A.F. Jankowski et al.: The softening factor $c_b$ of commercial titanium alloy wires

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The strain-rate sensitivity exponent $m$ and activation volume $v^*$ are often used to characterize the strain-rate sensitivity of strength behavior in metals and alloys. Complications can arise when the $m$ and $v^*$ values become indeterminate, due to factors such as an inherent scatter in the mechanical property data. The study of commercial Ti-alloy wires is considered wherein to overcome this limitation, the formulation of the Kocks–Mecking (K–M) model is modified to provide a parameter $c_b$ that characterizes the microstructural scale responsible for the observed plasticity and work hardening behavior. The softening factor $c_b$ is found to be independent of strain-rate for the Ti-alloy wires of this study. It is proposed that $c_b$ can offer a versatile and complementary computation to the activation volume $v^*$ since its formulation includes the yield and ultimate strength values along with the plastic strain. For the tensile testing of Ti-alloy wires, a low $c_b$-value of 14 is calculated for Ti-6Al-4V that is consistent with >10% plasticity during work hardening whereas a high $c_b$-value of 135 for Ti-6Al-7Nb corresponds with <4% plasticity.

Keywords: Softening factor; Work hardening; Plastic strain; Ti alloys

1. Introduction

Titanium (Ti) alloys are used in the aerospace, marine, chemical, athletic, and biomedical industries [1]. The quasi-static mechanical properties of grade 5 Ti-6Al-4V are similar to those of bone with excellent osseo-integration compatibility [2–4]. In addition, Ti alloys cause fewer allergic reactions [5, 6] than other implant materials. However, a low wear resistance coupled with the motion of implants relative to the implant site implies [7] deficient in-service lifetimes [8] as observed for total hip implants. Although oxide and ni tride coatings both increase surface hardness and improve corrosion resistance, Ti-implants remain at risk of tensile failure, or rejection by the body during osseo-integration when bone grows around the alloy implant. The use of niobium (Nb) as a solute addition is reported [9, 10] to enhance biocompatibility in comparison to Ti-6Al-4V alloy due to decreased toxicity and allergic reactions [11]. Beyond quasi-static tensile testing [12], the strain-rate sensitivities of commercial Ti, Ti-6Al-4V, and Ti-6Al-7Nb wires have received relatively little attention. Higher strain-rate loading conditions from $10^{-3}$ to $10^2$ s$^{-1}$ are of interest [13, 14] for biomedical applications. Thus, the assessment of strain-rate dependent strength for commercial-grade biocompatible Ti alloys is of practical interest [15] for improved reliability and longer life of both orthopedic and dental implants. For this purpose, the assessment of strength behavior for biocompatible Ti-alloys as a function of strain rate is pursued through the tensile testing of commercially available spooled wire. It is anticipated that complications may arise in the computational analysis of the strain-rate sensitivity exponent $m$ and activation volume $v^*$ as formulated from continuum models due to an inherent variability of the wire material and apparent scatter in stress–strain results.

The classic use of the Kocks–Mecking (K–M) model [16] provides an understanding of the work hardening rate of materials that undergo extensive plastic deformation. The stress–strain relationship from mechanical tests can be replotted in a different form. Specifically, a K–M plot de-
picts the variation of work hardening rate $\Theta$, i.e. $d\sigma/d\varepsilon$, with stress $\sigma$ where the work-hardening is evaluated at specific conditions of temperature and strain rate. Often, and of specific interest, are the progressive stages of plasticity that are observed, during homogeneous deformation, prior to localization. Here, a K–M plot may result [14, 16] with the appearance of a decrease in the work hardening rate in three distinct stages, i.e. III–IV–V. Recent applications of the K–M model include the assessment of work hardening behavior in Cr–Mo steel [17] and the evaluation of strain hardening to determine dynamic recrystallization [18]. Although numerous constitutive models have been developed to replicate the plasticity behavior, the formulation of an activation volume for the onset of plasticity is not addressed, as noted above. Starting with the K–M model, Morris Jr. has developed [19] a formalism for the operative scale of microstructure during work hardening as identified by the structural softening factor $c_b$. The tensile testing of commercial Ti-alloy wires is pursued across four-orders of magnitude in strain rate that enter the lower range of conditions of interest for biomedical application. The conditions selected for this study will suffice to benchmark the use of a 3-variable input to uniquely determine a structural scaling factor $c_b$ that proves indeterminate using fewer variables. The yield strength, plastic strain, and ultimate strength are now used to determine a characteristic softening factor $c_b$ to the microstructure, and its dependence on strain-rate.

