Isothermal Annealing of Shocked Zirconium: Stability of the Two-phase α/ω Microstructure

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Abstract

Under high pressure conditions, Zr undergoes a phase transformation from its ambient equilibrium hexagonal close packed α phase to hexagonal ω phase. Upon returning to ambient conditions, the material displays hysteretic behavior, retaining a significant amount of metastable ω phase. This study presents an in-situ synchrotron X-ray diffraction analysis of Zr samples shock-loaded to compressive peak stresses of 8 and 10.5 GPa and then annealed at temperatures of 443, 463, 483, and 503K. The evolution of the α phase volume fraction was tracked quantitatively, and the dislocation densities in both phases were tracked qualitatively during annealing. Upon heating, the reverse transformation of $\omega \rightarrow \alpha$ does not go to completion, but instead reaches a new

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metastable state. The initial rate of transformation is faster at higher temperatures. Samples shock-loaded to higher peak pressures experienced higher initial transformation rates and more extensive transformation. Dislocation content in both phases was observed to be high in the as-shocked samples. Annealing the samples reduces the dislocation content in both phases, with the reduction being lesser in the ω phase, leading to the postulation that transformation from $\omega \rightarrow \alpha$ is restricted by the pinning effect of dislocation structures within the ω phase. Electron backscatter diffraction analysis affirmed that the expected $(0\ 0\ 0\ 1)_\alpha \parallel (1\ 0\ \bar{1}\ 1)_\omega$ and $[1\ 0\ \bar{1}\ 0]_\alpha \parallel [1\ 1\ \bar{2}\ \bar{3}]_\omega$ orientation relationship is maintained during nucleation and growth of the α phase during the annealing.

Keywords: Zirconium, Synchrotron Diffraction, High Pressure, Phase Transformation, Annealing

1. Introduction

At ambient temperatures and pressures, zirconium is thermodynamically stable as a single phase, α , hexagonal close packed (HCP) crystal ($P6_3/mmc$, $c/a = 1.593$). Under high pressures, Zr and other transition HCP metals such as Ti and Hf undergo a phase transformation from the α phase to the simple hexagonal ω phase ($P6/mmm$, $c/a = 0.623$). The ω phase has been shown to display strong hysteretic behavior under both static and shock loading, with the high-pressure ω phase being retained after pressure is released [1, 2, 3, 4, 5, 6]. By shock-loading or deforming Zr to peak pressures above 7 GPa, as much as 80% of the ω phase has been retained after subsequent unloading to ambient pressures [7, 8, 9], and metastable nano-grained ω Zr

12 and Ti has been produced by high-pressure torsion [10, 11, 12, 13, 14]. This
13 is in contrast to the high-pressure phase transformation in Fe, where the ε
14 phase is not quenchable [15].

15 Modeling the dynamic deformation behavior of Zr and Ti necessitates
16 a robust understanding of the coupling between the mechanisms of plastic
17 deformation at high strain rates and the α/ω phase transformation at high
18 pressures. An important part of this understanding is the characterization
19 of the hysteresis of the transformation and the stability of the two-phase
20 microstructure. Cultivating this understanding is central to elucidating the
21 kinetics of the transformation that are applicable to dynamic conditions far
22 from thermodynamic equilibrium. The current work focuses on understand-
23 ing the mechanisms for arresting the reverse transformation post-shock and
24 the effects of temperature and peak shock pressure on the reverse transfor-
25 mation during subsequent annealing.

26 Investigations of the $\alpha \rightarrow \omega$ transformation in Zr and Ti have largely
27 been focused on determining the equilibrium pressure, crystallography, and
28 resulting mechanical properties and electronic structure of the ω phase [3,
29 16, 17, 18]. Multiple orientation relationships have been observed each with
30 one or more proposed mechanisms or transformation pathways [3, 6, 19, 20,
31 21, 22, 23]. Investigations of the kinetics of the phase transformation in Zr
32 have largely been limited to the forward $\alpha \rightarrow \omega$, with little found in the
33 literature concerning the reverse transformation of $\omega \rightarrow \alpha$ or the stability
34 of the dual phase microstructure. Recently, Brown et al. examined the
35 stability of the ω phase in shock-loaded Zr in-situ at a constant heating rate
36 of 3K/min from room temperature to ~ 620 K [24]. Based on the observation

37 of large initial dislocation densities (post-shock) in both the α and ω phases
38 and subsequent decrease in dislocation content in ω preceding the reverse
39 transformation, it was speculated that the hysteresis in shocked-samples is
40 due, at least in part, to the high concentration of defects in the ω phase
41 retarding the transformation and preventing the system from returning to
42 thermodynamic equilibrium after completion of the shock [24].

43 In the current work, X-ray diffraction techniques were used to charac-
44 terize the microstructural evolution of shock loaded Zr specimens during
45 isothermal annealing at low homologous temperatures. More specifically, the
46 measurements in this study include quantitative measurements of the growth
47 of the α phase and the subsequent stability of the two phase microstructures.
48 Dislocation density trends in both phases were monitored through the evo-
49 lution of the root mean square (RMS) strain ε_{rms} and related to the phase
50 transformation. The morphology of the microstructure was examined by se-
51 lected electron backscatter diffraction (EBSD) studies on the as-shocked and
52 partially annealed specimens.

