Compliance calibration for fracture testing of anisotropic biological materials

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A B S T R A C T

The compliance technique has been used to monitor crack length during fracture and fatigue testing of materials. Difficulties arise when this technique is applied to anisotropic biological materials such as bone. In this tutorial, two different methods of analyzing compliance calibration data are described: the standard ASTM method and a new approach developed by the authors specifically for anisotropic materials. An example is given showing how data from equine cortical bone can be analyzed. In this example, calibration tests were conducted on thirty-six three point bend specimens machined from the mid-diaphysis of six pairs of equine third metacarpal bones. Cracks were propagated in three orientations with respect to the long axis of the bone: transverse, longitudinal, and radial. Specimen compliance was determined for a crack range of 0.30 to 0.65 times the specimen width from load vs. crack opening displacement data. The results demonstrate that the ASTM method is not applicable to anisotropic biomaterials such as bone. Rather, it is necessary to develop separate compliance calibration equations for each crack propagation orientation investigated.

1. Introduction

The length of a propagating crack is needed for the determination of fracture toughness. The governing equation for fracture testing relates the stress intensity factor, $K_I$, to the measured crack length ($a$) or normalized crack length ($a/W$, where $W$ represents the width of the specimen), the applied stress ($\sigma$) and a geometry factor ($Y$) specific to the specimen.
shape and loading conditions:

\[ K_I = Y \sigma \sqrt{\pi a} \]  

(1)

For standard fracture toughness determinations, a specimen with a sharp crack or machined notch is monotonically loaded until catastrophic failure at which point \( K_I \) is equal to a critical value, \( K_c \), as given by the above equation. As the testing becomes more sophisticated, when other properties or phenomena are of interest to the investigator or the crack grows in a stable manner rather than catastrophically, it is necessary to monitor the crack length throughout the test. Similarly, under fatigue loading conditions it is desirable to know the rate at which the crack advances in a stable manner.

A variety of methods have been used to monitor a crack's progress throughout a fracture or fatigue test based on direct observation, changes in the mechanical response of the specimen, use of surface gages or the electrical characteristics of the specimen or surface film. Some are specific to certain types of materials and material conditions. For example, the electrical potential drop method, in which a current is passed through the specimen and the potential (voltage) drop is measured at points on either side of the advancing crack, is suitable only for metallic materials. A related technique involves sputter deposition of a carbon surface film on the test specimen (Ogawa and Suresh, 1991). During testing this carbon layer is connected to an electrical circuit where an increase in crack length is associated with a change in electrical resistance (Ogawa and Suresh, 1991). This technique results in accurate measurements but is not applicable to testing bone in a “natural” wet state due to the damage that would be done in the sputter coating process.

Numerous researchers have applied a variety of different techniques to determine the crack length during fracture testing of bone (Wright and Hayes, 1977; Behiri and Bonfield, 1984, 1989; Malik et al., 2002, 2003; Vashishth, 2004; Nalla et al., 2005). Behiri and Bonfield employed direct observation of the crack using a traveling microscope and a high speed camera to record images of the crack (Behiri and Bonfield, 1984, 1989). This technique monitors the edge of the crack on the specimen surface, but the relationship to crack length in the interior of the specimen is unknown. If the crack front does not remain straight, an investigator may under- or overestimate the actual length of the crack, leading to inaccurate fracture toughness values or fatigue crack growth rates.

A crack propagation gage has been applied to the surface of bone specimens, much like a conventional strain gage (MicroMeasurements, Malvern, PA) for the determination of crack length (Vashishth, 2004). The electrical resistance of the crack propagation gage changes as the crack propagates, allowing the determination of crack length during bone growth. Vashishth applied this technique to tests of bovine and antler cortical bone, using compact tension type (CT) specimens. This method is only applicable to specimens that are sufficiently large to accommodate the crack propagation gage. When smaller specimens are used, such as those with the single edge notch bend geometry, SE(B), there may not be sufficient area for attachment of a crack propagation gage.

A load-line compliance technique has been applied to human cortical bone specimens to determine crack length during stable crack propagation (Nalla et al., 2005). The method, originally described by Saxena and Hudak, used the mechanical compliance of a specimen to determine crack length (Saxena and Hudak, 1978). This method relates two easily measured quantities, applied load and specimen deflection, to obtain crack length. In its simplest form, the compliance is the ratio of the specimen deflection to the applied load. As the crack length increases, the specimen compliance also increases because the deflection for a given load increases. An equation describing the relationship between specimen compliance and crack length is ascertained through calibration so that measured compliance can be used to determine crack length. This method works particularly well in isotropic and elastic materials. It is advantageous for tests of bone because it obviates the need for visualizing cracks or attaching measuring devices to a bone surface. The primary shortcoming of this method is the use of the general compliance calibration equation found by Saxena and Hudak. These equations are appropriate for isotropic, elastic materials. However, bone is anisotropic and viscoelastic. Furthermore, successful application of load line displacement measurements is complicated by the need to exclude displacements of the testing machine.

