

Transformation and Twinning Induced Plasticity in Metastable Ti-Mo Alloys under High Strain Rate Deformation

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1 Abstract

The development of metastable titanium (Ti) alloys provides an unprecedented opportunity to expand their use in plasticity and damage critical applications like protective structures. However, such applications require knowledge of quasi-static to dynamic mechanical behavior, which is currently lacking. Here we perform in-situ, ultrafast synchrotron x-ray diffraction during high strain rate (Kolsky) pressure bar testing in tension and post-mortem electron microscopy to study TRansformation Induced Plasticity (TRIP) and TWinning Induced Plasticity (TWIP) in metastable Ti-Mo alloys. Two alloys, namely Ti-12Mo and Ti-15Mo (wt.%), were selected for study having different β -phase chemical stabilities. TWIP was observed in both Ti-12Mo and Ti-15Mo by in-situ synchrotron diffraction during high strain rate testing. Post-mortem microstructural characterization also revealed the presence of TRIP in Ti-12Mo. TWIP in Ti-15Mo was found to under-perform in terms of total elongation compared to TRIP/TWIP in Ti-12Mo. Ti-12Mo exhibited an average elongation of 17% compared to only 12% for Ti-15Mo with deformation at 1000 s^{-1} . TRIP resulted in significantly finer microstructure evolution and alleviated local strain accumulations in Ti-12Mo, suggesting TRIP can be used to tune available strength/ductility combinations in metastable Ti alloys under high strain rate deformation conditions.

2 Introduction

Ti alloys have been widely used in structural components in aerospace applications, due to their high strength to weight ratios. Recently, interest in a novel class of Ti alloys, namely metastable β alloys, has been increasing, due to promising work hardening and ductility behaviors [1, 2, 3, 4]. Metastable β Ti alloys have long been known to exhibit microstructure evolution during quasi-static deformation [5]. Only recently has the propensity for TRansformation Induced Plasticity (TRIP) and Twinning Induced Plasticity (TWIP) been studied with the aim of exploiting these

mechanisms to achieve desirable strength/ductility combinations by engineered work hardening response [6]. A similar approach has been successfully used to develop a myriad of useful advanced high strength steels for automotive applications, driven by the need for lightweighting and improved crash performance [7, 8]. Metastable TRIP/TWIP Ti alloys may similarly open the door to mechanical property combinations for the aerospace and defense sectors. For strong, ductile and tough metastable Ti alloys to become ubiquitous, however, understanding TRIP and TWIP and the effect of strain rate, temperature, and strain path are crucial.

Recent publications have highlighted that TRIP in Ti alloys is less favorable for obtaining higher yield stresses compared to TWIP for quasi-static tensile deformation [4, 9, 10]. On the other hand, TRIP has been revealed as a powerful mechanism for internal stress relaxation, specifically where twin/twin interactions occur [4] and at twin and secondary phase boundary interactions [9] where local stresses may be high. Nevertheless, the trend seems to be moving toward TWIP-dominant alloys, based upon our current understanding [3, 4, 11] from quasi-static mechanical testing and microstructure characterization. For the broader use of metastable Ti alloys to become a reality, specifically in applications requiring substantial plasticity or damage tolerance, quasi-static mechanical behavior may be a misleading experimental basis for alloy and microstructure design, given that plasticity and fracture are inherently dynamic processes in real-world scenarios. This implies the dynamic behavior of TRIP/TWIP alloys needs to be well-understood, especially if they are to be utilized in crash or blast resistant scenarios where energy absorption is essential. In cases such as these, there are no substitutes for high strain rate experiments.

Only a few high strain rate deformation studies have been published to date on metastable β Ti alloys, mostly concentrating on the strain rate sensitivity from quasi-static strain rates up to the low end of intermediate strain rates (10^{-5} to 10^{-1} s^{-1}) [12, 13, 14]. While the data presented in these studies are useful, they

do not extrapolate to high strain rate behaviors because different mechanisms like dislocation generation and motion may dominate in different strain rate regimes [15]; in addition temperature effects (i.e., adiabatic heating), pathway dependent microstructure evolution is also not well understood. Ahmed et al. [12] have published one of the only studies where strain rates up to 10^2 s^{-1} were tested on an $\alpha + \beta$ TRIP/TWIP Ti-10V-3Fe-3Al (wt. %) alloy. While their study was performed in compression, they concluded that TWIP was dominant over TRIP as strain rate increased, although all mechanisms were found to be active over the entire strain rate range regime studied (i.e., no TRIP suppression occurred). To the authors' knowledge, only two studies have looked at metastable β Ti alloys [16, 17] deformed at strain rates on the order of 10^3 s^{-1} and only in compression. Both studies found evidence of TRIP and TWIP in the deformed state; increased work hardening was provided by both mechanisms. Specifically, Yang et al.'s study on Ti-8.5Cr-1.5Sn (wt. %) [16] showed that TRIP and TWIP are a powerful deterrent to the formation of adiabatic shear bands. To date, no studies have systematically compared different alloys and chemical phase stabilities under high strain rate deformation conditions, which controls the microstructure evolution.

Here we investigate the microstructure evolution in two Ti-Mo alloys with different β -phase chemical stabilities using ultrafast synchrotron x-ray diffraction during high strain rate tensile deformation. Complementary post-mortem electron microscopy was also performed. **These results reveal the importance of TRIP in maintaining higher elongations, and thus the potential for energy absorption, and suggest that TRIP-capable alloys (Ti-12Mo) out-perform TWIP alloys (Ti-15Mo) at high strain rates, due to the central role that martensite plays in local stress relaxation.**

3 Methodology

3.1 Ultrafast Synchrotron X-Ray Imaging and Diffraction during High Strain Rate Deformation

Modified (Kolsky) pressure bar testing together with high brilliance, high-flux, ultrafast synchrotron x-ray imaging and diffraction was performed at sector 32-ID-B at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL). The details of the modified (Kolsky) pressure bar apparatus have been reported elsewhere [18, 19, 20]. A schematic of the experimental setup is shown in Figure 1 a), which provides the simultaneous capture of x-ray imaging, diffraction and mechanical test data. A Photron FASTCAM SA-Z type 2100K-M-64G camera using a LuAG:Ce scintillator (100 thick) and a 10X microscope objective (Mitutoyo long WD) at a frame rate of 50400 frames per second (fps) was used for imaging. Diffraction data were captured in full transmission geometry with a Shimadzu HPV-X2, coupled to an image intensifier and an LSO:Ce scintillator (300 thick), at a frame rate of 298507 fps. The mechanical test data were acquired with an oscilloscope at an acquisition rate of 10^8 Hz. For the x-ray setup, an undulator gap of 12 mm was used, providing a "pink-beam"-like condition with a maximum flux at a wavelength of 0.512 Å and a characteristic, asymmetric intensity profile around the harmonic energies, with a tail dropping off in intensity gradually toward the lower energies. A beam size of 2 mm wide by 1 mm in height was used to illuminate the gauge length of each tensile specimen. This large beam size, combined with the tail of the energy profile, lead to significant instrument and source broadening of the diffraction peaks, as discussed in the supplementary materials. The detector-to-sample distance was roughly 0.6 m, and the resulting pixel size was roughly 32 μ m on the detector. Different detector positions were used to obtain different resolutions and q-space ranges. The sample geometry is also provided in Figure 1b.

Standards of pure Al and pure Ta foil were used to calibrate the detector position.

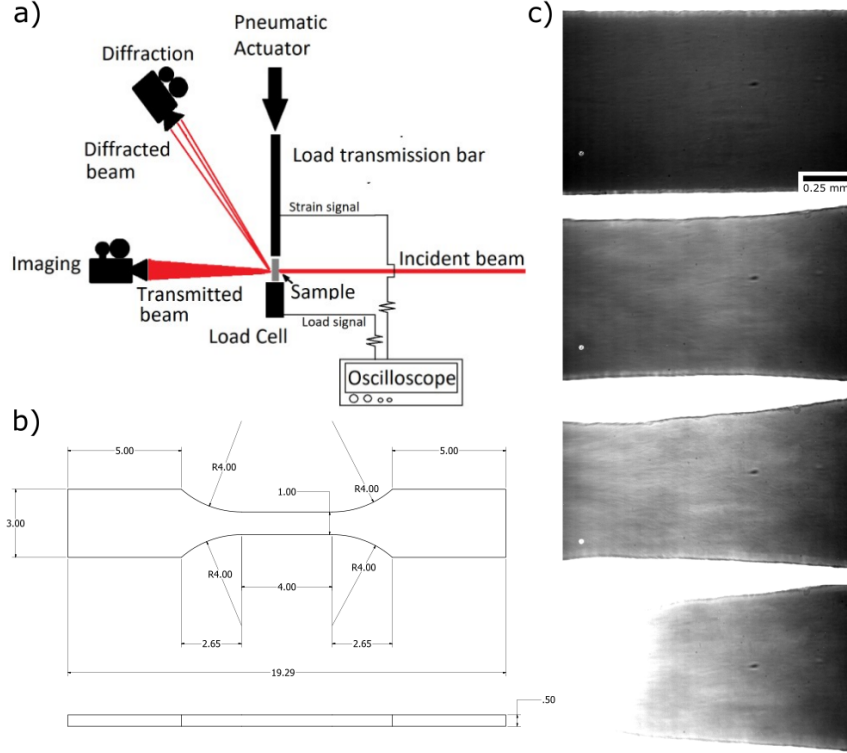


Figure 1: a) Schematic of the modified (Kolsky) pressure bar setup at the APS at ANL. b) Tensile test geometry used in this work (dimensions in mm). c) Example synchrotron x-ray radiographs obtained during tensile deformation, showing necking and fracture in the gauge length. This specimen was deformed at a strain rate of 10^3 s^{-1} . The observed contrast is associated with microstructure evolution.

