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The effect of microstructure and strain rate on the 25°C and 700°C compression deformation behavior of powder metallurgy processed Ti-45Al-2Nb-2Mn (at.%)-0.8 TiB₂ (vol.%) alloy

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Abstract

The effect of microstructure and strain rate on the room-temperature (RT) and 700°C compression deformation behavior of a powder metallurgy processed γ-TiAl intermetallic alloy, Ti-45Al-2Nb-2Mn (at.%)-0.8 (vol.%) TiB₂, was investigated. Samples were heat-treated to obtain a duplex two-phase α₂+γ microstructure and two nearly fully-lamellar α₂+γ microstructures with different lamellar spacings and γ-phase volume fractions. Compression experiments were performed to a minimum deformation of 10% true strain under strain rates of 10⁻², 10⁻³, 10⁻⁴, and 10⁻⁵ s⁻¹. The compression strength, strain rate sensitivity, colony size, interlamellar spacing, and microhardness were dependent on microstructure. The nearly fully-lamellar microstructures exhibited significantly higher compression strengths than the duplex microstructure for all the testing conditions. The strain rate sensitivity index (m), which ranged between 0 and 0.05, increased with increasing temperature, and for the 700°C deformation, m increased with increasing true strain. The apparent activation volumes, which ranged between 0-200b³ (where b is the Burger’s vector), decreased with increasing true strain at 700°C and increased with increasing true strain at RT. Scanning electron microscopy observations showed that cracking preferentially occurred within the γ phase and the extent of cracking increased with increased temperature, strain, and strain rate. Overall, the RT deformation was considered to be controlled by dislocation glide, while at elevated temperature, the likely thermally activated process controlling dislocation glide was associated with the forest junctions acting as pinning points.
Introduction

Two-phase intermetallic TiAl based alloys, consisting of \( \alpha_2 \) (TiAl, hexagonal close packed (hcp), DO19) and \( \gamma \) (TiAl, face centered tetragonal (fct), \( L_{10} \)) intermetallic compounds, have exhibited potential as high-temperature structural materials due to their low density and excellent creep, oxidation, and corrosion resistance [1-4]. The morphology of the microstructures, which is dependent on the processing conditions, has a critical influence on the mechanical behavior [3,4]. In general, microstructures containing only lamellar colonies, termed fully-lamellar (FL), are endowed with high strength as well as excellent creep resistance [3,4]. Duplex microstructures, in which different volume fractions of equiaxed \( \gamma \) grains are embedded in a matrix of lamellar colonies, are softer and usually possess higher RT elongation-to-failure (\( \varepsilon_f \)) [3,4]. For structural components, a reliable material design requires an understanding of the mechanical properties under different loading conditions. In particular, the work hardening behavior is an important factor in plastic deformation. Deformability, \( \varepsilon_f \), and toughness are intimately linked to the work-hardening capacity [5]. In the past several decades, the effect of strain rate on the deformation behavior of TiAl alloys has been investigated [6-9]. For example, the compression strength of a TiAl-Cr-V alloy increased at 1170ºC when the strain rate changed from \( 10^{-3} \) to \( 10^2 \) s\(^{-1} \) [6], and the flow stress of Ti-45Al-7Nb-0.4W(at.%) increased with the increase in strain rate (from \( 10^{-3} \) to \( 10^{-1} \) s\(^{-1} \)) for temperatures ranging between 1000ºC and 1200ºC [7]. \( \gamma \)-TiAl alloys exhibit a superplastic behaviour at temperatures higher than 1000ºC and low deformations [10]. This behavior can be separated into three sections as a function of temperature, as shown by U. Fröbel and Appel [11-12]. While these works were focused on the high-temperature deformation behavior, few studies have systematically investigated the mechanical behavior of TiAl alloys over a wide range of strain rates at room temperature (RT). In particular, Shu et al. [13] found that the addition of 2 (at.%) Mn
(where the Mn was mostly, if not always, in solid solution) increased the RT compression strength (sometimes up to 125MPa) for strain rates between $10^{-2}$ s$^{-1}$ and $10^{-5}$ s$^{-1}$.

Although the majority of deformation studies on TiAl-based intermetallics have involved castings and ingot metallurgy (IM) [3,14,15], powder metallurgy (PM) processing routes have been more recently explored for Ti-based intermetallics [16-20]. Some of the disadvantages associated with IM include casting defects, segregation of alloying elements, and the need for post consolidation heat treatments [21,22]. Techniques, such as counter-gravity and centrifugal casting, have been developed to overcome some of these drawbacks, though they require further heat treatment optimization [23]. PM offers several advantages such as refining the microstructure, improving structural and chemical homogeneity, incorporating heat treatment into the consolidation cycle, and directly obtaining final product shape requiring minimal post processing treatments, thereby reducing manufacturing times. Moreover, Voisin et al. [24] has proven that PM can be successfully used for obtaining high quality TiAl turbine blades.

The aim of this paper was to gain a more detailed insight into the relationship between the microstructure, strain rate, and the work-hardening behavior of PM processed Ti-45Al-2Nb-2Mn(at.%)-0.8TiB$_2$(vol.%) alloy (Ti4522XD) as such work has not been reported in the literature on this alloy. Compression tests were carried out to a minimum of 5% plastic strain using different strain rates ($10^{-2}$, $10^{-3}$, $10^{-4}$ and $10^{-5}$ s$^{-1}$) at RT and 700ºC. Compression testing was chosen over tension because the strain rate sensitivity of the flow stress and the occurrence of individual deformation mechanisms can be studied over a wider range of strain rates than tension, as compression does not restrict the low strain (~2-3%) plastic deformation at high strain rates ($10^{-2}$ to $10^{-3}$ s$^{-1}$), as is the case in tension. Prior to testing, the alloys were subjected to heat treatments to obtain different microstructural morphologies containing different colony sizes (CS), lamellar
spacings, and phase volume fractions. Thus, the effects of microstructural parameters on both the properties and deformation will be highlighted.

