33a.0 IN-SITU STUDIES OF STRAIN RATE EFFECTS ON PHASE TRANSFORMATIONS AND MICROSTRUCTURAL EVOLUTION IN BETA-TITANIUM ALLOYS (LEVERAGED)

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This project was initiated in Fall 2017 and is supported by the Office of Naval Research. Early support was received from A.J. Clarke's startup at the Colorado School of Mines (Mines). The research performed during this project will serve as the basis for a Ph.D. thesis for Benjamin Ellyson.

33a.1 Project Overview and Industrial Relevance

Titanium alloys are heavily used in the aerospace and biomedical industries for their high specific strength and good corrosion resistance. However, low work hardening and uniform elongation have limited their applicability in deformation controlled applications, where high absorbed energy or high formability are required. The ability to develop novel titanium alloys that exhibit high work hardening would broaden their applicability, such as lightweight blast-resistant armor, crash resistant structural components and high-complexity plastically formed parts. Recent work [33.1-33.3] on metastable β-titanium alloys has shown promising results, wherein high work hardening and uniform elongations were achieved through transformation induced plasticity (TRIP) and twinning induced plasticity (TWIP). These deformation mechanisms have been the subject of extensive study in ferrous alloys, while published work in other alloy systems, such as BCC titanium in particular, is limited in scope. The present project aims to study TRIP and TWIP effects in β-titanium to garner fundamental understanding of the intrinsic and extrinsic variables controlling TRIP and TWIP. The fundamental knowledge gained from this study will be used to develop an alloy design methodology that will enable the tailoring of microstructural evolution, deformation mechanisms, and mechanical response by means of alloying and processing. Specifically, Ti-10V-2Fe-3Al (wt.%) (Ti-1023) and Ti-15Mo (wt.%) were selected as candidate alloys for the study of TRIP and TWIP, respectively. Ti-1023 is well known to be a candidate TRIP alloy, as the orthorhombic martensite (α") phase forms in the matrix during deformation [33.4] and Ti-15Mo is known to be a candidate TWIP alloy, as {332} mechanical twinning is prevalent during deformation [33.5], at quasi-static deformation rates. Thus, each alloy will permit the study of the effect of TRIP and TWIP independently as a function of prior microstructure and strain rate.

33a.2 Previous Work

The previous reporting cycle's accomplishments consist mainly of quasi-static tensile testing of Ti-1023 and Ti-15Mo, including characterization of the microstructure before and after deformation. During beamtime at Sector 32-ID at the Advanced Photon Source (APS) at Argonne National Laboratory, in-situ diffraction and radiography measurements were conducted during Kolsky bar testing of Ti-1023 and Ti-15Mo in tension and compression. This beamtime was completed in February 2019. Quasi-static testing at Mines was aimed at identifying microstructural characteristics controlling transformation and twinning stress in tension. The results of the quasi-static testing will inform intermediate strain rate testing (10^{-1} 10^{0}/s) also conducted at Mines. Initial testing of Ti-1023 was aimed at ascertaining the effect of grain size on transformation stress.

Samples of Ti-1023, Ti-15Mo, Ti-35Zr-10Nb wt.% (Ti-3510) and equiatomic TiZrNb were tested in the Kolsky bar apparatus in tension and compression at Sector 32-ID at the APS. This beamtime allocation was the first of three allotted by a proposal submitted in 2018. The experiments consisted of high-rate (10^2 to 10^3 s^{-1}) deformation of samples, while simultaneously capturing radiography imaging and diffraction data on two separate high-speed cameras. Effectively, this allows for simultaneous measurement of microstructural evolution and transformation events occurring during deformation, along with stress-strain response. Table 33.1 summarizes alloys and heat treatments of the samples tested at the APS. Initial results from this data set point towards promising findings and will pave the way to more targeted experiments during the 2020-1 run cycle.

33a.3 Recent Progress

33a.3.1 Quasi-static Ti-1023 Tensile Testing and Aging Behavior

Shortly after the last reporting period ended, natural aging was found to occur in the Ti-1023 alloy in the β quenched state. This discovery lead to the formulation of artificial aging treatments that lead to significant gains (2.87 fold increase for peak-aging) in yield stress, without concurrent loss in ductility, as presented in Figure 33.1. The phenomena was successfully modeled by a single thermally activated mechanism from 75 to 250°C, with an activation energy of 55 kJ/mol using isothermal aging data from dilatometry. TEM characterization of aged and undeformed conditions confirmed the hypothesis that athermal ω-phase coarsens and re-distributes itself into short rows as aging proceeds, as presented in Figure 33.2. Lastly, over-aging of the material was found to lead to a total inhibition of TRIP, which presents a severe loss in ductility accompanied by only minor gains in strength. This over-aging behavior was found to be caused by the formation of preferential slip bands in the material that lead to heterogeneous plasticity and low work-hardening. A publication of these findings is currently undergoing internal review and will be the subject of a presentation at MS&T'19 in Portland, Oregon.

