

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

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6 Authors: B. Ellyson<sup>a\*</sup>, K. Fezzaa<sup>b</sup>, T. Sun<sup>b<sup>1</sup></sup>, N. Parab<sup>b</sup>, A. Saville<sup>a</sup>, C. Finfrock<sup>a</sup>,  
7 C.J. Rietema<sup>a<sup>2</sup></sup>, D. Smith<sup>a</sup>, J. Copley<sup>a</sup>, C. Johnson<sup>a</sup>, C.G. Becker<sup>a</sup>, J.  
8 Klemm-Toole<sup>a</sup>, C. Kirk<sup>c</sup>, N. Kedir<sup>d</sup>, J. Gao<sup>c</sup>, W. Chen<sup>c,d</sup>, R. Banerjee<sup>e</sup>, K.D.  
9 Clarke<sup>a</sup> A.J. Clarke<sup>a</sup>

10 <sup>a</sup> George S. Ansell Department of Metallurgical and Materials Engineering, Col-  
11 orado School of Mines, Golden, Colorado, <sup>b</sup> The Advanced Photon Source, Argonne  
12 National Laboratory, Lemont, Illinois, <sup>c</sup> School of Aeronautics and Astronautics  
13 engineering, Purdue University, West-Lafayette, Indiana, <sup>d</sup> School of Advanced Ma-  
14 terials Engineering, Purdue University, West-Lafayette, Indiana, <sup>e</sup> Department of  
15 Materials Science and Engineering, University of North Texas, Denton, Texas

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17 \* Corresponding author

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<sup>1</sup>Current affiliation: University of Virginia

<sup>2</sup>Current affiliation: Lawrence-Livermore National Laboratory

<sup>18</sup> **1 Abstract**

<sup>19</sup> The development of metastable titanium (Ti) alloys provides an unprecedented op-  
<sup>20</sup> portunity to expand their use in plasticity and damage critical applications like  
<sup>21</sup> protective structures. However, such applications require knowledge of quasi-static  
<sup>22</sup> to dynamic mechanical behavior, which is currently lacking. Here we perform in-situ,  
<sup>23</sup> ultrafast synchrotron x-ray diffraction during high strain rate (Kolsky) pressure bar  
<sup>24</sup> testing in tension and post-mortem electron microscopy to study TRansformation  
<sup>25</sup> Induced Plasticity (TRIP) and TWinning Induced Plasticity (TWIP) in metastable  
<sup>26</sup> Ti-Mo alloys. Two alloys, namely Ti-12Mo and Ti-15Mo (wt.%), were selected for  
<sup>27</sup> study having different  $\beta$ -phase chemical stabilities. TWIP was observed in both  
<sup>28</sup> Ti-12Mo and Ti-15Mo by in-situ synchrotron diffraction during high strain rate  
<sup>29</sup> testing. Post-mortem microstructural characterization also revealed the presence  
<sup>30</sup> of TRIP in Ti-12Mo. TWIP in Ti-15Mo was found to under-perform in terms of  
<sup>31</sup> total elongation compared to TRIP/TWIP in Ti-12Mo. Ti-12Mo exhibited an av-  
<sup>32</sup> erage elongation of 17% compared to only 12% for Ti-15Mo with deformation at  
<sup>33</sup>  $1000\text{ s}^{-1}$ . TRIP resulted in significantly finer microstructure evolution and allevi-  
<sup>34</sup> ated local strain accumulations in Ti-12Mo, suggesting TRIP can be used to tune  
<sup>35</sup> available strength/ductility combinations in metastable Ti alloys under high strain  
<sup>36</sup> rate deformation conditions.

<sup>37</sup> **2 Introduction**

<sup>38</sup> Ti alloys have been widely used in structural components in aerospace applications,  
<sup>39</sup> due to their high strength to weight ratios. Recently, interest in a novel class of  
<sup>40</sup> Ti alloys, namely metastable  $\beta$  alloys, has been increasing, due to promising work  
<sup>41</sup> hardening and ductility behaviors [1, 2, 3, 4]. Metastable  $\beta$  Ti alloys have long  
<sup>42</sup> been known to exhibit microstructure evolution during quasi-static deformation [5].  
<sup>43</sup> Only recently has the propensity for TRansformation Induced Plasticity (TRIP) and  
<sup>44</sup> TWinning Induced Plasticity (TWIP) been studied with the aim of exploiting these

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

53

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

69

70 Only a few high strain rate deformation studies have been published to date  
71 on metastable  $\beta$  Ti alloys, mostly concentrating on the strain rate sensitivity from  
72 quasi-static strain rates up to the low end of intermediate strain rates ( $10^{-5}$  to  
73  $10^{-1} s^{-1}$ ) [12, 13, 14]. While the data presented in these studies are useful, they