**Experimental Procedures**

The TiAl intermetallic alloy studied in this work had a composition of Ti-30.80Al-4.80Nb-2.85Mn-0.25B (wt.%), which is equivalent to Ti-44.70Al-2.03Nb-2.03Mn-0.91B (at.%). The starting material was gas atomized prealloyed powders produced at the Helmholtz-Zentrum für Material und Küstenforschung GmbH (Geesthacht, Germany) by electrode induction gas atomization (EIGA) [25]. The estimated cooling rate reached during the EIGA process was ~10⁵ °C/s, which was lower than that considered for rapid solidification processes, but higher than that typical of conventional gas atomization. The powders were consolidated by hot isostatic pressing (HIP) at 1200°C and 200 MPa during 4 hours at the Forschungszentrum Jülich (Jülich, Germany). Cylindrical specimens, nominally 15 mm in length and 10 mm in diameter, were electrodischarged machined from the as-HIP material. Some of the specimens were solution treated in a vacuum furnace (<10⁻⁵ torr) for 2 hours at 1300°C followed by furnace cooling, hereinafter referred to as HT1. Other specimens were vacuum sealed in quartz tubes and placed in a muffle furnace at 1300°C for 2 hours followed by water quenching, where the quartz tubes were broken upon entering the water. All of the water quenched samples were then heat treated in the same vacuum furnace mentioned above; some at 850°C for 8 hours followed by furnace cooling, hereinafter referred to as HT2, and others at 700°C for 8 hours followed by furnace cooling, hereinafter referred to as HT3. The heating rate for all samples heat-treated in the vacuum furnace was 10°C/min.

Uniaxial compression tests were carried out on the cylindrical specimens using a Gleeble 3800 equipment (Dynamic Systems Inc., Poestenkill, New York, USA) at RT and 700°C at strain...
rates of $10^{-5}$, $10^{-4}$, $10^{-3}$, and $10^{-2}$ s$^{-1}$. The compression axis was normal to a face of the cylindrical specimens. For the elevated-temperature experiments, the specimens were heated from RT to 700°C at a heating rate of 20°C/s and after holding at 700°C for 10 seconds, the samples were compressed to a minimum of 5% plastic deformation and after unloading, the samples were cooled from the deformation temperature with a cooling rate of 5°C/s. A thermocouple was spot welded in the middle of the sample at its periphery, and the temperature was controlled within ±2°C of the targeted 700°C temperature throughout the experiments. For some conditions, the experiments were repeated.

After the deformation, the samples were sectioned using a slow-speed diamond blade along the longitudinal direction and the cross section, and metallographically polished using colloidal silica (60 nm) for the finishing polish. Secondary electron (SE) and backscattered electron (BSE) SEM images, taken using either a Tescan Mira III (Brno, Czech Republic) Field Emission Gun (FEG) SEM, or a Philips XL30 SEM (now Thermo Fisher Scientific Hillsboro, Oregon), were used to examine the phase distributions and their morphologies. Electron backscatter diffraction (EBSD) of the $\gamma$ phase was performed using EDAX-TSL (Mahwah, NJ, USA) EBSD system. Phase volume fractions, which were determined with an accuracy of ±5% from approximately 20 BSE images acquired at different magnifications for each microstructure, were measured using ImageJ software. Grain size was determined using the mean line intercept method [26]. Energy Dispersive Spectroscopy (EDS) analysis within the SEM was performed to analyze the local chemical makeup of the microstructures.

For TEM specimen preparation, the deformed samples were mechanically ground to a thickness of ~100 μm, followed by electropolishing at -30°C, 40 V, for 10 s, using an electrolyte consisting of 60% methanol, 34% butoxyethanol, and 6% perchloric acid (vol.%). TEM imaging
was carried out in bright-field (BF) and high-angle annular dark field (HAADF) modes with a FEG S/TEM (Talos F200X, FEI) operated at an accelerating voltage of 200 kV. The resulting TEM images were used, in conjunction with SEM images, to estimate the lamellar spacings of the microstructures after deformation.

The compression tested samples were polished to a mirror finish before testing according to the ASTM Standard E92-17 for Vickers hardness using a 500 gf and a dwell time of 15 s. At least 10 separate measurements were recorded per alloy/aging combination and the averaged values were reported. The phase constituents of the alloys were examined by X-ray diffraction (XRD) using a Philips X’Pert-MPD System (Almelo, Netherlands) with Cu-Kα (λ=1.542 Å) radiation at a voltage of 40 kV and 40 mA.