As an alternative, Malik et al. validated and employed a compliance method based on direct measurement of crack opening displacement (COD) for bone specimens (Malik et al., 2002, 2003). Varadarajan et al. employed a similar technique while creating compliance calibration curves of ultra high molecular weight polyethylene which was irradiated at different doses (Varadarajan and Rimnac, 2006). Malik, et al. used the technique on equine third metacarpal bone material, with the crack propagating transverse to the longitudinal axis of the bone. They developed compliance calibration equations specific to bone rather than relying on standard published equations and demonstrated that different calibration equations were needed for material sampled from two different regions of the third metacarpal bone. Malik et al. subsequently applied the compliance method based on COD measurements to assess the rising R-curve fracture behavior of cortical bone (Malik et al., 2003). However, they suggested that the calibration method might also yield different calibration equations across subjects.

This tutorial describes a compliance calibration method which can be used to determine crack length based on COD and load for anisotropic biological materials. An example is included using equine cortical bone to show how this method can be applied to individual animals and for different crack propagation orientations (radial, longitudinal and transverse). The compliance method is sensitive to the elastic properties of the material. Due to the osteonal structure of equine cortical bone, its elastic behavior varies with orientation (that is, it is anisotropic). In addition, the elastic properties of bone vary by individual subject. Thus, it is desirable to create specific calibration equations for bone in each orientation in which it will be tested and for each subject. The results are also compared to results derived using uniform compliance calibration equations based on ASTM Standard E561 for single edge notch bend specimens, SE(B).
2. Methods

2.1. Overview

Three-point bending compliance calibration experiments were carried out on equine cortical bone at room temperature for SE(B) specimens using a MTS 810 servohydraulic test system (MTS Systems Corporation, Eden Prairie, MN). Crack opening displacement was measured by an MTS model 632.26 E-30 extensometer attached by elastic bands to thawed specimens as shown in Fig. 1. All three-point bending tests were run in COD control for the determination of specimen compliance over a range of generated crack lengths (eight crack lengths over a range of 0.30 < a/W < 0.65) for each specimen. Actuator displacement, time, COD, and load were measured (125 samples/s), the latter using an Interface 1010-AF force transducer (Interface, Scottsdale, AZ).

2.2. Compliance test

Specimens were mounted in a stainless steel three-point bend fixture with roller supports and pre-loaded to approximately 5 N (Fig. 1). For the initial crack length after pre-load, COD was increased at 0.0008 mm/s in a ramp configuration to a predetermined nominal load level of 50 N. A compliance measurement was made by unloading the specimen at a rate of 0.008 mm/s until the COD decreased by 0.005 mm. Finally, the COD was decreased at a rate of 0.008 mm/s until 80% of the nominal test load was reached. This unloading/reloading sequence was repeated two more times. The compliance measurements were made at an unload/reload ramp rate that was greater than the test loading rate to avoid errors due to anelastic strains while still using a rate that was slow enough to give an adequate number of data points from which to calculate the slope of the COD-load data line. Specimens then were removed from the fixture and the crack was advanced using a 0.2 mm thick razor saw (X-Acto, Columbus, Ohio) and crack length was manually measured with a traveling microscope. The nominal load level was reduced from 50 N for the shortest crack length to 30 N for the longest crack length to prevent specimen damage as compliance increased with increasing crack length.

2.3. Compliance determination for anisotropic materials

A typical COD-load record is shown in Fig. 2. The compliance of the specimen is defined as the slope of the COD-load data line. Such data often exhibit nonlinearity at the maximum and minimum values associated with the reversal of direction of the hydraulic actuator. Thus, the upper and lower 10% of the COD-load data were trimmed to avoid these nonlinear end effects and the average compliance (C) was calculated from the slopes of least squares regression fits on the decreasing (unloading) portions of the three compliance checks at each crack length. Using the approach of Saxena and Hudak (1978), the transform of the dimensionless compliance (Ux):

\[ U_x = \frac{1}{1 + \sqrt{BEC}} \]  

was correlated to normalized crack length (a/W) by a least squares polynomial regression:

\[ a/W = \lambda_0 + \lambda_1 U_x + \lambda_2 U_x^2 + \cdots + \lambda_n U_x^n \]  

where a is the crack length, W is the specimen width, E is the elastic modulus, B is specimen thickness, C is compliance and \( \lambda_n \) are regression coefficients. Saxena and Hudak found a fifth order polynomial fit their data. Similar to Malik’s findings (Malik et al., 2002), a linear form was found to fit the present data best.