129 Analysis of the raw diffraction data was performed using High Speed Polychromatic
 130 Diffraction (HiSPoD), a MATLAB program developed at sector 32-ID. The details
 131 of the program and its implementation are provided elsewhere [21]. The program
 132 was used to calibrate the beam position and detector/sample geometry, allowing for
 133 the calculation of a q-space map (radial coordinates) for integration of the diffrac-
 134 tion patterns.

135

136 The modified (Kolsky) pressure bar apparatus used strain gauges to capture the
 137 incident and reflected pulses. Due to space restrictions in the hutch, the load data
 138 were measured using a fast-response quartz load cell instead of a typical transmission
 139 bar. Measurements made during testing of a brittle ceramic indicated the decay time
 140 of the load signal was roughly $0.35 \text{ V}/\mu\text{s}$. In this study, strain rates of roughly 10^3
 141 and $2 \times 10^3 \text{ s}^{-1}$ were achieved. Stress and strain data were averaged for all specimens

of each alloy by averaging the stress value of each replicate for each strain increment.

3.2 Sample Preparation, Post-Mortem Microstructure Characterization and Quasi-static and Intermediate Strain Rate Testing

The Ti-15Mo specimens for testing at the APS were machined from a 15.8 mm diameter bar stock, whereas the Ti-12Mo specimens were wire electro-discharge machined from a rolled sheet. The flat specimens extracted from the round bar of the Ti-15Mo alloy likely exhibited slight variations in texture, due to the sample extraction strategy. In all cases, the specimens were ground to a final thickness of 0.5 mm using 320 grit SiC metallographic paper. The tensile specimens were wrapped in Ta foil and encapsulated under vacuum in quartz tubes to minimize contamination and oxygen pick-up. Ti-12Mo specimens were held at 1093 K for 30 min. Ti-15Mo specimens were held at 1073 K for 30 min. All samples were immediately quenched into room temperature distilled water by breaking each quartz capsule. The resulting microstructures for both alloys was the β -phase with an equiaxed grain structure and average grain size of roughly 35 μm for Ti-15Mo and 45 μm for Ti-12Mo, as determined by Electron Backscatter Diffraction (EBSD). The details of the EBSD measurements are provided later in this section. With a total gauge thickness of 0.5 mm and grain sizes in the range of 40 μm , we can assume that roughly 10 grains were present through thickness in each specimen. Additionally, it can be estimated that roughly 5×10^4 grains were in the gauge section, and only about half that number were illuminated by the beam. This number is considered a conservative upward-bound estimate, since a spherical grain shape was assumed, which leads to overestimating the number of grains present in a given volume.

Quasi-static tensile testing was performed at the Colorado School of Mines with an electromechanical Alliance load-frame at a strain rate of 10^{-3} s^{-1} using a 1 in

170 Shepic type extensometer. Intermediate strain rate tests were performed on a hy-
171 draulic MTS load-frame using a 1 in MTS blade-type extensometer. All quasi-static
172 and intermediate strain rate tests were performed using ASTM E8 standard [22]
173 subsize geometry tensile specimens with a gauge length of 25.4 mm and a cross
174 section of 3.175 mm by 6.35 mm.

175

176 An FEI Helios 600i dual-column Focused Ion Beam (FIB)/Field Emission Scan-
177 ning Electron Microscope (FESEM) was used to make lift-outs for Transmission
178 Electron Microscopy (TEM). TEM was performed in an FEI Talos F200X CTEM/STEM
179 at 200 kV. EBSD specimens were prepared by electropolishing at 20 V and 253 K,
180 using a mixture of 6 % perchloric and 4 % hydrochloric acids diluted with a 2 to
181 1 mixture of methanol and butoxyethanol. EBSD scans were performed in an FEI
182 Helios 600i dual-column FIB/FESEM at an accelerating voltage of 20 kV with 11
183 nA of current. No post-scan clean-ups were performed.

184

185 4 Results

186 4.1 Mechanical Properties

187 4.1.1 Quasi-Static and Intermediate Strain Rate Mechanical Properties

188 Engineering stress versus engineering strain curves obtained from quasi-static (10^{-3}
189 s^{-1}) and intermediate ($10^{-1} s^{-1}$) strain rate tests of Ti-15Mo are presented in Fig-
190 ure 2 a). A hundred fold increase in deformation rate leads to an increase in yield
191 stress and reduction in total and uniform elongation (Figure 2 a) and b)). Figure
192 2 b) shows true stress versus true strain of Ti-15Mo and Ti-12Mo. Ti-12Mo has
193 lower yield stress and overall strength at comparable strain levels, but greater uni-
194 form elongation and maximum work hardening rate (WHR) compared to Ti-15Mo.
195 WHR was determined using the first derivative of the stress ($\frac{d\sigma}{d\epsilon}$) and a running aver-
196 age over 50 points for smoothing. The main difference between the two alloys, apart

from composition, is the possible activation of TRIP in the Ti-12Mo alloy. TWIP was anticipated in Ti-15Mo, based upon phase stability. Composition likely plays a major role in the observed strength difference, as Mo is a potent solid solution strengthener in the β -phase of Ti [23]. The Ti-12Mo data shown in Figure 2 b) was chosen to reflect Ti-12Mo in the as-quenched, unaged state, where ω -phase hasn't significantly altered the mechanical response [24].

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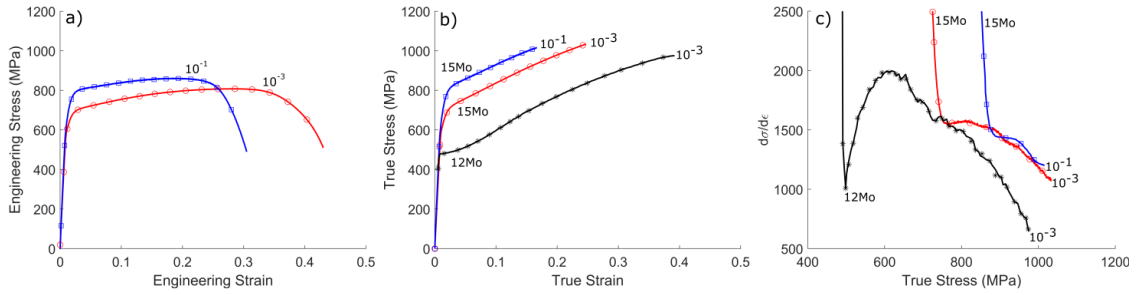


Figure 2: a) Engineering stress versus engineering strain curves for Ti-15Mo tested at quasi-static and intermediate strain rates of 10^{-3} and 10^{-1} s^{-1} . b) True stress versus true strain of Ti-15Mo and Ti-12Mo deformed in tension at a strain rate of 10^{-3} s^{-1} and Ti-15Mo tested at an intermediate strain rate of 10^{-1} s^{-1} . c) Instantaneous WHR as a function of true stress for the three conditions shown in b). The data for Ti-12Mo was taken from [11].

The true stress versus true strain response of Ti-15Mo is also shown in Figure 2 b) for quasi-static and intermediate strain rates. Interestingly, the WHR seems to be comparable between the two strain rates. However, an apparent reduction in uniform elongation correlates with a concurrent reduction in work hardening as flow stress approaches the ultimate tensile strength (UTS) of 1 GPa. Figure 2 c) shows the substantial difference in WHR at high flow stresses exhibited by both alloys. TRIP-capable Ti-12Mo exhibits an overall larger WHR over a larger stress range, but does not reach Ti-15Mo equivalent stress levels. The superior flow stress exhibited by Ti-15Mo is most likely due to the Mo solid solution strengthening in the matrix. When quasi-static and intermediate strain rates are considered for Ti-15Mo, it is clear that the WHR is similar for a given stress level after yielding. The work hardening behavior of Ti-15Mo does not appear to be strongly sensitive to strain rate. This finding supports the claim that plastic strain is mostly mediated

by dislocations, while work hardening is controlled by slip and twinning. This is discussed further later.

4.1.2 High Strain Rate Mechanical Properties

Figure 3 a) shows engineering stress versus engineering strain curves obtained from tensile testing of Ti-12Mo with the modified (Kolsky) pressure bar setup at the APS at ANL. Due to the inherent variability associated with these small scale, high strain rate tests, averaging of the curves was performed to obtain representative behavior. Two different strain rates were achieved during testing, roughly 10^3 and 2×10^3 s^{-1} . The difference between the two strain rates for the Ti-12Mo alloy is evident in Figure 3 a).

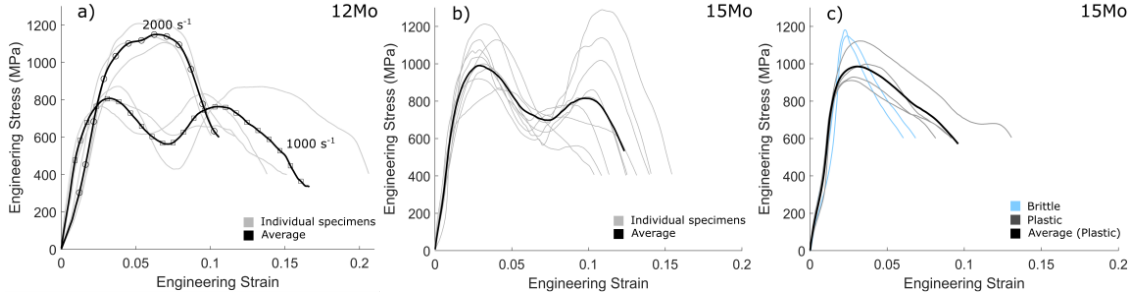


Figure 3: a) Engineering stress versus engineering strain curves for Ti-12Mo. The full black curves with symbols represent the average behavior of 5-10 tests, depending on alloy and strain rate. The stress-strain curves of individual specimens used in the average are shown in grey. b) Engineering stress versus engineering strain curves of Ti-15Mo deformed in tension at a strain rate of roughly 10^3 s^{-1} . Individual specimens are shown in grey, and the average behavior is shown in black. c) Engineering stress versus engineering strain curves of Ti-15Mo deformed at 2×10^3 s^{-1} . Curves in light blue represent outliers that exhibited no macroscopic ductility or "brittle" behavior, and were not used in the calculated average response shown in c).

