Mechanical Characterization of Brown and Grey Hair

Application Note

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Introduction

Most people who have some of both hair types perceive definite differences between pigmented and unpigmented (grey) hair. The vocabulary of common expression leads one to believe that the perceived differences are effectively *mechanical:* grey hair is said to be "stiffer," more "wiry," and generally more unruly. In this work, we demonstrate the experimental advantages offered by the combination of two nano-scale analytic tools from Agilent Technologies. The 8500 FE-SEM (Field-Emission Scanning-Electron Microscope) was used to make highly resolved diameter measurements, and the T150 Tensile Tester was used to do the mechanical testing.

Uncertainty analysis reveals that *diameter* is the most critical measurement in the determination of the Young's modulus of a fine fiber such as hair, both because the diameter is small and because the quantity is squared in the calculation of cross-sectional area. Thus, using the 8500 FE-SEM to measure diameter with a resolution of nanometers dramatically reduces the uncertainty of the resulting Young's modulus. (Most published studies on hair report using micrometers to measure hair diameter.) Furthermore, the 8500 FE-SEM is easy to use; the time for each diameter measurement was about 2 minutes.

The patented design of the T150 Tensile Tester confers several advantages for testing hair [1]. First, it has unparalleled accuracy and resolution, because the reaction force is generated electromagnetically, not by passive deflection of a spring. For the T150, the force capacity and resolution are 500 mN and 50 nN, respectively. Second, a dynamic oscillation can be superimposed upon the semi-static reaction force. This dynamic superposition has a number of advantages. It allows the measurement of modulus as a continuous function of strain, even after the onset of plasticity. This capability has been shown to be especially important for characterizing synthetic and natural polymer fibers, because the deformation mechanisms change as a function of strain. At low strains, the polymer chains unwind; at high strains, they stretch. For example, Blackledge et al. found that the silk of a black widow spider *(Latrodectus Hesperus)* has a modulus of 10 GPa at 10% strain, but a modulus of 30 GPa at 30% strain [2]. Another advantage of the dynamic oscillation is that it offers the capability of measuring loss modulus—the ability of the fiber to damp out energy.

Literature Review

Previous investigations into the physical properties of pigmented/unpigmented hair have yielded contradictory results. Recently, Kaplan et al. published a comprehensive study on the physical properties of pigmented and unpigmented hair [3]. They reviewed previous work and proposed that contradictory results arise from the fact that statistically significant differences between pigmented and unpigmented hair may be found for an individual, but that such differences may not be found in a population generally. For example, unpigmented hair may be significantly thicker for one person, and significantly thinner for another, so that the differences wash out when considered globally. They write:

> Restated simply, it is common to look at *global differences,* finding for example that grey hairs are not thicker than fully pigmented hairs when all fibres are pooled. In this study, we also look at individual differences to see if these hairs tend to be larger on an individual head. Differences that are strongly observable in some people, but not on average in the population we call *individual differences. Individual differences* do not describe the group, but do describe phenomena that may be real and important to those with the difference.

The importance of this distinction between global and individual difference was borne out by their results, and it should be remembered when comparing the results of the present work (which focuses on a single individual) to other studies.

Kaplan et al. acquired hair samples from individuals from a Mennonite community in Lancaster, PA. These subjects were chosen because they had similar ethnic backgrounds (Swedish); they were culturally prohibited from coloring or otherwise treating their hair; and the women covered their heads when outdoors, thus minimizing UV exposure. They acquired hair from 11 subjects (8 female, 3 male) between the ages of 53 and 65 who had "salt and pepper" hair. With respect to hair diameter, Kaplan et al. found grey hair had a significantly larger diameter, both globally and individually for four subjects. Young's modulus was measured under both dry and wet conditions. With respect to Young's modulus, they observed no global difference, but individual differences were observed in some subjects.

Experimental Method

Specimen Source

From a 41-year-old female, 24 hairs (N = 13 grey; N = 11 brown) were acquired. Hair had never been colored or otherwise chemically treated. Just prior to acquisition, hair was washed, but not conditioned. Hairs were cut close to the root, and care was taken to maintain orientation, root to end, so that testing specimens would all be obtained from the same place relative to the root.

Specimen Preparation

Individual hairs were mounted on cardstock across a 40 mm diamond, so that all had the same gage length (40 mm). Ends were fixed to the card using 5-minute epoxy.

Diameter Measurements

Using the Agilent 8500 FE-SEM, diameter measurements were made on 8 mm sections cut from just beyond the gage length. Hairs were mounted on SEM-compatible double-coated carbon conductive tabs on standard SEM pin stubs. Sections were mounted parallel to one another, six sections per stub. All micrographs were collected at 1 kV accelerating voltage. Diameter variation along the axis of the hair was minimal. The reported diameter for each hair was an average of three measurements.