Because of bone’s inherent anisotropic mechanical properties and the variability of the elastic modulus within the cortex, a unique method is required to determine the elastic modulus for use in the dimensionless compliance transform Eq. (2). Saxena and Hudak (1978) used an independent measure of the elastic modulus in their research on metallic C(T) specimens. This is more difficult in bone for a variety of reasons including the variability of the elastic modulus and the
limitations on the availability of bone specimens. Therefore the elastic modulus was determined in the same manner as was used by Malik (Malik et al., 2002).

A key assumption of Malik’s method is that specimens of the same geometry should follow the same $U_x$ vs. $a/W$ curve. Therefore, it was necessary to define a reference curve based on previously measured elastic modulus values of 15 GPa for the transverse orientation and 10 GPa for the longitudinal and radial orientations (Martin et al., 1998). These elastic moduli were used in Eq. (2) for the compliance check for the initial crack length, and subsequently in Eq. (3), resulting in $\lambda_0 = 1$ and $\lambda_1 = -3$ for all orientations. Once this curve was defined it was possible to individually determine the adjusted elastic modulus and calibration curve for each ith crack length such that the corresponding $U_{x,i}$ value was coincident with the reference curve given by $a/W = 1.00 - 3.00U_x$ as given by:

$$E_i' = \frac{1}{BC_i} \left( \frac{-3.00}{(a/W - 1.00)} \right) - \frac{1}{2}. \quad (4)$$

The elastic modulus for the specimen was then determined from the average for all compliance checks:

$$E' = \frac{1}{n} \sum_{i=1}^{n} E_i' \quad (5)$$

Once the adjusted elastic modulus had been calculated for each specimen it was possible to use this modulus in the calculation of the compliance calibration using Eqs. (2) and (3). This generated different coefficients, $\lambda_0$ and $\lambda_1$, specific to each calibration specimen.

### 2.4. Compliance determination using ASTM method

The method found in ASTM 561 is similar to the one outlined above with two distinct differences: (1) the equation that is used to calculate the transform of the dimensionless compliance is:

$$U_x = \frac{1}{1 + \sqrt{\frac{4BCW}{S}}} \quad (6)$$

and (2) the equation used to calculate $a/W$ is a fifth-order polynomial:

$$a/W = 0.999748 - 3.9504U_x + 2.9821U_x^2 - 3.21408U_x^3$$

$$+ 51.51564U_x^4 - 113.0314U_x^5 \quad (7)$$

As in the previous compliance calibration method it was necessary to determine the elastic modulus of each specimen. Since the first crack length was known for each specimen ($a/W$ of approximately 0.3) it was possible to solve Eq. (7) for the $U$ value that yields the correct $a/W$. Using this $U$ value it was possible to solve Eq. (6) for $E$ based on the measured compliance, $C$, for the specimen:

$$E = \frac{S}{BWC} \left( \frac{1}{U_x} - 1 \right)^2. \quad (8)$$

Once $E$ was known for each specimen based on the initial crack length, Eqs. (6) and (7) could be used to determine the crack length predicted by the ASTM compliance method for all subsequent extended crack lengths. These results were then compared with actual crack lengths using a t-test to determine whether the ASTM method yielded different results than the method developed by Malik.

#### 2.5. Compliance calibration experiment

To study the details of the compliance calibration approach, thirty six cortical bone specimens were machined from unilateral third metacarpal bones from 12 Thoroughbred horses (ages 2–7; Male, Female, and Gelding). Prior to machining, bones were debried of soft tissues, wrapped in saline soaked towels, and stored at $-20 \, ^\circ C$. SE(B) specimens were machined under saline irrigation to yield nominal dimensions of $L = 27 \, mm$, $W = 6 \, mm$, $B = 6 \, mm$, $h = 0.35 \, mm$, $a_0 = 0.3 \times W$, notch tip angle = 30 degrees (from vertical), and $S = 24 \, mm$ (Fig. 3) in accord with ASTM Standard E399 (ASTM, 1993). Each specimen was oriented within the metacarpal cortex so that the initial notch was aligned to propagate a crack in one of three orientations (radial, longitudinal or transverse) as illustrated in Fig. 4. In the transverse orientation, the crack propagated in the periosteal to endosteal direction because that is the direction in which cracks propagate in vivo. Similarly, the radial specimens were cut for periosteal to endosteal propagation to match the transverse specimens. Longitudinal specimens were cut for proximal to distal crack propagation.