Due to the inherently noisy nature of the high strain rate mechanical testing data, it is impossible to identify an exact uniform elongation and WHR. Even the exact moment of failure is hard to pinpoint, in large part due to the decay time of the load cell signal. To circumvent this difficulty, a cutoff stress equal to half the maximum stress was chosen as a comparative value for elongation (Figure 3). Sur-

234 prisingly, even at the highest strain rate, Ti-12Mo still exhibits measurable ductility.
235 The increase in flow stress exhibited between 10^3 s^{-1} and $2 \times 10^3 \text{ s}^{-1}$ is indicative
236 of the alloy resisting localization.

237

238 Figures 3 b) and c) show engineering stress versus engineering strain curves for
239 Ti-15Mo samples tested with the modified (Kolsky) pressure bar setup at strain
240 rates of 10^3 s^{-1} and $2 \times 10^3 \text{ s}^{-1}$. Figure 3 b) shows individual test specimen results
241 in light grey in the background; the average behavior is indicated in black. The
242 ductility and maximum stress vary amongst the individual test specimens at this
243 strain rate, with total elongation (cutoff stress set at 400 MPa) varying between 0.1
244 and 0.16. The variability in total elongation and maximum stress exhibited by these
245 specimens is, in part, due to slight variations in texture in the specimens mentioned
246 above. Another source of variability is the similarity between grain size and sample
247 thickness, i.e., only a few grains are present through thickness.

248

249 Curves for individual specimens of the Ti-15Mo alloy tested at the highest strain
250 rate of $2 \times 10^3 \text{ s}^{-1}$ were divided into 2 categories, as the mechanical behavior diverged
251 greatly. Specimens either exhibited limited ductility, or none at all, i.e., fracture oc-
252 curred before any plastic deformation could be measured. The latter are labeled in
253 light blue as "brittle" in Figure 3 c). Other specimens that exhibited macroscopic
254 plasticity were labeled as "plastic", and are represented in grey. They were used to
255 calculate the average curve shown in black. When comparing these data with those
256 collected at 10^3 s^{-1} , the yield stress is roughly equal, while elongation is decreased.
257 This behavior is indicative of having reached a strain rate at which localization is
258 consistently present at low plastic strain.

259

260 4.2 Ultrafast Synchrotron X-Ray Imaging and Diffraction 261 during High Strain Rate Testing in Tension

262 In-situ synchrotron x-ray diffraction data is presented for Ti-15Mo and Ti-12Mo in
263 Figure 4. In both cases, raw diffraction frames and integrated diffraction patterns
264 show clear evidence of grain refinement during deformation and texture evolution.
265 In Figure 4 a) and b), the first raw diffraction frames show large diffraction spots dis-
266 tributed radially, mostly centered around 4 nm^{-1} and 2.7 nm^{-1} in q-space. These
267 spots are the result of the initially large grain sizes in both alloys. As plasticity
268 evolves, the spots can be seen turning into full rings of diffuse intensity. The diffrac-
269 tion signal is a clear indication of microstructural evolution, leading to refinement
270 of the average size and change in orientation of the diffracting domains. The refine-
271 ment and re-orientation causes the diffracted photons to be more evenly distributed
272 in the ϕ , or azimuthal, direction on average. Secondly, the texture component of the
273 diffraction can also be seen to evolve. Figure 4 c) and d) show an integrated diffrac-
274 tion pattern as a function of engineering strain, where an integrated line-out for
275 each frame is calculated and stacked to form a heat map. It can be clearly seen that
276 the maximum intensity, which centers around 18° in 2θ space (or roughly 4 nm^{-1}
277 in q-space), rapidly shifts to form an increasing maximum near 12° (or roughly 2.7
278 nm^{-1} in q-space). These positions correspond to the $\{200\}_\beta$ and $\{110\}_\beta$ peaks,
279 respectively. The change in maximum intensity is indicative of a texture change
280 during deformation.

281

282 Possible explanations for this type of diffraction signal fall into two categories,
283 namely: 1) sub-grain formation and 2) deformation twinning. Theoretically, when
284 dislocation slip is active, cells eventually form, which upon further plastic straining,
285 evolve into sub-grains. Further slip tends to cause a rotation of the grains rela-
286 tive to the tensile axis, such that the slip planes move toward a low Schmid Factor
287 (SF), normally developing a $\{110\}$ type texture parallel to the tensile axis in BCC
288 crystals. On the other hand, deformation twinning will also lead to the same type

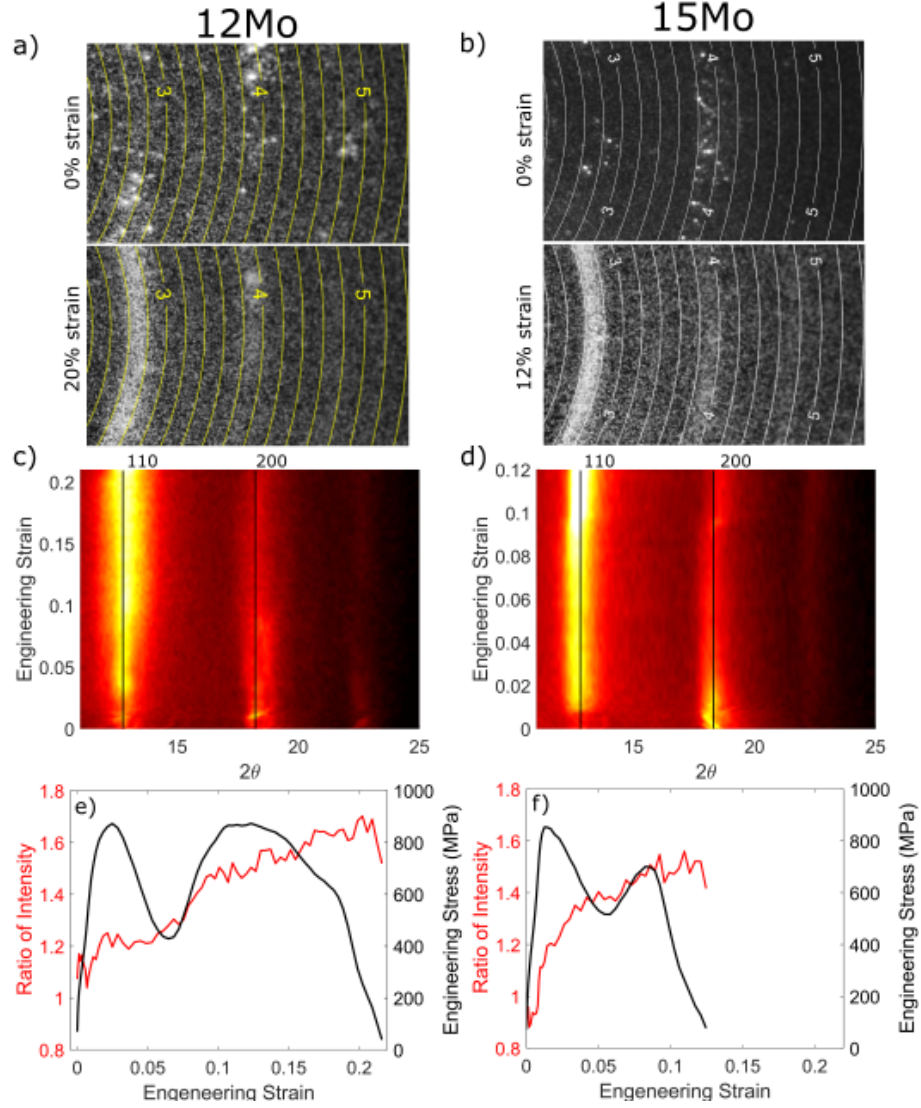


Figure 4: In-situ synchrotron x-ray diffraction during high rate tension testing. Raw diffraction frames showing the evolution from partial rings populated by spots to full diffuse rings in a) Ti-12Mo and b) Ti-15Mo. The white circular lines indicate contours of constant q , with 3, 4 and 5 nm^{-1} labeled and the equivalent strain is indicated next to the frame. Integrated diffraction pattern as a function of engineering strain for c) Ti-12Mo and d) Ti-15Mo. Black vertical lines indicate the theoretical position of β phase reflections and are labeled at the top of the map. Ratio of integrated intensity of the $\{110\}_{\beta}$ peak and the integrated intensity of $\{200\}_{\beta}$ peak shown in red on the left axis and engineering stress shown in black on the right axis, both as a function of engineering strain.

of "bulk-averaged" microstructure evolution and diffraction contrast. Indeed, the formation of a twin during plastic straining is the result of local re-orientation of a sub-volume within a grain to a specific twin relation. This process accumulated many times within a grain and over many 100s of grains will lead to a spots-to-ring type of diffraction signal, as shown. Due to the orientation dependence of deformation twinning during TWIP, a preferential texture may also evolve [25, 26]. In

summary, both alloys exhibit strong evidence of microstructural evolution, particularly grain refinement of the β -phase matrix.

In both integrated diffraction patterns presented in Figure 4 c) and d), intensity near the $\{110\}_\beta$ reflection becomes dominant over a large portion of the deformation. This indicates that plasticity is preferentially rotating the $\{110\}_\beta$ planes into the Bragg condition at the expense of others. No evidence of phase transformation was found for Ti-15Mo. Unfortunately, signs of deformation-induced phase transformation are not readily discernible in the diffraction data shown for Ti-12Mo. However, post-mortem characterization proves significant transformation occurred during deformation in Ti-12Mo, as shown in Figure 6, while it is completely absent in Ti-15Mo (Figure 13). Ti-12Mo forms a martensitic phase that is only a slight symmetry breakdown of the BCC β phase. As such, many diffraction peaks (especially those of highest intensity) either overlap or are very close to the β phase peaks in the region of the $\{110\}_\beta$ reflection. It is almost certain that the measured intensity for Ti-12Mo, as shown in Figure 4 a) and c), includes contributions from both phases, at least near the $\{110\}_\beta$ position. It should be noted that unequivocal evidence of transformation is not captured by the in-situ diffraction data, most likely due to angular resolution and dynamic intensity range limitations.

