The effect of temperature on the lattice spacing around a notched Zircaloy-2 specimen determined from X-ray diffraction

S. Read[1], S. Hanlon[1], G. McRae[1], C. Coleman[2], and B. Leitch[2]

[1] Carleton University, Ontario, Canada
(scottread@cmail.carleton.ca)

[2] Atomic Energy of Canada Limited, Chalk River, Ontario, Canada

A Master's Level Submission

Summary

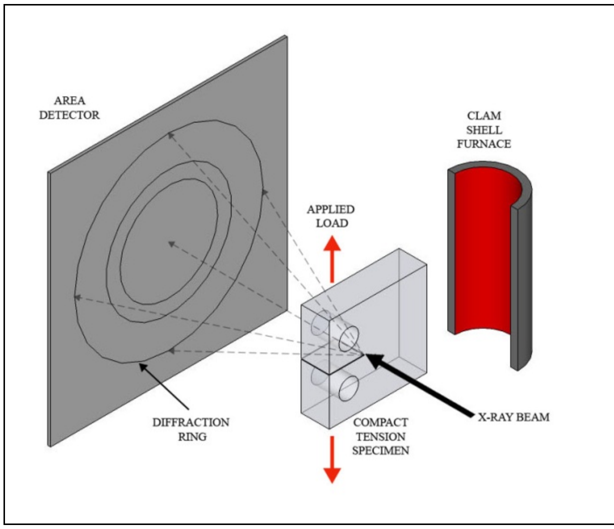
High intensityX-ray diffraction has been used to investigate lattice parameter and line width changes in a notched Zircaloy-2 compact tension specimen undergoing heating and cooling. It was found that the lattice spacing varied with temperature in part because of thermal expansion, but also because of annealing of residual stresses. Measurements made at many locations along the plane of the notch illustrate how the lattice spacing evolvesthroughout the specimen. Measurements of widths of the $(10\bar{1}0)$, $(11\bar{2}0)$, and (0002) diffraction peaksand comparisons of the line spacings,for both the a-parameter and c-parameter, with literature values of α–zirconium, at various temperatures,are used to demonstrate the effects of annealing.

1. Introduction

The experimentreported here shows how thelattice changeswithtemperatureand time around a notch in a Zircaloy-2 compact tension specimen. The $(10\bar{1}0)$, $(11\bar{2}0)$, and (0002) X-ray diffraction lines of α-zirconium were found to move because of thermal expansion. The diffraction lines were also affected by residual stress.Although the test specimen contains a notch, the effect of load will not be reported here; this study details the effects of temperature and provides a qualitative description of stress as a precursor to further study of hydrogen in Zircaloy-2 under thermo-mechanical loading.

2. Experimental Setup and Data Analysis

Lattice spacingsand line widths of the$(10\bar{1}0)$, $(11\bar{2}0)$, and (0002) planes were measuredwiththe X-ray diffraction apparatus at the Advanced Photon Source (APS) 1-ID beamline at Argonne NationalLaboratory (ANL) in Chicago, IL. Figure 1 shows a representation of the experiment. The X-ray beam wavelength was 14.42 pm. The area of the beam was50 μm × 50 μm. The beam was incident normal to a Zircaloy-2 compact tension specimen [1], as shown in Figure 1. The specimen was secured to a load frame, with 10 N, and surrounded by a furnace.Diffraction rings were collected withan amorphous silicon detector 2 m behind the specimen.The diffraction rings werecorrected for occasional bad pixels, and then integratedover 360°to produce plots of intensity as a function of lattice spacing. This study focuses on the movement and full-width-at-half-maximum (FWHM) line widthsof the$(10\bar{1}0)$, $(11\bar{2}0)$, and (0002)α-zirconium diffraction peaks that resultwhen the temperature of the specimen is cycled from room temperature to 350 °C,and then back to room temperature. Thetemperature-time historyis shown in Figure 2.The diffraction lines are fitted to a pseudo-Voigt profile; the error bars are standard errorsdetermined from the regressions.



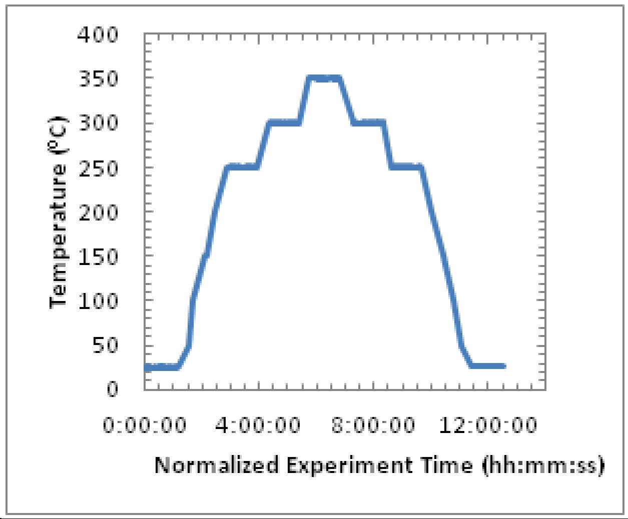


Figure 1 - Idealized experimental setup.

