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

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33A.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, 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 [33A.1-33A.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 (chemistry, microstructure) and extrinsic (temperature, strain rate, strain path) 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.

33A.2 Previous Work

The previous reporting cycle saw the completion of the initial quasi-static compression testing and microstructural characterization of the titanium alloy Ti-10V-2Fe-3Al wt.% (Ti-1023). The goal of this initial study was to rationalize the effect of solution heat treatments at temperatures above the β transus of Ti-1023 (795°C) on metastable phase evolution and deformation mechanisms in compression. The findings of this initial study indicated that grain size was a controlling factor in the yield stress and work hardening exhibited in compression. However, subsequent literature survey suggests that the heat treatment schedule used in this compression study and all other studies published to date varied the final temperature from which the samples were quenched to room temperature (RT). As such, different isothermal hold temperature lead to different grain sizes, but also affect athermal ω formation by altering the cooling rate during the quench. As such, it was rationalized that the heat treatment schedule for the quasi-static tension study would have to be formulated to decouple these effects of grain size and athermal ω. Thus, a heat treatment schedule with an initial homogenizing anneal and a final solution treatment from just above the β transus (850°C) would allow for the production of two microstructures yielding differing grain sizes, but with similar athermal ω formation, due to the final quench from a similar temperature. The heat treatment schedule is shown in Table 33A.1.

As previously reported, initial testing was completed to validate the proposed experimental plan. Samples of all three conditions (α/β produced by the 700°C-1h treatment, small grained (SG) fully β produced by the 850°C -1h treatment and big grained (BG) fully β produced by the 1100°C -3h treatment) were tested and compared. The true stress-true strain plots of the three tested previously mentioned conditions clearly show a difference in behavior among the large and small grained fully β specimens (1100°C -3h and 850°C -1h, respectively), and an even larger difference between both β samples and the α/β state (700°C -1h), which represents a fully chemically stable β matrix, as it does not exhibit TRIP/TWIP effects (an even larger difference in the stability of β phase as the latter does not show TRIP/TWIP effect).

33A.3 Recent Progress

33A.3.1 Quasi-static Tension Testing

Quasi-static tension testing of Ti-1023 in the SG and BG conditions has been completed. Analysis of the mechanical data, as well as post-mortem characterization of the specimens, is underway.

Transmission electron microscopy (TEM) characterization and X-ray diffraction (XRD) measurements confirm that the totality of deformation product seen at quasi-static strain rates is α'' martensite; no evidence of matrix twinning has been found. The observation of α'' martensite (and not twinning) supports the finding that Ti-1023 exhibits primarily transformation induced plasticity (TRIP) at quasi-static strain rates.

Initial analysis of the stress-strain data of the samples tested to failure indicates that the SG and BG mechanical behavior is not as markedly different as initial testing seemed to indicate. Specifically, the difference in yield stress and work hardening rate is decreased between the two conditions. This behavior is thought to be attributable to the 850°C water quench (WQ) treatment given to all conditions, as this step is the only difference between the two grain sizes As such, the original assumption that the cooling rate from the β regime affected the formation of atermal ω seems to be validated, although grain size still plays a significant role. In fact, atermal ω has been reported to mediate the formation of α'' during deformation in Ti-Nb binary alloys [33A.4].

Interrupted strain tension tests were performed to elucidate the formation of α'' martensite in the matrix. Tension tests to failure and compression tests to a final strain of 5-10% have demonstrated that the microstructure evolves very rapidly at the onset of plastic deformation to a point where post-mortem characterization becomes difficult. As such, interrupted tension tests were conducted to capture the microstructural state at early stages of deformation to characterize the formation and interaction of martensite laths with ω precipitates.

33A.3.2 APS Dynamic Experiments

Beamtime was allocated to this project in February 2019 at Sector 32-ID-B at the Advanced Photon Source (APS) at Argonne National Laboratory. The experiements consisted of Kolsky bar tests in compression and tension, while collecting in situ imaging and diffraction data from samples. The tests were performed at high strain rates on the order of 103 s-1. Multiple alloys were tested, including: Ti-1023, a Ti-15Mo wt.% binary alloy, and two medium entropy alloys - Ti55 Zr35 Nb10 and an equimolar TiZrNb alloy. The Ti-15Mo alloy was expected to undergo TWIP behavior [33A.5] since it is at the higher end of Mo equivalence. The two ternary compositions (TiZrNb alloys) were studied as candidate Ti-containing multi-principal element alloys (MPEAs) to form a link with the MPEA portion of this ONR project.

Simultaneous collection of mechanical, imaging and diffraction data will enable the comparison of microstructural evolution as a function of strain and strain path for given strain rates. An analysis of diffraction patterns during deformation will allow for the observation of new phase formation (e.g. TRIP), which is indicated by the appearance of new spots or rings in addition to those attributed to the matrix phase.. Broadening or filling out of diffraction rings attributed to the matrix phase may be indicative of crystallite domain size refinement. **Figure 33A.1** gives an example of imaging and diffraction data for a sample of Ti-15Mo tested in tension. **Figure 33A.1a** shows a diffraction pattern and image prior to deformation. **Figure 33A.1b** shows an image and diffraction pattern corresponding to fracture of the tensile specimen, indicating that the initial partial ring (prior to deformation) has filled in, possibly from crystallite refinement due to deformation twinning.

