Measurement of System Compliance Using Template Grip Technique and the Agilent T150

Application Note

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Introduction

The measurement of mechanical properties of single fibers requires special techniques for gripping in order to avoid damaging samples and compromising the sample properties of interest. ASTM and ISO standards address this issue and suggest a template-gripping procedure whereby small frames are constructed to hold single-fiber samples [1, 2]. Direct handling of the template (as opposed to the fiber) prevents induced stress concentrations and premature failure initiation sites. However, the influence of all material in the loading path must be properly reckoned in order to achieve accurate strain measurements. Thus, for a given system of fiber, grip, template, and glue, the extension from outside the sample must be determined and subtracted from the total measured extension. This is typically done by measuring the system compliance. In the literature the compliance test has been successfully applied to different grip and fiber systems by many authors [3-5]. This application note details how this test is done on the Agilent T150, according to ASTM C1557-03.

Compliance Theory

The total measurement of the stiffness for this analysis can be viewed as the effective stiffness of two springs in series, which is represented in Equation 1, where k_{eff} is what the T150 measures, k_s is the system stiffness or stiffness outside of the sample, and k_a is the sample stiffness.

$$1/k_{eff} = 1/k_s + 1/k_a \qquad (1)$$

Compliance is the inverse of stiffness, and thus, Equation 1 can be rewritten in terms of compliance, giving Equation 2.

$$C = C_s + C_a \qquad (2)$$

Here C is the total measured compliance, C_s is the system compliance, and C_a is the sample compliance, the determination of which is the goal of this analysis. However, C_s must be measured first and then subtracted from C to obtain C_a. Using Hooke's Law one can formulate an expression relating C_a to sample properties: gage length (l_0), cross sectional area (A), and modulus (E).

$$E = \frac{\sigma}{\varepsilon} = \frac{Fl_0}{A\Delta l} = k_a\frac{l_0}{A} = \frac{l_0}{C_aA} \qquad (3)$$

Combining the result of Equation 3 with Equation 2 gives Equation 4.

$$C = C_s + \frac{l_0}{EA} \qquad (4)$$

Equation 4 is the equation of a line. The independent variable is the gage length, l_0, and the dependent variable is the total measured compliance, C. The slope and intercept of this line are $1/EA$ and C_s, respectively. Thus, a value for system compliance, C_s, is achieved by testing a series of similar samples having different gage lengths and plotting the resulting compliance, C, against gage length. The y-intercept of the best linear fit to these data is the system compliance, C_s. Then, having an accurate value for C_S, the modulus of the fiber is calculated according to Equation 5, which is obtained by solving Equation 4 for E.

$$E = \frac{l_0}{(C - C_S)A} \qquad (5)$$

When testing fine fibers, it is tempting to assume that the system is infinitely stiff relative to the sample. In other words, it is tempting to assume that the first term on the right-hand side of Equation 4 is zero and that the measured compliance is entirely that of the sample. The appropriateness of this assumption increases with decreasing sample compliance; that is, the zero-system-compliance assumption is better for long fibers of low modulus. Unfortunately, these conditions are not always obtainable and corrections to experimental data must be made. The present work uses fine copper wire to demonstrate (1) the process of determining system compliance, and (2) the effect (on reported Young's modulus) of neglecting system compliance.

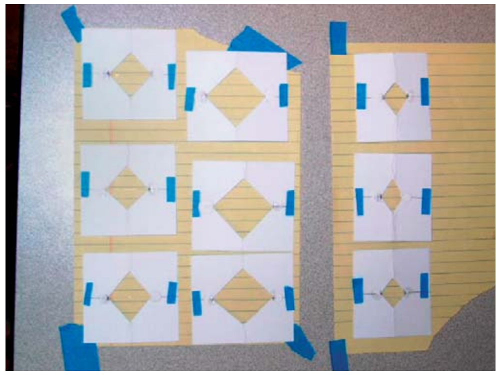

Figure 1. Samples prepared using paper templates. Samples bridge diamond shapes in template from left to right.

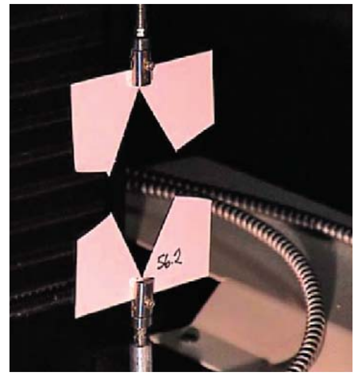

Figure 2. Paper template held with clamp grip.

Experimental Setup

Two kinds of copper wire, one having a diameter of 31.5±1.27 μm (AWG 48) and the other having a diameter of 44.7±1.77 μm (AWG 45), were used for these experiments (MWS Wire Industries, Westlake Village, CA). Three specimen lengths were prepared, at approximately 15, 25, and 35 mm to give the variation of the gage length needed for the system compliance measurement. Sample preparation followed the template gripping technique as seen in Figure 1 and outlined in the Agilent T150 Users' Manual. Samples were placed on the Agilent T150 UTM using the clamp grips as in Figure 2. Test parameters used for these experiments are listed in Table 1, and all samples were extended to tensile failure using the UTM-Bionix Standard Toecomp CDA method in NanoSuite. There were 9 tests for each kind of wire, 3 tests at 3 different gage lengths, as suggested by ASTM standard [1].