2. Morris, Jr. modification of Kocks–Mecking formulation

An approach to increase the strength of an alloy is through the thermal evolution of its microstructure. Heat treatments are used to selectively evolve a strengthening precipitate-phase from a homogeneous alloy. The precipitate particle may be coherent or incoherent with its matrix phase as dependent upon the processing method. An engineering application for structural materials is found for the case of ultra-high strength steels [19–23], i.e. the case for supersteels. A method to process inexpensive, low-alloy steels to very-high strength is to refine microstructure by incoherent phase transformations as, e.g., by application of thermo-mechanical controlled processing (TMCP) during the austenite–ferrite transformation on cooling. The presence of incoherent precipitate particles permits the matrix to deform plastically while maintaining its inherent ductility. Nanostructures in pure supersteels are unlikely since ultra-high strength via an ultra-refined grain size is not likely without an extreme loss of ductility. However, it can be shown that metallurgical approaches to mitigate limited ductility are available through the use of multiphase or metastable microstructures. To this end, a modified K–M model as developed by Morris, Jr. [19] considers the size effects present to optimize strength for the case of incoherent fine-grained ferrite. A structural parameter $c_{eb}$, i.e. a softening factor, is formulated through use of the yield strength $\sigma_y$, ultimate strength $\sigma_u$, and the plasticity $\varepsilon_p$ of the alloy. The inclusion of these three parameters can provide further insight into plastic deformation, in addition to or in lieu of the computation [24] of an activation volume $v^*$. The equations (1–10) that follow for the modified K–M model are formulated [19] beginning with consideration of the Hall–Petch equation which relates $\sigma_y$ to grain size $d$ as

$$\sigma_y = \sigma_0 + K_y \cdot d^{-1/2}$$

(1)

where $K_y$ is the Hall–Petch slope. Similarly, the cleavage fracture stress $\sigma_f$ for high-strength steels can be expressed as

$$\sigma_f = K_f \cdot d^{-1/2}$$

(2)

An equation used for predicting the ductile–brittle transition temperature $T_B$ is expressed as

$$T_B = T_o - K_B \cdot d^{-1/2}$$

(3)

If the strength is assumed to be a linear function of temperature, then the coefficient $K_B$ governs the ductile–brittle transition. $K_B$ is derived after subtracting Eq. (1) from (2), solving for $d^{1/2}$, and then substituting into Eq. (3) as

$$K_B = - (K_f - K_y) \cdot (d\sigma/dT)^{-1}$$

(4)

The coefficient $K_B$ is positive when $K_f > K_y$, as is the case for iron. The ultimate tensile strength $\sigma_u$ is found in the stress–strain diagram of Fig. 1 at the location where necking begins to occur. In the Considère criterion for determining tensile strength, a point on the true stress–true strain curve is graphically located that has a sub-tangent of unity. That is, $\sigma = \sigma_u$ when

$$d\sigma/d\varepsilon = \sigma/(1 + \varepsilon)$$

(5)

The onset of plastic instability occurs as the rate of specimen thinning exceeds the rate of strain hardening, i.e. when

$$d\sigma < (1 + \varepsilon)$$

(5a)

By taking the differential of Eq. (5a) with respect to $d\varepsilon$

$$d\sigma/d\varepsilon < 1$$

(5b)

The useful tensile ductility of a specimen is its uniform elongation, that terminates with the onset of necking, i.e. the plastic instability which occurs at the ultimate strength $\sigma_u$. The ultimate strength is determined in a test on a cylindrical specimen by the true stress $\sigma$, and the work hardening function $\Theta$ that equals $d\sigma/d\varepsilon$ according to the Considère

\[ \Theta = \frac{d\sigma}{d\varepsilon} \]

\[ \sigma = \sigma_0 + K_y \cdot d^{-1/2} \]

\[ \sigma_f = K_f \cdot d^{-1/2} \]

\[ T_B = T_o - K_B \cdot d^{-1/2} \]

\[ K_B = - (K_f - K_y) \cdot (d\sigma/dT)^{-1} \]

\[ \sigma = \sigma_u \text{ when } d\sigma/d\varepsilon = \sigma/(1 + \varepsilon) \]

\[ d\sigma < (1 + \varepsilon) \]

\[ d\sigma/d\varepsilon < 1 \]

\[ \Theta = \frac{d\sigma}{d\varepsilon} \]
criterion as
\[ \Theta_u = \left[ \frac{d\sigma}{d\varepsilon} \right]_u = \sigma_u \] (6)

The true strain \( \varepsilon \) at the instability point is determined by evaluating the integral from \( \sigma_y \) to \( \sigma_u \), that's given by the expression

\[ \varepsilon = \int \left( \frac{d\sigma}{d\sigma} \right) \cdot d\sigma \] (7a)

After substituting Eq. (6) into Eq. (7a), an expression for \( \varepsilon \) is then

\[ \varepsilon = \int \left[ \frac{1}{\Theta(\sigma)} \right] \cdot d\sigma \] (7b)

The work hardening function \( \Theta(\sigma) \) for many metals and alloys is described by a linear equation of the K–M form, as

\[ \Theta = \Theta_0 - c_b \cdot \sigma \] (8a)

where \( \Theta_0 \) is a constant, and \( c_b \) is the softening factor parameter. Rewriting Eq. (8a) to solve for \( \Theta_0 \) at \( \sigma = \sigma_u \), noting the equivalence of Eq. (6), it is found that

\[ \Theta_0 = \Theta + c_b \cdot \sigma \] (8b.1) \[ \Theta_0 = \Theta_u + c_b \cdot \sigma_u \] (8b.2) \[ \Theta_0 = \sigma_u + c_b \cdot \sigma_u \] (8b.3)

The work hardening function \( \Theta \) is determined from Eqs. (8a) and (8b.3) as

\[ \Theta = \sigma_u + c_b \cdot (\sigma_u - \sigma) \] (8c)

