An experimental system for high temperature X-ray diffraction studies with *in situ* mechanical loading

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An experimental system with *in situ* thermomechanical loading has been developed to enable high energy synchrotron x-ray diffraction studies of crystalline materials. The system applies and maintains loads of up to 2250 N in uniaxial tension or compression at a frequency of up to 100 Hz. The furnace heats the specimen uniformly up to a maximum temperature of 1200 °C in a variety of atmospheres (oxidizing, inert, reducing) that, combined with *in situ* mechanical loading, can be used to mimic processing and operating conditions of engineering components. The loaded specimen is reoriented with respect to the incident beam of x-rays using two rotational axes to increase the number of crystal orientations interrogated. The system was used at the Cornell High Energy Synchrotron Source to conduct experiments on single crystal silicon and polycrystalline Low Solvus High Refractory nickel-based superalloy. The data from these experiments provide new insights into how stresses evolve at the crystal scale during thermomechanical loading and complement the development of high-fidelity material models. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4793230>]

I. INTRODUCTION

Diffraction methods can be used to investigate the micromechanical state of bulk crystalline specimens. When these studies are conducted with thermal and/or mechanical loads applied *in situ*, processing and operating conditions can be mimicked and the material response can be probed in real time.^{1–5} These experiments paint a clearer picture of the microscale material response than more traditional diffraction-based residual stress studies which provide aggregate averaged lattice strain information, often near the surface of the sample.⁶ Lattice strain data measured in these experiments can be used to both validate and calibrate predictive, high fidelity material models.^{1,2} With data measured during both *in situ* mechanical and thermal loading, these models may eventually help build a more complete understanding of creep, fatigue, and the influence of each on microcrack initiation.

To conduct diffraction experiments on bulk crystalline specimens, neutrons and x-rays are typically employed. Neutrons interact predominantly with nuclei and thus undergo much less attenuation when compared to x-rays of similar energy, making neutron diffraction the ideal means of nondestructively measuring strain in large specimens and engineering components.⁷ However, neutron fluxes are significantly lower than those for x-rays, increasing data collection time.⁸ This is significant, particularly in lattice strain studies of polycrystalline materials, as diffracted intensity needs to be measured in many directions to determine the lattice strain tensor at each crystal orientation. For sample thicknesses on the order of millimeters, high energy x-rays from a synchrotron source form an effective measurement technique due to the penetrating power and high flux they afford.

The experimental system presented here was designed with such x-ray diffraction experiments in mind. The system

is capable of applying thermomechanical loading and reorienting specimens using two rotational axes to measure specimen response in many directions. The system has been used at the A2 experimental endstation at the Cornell High Energy Synchrotron (CHESS) to enable the interrogation of crystal scale material response using high energy x-ray diffraction. In the following, brief details on x-ray diffraction measurements are provided followed by the design requirements of the system, organized into two functions: thermomechanical loading and specimen reorientation. Next, the system design is fully detailed, with each function forming a section. Finally, a description of the experimental setup at CHESS is included, along with examples of data obtained.

II. X-RAY DIFFRACTION MEASUREMENTS

X-ray diffraction can be used to determine the spacing between lattice planes in a crystalline material. With loading applied *in situ*, changes in lattice plane spacing, and thus lattice strain, can be measured. A graphical representation of the diffraction geometry is shown in Fig. 1. The scattering vector, $\mathbf{s}$, is defined as the difference between the vectors representing the incident and diffracted x-rays. The diffraction condition is given by the Laue equations:

$$\mathbf{a}_1 \cdot \mathbf{s} = h, \quad (1a)$$

$$\mathbf{a}_2 \cdot \mathbf{s} = k, \quad (1b)$$

$$\mathbf{a}_3 \cdot \mathbf{s} = l, \quad (1c)$$

where $\mathbf{a}_1$, $\mathbf{a}_2$, and $\mathbf{a}_3$ are the crystal unit cell basis vectors, and h , k , and l are integers. Details of the derivation can be found in Refs. 8 and 9. The vector nature of these equations and the constraints on the scattering vector due to elastic scattering indicate that diffraction occurs when the crystal is oriented in

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FIG. 1. Schematic of transmission diffraction geometry.