Results

Prealloyed TiAl intermetallic alloy powder

XRD confirmed that the primary phase present in the gas atomized powders after cooling to RT was the α-Ti₃Al phase (a=2.8175 Å; c=4.6100 Å) [19]. The lack of the γ phase was consistent with the high cooling rate that the powders experienced. The average size of the powders, which exhibited a typical spherical morphology, was 51 μm, and the powder sizes measured at 10, 50 and 90% of the total powder volume were 17.3, 51.2 and 117.8 μm, respectively [19]. Powders larger than 50 μm typically exhibited an elongated dendritic structure likely due to the longer solidification period and slower cooling rates they experienced compared with the smaller powders. The borides precipitated with lacy, string-like shapes, likely due to lower solidification rates conditions. EDS analysis confirmed the heterogeneous microstructure of the powders with uneven Nb concentration and Mn concentrations [19]. Similar microstructures have been obtained
by Yang et al. [27]. Overall, the microstructural features were suggested to be a function of the individual powder cooling rates.

The gas atomized powders were heated to 1000°C and XRD analysis was performed at different temperatures during the heating in order to understand the change in their crystal structure as a function of temperature. The results showed a decrease in the intensities of the α peaks at 600°C compared to those obtained before heating. A further increase in the temperature to 800°C led to a greater decrease of the α-phase peaks combined with the appearance of both γ-TiAl and α2-Ti3Al peaks, suggesting that the metastable α phase started to transform into the α2+γ microstructure around 800°C [19]. At 1000°C, the transformation was complete and the α2 and γ peaks’ intensities were even higher. Thus, once the metastable powder is heated to temperatures above 800°C, γ forms and increasing the temperatures leads to stabilization of the α2 and γ phases according to the equilibrium phase diagram [28-30]. Therefore, in order to achieve a complete transformation during the consolidation of the previous metastable α powder, the sintering temperature must be equal or greater than 1000°C.

**As-HIP and as-HIP then Heat-Treated Microstructures**

The 1200°C HIP treatment transformed the metastable α phase into the α2 and γ phases as confirmed by XRD [19]. The as-HIP material developed a near γ microstructure in which the former powders were distinguishable, see Figure 1. The grains varied in diameter from 0.6-11.4 μm and the average grain size was 4.4±2.6 μm [19]. XRD of the HT1 and HT2 microstructures revealed that they were composed of only the α2 and γ phases. The HT1 microstructure exhibited a duplex microstructure combining lamellar colonies that were 98±34 μm in diameter and equiaxed γ grains with an average size of 4±1 μm, see Figure 2 (a). The HT1 lamellar CS ranged from 45 to
205 μm. In contrast, the HT2 sample exhibited a refined near fully-lamellar microstructure with an average CS of 33 μm, as observed in Figure 2 (b). The HT3 sample exhibited a refined near fully-lamellar microstructure with an average CS of 23 μm, as observed in Figure 2 (c). The volume fractions of the γ phase in the HT1, HT2 and HT3 microstructures were 0.84, 0.98 and 0.52, respectively. The volume fraction of the boride-rich phase in all the microstructures was less than 0.01. For TiAl alloys containing 45-48Al (at.%), the equilibrium α2 and γ phase volume fractions typically lie between 0.05-0.2 and 0.8-0.9, respectively [31], and this was similar for the HT1 and HT2 microstructures. However, the γ-phase volume fraction for the HT3 microstructure was quite low, 0.52, and this may have been a result of the lower temperature aging. In general, the HT2 microstructure exhibited the greatest volume fraction of the γ phase (0.98) and similar CS and lamellar spacings to that for the HT3 microstructure.

Figure 3 shows representative TEM images in BF and HAADF modes, corresponding to HT2 sample tested at 10⁻² s⁻¹ strain rate at RT, where lamellae are readily apparent. From the TEM images, the average lamellar spacing was ~ 12 ± 2 nm. It is noted that the SEM resolution was not adequate to accurately measure the lamellae thick and therefore TEM was required. Though we have not yet performed TEM on the HT3 microstructure, it is believed that the average lamellae spacings, independent of phase, was similar to that for the HT2 microstructure. Overall, in the colonies of all the microstructures, the α2 lamellar had smaller widths than those for the γ lamellae. In summary, HT2 microstructure exhibited the greatest volume fraction of the γ phase (0.98) and similar CS to that for the HT3 microstructure.

Compression Properties
The true stress versus true strain curves obtained from the compression tests under different strain rates at both temperatures evaluated are presented in Figure 4. It is noted that the absolute values of the true stress and true strain values are plotted rather than the actual values. In general, the strengths decreased with increasing strain rate. However, this was not the case for RT deformed samples which underwent the HT2 heat treatment, where the strength was not dependent on the strain rate and a near 0 value of the strain rate sensitivity index, m, was measured, as will be discussed later. The strengths decreased with increasing temperature. This decrease in strength was more dramatic for the near fully-lamellar microstructures (i.e. HT2 and HT3 samples). The heat-treated HT3 microstructure exhibited a larger drop in true flow stress at 700°C, where the decrease ranged from 78MPa to 340MPa, than the heat-treated HT2 microstructure.

The differences in the compression properties between the differently heat-treated samples were due to the phase volume fractions, morphology, and the lamellar spacings. The strengths of HT2 and HT3 samples with near fully-lamellar microstructures were significantly greater than those for the HT1 samples with duplex microstructures under similar conditions of deformation temperature, strain rate, and plastic deformation. For the 700°C deformation, the true flow stresses of the HT2 microstructures were at least 100MPa higher than those of the heat-treated HT3 microstructures and at least 115MPa higher than those of the heat-treated HT1 duplex microstructures, see Table I.

**Hardness of deformed samples**

There was no clear correlation between the hardness of the samples deformed to nominally 11% true strain and strain rate or temperature as the hardness values for a given set of heat-treated samples were similar for all strain rates and temperatures given temperature. Consistent with the
compression strength results, the HT2 samples exhibited higher hardness values than the HT1 samples, see Table I. They also exhibited higher hardness values than the HT3 samples, for which the hardness values ranged between 396-412 HV. In general, the duplex microstructure exhibited lower hardness values than the near fully-lamellar microstructures.

