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14. ABSTRACT

In previous AFOSR-funded (FA9550-06-0214) research, the PI's research laboratory developed new methods capable of characterizing the density of geometrically necessary dislocations (GNDs) with micrometer-scale spatial resolution over regions as large as one square millimeter. The PI has another concurrent grant (FA9550-09-1-0048)

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Final Performance Report

Award Number
FA9550-09-1-0453

Title of Project
(DURIP 09) Equipment Acquisition for the Study of Mechanical Behavior of Materials under High Temperatures and Extreme Conditions

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1 Introduction

The development of physics-based constitutive models for metals and ceramics that are able successfully to predict material behavior at high temperatures under monotonic and cyclic loading states, along with a prediction of the failure criteria and mechanisms is a critical requirement to minimize the length of the materials development cycle and also to determine safe operating parameters and lifetimes of existing systems. The engineering and scientific challenges to develop such constitutive models require extensive experimental characterization. The goal of this project was to bolster the experimental capability of the PI's laboratory at Columbia University to study the responses both metals and ceramic when subjected to high strain and high strain gradient loadings at high temperatures to support the development of a physics-based predictive capability of the fracture, fatigue, plasticity and strength properties of materials of interest to the AFOSR.

The PI's research laboratory has developed news methods that are capable of characterizing the density of geometrically necessary dislocations (GNDs) with micrometer-scale spatial resolution over regions as large as one square millimeter [1, 2, 3, 4, 5]. The method relies on inducing a plane deformation state into single crystals of a material of interest and then subsequently characterizing the plastic deformation by interrogating the rotation of the crystal lattice using Orientation Imaging Microscopy (OIM) based upon automated Electron Backscatter Diffraction (EBSD). Proper interpretation of the gradients of lattice rotation allows the rigorous lower–bound on the GND density to be determined.

An example of the capability that has been developed in the PI's laboratory is shown in Figure 1 where the lattice rotation associated with plastic deformation induced by a wedge indenter that penetrates a specially oriented single crystal of nickel so that a two–dimensional lattice rotation state is measured [5]. The gradients of lattice rotation are related to the Nye dislocation density tensor that is related, in turn, to the GND density. The goal of the experiments is to characterize the GND density on individual slip systems from such lattice rotation measurements.

Since, in general, there are more active slip systems than there are components of the Nye tensor, it can be shown that there is an infinity of possible solutions of GND densities that are consistent with the measured Nye tensor. However, the PI and co–workers have determined the lower bound solution to the total GND density that is consistent with the measured lattice rotation state. The actual GND density will be equal to or greater than the lower bound. If a Taylor–model for current yield strength—which assumes that yield strength is proportional to the square root of dislocation density—the GND lower bound can be interpreted as a lower of the local yield strength. Figure 2 shows the spatially–resolved (with a 3 micrometer spatial resolution) lower bound of total GND density associated with the lattice rotation shown in Figure 1.

2 Objective of Research

As delineated in the proposal for this DURIP grant, the objective of this research grant is to enhance the equipment available for research in the high temperature deformation of materials at high strains and high strain rates. This includes equipment necessary to