specific configurations with respect to a monochromatic beam of x-rays. Additionally, the integers h , k , and l are related to the set of lattice planes denoted by $\{hkl\}$, which satisfy the Laue condition when plane normal $\mathbf{c}$ is parallel to $\mathbf{s}$. The Laue condition is equivalent to the more familiar Bragg's law:

$$\lambda = 2d_{\mathbf{c}||\mathbf{s}} \sin \theta_{\mathbf{c}||\mathbf{s}}, \quad (2)$$

where λ is the x-ray wavelength, $d_{\mathbf{c}||\mathbf{s}}$ is the lattice plane spacing, and $2\theta_{\mathbf{c}||\mathbf{s}}$ is the angle between the transmitted and diffracted x-rays.⁸ The subscript $\mathbf{c}||\mathbf{s}$ denotes a diffraction measurement taken from the family of lattice planes with a normal $\mathbf{c}$, parallel to the measured scattering vector $\mathbf{s}$ (i.e., satisfying the Laue condition). In properly configured experiments on polycrystalline aggregates containing crystals of all orientations, the diffraction condition is always satisfied. Each $\{hkl\}$ forms a cone of diffracted x-rays, and rings of diffracted x-ray intensity can be collected on an area detector as shown in Fig. 1. In a single crystal experiment, planes satisfying Eq. (2) are fewer in number and the diffracted intensity form individual peaks (spots) on the detector.

The normal lattice strain component, $\epsilon_{\mathbf{c}||\mathbf{s}}$, for a particular scattering vector $\mathbf{s}$ is defined as

$$\epsilon_{\mathbf{c}||\mathbf{s}} = \frac{d_{\mathbf{c}||\mathbf{s}} - d_{\mathbf{c}||\mathbf{s}}^0}{d_{\mathbf{c}||\mathbf{s}}^0}, \quad (3)$$

where $d_{\mathbf{c}||\mathbf{s}}^0$ is the reference plane spacing. By reorienting the specimen with respect to the x-rays, a large number of unique strain measurements are made (each with a distinct $\mathbf{c}||\mathbf{s}$ combination). In experiments on polycrystalline aggregates, these measurements can be used to construct lattice Strain Pole Figures (SPFs) for each measured $\{hkl\}$ by plotting each strain value at the intersection of the respective scattering vector and a reference sphere.² The lattice strains measured during a single crystal experiment can be used to determine the complete strain state in the specimen.

Thus, these experiments are viable for probing the crystal scale material response during *in situ* thermomechanical loading. As useful examples, the authors recommend Refs. 1, 2, 7, 10, 11, and 12.

III. DESIGN REQUIREMENTS

The new experimental system was designed for use at the A2 experimental station at CHESS. The primary con-

straint on the design was that of x-ray diffraction: no part of the system can interfere with either the incident or diffracted x-ray beams. The value of $2\theta_{\mathbf{c}||\mathbf{s}}$ shown in Fig. 1 has a maximum value of 15° : a value based on the detector size ($410 \text{ mm} \times 410 \text{ mm}$) and the typical working distance in the A2 station of 700 mm. This distance was selected to balance lattice strain resolution and the number of $\{hkl\}$ s sampled. This $2\theta_{\mathbf{c}||\mathbf{s}}$ value translates to a 30° cone of diffracted intensity with its apex at the sample position, effectively forming a “dead zone”: any highly absorbing material that enters this cone obstructs the diffracted x-ray beams. This constraint significantly affected the design of both functions of the system: thermomechanical loading and specimen reorientation. The design requirements associated with each function are listed below.

(i) **Thermomechanical Loading:** The mechanical loading component of the system (the loadframe) should uniaxially load the specimen, often beyond the yield strength of the study material, while reorienting the specimen and without interfering with the heating process, disrupting the x-ray diffraction experiment, or buckling the specimen. The system should have the ability to apply both monotonic and cyclic loading in tension or compression. To most simply test compression specimens using platens, a vertical loading direction is required, as shown in Fig. 2. The furnace component of the system should heat the specimen uniformly while permitting loading and reorientation of the specimen, and without blocking the x-rays. The system should accommodate a variety of atmospheres including oxidizing, inert, and reducing so as to mimic processing and operating conditions. To prevent sensitive components on the loadframe from experiencing high temperatures, the furnace component also should include an adequate cooling system.

(ii) **Specimen Reorientation:** To make a sufficient number of diffraction measurements, the system should be able to reorient the specimen about two of its axes as depicted in Fig. 2, by a maximum of -60° to $+60^\circ$ (ω) and -5° to $+5^\circ$

FIG. 2. Schematic indicating the specimen loading and rotational axes.

FIG. 3. Schematic of the new system, indicating coordinate systems and rotational axes. The (X, Y, Z) coordinate system is defined by the gross translation stage and enables alignment of the system relative to the incident x-rays. The (X', Y', Z') coordinate system is defined by the roller bearing and dovetail translation stages and enables alignment of the specimen relative to the Z and X' rotational axes, respectively. The (x, y, z) coordinate system is defined by the test specimen. The machine is positioned such that x-rays travel out of the page (Y axis), through the specimen location at the center of the furnace.