53 2. Experimental Methods

54 2.1. Sample preparation

55 All samples were prepared from a high-purity crystal bar Zr (<100 ppm
56 impurities) which was upset forged, clock rolled, and annealed at 823K for
57 1 hour [25], producing a plate with a homogenous and fully recrystallized
58 microstructure, having an average grain size of 15-20 μm [25]. The plate
59 exhibits an in-plane isotropic crystallographic texture with a strong basal
60 component (>8 times uniform random distribution) nearly aligned with the

normal or through-thickness direction (TT) direction of the plate, with prism planes uniformly distributed about the in-plane directions of the plate. 5 mm thick, 25 mm diameter Zr disks were electrodischarge machined (EDM'ed) from the rolled plate with the symmetry axis of the disk parallel to the rolling normal (TT) direction for the shock loading. The specimens were then tightly fit into target assemblies specifically designed for shock loading/unloading experiments [26, 27]. The targets were impacted by 2.5 mm thick Zr flyer plates accelerated to velocities of 640 or 835 m/s, resulting in peak compressive stresses of 8 or 10.5 GPa respectively on the Zr samples [9].

From the soft-recovered [26, 28] shocked specimens, 1 mm thick $\times$ 3 mm diameter discs were EDM'ed for X-ray and microscopy examination. The disks were machined such that the disk axis was orthogonal to the shock direction and parallel to the in-plane direction of the original plate. The samples were cold mounted and prepared for microscopy by grinding with 2400 grit aluminum oxide paper and chemically polished with a solution of 45 H₂O : 45 HNO₃ : 10 HF to remove any surface damage caused by machining. The examined surface corresponded to the cross-section of the X-ray disks and contained both the shock direction and an in-plane sample direction. The same sample preparation is applied to samples that are subjected to heating during the in-situ X-ray diffraction experiments.

2.2. X-ray diffraction measurement

The in-situ heating measurements were completed on the 1ID-C beam line at the Advanced Photon Source (APS), Argonne National Laboratory [29]. A Cu sample fixture was constructed for the annealing experiments

to ensure uniformity of temperature. The sample holder was designed to facilitate the simultaneous annealing and characterization of two Zr samples, one subjected to 8 GPa shock pressure and the other 10.5 GPa shock pressure. The simultaneous heating of the two samples a) ensures that for a given annealing temperature, each sample is exposed to nearly identical thermal environments and b) greatly reduces the experimental time required to perform the experiment as the microstructural kinetics are slow compared to the collection time of diffraction patterns. Two X-ray through holes of diameter 2 mm were drilled in the 5 mm thick Cu plate, separated by 10 mm on-center. A 2 mm thick copper cover plate with two 3.3 mm diameter $\times$ 0.75 mm deep recesses was attached to the larger plate with spring clamps. A Zr disk sample was placed in each recess, with one recess containing a 8 GPa loaded sample, and the other containing a 10.5 GPa loaded sample. The spring clamps were tightened sufficiently to maintain contact and ensure good thermal conductivity, but not excessive so as to cause significant unknown stresses on the sample during heating. Since the Zr disks were slightly thicker than the recesses in the Cu plate, direct thermal contact with the Cu plate on both faces was ensured. The loaded sample holder was mounted on an MTS servo-hydraulic load frame in order to translate the two samples into and out of the beam in an alternating fashion. The samples and holder were heated in a focused optical furnace, and the temperature was manually controlled by monitoring reference thermocouples embedded in the thicker Cu plate. The Cu fixture provided an additional measurement of the temperature in contact with each sample. Given the well characterized coefficient of thermal expansion of Cu [30], passing the X-ray through the thin Cu backing

111 plate allowed for accurate tracking of the temperature change in the samples
112 from the change in Cu peak positions.

113 The 1ID beam line utilizes a monochromated beam from the standard
114 APS undulator and double-crystal Laue monochromator designed specifically
115 for high-energy X-rays [31]. The incident beam ($E = 86$ keV), was masked
116 to a $200\text{ }\mu\text{m} \times 200\text{ }\mu\text{m}$ cross-section before impinging on the sample/fixture
117 parallel to the cylinder axis of the samples. The X-ray beam penetrated 1.25
118 mm of the Cu backing plate as well as the Zr sample and was diffracted onto
119 a two-dimensional GE 41RT detector with 2048×2048 pixels ($0.200\text{ mm} \times$
120 0.200 mm pixel size) roughly centered on the straight-through beam. The
121 sample to detector distance is approximately 1500 mm, enabling 2θ coverage
122 of roughly $\pm 7^\circ$ or an approximate d-spacing from 1.0 to $3.5\text{ \AA}$. The angular
123 coverage of the detector was sufficient to collect up to 5 complete diffraction
124 rings for each phase.

125 During the in-situ annealing, the temperature was ramped as fast as pos-
126 sible ($\sim 1.5\text{ K/s}$) from room temperature to the annealing temperature while
127 avoiding significant overshoot. Samples shocked at both 8 and 10.5 GPa were
128 annealed at 443, 463, 483, and 503K (reference thermocouple temperature).
129 Samples were held at temperature until visual inspection of the collected
130 diffraction patterns showed no change over a 15 minute increment. The an-
131 nealing times varied with temperature from 2×10^3 to $> 2 \times 10^4$ seconds. The
132 503K sample (10.5 GPa) was then further heated at a higher temperature
133 773K for 30 minutes to produce a fully annealed sample with no measurable
134 retained ω phase. During characterization of the 463K samples, there was
135 an unplanned X-ray beam loss for approximately 25 minutes. During this

time the samples temperatures were maintained and data collection resumed upon beam restoration.