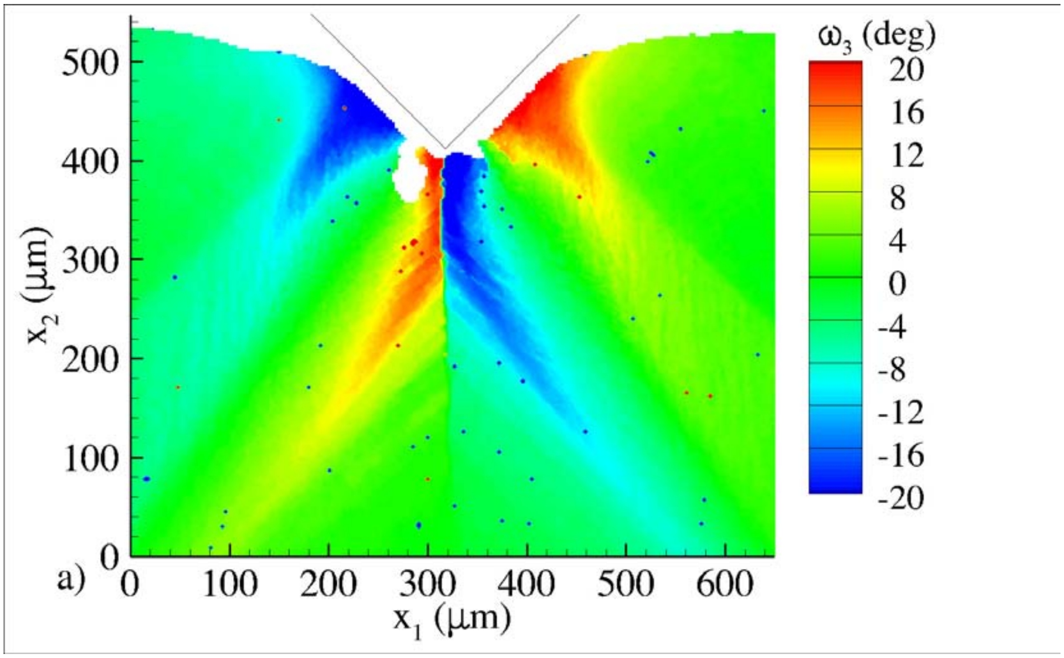

Figure 1: In–plane lattice rotation due to wedge indentation into single nickel crystal.

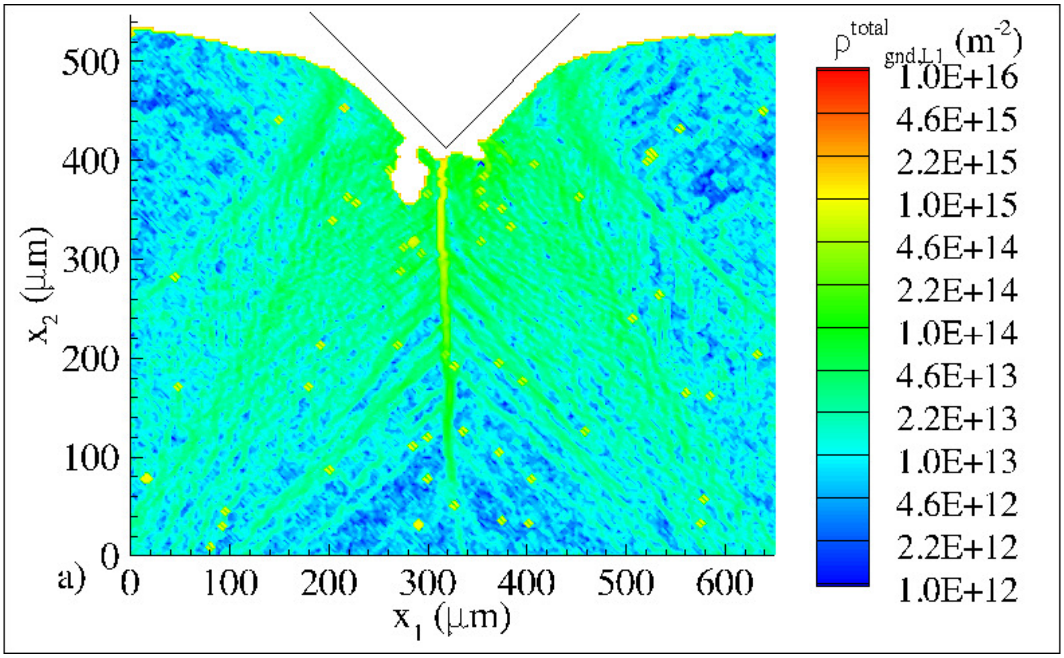

Figure 2: Lower bound on total GND density.

achieve the high temperatures as well as additional equipment for the characterization of the materials.