(χ). The distance between the intersection of the two rotational axes and the centroid of the specimen should be minimized, and this position should remain aligned relative to the x-rays during all rotations.

The experimental system was designed to satisfy the above constraints and objectives. A schematic of the complete design is shown in Fig. 3. Sections IV and V detail the design of each function, respectively: thermomechanical loading and specimen reorientation.

IV. THERMOMECHANICAL LOADING

A. Mechanical loading

The primary objective of the mechanical loading setup is to uniaxially deform the sample in tension or compression. To accomplish this, a loadframe (composed of a load cell and electromagnetic actuator shown in Fig. 3) capable of applying and maintaining loads up to 2250 N in tension or compression at a frequency of up to 100 Hz is used. A linear variable differential transformer (LVDT) monitors the grip displacement while a piezoelectric transducer monitors the applied load. Due to the sensitivity of lattice strain measurements to applied loading, load control is often used to ensure the desired load is maintained while measurements are made.

To ensure the load applied to the sample is uniaxial, the misalignment between the axes formed by the actuator and load cell is first quantified, then minimized. A thin sample with two strain gages on each side is placed in the grips of the loadframe, and the gages monitored while mounting slots

A. Gross translation stage: allows for translation in three orthogonal directions (X, Y, Z) to align the system relative to the incident x-rays

B. Rotation stage: actuator for rotation about the Z axis (ω)

C. Roller bearing translation stage: allows for translation in two orthogonal directions (X', Y') to align the sample relative to Z

D. Base plate: supports the rotation/loadframe assembly

E. Stepper motor: actuator for rotation about the X' axis (χ)

F. Right angle gearbox (not visible): transfers rotation from the stepper motor (E) to the driveshaft (G)

G. Drive shaft: enables rotation of the loadframe about X'

H. Pillow block bearing: supports the drive shaft

I. Dovetail translation stage: allows for translation in two orthogonal directions (Y', Z') to align the specimen relative to X'

J. Box beam: supports the loadframe and furnace (N)

K. Actuator: capable of applying loads up to ± 2250 N

L. Furnace translation stage: allows for alignment of the furnace relative to the x-rays

M. Furnace stepper motor: provides counter rotation about Z to ensure the furnace doesn't intersect with the x-rays

N. Furnace: heats the specimen to temperatures up to 1200 °C

O. Load cell: together with the actuator (K) forms the loadframe

in the actuator and load cell are adjusted (changing the positions of the ends of the specimen) until the strain readings are minimized.

1. Deforming the sample

Two separate specimen experimental configurations are used: one for specimens loaded in compression only and one for specimens loaded in tension and/or compression. For the compression loading configuration, two cylindrical platens are mounted in the grips of the actuator and load cell, and are used to transfer load to the specimen. Yttria-stabilized Tetragonal Zirconia Polycrystal (Y-TZP) was selected as the platen material for its low thermal conductivity and high modulus of elasticity: useful in preventing heat loss from the furnace and in increasing the buckling load, respectively.

Specimens undergoing both tensile and compressive loading are inserted into “pushrods” mounted within the grips which serve to hold the specimen in place inside the furnace. Low Solvus High Refractory (LSHR) nickel-based superalloy was chosen as the pushrod material for its high yield strength at the relevant temperatures. Both pushrods have a center bore feature which contains a “compressive rod” made of LSHR (refer to Fig. 4). The top section of each hole is tapped to allow two set screws to load the compressive rods which in turn compress both ends of the specimen against the pushrods, ensuring that no relative specimen movement takes place when the load transitions from tension to compression.

FIG. 4. Schematic depicting the tension/compression loading configuration, with the top pushrod shown semi-transparent.

2. Specimen design

Drawings of both the compression and tension/compression specimens are shown in Fig. 5. The specimen dimensions represent the optimization of four design objectives: (i) to create sufficient uniaxial stress to yield the material with the given loadframe, placing an upper bound on cross sectional area of the specimen; (ii) to ensure the platens/pushrods do not block the diffracted x-ray beams, placing a lower bound on the height and cross sectional area of the specimen; (iii) to ensure neither the platens/pushrods nor the specimen buckle, placing an upper bound on the specimen height and a lower bound on the cross sectional area of the specimen; and (iv) to conduct x-ray diffraction experiments in transmission.

