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Mechanical Testing of Bone with Non-Standard Techniques

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ABSTRACT

A non-standard technique for testing of bone was developed based on the wetting and penetration properties of quick-setting adhesives and adhesion between bone and a porous green body. Furthermore, tooling has been developed for the quick production of specimens and the quick locating and alignment of specimens in the gripper system during the consolidation process. This technique allows gripping to be performed within a relatively short time frame, reducing property variation during the time bone is held in dry air. Furthermore, the method lends itself to monitoring of extension by optical means, and measurement through miniature contact probes rather than extensometers. A prototype device was designed and built on the bed of a coordinate measuring machine with a repeatable precision of 0.25 micrometer and simultaneously verified with a second probing system at a resolution of 2.5 micrometers. The method also lends itself to improved reinforcement of the support areas of the tensile specimen in the third dimension and concentration of deformation within the gage area. A Finite Element Model of the tension specimen and the adhered gripper system was used in comparing the level of direct shear stress in the adhered areas "separation stress" to the stress in the gage length. The Method developed can provide information related to elastic and inelastic mechanical properties. Furthermore, properties can be measured related to primary, secondary and tertiary creep models for development of constitutive models needed in long-term loading of bone during orthodontic and other procedures.

INTRODUCTION

Mechanical testing of bone is a mature art that originally borrowed techniques, measurement systems, and equipment traditionally used in classical mechanics and engineering sciences for quantifying the mechanical behavior of metals. Some adaptations made to allow testing of polymers and rubbers have facilitated the quick deployment of testing experiments. Despite this history, some questions still remain unanswered, and some concerns still prevail. For example, concerns about non-linearity, frictional effects, localized high strains at the extremities of bone specimens, and other factors that are likely to cause errors were recently expressed (Grimm, 2000). Other concerns about shortcomings of single-axis standard testing equipment have been expressed (Steffen, 2000), and new systems with multiple axes of loading constructed in order to spearhead improvements in classical testing methods. Similarly, to permit measurement of deformations on specimens submerged in biological fluids, non-contact techniques have been implemented by incorporating 3-dimensional microscopic imaging techniques (Thomann et al., 2000).

Other motives, such as: development of techniques for in-vivo measurement of bone properties, measurements on individual patients, measurement of macromolecular properties, and establishment of non-contact non-destructive measurements, have lead to the development of acoustic, nanoindentation, optical tweezers, and other specialized techniques for mechanical characterization of bone (Luo and An, 1998, Turner et al., 1999, Windhagen et al., 1999, and Zysset et al., 1999). Along parallel lines, micro-testing techniques have been necessitated by the need for investigation of properties in various directions within the bone lamellae (Liu et al., 1999). At a larger scale, but still miniature specimens that could not be tested by conventional techniques, have been tested (Speirs et al., 1999) between compressive plates.

Alternatively, the need for measurement of macroscopic composite properties of long bones has lead to the development of customized procedures, and devices capable of testing whole-femoral bones in multiple directions perpendicular to a single axis (Bramen et al., 1998). Test modifications have not only affected uniaxial testing procedures, but also measurement of fatigue properties. Recent work (Griffin et al., 1999) has quantified differences in measured properties obtained through two different techniques, one using displacement control, and one using and equivalent force control.

Even more uniquely focused, needs for impact modeling of the cranial complex have lead to studies and development of methodologies for quantification of energy absorption characteristics of bone, a characteristic property dependent on non-linear behavior. Quantification of these properties and correlation with patient age was performed for cranial bone and suture bone by tensile and three-point bending (Margulies and Thibault, 2000). Energy dissipation properties of bone have also been studied with transient techniques, where an induced stress wave is observed to lead the resulting strain wave. Such an approach was used in an investigation of dampening characteristics of bone within the range of frequency of normal activities (Garner et al., 2000).