**Strain hardening exponent**

The strain hardening exponent (n) is an important parameter that reflects the work-hardening effect of the material during plastic deformation. It is readily apparent in Figure 4 (a-f) that the true stress-strain curves of the tested materials exhibited work-hardening, and therefore, the investigation of the n values is relevant in the current work. The n values were determined by two different approaches. First, by following the Hollomon relationship, which is the conventional and most widely used approach to evaluate the flow stress of a material in the plastic deformation region, and is given by:

\[
\sigma = K \varepsilon^n \tag{1}
\]

where K is the strength coefficient, \( \sigma \) is the true stress, \( n \) is the strain hardening exponent, and \( \varepsilon \) is the true strain. Only the plastic regime is used in the curve fitting to determine n. To better quantify n, Afrin et al. [32] proposed the relationship in Equation (2) below, which excludes the elastic portion of the true stress-strain curve:

\[
(\sigma - \sigma_y) = K (\varepsilon - \varepsilon_y)^n \tag{2}
\]

where \( \sigma_y \) is the yield stress and \( \varepsilon_y \) is the strain at yielding. As this was derived from the Ludwik equation [33], it will be referred as the modified Ludwik approach hereafter. The yield stress and the strain at yielding were determined from the conventional 0.2% strain offset of the true stress-
strain curves and used as inputs in the curve fitting of Equation (2). The \( n \) values calculated from the Hollomon and modified Ludwik approaches are given in Table I. The \( n \) values obtained from the Hollomon equation ranged between 0.078 and 0.241, whereas those obtained from the modified Ludwik equation ranged between 0.502 and 0.849. The \( n \) values obtained from the modified Ludwik approach were always larger than those obtained from the Hollomon approach, and they are plotted as a function of the strain rate in Figure 6 (d, e and f). Overall, there was no clear observed dependence of the \( n \) values on the strain rate, for any of the two approaches. However, the \( n \) values obtained from the modified Ludwik approach tended to increase with increasing strain rate for samples tested at 700 °C, and were generally lower than those for samples tested at RT.

**Strain rate sensitivity**

The true stress-strain values obtained from the compression tests were used to calculate the strain rate sensitivity \( (m) \). To determine this parameter, several methods can be used, such as determining the slope of the \( \log(\sigma) - \log(\dot{\varepsilon}) \) from instantaneous values, changing the deformation rate, or by performing stress-relaxation tests [11-13,34-38].

In the current work, the \( m \) values were calculated for a constant strain and test temperature using the following equation: [39,40]

\[
m = \frac{\partial \ln(\sigma)}{\partial \ln(\dot{\varepsilon})}_{\varepsilon,T}
\]  

(3)

where \( \sigma \) is the true flow stress and \( \dot{\varepsilon} \) is the corresponding strain rate. The \( m \) values were determined from the slope of the \( \ln(\sigma) - \ln(\dot{\varepsilon}) \) curve formed by the true flow stress values at the following true strain values: 5%, 6%, 7%, 8%, 9%, 10% and 11%, as observed in Figure 5. In this way, the \( m \) values were obtained as a function of the true strain and test temperature, and they are tabulated in
Table II. The m values for the HT1 samples ranged between 0.008 and 0.021 at RT and between -0.153 and 0.039 at 700°C. The m values for the HT2 samples ranged between -0.035 and -0.012 at RT and between 0.004 and 0.054 at 700°C. The m values for the HT3 samples ranged between -0.126 and 0.003 at RT and between -0.001 and 0.033 at 700°C.

Figure 6 (a, b and c) illustrates the evolution of m values as a function of true strain, for different heat-treated samples and testing temperatures. It was noted that for HT2 and HT3 samples, the m values obtained at 700 °C were always larger than those at RT, which was also true for HT1 samples above 7% true strain. In particular, the m values of HT1 samples tested at 700°C increased gradually with increasing strain, however, they decreased slightly with increasing strain for samples tested at RT. The trend of increasing m values at increasing strain holds true for HT2 and HT3 samples at both testing temperatures.

Apparent activation volume

The apparent activation volume, V*, of each of the microstructures were determined by the isostrain method using the following equation: [39]

\[ V^* = M k T \left( \frac{\partial \ln \dot{\varepsilon}}{\partial \sigma} \right) = \frac{M k T}{m} \sigma \]  

(4)

where m is the Taylor factor (equal to 3.06 for random textures), k is the Boltzmann’s constant and T is the absolute temperature, \( \sigma \) is the true flow stress, \( \dot{\varepsilon} \) is the strain rate, and \( m \) is the strain rate sensitivity. The V* values were determined in the range of 5-11% true strain, which belongs to the plastic regime, for each of the four strain rates tested, and their average values are provided in Table II, assuming a Burger’s vector, b, of 0.283 nm [41]. Note that V* values from HT2 and HT3 samples tested at RT were negative because of the corresponding negative m values, and thus, they lack from physical meaning. Figure 7 shows the evolution of the average V* values, calculated from
700°C tests for all strain rates, with increasing true strain, and the error bars represent their respective standard deviations.

