33.0 IN-SITU STUDIES OF STRAIN RATE EFFECTS ON PHASE TRANSFORMATIONS AND MICROSTRUCTURAL EVOLUTION IN BETA-TITANIUM AND MULTI-PRINCIPAL ELEMENT ALLOYS (LEVERAGED)

Benjamin Ellyson (CSM)
Faculty: Amy Clarke (CSM)
Industrial Mentor: TBD
Other Participants: Yaofeng Guo (CSM)

33.1 Project Overview and Industrial Relevance

Titanium alloys are heavily used in the aerospace and biomedical indsuctries for their high specific strength and good corrosion resistance. However, limited work hardening and uniform elongation have long limited their applicability in deformation controlled applications, where high abosrbed energy or high formability are required. The ability to develop novel titanium alloys that exhibit high work hardening would broaden their applicability to applications such as lightweight blast-resistant armor, crash resistant structural components and high-complexity plastically formed parts. Recent work [33.1-33.4] on metastable β-titanium alloys have 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, namely BCC titanium and high entropy alloys (HEAs) is mostly absent. The present project aims to study TRIP and TWIP effects in non-ferrous systems, specifically β-titanium and FCC HEAs, to garner fundamental understanding of the intrinsic and extrinsic variables controlling the expression of TRIP and TWIP effects. Although this project primarily focuses on β-Ti alloys, complementary experiments on HEAs will begin Summer/Fall 2018, either by a postdoc or via a new graduate student project. The fundamental understanding gained from this study will then be used to develop an alloy design methodology, allowing the specific tailoring of mechanical response and microstructural evolution by tuning transformation and twinning behavior by means of alloying and processing.

33.2 Previous Work

The project started in Fall 2018. As such, recent progress has been mostly restricted to literature review and preliminary material characterization. The literature review performed to date has revealed limited prior research on the subject of TRIP and TWIP in β-titanium. Only a dozen papers have been found, published by 2 or 3 different research groups, such as work by Sun, Brozek or Marteleur in the past 15 years [33.1-33.3]. Literature has been found on previous efforts to design low-modulus, super-elastic β-titanium alloys for biomedical implants [33.5], which have been harnessed to design TRIP and TWIP β-titanium alloys recently. This method reveals the effect of alloying elements on the chemical stability of the β phase (**Fig. 33.1**). Literature review was also concentrated on TRIP effects in Ti-1023 as material stock was readily available at CSM. This alloy has been shown to exhibit TRIP [33.6, 33.8] and TWIP [33.7] behaviors. Preliminary investigation was undertaken to find initial microstructural states that would promote TRIP and TWIP effects in Ti-1023. A schedule of solution treatments was developed to obtain a fully retained β structure. **Table 33.2** indicates the initial heat treatment schedule investigated. As of the last report, only the 1200°C helium quenched (HeQ) and 1100 °C slow cooled samples were presented. Optical microscopy of an etched sample, as well as transmission electron microscopy (TEM) investigation of a focused ion beam (FIB) liftout revealed a fully β structure with large amounts of the ω phase.

33.3 Recent Progress

33.3.1 Microstructural characterization

As of this report, all of the 8 heat treatments indicated in **Table 33.2** have been performed. The water quenched samples have been characterized by optical microscopy, while the helium quenched samples are still in preparation. The microstructure of the water quenched samples have revealed an inverse relationship between hold temperature of the solution treatment and the fraction of α'' athermal martensite present in the microstructure. The low temperature samples show an almost exclusively martensitic microstructure, while the sample treated at 1200 °C only exhibited martensite at the β grain boundaries. This trend is believed to be linked to increased grain size, which leads to decreased nucleation site density for the athermal martensitic transformation to occur upon quenching. Quantified results are in progress to correlate martensite phase fraction to grain size. In all cases, it has proven nearly impossible to produce a fully retained β microstructure and other heat treatment strategies are currently being investigated to mitigate martensite formation during quenching. These strategies include using other quenching media such as 5% saline brine, as recommended by the ASM handbook for solution treating Ti-1023, or may require an alloy with more ß stabilizers.

Another major concern that has arisen from this initial characterization is excessive grain growth during solutionizing. Literature [33.8] shows that there is an inverse trend between the propensity for mechanically induced transformation and grain size, such that excessively large grains hinder or even preclude the TRIP effect. Alternative heat treatments have been formulated which would produce retained β microstructures, while mitigating grain growth. These heat treatments include lower temperatures (800 to 900 °C), shorter times (no more than 0.5 h), and a more severe quench rate (5% saline brine). The samples have been produced and are awaiting characterization. Alternatively, thermomechanical processing may be necessary to obtain refined grain sizes from the as-received stock. Tests are planned to begin on the Gleeble at CSM.