74 do not extrapolate to high strain rate behaviors because different mechanisms like  
75 dislocation generation and motion may dominate in different strain rate regimes  
76 [15]; in addition temperature effects (i.e., adiabatic heating), pathway dependent  
77 microstructure evolution is also not well understood. Ahmed et al. [12] have pub-  
78 lished one of the only studies where strain rates up to  $10^2 \text{ s}^{-1}$  were tested on an  
79  $\alpha + \beta$  TRIP/TWIP Ti-10V-3Fe-3Al (wt. %) alloy. While their study was performed  
80 in compression, they concluded that TWIP was dominant over TRIP as strain rate  
81 increased, although all mechanisms were found to be active over the entire strain  
82 rate range regime studied (i.e., no TRIP suppression occurred). To the authors'  
83 knowledge, only two studies have looked at metastable  $\beta$  Ti alloys [16, 17] deformed  
84 at strain rates on the order of  $10^3 \text{ s}^{-1}$  and only in compression. Both studies found  
85 evidence of TRIP and TWIP in the deformed state; increased work hardening was  
86 provided by both mechanisms. Specifically, Yang et al.'s study on Ti-8.5Cr-1.5Sn  
87 (wt. %) [16] showed that TRIP and TWIP are a powerful deterrent to the formation  
88 of adiabatic shear bands. To date, no studies have systematically compared different  
89 alloys and chemical phase stabilities under high strain rate deformation conditions,  
90 which controls the microstructure evolution.

91  
92 Here we investigate the microstructure evolution in two Ti-Mo alloys with dif-  
93 ferent  $\beta$ -phase chemical stabilities using ultrafast synchrotron x-ray diffraction dur-  
94 ing high strain rate tensile deformation. Complementary post-mortem electron mi-  
95 croscopy was also performed. **These results reveal the importance of TRIP**  
96 **in maintaining higher elongations, and thus the potential for energy ab-**  
97 **sorption, and suggest that TRIP-capable alloys (Ti-12Mo) out-perform**  
98 **TWIP alloys (Ti-15Mo) at high strain rates, due to the central role that**  
99 **martensite plays in local stress relaxation.**

100

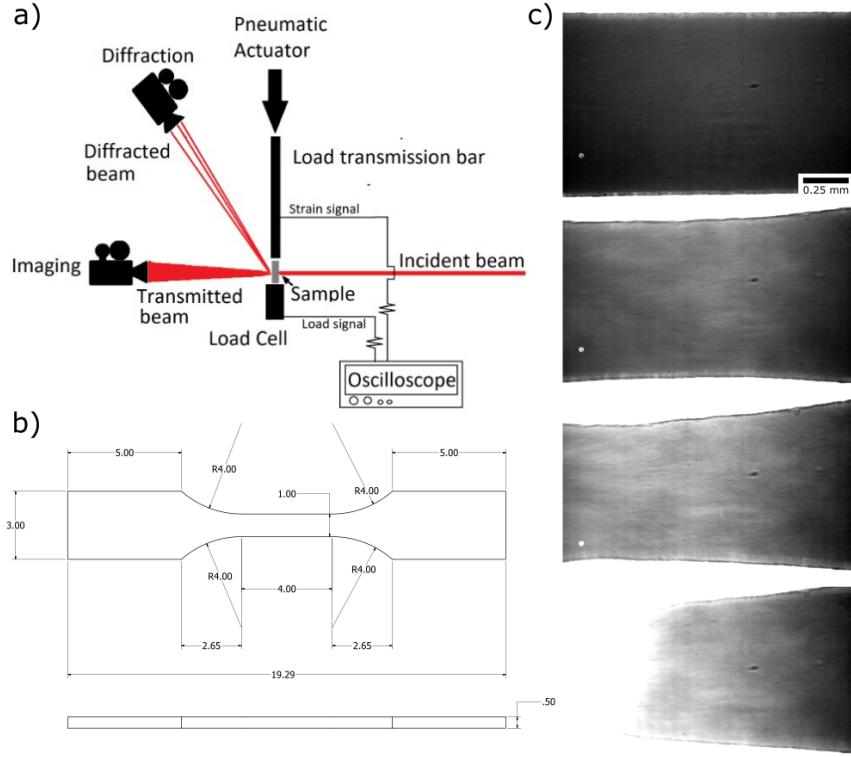
<sub>101</sub> **3 Methodology**

<sub>102</sub> **3.1 Ultrafast Synchrotron X-Ray Imaging and Diffraction**  
<sub>103</sub> **during High Strain Rate Deformation**