The $V^*$ values, which ranged between 10.6 and 210.4 b$^3$, equivalent to $2.4 \cdot 10^{-28}$ and $4.8 \cdot 10^{-27}$ m$^3$, for samples tested at 700°C, were relatively independent of stress, as similarly reported a previous study [35], and decreased with increasing true strain, as observed in Figure 7. These observations are similar to those from Kashyap et al. [35] who performed tensile tests at temperatures from 100-350°C, which represents 0.4-0.65T$_m$, for strain rates from $10^{-4}$ to $10^{-1}$ s$^{-1}$ for an ultrafine grained Al alloy. The 700°C compression experiments were estimated to be at 0.55T$_m$, which is within the normalized temperature range of those for Kashyap et al. [35]. The $V^*$ values at RT were either constant or slightly increasing with increasing true flow stress. Thus, there was a different dependence at lower temperatures compared with elevated temperatures.

**Compression cracking behavior**

In order to investigate the deformation behavior, BSE SEM photomicrographs of samples tested at RT and 700°C for all strain rates after reaching at least 11% true strain are provided in Figure 8, 9 and 10 for HT1, HT2 and HT3 samples, respectively. From Figure 8 and 9, it is apparent that the HT1 and HT2 samples tested at 700°C resulted in more cracking at a given strain level compared with those tested at RT. In general, the cracking tended to be confined within the dark $\gamma$ phase more than the $\alpha_2$ lighter phase, and this was especially true for the duplex microstructure exhibited after the HT1 heat treatment, as observed in Figure 8. Overall, the extent of cracking increased with increased temperature, strain, and strain rate. It is also noteworthy that in the HT1 samples tested at 700°C, the boride phase exhibited more cracking compared with that at RT, see Figure 8. Similar boride phase cracking has also been observed in convention $\alpha+\beta$ Ti alloys deformed at both RT and 455°C [42-48].
Discussion

Compression strength as function of temperature and strain rate

The results indicate that the near fully-lamellar microstructures were significantly stronger than the duplex microstructure, consistent with the literature [3,4,19]. The deformation temperature and strain rate had a significant effect on the true flow stress under all of the conditions tested. In general, the true flow stress increased with increasing strain rate and decreasing test temperature, as similarly observed for a high strength low alloy steel at strain rates from 0.1 to 10 s\(^{-1}\) between 800 and 1100°C [38]. This is because the dynamic softening dominates work hardening at higher temperatures [49,50]. Subsequently, the flow stress decreases with increased temperature and decreased strain rate until a balance with work hardening is achieved, and this explains the dynamic softening. The reason that the true flow stress values decrease with increasing deformation temperature is because higher temperatures and lower strain rates provide a longer time for energy accumulation and higher dislocation mobilities [51].

Deformation at high strain rates/low temperatures promotes the accumulation of dislocations within ultra-fine grains resulting in increased strain hardening [52] and the enhancement of the strain rate sensitivity to the flow stress [53]. It has been suggested that increased strain rate sensitivity to the flow stress increases uniform elongation by delaying macroscopic localization of plastic deformation [53,54]. Increased strain rate sensitivity has been attributed to the occurrence of grain boundary sliding and grain boundary diffusion in nanostructured metals [55,56], where both mechanisms can be active in ultrafine grained and nanostructured metals during plastic deformation, even at RT [55,56]. The higher \(m\) values observed at 700°C in the current work may be a result of sliding along the fine lamellae where grain boundary diffusion may have played a significant role as will be discussed later.
Strain rate sensitivity and strain hardening exponent

On the one hand, the strain rate sensitivity \( (m) \) is typically used to accurately predict the deformation behavior of the materials during loading [57]. On the other hand, the strain hardening exponent \( (n) \) can be used to rationalize on the amount of uniform plastic strain which the material can withstand before failure [8]. Since the elastic regime of the tested samples extends up to about 5% true strain, the \( m \) and \( n \) values were only considered meaningful above that value. In the current work, a difference in the \( m \) values was observed for each of the microstructures at the two different temperatures investigated, where higher \( m \) values were exhibited at 700ºC as compared to RT. This indicates a higher sensitivity to the deformation speed at elevated temperatures, as has been observed in other structural polycrystalline alloys [35].

The \( n \) values determined from the Hollomon and modified Ludwik relationships described earlier, confirmed the consistently higher values and more meaningful interpretation of the latter, in agreement with a previous work [32]. In general, the \( n \) values retrieved from the modified Ludwik approach decreased with increasing deformation temperature, as observed in Figure 6 (d, e and f). It should be noted that the \( n \) value results from a competition between work hardening and thermal softening, such that a decrease in softening (i.e. increase in work-hardening) leads to the increase in \( n \) value [58]. Thereby, the \( n \) value of materials exhibiting strain softening tends to increase with increasing deformation temperature, as it is the case of Ti-6Al-4V alloy [8], Ti60 alloy [58] and Ti17 alloy [59]. On the contrary, the \( n \) value of materials exhibiting strain hardening tends to decrease with increasing deformation temperature, as it is the case of the present study and several other works [60,61]. It is also evident from Figure 6 (d, e and f) that, for samples tested at 700ºC, the \( n \) values exhibited an increasing trend with increasing strain rate, and this is consistent with the trend observed in Ti60 alloy compression tested at 960ºC from \( 10^{-3} \) to \( 10^{-1} \) \( \text{s}^{-1} \) strain rates.
According to this analysis, it is concluded that the variation of the $n$ values is dependent on the deformation temperature and the strain rate during compression testing of the different heat-treated alloys.