Figure 4 e) and f) show the ratio of the integrated intensity of the $\{110\}_\beta$ position and the integrated intensity of $\{200\}_\beta$ position, as well as engineering stress, as a function of engineering strain. The ratio of integrated intensities provides a visual indication of the rate at which texture evolves as a function of strain. It is interesting to note that the ratio gradually increases from the onset of loading up to fracture in Ti-12Mo. When compared to the engineering stress response, the gradual increase in ratio of intensities correlates with a high strain to fracture and nearly-constant flow stress (disregarding the load oscillation caused by ringing in the sample). This contrasts strongly with the sharp initial increase exhibited by Ti-15Mo up to an

324 engineering strain of roughly 0.05, followed by a significant decrease in the slope
 325 from 0.05 to 0.12, where fracture occurs. The noted slow-down in texture evolution
 326 exhibited by the Ti-15Mo specimen also correlates with a decrease in the load re-
 327 sponse and lower strain to fracture, as compared to the Ti-12Mo specimen. These
 328 data appear to indicate that slower overall rate of microstructural evolution in the
 329 Ti-12Mo alloy allows for higher plastic strains to be reached before fracture occurs.

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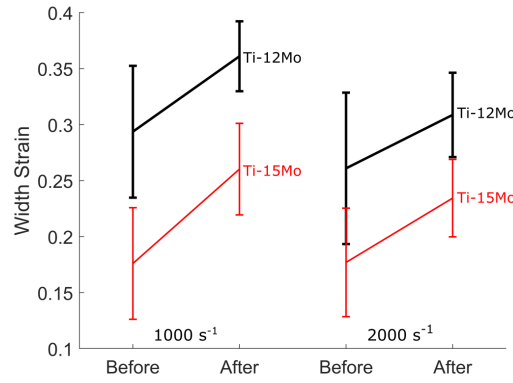


Figure 5: Comparison of the reduction in gauge section width taken from synchrotron x-ray imaging data. The average and standard deviation of the gauge section width at the narrowest point is shown in the last frame before fracture (Before) and after fracture (After) for both alloys tested at two strain rates (1000 and $2000\ s^{-1}$). Measurements were only included for samples where the entire width of the fractured gauge length was visible. When possible, measurements from both sides were averaged for a single specimen.

331 To help supplement the mechanical test data, synchrotron x-ray imaging data
 332 were used to measure and calculate reduction in area, or an approximate thereof
 333 (Figure 5). The radiography images allowed for the measurement of the reduction
 334 of the width of the gauge section, up to the moment of fracture, and the resulting
 335 fractured halves. Figure 5 shows the average (and standard deviation) of each alloy
 336 for both strain rates achieved during the in-situ experiments. The measurements
 337 indicated as "Before" were measured from the last frame, where the specimen is seen
 338 whole, whereas the measurements labeled "After" were obtained from a frame after
 339 fracture occurred. The average and error bars support the statement that Ti-12Mo
 340 exhibits higher ductility up to fracture at both 10^3 and $2 \times 10^3\ s^{-1}$.

341

4.3 Post-mortem Microstructure Characterization

The in-situ synchrotron x-ray diffraction data provide insights into the microstructure evolution that occurs during deformation. However, post-mortem characterization is necessary to understand the exact nature of the evolution, since diffraction is fundamentally a volume averaged technique. As such, fractured specimens from both alloys were characterized using EBSD and TEM to obtain important information about the fine-scale structural evolution.

4.3.1 TRIP/TWIP Ti-12Mo

Evidence of β -phase deformation twinning and transformation are present in the microstructure of deformed Ti-12Mo. Large deformation bands can be seen traversing grain boundaries with little re-orientation, plausibly due to accommodation in the neighboring grains [27, 28]. Figures 6 a) and b) show an IPF + IQ and Phase + IQ maps, respectively, of an EBSD scan where complex microstructure evolution can be seen, along with strong evidence of deformation twinning as the primary deformation product. Figure 6 c) shows an IQ map overlaid with $\{332\} \langle 113 \rangle$ twin boundaries. This map shows clear evidence of the concurrent presence of transformation and twinning in the deformed state, similar to the microstructure evolution reported for quasi-static deformation [1, 11]. Secondary twinning can also be seen to further sub-divide the β -phase matrix remaining between the primary twins in one of the grains on the left side of the map (indicated by the red box in Figure 6 c). Between the two black lines in Figures 6 b) and c) is the transition from untransformed primary twins in the bottom right to twins containing secondary transformation product, i.e. martensite in the upper portion of the same twinned laths. This is clear experimental evidence of secondary transformation within the primary twin accommodating further plastic deformation within the initially twinned crystal.

The bands contain more complex substructure that is too fine to be properly

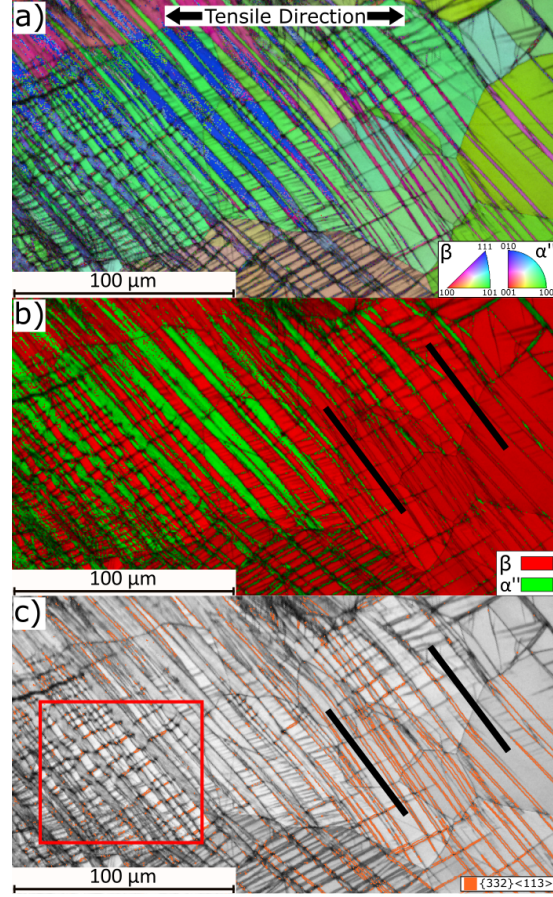


Figure 6: EBSD of a Ti-12Mo specimen deformed in tension at a strain rate of 10^3 s^{-1} taken outside the necked region of the gauge length. a) IPF + IQ map and b) Phase + IQ map c) IQ map with twin boundary overlay; orange represents $\{332\} \langle 113 \rangle$ twin boundaries. The phase map shown in b) clearly indicates the presence of martensite in the large bands traversing the grains, while the IQ map shown in c) shows the presence of $\{332\} \langle 113 \rangle$ twins. The red box highlights secondary twinning. The black lines in b) and c) highlights the transitions of primary twins forming internal secondary martensite. This EBSD scan shows how primary bands seemingly formed first as deformation twins, with subsequent internal transformation to martensite.

370 characterized by EBSD alone. A FIB liftout was produced to contain a cross section
 371 of one of these bands. Figure 7 shows the results of TEM characterization of one of
 372 these bands. The band exhibited a total of five of the six possible α'' variants, along
 373 with residual β -phase. Figures 7 b) through c) show Selected Area Diffraction Pat-
 374 terns (SADPs) taken from the same tilting condition that clearly show the relation
 375 between the β phase (Figures 7 b) and c)) and at least 2 of the martensite variants
 376 (Figures 7 c) and d)). Three other variants were also indexed using SADPs taken in
 377 other locations within this band, but are not shown here for conciseness. Figure 7 h)
 378 shows a centered dark field (DF) image taken using contributions from two different

379 martensite variants ($\{112\}_{\alpha''}$ and $\{200\}_{\alpha''}$), highlighting the complex sub-structure
 380 and distribution of martensite in this band. The level of microstructure evolution
 381 and locally accumulated plastic strain is such that partial rings and "smeared" spots
 382 were measured in Figure 7 g), which is indicative of high local misorientation and
 383 fine crystallite sizes.