2.3. X-ray data analysis

Each 2-D diffraction image was binned into $24 \times 15^\circ$ intervals in the azimuthal angle, η about the through beam using Fit2D [32] to make individual 1-D diffraction patterns with diffraction vectors nearly transverse to the incident beam ($\approx 2^\circ$ off) and transverse to the cylinder axis of the sample. As the samples were cut with their cylinder axis transverse to the shock direction, the 24 diffraction vectors sample orientations from the shock direction (TT) to an IP direction.

Whole pattern Rietveld refinements of the 1-D diffraction patterns were accomplished using GSAS software developed at Los Alamos National Laboratory [33]. The data was analyzed to determine the volume fractions, lattice parameters, peak variances of the α and ω phases, and the lattice parameter of the Cu backing plate in contact with the samples. Single diffraction peaks were analyzed for peak position, integrated intensity and peak width using the Rawplot subroutine of GSAS. The peaks were fitted with a pseudo-Voigt peak profile. The automated routines APSrunrep and APSspf [34] which call GSAS subroutines, were used to enable the analysis of the tens of thousands of diffraction patterns. Quoted uncertainties are based off the estimated standard deviations returned by GSAS. Examples of integrated 1D diffraction patterns in the as-shocked and fully annealed states have been shown previously in Brown et al. [24]. Little peak overlap between the two phases is present, allowing five unique peaks from each phase to be analyzed. Both the Rietveld and single peak fits are robust and the results are consistent

internally and with previous studies [24].

The thermal strains in the Cu plate, in conjunction with the known thermal expansion of Cu, were used to more accurately determine the temperature of the sample. In general, the temperature of the Cu plate in contact with the samples (taken to be the accurate measure of sample temperature) was in good agreement (within $\pm 2\text{K}$) with the reference thermocouples. Additionally the temperature difference between the two samples in the holder was $\leq 2\text{K}$.

Information concerning the defect state of the microstructure can be estimated from the diffraction peak breadth and variance, both of which are determined for the α and ω phases by the Rietveld refinements in GSAS. The diffraction peak variance is frequently assumed to be a sum of contributions from the RMS strain, ε_{rms} , which is associated with dislocations and finite crystallite domain or sub grain size (although other microstructural effects can also affect the peak breadth).

In this work we will use ε_{rms} as a qualitative measure of dislocation density in the α and ω phase. In the case of a dislocated crystal, ε_{rms} depends on the size of the integration distance used to calculate the average [35, 36, 37]. For the purpose of assessing trends and qualitative measurement, this dependence can be neglected and it can be assumed with reasonable accuracy that ε_{rms} is proportional to the dislocation density in each phase [24]. Unfortunately, quantitative measures such as diffraction line profile analysis are not applicable due to the limited knowledge of deformation in the ω phase (knowledge of the active slip systems and Burger's vectors are lacking) [24]. In previous work, Brown et al. performed quantitative line

186 profile analysis on the α phase during constant rate heating experiments and
187 found that the ε_{rms} scaled well with the computed dislocation density and
188 that trends in defect density evolution could be easily discerned from the
189 RMS microstrain [24].

190 Additionally the crystallographic texture of the as-shocked and fully an-
191 nealed samples was measured by rotating the samples $\pm 45^\circ$ in $\Delta\chi = 15^\circ$
192 increments in the incident X-ray beam. For each χ angle, the data was
193 binned in into 24 azimuthal bins and the complete set of histograms were re-
194 fined simultaneously with MAUD using the Rietveld method and E-WIMV
195 representation of the orientation distribution function with 10° resolution
196 [38]. The texture analysis, performed largely for validation against previ-
197 ously reported experiments, showed no new findings [24]. The as shocked α
198 and ω transformation textures were found to be nearly uniform (< 2 time
199 random), and the fully annealed α texture was also nearly random indicat-
200 ing no preferential variant selection during nucleation and growth. To be
201 succinct, the reader is referred to [24] for pole figures.

202 2.4. Electron Backscatter Diffraction

203 The samples were examined in an FEI XL-30 ESEM equipped with a TSL
204 DigiView EBSD camera. EBSD was performed on both the 8 GPa and 10.5
205 GPa samples, however the 10.5 GPa samples produced diffraction patterns
206 of poor quality with exceptionally noisy results. Although initial analysis of
207 the 10.5 GPa samples appears to be in qualitative agreement with the results
208 reported for the 8 GPa samples, they will not be shown until higher quality
209 results can be collected. Post collection, the 8 GPa data was moderately
210 cleaned up using the phase correlation clean up routine in the TSL OIM