The $m$ values measured at RT in the current work were similar to those measured at RT by others. For example, Shu et al. [13] measured $m$ values ranging between 0.009 to 0.024 at RT (for strains of 0.2%, 1.0%, and 2.0%) for both TiAl and TiAl-2Mn(at.%), where the Mn was mostly, if not fully, in solid solution. This is within the range of $m$ values calculated for the current alloy, and like that observed in Shu et al. [13], the $m$ values of the HT1 sample decreased with increasing plastic strain at RT (see Figure 7). The microstructures in that work were not described well, though they had subgrain structures and were processed by PM techniques. However, the $m$ values of the HT2 and HT3 samples tested at RT increased gradually with increasing plastic strain (see Figure 7), which might be due to a transition from dislocation-dominated plasticity to a combination of grain boundary diffusion and sliding, as proposed elsewhere [62]. Sabirov et al. [37] studied the RT compression behavior of ultrafine grain Al alloy Al6082 and the $m$ values decreased from 0.026 for a strain rate of $10^{-5}$ s$^{-1}$ to 0.013 for a strain rate of $10^{-2}$ s$^{-1}$. It should be noted that the $m$ value obtained for the lowest strain rate was comparable to that reported earlier for the case of tensile tests of the same material at similar strain rate ($m = 0.03$) [63].

Similarly, the $m$ values determined at 700°C in the present work were consistent with those reported in the literature in that the $m$ values increased with increasing deformation temperature and plastic strain (see Figure 7), and this was true for all the heat-treated samples. For example, the $m$ values were $\sim 0.13$ at 100°C, and $\sim 0.23$ at 250°C for ultrafine grained Al-Mg-Si [35]. In another study [58], during isothermal compression of the Ti60 alloy at 980°C, the reported $m$ values ranged between $\sim 0.15$ and 0.5 for strain rates between $10^{-3}$ and 10 s$^{-1}$. Similarly, the $m$
values obtained from isothermal compression of Ti-6Al-4V alloy varied from ~0.10 to 0.40 for testing temperatures from 830 to 1030°C [8], which are comparable to the $m$ values obtained in this work at 700°C.

The factors that have been shown to have the greatest influence on the $m$ values are deviations from stoichiometric composition, grain size, phase distribution, impurity content, supersaturation of vacancies and deformation temperature [12]. The different responses of the same alloy at the two different temperatures examined in the current work are expected to be a result of the microstructural differences. The duplex microstructure exhibited less differences between the $m$ values measured at RT and 700°C. Thus, it is expected that the larger fraction of equiaxed γ grains exhibited by the duplex microstructure played a role in this behavior. Although larger differences in the $m$ values at the different temperatures were exhibited by the near fully-lamellar microstructures, the difference in $m$ values for the HT3 heat-treated microstructure was smaller, and this may have been a result of the lower volume fraction of the γ phase exhibited by this microstructure. Thus, the softer γ phase may be more sensitive to temperature changes than the harder α2 phase.

The HT2 microstructure exhibited the greatest difference in the $m$ values at the two different testing temperatures. Particular noteworthy is that the $m$ values at RT were close to 0 in the HT1 sample, and negative in the HT2 and HT3 samples. Negative $m$ indicates a softening mechanism influenced by dynamic strain aging [64], which has been reported earlier in Ti alloys undergoing stress-induced martensitic transformation during deformation [65]. For two-phase TiAl intermetallics, at RT the dislocation velocity has been found to be controlled by a combined operation of lattice friction, jog dragging, and localized pinning [12]. The glide resistance provided
by these mechanisms gradually decreases with increasing temperature. Deformation at temperatures above 1173°C is dominated by dislocation glide and climb, with the relative contributions depending on temperature and strain rate [66]. Deformation in the 450-477°C range for TiAl intermetallics is characterized by discontinuous yielding, negative strain rate sensitivity and strain aging effects. These phenomena are usually associated with dislocation locking according to the Portevin-LeChatelier effect [67] and this has been assumed to be the case for TiAl [68-72]. Thus, low m values (and even negative m values) are normally associated with the locking of dislocations due to the Portevin-LeChatelier effect and discontinuous yielding [12].

Although this has been observed in TiAl intermetallics between 723-1023°C, the mechanisms are not completely understood due to the microstructural complexity in such alloys. m values near 0 have also been observed in other alloys. For example, Dudamell et. al [73] found low m values (and even negative m values) at 0.1 strain for Mg-1Mn-1Nd(wt.%) in 200°C compression tests for strain rates between 5·10⁻⁴ s⁻¹ and 5·10⁻² s⁻¹, and this was related to dynamic strain aging and deformation-induced twinning. When dislocations move with a certain velocity associated with a certain strain rate, if the solute diffuses at a similar velocity, then the solutes can attach to dislocations and align along them causing dislocation drag on a gliding dislocation. This will cause hardening. However, when the strain rate is fast enough to allow a gliding dislocation to jump into another dislocation, then this will result in less hardening because there will be less dislocation drag as the dislocation that jumped will jump out of the solutes that are dragging it. Thus, with increased strain rates, softening and serrations may occur, which could lead to low or even negative m values. Therefore, this may explain the negative m values obtained in the HT2 and HT3 samples deformed at RT.