33a.3.2 APS Dynamic Experiments

Data analysis has begun for the different data sets produced during our initial February 2019 beamtime. First and foremost, a generalized Matlab script was developed for reducing the mechanical data to obtain stress-strain curves from the oscilloscope data files. In essence, the raw data contains only the time-resolved strain pulse and load pulse measured by the strain gages and load cell, respectively, recorded by the oscilloscope. Accordingly, significant post-processing and timing calibration must be made to turn these data into a valid stress-strain curves. This script can be implemented directly from the raw data and will prove an invaluable asset during the upcoming beamtime, as the stress-strain curves will be readily available during testing for on-the-fly adjustments and decision-making for increased efficiency during beamtime. An example output stress-strain curve is presented in Figure 33.3a.

Radiogaphy and diffraction data have been post-processed and compiled into time-resolved animations, which allows for high-throughput screening of interesting candidates for more detailed microstructural characterization. The radiography data is especially useful for the timing of the mechanical and diffraction data. Lastly, HiSPod, a Matlab toolbox for synchrotron diffraction data analysis has been used to integrate the diffraction data into time-resolved diffraction patterns. The time-resolved diffraction data is indispensable to correlating microstructural evolution to stress-strain data, as stress and strain signal are also measured as a function of time. An example of a "frame"-resolved integrated diffraction pattern is presented in Figure 33.3b, where the vertical axis represents the number of frames taken from the start of deformation.

At this time, diffraction, mechanical and radiography data post-processing is nearing completion and analysis is underway. The biggest accomplishment has been screening through roughly 80 sample data sets to eliminate faulty, incomplete or otherwise unusable data. The current hurdle is timing all three data streams to correlate diffraction information with stress-strain curves.

33a.3.3 Tensile Testing of Ti-15Mo and Ti-1023

Quasi-static tensile testing of Ti-15Mo is underway. Experiments are currently aimed at identifying microstructural mechanisms that control twinning stress and work hardening in this alloy. Upcoming efforts will look into the effect of low volume fractions of α phase on twinning and mechanical behavior.

Intermediate strain rate testing has also begun for both alloys. Ti-1023 samples have been tested in the as-quenched, fully β and peak-aged state (900 s at 150°C), while the Ti-15Mo samples have only been tested in the β-quenched state. Mechanical data has been fully analyzed and microstructural characterization by light optical and transmission electron microscopy (LOM and TEM, respectively) are currently underway.

Mechanical data initially indicates that the aging mechanism does not interact strongly with strain rate. In essence, the gain in strength and loss of ductility as strain rate increases is similar between the as-quenched and aged samples, as can be seen in Figure 33.4. These data seem to indicate that the low-temperature aging of ω-phase is a promising strengthening mechanism for TRIP titanium alloys, even at higher rates of straining.

33a.4 Plans for Next Reporting Period

The work planned for the next reporting period is to conclude intermediate strain rate testing at Mines, which will consist mostly of 10^0 s^{-1} testing of both alloys. This testing will then be followed by LOM and TEM characterization of interrupted and fractured tensile specimens to elucidate how microstructural evolution changes in each alloy as a function of strain rate and β-phase stability.

In summary, the following work is planned in the upcoming months:

- Analysis of the APS dynamic data for Ti-1023, Ti-15Mo, Ti_{55} Zr_{35} Nb_{10} and an equimolar TiZrNb alloy;
- Tensile testing at 10^0 s^{-1} of Ti-1023 and Ti-15Mo;
- Preparation for upcoming beamtime at the APS during the 2020-1 run cycle; existing data will be analyzed and used to inform conditions to test in next round;
- LOM and TEM characterization of intermediate strain rate specimens;
- Bulk Kolsky bar testing in Compression at Los Alamos National Laboratory (LANL);
- In-Situ Quasi-static testing of Ti-1023 at Lawrence-Livermore National Laboratory (LLNL).

33a.5 References

[33.1] C. Brozek, et al., A β-titanium alloy with extra high strain-hardening rate: design and mechanical properties, Scripta Materialia 114 (2016): 60-64.

[33.2] F. Sun, et al., A new titanium alloy with a combination of high strength, high strain hardening and improved ductility, Scripta Materialia 94 (2015): 17-20.