384

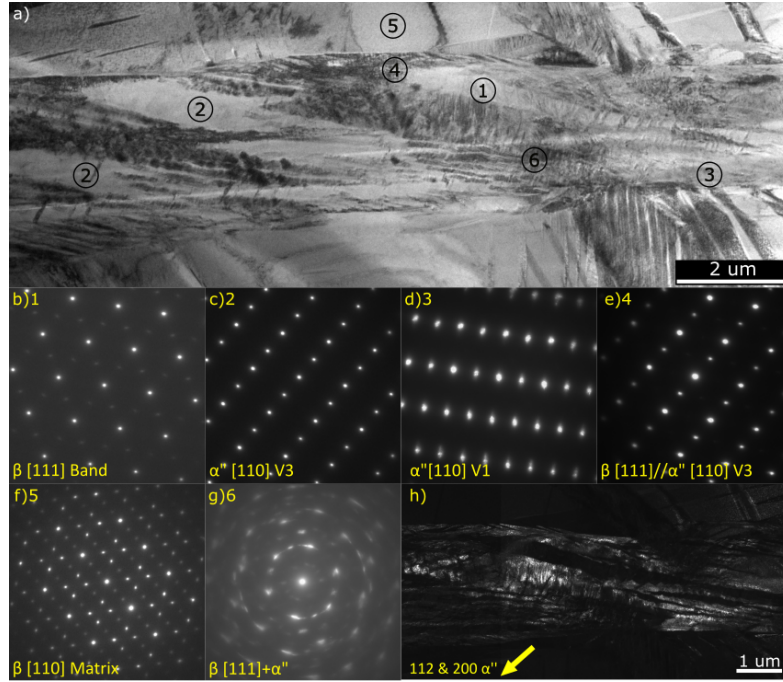


Figure 7: TEM of a large primary deformation band. a) Bright Field (BF) overview of the band with the location of selected area diffraction patterns (SADPs) taken in the same tilt condition indicated by the numbered circles (b-e). b) SADP of the β phase matrix taken parallel to the $[111]_{\beta}$ Zone Axis (ZA). c) SADP of martensite taken down the $[110]_{\alpha''}$ ZA. d) SADP of martensite taken down the $[110]_{\alpha''}$ ZA. e) SADP containing patterns from both the β phase matrix taken parallel to the $[111]_{\beta}$ ZA and of martensite taken down the $[110]_{\alpha''}$ ZA, as shown in d), highlighting the orientation relationship between the matrix and the martensite. f) SADP taken in the β phase of the grain, outside the main band and parallel to the $[110]_{\beta}$ ZA of the β phase. g) SADP of a region exhibiting fine sub-structure, composed of multiple variants of martensite, along with the $[111]_{\beta}$ ZA of the band. h) Two-beam dark field (DF) image taken using contributions from two different martensite variants. G-vectors used to form the image are indicated on the image.

385 Evidence of secondary and tertiary transformation product is also present in the
 386 matrix of the Ti-12Mo alloy. Figure 6 shows some of the secondary product to be
 387 $\{332\} < 113 >$ deformation twins. Secondary twinning and subsequent internal
 388 secondary transformation are also activated. Interestingly, TEM also reveals that
 389 secondary transformation is activated in the matrix, once primary twinning has

390 already subdivided the grains. Figure 8 a) shows a bright field (BF) overview of
 391 secondary transformation in the matrix; this image was taken in the tilt condition
 392 used to produce to the SADP shown in Figure 8 b). It can be seen that the matrix
 393 of the grain and the smaller laths are down zone, showing dark contrast and signi-
 394 fying the smaller laths are contributing the SADP shown in Figure 8 b). Figures
 395 8 c) through f) further support this, as the DF and BF images clearly show the
 396 smaller laths belong to the phase contributing to this pattern. It should be noted
 397 that contrast is obtained from the matrix in the DF images, due to overlap with
 398 the ω phase reflection of the matrix, as seen in Figure 8 b). The ω phase is known
 399 to be present in the β -phase matrix of Ti-12Mo [24]. These TEM images strongly
 400 support the fact that martensite laths formed directly from the β -phase matrix, not
 401 within a β twin.

402

403 4.3.2 TWIP Ti-15Mo

404 Figure 9 a) shows an IPF map of an EBSD scan taken from the gauge section of
 405 a Ti-15Mo specimen deformed in tension at a strain rate of roughly 10^3 s^{-1} . The
 406 IPF map shows a significant amount of microstructure evolution occurred during
 407 deformation. Large deformation product can be seen traversing the grains, and, in
 408 some instances, traversing multiple grains. These lath-like features have been iden-
 409 tified as twins. This type of accommodative twinning crossing grain boundaries has
 410 been reported before in Ti-15Mo after quasi-static strain rate testing [27, 28]. In-
 411 deed, Figure 9 b) shows an IQ map of the same scan, but with twinning boundaries
 412 overlaid in color, showing the majority of twins indexed are of the $\{332\} \langle 113 \rangle$
 413 type, equivalent to $\Sigma 11$ coincident site lattice (CSL) boundaries. All primary twins
 414 indexed are $\{332\} \langle 113 \rangle$ type.

415

416 Misorientation line profiles across twin boundaries further confirm this finding,
 417 as the characteristic $\Sigma 11$ misorientation of 50.5° about the $[110]_\beta$ is found. Two

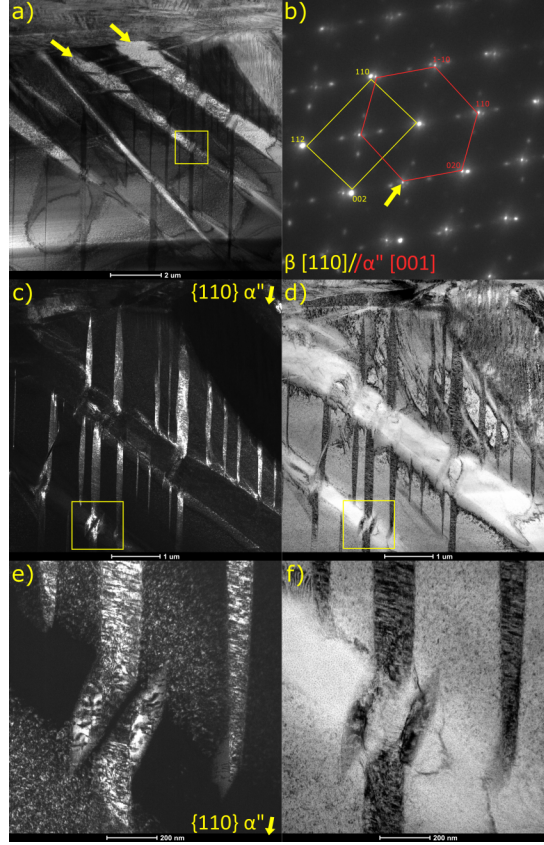


Figure 8: TEM of secondary transformation product in Ti-12Mo deformed in tension at a rate of 10^3 s^{-1} . a) BF image showing an overview of the characterized zone. The arrows indicate two martensite laths that visibly traverse another variant, as shown in c-f). b) SADP of the matrix and the laths that appear dark in a), taken parallel to the $[110]_{\beta}$ ZA of the β phase. This SADP was taken in the same tilt condition as the BF image shown in a). c-f) A DF and BF image pair taken using the reflections indicated by an arrow in b). c) and d) are a DF and BF image pair, respectively, highlighting the distribution of the martensite variant indexed in b). e) and f) are a DF and BF image pair, respectively, showing an expanded view of the region highlighted in c) and d). The same region is highlighted in a), for clarity. These images clearly show the first variant of martensite (indicated in a), shearing the illuminated martensite laths.

examples are presented in Figures 9 c) and f), with corresponding insets numbered 1 and 2 in a) and line-profiles shown in d) and e), respectively. Specifically, it can be seen in Figure 9 d) that the larger twins present internal or secondary twinning also of the $\{332\} < 113 >$ type. Interestingly, some of the boundaries are also identified as $\{112\} < 111 >$ twin boundaries, equivalent to $\Sigma 3$ CSL boundaries. Figure 9 d) indicates the presence of secondary twinning in a twinned grain, which is further discussed in the supplementary materials.

425

Interestingly, the area fraction of twins is significantly lower in the high strain

426

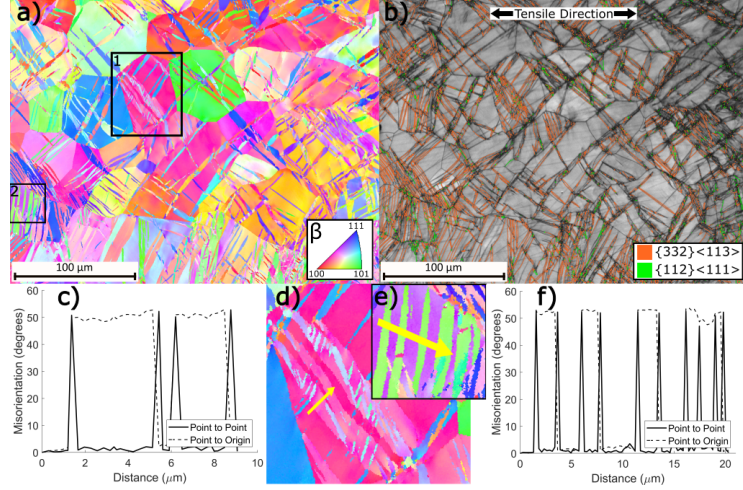


Figure 9: Evidence of primary twinning in an EBSD scan of a Ti-15Mo sample deformed in tension at a strain rate of 10^3 s^{-1} . This scan was taken in the gauge near the necked area. a) An IPF map. The areas highlighted by the black boxes numbered 1 and 2 are presented as insets in d) and e), respectively. b) An IQ map with twin boundaries overlaid: orange represents $\{332\} < 113 >$ twin boundaries, while green represents $\{112\} < 111 >$ twin boundaries. c) The misorientation profile taken along the yellow arrow indicated in d) (corresponding to the inset numbered 1 in a)). f) The misorientation profile taken along the yellow arrow indicated in e) (corresponding to the inset numbered 2 in a)).

rate deformation condition than after deformation at quasi-static strain rates. Studies on Ti-15Mo by Min et al. [29, 30] show the area fraction of twins is roughly 0.5 on average, and is up to 0.8 in favorably oriented grains. However, the EBSD scans shown here after high strain rate deformation reveal the twin area fraction, measured to be 0.13, is substantially lower compared to after quasi-static strain rate deformation [27], most likely due to lower plastic strain levels reached before fracture. This implies that TWIP is less active as strain rate increases in Ti-15Mo. In-situ deformation studies [30] reveal this is most likely due to $\{332\} < 113 >$ twins being formed as a function of strain, specifically in preferably oriented grains, and not stress within grains. If this is true and twinning is active over a smaller plastic strain range at higher strain rates, it would lead to a lower twin fraction, even if comparable stress levels were reached. The high strain rate ductility is substantially lower than after quasi-static strain rate testing. While the fundamental dependency is still unclear, the data presented here support this trend. This may have significant implications for the usefulness of TWIP Ti alloys for high strain rate applications.