For the case of an incoherent fine-grained ferrite, it's assumed that the hardening behavior is approximated by a single constitutive equation since there are no competing effects from the microstructure. By substituting Eq. (8c) into Eq. (7b), the strain \( \varepsilon \) is derived from \( \sigma_i \) to \( \sigma_u \) as a function of the reduced stress \( \sigma^* \) as

\[ \varepsilon = \left( \frac{1}{c_b} \right) \cdot \ln \left[ \frac{1 + c_b \cdot (1 - \sigma^*)}{1 + c_b \cdot (1 - \sigma^*_u)} \right] \] (9)

where \( \sigma^* = a \alpha \sigma_u \). The true strain \( \varepsilon_p \), from the yield point to the instability, can then be determined by evaluating the solution to the integral from \( \sigma_i \) to \( \sigma_u \) as

\[ \varepsilon_p = \left( \frac{1}{c_b} \right) \cdot \ln \left[ 1 + c_b \cdot (1 - \sigma_y^*) \right] \] (10)

where \( \sigma_y^* = \sigma_y \). In general, the observed trend is that as \( \sigma^* \) approaches one, the corresponding value for \( \varepsilon_p \) goes to zero. This is a boundary condition, i.e., \( \sigma^* \) equals one when \( \sigma_y = \sigma_u \) and Eq. (10) gives a corresponding value of zero for \( \varepsilon_p \) equaling \( (1/c_b) \cdot \ln[1] \). Again, \( \varepsilon_p \) represents the amount of plastic strain between the yield point and ultimate strength.

With respect to application of nanostructured supersteels, the optimal dual-phase structure [19] for enhanced strength and plasticity could be composed of small martensitic islands intermixed with incoherent ferrite. Although the modified K–M model is developed for determining the softening factor of two-phase alloys, there is no exclusion that prohibits application to the plastic behavior of complex alloy structures as well. The experimental measurement of \( \sigma_y, \sigma_u, \text{ and } \varepsilon_p \) allows for the determination of whether or not the underlying structural parameter, i.e., the softening factor \( c_p \), is invariant with the applied strain rate.

3. Experimental methods

The materials used in the tensile experiments were pure Ti (0.994 purity), Ti-6Al-4V (grade 5), and Ti-6Al-7Nb welding wires as-received in spool form. The pure Ti and Ti-6Al-4V wires were hot drawn, whereas the Ti-6Al-7Nb wire was cold drawn. Metallographic samples were prepared in directions both (axial) and transverse (radial) to the 1.0–1.2 mm diameter wire axis. A 0.5 μm colloidal diamond suspension was used to produce the final polished surface. The materials were examined using a Rigaku Miniflex for X-ray diffraction (XRD) analysis, a JEOL JSM7600F scanning electron microscopy (SEM), and a JEOL JXA-8200 electron-probe micro-analyzer (EPMA). The samples were tested in tension over a range of strain rates produced using timed displacement of crosshead beams in a Test Resources® universal tester.

XRD scans of polished axial and radial surfaces of each material were conducted using a diffractometer operated in the 0/20 mode with monochromatic CuKα radiation as generated at 30 kV/20 mA. The XRD scans were collected over a 2θ-range of 30°–90° using a Δθ increment of 0.05° and 2 s dwell-time interval. The scans revealed the crystalline Bragg reflections of the constituent alloys, the presence of texturing along the drawn wire axis, and the possible presence of multiple phases.

The microstructural features of the samples were revealed from the axial and radial surfaces with the secondary-electron imaging mode of an SEM. The low-angle, secondary-electron detector (below-the-lens) imaging (LEI) mode enhanced surface relief from the polished samples that were treated using a low-contrast, Kroll’s etchant – a dilute acid solution of 6% nitric and 2% hydrofluoric as swabbed onto each sample surface for 10–15 s. A mean linear-intercept calculation was used to determine the average size of the constituent microstructure. The size calculation can include a shape factor since the grains are assumed to be a geometric variant of spheres–to-cubes. The elemental composition of each alloy was determined both by energy dispersive spectroscopy (EDS) with the SEM using characteristic X-rays, and by wavelength dispersive spectroscopy (WDS) with a EPMA.

The mechanical properties were determined using tensile test wire samples each 250 mm in length. The diameters of each Ti, Ti-6A-4V, and Ti-6Al-7Nb wire were measured where nominal values were 1.19 mm, 0.88 mm, and 1.02 mm, respectively. The wire samples were tensile mounted according to ASTM standards using a wire spool grip to produce a gage length of 92–122 mm. Loading occurs over a strain rate range of 10−3 to 10−1 s−1. To achieve this range, a nominal total displacement of 24 mm was applied over time intervals that range from 5 to 4000 s. These values were consistent with limits for the minimum and maximum velocities of the tensile test instrument. Tensile testing was used to determine the plastic strain \( \varepsilon_p \), yield stress \( \sigma_y \), and ultimate strength \( \sigma_u \) from measurements of load \( P \), initial wire diameter, gage length, and displacement of the wire during loading. A linear regression analysis was used to isolate the elastic loading regime. For consistency with the modified K–M model, it
was considered that the wire begins to yield at the proportional limit – to permit full measurement of plasticity during work hardening. Once the non-linear behavior began, the measure of plastic strain to failure was recorded.

4. Experimental results and analysis

The microstructure and composition of each wire material were examined using X-ray diffraction and electron microscopies. Tensile testing was used to determine the value for the softening factor $c_b$. Comparison of $c_b$ with the activation volume for plastic deformation was made along with its variation with the strain-rate.