In the particular case of testing of bone harvested from small animals and young children, initial sample size and shape is an issue of critical concern. Furthermore, the sensitivity and resolution of standard testing systems, creates further challenges that render testing a laborious practice and testing procedures an evolving science. This work focuses on a method for testing of small and non-standard bone samples, that makes use of mounting practices with no clamping forces and deflection measurement without the use of contact extensometers.

METHODS

Figure 1 illustrates the main components of the system with the loading sub-system removed for ease of visualization. Removable attachments have been designed and built for addition to the granite plate (A). This plate is used as the reference surface and foundation of a Coordinate Measuring Machine (CMM). The attachments convert the machine to a measurement system for deformation of sample bone under transient or static loads. Machine software options have been selected to allow for timed collection of data as well as instantaneous selective measurements. The system completed to date allows for creep testing with dead weights, and tensile testing with discrete weights. Future plans include the addition of a programmable motorized loading device that will be integrated into the system for synchronized experimentation.

Deformation Measurement System

The Brown and Sparpe MicroVal 3-axes CMM used as the main frame for the new measurement system was not permanently modified for the purpose of the experiments. Instead, an additive sub-system was built that can be easily removed to restore the machine to its original condition and function. The attachment plate (B) carries all of the components that have been added to the base system. Arrows (X), (Y), and (Z) designate the corresponding degrees of freedom along which there is an active measurement

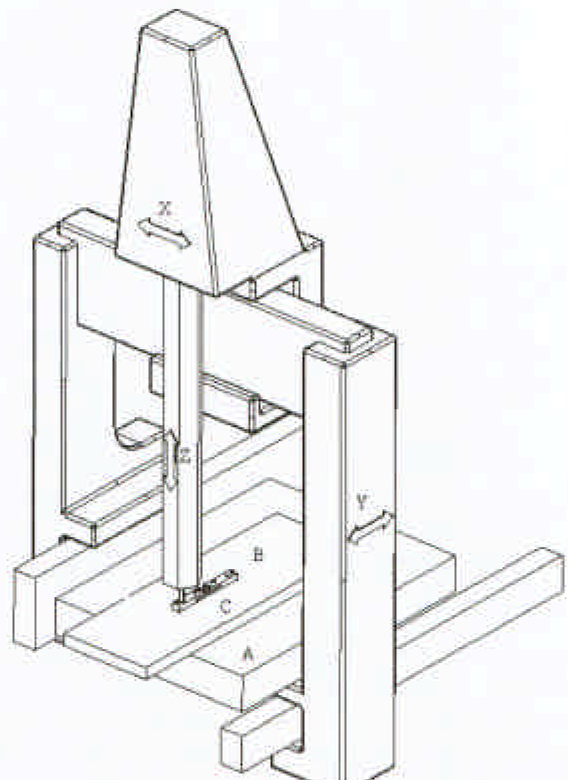

Figure 1. Main components of the coordinate measuring machine used to measure specimen deformation.

system. Each of the axes of motion is supported by multiple linear air bearings. When a set of air bearings is switched-off, the corresponding axis is immobilized without disabling the measurement instrumentation. This feature allows for measurement and reporting of any error that may be caused due to side-loading deflections of the specimen. The (Z) axis extends telescopically in a direction perpendicular to the plane of plates (A) and (B). The extending arm has a hexagonal shape, and is supported at any one time by a minimum of 12 air bearings. This member also carries a *9.5 (mm)* bore for attachment of probes. This bore is used for attachment of the vertical pole of the loading jaw of the testing fixture (C). A more elaborate illustration of the testing fixture (C) is shown in Figure 2.

System Resolution and Measurement Verification Method. Loading of the specimens is carried out in the direction of the Y-axis. During the course of an experiment, the X and Z-axes are locked in place, while the Y-axis is free to move. Displacement of each of the CMM axes requires a force that is dependent on the acceleration at which a test is conducted, but for accelerations below $10\ (m/s^2)$ the force is below $0.02\ (N)$. When used in conjunction with its full software drivers and a rigid probe such as the vertical pole (D) of the loading Jaw, the system is capable of recording deflections of the order of magnitude of *0.25(micrometers)*. For this precision to be obtained, the machine was located in a laboratory with a temperature maintained at $22^{o}\ C\ (\pm 0.5^{o}\ C)$. When used with a dynamic probing system such as a laser, or a RENISHAW sensor probe, the system's repeatability is reduced to the precision inherent in those sub-systems.