<sub>104</sub> Modified (Kolsky) pressure bar testing together with high brilliance, high-flux, ul-  
<sub>105</sub> trafast synchrotron x-ray imaging and diffraction was performed at sector 32-ID-B  
<sub>106</sub> at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL). The  
<sub>107</sub> details of the modified (Kolsky) pressure bar apparatus have been reported elsewhere  
<sub>108</sub> [18, 19, 20]. A schematic of the experimental setup is shown in Figure 1 a), which  
<sub>109</sub> provides the simultaneous capture of x-ray imaging, diffraction and mechanical test  
<sub>110</sub> data. A Photron FASTCAM SA-Z type 2100K-M-64G camera using a LuAG:Ce  
<sub>111</sub> scintillator (100 thick) and a 10X microscope objective (Mitutoyo long WD) at a  
<sub>112</sub> frame rate of 50400 frames per second (fps) was used for imaging. Diffraction data  
<sub>113</sub> were captured in full transmission geometry with a Shimadzu HPV-X2, coupled to  
<sub>114</sub> an image intensifier and an LSO:Ce scintillator (300 thick), at a frame rate of 298507  
<sub>115</sub> fps. The mechanical test data were acquired with an oscilloscope at an acquisition  
<sub>116</sub> rate of  $10^8$  Hz. For the x-ray setup, an undulator gap of 12 mm was used, providing  
<sub>117</sub> a "pink-beam"-like condition with a maximum flux at a wavelength of 0.512 Å and  
<sub>118</sub> a characteristic, asymmetric intensity profile around the harmonic energies, with a  
<sub>119</sub> tail dropping off in intensity gradually toward the lower energies. A beam size of 2  
<sub>120</sub> mm wide by 1 mm in height was used to illuminate the gauge length of each tensile  
<sub>121</sub> specimen. This large beam size, combined with the tail of the energy profile, lead to  
<sub>122</sub> significant instrument and source broadening of the diffraction peaks, as discussed  
<sub>123</sub> in the supplementary materials. The detector-to-sample distance was roughly 0.6 m,  
<sub>124</sub> and the resulting pixel size was roughly 32  $\mu$ m on the detector. Different detector  
<sub>125</sub> positions were used to obtain different resolutions and q-space ranges. The sample  
<sub>126</sub> geometry is also provided in Figure 1b.

<sub>127</sub>

<sub>128</sub> 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  $10^3 \text{ s}^{-1}$ . 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

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

143

144 **3.2 Sample Preparation, Post-Mortem Microstructure Char-**  
145 **acterization and Quasi-static and Intermediate Strain Rate**  
146 **Testing**

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

167

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

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

<sup>175</sup>

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

<sup>184</sup>

## <sup>185</sup> 4 Results

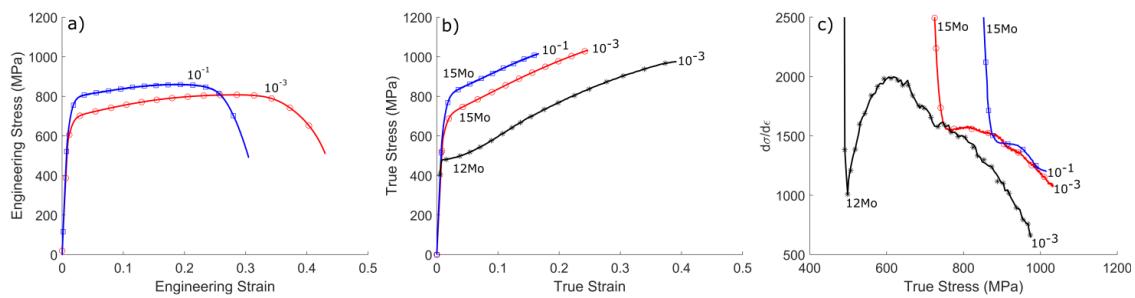
### <sup>186</sup> 4.1 Mechanical Properties

#### <sup>187</sup> 4.1.1 Quasi-Static and Intermediate Strain Rate Mechanical Properties

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

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

203



**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].

204 The true stress versus true strain response of Ti-15Mo is also shown in Figure  
 205 2 b) for quasi-static and intermediate strain rates. Interestingly, the WHR seems  
 206 to be comparable between the two strain rates. However, an apparent reduction  
 207 in uniform elongation correlates with a concurrent reduction in work hardening as  
 208 flow stress approaches the ultimate tensile strength (UTS) of 1 GPa. Figure 2 c)  
 209 shows the substantial difference in WHR at high flow stresses exhibited by both  
 210 alloys. TRIP-capable Ti-12Mo exhibits an overall larger WHR over a larger stress  
 211 range, but does not reach Ti-15Mo equivalent stress levels. The superior flow stress  
 212 exhibited by Ti-15Mo is most likely due to the Mo solid solution strengthening in  
 213 the matrix. When quasi-static and intermediate strain rates are considered for Ti-  
 214 15Mo, it is clear that the WHR is similar for a given stress level after yielding. The  
 215 work hardening behavior of Ti-15Mo does not appear to be strongly sensitive to  
 216 strain rate. This finding supports the claim that plastic strain is mostly mediated