**Apparent activation volume**

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The activation volume ($V^*$) is widely used to determine the possible plastic deformation mechanisms [74-76]. Like the $m$ values, the $V^*$ values were only considered meaningful within the regime of plastic deformation, which begins at approximately 5% true strain for all samples tested in the current work. In Shu et al. [13], the average $V^*$ values of the TiAl and TiAl-2Mn(at.%) microstructures were 22.5 $b^3$ and 24.6 $b^3$, respectively. The $V^*$ calculations they used were based on a constant that was approximately half that used in the current work. Incorporating their data into equation (3), their revised $V^*$ values (~ 45-50 $b^3$) fall within the range of $V^*$ values reported here for samples tested at 700°C for ~ 0.07-0.08 true strains, as observed in Table II and Figure 7. According to the literature [77,78], such low $V^*$ values are often associated with dislocation glide. It should be recalled that the $V^*$ values obtained for HT2 and HT3 samples tested at RT were negative, as were their corresponding $m$ values.

An interesting feature of plastic deformation of the material is the decrease of the activation volume with increasing true stress which occurred for the 700°C deformed samples. This behavior, particularly the inverse proportionality of the activation volume to stress, is consistent with the assumption that the thermally activated process controlling dislocation glide is associated with the forest junctions acting as pinning points [39]. The magnitude of the activation volume (i.e. between 0 to 200 $b^3$, see Table II) is semi-quantitatively consistent with the forest junction mechanism, as well. Assuming a $V^*$ value of 50 $b^3$, the spacing, $L$, between the pinning points estimated on the basis of the formula [79].

$$V^* = \frac{2}{3} b^3 \left( \frac{d}{b} \right) \left( \frac{d}{b} \right)$$

(5)

is 400$b$, if the stress-dependent width of the junction, $d$, is estimated to be 0.1875$b$. The typical spacing between the forest junctions, $\rho^{1/2} \approx 10^{-7} m \approx 400b$; is quantitatively similar. Thus, the overcoming of the forest junctions by gliding dislocations can be considered as the most plausible
thermally activated glide mechanism. It is noted that full thermal activation analysis, namely, evaluation of the activation energy for the mechanism governing dislocation glide as well as the dynamic recovery mechanism, which controls steady-state deformation, generally is a useful tool to gain further insight in the possible deformation mechanisms [80].

**Activation energy**

To calculate the activation energy, the relationship between the flow stress ($\sigma$), $\dot{\varepsilon}$, and $T$ during hot deformation can be expressed using the following relationship:

$$\dot{\varepsilon} = A \sigma^{(1/m)} \exp \left(- \frac{Q}{RT} \right)$$  \hspace{1cm} (6)

where $\dot{\varepsilon}$ is the strain rate, $\sigma$ is the true flow stress, $m$ is the strain rate sensitivity, $A$ is a material constant, $R$ is the universal gas constant, $T$ is the absolute temperature and $Q$ is the activation energy. Taking natural logarithm on both sides of equation (6), it can be expressed as:

$$ln = ln(A) + \frac{1}{m} ln(\sigma) - \frac{Q}{RT}$$  \hspace{1cm} (7)

In the current work, $Q$ values were calculated for a constant strain rate, by applying partial derivative of equation (7) as follows:

$$Q = \frac{R}{m} \left[ \frac{\partial ln(\sigma)}{\sigma(\dot{\varepsilon})} \right]_T = \frac{R}{m} \left[ \frac{ln(\sigma_2/\sigma_1)}{(T_2^{-1} - T_1^{-1})} \right]_T$$  \hspace{1cm} (8)

where $\sigma_2$ and $\sigma_1$ are the values of the true flow stress corresponding to the testing temperatures $T_2$ and $T_1$, respectively. The $Q$ values were determined using: $T_1=700^\circ$C, $T_2=RT=25^\circ$C, their respective true flow stress values at 10% true strain, and the calculated $m$ values. The computed $Q$ values are given in Table I, and range between 19-38 kJ/mol, 6-36 kJ/mol and 16-47 kJ/mol for HT1, HT2 and HT3, respectively. For the three samples, there is consistent trend of increasing $Q$ with decreasing $\dot{\varepsilon}$, which agrees with that obtained from compression tests performed at 850-
1150°C in Ti-15-3 alloy at $10^{3}$–$10^{4}$ s$^{-1}$ strain rates [81]. No significant differences were observed in the $Q$ values obtained at different flow stress values within the range of 5-10% true strain.

For TiA15 (Ti-6Al-2Zr-1Mo-1V (wt.%)), Yasmeen et al. [82] measured the $Q$ values for tensile deformation between 880-920°C and strain rates of $10^{-2}$ to $5 \cdot 10^{-4}$ s$^{-1}$. In the early stages of deformation, they measured $Q=256$ (at a true strain of 0.25) and $Q=276$ kJ/mol (at true strain of 1). In the current work, the $Q$ values ranged between 0-47kJ/mol, and there was no clear relationship between deformation or microstructure and the $Q$ values. These values are roughly half of the self-diffusion activation energy values of both $\alpha$-Ti (204 kJ/mol) and $\beta$-Ti (161 kJ/mol) [83]. These values do not lie in the range of activation energy values for dislocation creep of $\alpha$-Ti (200- 360 kJ/mol) [84]. Thus, the activation energy values more represented grain boundary diffusion processes suggesting that the lamellar boundaries may be playing an important role in the deformation behavior. It has been shown that the dislocations can move by glide or climb, depending on the temperature range, and that some dynamic strain aging occurs at intermediates temperatures [85]. In lamellar alloys, the interaction at interfaces is found to play a determining role [85]. As mentioned in the first part of the discussion, the higher $m$ values measured during the 700°C deformation may be a result of sliding along the fine lamellae, where grain boundary diffusion may have played a significant role, and this could be supported by the low $Q$ values, which are more representative of grain boundary diffusion.