[33.3] M. Marteleur, et al., On the design of new β-metastable titanium alloys with improved work hardening rate thanks to simultaneous TRIP and TWIP effects, Scripta Materialia ,10 (2012): 749-752.

[33.4] T.W. Duerig et al., Formation and reversion of stress induced martensite in Ti-10V-2Fe-3Al, Acta Metallurgica 30.12 (1982): 2161-2172.

[33.5] X. Min et al., Mechanism of twinning-induced plasticity in β-type Ti–15Mo alloy, Scripta Materialia 69.5 (2013): 393-396.

33a.6 Figures and Tables

Table 33.1 Heat treatment and alloys for the APS testing.

Alloys (wt.%)	**Heat Treatments (Expected Microstructure)**	
Ti-10V-2Fe-3Al	As-received (α+β)	850-1h-WQ (β+ω)
Ti-15Mo	As-received (α+β)	800-1h-WQ (β+ω)
Ti-35Zr-10Nb	700-1h-FC (α+α'')	700-1h-WQ (α'')
TiZrNb	700-1h-WQ (β)	--

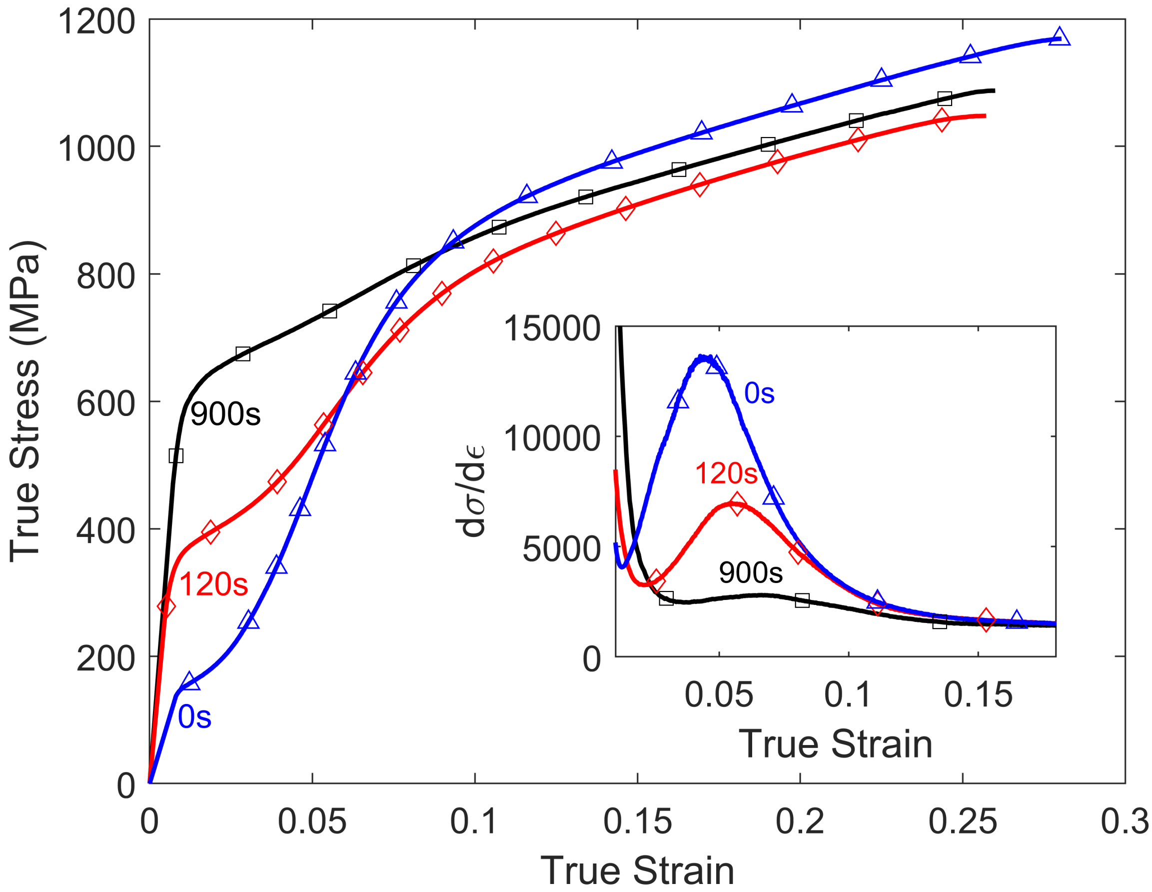


Figure 33.1. True stress strain plots of Ti-1023 aged at 423K (150 °C) for the number of seconds labeled next to the curves. Inset presents the work hardening curve of the corresponding stress-strain curves.