Each specimen was machined from one of four diaphyseal locations: proximodorsal, proximolateral, distodorsal, or distolateral (Fig. 4). One specimen for each crack orientation, transverse, longitudinal, or radial, was taken from the third metacarpal bone of the left or right leg for each horse (the contralateral bone was reserved for R-curve fracture specimens for a subsequent study). The radial and longitudinal specimens were always taken from the same region (either dorsal or lateral), with one specimen coming from the proximal portion of the middle of the diaphysis and the other specimen from the distal portion. The transverse specimen was taken from the remaining region, and from either the proximal or distal portion of the middle of the diaphysis. Specimen layout between left and right legs and distal and proximal locations was switched between successive horses. The distribution of specimens was designed to be completely balanced (6 from each region/orientation combination).

Compliance calibration equations have been shown to differ by bone region (Malik et al., 2002), and due to the anisotropic nature of bone it was assumed that they would also differ with crack propagation orientation. Therefore it...
was necessary to perform a statistical analysis to determine which factors affect the compliance calibration coefficients. A mixed model analysis of variance (SAS, Cary, NC) was used to assess the effects of bone region (dorsal, lateral), horse age, elastic modulus, orientation (transverse, longitudinal, radial), and horse (as a repeated measure), and the two way interactions between region and orientation and between age or the region*orientation interaction (Tables 1–3). Both $\lambda_0$ and $\lambda_1$ were statistically different between longitudinal and transverse orientations ($p = 0.012$ and $0.011$ respectively). Differences for $\lambda_0$ and $\lambda_1$ between radial and transverse orientations ($p = 0.109$ and $0.068$, respectively), and between radial and longitudinal orientations ($p = 0.203$ and $0.366$, respectively) were not statistically significant. Fig. 6 shows the six individual compliance calibration fits for a specific region–orientation (lateral–transverse) combination. These results support the initial hypothesis that different crack propagation orientations would result in different compliance calibration coefficients.

When the ASTM calibration equation is used to calculate crack length it underestimates the actual crack length by as much as an $a/W$ of $0.02$. Fig. 7 shows an example of this result for the transverse orientation in the dorsal region. When the results of the ASTM calibration equation and the individual compliance calibration equation are compared they are found to be significantly different (t-test p-value of $<0.0001$). This finding supports the need to perform a set of compliance calibration experiments on bone in order to obtain the most accurate crack length predictions during future R-curve testing.

4. Discussion

The results of the example for bone demonstrate that the standard ASTM compliance calibration equation does not accurately predict the crack length of anisotropic specimens of a biological material based on its compliance. This conclusion is reinforced by the fact that the compliance calibration results differ with crack propagation orientation, showing that it is necessary to build an individual compliance calibration curve to accurately predict crack lengths in fracture testing of cortical bone rather than using a single equation as given by the ASTM standard. The ASTM compliance calibration equation consistently underestimates the length of the crack for any given experimental compliance measurement. Although this error is as small as an $a/W$ of $0.02$, it is considerable because this small error will be magnified in the calculation of the fracture toughness for R-curve experiments. Because different orientations

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**Table 1 Summary of ANOVA for $\lambda_0$ and $\lambda_1$.**

<table>
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<tr>
<th>Parameter</th>
<th>Effect</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
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<td>$\lambda_0$</td>
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</tr>
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<td></td>
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<td>Age</td>
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<td>Region*Orientation</td>
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<td>Orientation'E</td>
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<td></td>
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<td></td>
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<td>0.4103</td>
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*aStatistically significant.*
Malik has shown that compliance calibration methods are applicable to bone if the anisotropic elastic properties are taken into consideration (Malik et al., 2002). In the current research the two coefficients of the compliance calibration equation were found to depend on both orientation and elastic modulus, supporting the hypothesis that separate compliance calibrations are necessary for each orientation.

Elastic modulus was included as a factor in the statistical analysis because it was found to differ significantly between orientations ($p < 0.05$). In the current study the average elastic modulus of the transverse orientation was found to be 13.13 GPa, whereas the average moduli for the radial and longitudinal orientations were 7.74 and 8.73 GPa respectively. These values are similar to the initially assumed values of 15 GPa for the transverse orientation and 10 GPa for longitudinal and radial orientations. The orientation specific moduli derive from the anisotropic nature of bone tissue. Osteons, which are usually oriented approximately longitudinally, result in different material properties when bone is loaded in different orientations (Behiri and Bonfield, 1989; Martin et al., 1998). The interaction between orientation and elastic modulus (orientation*modulus) was investigated and found not to be significant, suggesting that these factors are independent (Table 1).