4.1 Structure and Composition

SEM micrographs were obtained in the LEI imaging mode using a working distance of 8 mm, and a 5 keV incident electron beam. The LEI mode enhanced definition of the grain structure and the appearance of precipitate phases. Each sample was viewed along the axial and radial directions, as shown in Fig. 2. The pure Ti wire has an elongated grain structure in the Fig. 2a axial view, and a refined grain structure in the Fig. 2d orthogonal, radial view. Particles are present that could be attributed [25–27] to a Ti-hydride and/or impurity-stabilized $\beta$-phase. A primary $\alpha$-phase structure is seen in Fig. 2b and e Ti-6Al-4V and Fig. 2c and f Ti-6Al-7Nb samples as was found in prior studies [28, 29]. An estimate is made for the grain size $h_g$ of the matrix without the shape factor since the true shape geometry of the grains is unknown. The radial images were measured to provide mean linear-intercept $h_g$ values of: 1.1 ± 0.1 $\mu$m for the elongated structure of pure Ti; 3.1 ± 0.3 $\mu$m for the equiaxed structure of Ti-6Al-4V; and 1.9 ± 0.2 $\mu$m for the slightly elongated structure of Ti–6Al–7Nb. Microstructures similar to the cold-drawn condition observed in Fig. 2b and e are found [25, 26] in forged samples. Surface relief is evident with a second particulate phase at, or near, the grain boundaries.

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**Table 1. Weight % analysis of the Ti-alloy wires from EDS and WDS analysis.**

| X-ray line | Ti-6Al-4V | Ti-6Al-7Nb |
|-----------|-----------|-----------|
|           | matrix    | particle  | matrix    | particle |
|           | WDS EDS  | EDS       | WDS EDS  | EDS |
| Ti-K$_{\alpha}$ | 89.20 92.9 | 82.5 | 85.80 | 86.4 | 74.1 |
| Al-K$_{\alpha}$ | 6.30 3.4 | 2.6 | 6.27 | 6.2 | 4.7 |
| V-K$_{\alpha}$ | 4.45 3.7 | 14.1 | 0.25 | – | – |
| Nb-L$_{\alpha}$ | – – | – | 7.41 | 7.4 | 18.2 |
| Fe-K$_{\alpha}$ | 0.05 – | 0.8 | 0.27 | – | 3.0 |
For example, β-precipitates are seen [30] at grain boundaries in a primary (90%) α-phase matrix for Ti-6Al-4V heated to 550 °C, held at temperature, and then slow cooled. Similar images with a more-refined grain structure are seen in images of the Ti-6Al-7Nb alloy wire, Fig. 2c and f.

Composition analysis of the matrix and particulate phases, as seen in Fig. 2e and f was first conducted using EDS analysis. The characteristic X-ray peaks up to 10 keV were collected as emitted from the matrix and particle phases. The X-ray maps of interest used in the analysis were the 1.49 keV Al-Kα1, 2.17 keV Nb-Lα1, 4.51 keV Ti-Kα1, and 4.95 keV V-Kα1. The weight percent values are listed in Table 1 as computed using corrected intensities of the characteristic X-rays. A measured precision of ±0.04 wt.% occurs for this EDS analysis. It is seen that the Al content is slightly less in the matrix than the nominal value for the Ti-6Al-4V wire. In general, X-ray composition maps of the Ti-6Al-4V and Ti-6Al-7Nb second phase particulates indicate a diffuse enhancement of V and Nb, respectively, that may be evidence for β-segregation [26]. In addition, the presence of characteristic 0.62 keV L111b, 0.71 keV L111ab, 0.72 keV L11ab, 6.40 keV Kα, and 7.06 keV Kβ peaks for Fe are found in the EDS spectra from isolated particles. These EDS wt.% composition values listed in Table 1 are consistent with prior WDS microprobe measurements [31] of α- and β-phases in a (grade 5) Ti-6Al-4V alloy, where the matrix was found to be 6.7 wt.%Al-1.4 wt.%V and the particles were 2.9 wt.%Al-15.4 wt.%V-1.3 wt.%Fe. The present WDS compositions are taken from matrix areas of polished (but not etched) wire cross-sections – where 5 are from the radial, and 10 are from the axial sections of each sample. A statistical difference was not found between the composition values of the radial and axial sections. A precision of ±0.04 wt.% was measured for the WDS analysis. The WDS composition values listed in Table 1 appear equivalent to the nominal values of each wire material. In addition, WDS measurements of the pure Ti wire reveal an average impurity composition of 0.09 wt.%Al-0.30 wt.%V-0.02 wt.%Nb-0.05 wt.%Fe.

The Ti wire is a polycrystalline, hexagonal close-packed (hcp) structure, i.e. the α-phase. Basal plane (00.2) texturing along the axial direction is evident in the XRD scan of Fig. 3. Additional reflections in Fig. 1 for the pure Ti wire match those reported [32, 33] for the α-phase, where the peak positions are computed using lattice parameters \(a = 0.2952 \text{ nm} \) and \(c = 0.4681 \text{ nm}\). The XRD scan from the radial direction has a (10.0) texture, complementary to the (00.2) texturing found for the axial scan.

