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Effect of V-Induced Lattice Distortion on Tensile Creep Properties of Nb-Ta-Ti-V Refractory
Complex Concentrated Alloys

By

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THESIS

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Table of Contents

Acknowledgements.....	iv
Abstract	v
Chapter 1. Introduction	1
1.1 Refractory complex concentrated alloys	1
1.2 Deformation behavior of RCCAs.....	1
1.3 Goals of this thesis	2
1.4 References	3
Chapter 2. Tensile Creep Properties of NbTaTi and NbTaTiV Refractory Complex Concentrated Alloys.....	5
2.1 Introduction	5
2.1.1 Refractory complex concentrated alloys	5
2.1.2 Creep properties of RCCAs.....	7
2.1.3 Creep Mechanisms of RCCAs.....	8
2.1.4 Scope of this work	9
2.2 Experimental	10
2.3 Results and Discussion.....	12
2.3.1 Initial microstructure and phase stability.....	12
2.3.2 Creep results and microstructure evolution	17
2.3.3 Creep mechanisms	26

2.4 Conclusions	30
2.5 Acknowledgements	31
2.6 References	31
Chapter 3. The Effect of Lattice Distortion on Tensile and Creep Properties of the NbV Alloy.	37
3.1 Introduction	37
3.1.1 Complex concentrated alloys.....	37
3.1.2 Strengthening in CCAs	37
3.1.3 Scope of this work	38
3.2 Experimental	39
3.3 Results and Discussion.....	40
3.3.1 Initial microstructure	40
3.3.2 Mechanical properties.....	42
3.3.3 Lattice distortion and solid solution strengthening.....	45
3.3.4 Lattice distortion and creep properties	54
3.4 Conclusions	57
3.5 References	57
Chapter 4. Summary and Future Work	61

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Abstract

Refractory complex concentrated alloys (RCCAs) are a relatively novel field of alloys that retain high strength at elevated temperatures, but their creep properties have been scarcely reported. This work investigates the tensile creep behavior of NbTaTi, NbTaTiV, and NbV alloys. In these alloys, lattice distortion increases along these compositions' gradient of V concentration because all of the component elements have similar atomic sizes except for V, which is smaller in these BCC alloys. Higher concentration of V in the composition is correlated with an improvement in the creep resistance of the alloy. The increase in V is also associated with a transition to edge dislocation-controlled deformation behavior of the alloys under creep. Further work was done with NbV to study the effects of isolated, maximized lattice distortion on overall strength. Extreme lattice distortion in NbV further increased the tensile yield strength compared to other Nb-Ta-Ti-V system alloys, showing that this effect matters beyond creep properties. The long duration of creep testing also uncovered phase instability with Ti containing compositions, as Ti-rich secondary phases were found in specimens after testing. However, these secondary phases improved the creep resistance of the material, revealing the importance of multi-phase design. This work demonstrates how both lattice distortion and multi-phase design are critical considerations for engineering high strength, creep resistant RCCAs.

Chapter 1. Introduction

1.1 Refractory complex concentrated alloys

The ever increasing global energy demand drives the need for materials that retain extensive strength at increasingly high temperatures. Current materials in high temperature structural applications such as the blades in gas turbines are currently reaching their limit. Ni based superalloys are the modern solution for this application [1], but in order to improve the efficiency of the turbine, the operating temperature must be increased [2]. This means that Ni based superalloys are limited by their melting point of about 1500 K [1]. An emerging field of materials that have melting temperatures exceeding that of Ni based superalloys are refractory complex concentrated alloys (RCCAs). These materials are typically made up of transition metal elements from groups IV to VI on the periodic table in comparable concentrations, rather than having a single element that takes up the majority of the composition [3], [4]. Importantly, the melting temperatures of these materials can exceed that of Ni based superalloys, thus being prime candidates for being replacements. However, the fundamental mechanisms behind the properties of RCCAs are not fully understood, so informed alloy design cannot be fully optimized. Many works are ongoing to better understand the mechanical behavior of RCCAs at elevated temperatures.

1.2 Deformation behavior of RCCAs

RCCAs have been demonstrated to have high strength at elevated temperatures. RCCAs typically exhibit the body-centered cubic (BCC) crystal structure [4], [5], [6]. BCC metals generally have their mechanical properties governed by screw dislocation motion [7]. Thus, limiting the rate at which these dislocations can move greatly increases the strength of these materials. The addition of solute atoms at these dislocations changes the local energy landscape, thus leaving some

sections of a dislocation pinned, requiring more energy to overcome a barrier. This is one of the fundamental strengthening effects present in RCCAs, and these are more prominent due to the high concentration of all elements in their compositions [7], [8], [9]. However, it has been revealed that edge dislocation motion should not be omitted from consideration in strengthening. At high temperatures, the contribution of edge dislocation motion to strength has been postulated [10], [11]. Thus, it is reasonable to hypothesize that intentionally inducing edge dislocation control in RCCAs may be a viable way to improve high temperature strength. This could be possible due to interactions of certain solute atoms with the edge dislocation cores. One effect of specific solute atoms in RCCAs is lattice distortion, where atomic size misfit causes the regular BCC lattice to slightly distort from its ideal geometric parameters [12]. Since edge dislocation stress fields have a hydrostatic component whereas screw dislocations do not [13], lattice distortions caused by excessively larger or excessively smaller atoms may cause more interactions with edge dislocations, thus limiting their movement. This connection has been postulated in Refs. [12], [14] with the equiatomic alloy Nb-Ta-Ti-V system. In this composition, the elements Nb, Ta, and Ti all have similar effective lattice parameters ($d \approx 2.86 \text{ \AA}$), whereas V is noticeably smaller ($d \approx 2.62 \text{ \AA}$). Thus, the lattice distortion is essentially controlled by the content of V. In Ref. [14], increasing the content of V in this system correlates with increasing the tensile yield strength of the alloy across the temperature range 298 K to 1573 K. Whether this connection between lattice distortion and edge dislocation motion also applies to low strain rate, low stress situations such as creep, is a key investigation in this work.

