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# A MECHANICAL LOADING/SYNCHROTRON X-RAY DIFFRACTION SYSTEM FOR IN-SITU DETERMINATION OF LATTICE STRAINS

## F49620-03-1-0322

## Final Report

## March, 2005

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## Abstract

A mechanical testing / synchrotron x-ray diffraction system was fabricated in this DURIP grant. The key elements of the system are a high frequency uniaxial loading machine, a precision diffractometer support unit and an x-ray shutter (chopper). The system will be employed within synchrotron experimental facilities to measure lattice strains during cyclic deformation experiments as a means to understand the evolution of crystal stresses.

## 1 Introduction

In this DURIP funded project, we built a diffractometer / mechanical testing machine for conducting in-situ synchrotron x-ray diffraction / mechanical testing experiments. In particular, we seek to measure lattice strains using x-ray diffraction concurrent with cyclic loading experiments to build an understanding of the evolution of crystal stresses during mechanical loading excursions like fatigue conditions. The experimental setup configured for the A2 experimental station at the Cornell High Energy Synchrotron Source (CHESS) is depicted in Figure 1. The desired x-ray wavelength (energy) is chosen using the monochromator crystals. Ion chambers measure the x-ray flux at several points and a series of slits are employed to progressively reduce the size of the beam. A linear shutter controls passage of the beam to the experiment. The rotating shutter (chopper) is used to synchronize the beam with the mechanical loading. The diffractometer positions the specimen relative to the beam and the diffracted x-rays are collected on the MAR area detector. In the sections below, we describe the design process for the components developed under the grant, give a detailed description of the system then show the initial employment of the system components and some preliminary results.

## 2 Design Objectives

There are issues related to the mechanical testing, diffraction physics and lattice strain determination components of the experiment that drove the design of the system. These are ellucidated below.
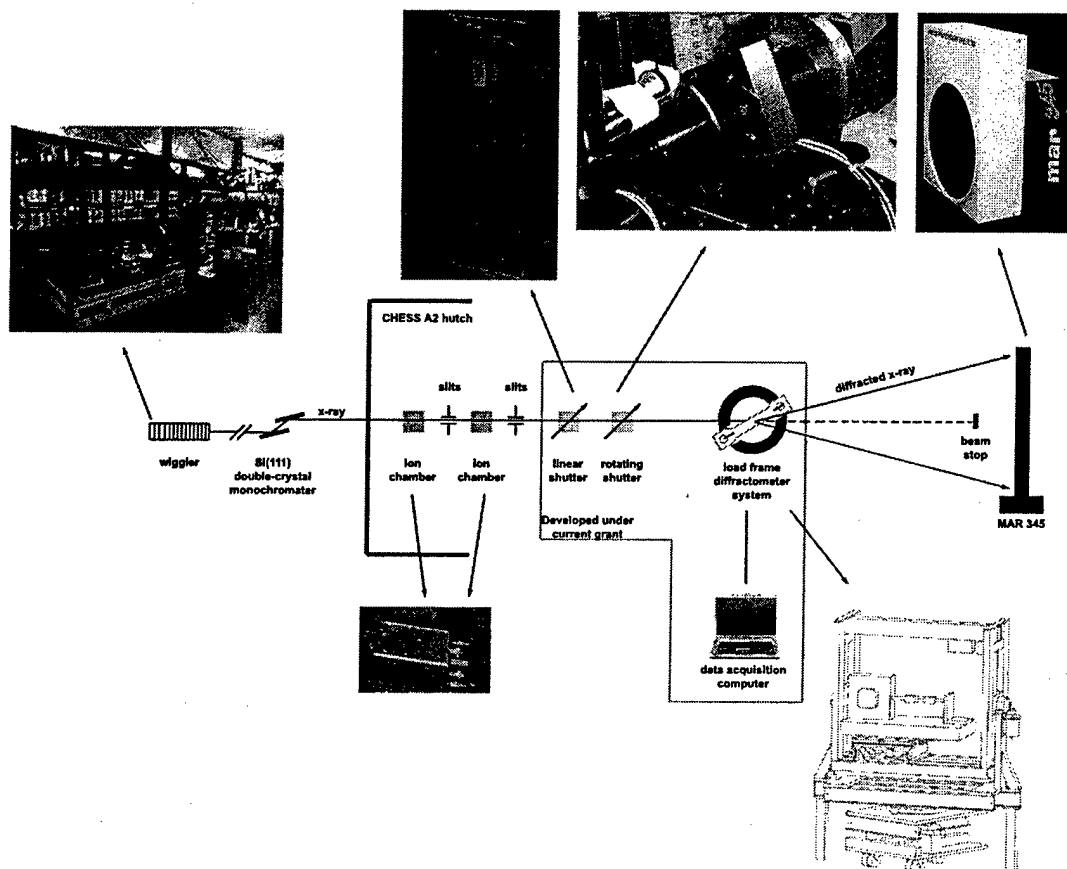
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Figure 1: Schematic of the mechanical testing / synchrotron x-ray diffraction setup configured for the A2 experimental station at CHESS