The addition of alloying elements as Al, V, and Nb to the α-phase of Ti can produce multiple phases and slightly change [25, 34–37] the lattice parameters of the α-phase to \(a = 0.292–0.293 \text{ nm} \) and \(c = 0.466–0.472 \text{ nm}\). The possible increase in \(c\) and slight decrease in \(a\) for the α-phase Ti alloy wires will tend to shift the (00.2) peak to a lower 2θ position, and increase the 2θ position of the (20.1) peak, both of which are seen in Fig. 3 when comparing the alloy to pure Ti scans. In addition, the presence of a face-centered-cubic (fcc) Ti(Al) γ-phase is reported [38–41] for nanocrystals and severely plastically-deformed, ball-milled powders. The position of the γ-phase (111) and (200) reflections in Fig. 3 are computed using a lattice parameter \(a = 0.409 \text{ nm} \) [38, 40], whereas a 0.440 nm value gives an (111) reflection at a 2θ position of 44.2°. Alternatively, the 44.2° peak position can be fitted to a common Ti-hydride phase. The peak positions for a body-centered-cubic (bcc) β-phase in the Ti-6Al-4V [37] and Ti-6Al-7Nb [34] alloy wires are shown in Fig. 3 as they would appear using a lattice parameter \(a = 0.329 \text{ nm}\). No distinct β-phase peaks are observed in the XRD scans of Fig. 3, except possibly a very diffuse (110) reflection.

4.2 Mechanical properties

A single wire tensile test contains regions that include: an initial nonlinear, pre-loading range where the wrapped ends of the wire are brought under tension to remove slack; a linear loading range of Hookean elastic behavior; and a plastic deformation range where yielding and strain hardening occur until failure. The logarithmic variation of change [25, 34–37] in yield strength \(\sigma_y\) with increasing strain rate is used to determine the coefficient \(m\) for strain-rate sensitivity through the
The yield strength will vary due to several factors that include surface finish and grain size, i.e. the Hall–Petch effect. However, specific effects of grain size and surface finish are not considered in this analysis. A typical tensile load \( P \) versus displacement \( z \) curve is shown in Fig. 4 for a 1.19 mm dia. pure Ti wire as tested at a strain rate of \( 1.19 \cdot 10^{-2} \) s\(^{-1}\). The Fig. 4 load–displacement \( P-z \) data is plotted that includes some of the initial pre-loading regime. The dashed-linear (red) curves are used to extrapolate the position of the origin (i.e. zero elastic displacement); and determine the proportional yield limit \( P_y \), the ultimate strength \( P_u \), and the amount of plastic strain.

Tensile test results of representative samples for each wire material are shown in the Fig. 5 stress–strain plots with corresponding strain rates indicated in the legend. The top curve set is for Ti-6Al-7Nb, the middle set is for Ti-6Al-4V, and the bottom curve set is for pure Ti. The yield point is determined using the proportional limit, rather than offset method, since the effects of work-hardening are significant for the wire tests. The tensile strengths measured are: 570–680 MPa for commercially pure Ti; 710–830 MPa for the Ti-6Al-4V alloy; and 850–1070 MPa for the Ti-6Al-7Nb alloy. The amount of plastic strain is reduced from greater than 0.10 for the Ti and Ti-6Al-4V wires, to less than 0.04 for Ti-6Al-7Nb. In general, the Fig. 5 stress–strain plots show that the yield strength \( \sigma_y \) increases slightly with increasing strain rate. Limited ductility is expected and found for the cold drawn, i.e. Ti-6Al-7Nb, wire material.

An alternative means to represent the stress–strain results for the Ti-alloy wire tensile tests is through the use of a K–M plot. The representative curves in Fig. 6 are plotted from several stress–strain curves of Fig. 5. The stress range shown in Fig. 6 is plotted as it begins with a rapid decrease in the coefficient \( \Theta \) equaling \( d \dot{\varepsilon}/de \) for the work hardening rate. A progressive decrease in the slope of the Fig. 6 curves is found through a succession of approximately linear regions. The three classic K–M work hardening stages are found for the Ti-6Al-4V curve in Fig. 6 for the test conducted at a \( 4.57 \cdot 10^{-2} \) s\(^{-1}\) strain rate. These linear regions correspond to applied stress levels up to 850 MPa for stage III, from 850 to 975 MPa for stage IV, and from 975 MPa to necking for stage V. The saturation stress is seen to increase through III as the applied strain rate increases, i.e. the K–M curve for a material shifts to higher stress with increasing strain rate. The presence of at least stages III–IV appear for the pure Ti wire. However, with its reduced plasticity, only stage III is apparently observed for the Ti-6Al-7Nb wire, as is reported elsewhere [18] for similar Nb-addition Ti–Al alloys. Stage III is associated with the trapping of dislocations, whereas the saturation stress is a result of dislocation annihilation. As for grade-1 pure Ti, in general, the transition from stage III–IV occurs at a few percent of plasticity, and from stage IV to V beyond \( \sim 10\% \).