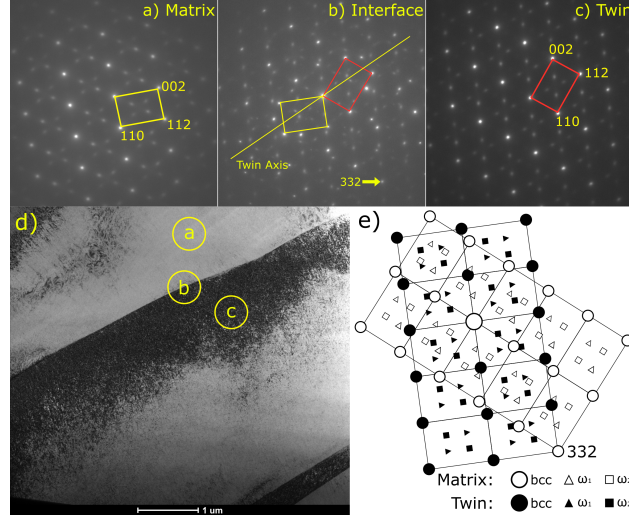


Figure 10: TEM of a $\{332\} \langle 113 \rangle$ twin interface taken from the gauge section of a TWIP Ti-15Mo specimen deformed in tension at 10^3 s^{-1} . a) SADP pattern of the matrix down the $[110]_\beta$ ZA. The β phase matrix reflections are labeled in the first Brillouin zone. The projected reciprocal unit cell is outlined for clarity. b) SADP pattern of the twin/matrix interface looking down the $[110]_\beta$ ZA of both crystals. The projected reciprocal unit cell is outlined for the twin and matrix crystal, as well as the twinning axis. The twinning plane is also labeled. c) SADP pattern of the twin looking down the $[110]_\beta$ ZA. The β phase matrix reflections are labeled in the first Brillouin zone. The projected reciprocal unit cell is outlined for clarity. d) A two-beam bright field image taken using the $g=110_\beta$ diffraction condition. The position where the patterns in a), b) and c) were taken is indicated in d). e) Schematic providing the key to interpreting the diffraction pattern shown in b).

443 To provide further proof of the nature of the deformation twins found in Ti-15Mo
 444 samples, TEM characterization was conducted on a FIB liftout taken at a primary
 445 deformation twin interface. Figure 10 shows SADPs indexed as a $\{332\} \langle 113 \rangle$
 446 twin pattern, taken from the interface of a primary deformation twin. Figures 10 a),
 447 b) and c) present SADPs from the matrix, interface and twin crystals, respectively.
 448 In Figure 10 b), the twin axis and twin plane ($g = \{332\}_\beta$) are highlighted, as well
 449 as the outline of the projection of the reciprocal unit cell (first Brillouin Zone) of
 450 each crystal. Also of note in the two-beam BF image in Figure 10 d) is the high
 451 density of dislocations visible near the twinning interface. Although not shown here,
 452 this is a commonly observed feature in many $\{332\} \langle 113 \rangle$ twins [31, 32] deformed
 453 at quasi-static strain rates and confirmed by TEM in this work.

454

455 5 Discussion

456 5.1 TWIP in BCC Versus FCC

457 It is well known that for TWIP face centered cubic (FCC) steels, the total contri-
458 bution to plastic strain from deformation twinning is roughly on the order of 3-5
459 % [8]. Zhou et al. [27] and Min et al. [29, 30] found that $\{332\} < 113 >$ twins
460 contribute much more to the total strain in TWIP Ti alloys, and that certain grains
461 when oriented near the $[\bar{1}22]$ axis, could provide up to 80% of the plastic strain, as
462 twin area fraction in these grains rapidly reaches 0.8. Although certain preferably
463 oriented grains accommodate most of the plastic strain by $\{332\} < 113 >$ twinning,
464 averaged over all measured grains, the twin area fraction reached a maximum of
465 roughly 0.5. The higher twin fraction implies the total contribution of twinning in
466 $\{332\} < 113 >$ TWIP β Ti alloys is larger than in TWIP steels.

467
468 TWIP strongly affects the WHR, as shown in TWIP steels, through the so-
469 called dynamic Hall-Petch effect, i.e., a dynamic refinement of the mean free path
470 of dislocations. This appears to be the case in TWIP Ti alloys also. Min et al.
471 were able to explain in large part the contribution of TWIP to the WHR using the
472 dynamic Hall-Petch effect in Ti-15Mo [29]. Secondly, Min et al. and Zhou et al.'s
473 study on Ti-15Mo [29, 30, 27] highlight how $\{332\} < 113 >$ twins seem to form by
474 accommodating the imposed strain, not stress. Specifically, Min et al. [30] showed
475 that on a grain by grain basis, twinning was almost completely absent in grains
476 oriented preferentially for slip, even if the local stresses within those grains were
477 high. This might explain the similarities between the stress-strain response of the
478 Ti-15Mo specimens at 10^{-3} and 10^{-1} s^{-1} . This is further highlighted when the WHR
479 is shown as a function of true stress (Figure 2 c), where it can be seen that the two
480 curves overlap once the yield stress has been reached. In essence, Ti-15Mo exhibits
481 similar work hardening for a similar flow stress, regardless of the strain rate at which
482 it is deformed. Once the capacity for twinning is exhausted, the normal, decreasing

work hardening of slip-dominant deformation takes over. From this stage onward to fracture, the work hardening curves truly match up independent of strain rate, as would be expected for a slip-dominant BCC crystal. As such, it seems as though the ability to exhibit deformation twinning is exhausted over a smaller range of flow stresses, and lower absolute levels of plastic strain result as strain rate is increased.

In FCC crystals, the yield stress, in general, is not highly sensitive to strain rate or temperature, due to the mostly athermal barrier for slip. However, work hardening is highly temperature sensitive, as the stacking fault energy (SFE) decreases with temperature and cross-slip is discouraged. Difficult cross-slip leads to an increased back-stress on slip systems, promoting hardening and retarding dynamic recovery. It is well reported that low SFE FCC materials exhibit increased work hardening and ductility at reduced temperatures [33, 34]. Hence, in FCC TWIP steels, the low SFE and deformation twinning itself both contribute to increasing work hardening as thermal activation is reduced (either by reducing temperature or increasing strain rate).

When BCC TWIP Ti alloys are considered in the same light, the conclusion is less promising. The stress necessary for slip has a strong thermal component, due to the core structure of screw dislocations in BCC alloys [35, 36]. However, work hardening is not strongly dependent on strain rate. When the contribution of TWIP is considered, decreasing thermal activation from increased strain rates should theoretically cause only small differences in twinning activity, due to the athermal nature of the twinning stress [37, 38, 8]. However, the result is still an altogether reduced capacity to accumulate plastic deformation as strain rate increases (or temperature decreases), as the contribution to WHR from twinning stays relatively constant per plastic strain increment. The BCC structure of TWIP Ti alloys, compounded with reduced twinning activity at increased strain rates, makes TWIP Ti less promising for high strain rate applications.

513 5.2 TRIP Versus TWIP at High Strain Rates: Work Hard- 514 ening and Strain Relaxation

515 Although we do not have comparable intermediate strain rate data available for
516 TRIP/TWIP Ti-12Mo, we can turn to TRIP/TWIP Ti-10V-3Fe-3Al (wt.%) and
517 TRIP Ti-10V-2Fe-3Al (wt.%) data available from Ahmed et al. and Ma et al., re-
518 spectively, to understand the effect of increasing from quasi-static to intermediate
519 strain rates on work hardening provided by TRIP [12, 13]. In both cases, the shape
520 of the stress-strain response and resulting WHR is unchanged by increasing strain
521 rate. Ma et al. reports the true strain at which yield and minimum WHR occur do
522 not significantly change over 5 orders of magnitude of strain rate.

523

524 When the mechanical testing data presented in Figure 3 is considered, specifically
525 comparing both alloys, it seems as though Ti-12Mo is more efficient at accommodat-
526 ing the imposed high strain rate deformation than Ti-15Mo. This is also supported
527 by the x-ray imaging used to determine the reduction in width up to fracture, as
528 shown in Figure 5. Both the mechanical testing data and x-ray imaging show that
529 Ti-12Mo exhibits higher ductility at both strain rates compared to Ti-15Mo up to
530 fracture. The increased ductility associated to TRIP also correlated with a more
531 gradual rate of microstructural evolution as a function of strain (Figure 4 e) and f).
532 Secondly, Ti-12Mo exhibits a larger increase in flow stress for the same increase in
533 strain rate compared to Ti-15Mo, indicating that Ti-12Mo has a higher strain rate
534 sensitivity (Figure 3). It also appears as though adiabatic heating does not have a
535 significant effect on TRIP in Ti-12Mo, as discussed in the supplementary materials.
536 A lower concentration of Mo leads to a less stable β -phase. Both alloys exhibit
537 TWIP during deformation. However, Ti-12Mo also exhibits TRIP. In fact, when
538 the results shown in Figure 7 are considered, it seems as though TRIP plus TWIP
539 leads to the formation of a much finer microstructure than TWIP alone produces

540 in Ti-15Mo. The exact reasons for this are still unknown, but martensite appears
541 to form sub-micron and even nanometer scale features that result in more efficient
542 accommodation of local strains, which maintains hardening to higher strains.

543

544 As stated in recent studies on TRIP/TWIP β Ti alloys [9, 11, 26], martensitic
545 transformation seems to be an important mechanism for alleviating local stresses
546 and elastic strains caused by concentrated microstructure evolution, specifically at
547 the intersection of secondary and tertiary deformation products (Figure 6). TWIP
548 in both alloys contributes to increasing the work hardening during plasticity, and
549 the effects reported at quasi-static strain rates are still observed at higher strain
550 rates. However, it seems that alleviating the local build-up of strain and resulting
551 stress is a more prominent concern in TWIP BCC alloys deformed at higher strain
552 rates. TRIP, on the other hand, serves to alleviate the local build-up of strain on
553 the sub-micron scale in Ti-12Mo, which allows for refinement of the microstructure
554 and dislocation density to increase before dynamic recovery, local strain, and stress
555 cause localization and fracture.