**Deformation observations**

The cracking, which preferentially occurred within the equiaxed $\gamma$ phase, could be due to the buildup of stress concentrations caused by dislocation pileup, both within the grains and at the grain boundaries and lamellar phase interfaces. The balance between work hardening and dynamic softening led to lower stresses achieved at elevated temperatures, as explained earlier, and the
buildup of the stress concentrations could lead to the cracking, which occurred to a lesser extent at RT. The γ phase in particular exhibited a greater extent of cracking than the lamellar colonies. This is not unexpected as the γ phase is softer than the α₂ phase and therefore would be expected to crack prior to the α₂ phase. In addition, the limited deformation exhibited by α₂-Ti₃Al-based alloys, which can be attributed to the lack of independent slip systems, has been well-documented in the literature [86]. Hence, the von Mises criterion is not satisfied. Thus, the deformation of α₂+γ TiAl alloys is considered to mainly be carried by the γ phase, and this could explain the preferential cracking exhibited within this phase. In addition, because most of the deformation is considered to be carried by the γ phase, the activation volume analysis used is this work, with only the γ phase being considered, is justified.

Summary and Conclusions

Duplex and near fully-lamellar Ti-45Al-2Nb-2Mn(at.%)-0.8TiB₂(vol.%) microstructures, obtained through different heat treatments, were subjected to compression tests at strain rates ranging between 10⁻² s⁻¹ to 10⁻⁵ s⁻¹ at RT and 700°C. The strain rate sensitivity of the flow of stress was estimated and the associated deformation mechanisms were discussed. With increased strain rate, the flow stress increased for each of the microstructures evaluated. For all the strain rates and temperatures examined, the near fully-lamellar microstructures exhibited higher strengths than the duplex microstructure.

The strain rate sensitivity exponent, which ranged between 0 to 0.05, was not strongly dependent on true stress and increased with increasing temperature. For the 700°C deformation, m increased with increasing true strain. Scanning electron microscopy observations showed that cracking preferentially occurred within the γ phase and the extent of cracking increased with increasing temperature, strain, and strain rate. The activation volume, which ranged between 0-
200 h\(^1\), was found to be inversely proportional to the flow stress at 700\(^\circ\)C. This dependence was shown to be qualitatively consistent with the assumption that dislocation glide is controlled by the thermally activated overcoming of forest junctions. Overall, the RT deformation was considered to be controlled by dislocation glide, while at elevated temperature, the likely thermally activated process controlling dislocation glide was associated with the forest junctions acting as pinning points.

**Data availability statement**

The raw data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

**Acknowledgments**

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Figure 5: Evolution of the activation volume (V*) in the range of 5-11% true strain for samples HT1, HT2 and HT3 during compression testing at 700°C. Data points and error bars represent, respectively, the average V* values and their corresponding standard deviations for tests performed at 10^{2} s^{-1}, 10^{3} s^{-1}, 10^{3} s^{-1} and 10^{2} s^{-1} strain rates. Note that V* is expressed in terms of the Burger’s vector (b), which is assumed to be 0.283nm [41].

Figure 6: Evolution of the strain rate sensitivity values, m, as a function of the true strain for samples (a) HT1, (b) HT2 and (c) HT3, and evolution of the strain hardening exponents, n, determined from the modified Ludwik relationship, as a function of the strain rate for samples (d) HT1, (e) HT2 and (f) HT3 during compression testing at RT and 700 ºC.

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Figure 8: BSE SEM photomicrographs of the cross-section of samples HT1 after deformation to ~12% true strain at (a and b) 10^{-2} s^{-1}, (c and d) 10^{-4} s^{-1}, (e and f) 10^{-4} s^{-1} and (g and h) 10^{-2} s^{-1} strain rates during compression testing at RT and 700°C, respectively. Note the white boride-rich needles dispersed within the α2 (bright) and γ (dark) phases. Cracking preferentially occurred in the γ-phase in (a and b). Note the cracking of the boride-rich needles in (d). Cracking preferentially occurred in the γ-phase in (e, f, g and h), and more cracking was exhibited after the elevated-temperature deformation (f and h).

Figure 9: BSE SEM photomicrographs of the cross-section of samples HT2 after deformation to ~13-15% true strain at (a and b) 10^{-5} s^{-1}, (c and d) 10^{-4} s^{-1}, (e and f) 10^{-4} s^{-1} and (g and h) 10^{-2} s^{-1} strain rates during compression testing at RT and 700°C, respectively. Note the white boride-
rich needles dispersed within the α2 (bright) and γ (dark) phases. More cracking was exhibited after the elevated-temperature deformation.

Figure 10: BSE SEM photomicrographs of the cross-section of samples HT3 after deformation to ~15-18% true strain at (a and b) 10-5 s⁻¹, (c and d) 10-4 s⁻¹, (e and f) 10-3 s⁻¹ and (g and h) 10-2 s⁻¹ strain rates during compression testing at RT and 700°C, respectively. Note the white boride-rich needles dispersed within the α2 (bright) and γ (dark) phases.
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<table>
<thead>
<tr>
<th>Heat treatment</th>
<th>Test temperature</th>
<th>Strain rate (s⁻¹)</th>
<th>True Yield stress (MPa)</th>
<th>True Yield strain</th>
<th>n</th>
<th>HV (kJ/mol)</th>
<th>Q (kJ/mol)</th>
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<th>m</th>
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<td>11</td>
<td>0.00821</td>
<td>0.03940</td>
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