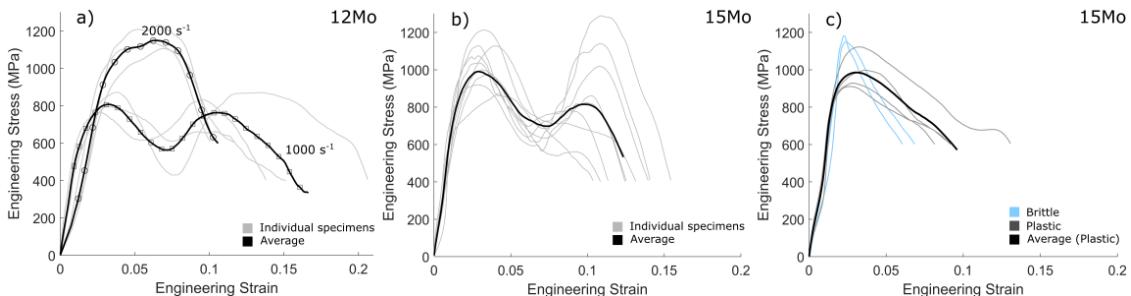
217 by dislocations, while work hardening is controlled by slip and twinning. This is  
218 discussed further later.

219

#### 220 4.1.2 High Strain Rate Mechanical Properties

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

228



**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  $2 \times 10^3 s^{-1}$ . 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).

229 Due to the inherently noisy nature of the high strain rate mechanical testing  
230 data, it is impossible to identify an exact uniform elongation and WHR. Even the  
231 exact moment of failure is hard to pinpoint, in large part due to the decay time of  
232 the load cell signal. To circumvent this difficulty, a cutoff stress equal to half the  
233 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  $10^3 \text{ s}^{-1}$  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  $10^3 \text{ s}^{-1}$  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

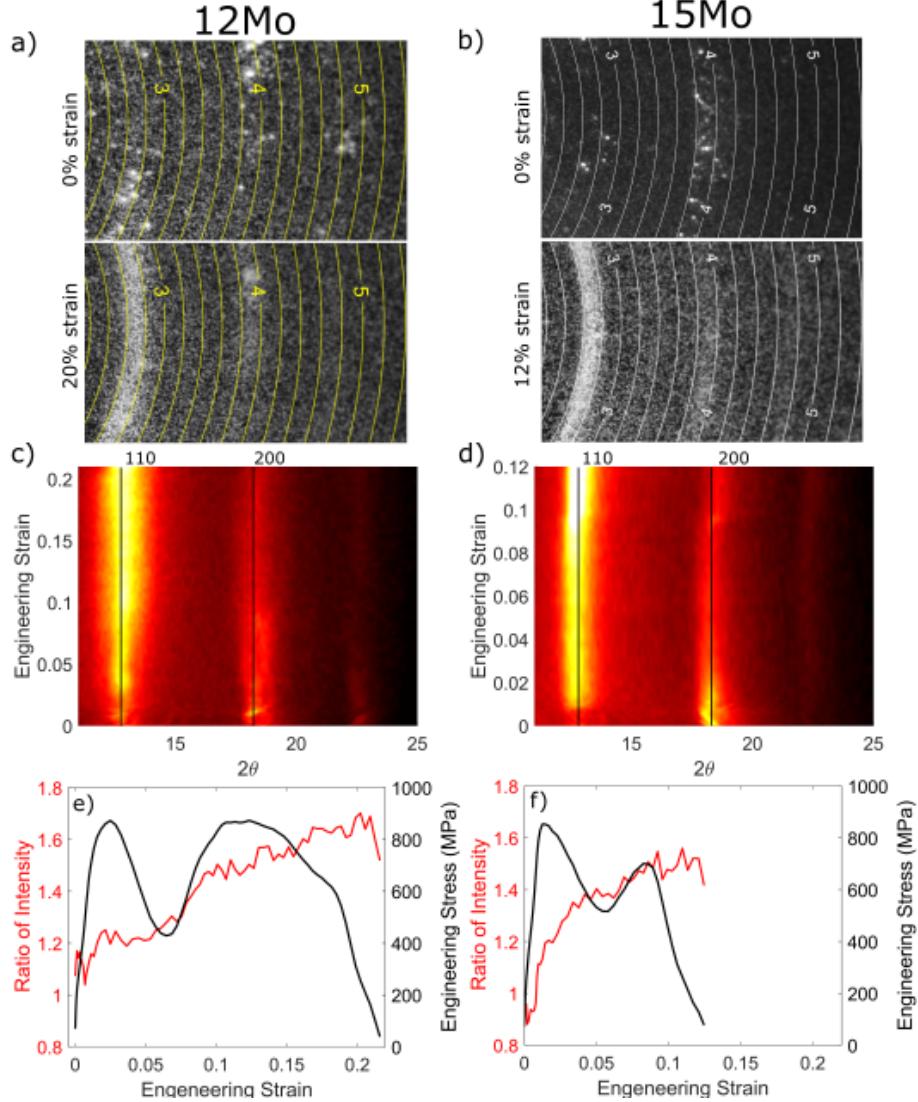
249 Curves for individual specimens of the Ti-15Mo alloy tested at the highest strain  
250 rate of  $2 \times 10^3 \text{ s}^{-1}$  were divided into 2 categories, as the mechanical behavior diverged  
251 greatly. Specimens either exhibited limited ductility, or none at all, i.e., fracture oc-  
252 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  $10^3 \text{ s}^{-1}$ , 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  $4 \text{ nm}^{-1}$  and  $2.7 \text{ nm}^{-1}$  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  $\phi$ , 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  $18^\circ$  in  $2\theta$  space (or roughly  $4 \text{ nm}^{-1}$   
277 in q-space), rapidly shifts to form an increasing maximum near  $12^\circ$  (or roughly  $2.7$   
278  $\text{nm}^{-1}$  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  $\beta$  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.

