High Energy X-ray Micro-tomography for the characterization of thermally fatigued GlidCop specimen

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The expected increases in the thermal power of X-ray beams produced at the third-generation synchrotron radiation facilities may exceed the heat-load capabilities of the existing high-heat-load components with which the beams interact. An X-ray beam shutter is an example of such components. Typically made of GlidCop, it is used, as needed, to block the X-ray beam from entering the experimental area. Analyses show that the planned 50% increase in storage ring beam current (and thus beam heat load) at the Advanced Photon Source (APS) will result in plastic strain in the shutter limiting its operational life. In order to develop a predictive model to estimate the number of cycles to failure, cooled GlidCop samples were thermally cycled 10,000 times under the high heat load of an X-ray beam from two inline APS undulators. Samples are examined for cracks, and crack size information is extracted for use in the model. In this paper, following some introductory remarks, we report on the characterization of the micron-size fatigue cracks in a sample GlidCop using high-energy X-ray absorption and phase-contrast tomography. It is shown that the current set up at beamline 1-ID at the APS is capable of nondestructive reconstruction of internal structure of the fatigued part with a resolution of 3 µm, which may further be improved to about 1 µm.

1. Introduction

High-energy third-generation synchrotron X-ray facilities use insertion devices (undulators and wigglers) to generate collimated intense and tunable hard X-ray beams for a broad range of scientific and engineering applications. Each polychromatic (white) X-ray beam leaving the exit ports around the storage ring pass through the ‘front end’ where masks, slits, and shutters are used, respectively, to spatially confine, define, or block the beam. From there a beam can enter the experimental area where user-controlled filters, windows, slits, shutters, mirrors, monochromators, lenses, etc. are used to condition the X-ray beam as required for each experiment.

A feature of white X-ray beams is their combined high power and high heat flux [1]. An examination of the dependencies of the total power and the peak heat flux of a beam at some distance (D) from the source are instructive. The total power \( P \propto E^2 B^2 I L \) is dependent on the ring energy squared (\( E^2 \)), device magnetic strength (B), the electron beam current (I), and the device length (L). The peak heat flux, \( F_0 \propto E^4 B I L / D^2 \), on the other hand, is dependent on the fourth power of the ring energy. So, all other things being equal, a 7-GeV ring produces beams with 4 times more power and...
16 times more heat flux in comparison with a 3.5-GeV storage ring. The combined high total power and heat flux can make the thermal management of those front-end and beamline components, which directly interact with the x-ray beams, quite challenging.

The upgrade plans at the Advanced Photon Source (APS) usher in a number of new or enhanced source configurations (e.g., multiple undulators including superconducting devices, higher beam current), resulting in, among other things, the generation of yet more powerful X-ray beams [2]. For example, whereas presently the total power and heat flux (20 m from the source) of a single Undulator A the APS is about 6 kW and 400 W/mm², respectively, the combination of higher beam current and the multiple and/or more powerful sources in the longer straight sections of the storage ring will result in heat loads approaching 20 kW and 1600 W/mm², respectively. With such high thermal loads, the temperature and stress in the high-heat-load front-end components, such as the X-ray shutters, will exceed the current design criteria set for their safe and long-life operation. This gives rise to the question of whether the current criteria limiting the maximum temperature and stress in the component can be relaxed and if so, to what extent and on what basis? If the components cannot be shown to last the expected life of the facility, they would either have to be periodically replaced with similar or new designs, as appropriate. These issues have considerable practical and economic impacts.

2. Thermal Fatigue

Although efficient cooling has been a key consideration in the design of high-heat-load front-end components, incident X-ray beams with higher power can induce significantly higher thermal gradients and thus thermal stress and strain in the components. When the stresses are below the elastic limit of the substrate material, the components could survive a large number of thermal cycles of beam on/beam off, because the strains are fully reversible. With stress levels exceeding the elastic limit, during the heating-up phase the component can go through multiaxial plastic compression. Upon cooling, the elastic stress and strain unload but the plastic components do not. With additional thermal cycles and the accumulation of stress, crack nucleation producing microcracks can commence. These microcracks accumulate and join, resulting in fatigue cracks. If the cracks are not arrested and deepen to reach component’s cooling channels, water contamination of the storage ring can occur. Such a failure, which can potentially shut down the storage ring for some time, underlies the critical need for understanding thermal fatigue in high-heat-load components.

Since the number of thermal cycles these components undergo during their anticipated life span of about 20 years is finite and on the order of 10,000, it is desirable to develop criteria (such as limits on the temperature and stress in the component) to ensure the survival of the component over that many cycles. Intuitively, the higher the beam heat loads, the higher the stress, and the fewer the number of cycles to failure. In practice, specific failure criteria and a reliable model to predict the allowable stress for a 10,000-cycle lifetime are necessary.