The effect of strain-rate sensitivity on strength is quantified using a classic continuum approach with the In-scale relationship of the Dorn equation, as

\[
m = \frac{d(\ln \sigma_y)}{d(\ln \dot{\varepsilon})}
\]

The data plots of \( \ln \dot{\varepsilon} \) versus \( \ln \sigma_y \) for all of the wire tensile tests are shown in Fig. 7. The scatter in the data is anticipated and is attributable to the variation in the samples cut from the spool of wire material, i.e. the commercial grade wire materi-
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Fig. 7. The exponent \( m \) for the strain-rate sensitivity is the slope of the linear-curve fit of the ln–ln plots for Ti-6Al-7Nb (top curve), Ti-6Al-4V (middle curve), and pure Ti (bottom curve).

Fig. 8. The activation volume \( t \) is computed from the slope of the linear curves plotted in Fig. 7 for the strain-rate sensitivity is plotted as function of the strain rate during wire tensile testing of Ti-6Al-7Nb (green data), Ti-6Al-4V (red data), and pure Ti (blue data).

Fig. 9. The softening coefficient \( c_b \) is plotted as function of the strain rate during wire tensile testing of Ti-6Al-7Nb (green data), Ti-6Al-4V (red data), and pure Ti (blue data).

Fig. 10. The softening coefficient \( c_b \) is plotted as a function of the strain rate during wire tensile testing of Ti-6Al-7Nb (green data), Ti-6Al-4V (red data), and pure Ti (blue data).

The activation volume \( v^* \) for the onset of plasticity can be computed from the strain rate sensitive strength behavior using the following Cahn–Nabarro [24] relationship, as

\[
v^* = (k_B \cdot T) \cdot \left[ \frac{d(\ln \dot{\epsilon})}{d(\sigma_y)} \right]
\]

At room temperature, the product of Boltzmann’s constant \( k_B \) and temperature \( T \) equals 4.142 \cdot 10^{-21} \text{ J} \text{ mol}^-1 \text{ K}^{-1} \text{ nm}^3. \text{ The activation volume } v^* \text{ is the product of } k_B \cdot T \text{ with the slope of linear curves plotted in Fig. 8 for } \sigma \text{ versus } \ln \text{. The values of } v^* \text{ computed for Ti and Ti-6Al-4V are } 0.207 \pm 0.117 \text{ nm}^3, \text{ and } 0.230 \pm 0.090 \text{ nm}^3, \text{ respectively. These } v^* \text{ values correspond to } 26 - 29 \text{ dislocation volumes (of } b^* \text{) when assuming a typical Burger’s vector } b \text{ equal to } 0.2 \text{ nm. A value of } v^* \text{ for the Nb alloy is indeterminate using eqn. (12) since } \dot{\sigma}(\sigma_y) \sim 0 \text{ (hence } m_{f6Al7Nb}= 0), \text{ i.e. there is no statistical variation of strength } \sigma_y \text{ with strain rate.}

The modified K–M relationship provides a measurement, in addition to \( v^* \), for evaluating the scale of the underlying microstructure responsible for plasticity during tensile testing. The softening coefficient \( c_b \) is determined by fitting Eq. (10) with measured values of the plastic strain, yield, and ultimate strength using tensile test results such as those shown in the Fig. 5 curves. The variation of \( c_b \) with the strain rate is plotted in Fig. 9. The \( c_b \)-values are seen to be approximately constant across the strain rate range used in these tensile tests. The strain-rate invariant \( c_b \)-values are evidenced by the near-zero slope to the linear curve fit of versus \( c_b \) for each material. The constant \( c_b \) value as a function of is analogous to the constant value for the activation volume \( v^* \). Although no activation volume was determined for Ti-6Al-7Nb from the scattered data plot in Fig. 8, it is possible to determine a \( c_b \)-value using Eq. (10). The \( c_b \)-values for pure Ti, Ti-6Al-4V, and the Ti-6Al-7Nb wires are 19, 14, and 135, respectively. The parameters computed for the strain-rate sensitivity factor \( m \) using Eq. (11), the activation volume \( v^* \) using Eq. (12), and the softening factor \( c_b \) as fit using Eq. (10) are listed for all of the wire materials in Table 2.

5. Discussion

The microstructures of the wires revealed in the analysis of SEM images shown in Fig. 2 are consistent with an \( \alpha \)-phase matrix, and a dispersion of impurities that decorate grain boundaries. The orthonormal XRD scans of Fig. 3 show crystallographic texturing consistent with a wire-drawn stock material. A refined grain size of a few microns in the microstructure results from wire drawing process as well. There is a V and Nb enrichment found in the particulates, which may have some inhomogeneity in structure including some variations along the gage length in composition, diameter, surface finish, and grain size. The mechanical behavior of the test material is of interest with all of the inconsistencies therein, since an objective is to determine whether or not the use of the softening factor \( c_b \) introduced in Eq. (8a) provides insight to the scale of the underlying structure responsible for the strain-rate dependent strength behavior. From the slope of the linear curves in Fig. 7, the strain-rate sensitivity values for the Ti and Ti-6Al-4V wires are positive with similar exponents of \( m_{Ti} = 0.014 \pm 0.006 \), and \( m_{f6Al4V} = 0.014 \pm 0.008 \), respectively. A near zero, i.e. perhaps a negative, exponent \( m_{f6Al7Nb} = -0.008 \pm 0.007 \) is computed for the full Ti-6Al-7Nb data set with its inherent scatter in the tensile data.
as measured using EDS and listed in Table 1 where the composition values are similar to those reported for microprobe measurements of Ti-6Al-4V alloys [31].