## 2.1 Mechanical Testing Fidelity

The critical link between the lattice strains (micromechanical response) and the macroscale boundary value problem is the applied load at the test machine / specimen interface. The final understanding gained hinges on our ability to quantify the specimen loading conditions at the time of the diffraction experiment. In short, we want to conduct a sound mechanical test - free from stress gradients throughout the gage section - and well controlled. To do this we must be cognizant of details like specimen alignment, transducer selection and machine control in the design of the system. In this particular case, we also sought to conduct cyclic tests in-situ within the x-ray experimental station. Because we have a limited amount of beam time, we need to run the tests at moderately high frequencies (50-100Hz) in order to complete the experiments. Finally, we need the ability to pass the direct and deflected x-rays through the specimen. This puts constraints on grip design. In summary, on the mechanical testing side of the experiments, we need:

- high frequency mechanical cycling capabilities,

- transducers for strain, load and displacement including requisite control and data acquisition capabilities for high frequency,

- specimen alignment system and

- specimen support fixtures (grips) that allow for optimal with x-ray transmission.

## 2.2 Strain Pole Figure Coverage

Our primary experimental objective in these experiments is the production of lattice strain pole figures while the specimen is loaded. In terms of the geometric configuration of the specimen relative to the incoming x-rays and detector, this goal is completely analogous to the one employed in lab-source x-ray goniometry to produce conventional orientation intensity pole figures. Instead of pole intensity, in this case, the relevant scalar field is the normal strain determined using the change in crystal lattice spacing for a particular family of planes ($\{hkl\}$). As in the lab source experiment, we seek overall coverage of the lattice strain pole figure. Bragg's law and the x-ray energy enable us to design the optimal geometric relationships between the experimental components to obtain this goal. One of the advantages of using x-rays for the diffraction experiments is the ability to employ area detectors such as the MAR detector shown in Figure 1. By rotating the specimen, "rings" of lattice strain data can be produced, eventually filling in the strain pole figure. This is depicted schematically in Figure 2. A series of rings for each $\{hkl\}$ can be produced by holding the mechanical load constant and conducting diffraction experiments at various values of $\omega$. Rotation about the specimen loading axis produces rings that extend across the pole figure in a direction orthogonal to that shown in Figure 2. By employing a combination of rotations, adequate pole figure coverage can be obtained. A main objective in the design of the diffractometer was producing the ability to rotate the sample about both the loading and transverse directions.

## 2.3 Sychronization of x-ray beam exposure and mechanical load

Along with the precise positioning and rotational degrees of freedom provided by the diffractometer, the ability to capture lattice strains in "real time" snapshots while the load is continuously cycling represents the most unique aspect of the proposed experimental capabilities of this system. By assembling these snapshots across the fatigue life of the specimen, a chronological picture of the evolution of the lattice strains - and ultimately crystal stresses - will emerge. The challenge is to synchronize the x-ray exposure with the loading cycle, which is operating at 50 -100 Hz. One manner in which this can be accomplished is by employing an x-ray shutter or "chopper" that can be synchronized with the mechanical loading cycle.