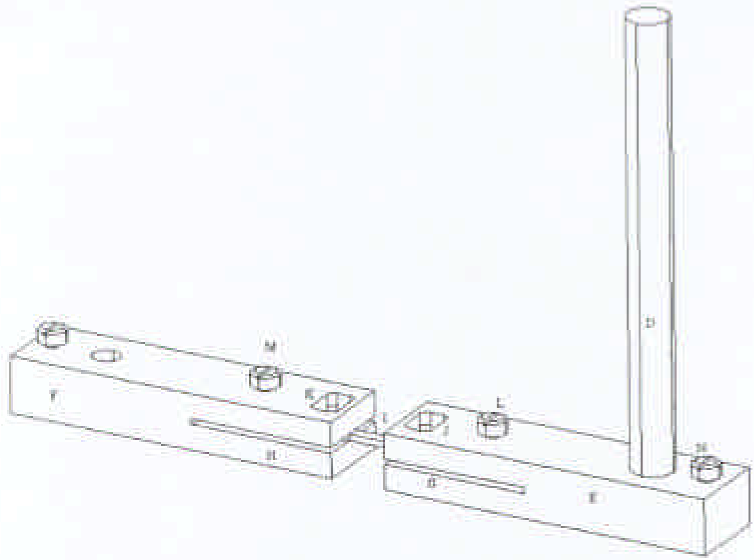

Figure 2. Detail view of the stationary and loading jaws of the testing fixture (c).

To ensure that the measured deflection is instantaneously verified, a second measurement system (Fowler Digi-max 54-568-000) was added to the fixture. This system allows the measurement of motion of the loading jaw (E) relative to the stationary jaw (F) with a resolution of *1 (micrometer)*. Verification is carried out manually at intervals corresponding to 4 measurements of the CMM system. An electronic connectivity feature is available on this instrument as well, but it is currently disabled.

Description of the Mounting Procedure. The Slits (G) and (H) in the loading and stationary jaws, respectively, have a free-standing opening of *1.5 (mm)*. This opening can be slightly increased by adjusting screws (L) and (M). This adjustment is necessary to allow customizing the jaws to the thickness of a particular bone sample (I). The opening is increased enough to allow stress-free insertion of the sample. The spring-back response of the jaws is not expected to apply a normal load for holding purposes, but merely to lightly touch the specimen. During installation, a *12 (mm)* Grade 1 gage block is inserted in the opening between the stationary and the loading jaws to properly space them, and to provide an alignment reference for the specimen. The jaws, the gage, and the specimen are temporarily affixed on a magnetic plate covered with disposable vinyl sheet, while permanent mounting takes place.

For permanent fixation, a low viscosity Cyanoacrylate adhesive with good wetting characteristics was selected and for a main support material (filler) a highly purified Aluminum Oxide powder with a sub-micron particle size. Apertures J and K are initially filled with two drops of adhesive, and a very light dusting of powder is immediately applied. 10 seconds afterwards, the powder is scribed with a sharp object to test its consistency, and upon setting, the two apertures are filled with tightly packed powder for an approximate depth of *0.5 (mm)*. The power has the tendency to conglomerate into a green body that cannot be easily removed. Three drops of adhesive are dropped to completely soak the powder. The process of packing and soaking powder is repeated at least 3-5 times for a minimum accumulation of *1.5 (mm)*. Every time fresh powder is added, the previous layer is checked for hardness by scribing. After one side is complete, the entire assembly is turned over, and the opposite site is processed in the same manner.