211 Analysis software.

212 **3. Results**

213 *3.1. In-situ X-ray diffraction experiments*

214 After shock loading, the initial α volume fraction for the material shocked
215 to 8 and 10.5 GPa is 0.39 ± 0.02 and 0.18 ± 0.03 respectively. This is in good
216 agreement with previous studies on the same material, although with slightly
217 higher sample to sample variance [24]. $t = 0$ s is taken as the time when the
218 temperature reaches 95% of the target annealing temperature. Comparing
219 the initial volume fractions shown in Figures 1a and 1b with those reported
220 above, it is seen that some degree of phase-transformation occurred during
221 the temperature ramp up. The evolution of the α phase fraction in both
222 samples follow the same general trend during the isothermal hold. The initial
223 rates of transformation (represented by the slopes of the curves in Figure 1a
224 and 1b) are relatively high at sufficiently high temperatures but decrease
225 with time. The α -phase volume fraction grows and saturates at a steady
226 state level that is less than one, instead reaching another metastable state
227 containing both α and ω phases. The reader can further verify this in Figure
228 2 which displays the integrated spectra and corresponding Rietveld fittings
229 for 10 GPa samples heated at 230C prior to and after the heating. the Both
230 the initial transformation rate and extent of the $\omega \rightarrow \alpha$ transformation are
231 strongly temperature dependent, with both increasing with temperature.

232 Interestingly, the transformation rate is also a function of the post-shock
233 condition of the material. The reverse transformation from ω to α occurs
234 more rapidly in the 10.5 GPa shocked sample in contrast with the 8 GPa

shocked sample. Figures 3c and 3d illustrate the described effect of the pre-shocked peak pressures on the $\omega \rightarrow \alpha$ transformation. At higher temperatures, the α concentration in the two samples crossover, and the final steady-state α concentration is higher in the 10.5 GPa shocked material, despite initially having a lower α volume fraction. Given sufficient time, the transformation stabilizes or saturates at a higher α fraction than the 8 GPa sample. Figure 3d also highlights the degree of transformation which occurred during the temperature ramp to the final annealing temperature. Despite ramping as quickly as the experimental setup allowed, changes in volume fraction of up to 0.05 (for the 503K samples) were observed during the temperature ramp.

Figure 4 shows the initial transformation rates of the 8 and 10.5 GPa shocked samples as a function of temperature. Transformations occurring during the temperature ramp-up is neglected in this calculation. The initial transformation rates increase with temperature for both samples as expected. Interestingly, a crossover trend is once again seen in the transformation rate as temperature changes. The sample shocked to 8 GPa starts with a higher initial transformation rate at the lowest annealing temperature (443K) but is eventually surpassed by the 10.5 GPa at higher temperatures.

Figure 5 shows the time evolution of the RMS strain in both phases for material shocked to 10.5 GPa. Recall that the RMS strain ε_{rms} serves as an indicator of the dislocation density. The initial ε_{rms} values in the 4 samples shocked to its respective pressure displayed good repeatability, with $\varepsilon_{rms} \approx 0.01250 \pm 2 \times 10^{-5}$ for both 8 GPa and 10.5 GPa cases, indicating that the initial dislocation density in the ω phase was not shock pressure sensitive.

260 In contrast, the RMS value for the α phase was $\sim 15\%$ higher in the 10.5
261 GPa shocked samples than in the 8 GPa shocked sample (≈ 0.017 vs ≈ 0.014
262 respectively).

263 As described in Section 2, in order to explore the effects of thermal his-
264 tory on the microstructure evolution, a second set of samples was heated to
265 503K but at a slower heating rate (0.25K/s instead of 1.5K/s). Figure 6
266 shows a comparison of the evolution of α volume fraction for the 10.5 GPa
267 samples. The 8 GPa sample displayed similar behavior and will be omitted
268 for brevity. As seen in the figure, the fast-ramp sample showed a higher
269 initial transformation rate, transforming to a greater extent (higher α frac-
270 tion), and saturating to the new metastable state faster than the slow-ramp
271 samples. Furthermore, due to the slow heating-rate, a significant amount of
272 transformation is observed before the sample reaches 503K (negative time in
273 Figure 6 indicates heating). The differences in behavior clearly show that
274 the microstructure evolution in the two-phase material is strongly dependent
275 on the thermal history and the final temperature, further highlighting the
276 non-equilibrium nature of the microstructural state.

277 3.2. Microstructure Characterization

278 Figure 7 shows orientation and phase maps of $90 \times 90 \mu\text{m}$ regions in
279 the (8 GPa) as-shocked and annealed samples. For the annealed specimens,
280 samples were heated at 443K and 463K for a total duration of 21,336 and
281 19,145 seconds respectively. As seen in Figure 7b, the as-shocked α phase
282 grains are lamellar with grains that are reminiscent of deformation twins or
283 fine martensite. The ω grains (Figure 7c) make a relatively equiaxed matrix
284 in which the α lathes are embedded. The circled α grains in the as-shocked α

285 phase orientation map (Figure 7b) are hypothesized to be the un-transformed
 286 remnants of original pre-shock microstructure. This hypothesis is based on
 287 the morphological differences (equiaxed vs lath) and the presence of $\{10\bar{1}1\}$
 288 compression twins. Figure 7d confirms the lath like α grains exhibit an
 289 $(0\ 0\ 0\ 1)_\alpha \parallel (1\ 0\ \bar{1}\ 1)_\omega$ and $[1\ 0\ \bar{1}\ 0]_\alpha \parallel [1\ 1\ \bar{2}\ \bar{3}]_\omega$ orientation relationship
 290 with the equiaxed parent ω , as reported in previous studies on shocked Zr
 291 [21]. That this relationship is not observed in the circled grains in Figure 7b
 292 supports that they are untransformed remnants of the original structure and
 293 provides further evidence for the hypothesis that, during the shock, nearly
 294 all of the initial α microstructure is transformed to ω and that majority of
 295 the observed α grains post-shock are children of the ω structure [39].