The relatively tall specimens allow diffracted x-ray beams to clear the platens/pushrods while the small cross sectional areas allow both the specimens to yield without buckling and the x-rays to penetrate the specimens (using 50 keV x-rays). Each of these design objectives must be considered before conducting experiments on a new material of interest.

B. Specimen heating

Conducting experiments at elevated temperatures was one of the most important design requirements for the new system. The heating assembly used to accomplish this consists of two sub-assemblies: a custom furnace and a separate “environment assembly” which controls the specimen environment. Details on the design of both the furnace and specimen environment assembly are presented below.

1. Furnace design

The furnace utilizes radiative heating in the form of a custom resistance heater. As illustrated in Fig. 6, the resistance heater is composed of an alumina cylinder (hereafter referred to as the “core”) with resistance wire, made from a FeCrAl alloy, wrapped around grooves on the exterior of the core.

The specimen is located at the center of the furnace core, although the specimen, platens/pushrods, and environment assembly are not directly connected to the furnace. As a result, the specimen can be rotated about Z while the furnace remains fixed relative to the x-rays. This allows the incident x-rays to pass through a small inlet hole in the core and diffracted

FIG. 5. Specimen drawings with the dimensions in millimeters for (a) compression specimens and (b) tension/compression specimens.

FIG. 6. Cross sectional view of the furnace.

x-rays, at a maximum $2\theta_{\text{c||s}}$ of 20° , through a conical outlet hole (refer to Fig. 6 for a cross sectional view of the core and to Fig. 1 for the beam geometry).

To isolate the core thermally, it is surrounded by alumina-based cement and placed within alumina-silica insulation, a material which does not add appreciably to the background of the diffraction pattern. The alumina-silica insulation is held within a stainless steel cylinder with water cooling caps attached above and below, as depicted in Fig. 6, which serve to further contain heat from the core. The design of the caps includes a labyrinth and pockmarks on the base to allow the fluid to reach turbulence quicker.

To monitor the temperature at critical points in the system, Type R (Pt 13% Rh/Pt) bare wire thermocouples, 0.25 mm in diameter, are used for their accuracy at high temperatures, fast response, and minimal heat loss. One control thermocouple is located adjacent to the sample, and four safety thermocouples are located at two points on the exterior of the core and two points on the grips. A microprocessor-based temperature controller uses the control thermocouple as input to turn on and off a solid state relay (calibrated to $\pm 2^\circ\text{C}$). This relay is connects to a variable autotransformer which serves as the power supply for the heater.

2. Specimen environment assembly

As discussed in Sec. III, it is necessary to control the specimen environment and accommodate oxidizing, inert, and reducing atmospheres. To contain the specimen environment, a fused quartz tube is used as shown in Fig. 7. Amorphous fused quartz was chosen for its low coefficient of thermal expansion, low thermal conductivity, chemical stability, and low x-ray absorption at high energy.¹³ This combination of properties is ideal for minimizing heat loss from the furnace, enabling a range of specimen atmospheres, and minimizing x-ray intensity losses.

As the specimen is not connected to the furnace, the environment assembly attaches directly to the platens/pushrods which pass through the center of the furnace. To accomplish this, the fused quartz tube seals onto a pair of stainless steel bellows attached to the platens/pushrods. Mating of the fused quartz to each bellows is achieved using a screw-on cap

FIG. 7. Schematic of the specimen environment assembly with a tension/compression specimen (bottom components are shown semi-transparent).

that applies axial force to a high temperature O-ring which sits in a groove beneath the bellows. This axial force compresses the O-rings onto grooves in the fused quartz tube to attach the tube to the bellows and to seal the environment. Split clamps are used to seal a flange on the bellows to the platens/pushrods, as shown in Fig. 7.

To prevent heat from the furnace from reaching the load cell or the actuator, the gas used for the specimen atmosphere doubles as a cooling system by reducing the temperature of the ends of the platens/pushrods. To accomplish this, each bellows assembly has an inlet that allows the chosen gas or combination of gases to enter the system from the storage tank, flow across the ends of the platens/pushrods and out through an outlet connected to a relief valve, as indicated in Fig. 7.

V. SPECIMEN REORIENTATION

As discussed in Sec. III, the specimen needs to be reoriented with respect to the x-rays using two rotational axes in order to measure diffracted intensity in many directions. For our purposes, reorientation is defined as rotation of the specimen that produces a minimal amount of translation of the specimen centroid. To create this motion precisely, three degrees of freedom per rotational axis are required: one rotational axis in the form of a rotary actuator and two orthogonal, translational axes for alignment, in the form of a two-axis translation stage. Therefore, the reorientation design function can be further resolved into two functions: rotation and alignment, as detailed below.