The multiaxial thermal fatigue phenomenon encountered is poorly understood, complex, multifaceted, and statistical in nature [3-5]. A sufficient number of test samples are required to develop and validate a predictive model. Several heuristic models [6] have been proposed, but they require reliable test data to determine a set of constants in the equations. Additional case-specific tests are needed for strict verification. Strain, which is the critical parameter in the leading theoretical models [7] for fatigue life prediction is difficult to measure directly, so our approach has been to directly measure the temperature variations during cyclic loading and to estimate the strain from elastic-plastic finite element simulations validated by the measured temperature.
2.1. GildCop®

GildCop® [8], a dispersion-strengthened copper alloy, is the material of choice for many high-heat-load front-end components. The AL-15 (UNS-C15715) variety (with ~0.15% by weight Al2O3) used at the APS has a thermal conductivity 92% that of high purity copper, yet, unlike the latter, it maintains its mechanical strength at high temperatures. Its yield and ultimate tensile strengths are comparable to mild steel. The combination of high conductivity and high-temperature strength makes it a suitable material for X-ray absorbers on high-heat-load beamlines. Although absorbers are designed to intercept incident beams at very shallow angles to reduce the absorbed heat flux and thus temperature and stress, the expected increases in beam power will result in plastic deformation of GildCop. Fatigue properties are then required, yet the available data on this material is scant. For this reason and following earlier work [9-12], APS has embarked on a thermal fatigue study taking advantage of the parasitic beam time from a high-heat-load beamline under construction.

3. Thermal fatigue experiments

Estimation of thermal fatigue life is typically made by cyclical thermal loading of a large number of samples to a specific stress level, cycling until they fail, and repeating this for various stress levels until a set of data relating the stress level in the part to the number of cycles to failure is obtained. In the present in-vacuum study, because of the difficulty of accessing the samples intermittently, they were all cycled for a fixed number of cycles and then examined for cracks and crack size.

![Figure 1: From left clockwise: Four GildCop samples shown mounted on the sample holder assembly that is inserted inside the vacuum chamber; a cartoon of the X-ray beamline with the test chamber and the X-ray beam from two undulators striking a test sample on the sample holder; the 4 mm × 5 mm footprint surface area of a fatigued sample after 10,000 cycles with an absorbed X-ray power of 1185 W showing multiple cracks in a 1.2 mm × 4.3 mm area.](image)

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A total of 45 GlidCop samples, each 100 × 25 × 22 mm³, were prepared and brazed, four at a time, onto a cooling line mounted in a vacuum chamber (Fig. 1, left). Each sample was subjected to a normal incident 4.5 mm × 4.5 mm X-ray beam for 10,000 cycles. Using an upstream shutter, the beam was 1 second on and 9 seconds off (a sufficient number of times for it to reach 90% of the respective steady state conditions). The beam is from two inline undulators (Fig. 1, right, top) whose gaps were changed to provide various thermal loads, ranging from 674 W to 2250 W, on different GlidCop samples. The estimated maximum surface temperature ranges from 325° to 1096°C.

Visual examination of the tested samples showed a variety of responses to varying power loads including incipient cracking, multiple surface cracking, deep cracking, “exfoliation,” ablation, surface extrusion, and finally melting. The crack size and depth in the samples (in the deep cracking regime) provide the necessary data for the leading predictive thermal fatigue models [6,13].

Measuring micron-size cracks usually requires progressive slicing and polishing of the sample. In the process, samples are destroyed. In the present study, cracks were typically confined to an area of about 5 mm × 5 mm beam around the footprint, and a few mm deep. Instead of the traditional technique, we investigated the possibility of measuring the micocracks in Glidcop nondestructively using the high-energy X-rays beams available the APS, as reported in this paper.

4. Wide field-of-view microtomography

The APS 1-ID beamline utilizes a 33-mm period undulator source. It is equipped with a cryogenic high-energy monochromator that provides a 2 mm × 2 mm collimated monochromatic x-ray beam. The energy bandwidth of the monochromatic beam is about 10⁻³, allowing both microtomography and diffraction experiments.

Figure 2: For a sample larger than the available field of view (FOV), horizontal segments are first scanned, one FOV at a time, with some overlap, as shown. The sample is then translated vertically and the process repeated. The 7.5× long working distance objective (with a 1× secondary lens not shown) images the 25-μm-thick scintillator (at 2.5 mm from the specimen) onto the CCD. For phase-contrast tomography, the scintillator and all optical components are translated 600 mm downstream.
In a typical study, a sample is mounted and centered on a rotation axis and is rotated by at least 180°, acquiring projection images while the entire width of the sample is kept within the field-of-view (FOV) of the detector. Samples not wider than the FOV can be scanned and then translated vertically, if necessary, to scan other parts and stitch the reconstructed volumes. Thus it is often the sample width and not length that results in complications.

With high-energy X-rays, the penetration power of a beam is sufficient to allow the examination of samples thicker than the beam size in the tomographic scans. Thus, several smaller side-by-side scans are made, and they are then stitched digitally to arrive at what amounts to a wide field-of-view single scan. To accomplish this, the rotation axis is translated perpendicular to the beam to allow scanning of a different part of the sample in the beam. Resulting projection images belonging to the same angular position are treated as parts of a large panoramic view of the sample and are properly stitched.