The setting time for the individual layers of adhesive varies between *5 and 15 (sec)* depending on the thickness of the layer of powder. The shelf life of the compound is several months, while the mounting process only lasts a few minutes. It is expected that on some experiments it will be necessary to mount a specimen, and refrigerate it immediately afterwards while other steps in the testing process were being carried out.

Force Measurement. The current setup was originally prepared for the purpose of mechanical characterization of bone under constant stresses, or more specifically for quantification of creep behavior under constant load. The same device is currently evolving into a test-setup for quantification of creep behavior under constant strain, where stress relief is being monitored, and a true stress-strain testing cell, through the incorporation of motion and force control. Figure 3 illustrates the current setup inside a container, and a pulley system where weights are suspended.

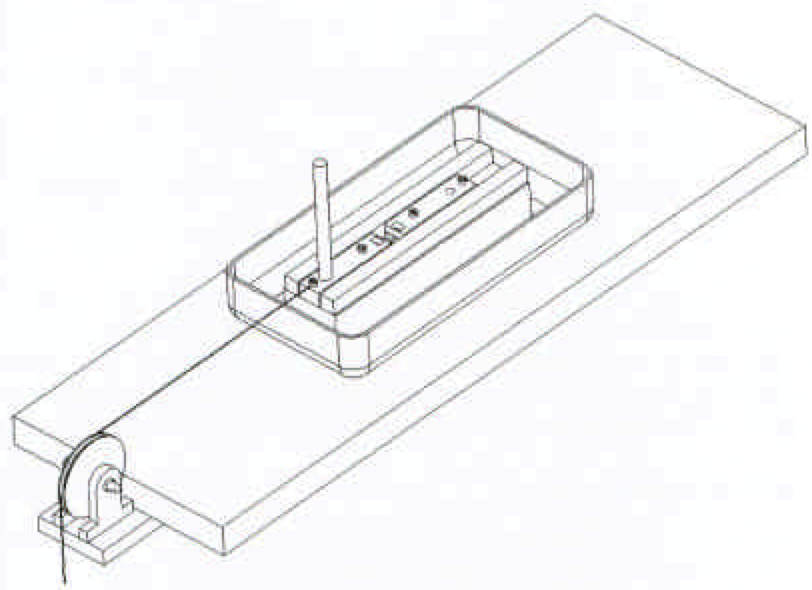

Figure 3. Assembled testing setup for constant stress creep testing.

Sample Cutting Method

In specimen preparation for polymeric materials, it is often easier to prepare samples with puching dies rather than with conventional machining practices. Dies provide more repeatable dimensions, a lower cost, a quicker practice, and a more efficient utilization of available materials. The benefits of this method also seem particularly helpful in the case of mechanical characterization of bone where available materials are naturally scarce. Figure 4 shows a sample die fabricated for use on preparation of standard dog-bone flat specimens. The rake angle on the die part and the flat configuration of the punch are intended for production of a flat blank and transfer of the stresses on the balk of the bone, not the separated specimen. Die preparation is an evolving art that seems to require use of wire electro-discharge machining, a process that results in steep rake angles that are needed in minimizing residual stresses and accumulated damage of the test sample. Current experiences also point to the need for a 3-Dimensional rake surface around the entire circumference rather than the single slope plane that is traditional in die manufacture. A commercial vendor was also identified with capabilities for overnight production and

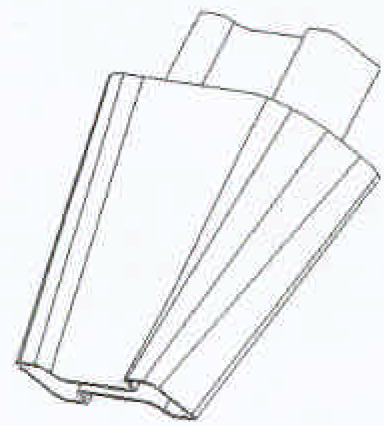

Figure 4. Schematic diagram of a punch and die-set used in specimen preparation.

delivery of dies for cases of experiments where preserved bone should not be refrigerated for periods in excess of one day.