296 Figures 7e, 7f, and 7g show the microstructure after partial annealing at
 297 443K. As displayed, the α laths have coarsened considerably relative to the
 298 as-shocked microstructure. The area fraction of α from EBSD analysis in
 299 the partially annealed structure is 0.41 as opposed to 0.32 in the as-shocked
 300 structure. As measured orthogonal to the long axis of the α grain, the average
 301 lath thickness of the 443K anneal material is $0.79 \pm 0.30\ \mu\text{m}$ vs $0.51 \pm 0.21\ \mu\text{m}$
 302 in the as-shocked structure. This analysis disregards the 3D orientation of
 303 the laths with respect to the cutting plane, and is not meant to represent
 304 a true stereological width of the α grains, rather it is meant as a simple
 305 comparative metric between the as-shocked and annealed microstructures
 306 (assuming that the crystallographic texture of the α phase is fairly uniform
 307 and that there is no preferential growth of different α variants at different
 308 temperatures). In contrast, the material heated at 463K (Figures 7i, 7j, and
 309 7k) has a microstructure composed of both coarsened and very fine laths. In

fact, the average lath thickness is nearly identical to the as-shocked material
 (0.50 $\pm$ 0.16 μ m) despite transforming to 0.74 area-fraction α . This implies
 that both growth of the existing α grains and nucleation of new α laths
 is occurring at 463K, but that no or very limited nucleation of new α is
 taking place at 443K and that the transformation is progressing completely
 by the growth of existing α grains. Figures 7i and 7l, show that the expected
 α/ω orientation relationship is maintained during the nucleation and growth
 process.

4. Discussion

From purely thermodynamic considerations, the α and ω phases in a pure
 Zr material should not co-exist at standard temperature and pressure (STP)
 conditions. Despite the α phase being the equilibrium structure at STP, it
 is well established that post shock microstructures with ω fractions $\geq$ 0.8
 are metastable at STP conditions for years following a shock induced phase
 transformation [9].

The working hypothesis in developing the above experimental study was
 that the complex dislocation state in the ω phase arrests the reverse trans-
 formation, and that the reduction of dislocation density in the ω phase with
 sufficient heating allows the reverse transformation to advance. These ar-
 resting dislocation structures are postulated to have been mobile in the pre-
 shock microstructure but are likely sessile in the ω phase. Several critical
 questions directly follow from this working hypothesis. These include: i)
 What is the asymptotic behavior when the material is isothermally heated?
 ii) How sensitive is the material response to thermal history and heating

334 rate? and iii) What is the balance between nucleation of new α grains and
335 growth of existing α grains? The heating experiments described above were
336 developed to begin addressing these questions and particularly to provide the
337 required data for the development of microstructure sensitive kinetic models
338 of the transformation. The X-ray and EBSD analysis results indicate a more
339 complex transformation behavior than suggested by earlier experiments and
340 providing critical insight into the microstructure evolution during heating.

341 Several distinct martensitic or shear/shuffle transformation mechanisms
342 have been proposed for the forward transformation [3, 6, 19, 20, 21, 22].
343 Focusing on Figure 3a and 3b, attention is immediately drawn to the tem-
344 perature dependence of the initial α volume fraction growth rates and the
345 extent of reverse transformation from $\omega \rightarrow \alpha$. In general, a strong tempera-
346 ture dependence of transformation rate between two phases without change
347 in chemical composition would indicate a massive or short range-diffusional
348 type mechanism; where the rate would be directly related to the difference in
349 phase fraction. However, comparing the heating of samples in Figures 3c and
350 3d reveals a cross over of α volume fractions between the two different shock
351 loaded samples. Despite having similar α phase concentrations at the cross-
352 ing point, the transformation rates still differ, indicating that the driving
353 force is not solely based on the concentration difference between the phases,
354 but also on the microstructure of the material. Keeping this in mind, the
355 observation of initially high transformation rates followed by a plateau to a
356 metastable state with significantly retained ω also indicates a limiting of the
357 reverse transformation at STP conditions due to the microstructural state
358 of the ω phase. This idea, taken together with the low self-diffusivity of Zr

at a low homologous temperature ($10^{-23}m^2/s$ at 800K)[40], strongly points to a shear mechanism for the the reverse transformation as well. One can then deduce that the temperature dependency is sourced from dislocation of defect pinning within the ω phase, although the exact dislocation reactions and mechanisms, e.g. local dislocation annihilation, cutting or unlocking, which facilitate the transformation are still unknown.