33.3.2 Compressive Testing

TRIP and TWIP effects are inherently mechanically induced, such that mechanical deformation is necessary to elucidate the metastable β phase's propensity to exhibit them. Compressive tests were intitially proposed as a "high-throughput" method to screen the candidate solution treatments for TRIP or TWIP effects, as heat treatment samples could be directly tested in the load frame available in-house at CSM. Typical signs of transformation or mechanical twinning are a near constant stress plateau and increasing strain hardening rate, respectively. It is thus possible to screen samples for interesting microstructural evolution on the basis of the strain hardening rate shown during compression. **Fig. 33.3** shows strain hardening curves for 5 of the 10 investigated solution treatments. Most samples show no sign of transformation plateau (near-zero strain hardening rate) or twinning induced hardening (increasing strain hardening rate). Only the 1200°C-2h-HeQ sample shows signs indicative of twinning, as strain hardening plateaus following yielding and increases near the end of plastic straining (roughly 10% strain). These results are consistent with literature in that large β grain size precludes transformations, but promotes mechanical twinning. In this case, the 1200°C-2h-HeQ heat treatment presents the largest β grains and lowest martensite phase fraction, providing the highest uninterrupted β crystallite domain size for twinning to occur, thus lowering the twinning stress. Microstructural investigation of the compressed samples is underway and should confirm the trends in strain hardening behavior noted in the compression curves.

33.4 Plans for Next Reporting Period

By the next reporting period, extensive progress on the study of Ti-1023 is expected. The tensile testing phase should be near completion and the investigation into the effect of strain rate on microstructural evolution will be well underway. The heat treatment, processing and compression phase of this study will be complete by June and should lead to a first publication. However, the results from this phase are necessary for the initiation of the tension testing phase. Also, the second candidate alloy system, Ti-15Mo is in the process of acquisition from ATI. This alloy will be used specifically for more fundamental studies on the extrinsic factors controlling microstructural evolution, such as strain rate, temperature and strain path. This alloy will be subject to in-situ experiments to gain a better understanding of the interplay of the different mechanisms responsible for plasiticity in this alloy.

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

- Beam-time request submitted to APS for Fall 2018. Proposed experiments include high-strain rate diffraction and imaging experiments on Ti-1023 and Ti-15Mo in a Kolsky bar setup. Quasi-static straining experiments to monitor phase transfromations and twinning may also be pursued at the Cornell High-Energy Synchrotron Source (CHESS) and/or the APS or Los Alamos Neutron Science Center (LANSCE) at Los Alamos National Laboratory.
- Experimental work planned
    - Finish the compressive tests for all of the heat treatments planned (**Table 33.2**) to indentify the most promising conditions for TRIP and TWIP in Ti-1023;
    - Investigate strain rate effects on TRIP and TWIP of Ti-1023 in tension using candidate heat treatments found in the compressive study;

- Begin characterizing the Ti-15Mo alloy provided by ATI to identify heat treatment response and microstructural evolution;
- Continue literature review aimed at formulating analytical and numerical methods to inform alloy design, including refining the Bond order (Bo) and Mean d-orbital energy (Md) models [33.5].

33.5 References

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

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[33.3] Marteleur, Matthieu, 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* 66.10 (2012): 749-752.

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

[33.5] Morinaga, M., et al. "Theoretical design of titanium alloys." *Sixth World Conference on Titanium. III.* 1988.

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

[33.7] Ahmed, Mansur, et al. "Strain rate dependence of deformation-induced transformation and twinning in a metastable titanium alloy." *Acta Materialia* 104 (2016): 190-200.

[33.8] Bhattacharjee, A., et al. "Effect of β grain size on stress induced martensitic transformation in β solution treated Ti–10V–2Fe–3Al alloy." *Scripta Materialia* 53.2 (2005): 195-200.