556

557 One major caveat to consider is the absence of nanoscale twin structures in Ti-
558 15Mo. Considering the evidence shown herein and by other quasi-static studies on
559 Ti-12Mo and Ti-15Mo alloys [1, 11, 27, 29], Ti-12Mo is clearly able to form much
560 finer microstructures during plastic deformation, even at higher strain rates. Re-
561 cent publications on multi-modal TWIP alloys, where both $\{332\} < 113 >$ and
562 $\{112\} < 111 >$ twinning modes are concurrently active, like Ti-7Mo-3Cr (wt. %) [39],
563 Ti-4Mo-3Cr-1Fe (wt. %) [3], or Ti-Mo-Zr ternaries [40, 41], exhibit a structural
564 refinement that is greater than found in Ti-15Mo. Testing multi-modal TWIP alloys
565 at higher strain rates would help to shed light on the role of TWIP versus TRIP in
566 work hardening versus strain relaxation.

567

6 Conclusions

We have used a combination of ultrafast synchrotron x-ray imaging and diffraction during high strain rate tensile testing and post-mortem characterization to compare and contrast the microstructure evolution of TRIP/TWIP Ti-12Mo and TWIP Ti-15Mo for the first time. These results provide key insights into the contributions of TRIP/TWIP for increased ductility in metastable Ti alloys during high strain rate deformation. The following conclusions can be drawn from this work:

- Quasi-static and intermediate strain rate tensile testing of TWIP Ti-15Mo showed that while TWIP contributes strongly to increasing work hardening, the intrinsic strain rate sensitivity of dislocations in the BCC crystal limit the maximum strength reached. For low to intermediate strain rates, once the contribution to work hardening from twinning is exhausted, slip takes over and the WHR curves overlap up to fracture, resulting in a severe reduction in elongation during tensile deformation with increasing strain rate.
- Ti-12Mo differs from Ti-15Mo during high strain rate testing, due to TRIP activation during deformation. TRIP has been shown to alleviate local elastic strain resulting from microstructure evolution during deformation. TWIP is still active as a primary and secondary deformation mechanism, however, in Ti-12Mo. The high strain rate mechanical testing data clearly shows that Ti-12Mo exhibits, on average, a higher total elongation at both strain rates.
- The activation of TRIP is believed to accommodate a portion of the imposed deformation and concurrently alleviates local strains from extensive twin formation. The combination of TRIP and TWIP appears to offer superior combinations of strength and ductility at high strain rates over TWIP alone for metastable Ti alloys.

This study shows that TRIP is crucial to maintaining high elongations during high strain rate mechanical testing compared to TWIP alone in metastable β Ti

595 alloys. Implications of this study potentially shift the focus from TWIP-dominant
596 alloys to optimized combinations of TRIP/TWIP for alloy, microstructure, and prop-
597 erty design.

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617 **8 Declaration of Competing Interests**

618 The authors have no competing interests to declare.

619 9 Data Availability

620 The Authors will make data available upon reasonable request.

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747 10 Supplementary Materials

748 10.1 Mechanical Discussion

749 According to Meyers [15], Kolsky pressure bar equations to obtain the strain rate
750 and strain signal from strain gauge data are as follows:

$$\dot{\varepsilon}(t) = -\frac{2C_0}{L}\varepsilon_R(t) \quad (1)$$

$$\varepsilon(t) = -\frac{2C_0}{L} \int \varepsilon_R(t) dt \quad (2)$$

751 where C_0 is the speed of sound in the incident bar, L is the length of the sam-
752 ple and $\varepsilon_R(t)$ is the amplitude of the reflected pulse as a function of time. These
753 two equations allow for the calculation of the "imposed strain" signal acting on the
754 mobile side of the bar setup, i.e., analogous to the crosshead in a conventional load
755 frame. Typical examples of strain versus time signal are shown in Figure 11 c).
756 For both strain rates, the rise time of the load pulse is roughly 50-60 μs , which
757 corresponds to roughly 0.02 imposed strain. This implies that the sample has not
758 reached a constant velocity until this strain level has been reached, and should be
759 kept in mind when interpreting the mechanical property results.

760

761 In a conventional Split-Hopkinson bar setup, the fixed-side of the sample would
762 be attached to an additional slender bar for measuring the transmitted strain pulse
763 and stress measurements. In our case, the size limitation of the hutch at the beamline
764 imposed the use of a load cell instead. Load cells directly convert voltage to newtons,
765 such that the signal need only be divided by the area of the specimen gauge section
766 (A_s):

$$\sigma(t) = \frac{F(t)}{A_s} \quad (3)$$

767 In our case, because we use a load cell instead of a transmission bar, this side

768 is assumed to be an "anvil", and thus a fixed boundary with an infinite impedance.
 769 For this assumption to be valid, it implies that the stress pulse undergoes perfect
 770 reflection upon reaching the fixed end. The boundary conditions for reflection of a
 771 stress wave against a rigid body are that the particle velocity must remain 0 in the
 772 rigid body, while the reflected pulse and stress will be equal in magnitude and sign
 773 to that of the incident pulse [15]. In this work, the reflected pulse has important
 774 variations in the plateau region, leading to the conclusion that motion was trans-
 775 mitted to the load cell. This implies that the fixed boundary condition assumption
 776 was not fully achieved.

777

778 Another aspect to consider is the intrinsic impedance of the samples which is
 779 higher than that of the incident bar. As per Meyers [15], when a stress wave arrives
 780 at an interface, equilibrium of forces and velocities must be maintained:

$$\sigma_I A_0 + \sigma_R A_0 = \sigma_T A_s \quad (4)$$

781 and

$$U_{0I} + U_{0R} = U_{sT} \quad (5)$$

782 where σ denotes stress, A denotes area, and U denotes particle velocity. Sub-
 783 scripts I, R and T represent the incident, reflected and transmitted components, and
 784 subscript 0 and s refer to the incident bar and the sample, respectively. Knowing
 785 that:

$$\sigma = \rho \frac{dx}{dt} U \quad (6)$$

786 and, in a slender bar, where lateral confinement is absent and dispersion is ne-
 787 glected:

$$\frac{dx}{dt} = V_{long} = C = \sqrt{\frac{E}{\rho}} \quad (7)$$

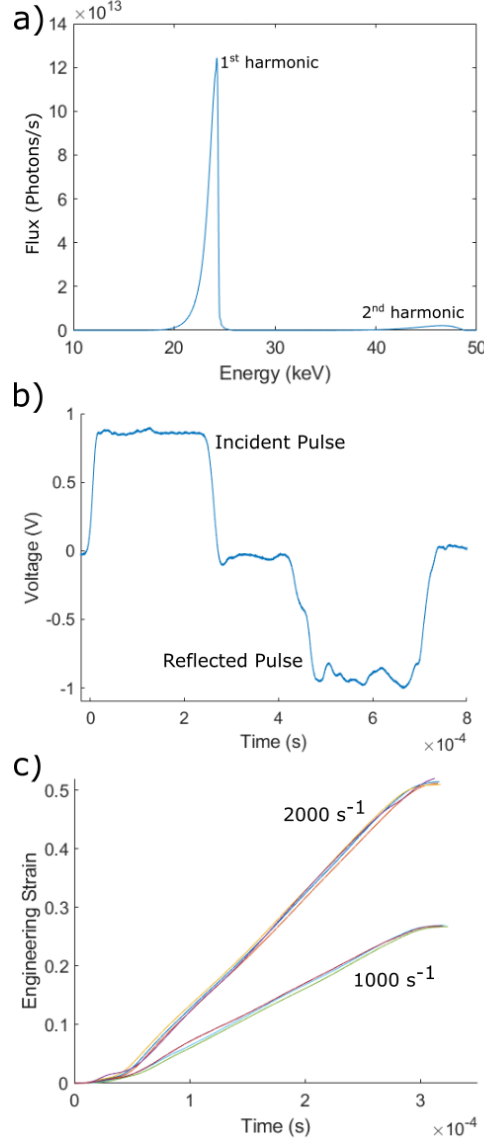


Figure 11: a) Energy spectrum for the pink beam used for in-situ synchrotron imaging and diffraction. b) Raw voltage versus time profile of the strain gauge signal for a typical test, showing the incident and reflected pulses. c) Integrated reflected pulse signal, showing strain versus time for both strain rates used in the high strain rate modified (Kolsky) pressure bar experiments.

Such that for an Al incident bar, $C_0 = 5051 \text{ ms}^{-1}$ ($C_0 = 5091 \text{ ms}^{-1}$ measured),
for a β phase Ti sample $E = 80 \text{ GPa}$, and $\rho = 5199 \text{ kg/m}^3$ and $\rho = 5372 \text{ kg/m}^3$
for Ti-12Mo and Ti-15Mo, respectively. Taking an average density of $\rho = 5280$
 kg/m^3 , the velocity of a longitudinal wave is $C_s = 3891 \text{ m/s}$. The impedances (or
the product ρC) of the incident bar and the sample are thus $1.437 \times 10^7 \text{ kg/m}^2\text{s}$
and $2.07 \times 10^7 \text{ kg/m}^2\text{s}$, respectively. The cross-sectional area of each also acts as a

794 geometric impedance. Using equations 4 and 5, it can be shown that:

$$\frac{\sigma_R}{\sigma_I} = \frac{A_0\rho_0C_0 - A_s\rho_sC_s}{A_s\rho_sC_s + A_0\rho_0C_0} \quad (8)$$

795 The area of the incident bar is 126 mm^2 and the cross-section of the gauge of
796 the specimens is 0.5 mm^2 . Unsurprisingly, when the contribution of geometry is
797 considered, the ratio σ_R/σ_I is close to unity, because the total impedance of the
798 sample is low relative to that of the incident bar. As such, assuming the sample has
799 a negligible total impedance relative to the incident bar is reasonable. To support
800 this assumption, Figure 11 b) shows the magnitude and duration of the reflected
801 pulse is similar to the incident pulse (neglecting the apparent wave dispersion).