A dislocation mediated shear mechanism is consistent with very recent atomistic modeling by Zong et al., informed by preliminary analysis of the data presented here [41]. In that work the evolution of ω fraction is fit to a modified Kohlrausch-Williams-Watts equation [42] or stretched exponential of the form $\eta(t) \propto t^{-\alpha} \cdot \exp\left(-\left(\frac{t}{\tau(T)}\right)^\beta\right)$, where η is the ω fraction, $t^{-\alpha}$ is an pre-factor that accounts for non-thermally activated events, T is the absolute temperature, and τ is the relaxation time or time for $\approx 37\%$ of the transformation to take place. The produced fits show that the α was close to zero indicating that that the thermal contribution to the kinetics was small and the exponent $\beta \approx 0.5$ indicating that the kinetics are analogous to other “glassy” or “frustrated” systems and not consistent with Johnson-Mehl-Avrami-Kolmogorov nucleation and growth [43, 44]. The predicted kinetics of the $\omega \rightarrow \alpha$ transformation in Ti with a simulated shocked microstructure via molecular dynamics was qualitatively consistent with the experimental results for Zr. However, in that work Zong suggests that nucleation is energetically preferable to growth by interface migration, which is inconsistent with our EBSD results which show significant growth at 443K without observable nucleation.

Recall that the RMS strain values qualitatively track the dislocation den-

384 sity in each phase. Upon heating, ε_{rms} decreases quickly in both samples and
 385 both phases as evidenced in Figure 5. The evolution of dislocation densities
 386 in both phases are distinct – the rate at which ε_{rms} decreases in the α phase is
 387 strongly temperature dependent, whereas the decrease of ε_{rms} in the ω phase
 388 showed minimal temperature dependence, with the exception of the heating
 389 at 443K. This points towards distinct mechanisms behind the evolution of
 390 the microstructure in the individual phases.

391 For the α phase, we suggest that the reduction of the dislocation density
 392 is due to the creation of incipient α phase material with a relatively lower
 393 dislocation density, resulting in a reduction of the average dislocation den-
 394 sity in the α phase. If we assume that the α phase present following the
 395 shock (ν_{α_0}) has a fixed RMS strain (ε_{α_0}) and that the incipient α (ν_{α_i}) also
 396 has a fixed lower RMS strain (ε_{α_i}), then the average RMS strain could be
 397 approximated by a weighted average of the two components, given by:

$$398 \quad \varepsilon_{rms,\alpha} = \frac{(\varepsilon_{\alpha_0}\nu_{\alpha_0} + \varepsilon_{\alpha_i}\nu_{\alpha_i})}{\nu_{\alpha_i} + \nu_{\alpha_0}} \quad (1)$$

399 Using an appropriate initial volume fraction of α for each material – 0.4 and
 400 0.2 in the material shocked to 8 and 10.5 GPa respectively, and likewise as-
 401 signing RMS strain values of 0.014 and 0.016 to the as-shocked α and 0.0035
 402 to the incipient α , an RMS strain averaged over the α phase is calculated
 403 and plotted in Figure 8. The simple calculation fits the data observed during
 404 heating of the material shocked to 10.5 GPa very well, and is reasonable for
 405 the 8 GPa material. It is worth re-stating at this point that the curve for
 406 annealing at 443K remains an exception, reinforcing the notion of a different
 407 mechanism (growth vs nucleation) for the reverse transformation depend-
 408 ing on a temperature threshold. More on this mechanism difference will be

409 presented in the discussion of EBSD results.

410 Figures 8b and 8d show the evolution of the α volume fraction as a func-
411 tion of the ω RMS strain. The arrows indicate the direction of evolution
412 on these plots. Notice that the dislocation density in the ω phase decreases
413 considerably before the reverse transformation begins in earnest, indicated
414 by the significant increase in α . Indeed, it appears that the defect level in
415 the ω microstructure must be reduced to a critical value, corresponding to an
416 RMS strain of approximately 0.01, for the initiation of the reverse transfor-
417 mation. The mechanism behind the removal of these arresting dislocations
418 is still unknown, but it can be further inferred that the energy required to
419 remove the arresting dislocations in the ω phase contributes significantly to
420 the nucleation energy barrier.

421 In comparing the microstructure states for different shock loadings, the
422 initial α dislocation density was about 15% higher in the 10.5 GPa sample
423 than in the 8 GPa sample.(Figures 5a and 5c). During the initial stages of
424 the shock prior to the transformation from α to ω , the initial α microstruc-
425 ture undergoes substantial plastic deformation and accumulates a significant
426 dislocation density. On transforming to ω , these accumulated defects become
427 immobile or have very limited mobility. The difference in initial dislocation
428 density with shock pressure in the α phase can be understood on the basis
429 that reverse transformation occurs simultaneously during the shock loading,
430 nucleating incipient α with a significantly lower density than the parent ω .
431 This supposition is supported for the initial EBSD analysis which shows much
432 less lattice curvature in the newly transformed lenticular α grains post shock
433 than in the equiaxed heavily deformed and twinned grains retained from the

original microstructure. The difference in as-shocked dislocation density with shock pressure is likely due to a combination of the reverse transformation occurring to a lesser extent in the 10.5 GPa samples during the shock itself, a higher fraction of the heavily deformed α being retained from the initial microstructure and additional plastic deformation in the re-transformed α in the 10.5 GPa sample. Additional EBSD and other microscopy is required to deconvolve and individually evaluate these effects.