These objectives were accomplished by purchasing and installing all the equipment budgeted in the proposal. In addition, one additional mechanical loading testing system was obtain beyond that requested. The capabilities and performance specifications of each piece of equipment is described in the remainder of this report.

3 Equipment Purchased

The main equipment to purchase was a high temperature furnace ($> 1000°$ C) for an existing electrically-actuated Instron load frame, a high accuracy load frame to be used for subsequent testing of localized regions of material deformed at high temperatures, as well as upgrades to an existing Agilent nanoindenter to equip the device for subsequent characterization of materials deformed at high temperatures.

3.1 High Temperature Furnace

A high temperature furnace from Instron (3117-200 model with a 3117-203 M/S Controller) was purchased and mounted onto an existing servo–electric mechanical testing load frame. The furnace has three zones with a split configuration with embedded heating elements. The maximum nominal specimen temperature is 1000° C and the minimum nominal specimen temperature is is 500° C. The nominal power rating of the furnace is 3 kW with a master-slave temperature control system. In addition, a stand-alone computer to control the load frame and to measure the specimen as a function of time throughout an experiment was obtained.

A pair of reverse stress pull–rods were also purchased to allow both compression and tensile loading to be applied at the high temperatures. To facilitate compression tests, high temperature compression platens were purchased with a maximum load of 10 kN at 1000° C. A high temperature strain gauge extensometer with a 25 mm gauge length was installed. The alumina chisel end rods attach to a specimen using ceramic wrap–around cord along with a spring attachment. Compressive strains as large as -5% and tensile strains as large as 10% can be measured. A closed–loop water chiller was obtained in order to provide cooling for the furnace.

Figure 3 shows the high temperature furnace after installation on the existing Instron. The furnace in its closed configuration is shown in Figure 3a and the furnace in its split configuration showing the embedded heating elements as well as load train with high temperature platens is shown in Figure 3b.

3.2 Ultra High Precision Materials Testing System

An ultra precision and low force materials mechanical testing system that allows experiments to be performed on millimeter scale crystals was purchased as a part of the DURIP. It is a model 5848 system from Instron with a static load capacity of 2 kN and a dynamic capacity of 400 N with a displacement resolution better than 50 nm. Data can be obtained from

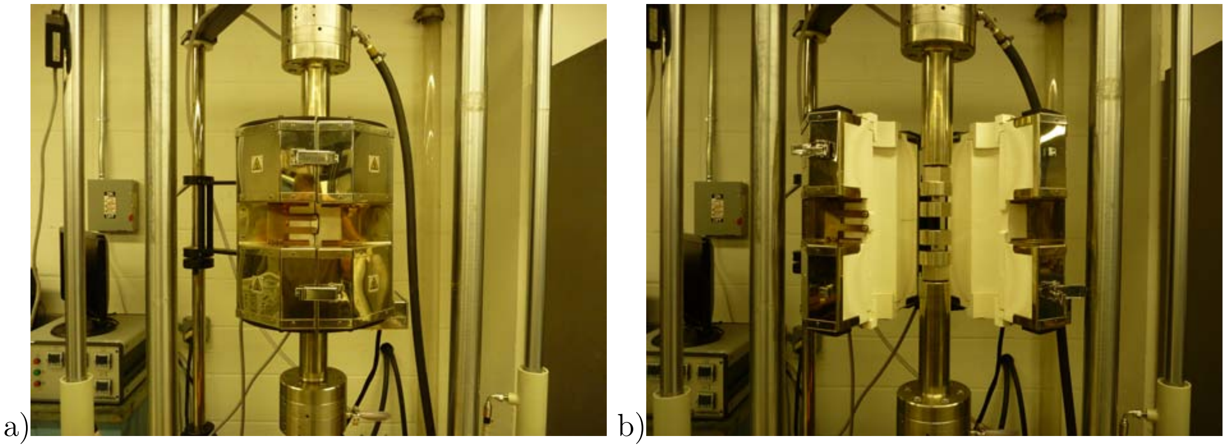

Figure 3: High temperature furnace installed onto existing Instron servo–electric mechanical testing load frame: a) Furnace in its closed configuration; b) Furnace in split configuration showing embedded heating elements as well as load train with high temperature platens.

experiments at a rate of 5 kHz and the PID controller has a 5 kHz control loop update. A pair of 50 mm diameter compression platens was purchased that has a 50 mm spherical seat compression jig to avoid introduction of a bending moment during a compression experiment. A PC computer with the BlueHill 2 Materials Testing software was obtained as well.