The anisotropic nature of bone described above is believed to be responsible for the differences found in $\lambda_0$ and $\lambda_1$ for different crack propagation orientations. For both $\lambda_0$ and $\lambda_1$, the only significantly different orientations are longitudinal and transverse ($p < 0.05$). No significant differences in $\lambda_0$ and $\lambda_1$ were found when the radial and transverse (or radial and longitudinal) orientations were compared. It is important to note that not only is the microstructure different in the transverse orientation compared to the other two, but the elastic modulus is significantly different ($p < 0.001$). Both the radial and longitudinal moduli are different from
the transverse modulus ($p < 0.0001$) but the radial and longitudinal moduli are not significantly different from each other ($p > 0.05$). This result suggests that something other than just the difference in elastic modulus is responsible for the differences in $\lambda_0$ and $\lambda_1$ between orientations, e.g., the directionality of the osteons in the bone. Finding other potential differences than the elastic modulus is similar to the conclusion reached by Malik regarding regional differences, although he only investigated a single crack propagation orientation.

Previously, regional differences in $\lambda_0$ and $\lambda_1$ were found (Malik et al., 2002), whereas the present results revealed no regional differences. A possible explanation for this inconsistency is microstructural differences, or lack thereof, between the regions. Neither Malik’s nor the current study involved histologic analysis of the bone structure. Martin has reported significant differences between regions in the microstructure of equine cortical bone (Martin et al., 1996). These microstructural differences may have led to the differences in $\lambda_0$ and $\lambda_1$ that Malik’s study revealed. The same microstructural differences are expected in the present study.

Table 2

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<th>Horse</th>
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<th>$\lambda_1$</th>
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<td>0.90</td>
<td>-2.37</td>
<td>12.14</td>
</tr>
<tr>
<td>2373</td>
<td>L</td>
<td>$T^a$</td>
<td>0.95</td>
<td>-2.68</td>
<td>14.23</td>
</tr>
<tr>
<td>2361</td>
<td>L</td>
<td>$T^a$</td>
<td>0.96</td>
<td>-2.77</td>
<td>11.76</td>
</tr>
<tr>
<td>2408</td>
<td>L</td>
<td>$T^a$</td>
<td>0.96</td>
<td>-2.76</td>
<td>12.31</td>
</tr>
<tr>
<td>2402</td>
<td>L</td>
<td>$T^a$</td>
<td>0.92</td>
<td>-2.55</td>
<td>12.47</td>
</tr>
</tbody>
</table>

![Fig. 6](image1.png)

Table 3

<table>
<thead>
<tr>
<th></th>
<th>$\lambda_0$</th>
<th>$\lambda_1$</th>
<th>E (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transverseb</td>
<td>0.96 (0.07)</td>
<td>-2.758 (0.46)</td>
<td>13.134 (1.91)</td>
</tr>
<tr>
<td>Longitudinalb</td>
<td>0.983 (0.05)</td>
<td>-2.938 (0.33)</td>
<td>8.739 (1.61)</td>
</tr>
<tr>
<td>Radialb</td>
<td>0.935 (0.03)</td>
<td>-2.69 (0.21)</td>
<td>7.744 (1.31)</td>
</tr>
<tr>
<td>Dorsal</td>
<td>0.97 (0.06)</td>
<td>-2.838 (0.36)</td>
<td>10.152 (2.38)</td>
</tr>
<tr>
<td>Lateral</td>
<td>0.947 (0.06)</td>
<td>-2.742 (0.35)</td>
<td>9.643 (3.37)</td>
</tr>
</tbody>
</table>

![Fig. 7](image2.png)

However, the current results are based on fewer specimens than Malik’s results as well as smaller specimen volumes that are more localized to the region of interest. These differences in test methodology could account for differences in the influence of region. Regardless of the source of these differences, or lack thereof, this difference in results further supports the need for individual compliance calibration equations paired with fracture or fatigue experiments.
5. Conclusions

The purpose of this tutorial was to describe a compliance calibration technique for measuring crack length in an anisotropic biological material. The compliance calibration method was contrasted with the ASTM standard method to determine if it was necessary to perform individual compliance calibrations. The ASTM method did not accurately predict crack lengths in any crack propagation orientation of equine cortical bone. Also, it was found that compliance calibration equations from different orientations are statistically different, thereby supporting the need for individual compliance calibration equations when using the compliance technique on anisotropic biomaterials.

References