RESULTS

This system is currently in a development stage, and most experiments conducted relate to assessment of precision, and qualification of various sub-systems. Upon completion, this phase will allow a comparison with standard techniques, and a quantification of the advantages in the areas of testing small samples, testing on odd-shapes, and measurement with no contact.

Fracture Strength of Filler Bond

Range-finding experiments were conducted to quantify the strength of the adhesive used in conjunction with a filler green body. For the purpose of these experiments, six samples were fabricated to a random thickness by using green body material, and then manually cut to a reduced thickness. After mounting in the jaws, they were tested to fracture and force-displacement measurements recorded. In all specimens, fracture occurred in the gage area, and no shear separation was observed in the adhered tension stubs. Table 1, lists the thickness of each sample, the location of fracture from the center of the gage length, and the fracture stress.

Computed Shear Separation Stress

The specimens manufactured with adhesive and filler mixture were modeled as elastic finite element models, in order to obtain an order-of-magnitude indication of the level of shear stress on the interface surface between adhesive and tension stabs, relative to the stress in the gage area. Figure 5 illustrates the model used and the distribution of Von Mises stress after a *0.2%* effective strain. Considering symmetry about 3 planes and displacement loading, the numerically calculated separation stress is found to remain well below the stress in the gage area. In the particular setup modeled, the separation stress is only *15%* of the stress in the gage area, even though the area of the stubs was only partially adhered (*52%* coverage) with a circular aperture of *6 mm* diameter rather than the standard rectangular shape.

Table 1. Measured Thickness and Location of Fracture for Tested Adhesive Samples.

ADHESIVE QUANTIFICATION

Sample Number	Thickness (mm)	Distance of Fracture from Mid-Point (mm)	Fracture Stress (MPa)
1	2.0	2.0	17.5
2	2.3	0	17.67
3	2.3	0	20.11
4	2.3	2.7	13.4
5	2.3	2.5	9.75
6	2.3	2.7	18.89

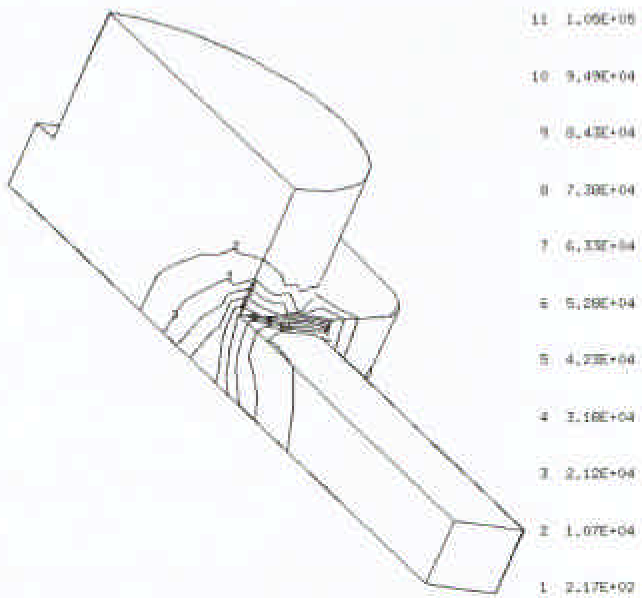

Figure 5. Von Mises stress distribution on symmetric model of tensile specimen adhered to cylinders.

CONCLUSIONS

One of the features of the method and test system developed was reduction of the normal holding forces on the bone sample through adhesion with a green body. The bond strength of the composite cyanoacrylate adhesive and the green body was tested and found to be adequate for testing of samples approximately dimensioned as D1708 standard miniature tensile specimens. The measured fracture strength of the composite averages at *16.22 MPa*. Preliminary tests have also been performed on odd-shaped animal bone samples for the purpose of assessing the suitability of the bond on wet bone. Even though the tests have not been fully quantified, the outcome has been encouraging.

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