A. Rotation

To measure the diffracted intensity of a sufficient number of crystal planes in most materials, a maximum rotation of

$\omega = \pm 60^\circ$ and $\chi = \pm 5^\circ$ is required (refer to Fig. 2 for a depiction of each rotation). The set of crystal orientations of a polycrystalline aggregate sampled by each measurement is described in detail in Ref. 1. In Fig. 3, the system is shown at the zero point of both rotations, with the Z axis (concentric with the rotation stage) defining ω rotation, and the X' axis (collinear with the drive shaft) defining χ rotation.

To rotate the specimen (and all components on the base plate in Fig. 3) 120° in ω about Z, a single axis rotation stage is used. Lead blocks are placed on the base plate to ensure the rotation stage experiences a uniformly distributed load.

The assembly used to rotate the specimen (and all components attached to the box beam in Fig. 3) 10° in χ about X' is composed of a stepper motor and a 100 to 1 right angle gearbox. A custom drive shaft is connected to the gearbox and supported by two tapered roller bearing housed units, then attached to the dovetail translation stage and box beam (see Fig. 3). As the actuator weighs 34 kg, a counterweight is attached to the top of the box beam. The counterweight is of appropriate weight and distance from the rotational axis to minimize the torque that the gearbox and stepper motor would otherwise experience.

1. Furnace rotation

As described in Sec. IV B, the furnace is mounted separately to allow only the specimen and environment to rotate up to 120° in ω with respect to the x-rays. To prevent the furnace from obstructing the x-rays, it is rotated about z (specimen axis) counter to the ω rotation and is attached to the box beam, allowing it to also rotate about X'. Refer to Fig. 6 for a cross sectional view of the furnace and Fig. 8 for a schematic of the furnace rotation.

To counter rotate about z, the furnace is supported by two bearings and connected to a spur gear driven by a dedicated

FIG. 8. Schematic of furnace rotation. Components shown in gray (the furnace and gear) counter rotate in ω while all components rotate in χ .

A. Mount to box beam
B. Furnace translation stage
C. Furnace stepper motor
D. Pushrod
E. Furnace gear
F. Furnace
G. Temperature controller

FIG. 9. Picture of the system after ω rotation: the stepper motor counter rotated the furnace about z to ensure it remained aligned with respect to the x-rays. The incident and transmitted beams are shown in green, refer to Fig. 1 for a schematic.

stepper motor, depicted in both Figs. 8 and 9. The motor parameters of the rotation stage and furnace stepper motor are configured such that each rotates exactly opposite to the other, at the same speed. A graphite/metal alloy was selected as the bearing material for the furnace as it is a self-lubricating alloy that functions well at high temperatures and low angular speeds.

B. Alignment

Alignment is required for each rotational axis so as to minimize the region traced by the centroid of the specimen during rotations: referred to as the “sphere of confusion” in diffraction experiments. Inability to properly align the system prior to an experiment can introduce error into measurements and significantly complicate the data reduction process. Two separate alignment configurations are used, defined as the system alignment and the specimen alignment. The former aligns the specimen centroid with the intersection of Z and X', and aligns this position with respect to the x-rays. This system alignment is typically conducted preceding synchrotron beamtime. The specimen alignment

FIG. 10. Specimen alignment setup for (a) compression specimens (shown semi-transparent) and (b) tension/compression specimens.

ensures that the specimen centroid is aligned relative to the loading axis, and is used prior to each experiment.

1. System alignment

To align the system with respect to the x-rays, distance is minimized both between the specimen centroid and each rotational axis, and between the specimen centroid and the beam of incident x-rays. The furnace is then positioned relative to the beam, such that x-rays pass through both the furnace inlet and outlet during all rotations (refer to Fig. 6). The (X, Y, Z) coordinate system enables translation of the system with respect to the incident x-rays and the (X', Y', Z') coordinate system enables translation to align the specimen with respect to each rotational axis. A depiction of these coordinate systems and further detail can be found in Fig. 3.

2. Specimen alignment

To further reduce the diameter of the sphere of confusion, care is taken to align the specimen centroid with respect to the loading axis (parallel to z). For compression specimens, soft, elastic alignment caps with cutouts matching the specimen geometry were created to fit over the top of the platens (depicted in part (a) of Fig. 10). The sample is placed within this cutout to align it with respect to the loading axis. The caps are not removed prior to the experiment as resistance from the caps to Poisson expansion in the sample is negligible. To align tension/compression samples, a collar fits over both pushrods and the sample only when they are aligned, as shown in part (b) of Fig. 10. Once alignment has been achieved, the compressive rods are tightened down, the collar is removed, and the pushrod/sample assembly is loaded into the system.