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

295 summary, both alloys exhibit strong evidence of microstructural evolution, particu-  
296 larly grain refinement of the  $\beta$ -phase matrix.

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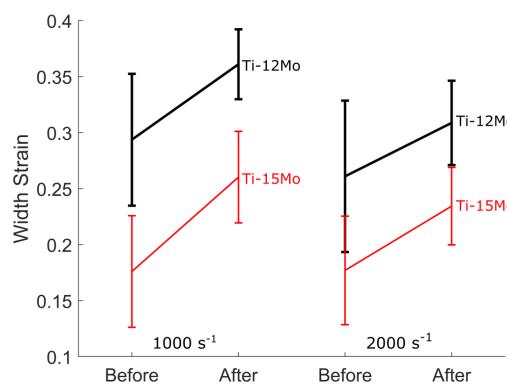
298 In both integrated diffraction patterns presented in Figure 4 c) and d), intensity  
299 near the  $\{110\}_\beta$  reflection becomes dominant over a large portion of the deforma-  
300 tion. This indicates that plasticity is preferentially rotating the  $\{110\}_\beta$  planes into  
301 the Bragg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

314

315 Figure 4 e) and f) show the ratio of the integrated intensity of the  $\{110\}_\beta$  posi-  
316 tion and the integrated intensity of  $\{200\}_\beta$  position, as well as engineering stress, as  
317 a function of engineering strain. The ratio of integrated intensities provides a visual  
318 indication of the rate at which texture evolves as a function of strain. It is interesting  
319 to note that the ratio gradually increases from the onset of loading up to fracture in  
320 Ti-12Mo. When compared to the engineering stress response, the gradual increase  
321 in ratio of intensities correlates with a high strain to fracture and nearly-constant  
322 flow stress (disregarding the load oscillation caused by ringing in the sample). This  
323 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.

330



**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 ( $1000\text{ s}^{-1}$  and  $2000\text{ s}^{-1}$ ). 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  $2 \times 10^3\text{ s}^{-1}$ .

341

342 **4.3 Post-mortem Microstructure Characterization**

343 The in-situ synchrotron x-ray diffraction data provide insights into the microstruc-  
344 ture evolution that occurs during deformation. However, post-mortem characteri-  
345 zation is necessary to understand the exact nature of the evolution, since diffraction  
346 is fundamentally a volume averaged technique. As such, fractured specimens from  
347 both alloys were characterized using EBSD and TEM to obtain important informa-  
348 tion about the fine-scale structural evolution.