33.6 Figures and Tables

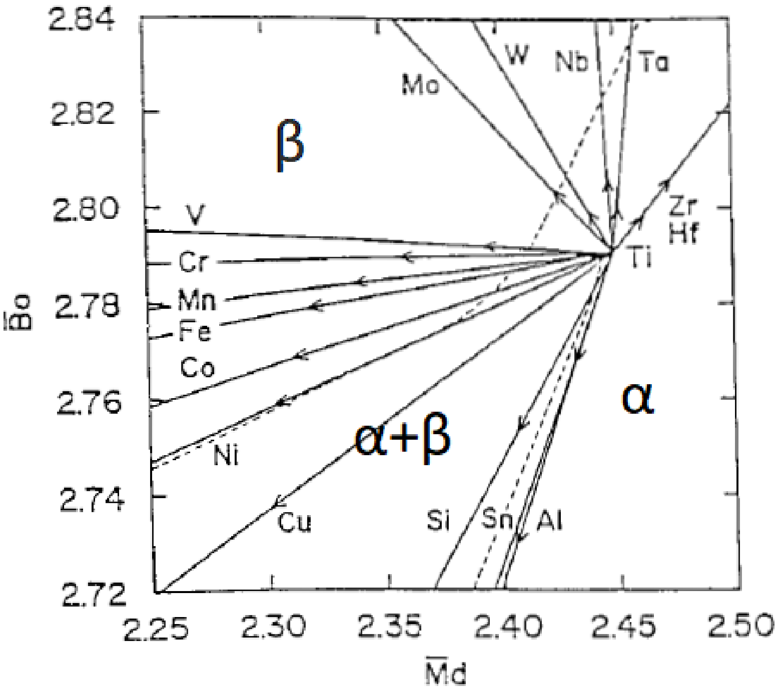

Figure 33.1 Alloying vectors showing the effect of solute concentrations on the stability of the β phase in terms of average Bond order (Bo) and Mean d-orbital energy (Md). These results were obtained from numerical simulations of titanium atom clusters. [33.5]

Table 33.1 Initial heat treatment schedule investigated for Ti-1023.

Temperature (°C)	Time (h)	Quench
900	2	Water or Helium
1000	2	Water or Helium
1100	2	Water or Helium
1200	2	Water or Helium

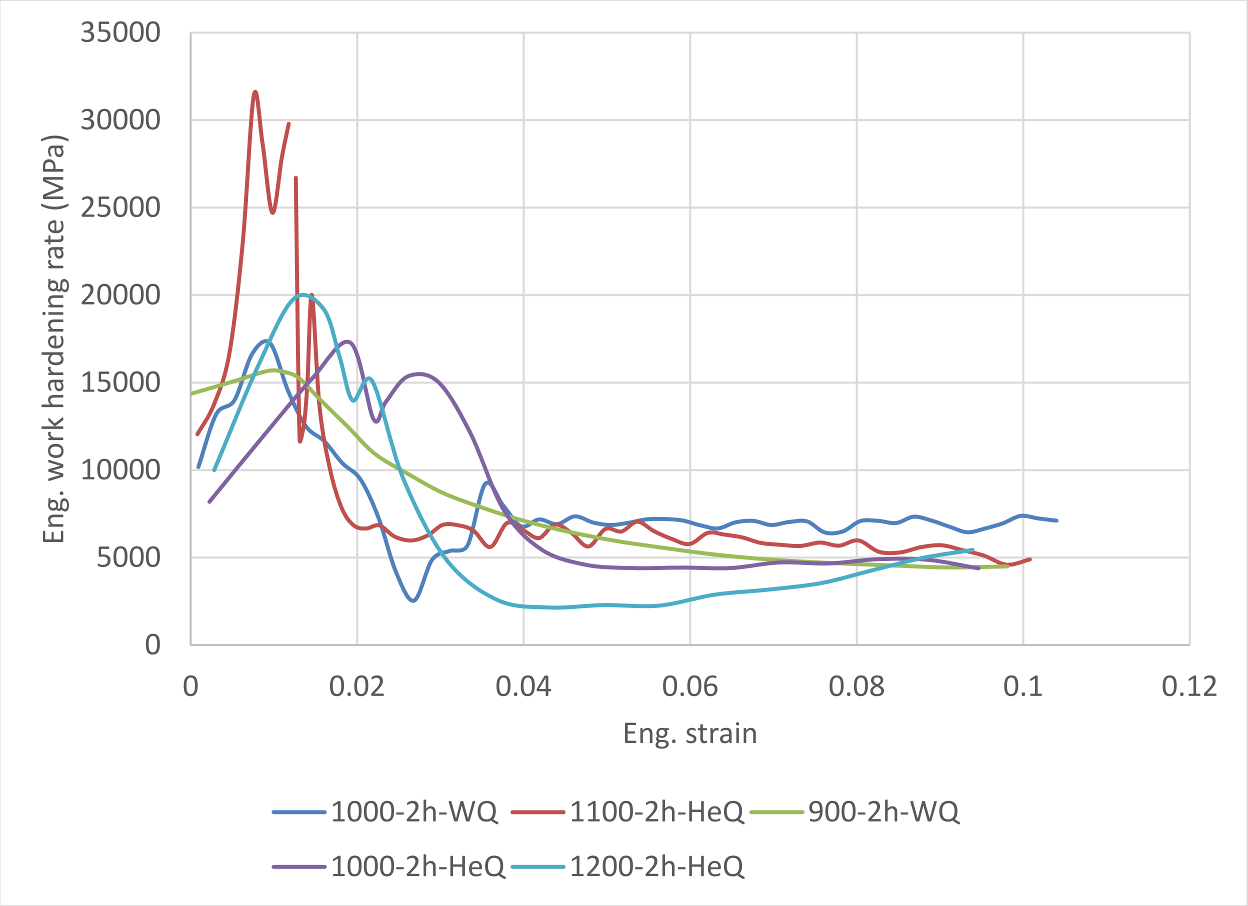

Figure 33.2: Instantaneous work hardening rate as a function of strain for all five samples tested. Note the increasing rate for the 1200-2h-HeQ sample from 0.06 to 0.1 strain.