33A.3.3 Initial Characterization of Ti-15Mo

Samples of Ti-15Mo have been solution heat treated to produce a fully retained β microstructure of equiaxed grains. Material was subsequently rolled to 30% reduction in thickness to introduce plastic deformation. The deformed sample exhibited evidence of twinning product with optical microscopy. Quasi-static tensile tests are planed in the near future.

33A.4 Plans for Next Reporting Period

During the next reporting period, a quasi-static tension study of the Ti-15Mo alloy provided by ATI will be performed using similar conditions employed for Ti-1023. Analysis of the quasi-static data already produced for Ti-1023 will also be completed. This analysis will inform the experimental conditions for future intermediate strain rate testing of both alloys using a custom MTS servo-hydraulic load frame at Mines.

Because early results from Ti-1023 indicate that the ω phase mediates TRIP and TWIP effects, further literature review on this subject is planned to inform an upcoming experimental plan. The next series of experiments will combine dilatometer testing at various cooling rates and low temperature aging treatments to ascertain the effect of ω phase formation and evolution on subsequent deformation mechanisms. Tensile testing will be performed to link the dilatometer measurements to changes in mechanical behavior.

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

- Analysis of APS dynamic data for Ti-1023, Ti-15Mo, Ti55 Zr35 Nb10, and equimolar TiZrNb alloys;
- Analysis of quasi-static tensile testing of Ti-1023;
- Quasi-static tensile testing of Ti-15Mo;
- Intermediate strain rate testing of Ti-1023 and Ti-15Mo;

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] Moffat, D. L., et al. The compctition between martensite and omega in quenched Ti-Nb alloys. Metallurgical Transactions A 19.7 (1988): 1677-1686.6.

[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 33A.1: Heat treatment schedule for tensile study of Ti-1023.

Temperature (°C)	Time (h)	Quench	Comment
700	1	Water	α/β homogenizing step
1100	3	Water	Optional grain growth step for large grain samples
850	1	Water	Homogeninzing solution treatment (athermal ω phase)

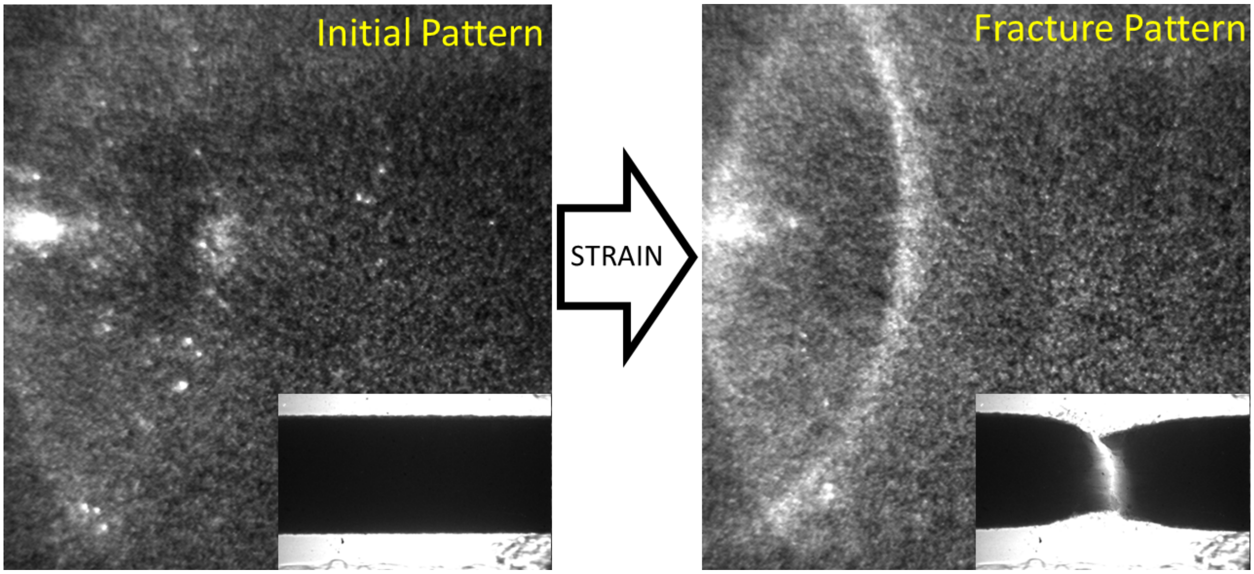

Figure 33A.1 (a, left) Evolution of the diffraction pattern of a Ti-15Mo sample from the initial state (spotty pattern on the left) until (b, right) fracture (continuous ring pattern on the right), with the corresponding mechanical test images as insets. Strain rate was roughly 102.