3.3 Upgrade to Agilent G200 Nanoindenter

Prior to receiving the DURIP grant, the PI had purchased an Agilent G200 nanoindenter (cf. Figure 5a), but without many of the available features that are necessary for the characterization for high temperature. Thus, a number of attachments for the nanoindenter were purchased using the DURIP.

The nanoindenter attachments are being used for several purposes related to high temperature materials research. Specifically, one purpose is to perform spatially–resolved maps of the hardness of the deformed material. Since the hardness of the material is related to yield stress, this ability allows the correlation between local yield stress and the total GND lower bound to be obtained. In addition, another purpose is to perform nanoindentation at intermediate temperatures of up to 350° C.

3.3.1 Dynamic Control Module

The Dynamic Control Module (DCM) attachment (cf. Figure 5b) for the nanoindenter was purchased with this DURIP. The DCM adds the critical capability to perform indentations with load resolution of $\leq 1\,\mathrm{nN}$ and a sub-Angstrom spatial resolution. Such a high resolution capability allows the PI's group to characterize the hardness of the deformed material with indentations of only a few hundred of nanometers in depth.

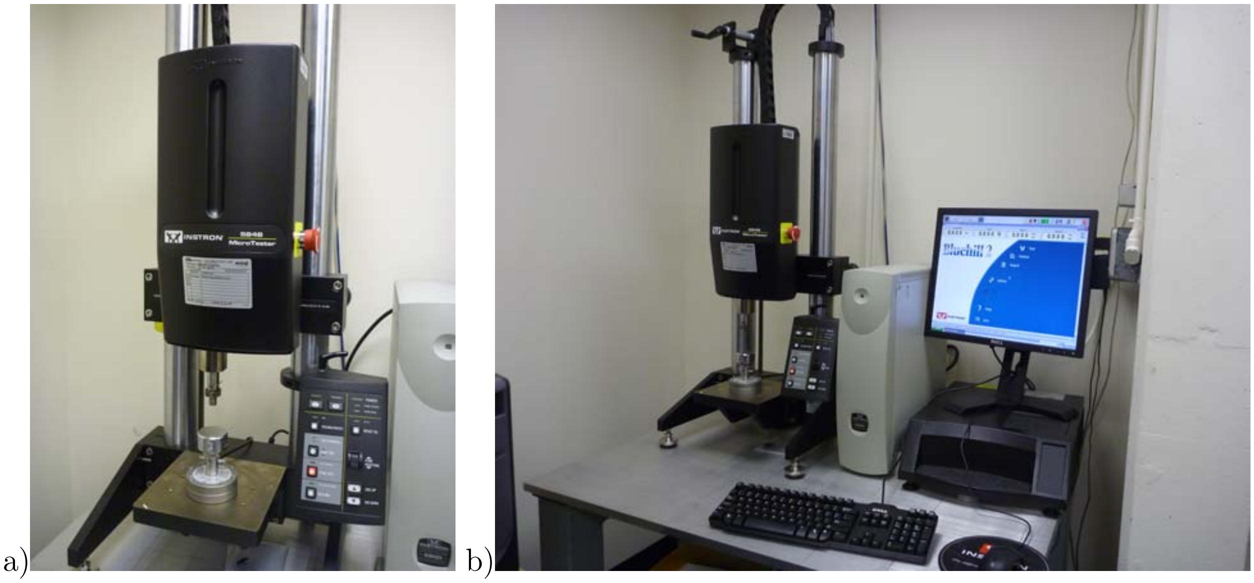

Figure 4: Model 5848 Instron: a) Close–up view shows the load frame and the lower compression platen; b) System combined with control computer after installation.