The sphere of confusion of the system is approximately 50 μm in diameter after both system and specimen alignment—sufficient to ensure the portion of the specimen near the centroid, far from the loading surfaces, is located at the center of x-ray flux during typical studies on bulk single crystal specimens and polycrystalline aggregates (using a 200 μm –400 μm beam of square cross section). This value serves as the lower bound on allowable beam sizes when mon-

itoring the response, during rotations, of a single embedded grain in an aggregate.

VI. APPLICATION

The system described above, capable of use at any synchrotron radiation facility, has thus far been used at the A2 experimental endstation at CHESS to interrogate single crystal silicon and polycrystalline LSHR nickel-based superalloy: a fcc alloy developed by NASA for high temperature turbine disks. Silicon specimens were monotonically compressed and heated in a nitrogen environment while diffraction measurements were made. These experiments were primarily conducted to develop a methodology for monitoring microscale deformation processes in single crystals. In studies on LSHR, specimens were subjected to fully reversed loading cycles and heated in an oxidizing environment while diffraction measurements were made. These experiments help to reveal a more complete picture of the stress state in LSHR at high temperature and complement previous work that focused on measuring lattice strain pole figures at room temperature.^{1,2} The following sections detail the experimental conditions at the A2 beamline and the procedure for conducting an experiment, along with examples of measured data.

A. Experimental conditions

The Cornell Electron Storage Ring (CESR) provides a 5.3 GeV beam of electrons which pass through a 49 pole wiggler, producing x-rays for the A2 beamline. The full beam path is depicted schematically in Fig. 11.^{1,14} An x-ray energy of 50 keV is selected, using a double-bounce silicon monochromator, from the wide range of energies emanating from the wiggler. The desired beam cross section, a 200 μm –400 μm square, is obtained using two sets of tungsten slits. To control the specimens exposure time to the beam and to monitor the x-ray flux, a linear shutter and a pair of ion chambers, respectively, are used. After entering the experimental station, the beam passes through a helium-filled flight chamber and a collimator (a lead cylinder) to minimize the amount of spurious x-rays. After leaving the collimator, the x-rays strike the specimen mounted in the experimental system. To record

FIG. 11. Schematic of components in the beam path of the A2 experimental station at CHESS. Components downstream of the monochromator are located within the station. The experimental system applying *in situ* thermomechanical loading to the specimen is not shown.

diffracted intensity, an area detector with a 2048×2048 array of $200 \mu\text{m}$ square pixels sits downstream of the specimen, at a working distance between 400 mm and 1400 mm. This distance is typically 700 mm, selected to balance lattice strain resolution and the number of $\{hkl\}$ s sampled: far from the specimen, positional changes in the diffracted intensity at the detector from lattice strain are greater, though diffracted intensity is measured from fewer $\{hkl\}$ s.

B. Experimental procedure

Subsequent to any study conducted using the system, the distance between the specimen centroid and detector is precisely quantified for each specimen orientation. To accomplish this, a dummy specimen with a circular void at its centroid is filled with a calibrant powder (a material with a highly resolved lattice parameter), wrapped in x-ray transparent polyimide tape, and mounted to the platens/pushrods. The diffraction of the unstrained calibrant powder is used to measure each distance. The dummy specimens used for alignment in each experimental configuration are shown in Fig. 12.

To measure the specimen-to-detector distance, an entire rotation sequence is run with the powder-filled dummy specimen loaded in the system. A rotation sequence is defined as a maximum of -60° to $+60^\circ$ of ω rotation, with measurements made every degree. At each one degree step of ω rotation, the specimen is rotated a variable amount in ω and χ (rocked) to ensure a sufficient number of crystals are illuminated.

To complete the setup, the specimen of interest is aligned as described in Sec. V B 2, and loaded into the system along with the environment assembly and furnace. Calibrant powder is then placed within an x-ray transparent container and mounted to a separate rail. The diffraction of this powder aids in data reduction, and enables tracking of the center of flux in the x-rays which can shift slightly during the course of an experiment.

Next, the experiment is conducted as follows:

1. The rotation sequence, rocking in both ω and χ at each step, is again run to record the diffracted intensity of the sample at zero load/room temperature. This set of data is used as a reference to determine strain due to thermomechanical loading.
2. Once the sample has reached the first temperature set-point, a zero load/at temperature set of diffraction data is collected (again running through the full rotation sequence).

FIG. 12. View of the dummy specimen used for alignment in (a) the compression loading configuration and (b) the tension/compression loading configuration. The circular void in each specimen is filled with unstrained calibrant powder and sealed with polyimide tape.