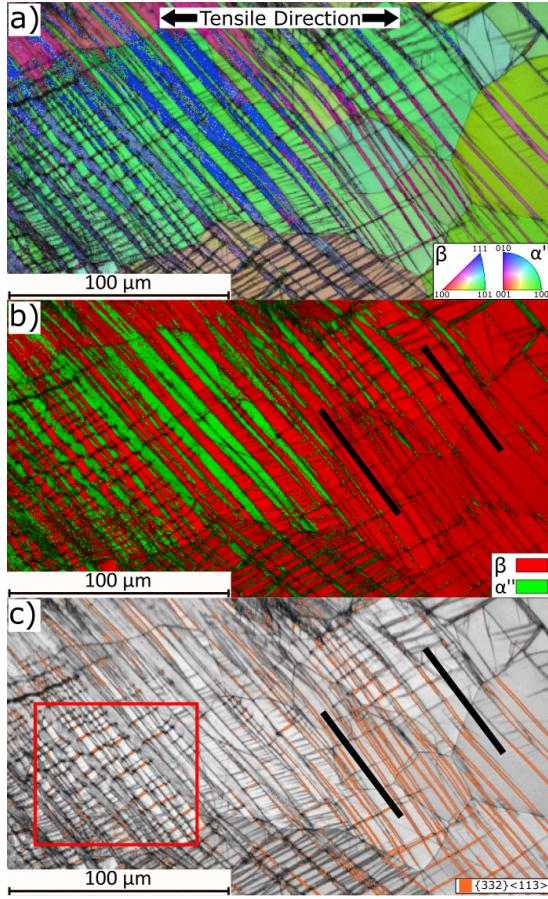
349

350 **4.3.1 TRIP/TWIP Ti-12Mo**

351 Evidence of  $\beta$ -phase deformation twinning and transformation are present in the  
352 microstructure of deformed Ti-12Mo. Large deformation bands can be seen travers-  
353 ing grain boundaries with little re-orientation, plausibly due to accommodation in  
354 the neighboring grains [27, 28]. Figures 6 a) and b) show an IPF + IQ and Phase  
355 + IQ maps, respectively, of an EBSD scan where complex microstructure evolution  
356 can be seen, along with strong evidence of deformation twinning as the primary de-  
357 formation product. Figure 6 c) shows an IQ map overlaid with  $\{332\} < 113 >$  twin  
358 boundaries. This map shows clear evidence of the concurrent presence of transfor-  
359 mation and twinning in the deformed state, similar to the microstructure evolution  
360 reported for quasi-static deformation [1, 11]. Secondary twinning can also be seen  
361 to further sub-divide the  $\beta$ -phase matrix remaining between the primary twins in  
362 one of the grains on the left side of the map (indicated by the red box in Figure 6  
363 c). Between the two black lines in Figures 6 b) and c) is the transition from un-  
364 transformed primary twins in the bottom right to twins containing secondary trans-  
365 formation product, i.e. martensite in the upper portion of the same twinned laths.  
366 This is clear experimental evidence of secondary transformation within the primary  
367 twin accommodating further plastic deformation within the initially twinned crystal.

368

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

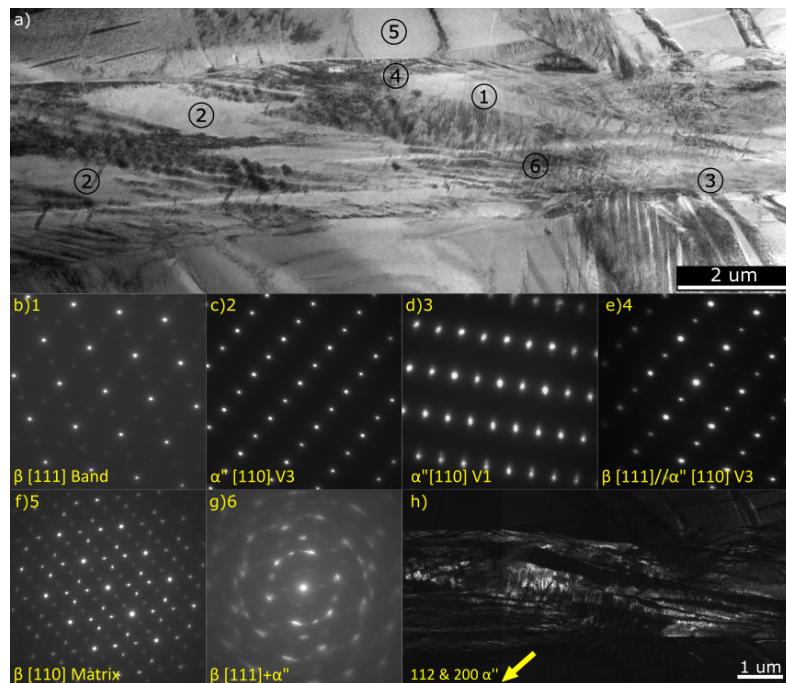


**Figure 6:** EBSD of a Ti-12Mo specimen deformed in tension at a strain rate of  $10^3 \text{ s}^{-1}$  taken outside the necked region of the gauge length. a) IPF + IQ map and b) Phase + IQ map c) IQ map with twin boundary overlay; orange represents  $\{332\} <113>$  twin boundaries. The phase map 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  $\{332\} <113>$  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  $\alpha''$  variants, along  
 373 with residual  $\beta$ -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  $\beta$  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  $\beta$  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 d) SADP containing patterns from both the  $\beta$  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  $\beta$  phase of the grain, outside the main band and parallel to the  $[110]_{\beta}$  ZA of the  $\beta$  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

already subdivided the grains. Figure 8 a) shows a bright field (BF) overview of secondary transformation in the matrix; this image was taken in the tilt condition used to produce to the SADP shown in Figure 8 b). It can be seen that the matrix of the grain and the smaller laths are down zone, showing dark contrast and signifying the smaller laths are contributing the SADP shown in Figure 8 b). Figures 8 c) through f) further support this, as the DF and BF images clearly show the smaller laths belong to the phase contributing to this pattern. It should be noted that contrast is obtained from the matrix in the DF images, due to overlap with the  $\omega$  phase reflection of the matrix, as seen in Figure 8 b). The  $\omega$  phase is known to be present in the  $\beta$ -phase matrix of Ti-12Mo [24]. These TEM images strongly support the fact that martensite laths formed directly from the  $\beta$ -phase matrix, not within a  $\beta$  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  $10^3 \text{ s}^{-1}$ . 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  $\{332\} < 113 >$   
413 type, equivalent to  $\Sigma 11$  coincident site lattice (CSL) boundaries. All primary twins  
414 indexed are  $\{332\} < 113 >$  type.