Test Parameter	Value
Strain Rate	1.0e-4/s
Delta for Data	0.001 mm
Harmonic Force	4.5 mN
Harmonic Frequency	20.0 Hz
Tension Trigger	500.0 μN

Table 1. Test parameters.

Results and Discussion

The compliance measurements for AWG 48 and AWG 45 copper fibers—mounted on paper templates using cyanoacrylate glue and held by clamp grips—gave system compliances of 3.682e-5 m/N and 1.607e-5 m/N, respectively. The determination of these system compliances by linear extrapolation is seen in Figures 3 and 4 using the test data listed in Tables 2 and 3. The linearity of the data in Figures 3 and 4 is evidence of consistent mounting, and this is essential for the accurate determination of system compliance. (Inconsistencies which might cause non-linear data and compromise system-compliance determination include template variation, fiber-glue interface weakness, poor gripping, and fiber misalignment.)

So why do the two different gages of wire result in different values for system compliance? It is important to remember that for template-mounted samples, the part of the fiber which is outside

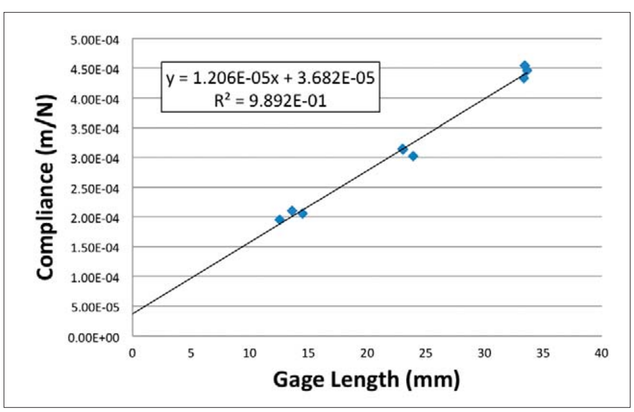

Figure 3. AWG 48 (31.5 μm) copper fiber with paper template and cyanoacrylate glue.

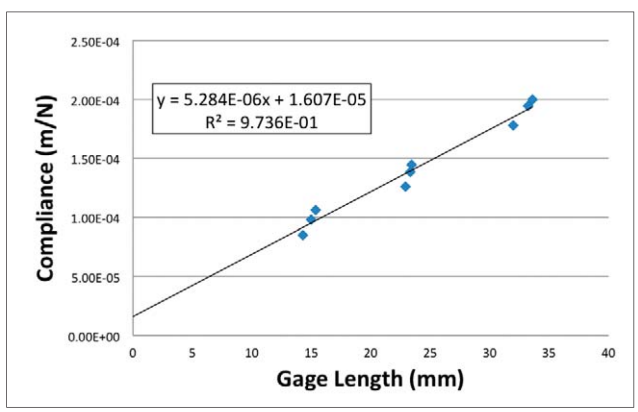

Figure 4. AWG 45 (44.7 μm) copper fiber with paper template and cyanoacrylate glue.

Test	Dia. (µm)	Gage Length (mm)	E (GPa)	C (m/N)	C_a (m/N)	E (compliance corrected, GPa)
1	31.5	14.5	90.4	2.058E-04	1.6893E-04	110.1
2	31.5	12.55	82.5	1.953E-04	1.5844E-04	101.6
3	31.5	13.61	83.1	2.102E-04	1.7341E-04	100.7
4	31.5	23.04	94.3	3.134E-04	2.7661E-04	106.9
5	31.5	23.91	101.5	3.024E-04	2.6560E-04	115.5
6	31.5	23.01	93.8	3.148E-04	2.7801E-04	106.2
7	31.5	33.35	98.8	4.331E-04	3.9626E-04	108.0
8	31.5	33.43	94.4	4.544E-04	4.1759E-04	102.7
9	31.5	33.63	96.7	4.461E-04	4.0930E-04	105.4

Table 2. AWG 48 Cu compliance test results.

Test	Dia. (µm)	Gage Length (mm)	E (GPa)	C (m/N)	C_a (m/N)	E (compliance corrected, GPa)
1	44.7	31.98	114.5	1.779E-04	1.6188E-04	125.9
2	44.7	33.23	108.9	1.945E-04	1.7844E-04	118.7
3	44.7	33.6	107.1	1.999E-04	1.8379E-04	116.5
4	44.7	22.92	115.9	1.260E-04	1.0997E-04	132.8
5	44.7	23.32	107.3	1.384E-04	1.2238E-04	121.4
6	44.7	23.43	103.3	1.445E-04	1.2848E-04	116.2
7	44.7	14.29	107.0	8.512E-05	6.9051E-05	131.9
8	44.7	14.98	97.4	9.804E-05	8.1967E-05	116.5
9	44.7	15.36	92.2	1.061E-04	9.0070E-05	108.7

Table 3. AWG 45 Cu compliance test results.