## 3 System Description

The final design consists of a combination of purchased and in-house manufactured components. The load frame / diffractometer system is shown Figure 4 along with detailed drawings of some its main components
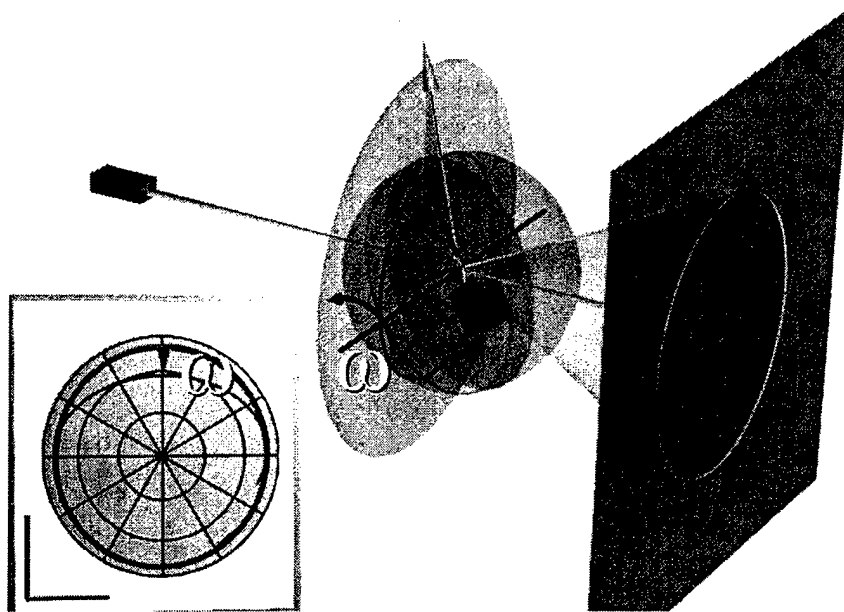
Figure 2: Schematic of the lattice strain experiment that shows the ring of lattice strain data obtained using the area detector and the manner in which additional rings of data are produced by rotating about the specimen transverse direction (omega)

## 3.1 Load Frame

Mechanical loading is accomplished using an electomagnetic actuation system manufactured by the EnduraTEC Group of the Bose corportation. The system is shown in Figure 3 The actuator has a capacity of 2000 Newtons at 100Hz and can be operated in load, displacement or strain control. Data can be acquired on all three channels at a rate of 3 kHz. A set of low profile grips were developed to accommodate x-ray transmission.

## 3.2 Diffractometer

Positioning of the load frame relative to the direct and diffracted beam is accomplished using the diffractometer system shown in Figure 4. This system, which was designed and manufactured at Cornell, consists of two rotational degrees of freedom and positioning capabilities to bring the axes of the specimen into coincidence with these rotational axes. As depicted in Figure 2, these rotations produce rings of strain data that gradually fill in the strain pole figures. The system was designed so that the center of the gage section of the specimen moves less than 10 $\mu$m at the full extent of the Y and Z rotations.
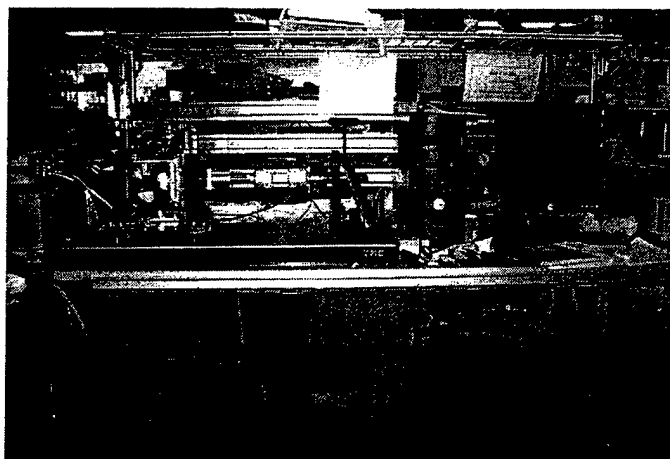
Figure 3: EnduraTEC mechanical testing system.

## 3.3 Rotating x-ray shutter - chopper

The x-ray chopper assembly is shown in Figure 1. The main component is a rotating tungsten disk with slots positioned to allow x-rays to pass twice per rotation. The size of the slots and the angular velocity of the disk dictate the x-ray exposure time. Since the load is constantly varying, we seek short exposure times. The chopper cycle is brought into coincidence with the load by rotating the motor poles.

## 4  Recent Results

The EnduraTEC load frame was employed without the chopper and diffractometer during a recent run at the A2 experimental station at the Cornell High Energy Synchrotron Source (CHESS). We ran load controlled fatigue experiments on rolled copper sheet specimens. One of the load histories is shown in Figure 5. The load frame is shown positioned within the hutch in Figure 6. Also shown in this figure is the computer controlling the fatigue experiment - which was cycling the specimen at 90 Hz. In these experiments, cycling was interrupted at prescribed numbers of cycles - the load was held - and an x-ray diffraction experiment was conducted. In this manner the evolution of lattice strain was tracked over the life of the specimen. Figure 7 shows the evolution of the lattice strain in the loading and transverse directions for several $\{hkl\}$s in a specimen subjected to the loading depicted in Figure 5. It is difficult to draw too many conclusions from these results but we see definite changes as the specimen nears its fatigue life.