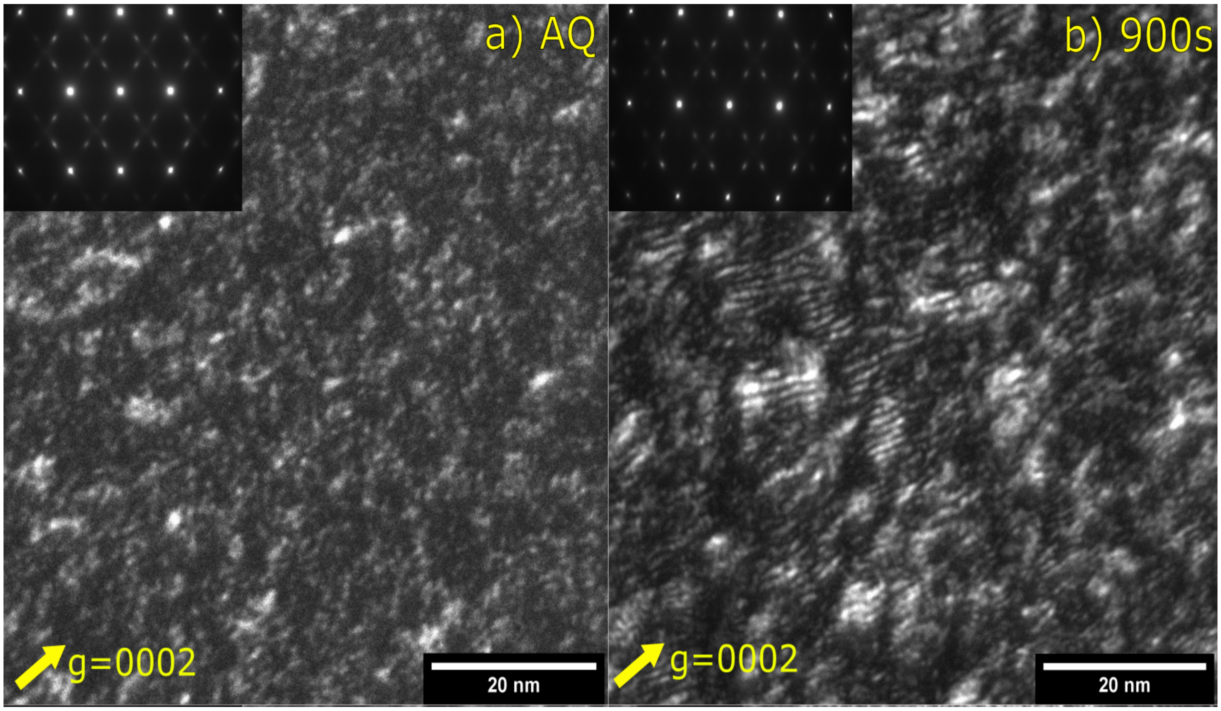
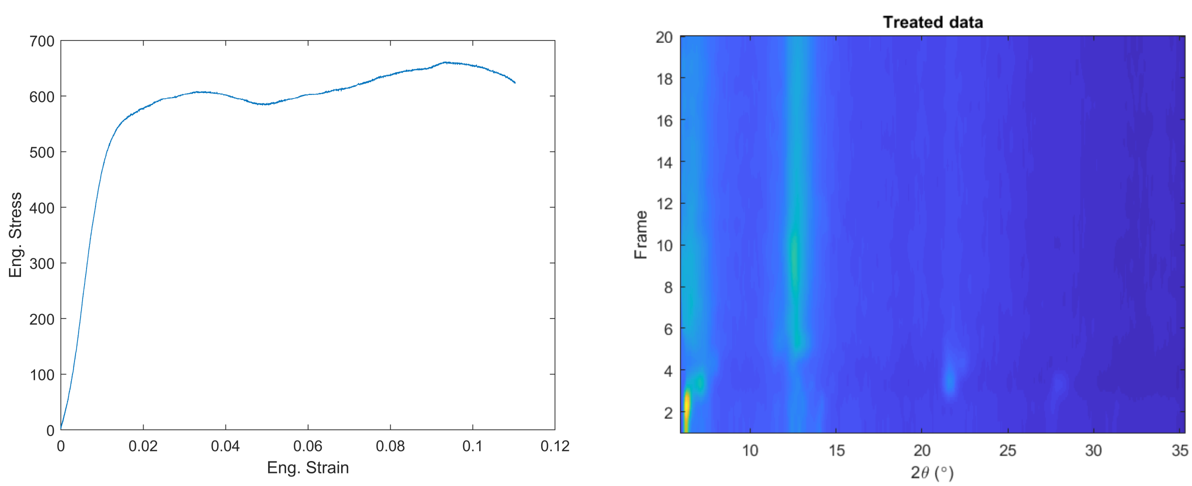


Figure 33.2. TEM two-beam dark field images of ω-phase precipitates in the a) as-quenched state and b) aged for 900s at 423K (150 °C) (peak-aged) state. Selected area diffraction patterns of the 110 zone axis are also included as insets.



