \sim 0.008$), whereas the straight samples had a skewed distribution with significantly lower birefringence values (μ = 0.005–0.006).

The broad spectrum of birefringence values within individual fibers suggested that fibers within the same hair tress might respond differently to heat, depending on their initial degree of keratin organization. A statistical t-test showed that the silicone-treated samples were not significantly different from the control samples. But there were significant differences between the samples (B, C, D, and E) treated at different temperatures.

Table III
Summary Table

			Tensile test	DSC		Polarized Microscopy
Hair temperature	Number of cycles		Wet Young modulus ($/10^9$ Pa)	T_d (°C)	ΔH (J/g)	Birefringence ($\times 10^3$)
		Control	1.80 ± 0.29	174.4 ± 0.28	2.81 ± 0.51	7.7± 2.4
122°C	10	Silicone	1.73 ± 0.35	174.0 ± 0.15	4.26 ± 0.51	7.9 ± 2.2
		Control	1.42 ± 0.25	172.9 ± 0.30	3.58 ± 0.16	6.4 ± 1.8
144°C	10	Silicone	1.40 ± 0.2	172.0 ± 0.31	3.65 ± 0.15	7.1 ± 1.9
		Control	1.31 ± 0.23	173.1 ± 0.60	2.84 ± 0.50	5.9 ± 2.0
154°C	5	Silicone	1.21 ± 0.33	172.8 ± 0.83	2.59 ± 0.34	5.4 ± 2.1
		Control	1.11 ± 0.31	169.9 ± 0.38	4.81 ± 0.11	5.8 ± 1.7
175°C	2	Silicone	0.95 ± 0.37	168.0 ± 0.71	2.02 ± 0.11	5.4 ± 1.8

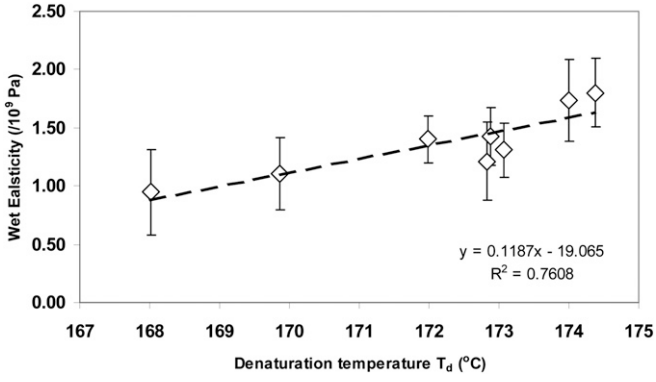

Figure 9. Wet elasticity versus the denaturation temperature (T_d) for the curly hair samples ironed multiple times shown in Table III. {Note: Test data. Actual results may vary.}

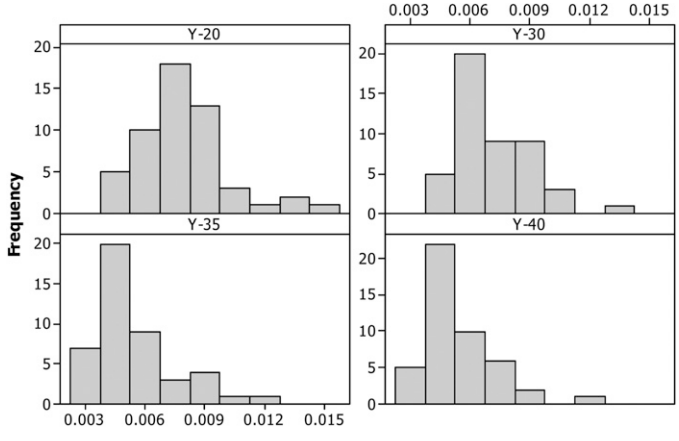

Figure 10. Histograms of birefringence values of fibers obtained from silicone treated tresses ironed multiple times. Y-20 = 10 iron cycles at 122°C, Y-30 = 10 iron cycles at 144°C, Y-35 = 5 iron cycles at 154°C, and Y-40 = 2 iron cycles at 175°C. {Note: Test data. Actual results may vary.}

IRONING AT THE ONSET TEMPERATURE (T = 154°C)

An experiment conducted at the onset temperature (154°C) for three iron cycles is shown in Figure 11 and Table IV. DSC data showed no significant change of T_d but a 40% decrease in enthalpy in both samples. However, straightening was much better with silicone because of a better fiber alignment during setting.