415

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

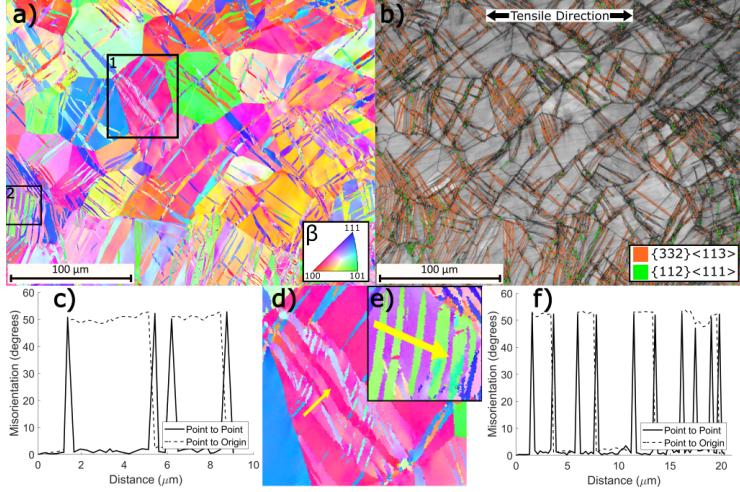


**Figure 8:** TEM of secondary transformation product in Ti-12Mo deformed in tension at a rate of  $10^3 \text{ s}^{-1}$ . a) BF image showing an overview of the characterized zone. The arrows indicate two martensite laths that visibly traverse another variant, as shown in c-f). b) SADP of the matrix and the laths that appear dark in a), taken parallel to the  $[110]_\beta$  ZA of the  $\beta$  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.

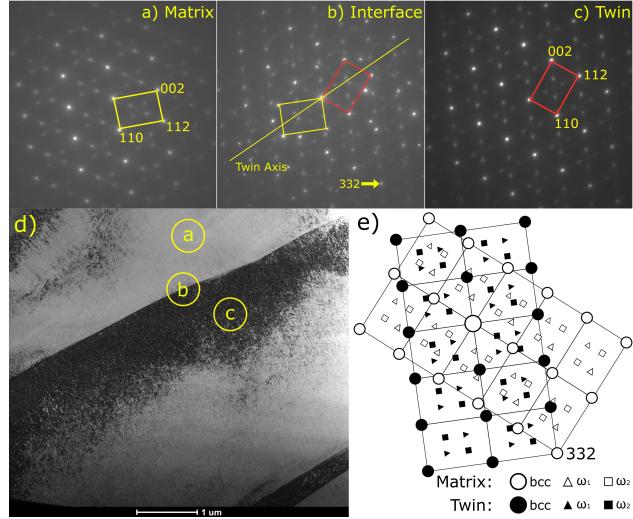
425

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



**Figure 9:** Evidence of primary twinning in an EBSD scan of a Ti-15Mo sample deformed in tension at a strain rate of  $10^3 \text{ s}^{-1}$ . 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  $\{332\} < 113 >$  twin interface taken from the gauge section of a TWIP Ti-15Mo specimen deformed in tension at  $10^3 \text{ s}^{-1}$ . a) SADP pattern of the matrix down the  $[110]_\beta$  ZA. The  $\beta$  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  $\beta$  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  $\{332\} < 113 >$   
 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  $\{332\} < 113 >$  twins [31, 32] deformed  
 453 at quasi-static strain rates and confirmed by TEM in this work.

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  $\beta$  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

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

488

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

499

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

513 **5.2 TRIP Versus TWIP at High Strain Rates: Work Hardening 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.

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  $\beta$ -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  $\beta$  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. %)  
563 [39], 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

568 **6 Conclusions**

569 We have used a combination of ultrafast synchrotron x-ray imaging and diffraction  
570 during high strain rate tensile testing and post-mortem characterization to compare  
571 and contrast the microstructure evolution of TRIP/TWIP Ti-12Mo and TWIP Ti-  
572 15Mo for the first time. These results provide key insights into the contributions of  
573 TRIP/TWIP for increased ductility in metastable Ti alloys during high strain rate  
574 deformation. The following conclusions can be drawn from this work:

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

593 This study shows that TRIP is crucial to maintaining high elongations during  
594 high strain rate mechanical testing compared to TWIP alone in metastable  $\beta$  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  $\mu s$ , 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 stain 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