(a) (b)

Figure 33.3. Example of a) high-rate mechanical testing data and b) diffraction from a Ti-1023 specimen obtained at the APS.

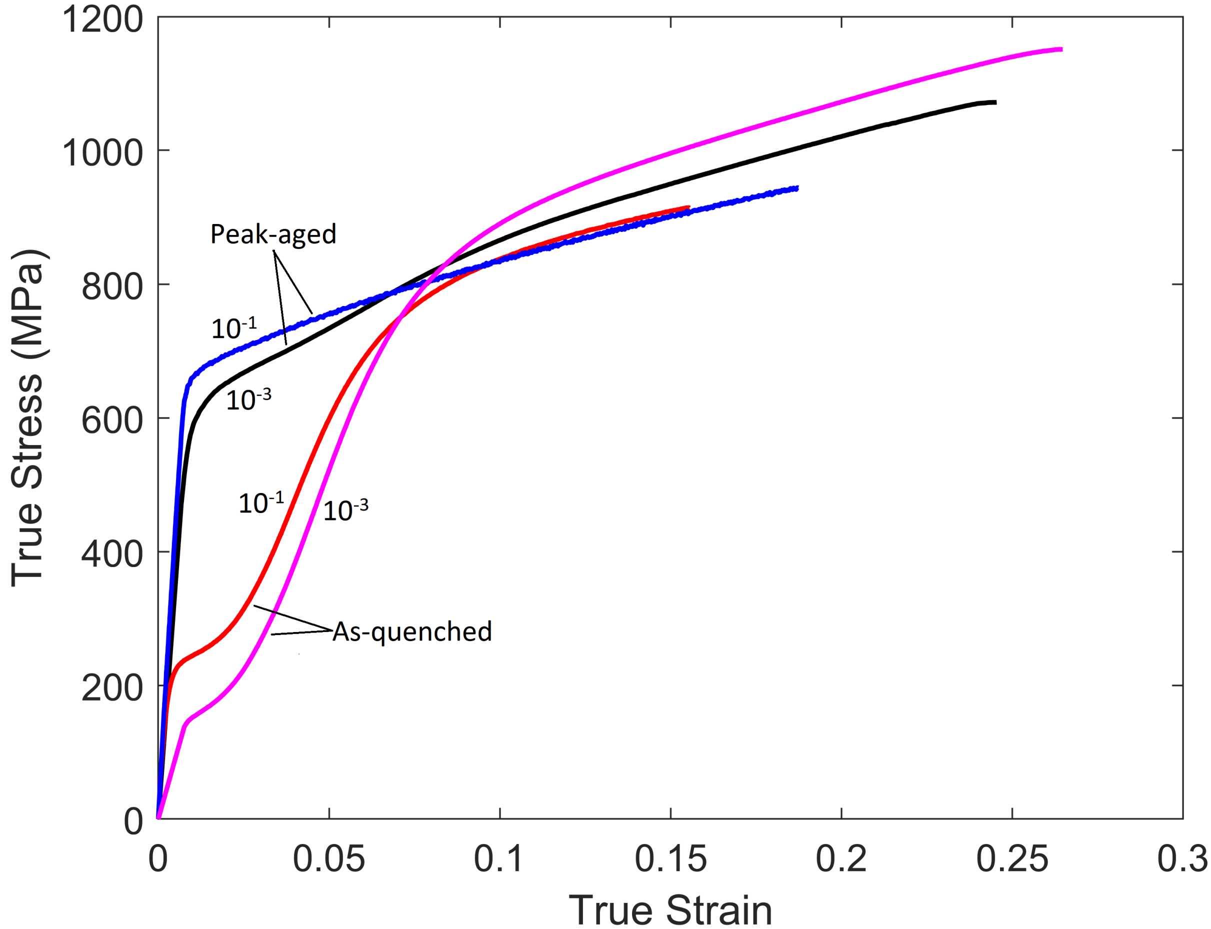


Figure 33.4. True stress-strain plot of Ti-1023 tensile specimens tested at 10^{-3} and 10^{-1} in the as-quenched and peak-aged conditions.