3. The sample is then brought to and maintained at the first prescribed macroscopic stress, while the rotation sequence is run. A loaded/at temperature diffraction pattern is measured at each of the rotational steps. Once complete, the specimen is brought to the next prescribed stress point or the next temperature setpoint, and the process is repeated.
4. Once the experiment is complete, the powder-filled dummy specimen is again loaded in and diffraction measurements are made for a full rotation sequence to verify that the center of rotations did not shift during the course of the experiment.

C. Measured data

To demonstrate the performance of the new system, data obtained from studies of silicon and LSHR are included. Macroscopic stress as a function of strain, in addition to lattice strain measurements depicted on SPFs, are shown for each material.

1. Single crystal silicon results

A silicon sample, with the [111] direction aligned with the loading axis, was compressed monotonically to 750 MPa with loading paused to conduct lattice strain measurements at 250 MPa increments using a $200\ \mu\text{m} \times 200\ \mu\text{m}$ beam of 50 keV x-rays. The sample was unloaded, heated to 200 °C, and the previous loading history repeated, again recording diffraction measurements at three points.

Within the portion of a single crystal sample illuminated by the x-ray beam, the strain state is assumed to be homogeneous as this portion of the sample is sufficiently far from the two loading surfaces. This implies that each lattice strain measurement can be interpreted as the projection of the strain state (a tensor value) along the scattering vector direction:

$$\epsilon_{\mathbf{c}||\mathbf{s}} = \hat{\mathbf{s}} \cdot \tilde{\epsilon} \cdot \hat{\mathbf{s}}, \quad (4)$$

where $\tilde{\epsilon}$ is the lattice strain tensor, $\hat{\mathbf{s}}$ is a unit vector parallel to the scattering vector, and $\mathbf{c}$ is the normal of planes from which measurements are made, parallel to $\mathbf{s}$.

To efficiently depict measured lattice strains, all measurements at each prescribed stress and temperature point were projected onto a single pole figure, shown in Fig. 13. As expected, compressive lattice strains with the largest magnitude were in the loading direction (z), with the Poisson effect producing strains of opposite sign in the orthogonal directions (x, y).

Six lattice strain measurements are necessary to uniquely solve a three-dimensional set of “Rosette equations” (Eq. (4)) for the lattice strain tensor. To minimize the effect of error in individual lattice strain measurements, more than six measurements are typically made, and a least squares optimization is used to determine a best fit to the data. To ensure low error in lattice strain tensor components, 10–12 lattice strain measurements which span the pole figure are usually required.⁴ Here, 27 measurements were made which span approximately 240° of the

FIG. 13. Pole figures of lattice strains measured in single crystal silicon, at each prescribed macroscopic stress and temperature point. Each pole figure contains data from multiple sets of lattice planes ($\{hkl\}$ s). Load was applied along the z axis as shown in Fig. 3.

pole figure. The resulting, optimized normal strain tensor values were plotted as a function of macroscopic load, shown in Fig. 14. The measured strain tensors are in good agreement with values calculated using anisotropic Hooke’s law with established temperature dependent elastic compliance values and the known uniaxial stress state, shown as dashed lines.¹⁵ The per-component errors were estimated using the reference set of lattice strain measurements made while the sample was at zero load, in a method similar to Ref. 16. Dynamic diffraction effects from the near-perfect silicon crystal introduce error into lattice strain measurements,¹⁷ however with a large number of strain measurements made, lattice strain tensor values can still be determined.

2. Polycrystalline LSHR results

A polycrystalline LSHR sample at 550 °C was subjected to 50 fully reversed loading cycles, with diffraction measurements made during cycles 1, 2, 10, and 50 using a $400\ \mu\text{m} \times 400\ \mu\text{m}$ beam of 50 keV x-rays. During measurement cycles, the sample was deformed to each prescribed macroscopic stress and the load decreased to 90% before making diffraction measurements, so as to lessen the effects of creep during the measurements.¹⁸ Approximate macroscopic stress-strain values at which measurements were made during the first cycle are shown in Fig. 15, corresponding to prescribed macroscopic stress values of 0 MPa, ± 400 MPa, ± 800 MPa, and ± 1150 MPa.

FIG. 14. Optimized normal strain tensor components at prescribed macroscopic stress points, determined from lattice strains in single crystal silicon at (a) 25 °C and (b) 200 °C. Included theoretical stress-strain curves are calculated from the following elastic constants:¹⁵ (a) $C_{11} = 165.64$ GPa, $C_{12} = 63.94$ GPa, $C_{14} = 79.51$ GPa; (b) $C_{11} = 162.52$ GPa, $C_{12} = 62.68$ GPa, $C_{14} = 78.19$ GPa.