Figure 2 - Temperature history for experiment.

Thelattice spacing determined from the$(10\bar{1}0)$ and$(11\bar{2}0)$ planes pertain to the a-parameter, while thelattice spacing of the(0002) plane relates to the c-parameterof the hexagonal-close packed (HCP) structure of α-zirconium. The value reported for the a-parameter is an average of the values determined from the$(10\bar{1}0)$ and$(11\bar{2}0)$ planes.

3. Results: Temperature and Residual Stress Dependence

The lattice parameters and linewidthsdetermined for the temperatures depicted in Figure 2 show that the Zircaloy-2 sample underwent annealing during the experiment:

(1) The difference between the measured and expected lattice parameters[2]decreases as the sample is heated from room temperature to 350 °C, and the difference remains small during cooling from 350 °C to room temperature;
(2) Residual stressin the region closest to the notch (0 to 0.5 mm) relaxes as the sample is heated from room temperature to 350 °C, and remains relaxed as the sample is cooled;
(3) The line widths of the diffractionpeaks are narrower after heating.

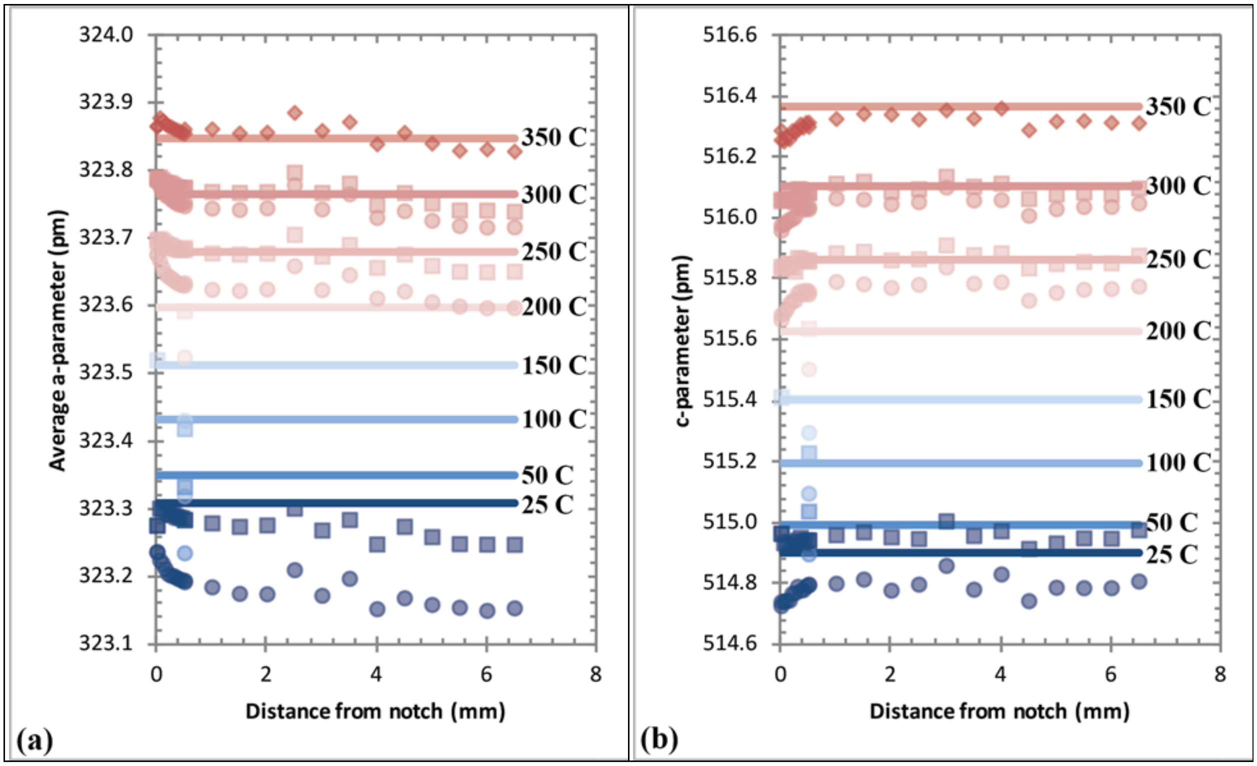


Figure 3 – Determined lattice parametersas a function of distance from a notch for different temperatures during the heat-cool cycle: (a) 'average' a-parameter, and (b) c-parameter. Circles are for heat up, squares for cool down. Solid lines are predictions at the specified temperature based on [2].