Sample	E	STD	E (compliance corrected)	STD (compliance corrected)
AWG 45 Cu	110.9	6.7	120.9	7.9
AWG 48 Cu	92.8	6.5	106.4	4.6

Table 4. Average Young's modulus (GPa) without and with compliance correction.

the gage length actually constitutes part of the gripping system. Thus, for a fiber which is stiffer, either because it has a larger diameter or because it has a higher modulus, the system stiffness will be greater. This is why the system compliance is lower (system stiffness is higher) for the larger AWG 45 wire than for the AWG 48 wire. This difference illustrates the importance of evaluating system compliance for each unique combination of fiber, glue, template, and grip.

For these samples, system compliance was not negligible. Table 4 lists the measured average Young's moduli with and without system-compliance correction. The average Young's modulus of AWG 45 copper increased 9.0% from 110.9 GPa to 120.9 GPa, while the average Young's modulus of AWG 48 copper increased 14.7% from 92.8 GPa to 106.4 GPa.

Other sources give the Young's modulus of copper as 110-128 GPa [6]. The value obtained for the AWG 45 wire is within this range, but the value obtained for the AWG 48 wire is not. The allowable variation in diameter for the AWG 48 gage may explain the low value for Young's modulus. For fibers, the Young's modulus obtained by tensile testing is very sensitive to diameter, both because the diameter is small, and because the value is squared in the calculation of cross-sectional area. If the true diameter of the AWG 48 wire were 30.23 µm—the lower limit of the range cited by the manufacturer—then the Young's modulus would be about 115 GPa. Highly resolved direct measurements of diameter would shed light on this issue. Because copper is a very soft metal, a second possible explanation is that there may be a small degree of plasticity in the stress-strain data, even in the range that is considered elastic.

The values for compliance-corrected Young's modulus in Tables 2 and 3 were determined using Equation 5. However, it is also possible to incorporate the new values for system compliance directly into the NanoSuite sample file, re-calculate strain accordingly, and determine compliance-corrected Young's modulus as the slope of the corrected stress-strain data. Figures 5 and 6 show such data before and

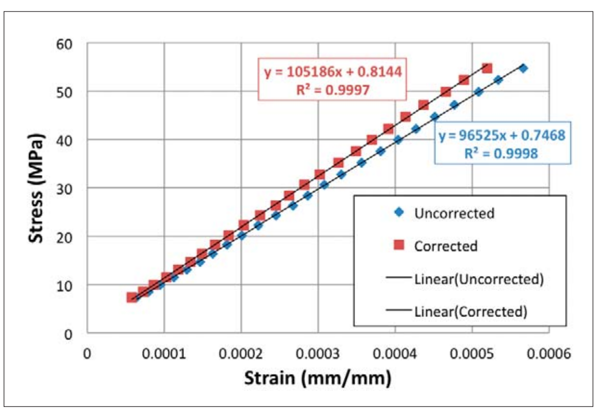

Figure 5. Corrected strain for AWG 48 Cu Test 9.

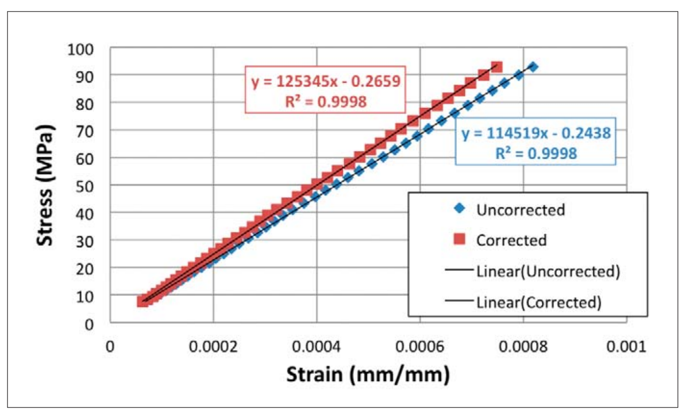

Figure 6. Corrected strain for AWG 45 Cu Test 1.

after correction as well as the moduli derived therefrom. The procedures for implementing compliance correction within NanoSuite are provided in the T150 Users' Manual.

Conclusions

This work demonstrates a procedure for determining system compliance and accounting for it in the determination of strain and Young's modulus. For the samples tested in this work, system compliance was not negligible. Once system compliance was properly accounted, the Young's moduli for AWG 45 and AWG 48 copper wire were measured to be 120.9 GPa and 106.4 GPa, respectively. (The most likely explanation for the low value for the AWG 48 wire is a value for diameter that is too high.) Although this work presents a procedure by which system compliance may be determined, the obtained values are only appropriate for the grip, template, glue, and sample combinations examined. For the best results, system compliance should be determined by this method for each unique combination of grip, template, glue, and sample.

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