For the present tomography experiment, a $5 \times 5 \times 5 \text{mm}^3$ volume of the larger sample was harvested. It includes the surface area exposed to the incident beam (beam footprint, which also has cracks). Seemingly, the fatigue cracks were confined to this volume as well. An 88-keV beam was selected to have sufficient transmitted intensity for detection through the diagonal of the square cross section and sufficient intensity resolution to resolve the smallest features in the sample, which is ultimately determined by the dynamic range and the noise in the CCD camera. In order to have both high optical resolution as well as good X-ray-to-visible-photon conversion, a 25-µm-thick LuAG:Ce single crystal scintillator was used (Figure 2).

To reconstruct the sample reported in this paper, four horizontal FOVs, each 1.95 mm wide and 1.8 mm tall, were combined into one 1.8 mm tall $\times$ 7 mm wide FOV for each angular position with 0.1° step over 180°. The FOVs were overlapping by about 100 µm to assure the proper parameterization of the stitching of the projections. The overall 3D spatial resolution of the measurement after the processing procedure is estimated to be about 3 µm with 1 µm/voxel sampling. The volume of the data acquired during one wide FOV scan was about 57 GB with 16-bit images. This increased to 460 GB with 32-bit floating-point during preprocessing and image reconstruction. To speed up processing and for further analysis, postprocessing algorithms including cropping to the region of interest, selecting the relevant range of the intensity scale, and reducing to 8-bit resolution reduced the data to about 15 GB. Since the physical resolution is about three times lower than the sampling for general visualization purposes, a re-binning of the 3-D reconstruction with $3 \times 3 \times 3$ averaged voxels reduced the data to about 0.5 GB without any significant loss of the image quality in this instance, yet providing for speedy 3-D visualization. In the present paper the Gridrec reconstruction algorithm was used [14-15].

Each tomography scan took about 30-45 minutes, and the full reconstruction of the entire set of scans with the necessary stated overlapping measurements took about six hours.

Tomographic images of the specimen with a resolution of 3 µm were thus obtained. Figure 3, as an example, shows a 100-µm-tall slice of the $5 \times 5 \times 5 \text{mm}$ thermally fatigued GlidCop sample. There are several reasons for the 3-µm resolution limit with the present setup, the main one being the scintillator. The scintillator is 25 µm thick whereas the depth of focus of the 7.5× Mitutoyo infinitely corrected long-working-distance microscope objective (Edmond Optics, NT66-383) is about 6 µm. A thinner scintillator could reduce some of the blur for better resolution, but it would be at the expense of a substantially increased scan time. Another factor limiting the resolution is the X-ray source size. A modified setup that uses focusing optics to create a fine virtual source could further improve
resolution. With these two improvements, a 1-µm resolution in absorption tomography may be achievable.

Smaller features, smaller than the pixel resolution of the detector, can be seen in phase-contrast tomographic images obtained by translating the scintillator and all downstream components 600 mm away from the sample and repeating scanning and reconstruction steps. Figure 4, as an example, shows a slice through the sample revealing a surprising range of internal features and interfaces. For example, there appear to be internal microcracks that do not originate at the free (heated) surface. Examination of additional fatigued samples subjected to different heat loads is planned for a better understanding of the role of internal stress in the creation and closing of internal microcracks. Tomographic evaluation thus provides, nondestructively, quantitative data (crack size/depth) as well as insights for a better qualitative understanding of stress, fracture, and high-temperature thermal fatigue in this important material.

5. Conclusions

Understanding the fatigue properties of GlidCop is essential for the development of fatigue models for use in the analysis and design of high-heat-load components including photon shutters at third-generation synchrotron X-ray facilities. The present work has demonstrated the feasibility of obtaining detailed quantitative and qualitative information about thermally fatigued samples using absorption and phase-contrast tomography. The spatial resolution is influenced by various factors including optics, scintillator type and thickness, beam energy, mechanical systems, and reconstruction algorithm. The current 3-µm-resolution absorption tomography is sufficient for the current study. Overcoming limitations imposed by the scintillator thickness, source size, and optics can further improve it. Furthermore, image reconstruction can be improved with the availability of a larger beam to reduce overlapping image collection. The phase-contrast tomography results reported here point to the possibility of detecting yet smaller features, but further work is needed to both verify the present results and provide quantitative information about the features seen.

Figure 3: An absorption X-ray tomographic reconstruction showing a 100-µm-tall slice of the 5×5×5 mm cube thermally fatigues Glidcop sample. The bright traces are artifacts and correspond to the areas near the stitching of the images. The front surface corresponds to the beam footprint area and the dark green is the air volume around the sample. The gray area shows a slice of the specimen and a crack.
If another opportunity for further fatigue study using high-heat-load X-ray beamlines presents itself, it would be desirable to use high-energy photons to monitor crack initiation and propagation in a phase-contrast imaging setup composed of a scintillating crystal and a mirror set to re-direct the image out of the vacuum chamber and onto a CCD camera. Photons with energies over 200 keV present in the X-ray beam would propagate through the thickness of the test samples.

Figure 4: This image shows a 0.5-mm-tall (5-mm-wide) cut from the phase-contrast tomographic reconstruction of the specimen, providing a closer look at its interior. The features surrounding the main crack appear to be internal cracks, generally in the same direction.

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