Point (1) is illustrated in Figures 3 and 4. Figure 3(a) and 3(b) show how the lattice parameters change as the beam is moved relative to the notch for the temperature cycle (Figure 2). The solid lines in these plots are expected values for fully annealed single crystal zirconium [2]. Figure 4 shows that the differences between the current measurements and those expected from fully annealed material decrease during heat-up, becoming effectively zero at the highest temperature, and then remain small during cooling.

Point (2) is demonstrated in Figure 5, which is scaled to show the near notch region of Figure 3.At low temperatures, the a-lattice parameter increases relative to the bulk values at the notch, and the c-lattice parameter decreases. The absolute differences between these values at the notch and in the bulk decrease with temperature, and become small at the highest temperature, and remain small as the sample is cooled. Thus, the lattice relaxes with temperature to produce a uniform 'annealed' structure.

In addition, Figure 5 also shows that the a and c lattice parameters are higher and lower than their respective bulk values, which is in accord with Poisson's ratio for this textured material.

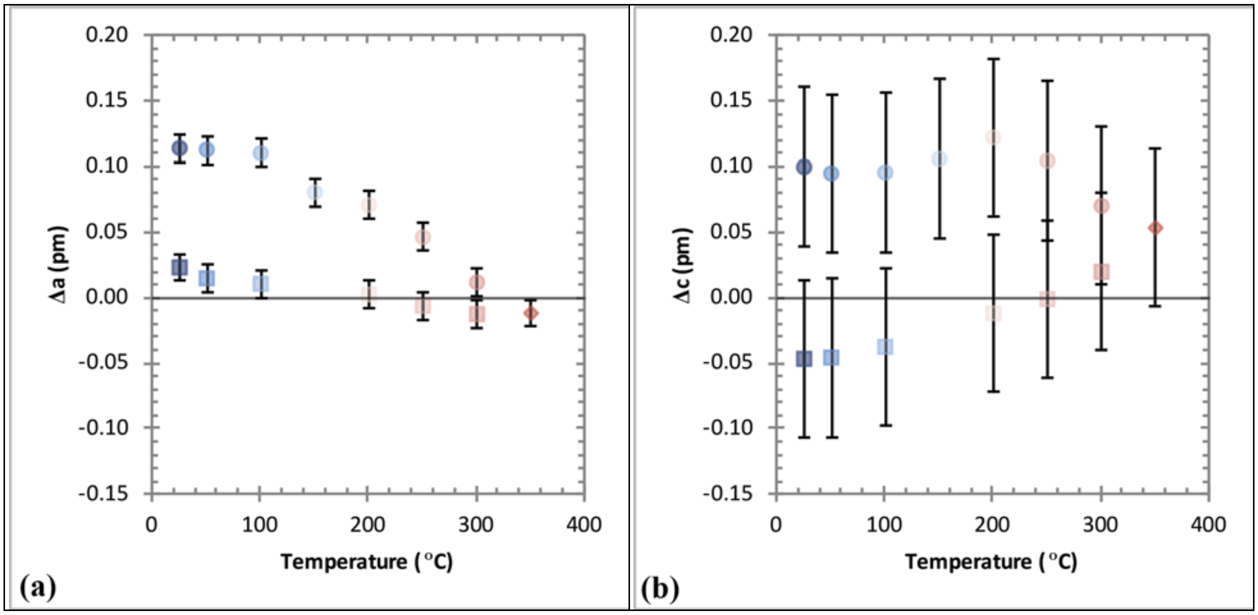


Figure 4 – Differences between the expected lattice parameters (fully annealed single crystals [2]) and the measured (a) a-parameter(b) c-parameter, at 0.5 mm from the notch. Circles represent the measurements made during heat up, while squares correspond to cool down.