Unlike the α phase, the initial dislocation density for the ω phase was approximately equal in both the 8 GPa and 10.5 GPa shocked samples (Figures 5b and 5d). It seems possible that the defect content in the ω saturates and does not further increase with increased peak pressure, however there is insufficient information to confirm this. Despite having similar initial dislocation densities in the ω phase for both shock pressures, the transformation rates and extent of transformation for different shock pressures were different. The most likely explanation is that the dislocation state of both samples are significantly different, thus requiring affirmation in a future study to determine the type and nature of dislocations present and their likely precursors in the pre-transformed α . VISAR (Velocity Interferometer System for Any Reflector) analysis of the data from the shock experiments suggests that while both samples were above the transformation pressure for the same amount of time ($\approx 0.7\mu s$) [39], the 8 GPa sample took almost this entire time to complete the forward transformation while the 10.5 GPa sample completed the forward transformation in $\approx 0.1\mu s$. Due to the more rapid nature of the shock transformation in the 10.5 GPa sample, the dislocations in the original α microstructure may have been “caught” in the transformation and become

459 sessile in the ω phase (stuck on planes with limited mobility). In contrast,
 460 slower transformation in the 8 GPa sample allowed more time for the α
 461 dislocations to move and react and avoid being trapped during shock trans-
 462 formation. Upon unloading, regions with dislocation densities lower than the
 463 critical dislocation density serve as nucleation sites for new α , leaving behind
 464 ω regions with high dislocation density. It is likely that the slower moving
 465 transformation front in the 8 GPa samples pushed or swept dislocations in
 466 front of it, leaving larger regions of low defect density ω which could readily
 467 nucleate α immediately after the shock. An interesting thought to pursue in
 468 the future is that of the ω regions in the 8 GPa sample having more complex
 469 dislocation structures and locks than the 10.5 GPa sample due to the amount
 470 of time afforded for dislocation kinetics during the forward transformation
 471 during shock. Furthering this thought, although the average dislocation den-
 472 sity of both differently shocked samples in the retained ω are closely similar
 473 and above a certain critical threshold, the 10.5 GPa sample would contain
 474 more regions with simpler structures and marginal stability which is more
 475 easily overcome during annealing. While speculative, this theory would ac-
 476 count for the higher initial transformation rates at all temperatures and the
 477 higher α fraction saturation meta-stable state for the 10.5 GPa sample. Con-
 478 firmation of this hypothesis would require significant resources allocated to
 479 defect microscopy to determine the local dislocation state in the shock loaded
 480 material.

481 The dependence of the hysteresis on thermal history and heating rate
 482 (see Figure 6) further suggests that the mechanisms by which the arresting
 483 dislocations become mobile or are annihilated is complex or can take multiple

484 paths. Samples heated at 1.5 K/s transformed to a more significant degree
485 than the samples heated at 0.25 K/s, indicating that the various paths are
486 not equivalent in a kinetic sense. When heated at a slow rate, the defects may
487 react by a variety of different pathways and possess a microstructure different
488 to that of the as-shocked state when the sample finally reaches the target
489 temperature. At higher temperature ramp rates, there is insufficient time for
490 these initial low temperature reactions to occur and the dislocation structure
491 at the target temperature becomes more representative of the as-shocked
492 microstructure. As more energy becomes available at higher temperatures,
493 it is likely that more complete dislocation reactions are possible. At slower
494 ramp rate, significant dislocation debris or various locks may be formed with
495 energy barriers which require even higher temperatures to overcome.

496 Figures 7f and 7j compare the α phase of 8 GPa peak pressure samples
497 heated at 443K and 463K respectively. The samples at 443K did not show any
498 visible evidence of nucleation of new α , in contrast with the samples at 463K
499 which displayed significant nucleation of lenticular α laths. Knowing that
500 growth in α volume fraction occurs at both these temperatures, albeit with
501 differing rates (refer to Figure 3b), it can be inferred that there is a higher
502 energy barrier associated with nucleation in comparison to that of growth
503 from existing α . This is again counter to the predictions of Zong et al [41],
504 demonstrating the need for further in-situ experimental studies to inform
505 the theory and modeling. At sufficiently high temperatures, nucleation of
506 α from ω parents dominates the reverse transformation, with the growth of
507 the original α happening simultaneously. Unsurprisingly, the nucleation of
508 α from ω parents occurs more readily near an existing α/ω interface than in

509 the bulk of a ω grain or ω/ω grain boundary. However, in conjunction with
510 the working hypothesis, this also means that mechanism responsible for the
511 removal of arresting dislocations in the ω phase also occurs at similar sites.

512 As a final mention, the results derived from EBSD analysis of the as-
513 shocked microstructure reaffirmed the preferred orientation relationship ini-
514 tially observed by Song et al. [21]. This orientation is maintained throughout
515 growth of the α phase. In samples heated 463K and above, the newly nu-
516 cleated lenticular α grains are quite similar to deformation twins in Zr, with
517 respect to both their morphology and in the propensity to apparently initiate
518 at interfaces and quickly propagate across the grain and terminate at either
519 another ω grain boundary or another α lath.

520 5. Conclusions

521 In-situ X-ray diffraction experiments were undertaken to better under-
522 stand the stability and transformation behavior of α/ω microstructure result-
523 ing from shock loading in pure Zr. Under standard conditions, the α phase
524 is clearly the equilibrium phase. However, these experiments suggests that
525 microstructure elements, presumably complex dislocation structures, arrest
526 the reverse transformation and prevent the system from returning to equi-
527 librium following shock loading, leading to samples with significant retained
528 ω phase.