802

803 Lastly, it should be stated that at least 3 reverberations are necessary to obtain
804 a state of homogeneous stress in the sample [15]. Considering the velocity of a lon-
805 gitudinal elastic wave in the sample is $C_s = 3891 \text{ ms}^{-1}$ and 3 times the gauge length
806 is equal to 12 mm , roughly $3 \text{ } \mu\text{s}$ are necessary for a homogeneous state of stress to
807 arise in the sample.

808

809 These discussion points ultimately show that many of the assumptions used in
810 the stress-strain analysis of a "Split-Hopkinson pressure bar" type of setup are re-
811 spected. First, seeing as how the pulse length relative to the duration of the test is
812 high and the sample impedance is negligible relative to that of the bar and anvil,
813 little energy loss is exhibited by the elastic pulse as it deforms the sample and is
814 reflected, meaning that negligible acceleration is felt by the bar during the test. In
815 fact, most of the deviation exhibited seems to be the result of motion within the
816 load cell on the anvil side and slack in the threads during loading. Coupled to the
817 relatively rapid rise time and short time required to establish a homogeneous stress
818 state in the gauge length implies the assumption of a constant strain rate uniaxial
819 tensile stress state is acceptable (at least from 2% strain to fracture). Indeed, it
820 seems that most of the artifacts in the mechanical testing data may arise from the

821 compliance of the load cell.

822

823 10.2 The Role of Adiabatic Heating on TRIP

824 Heat accumulation from plastic work becomes a concern when the characteristic
825 time for the deformation event to occur becomes smaller than the time for the
826 generated heat to diffuse away from the sample. This condition can be considered
827 as quasi-adiabatic, wherein the event can be considered to occur in the absence of
828 heat transfer to the environment. A useful equation for determining the threshold
829 is the characteristic diffusion distance for a given time:

$$d_t = 1.2\sqrt{(\alpha t)} \quad (9)$$

830 where :

$$\alpha = \frac{k}{\rho c_p} \quad (10)$$

831 where α is thermal diffusivity, d_t is the characteristic diffusion distance, t is time
832 (equivalent to strain rate in this case), and k is thermal conductivity, ρ is density
833 and c_p is specific heat. Typical values for β Ti alloys were used for these calculations,
834 including: $k = 7.8 \text{ W/mK}$, $\rho = 5285 \text{ kg/m}^3$ (average density for T-12Mo and Ti-
835 15Mo), $c_p = 525 \text{ J/kgK}$. These parameters give a thermal diffusivity $\alpha = 2.81 \times 10^{-6}$
836 m^2/s , which is almost an order of magnitude lower than mild steel ($\alpha = 18.8 \times 10^{-6}$
837 m^2/s) and is still lower than that of 304 stainless steel ($\alpha = 3.5 \times 10^{-6} \text{ m}^2/\text{s}$) [42].
838 As expected, this results in quasi-adiabatic conditions being reached at lower strain
839 rates than for a mild steel sample (AISI 1010) of the same dimensions (Figure 12).
840 The two horizontal lines in Figure 12 represent the thickness of the gauge section for
841 the intermediate strain rate tensile specimens (6.35 mm) and the high strain rate
842 (in-situ) tensile specimens (0.5 mm).

843

844 Adiabatic heating has been found to be an important consideration in the strain

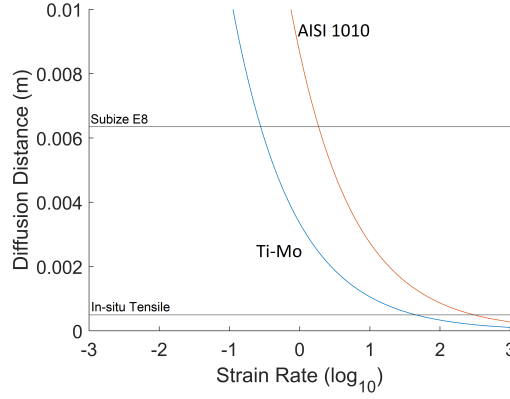


Figure 12: Characteristic thermal diffusion distance versus strain rate for averaged Ti-Mo composition, equivalent to Ti-13.5Mo (wt %) and AISI 1010 mild steel.

rate sensitivity of TRIP in steels [43, 44], which stems from the thermodynamics of TRIP. In general, The higher the testing temperature, the lower the undercooling relative to M_s , and more mechanical energy is needed to to drive the transformation.

848

If it is known that quasi-adiabatic conditions subsist during testing and calculating the temperature rise induced during testing is a matter of calculating the plastic work produced during the deformation and converting that energy to heat. As such, integrating the area under the true stress versus true strain curve gives the total temperature rise as follows:

$$\Delta T = \beta \int \frac{\sigma(\varepsilon)d\varepsilon}{\rho c_p} \quad (11)$$

where β is the fraction of work converted to heat, taken as 0.9 here, and $\sigma(\varepsilon)$ is the true stress response as a function of true strain. As discussed previously, the mechanical data available from high strain rate testing is highly variable. Thus, using the stress-strain data to calculate an exact temperature rise versus strain would lead to large uncertainty. However, the calculated values are a useful estimate for the purpose of discussion. The calculated total values vary between 25 and 35 K for both alloys at both strain rates. This temperature rise is also considered as a high-end conservative estimate, as the entire stress-strain curve was used for integration, which includes any post-uniform elongation that occurred.

863 A total temperature rise of roughly 30 K is not considered to have a large effect on
 864 TRIP and martensite undercooling. This value is comparatively small, due to Ti's
 865 low density and specific heat. The product of the two quantities (ρc_p) gives a relative
 866 indication of the temperature rise obtained for an increment of plastic work. This
 867 parameter is 40% higher in steels, which helps to explain why adiabatic heating may
 868 be a more important factor in the strain rate sensitivity of TRIP steels [44]. Lastly,
 869 it should also be noted that the temperature dependence of TRIP stress in Ti alloys
 870 is still not well understood. The presence of athermal ω phase in the undeformed
 871 state competes with deformation-induced martensite and adds a degree of freedom
 872 in determining the mechanical component of the free energy for transformation.

873

874 10.3 Secondary Twinning in TWIP Ti-15Mo

875 Figure 13 a) shows an IPF + IQ map with twinning boundaries overlaid and Figure
 876 13 b) shows a representative schematic of the band in the region enclosed by the
 877 dashed circle in a). The schematic clearly shows that $\{112\} < 111 >$ twin bound-
 878 aries are only present when two variants of the secondary $\{332\} < 113 >$ intersect.
 879 Figure 13 c) shows a misorientation profile for the line indicated by an arrow and
 880 labeled 1 in a). This line profile indicates that each of these parallel secondary twin
 881 boundaries present a misorientation characteristic of a $\{332\} < 113 >$ twin. On
 882 the other hand, the misorientation line profile indicated by the arrow labeled 2 was
 883 chosen to intersect both $\{332\} < 113 >$ and $\{112\} < 111 >$ boundaries. Indeed,
 884 Figure 13 d) shows that the misorientations of 50.5° and 60° are measured where
 885 the $\{332\} < 113 >$ and $\{112\} < 111 >$ twin boundaries are expected, further sup-
 886 porting this finding.

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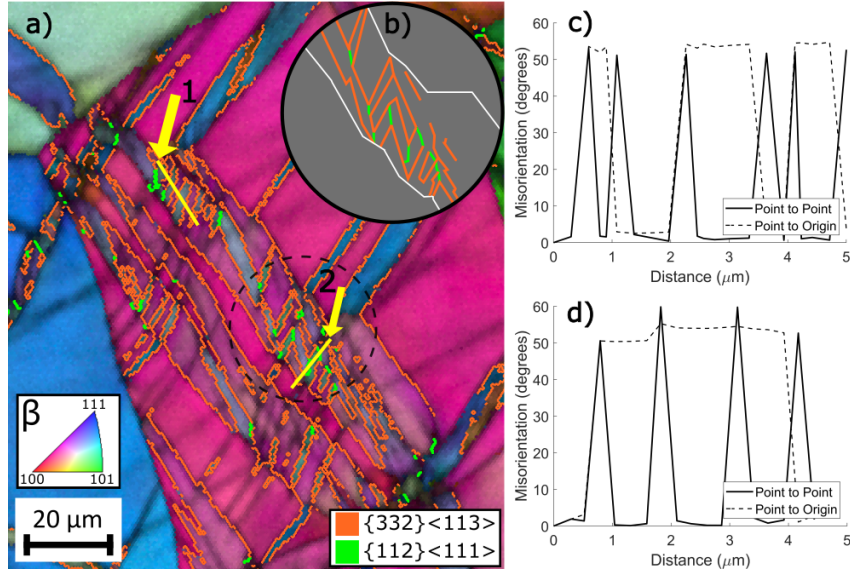


Figure 13: Detailed IPF map, highlighting the secondary twinning structure in Ti-15Mo. a) IPF + IQ map. Twin boundaries are also overlaid: orange represents $\{332\} \langle 113 \rangle$ twin boundaries, while green represents $\{112\} \langle 111 \rangle$ twin boundaries. Yellow arrows labeled 1 and 2 point to line profiles shown in c) and d), respectively. b) Schematic of secondary boundaries contained within the larger primary twin. The region the schematic is indicated by the dashed circle in a). The color scheme for boundaries is the same as in a). c) Misorientation profile for the line indicated at 1 in a). d) Misorientation profile for the line indicated at 2 in a)