INTRODUCTION
Over the past twenty-five years, and especially in the last decade, nano-scale instrumented indentation (or “nanoindentation”) has become the most widely accepted method for measurement of the elastic and plastic properties of thin films and small volumes. The widespread acceptance of nanoindentation for hardness and modulus measurement has recently been codified in recent ISO (2003) and ASTM (2007) documentary standards for instrumented indentation calibration and testing.

Nanoindentation is a primary source of experimental data that alternately inspires or supports theories of length-scale dependent phenomena, especially those that suggest that strength controlled by dislocation nucleation and motion is enhanced at the micro-scale. However, these sorts of experiments are dogged by noise, imprecision, and difficulty in instrument calibration. Marked improvements in temporal, force and spatial resolution will have the capability to explore discrete plasticity [1,2] and nanoscale fracture phenomena [3,4] with very acute indenters [5].

The goal of the development program presented in this paper is to establish a directly SI-traceable platform for nano-scale mechanical metrology. Such a system will advance nanomechanical measurement science by reductions in force and displacement noise with accompanying fast servo loops of force and displacement using state of the art real-time and field-programmable gate array (FPGA) based control. Incorporation of the latest force and displacement sensing techniques could reveal nanomechanical phenomena (especially coupled elastic-plastic-fracture phenomena) that are not accessible to commercial nanoindentation instruments or cantilever-based nanomechanical instruments (scanning probe- or atomic force- microscopes, for example).

This paper presents preliminary studies of nanomechanical device designs for incorporation into a test-bed instrument. Each component not only provides function but contain known uncertainties or a specific methodology to ascertain uncertainty values.

TYPICAL NANOINENTER DESIGN ISSUES
Figure 1 is a simplified diagram of a nanoindenter in which the force and measurement loops follow a common path. It can be seen that there are a number of compliances around the force loop, the most obvious being frame compliance. In theory, the frame should provide high stiffness reference for instrument and be immune to any environmental change such as temperature, humidity etc. Not only typical frame deforms with applied load but because it is usually an assembly that relies on friction and applied preloads, it will, at some level of precision, contain hysteresis. Other contributions to compliance around the force
loop include the specimen, specimen to holder interface and force and displacement sensing will introduce uncertainties that may be difficult to predict.

There is also a compliance between the specimen and positioning stage table. A worst case scenario is when it is simply placed on the table. In this case it relies on three point contact formed from peaks of roughness and form of both surfaces. The contact area most probably is based on elastic averaging in an unpredictable manner which again varies with applied load and position of the indenter tip contact. A better, and commonly used practice, is to bond the specimen to a holder with glue or hot wax which forms a more linear interface characteristic. Both methods require time for the holder to reach equilibrium in the instrument.

The positioning stage and specimen holder will also add to nonlinearities of the instrument. Most positioners do not exhibit linear behavior (i.e. constant compliance) with applied load and, because they contain actuators, generate heat that will soak through the instrument causing drift during experiments.

Temperature change effects will require instrument calibration before each experiment to linearize and predict drifts which, for extremely accurate measurements, might be neither practical nor, in some designs, possible. Wherever the metrology loop has the same path as the force loop each disturbance is directly coupled with measurement which, in most cases, cannot be extracted with predictable uncertainties.

Other than these issues that directly relate to the mechanical design of the instrument there remain a number of implementation issues that are still considered challenging for nanoindentation. Briefly, these include; prediction of areas functions for compensating indenter tip geometric effects, creating an accurate time-base for measurement processes, control stability, tip to specimen contact detection, signal to noise improvements to detect dynamic material deformation phenomena during indentation, accuracy to enable absolute parameter extraction, temperature stability and control, stiffness and force calibration, standard reference materials (particularly for softer materials) and a lack of complimentary methods to name a few.

CONCEPTUAL DESIGN
To achieve traceability each of the previously described phenomena has to be quantified. To eliminate and alleviate some of these challenges it has been necessary to design an instrument in which all significant uncertainties can be evaluated.

The principal of operation of this indenter (Figure 2) incorporates separation of force and measurement loops as far as is deemed possible. In particular, atomic force microscopy (AFM) type displacement sensing is directly referenced to the surface of the specimen. This forms a virtually rigid reference for penetration measurement of indenter tip into specimen surface while introducing force uncertainties of known value. Measurement is achieved by keeping the AFM sensor in contact with the specimen surface relatively close to indenting region but far enough that indentation induced surface deformations are not detected. Controlling its position with PZT actuator will enable maintenance of a constant force applied to the specimen surface. Ideally with sufficient sensitivity and stability of this sensing element it should be possible maintain a constant force with uncertainties being quantified from the controller error signal. By balancing between

![FIGURE 2: Block diagram of simplified current design showing main components and separation of force and metrology loops.](image-url)
contact and adhesion, positive, negative, or even zero, forces may be used as the null value.