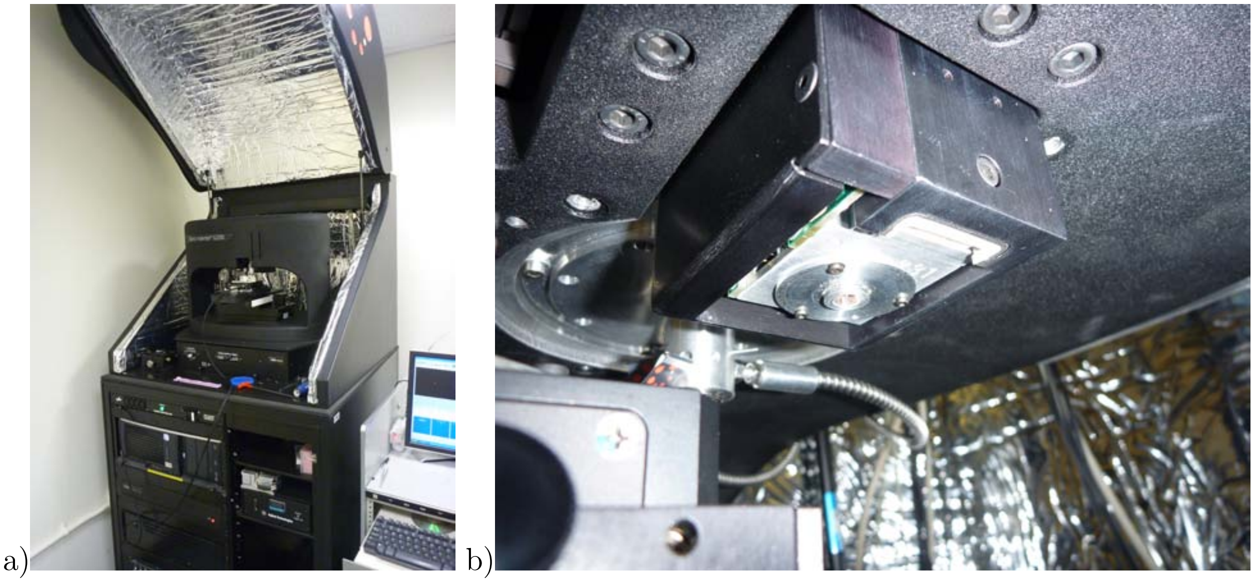

Figure 5: Model G200 Agilent Nanoindenter: a) Image of nanoindenter inside vibration and temperature isolation chamber; b) Close-up view of Dynamic Control Module.

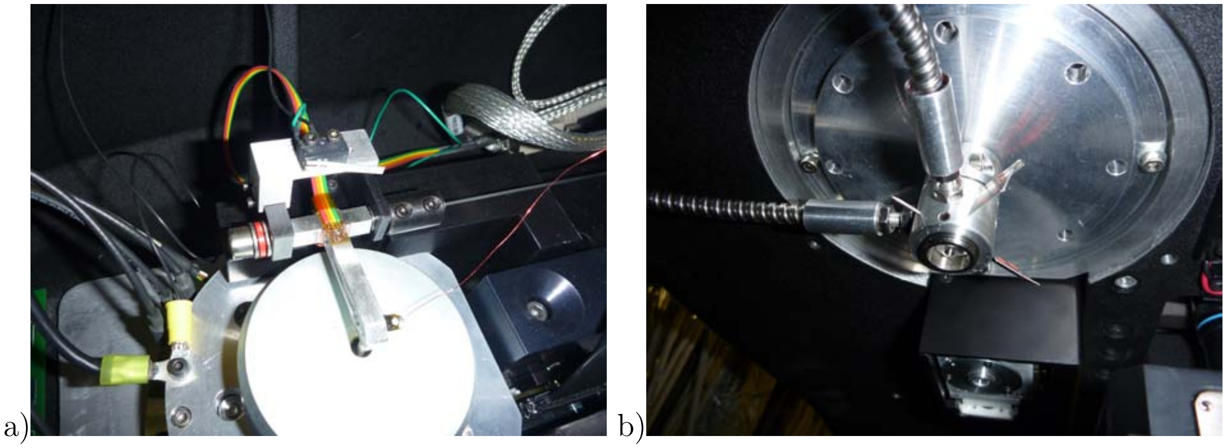

Figure 6: a) Mechanism to activate high load option; b) Details of lateral force option on G200 Agilent nanoindenter.