## 5  Summary

The nature of material characterization and property determination experiments needs to change to keep pace with the level of detail now possible within simulations. More importantly, a new generation of experiments is necessary to validate and calibrate material models that explicitly represent internal structures and their evolution. It is no longer acceptable to only match stress-strain curves to validate material models. Quan-
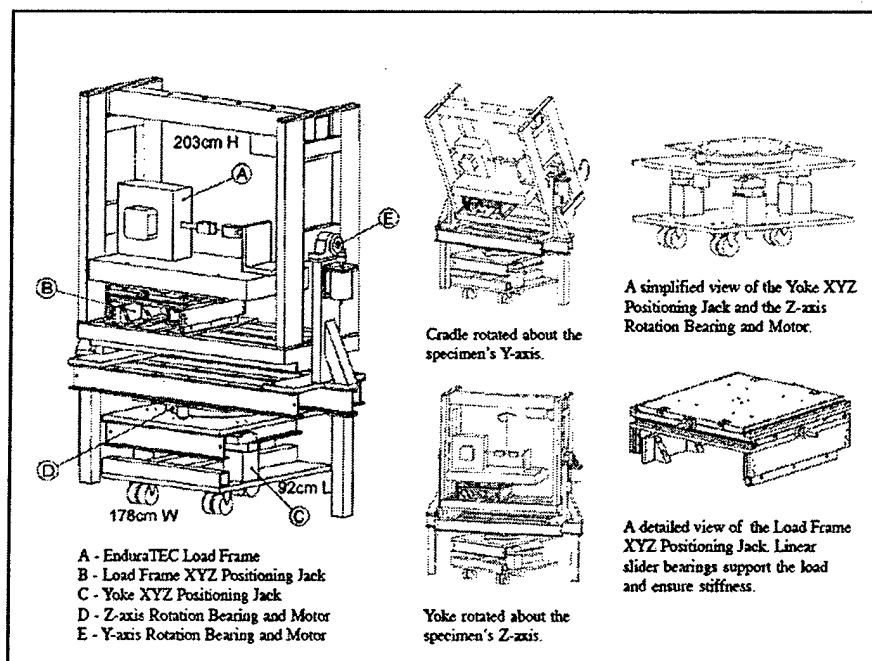
Figure 4: Load frame / diffractometer system showing rotational capabilities and associated subsystems.

titative characterization experiments - ones that actually measure relevant structures, their attributes and distributions, preferably in real time during thermomechanical loading episodes - can be used to understand physical processes and can provide vital information for qualification of structure-based material models. We have fabricated an experimental system that will enable us to measure the evolution of lattice strains *during* loading excursions. In particular, we will be able to employ this system concurrently with synchrotron x-ray diffraction to measure lattice strains (and determine crystal stresses) in real time during cyclic loading histories. To our knowledge, these experiments will be unique. It is our belief that the knowledge gained from these tests will directly contribute to our understanding of material state at the instant a fatigue crack initiates.

## 6  Publications

Since the system is just coming on line, no publications have come out of the work. However, the data from Figure 5 has been presented at two conferences.

## 7  Personnel

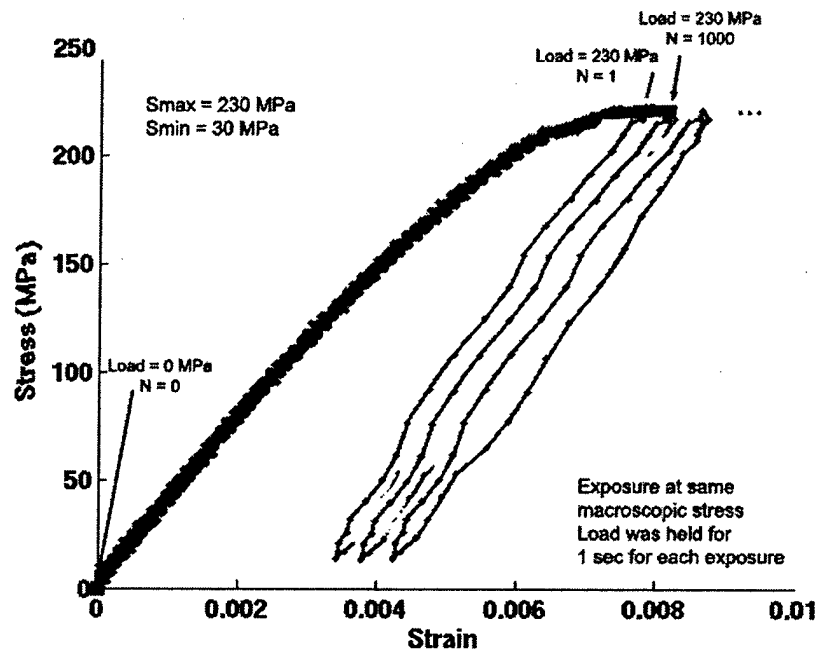This is an equipment grant. No personnel were supported.