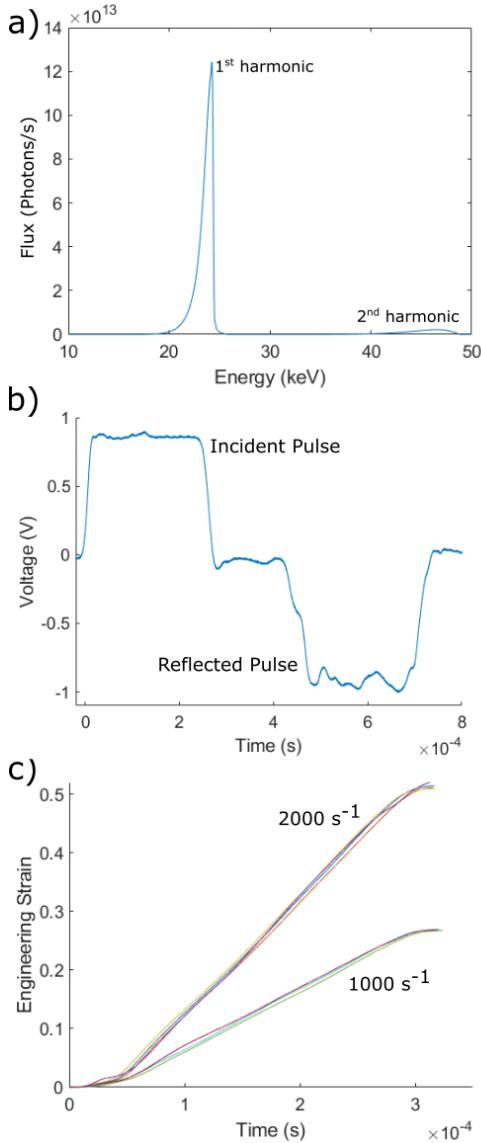
$$U_{0I} + U_{0R} = U_{sT} \quad (5)$$

782 where  $\sigma$  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  $\beta$  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  $\rho = 5280\text{ kg/m}^3$ , the velocity of a longitudinal wave is  $C_s = 3891\text{ m/s}$ . The impedances (or the product  $\rho C$ ) 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  $126 \text{ mm}^2$  and the cross-section of the gauge of  
796 the specimens is  $0.5 \text{ mm}^2$ . Unsurprisingly, when the contribution of geometry is  
797 considered, the ratio  $\sigma_R/\sigma_I$  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  $12 \text{ mm}$ , roughly  $3 \mu\text{s}$  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:

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

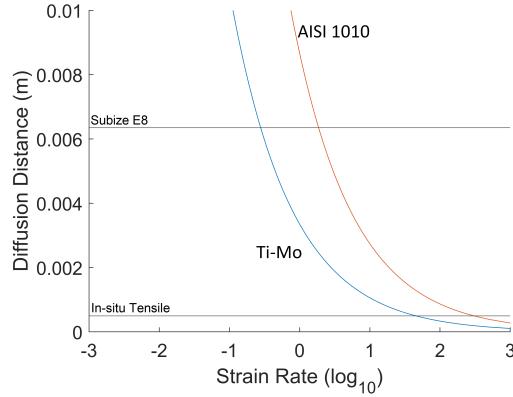
830 where :

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

831 where  $\alpha$  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,  $\rho$  is density  
833 and  $c_p$  is specific heat. Typical values for  $\beta$  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  $\text{m}^2/\text{s}$ , which is almost an order of magnitude lower than mild steel ( $\alpha = 18.8 \times 10^{-6}$   
837  $\text{m}^2/\text{s}$ ) 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.

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

848

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

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

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

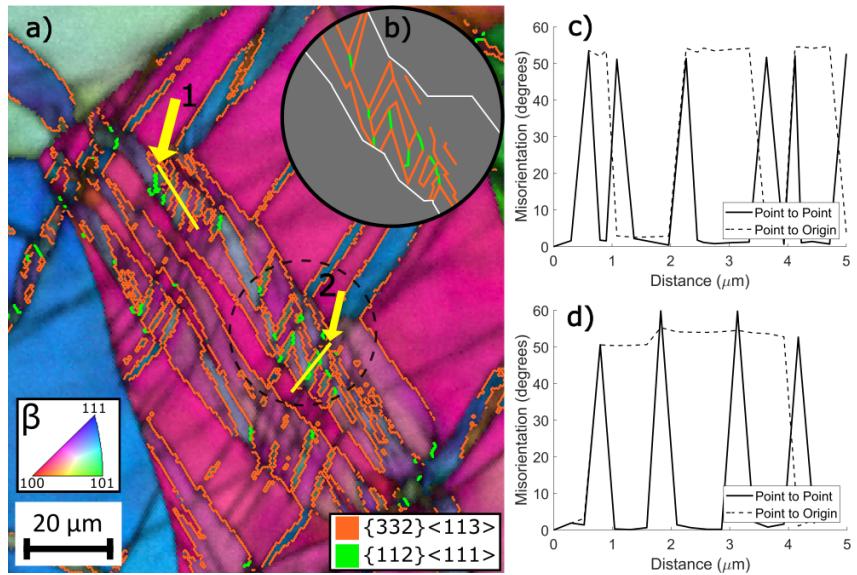
863 A total temperature rise of roughly 30 K is not considered to have a large effect on  
864 TRIP and martensite undercooling. This value is comparatively small, due to Ti's  
865 low density and specific heat. The product of the two quantities ( $\rho c_p$ ) gives a relative  
866 indication of the 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  $\omega$  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.

887



**Figure 13:** Detailed IPF map, highlighting the secondary twinning structure in Ti-15Mo. a) IPF + IQ map. Twin boundaries are also overlaid: orange represents  $\{332\} < 113 >$  twin boundaries, while green represents  $\{112\} < 111 >$  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)