The wire is a commercial product that has inherent structural and composition variations which leads to an inherent scatter in the range of tensile behavior for each type of material as seen in the data plots of Fig. 7 and 8. Specifically, the Ti-6Al-4V yield strength ranges from 710–830 MPa, and the Ti-6Al-7Nb wires have yield strengths of 850–1070 MPa. Tensile test results for the Ti-6Al-7Nb wires are consistent with an equiaxed α-phase microstructure [29] that has a concurrent strength of 910–980 MPa. The plasticity of the hot-drawn pure Ti and Ti-6Al-4V wires exceeds 10%, whereas the cold drawn Ti-6Al-7Nb wires is less than 4%. The limited ductility of the Ti-6Al-7Nb wires is consistent with its cold-drawn processing versus the greater plasticity found for hot-drawn pure Ti and Ti-6Al-4V wires.

The strain rate sensitivity coefficients m computed using Eq. (11) for the Ti-alloy wires are low, as seen in Fig. 6, at less than 0.015 for all samples over a strain rate range of 10^{-5}-to-10^{-1} \text{s}^{-1}. This behavior is typical for pure Ti and Ti-6-4 with m values of 0–0.03 that can be computed [42] across each stage of work hardening. Activation volumes \(v^*\) of 26–29 (\(b^3\)) dislocations are determined using Eq. (12) from a Cahn–Nabarro analysis [24] for the onset of plasticity in Ti-6Al-4V and pure Ti, respectively. However, the \(v^*\)-value is indeterminate for the Ti-6Al-7Nb data using Eq. (12) since \(\bar{\sigma}(\varepsilon)\sim0\). Despite this shortcoming, the modified Morris, Jr. model [19] proves useful to evaluate the scale of microstructure responsible for plastic deformation. The softening factor \(c_b\) is computed using Eq. (10) as a function of the yield strength \(\sigma_y\), ultimate strength \(\sigma_u\), and plastic strain \(\varepsilon_p\). A constant value for \(c_b\) is revealed with respect to a variation in strain rate for each wire material in the Fig. 9 data plots. The softening factor \(c_b\)-values of 14, 19, and 135 are computed for Ti-6Al-4V, pure Ti, and Ti-6Al-7Nb, respectively. Although the SEM images of Fig. 2 show similar microstructures, the lower \(c_b\) value is consistent with a greater amount of work hardening and plasticity for Ti-6Al-4V. Conversely, rapid work hardening behavior is observed for the Ti-6Al-7Nb alloy leading to its larger \(c_b\) value. This later result would indicate a greater \(v^*\) value as well, that would be consistent with a near zero value in the computation of \(m\) for Ti-6Al-7Nb, as shown in Fig. 7.

The microstructure for the drawn wire would be consistent with elongated grains, wherein cell interiors would have low dislocation density and the grain boundaries would have high dislocation density. To assess deformation mechanisms that are operative during plasticity, a K–M work hardening rate analysis is reported for measurements of grade-1 pure Ti under tension [43]. For tensile deformation, the stage III with a rapid, continuous decrease in \(\Theta\) has dislocation glide. This is followed by twinning in stage IV, until saturation of that particular system, as e.g., (10,0), occurs. Further twinning continues in stage V as deformation shifts to a new system as, e.g., (11,0). Although the commercial wires for this study do not show the extensive plasticity reported for grade-1 pure Ti [43], these three stages are observed as seen in Fig. 6. The appearance of multiple stages of work hardening is readily seen as well for pure Ti in the stress–strain results of tensile tests at various strain rates [42]. The tensile behavior of the Ti and Ti-6-4 wires is consistent with this reported behavior wherein Stage III of the K–M behavior is associated with the trapping of dislocations. In addition, the presence of stages III and IV is reported [44] in a K–M plot for the work hardening behavior of Mg–Li alloys under tension. The strain to failure of 6–16% is not extensive but is similar to the commercial wires tested in the present study.

Modeling efforts have evolved to further evaluate the strain hardening rate effects in plastically deformed metal alloys beyond the basic K–M formulation. Extension of the K–M model to include the dependence of solute concentration on cross slip is considered in the hardening behavior of the Fe–Al alloy system under tension [45]. The interplay, i.e., coupling, between the edge and screw dislocation density at cell walls and within the cells, respectively, is used in modeling efforts to evaluate the dynamics of plasticity beyond stage III [42, 46–48]. Edge dislocations climb in the cell walls whereas screw dislocations undergo cross-slip within the cell interiors where the interplay between these two attributes is assumed to occur under the same total strain rate. Efforts to develop strain-hardening models for Ti–Al alloy behavior using K–M plots [18] again evidence a sequence of deformation modes similar to this study for Ti-6Al-7Nb alloy wires, wherein an initial rapid work hardening is observed. Whereas an extended range of plasticity beyond initial yielding is reported for the hot deformation of Ti–Al alloys that have a complex microstructure with multiple phases which undergo recrystallization [18], this scale of behavior is not seen in Fig. 5 for the room-temperature testing of the hot-drawn, single-phase Ti-alloy wires of this study. The current model [19] is not intended to simulate the details of strain hardening wherein the evolution of deformation modes with applied stress evidence changing mechanisms of dislocation-based deformation. The derivation presented is intended to provide a scale for the microstructure in the coefficient \(c_b\) that underlies the deformation behavior from yielding to the ultimate strength as evaluated at the plastic instability using the Considère criterion. Whereas \(c_b\) can be uniquely determined, a traditional approach to evaluate an activation volume for deformation proves problematic for the deformation behavior for the untreated, commercial Ti alloy wires. The Morris, Jr. model [19] provides another method to assess the work hardening behavior, and is capable of providing a microstructural parameter \(c_b\) that unifies the complex behavior of Ti-alloy wires. The use of the Morris, Jr. model may