Mechanical Testing

Fibers were tested mechanically using an Agilent T150 Tensile Tester with Continuous Dynamic Analysis (CDA) option. The test method "UTM-Bionix Standard Toecomp CDA.msm" was used for all testing. After fixing the template in the grips, the sides of the template were cut away to release the fiber for testing. Fibers were extended using a strain rate of 0.0027/sec. Although we intended to test the fibers to failure, this was not possible, either because the fiber pulled out of the epoxy or because the instrument did not have enough force capacity. Thus, tests were terminated at pullout or force saturation, whichever happened first.

During the entire test, an oscillating load was superimposed upon the constant-strain-rate loading. The amplitude of the force oscillation was

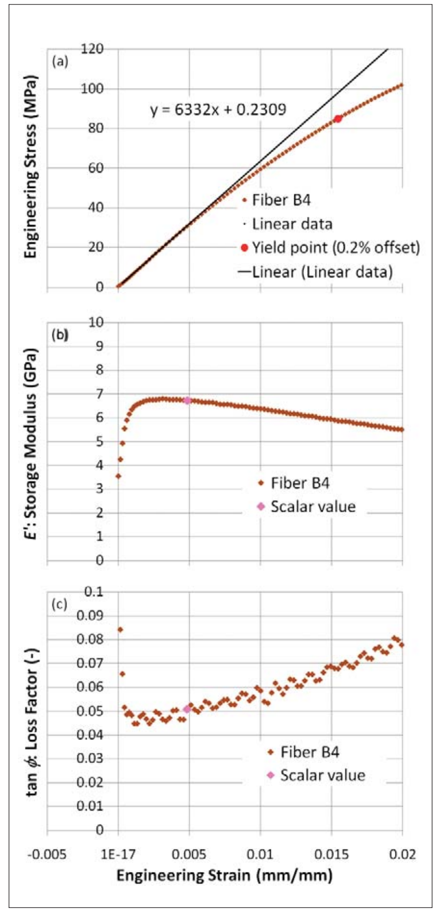

Figure 1. For one brown hair (B4), these are the simultaneous traces of (a) engineering stress, (b) storage modulus, and (c) loss factor, all as a function of engineering strain. The Young's modulus derived from the linear part of the stress-strain curve (a) is 6.33 GPa.

F_0 = 4.5 mN and the frequency was 20 Hz. The resulting displacement oscillation, z_0, and the phase shift, ϕ, between force and displacement were measured by means of a frequency-specific (lock-in) amplifier.

Analysis

Semi-statically, Young's modulus (E) is calculated as the slope of the engineering stress-strain curve during elastic deformation:

$$E = \frac{\delta}{\varepsilon} = \frac{P/A}{\Delta L/L} = \frac{P}{\Delta L} \cdot \frac{L}{A}, \qquad \text{(Eq. 1)}$$

where P is the force in the fiber, A is the cross-sectional area, L is original fiber length (gage length) and ΔL is the change in length. Figure 1a shows a typical stress-strain curve for hair and the Young's modulus derived from it.

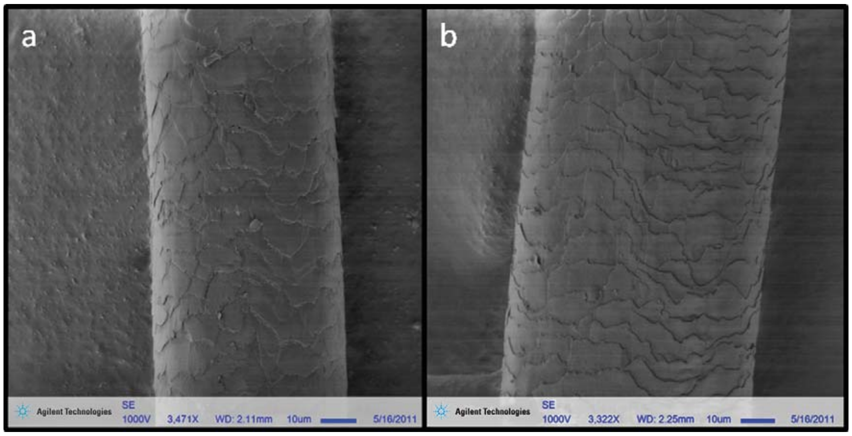

Figure 2. (a) Typical brown hair (B9; D=53.9 μm) and (b) typical grey hair (G2; D=74.7 μm). Magnification is the same in both images. These particular fibers were chosen, because their diameters were close to the averages for their respective groups.