Diffraction measurements of both the thermal expansion coefficient and lattice strain are depicted on pole figures for characteristic lattice planes ($\{hkl\}$ s). Methods of x-ray diffraction were used to quantify the thermal expansion coefficient in place of more common techniques. The thermal expansion coefficient associated with each scattering vector, $\alpha_{\mathbf{c}_{||}\mathbf{s}}$, is defined as

$$\alpha_{\mathbf{c}_{||}\mathbf{s}} = \frac{\epsilon_{\mathbf{c}_{||}\mathbf{s}}}{\Delta T}, \quad (5)$$

where $\epsilon_{\mathbf{c}_{||}\mathbf{s}}$ is the strain resulting from a temperature change ΔT , and is as defined in Eq. (3). By measuring the lattice plane spacing at zero load in an LSHR specimen both at room tem-

FIG. 15. Macroscopic stress-strain values of diffraction experiments, conducted on an LSHR alloy at 550 °C. Deformation began at the origin A and ended at B. Dashed lines connecting the diffraction points give a rough outline of the macroscopic stress-strain behavior.

perature and 550 °C, the thermal expansion coefficient was calculated for each scattering vector in an entire rotation sequence (detailed in Sec. VI B). Data for each measured $\{hkl\}$ can be visualized in a pole figure by plotting the value of $\alpha_{\mathbf{c}_{||}\mathbf{s}}$ at the intersection of the respective scattering vector and a reference sphere. Part (a) of Fig. 16 contains $\alpha_{\mathbf{c}_{||}\mathbf{s}}$ pole figures for the $\{111\}$, $\{200\}$, $\{220\}$, and $\{311\}$ planes. The mean $\alpha_{\mathbf{c}_{||}\mathbf{s}}$ was $13.5 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$, in good agreement with Ref. 19. The coefficient of thermal expansion for cubic materials is isotropic, i.e., identical for all $\{hkl\}$ s, in all directions. The variation in $\alpha_{\mathbf{c}_{||}\mathbf{s}}$ was relatively small (standard deviation of $0.4 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$) and likely the result of slight oscillation in the furnace temperature.

Lattice strains measured in an LSHR specimen during thermomechanical loading can be used to construct pole figures in the same fashion as described above. Pole figures containing lattice strains measured at a temperature of 550 °C and a macroscopic stress of 1150 MPa in tension and compression are shown in part (b) of Fig. 16. The pole figure coverage, i.e., the number of diffraction measurements made, was optimized based on the conditions of the experiment: the area detector size, experimental system geometry, and sample to detector distance. Further details on lattice strain measurements and their significance in validating and calibrating material models can be found in Refs. 1, 2, 10, and 11.

VII. SUMMARY AND CONCLUSIONS

An experimental capability has been described that enables monitoring, in many directions, of the evolving crystal scale response of crystalline materials to thermal and mechanical loads. Central to this capability is an experimental system which both reorients specimens and applies thermomechanical loading during x-ray diffraction experiments. The system satisfies all design objectives: to heat the specimen uniformly in a controlled environment while applying *in situ* mechanical

FIG. 16. LSHR pole figures for four characteristic $\{hkl\}$ s at 550 °C, containing (a) thermal expansion coefficient data and (b) lattice strain data at a macroscopic stress of 1150 MPa in tension (upper set of pole figures) and compression (lower set). Load was applied along the z axis as shown in Fig. 3.

loading, reorienting the specimen over a large angular range, and not obstructing the incident or diffracted x-rays. Key elements of the system include:

- A loadframe with electromagnetic actuation that supports two specimen experimental configurations: one for specimens loaded in compression only and one for specimens loaded in tension and/or compression.
- A furnace with a custom resistance heater and a dedicated cooling system that does not obstruct incident or diffracted x-rays.
- A specimen environment that accommodates a range of atmospheres.
- Two rotational axes with alignment stages for the purpose of rotating the specimen relative to the x-rays, while minimizing translation of the specimen centroid.

Thus far studies, conducted at CHESS, have focused on single crystal silicon and polycrystalline LSHR nickel-based superalloy, and have demonstrated the system is capable of enabling precise measurement of lattice strains. The primary goal of the experimental work is to produce key data for the development of high fidelity material models. Given the systems ability to measure material response to *in situ* mechanical loading applied at elevated temperatures, data collected could in time lend more complete perspective on the

influences behind and preventative measures towards reducing microcrack initiation.

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