3.3.2 High Load and Lateral Force Options

The wedge indentation for experiments used in Figure 1 and Figure 2 has been performed using a standard mechanical loading testing frame. The indentations are approximately 200 μm in depth. In order to allow the wedge indention to be performed via a nanoindenter, a high load option for the G200 Agilent nanoindenter was purchased. The attachment, shown in Figure 6a has a maximum load of 10 N while maintaining a displacement resolution of 1 Angstrom. The maximum indentation depth is as much as 500 μm. A lateral force option was also purchased as a part of the DURIP grant; the module is shown in Figure 6b. The lateral force option has a lateral force resolution of better than 2 nN with a maximum allowable force of 250 mN.

3.3.3 Nano Vision

A nanoindenter measures the force vs. displacement response of a diamond tip as it is plunged into a material. However a traditional nanoindenter performs a measurement in one position on a material before the tip must be moved to a new location. The Nano Vision attachment implements the ability to move the tip relative to the material surface automatically so that spatially–resolved measurements of quantities of interest can be obtained. In addition, this feature allows the surface profile of a material to be measured.

A Nano Vision option was purchased for the G200 Agilent nanoindenter. It has a maximum range of travel of 100 μm in each of two in–plane directions with a feedback–controlled positioning accuracy of better than 2 nm with a resonant frequency greater than of 120 Hz. It is fully integrated into the operating software so that automatic scans can be obtained with any of the system hardware on the nanoindenter.

3.3.4 Local Heating Stage

In order to allow characterization of materials at intermediate temperatures, a local heating stage was purchased for the G200 Agilent nanoindenter. This heat stage has an operating

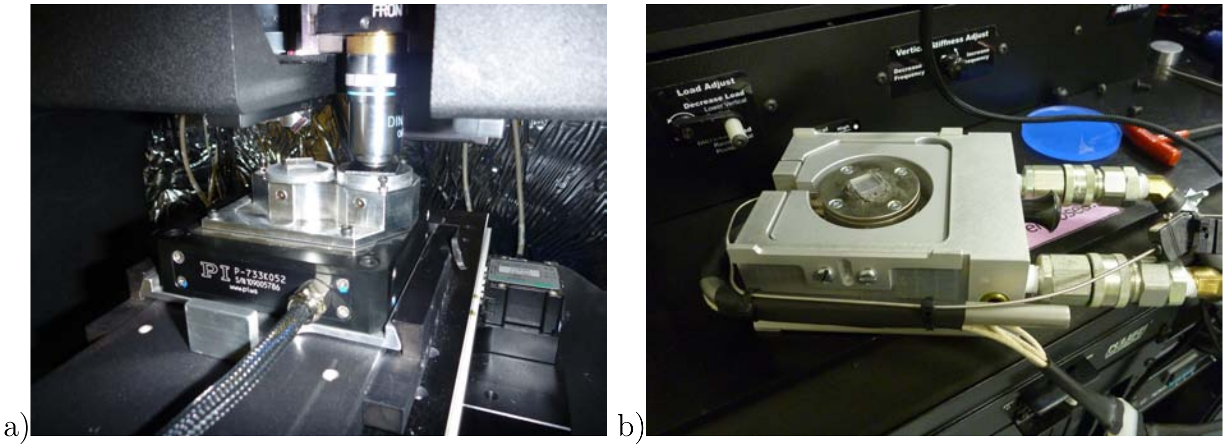

Figure 7: a) Nano Vision attachment for G200 Agilent nanoindenter to allow scanned measurement of different variables associated with nanoindentation; b) Local heating stage attachment for nanoindenter to allow measurements at temperatures up to 350° C.

temperature between ambient and 350° C. Corrosion is inhibited in the nanoindenter at these temperatures by flow of argon gas. The heat is removed via a liquid cooling system and the nanoindenter head is protected from the heat by a specially–designed shield. The heat stage, shown in Figure 7b, can be used for the characterization of plastic deformation in single crystals at elevated temperatures.