DISCUSSION AND CONCLUSION

The progressive, irreversible straightening of curly hair was a function of temperature, occurring faster at higher temperatures than at lower temperatures. The birefringence data suggested that the straightening was related to a gradual decrease of the microfilament organization (i.e., decrease of birefringence). This was induced by heating the fibers above a threshold temperature while holding the fibers straight. The silicone did not

Figure 11. Tress photograph after 3 iron cycles at 154°C, 1 wash, and storage for 30 min at 90% RH. {Note: Test data. Actual results may vary.}

significantly affect the course of microfilament denaturation, but it improved the quality of straightening. By forming a film on the hair, the silicone treatment enhanced the slip between fibers, allowing a regular packing and alignment under the gliding action of the iron. This kept the fibers in a more aligned conformation.

As shown by DSC analysis, the keratin denaturation temperature depended on moisture. In particular, denaturation temperature increased significantly at reduced moisture levels (<10%). During one ironing cycle, hair moisture level decreased to a point where the denaturation temperature was higher than the hair temperature. At each ironing cycle, moisture was restored to further modify the keratin organization toward a straight conformation. The hair ironing process presented some analogy with the permanent set of wool using boiling water or steam (4). In the case of wool, where water and strain were necessary to cause the keratin microfilament transition, the extension strain needed to be maintained for a sufficient time to produce irreversible setting. In the case of hair ironing, a similar process occurred by iteration, restoring water at each cycle.

The shift of denaturation temperature, T_d, has been interpreted in different ways in the literature. It could be due to a plasticizing effect or loss of cross-linking density. For example, it was shown that bleaching, by causing a loss of cross-linking density in the matrix (higher swelling, lower wet elasticity), leads to a decrease of the keratin denaturation temperature (5,13). Here, since the decrease of the denaturation temperature, T_d, was correlated with a decrease of the wet elasticity, a loss of cross-linking density might have occurred in the matrix. The nonlinear decrease of T_d as ironing temperature increased, shown by both the iron and the multiple DSC heat and cool cycle experiments, suggested that the rate of disulfide bond scissions sharply increased as heat cycle

Table IV
DSC Analysis for Curly Hair Samples Ironed for Three Cycles at 154°C

	T_d (°C)	ΔH (J/g)
Initial	172 ± 1	5.7 ± 0.6
3× control	171.7 ± 0.2	3.3 ± 0.2
3× silicone	172.2 ± 0.5	3.7 ± 0.4

temperature exceeded 150°C. Disulfide bond scissions facilitated the keratin denaturation, as suggested by Istrade (14). Therefore, the straightening occurred more rapidly at 175°C than at lower temperatures. Interestingly, at low temperature, provided the fibers were held straight multiple times, change in the microfilament organization (decrease of birefringence) and the fiber reshaping occurred despite the low number of disulfide scissions (small T_d shift). An amino acid analysis would be useful to determine whether the scission of disulfide bonds was indeed different between the samples produced at low and high temperatures.

Progressive thermal straightening may be a promising method to achieve permanent smoothing of curly hair without chemical treatment. Ironing at the onset temperature (~154°C), before substantial disulfide bond scission occurred, seemed to be a good compromise between process speed, straightening performance, and hair integrity (i.e., reduced loss of cross-linking). In that transition region, the silicone increased the process efficiency, allowing the hair to be straightened at lower temperature.

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