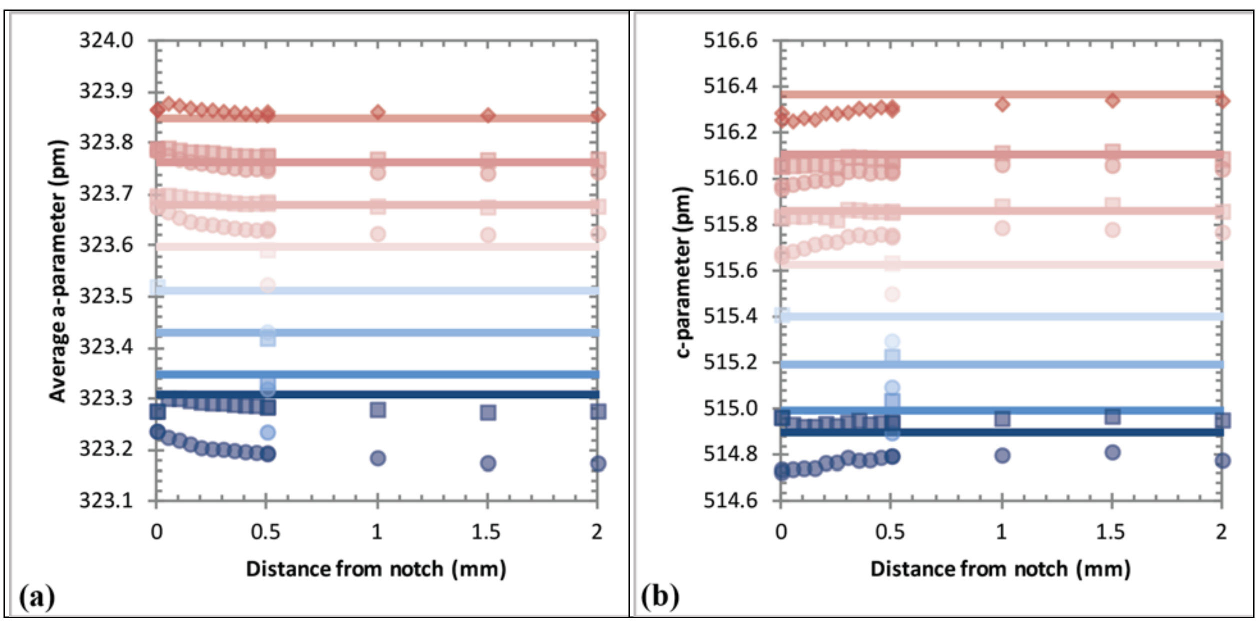


Figure 5 - The near-notch region of Figure 3 (See Figure 3 caption for details).

Point (3) is illustrated in Figure 6, which compares the line widths for the $(10\bar{1}0)$diffraction peak over the heat-cool cycle. The line widths are narrower during cooling, which is consistent with annealing of lattice damagethat broadens diffraction lines [3].

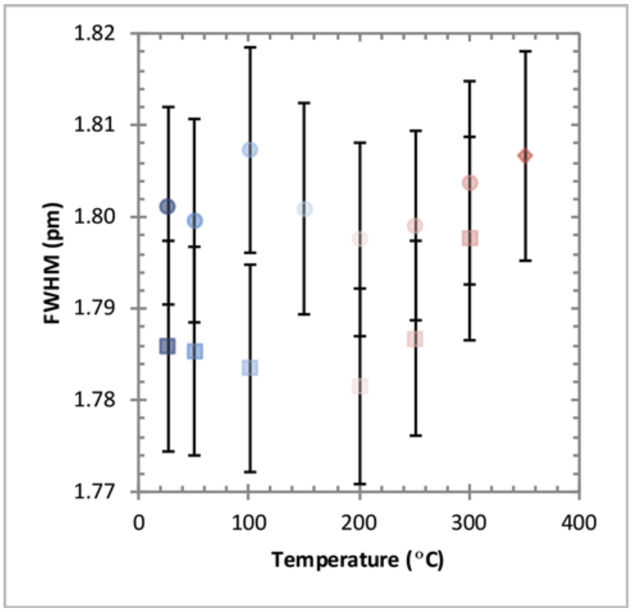


Figure 6 - Line widths for the $(10\bar{1}0)$ diffraction peak at the various temperatures of Figure 2. (See the caption for Figure 3 for an explanation of the data representation.)

4. Conclusion

Lattice spacingsand line widths for a Zircaloy-2 compact tension specimen under thermalcyclinghave been determined from X-ray diffraction using the Advanced Photon Source at Argonne National Laboratory. Determined lattice spacing and X-ray diffraction line widths showed evidence of annealing of residual stressesupon heating, and relatively constant values on cooling that were comparable with thermal-expansion expectations from previous work on fully annealed single crystal zirconium.

5. References

[1] ASTM Standard E647, 2000. "Standard Test Method for Measurement of Fatigue Crack Growth Rates," ASTM Int., West Conshohocken, PA, 2000, DOI: 10.1520/E0647-00.

[2] J. Goldak. "Lattice Parameters, Thermal Expansions, and Grüneisen Coefficients of Zirconium, 4.2 to 1130 °K",*Physical Review*, 144(2) 478-484, 1966.

[3] D. Balzar. "X-Ray Diffraction Line Broadening: Modeling and Applications to High T_c Superconductors". *Journal of Research of the National Institute of Standards and Technology*, 98(3), 321-353, 1993.

Acknowledgements

The authors wish to thank Jonathan Almer of APS and Kimberly Colas (Penn State) for discussions.