Figure 5: Cyclic stress-strain history from a recent experiment at CHESS.
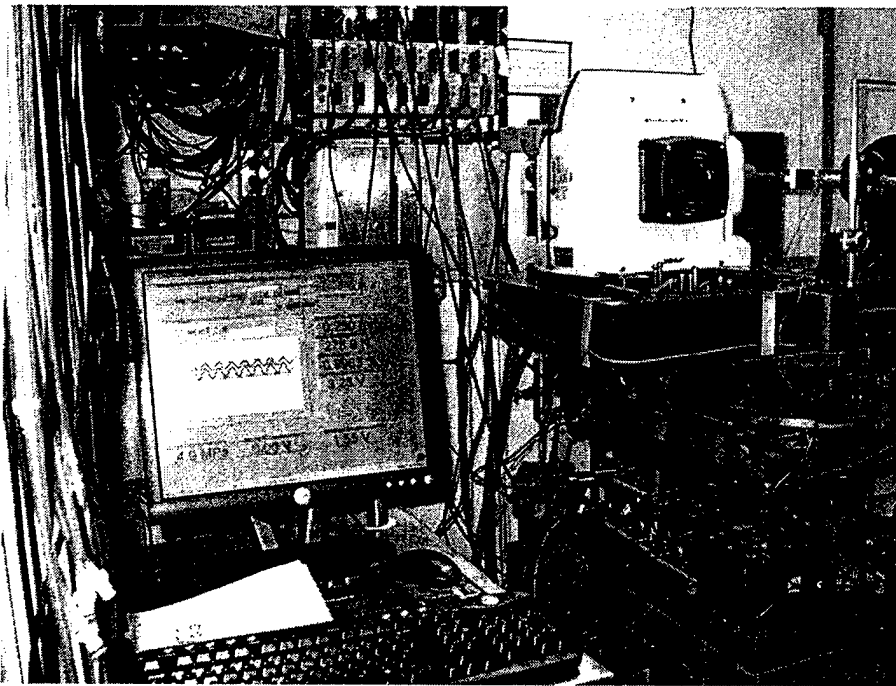


Figure 6: EnduraTEC load frame shown positioned within the A2 experimental station at CHESS.
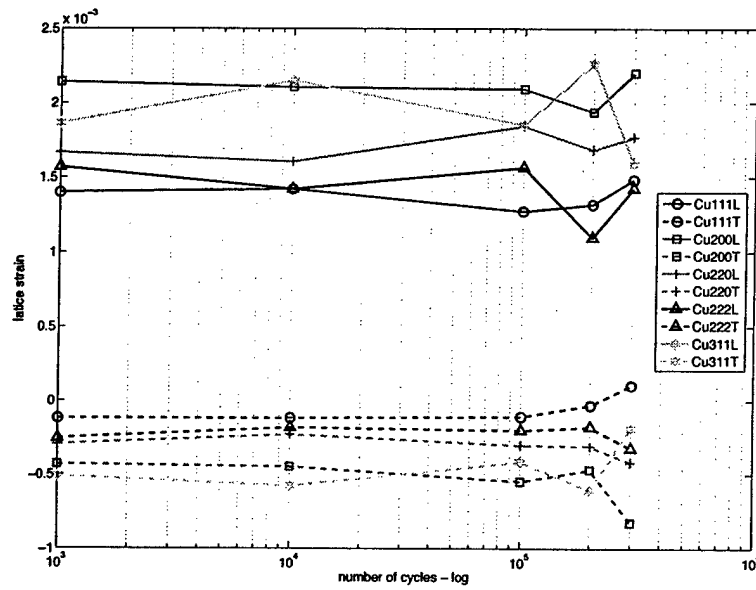
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Figure 7: Lattice strain in the loading and transverse specimen directions as a function of cycle number for the specimen subjected to the loading histories in Figure 5.