529 The current experiments quantitatively monitored that phase evolution
530 during isothermal annealing and semiquantitatively followed the evolution of
531 dislocation densities (via the evolution of RMS strain values) during isother-
532 mal annealing at 443, 463, 483, and 503K. At sufficiently high temperatures,

533 the initial transformation occurred rapidly, quickly slowing down to reach
534 a new metastable equilibrium state. Further, it was found that the sam-
535 ples shocked to higher peak pressures transformed at higher initial rates and
536 asymptotically attained higher α fractions despite starting with significantly
537 lower α in the as-shocked state.

538 The RMS strain was related to dislocation density and used to capture
539 the evolution of microstructure of the α and ω phases in a semi-quantitative
540 manner. The analysis suggests a high dislocation density in both the α and ω
541 phases following shock loading. The dislocation density of the α phase drops
542 significantly during heating, however we suggest that this is likely due to the
543 growth of new α phase with low dislocation density rather than significant
544 recovery or annealing in the α phase during heating. In contrast, there is
545 an observed drop in dislocation density in the ω phase with a lesser extent
546 compared to the α phase. It is postulated that the transformation is arrested
547 due to complex dislocation structures in the ω phase “pinning” or preventing
548 the reverse transformation. This is supported by the observation that the
549 reverse transformation initiates at a critical value of the RMS strain in the
550 ω phase. As these complex dislocation structures are removed locally, the
551 material proceeds to transform to α . The relatively high concentration of
552 defects in the ω phase following heating, along with the observation of a
553 metastable state with retained ω reflects that the material is still prevented
554 from transforming completely back to α .

555 Some initial EBSD was performed to characterize the morphology of the
556 as-shocked and partially annealed samples. The expected α/ω orientation
557 relationship is observed in the as-shocked state and this relationship is main-

558 tained throughout the transformation (growth and nucleation). Further, it
559 was found that at 443K, only the growth of the existing α phase was ob-
560 served while at 463K both nucleation of new α and growth of existing α
561 grains were readily observed. This suggests that nucleation of new α grains
562 requires overcoming a significant energy barrier or equivalently, the growth
563 of existing α happens via a lower energy pathway than nucleation.

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Figure 1: The evolution of the α volume fractions during isothermal heating of the material shocked to (a) 8 GPa and (b) 10.5 GPa peak pressures as a function of time at varying temperatures. Markers indicate volume fractions from X-ray analysis, solid lines indicate 2nd order rational function fit $((c_1 t^2 + c_2 t + c_3)/(t + c_4))$ to the experimental data. For visual clarity, markers are only shown every 10 or 15 data points. The large gap between markers in the 463K experimental data is due to the X-ray beam loss described in section (2.2). The time $t = 0$ s corresponds to when the sample reaches 95% of the target temperature, and negative time corresponds to the heat-up phase.

(a) 245C Spectra at $t = 0$ Fs

(b) 245C Spectra at $t = 3523$ s

Figure 2: The integrated X-ray spectra and applied Rietveld fittings for 10 GPa samples subjected to 230C heating (a) prior to heating and (b) after heating for 3523 secs. Note: The Y-axis intensity is square-root of the original intensity.

(a) 443K

(b) 463K

(c) 483K

(d) 503K

Figure 3: Comparing the effects of peak pressure on evolution at a fixed temperature of (a) 443K, (b) 463K, (c) 483K, (d) 503K. Different time scales are applied here.

Figure 4: Semilog plot showing initial α fraction growth rates with respect to temperature.

Figure 5: Comparison of the evolution of the RMS strains in both α and ω phase in the annealing of samples shocked to a peak stress of 8 GPa, (a)-(b) and 10.5 GPa (c)-(d). For visual clarity, markers are only shown every 10 or 15 data points. Note again that the RMS strain is an indicator of dislocation density.

Figure 6: Comparison of the evolution of the α fractions for different ramp rates to 503K. The slower ramp rate was at 0.25 K/s, the faster ramp rate was at 1.5 K/s.

Figure 7: EBSD images obtained from OIM Software, with the shock direction normal to the analyzed surface. (a)-(d) correspond to the non-annealed sample, (e)-(h) correspond to sample annealed at 443K, and (i)-(l) correspond to sample annealed at 463K. The Phase and Interface figures (d), (h), and (l) are color coded as such: α -white, ω -light green. The blue lines mark interfaces with the $(0\ 0\ 0\ 1)_\alpha \parallel (1\ 0\ \bar{1}\ 1)_\omega$ and $[1\ 0\ \bar{1}\ 0]_\alpha \parallel [1\ 1\ \bar{2}\ \bar{3}]_\omega$ orientation relationship.

(a) ϵ_{rms} in α phase (8GPa)

(b) ϵ_{rms} in ω phase (8GPa)

(c) ϵ_{rms} in α phase (10.5GPa)

(d) ϵ_{rms} in ω phase (10.5GPa)

Figure 8: Comparison of the evolution of RMS strain relative to the α phase volume fraction evolution in the annealing of samples shocked to a peak stress of 10.5 GPa in the (a)–(c) α phase and (b)–(d) ω phase. The theoretical fit in (a) and (c) is obtained using a weighted average method over the original and incipient α phase.