The mutual load between the indenter tip and surface is measured via deflection of a flexure based load cell using either a sub-nanometer resolution fiber optic interferometer [6] or capacitance gauging. The indentation depth is measured as a difference between nulling sensor and load cell position measured by double differential capacitance gauges disposed symmetrically about the load cell platform, see Figure 2.

The nanoindenter head is designed to be as symmetrical and monolithic as possible to reduce uncertainties associated with assembled structures and reduce influence of thermal distortion on instrument operation. Being in the region of the instrument where force and measurement loop coincide, the goals of the indenter tip holder are to produce stiffness values of greater than $10^7$ N·m$^{-1}$ with insignificant hysteresis while enabling a relatively simple indenter replacement with the indenter tip having known orientation relative to the instrument coordinate frame.

Calibration methods will include interferometric measurement for displacement and combined interferometry and dead weight loading for force. It is envisaged that dead weight loads at nanogram levels will utilize electrolytic removal of known mass values.

**PRINCIPLE OF OPERATION**

The schematic representation of a typical measurement sequence is shown in Figure 3. In the first stage of motion the actuator 1 displaces the indenter frame 2 towards the specimen until AFM sensor 6 will detect contact with the surface. In the second stage while actuator 1 continues its motion, PZT 2 will contract to keep constant force applied from AFM sensors by following the specimen surface. In a third stage, the continued motion of the instrument brings indenter tip 4 into contact with specimen surface which is detected by load cell 3. Stage four is the actual indentation process where actuator 1 provides needed displacement, sensor 6 and servo loop 2 tracks the surface compensating for any stage, frame, instrument etc. parasitic deformation. The force is measured by load cell 3 and indentation displacement is measured by differential capacitance gauge 5.

The prototype design is shown in a Figure 4. The assigned numbers follow the same
convention from Figure 3 to enable identification of the functional components of this design. The fully symmetrical design comprises of main body with pockets for associated preamplifiers and conditioning circuitry to achieve minimized signal paths. Following the numbers: 1 is main PZT, 2 is PZT for driving servo loop around nulling mechanism 5, 3 is the load cell with differential capacitance gauge and laser fiber optics laser interferometer[6] achieving picometer resolution, 6 is an extension arm of the nulling sensor that incorporates a fine soft-hard spring mechanism for adjusting the AFM tip relative to that of the indenter, 7 is the indenter tip holder with the clamp mechanism shown in bottom right corner of Figure 4. In this mechanism a typical diamond tip is attached to the threaded shaft A which is then manually screwed into nut B. The preload is then increased by the in-built leverage of the nut B with a further load imposed by the fine thread screw C. Also an adapter is available for mounting of other types of commercially available indenters.

The instrument comprises the precision indenter head and a coarse translation stage for specimen positioning. The coarse translation stage is mounted below the indenter head and labeled xyz stage, in Figure 3. This coarse stage provides a displacement range of 150 mm in X, 100 mm in Y and 5 mm in Z to enable selection of indent location and also to position the specimen vertically to bring its surface within the 10 µm range of the indenter head.

To further improve operation and stability of the instrument a vacuum chamber has been built providing seismic and acoustic isolation from the laboratory environment and further thermal insulation to reduce temperature changes during measurement. The vacuum chamber will also provide a Faraday cage to reduce electronic noise and should further improve measurement resolution. Resolutions in the region of tens of picometers for displacement over a range of few micrometers and force measurements lower than 50 nN over a 10 mN range are expected.

Figure 5 is a greatly simplified diagram of the entire instrument. The brain of the instrument is a PXI chassis, which includes an independent microelectronic controller. Critical measurement timing and data collection are controlled by a FPGA card operating at a 40 MHz clock speed enabling event timing at these speeds and hundreds of kHz for PID control. Less critical calculations will be managed by PXI real-time with 18-bits A/D such as control of nanopositioning motor for a coarse Z displacement. All other operations such as control of vacuum system, precision XY stage and cameras are carried out using a host computer.

REFERENCES