The ratio $P/\Delta L$ is the semi-static stiffness, S. The dynamic stiffness, S', is calculated from the amplitude ratio, F_0/z_0, and the phase shift, ϕ, as

$$S' = \frac{F_0}{z_0}\cos\phi \qquad \text{(Eq. 2)}$$

Dynamic Young's modulus (a.k.a. 'storage modulus') is calculated by replacing the semi-static stiffness in $(P/\Delta L)$ in Eq. 1 with the dynamic stiffness of Eq. 2, giving

$$E' = \frac{\delta}{\varepsilon} = \frac{P/A}{\Delta L/L} = \frac{F_0}{z_0}\cos\phi \cdot \frac{L}{A}. \qquad \text{(Eq. 3)}$$

Likewise, we may define a loss modulus, E'', which characterizes the ability to damp out energy as

$$E'' = \frac{F_0}{z_0}\sin\phi \cdot \frac{L}{A}. \qquad \text{(Eq. 4)}$$

Practically, it is more useful to consider the value of the loss modulus in relation to the storage modulus. The loss factor is the ratio of the loss modulus to the storage modulus:

$$Loss\ Factor \equiv E''/E' = \tan\phi. \qquad \text{(Eq. 5)}$$

If the loss factor is close to zero, then the material is substantially elastic. If it is close to one, then the loss capacity is equal to the storage capacity.

The storage modulus and loss factor are calculated continuously during stretching. These channels are plotted for a typical fiber in Figures 1b and 1c. In order to report scalar dynamic properties, values of E' and $\tan\phi$ were taken at the elastic strain corresponding to the upper limit of the range over which Young's modulus was computed, i.e. at the pink diamond. These scalar values of E' and $\tan\phi$ are designated as $E'|_2$ and $\tan\phi|_2$, respectively.

For each reported result (E, S, $E'|_2$, and $\tan\phi|_2$), the significance of the difference between grey and brown hair was judged according to a two-tailed t-distribution test at the level of 95% confidence.

Results and Discussion

We found that the diameter of unpigmented (grey) hair was 36% larger than the diameter of brown hair. Figure 2 shows FE-SEM images of (a) one brown hair and (b) one grey hair. These two particular hair fibers had diameters that were close to the averages for their respective groups. No differences were observed in the cuticle morphology between the 24 tested hairs. Also, no mineral deposits or hair care product residue was observed.

Figure 1 shows traces of (a) stress, (b) storage modulus, and (c) loss factor as a function of strain for a single brown hair (B4). Traces for other fibers were similar in shape. Scalar values for E, S, $E'|_2$ and $\tan\phi|_2$ were determined as described in the Analysis section; these results, along with diameter measurements, are summarized in Table 1.

Grey hair had a lower Young's modulus than brown hair. Although Kaplan et al. did not find a global difference in Young's modulus, they did find significant differences for some individuals. Even though the grey hair tested in this work had a lower Young's modulus, it had a higher stiffness due to its larger diameter. Thus, for this subject, grey hair was significantly *stiffer*, not because it had a greater intrinsic elasticity as quantified by the Young's modulus, but rather because the grey hair was just more course. These results are in line with the global conclusions of Kaplan et al. and the anecdotal sense that grey hair is stiffer.

Even as the fibers began to yield plastically, they retained some elasticity as indicated by the storage modulus (Figure 1b). The gradual decrease in storage modulus with increasing strain indicates that as stiffer bonds break and manifest plastic yield, more compliant bonds increasingly govern the elastic response.

The damping in these fibers is small (less than 5% of the storage modulus at the end of the linear regime) but the increase with strain is interesting (Figure 1c). The bonds that persist to higher strains are more viscoelastic in nature.

Quantity		**Brown (N = 11)**		**Grey (N = 13)**		**Significant difference?**
	Units	**Value**	**Std. dev.**	**Value**	**Std. dev.**	
Diameter	um	56.2	10.0	76.5	12.1	yes
E	GPa	7.215	0.927	6.315	0.815	yes
$E'\|_2$	GPa	7.661	0.937	6.662	0.886	yes
$\tan\phi\|_2$	—	0.048	0.003	0.046	0.005	no
Stiffness	N/m	446.6	116.7	722.2	171.1	yes

Table 1. Summary of results.

It must be noted that the Young's moduli measured in this work were quite a bit higher than those reported by Kaplan et al. Their values were all in the range of 3.0–4.5 GPa. Our values were in the range of 6.0–7.5 GPa. If this difference is real, one explanation might be the younger age of the present subject. If it is an experimental artifact, then the close agreement between our static and dynamic results gives the present work credence.

Conclusions

For one person, grey hair was found to be stiffer than brown hair, not because the Young's modulus was larger, but because the diameter was larger by 36%. These results are consistent with previous studies, leading to the suspicion that hair products aimed at changing the composition of grey hair are unlikely to be effective in changing stiffness. This work demonstrates a process for determining the mechanical properties of hair that should be useful for evaluating the effects of various hair treatments, both common and novel.

The 8500 FE-SEM and the T150 Tensile Tester together comprise a powerful experimental package for testing hair and other fine fibers, offering accurate and highly resolved dimensional and mechanical measurements.

References

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