3.3.5 TestWorks 4 Software

In order to have maximum flexibility for the development of software for control of experiments, an add–on software feature called TestWorks 4 Explorer Level Software package was purchased. It allows the loading and unloading rates to be varied, the load–hold length to be specified, as well as the ability to change the order and number of experimental segments. In addition, the data acquisition rates can be modified.

3.4 Model 5569AInstron

The final major piece of equipment obtained through the DURIP grant is a model 5569A Instron, which is a table–mounted materials testing system. It is a screw–driven servo–electric system with a maximum load capacity of 50 kN with testing speeds within the range of 0.001 to 500 mm/min. The maximum rate of data acquisition is 40 kHz. A pair of wedge action grips with a capacity of 50 kN was obtained as well. Finally, the BlueHill 2 Materials Testing Software also came with the model 5569A Instron. Figure 8 shows the mechanical testing system after installation was complete.

4 Costs

The total purchases from Agilent were $194,694.60. The total purchases for the furnace and other equipment from Instron were $137,829.40.

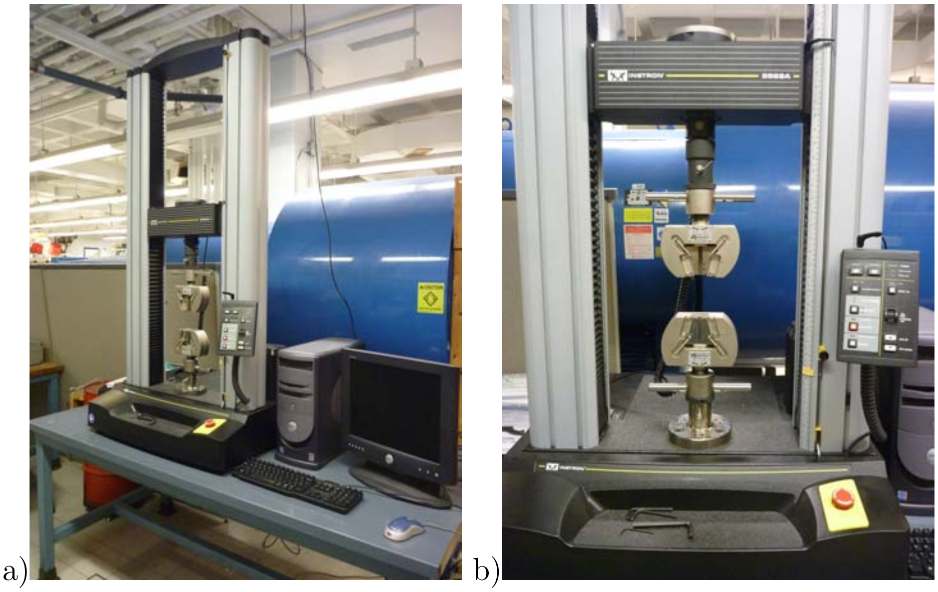

Figure 8: a) Model 5569a Instron servo–electric materials testing system: a) Overall view of materials testing system; b) Close–up view of load frame.

5 Conclusions

The DURIP grant initiated on 1 June 2009 and finished 12 months later on 31 May 2010. As per the requirements of a DURIP grant, the resources provided by the AFOSR were used entirely to purchase scientific equipment; thus no graduate students were supported. Given the short–term length of the grant, there are no publications as of the date of the submission of this final report. However several projects are currently underway which will acknowledge this grant. The experimental equipment purchased with this DURIP will be used to carry out the experimental aspects of a concurrent grant that the PI also has with the AFOSR (FA9550-09-1-0048).

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