| wire material   | strain-rate sensitivity factor m | activation volume \(v^*\) (nm\(^3\)) | softening factor \(c_b\) |
|-----------------|----------------------------------|----------------------------------------|-------------------------|
| Ti              | 0.014 ± 0.006                    | 0.207 ± 0.117                          | 19                      |
| Ti-6Al-4V       | 0.014 ± 0.008                    | 0.207 ± 0.117                          | 14                      |
| Ti-6Al-7Nb      | −0.008 ± 0.007                   | 0.230 ± 0.090                          | 135                     |
be particularly suitable for the assessment of the observed variation [49] in mechanical behavior of alloys including Ti-6Al-4V produced by additive manufacturing wherein a characteristic microstructure may be identified through use of a parameter as the $c_b$-coefficient.

6. Conclusions

A greater understanding of the microstructure associated with the mechanical behavior of commercial Ti-alloy wires is of interest for a variety of applications such as structural implant biomaterials. Analysis of the mechanical behavior of commercially available Ti, Ti-6Al-4V, and Ti-6Al-7Nb wires is conducted for tensile tests conducted over strain rates of $10^{-5}$–$10^{-1}$ s$^{-1}$ to further exploration of strength behaviors of interest.

1. Yield strengths are measured that are material dependent which vary from 570 to 1070 GPa. The wide range of materials that exhibit less plasticity and a rapid strain hardening parameter as the softening factor $c_b$, that provides insight to the underlying scale of microstructure in these wires as computed using the yield stress and ultimate strength along with the plastic strain during work hardening.

2. Small $c_b$ values are consistent with greater plasticity and work hardening, whereas large values are consistent with materials that exhibit less plasticity and a rapid strain hardening behavior. In this study, a high $c_b$-value of 135 is computed for Ti-6Al-7Nb that rapidly strain hardens with $<$4% plastic strain, whereas a low $c_b$-value of 14 is found for Ti-6Al-4V that strain hardens with a greater ductility of 10% plastic strain.

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References

[1] N. Poondla, T.S. Srivatsan, A. Putnaik, M. Petraroli: J. Alloys Compd. 486 (2009) 162 – 167. DOI:10.1016/j.jallcom.2009.06.172
[2] L. Le Guennec, A. Soueidan, P. Layrolle, Y. Amouriq: Dental Compd. 22 (2001) 1253 – 1262. DOI:10.1016/S0142-9612(99)00275-1
[3] P.C. Collins, B. Welk, T. Searles, J. Tiley, J.C. Russ, H.L. Fraser: J. Biomed. Eng. 30 (1985) 334 – 339. DOI:10.1016/0142-9612(88)90024-5
[4] M. Browne, P.J. Gregson: Biomater. 21 (2000) 385 – 392. DOI:10.1016/S1359-6454(03)00239-8
[5] M. Bambach, I. Sizova, S. Bolz, S. Weiβ: Metals 6 (2016) 204 – 222. DOI:10.3390/met6090204
[6] A.F. Jankowski et al.: The softening factor $c_b$ of commercial titanium alloy wires

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A.F. Jankowski et al.: The softening factor $c_b$ of commercial titanium alloy wires

[42] W.D. Nix, J.C. Gibeling, D.A. Hughes: Metall. Trans. A 16 (1985) 2215–2226. DOI:10.1007/BF02670420
[43] H. Becker, W. Pantleon: Comput. Mater. Sci. 76 (2013) 52–58. DOI:10.1016/j.commatsci.2013.03.028
[44] Y. Zou, L. Zhang, Y. Li, H. Wang, J. Liu, P.K. Liaw, H. Bei, Z. Zhang: J. Alloys Compd. 735 (2018) 2625–2633. DOI:10.1016/j.jallcom.2017.12.025
[45] O. Bouaziz, D. Barbier, J.D. Embury, G. Badinier: Philos. Mag. 93 (2013) 247–255. DOI:10.1080/14786435.202.704419
[46] G.V.S.S. Prasad: An Improved Dislocation Density Based Work Hardening Model for Al-Alloys, Dissertation Master of Science, Georesources and Materials Engineering, RWTH Aachen University, Aachen, Germany (2007) pp. 3–34.
[47] K.F. Karhausen, F. Roters: J. Mater. Process. Technol. 123 (2002) 155–166. DOI:10.1016/S0924-0136(02)0081-X
[48] G.V.S.S. Prasad, M. Goerdeler, G. Gottstein: Mater. Sci. Eng. A 400–401 (2005) 231–233. DOI:10.1016/j.msea.2005.03.061
[49] S. Gorsse, C. Hutchinson, M. Gouné, R. Banerjee: Sci. Technol. Adv. Mater. 18 (2017) 584–610. PMid:28970868; DOI:10.1080/14686996.2017.1361305

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Bibliography
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