1.3 Goals of this thesis

The overall aim of these works is to provide an understanding of the deformation properties of RCCAs under tensile creep and to detail how alloy design choices such as elemental composition

fundamentally have an effect on the mechanical response of these solid solutions. The first research chapter discusses the effect of lattice distortion induced by V in the Nb-Ta-Ti-V system to tensile creep properties, including creep rates, exponents, activation energies, and dislocation behaviors. This study also investigates the effects of phase stability on the creep properties of RCCAs. The second research chapter takes this one step further, aiming to maximize the lattice distortion of the system by simplifying the system to a NbV binary alloy to determine whether lattice distortion is the main governing factor in the creep resistance and strength of single phase RCCAs.

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Chapter 2. Tensile Creep Properties of NbTaTi and NbTaTiV

Refractory Complex Concentrated Alloys

M. Lau, T. Zakia, A. Stratton, A. Ni, and M. Zhang, “Tensile creep properties of NbTaTi and NbTaTiV refractory complex concentrated alloys,” *Acta Mater.*, vol. 308, p. 122021, 2026, doi: <https://doi.org/10.1016/j.actamat.2026.122021>.

2.1 Introduction

2.1.1 Refractory complex concentrated alloys

The pursuit of alloys capable of operating at temperatures exceeding those of Ni-based superalloys, aimed at improving turbine engine efficiency, has recently directed research attention toward refractory systems with inherently higher melting points [1]. A newly developed and important class of refractory alloys designed for this purpose is known as refractory complex concentrated alloys (RCCAs), also referred to as refractory multiple principal element alloys (RMPEAs) or refractory high-entropy alloys (RHEAs) [2]. These alloys are characterized by containing substantial concentrations (> 10 at. %) of at least three refractory elements, typically drawn from Groups IV to VI of the periodic table (e.g., Ti, V, Cr, Zr, Nb, Mo, Hf, Ta, and W). Initial studies on RCCAs have identified compositions with promising compressive strength across 1073–1873 K. For example, the $\text{AlMo}_{0.5}\text{NbTa}_{0.5}\text{TiZr}$ alloy, which exhibits a BCC + B2 dual-phase structure (hence termed a refractory high-entropy superalloy), shows a yield strength exceeding 1.5 GPa at 1073 K [3], while the single-phase BCC MoNbTaWV alloy retains a yield strength above 400 MPa even at 1873 K [4]. Despite their high compressive strengths at elevated temperatures, these alloys are constrained by low tensile ductility and fracture toughness even extended to high temperatures, stemming from intrinsic brittleness and grain boundary embrittlement [5-7]. This

limitation substantially restricts the direct use of these alloys, as it complicates metal forming, machining, and compromises damage tolerance.

In contrast, the HfNbTaTiZr and NbTaTiV systems are currently the only RCCAs demonstrated to exhibit extensive tensile ductility [8], [9], including at room temperature. HfNbTaTiZr can achieve tensile strains exceeding 15% [10]. More recently, a derivative alloy, Nb₄₅Ta₂₅Ti₁₅Hf₁₅, has demonstrated a high fracture toughness above 200 MPa·m^{1/2} at room temperature, accompanied by a tensile ductility of more than 35% [11]. Furthermore, tensile tests on as-cast NbTaTiV have reported a tensile ductility surpassing 40% [8]. In addition to their high ductility, coarse-grained, recrystallized HfNbTaTiZr exhibits a yield strength above 1 GPa at room temperature [10], whereas Nb₄₅Ta₂₅Ti₁₅Hf₁₅ with a similar microstructure has a lower yield strength of 600 MPa [11]. However, elevated-temperature strengths of these alloys decrease to ~400 MPa at 1073 K and below ~100 MPa at 1473 K [11], [12], remaining lower compared to Ni-based superalloys [13]. On the other hand, NbTaTiV exhibits a room-temperature strength of ~1.3 GPa and retains a strength above 600 MPa at 1173 K [12], making it substantially stronger than the HfNbTaTiZr family and comparable to many Ni-based superalloys. Specifically, Yin et al. [14] concluded that vanadium (V) is an optimal strengthening element for RCCAs, as its significantly smaller atomic size compared to most other refractory elements introduces substantial solute misfit volumes into the system, elevating solid solution strengthening. In addition, this misfit volume can alter the deformation mechanism of NbTaTiV. As Lee et al. [12] demonstrated, its yield strength is governed by the glide of edge dislocations, in contrast to most BCC RCCAs and conventional refractory alloys, where strength is typically controlled by screw dislocation motion. To date, extensive experimental evidence has shown that the strength of the HfNbTaTiZr system is controlled by screw dislocation glide from room temperature to elevated temperatures

[15-17]. Consequently, the role of V alloying, and its influence on shifting the deformation mechanism from screw to edge dislocation-controlled strength, including creep strength, must be thoroughly understood to enable enhanced high-temperature performance.

2.1.2 Creep properties of RCCAs

Resistance to creep deformation is a critical criterion for the selection of structural materials intended for high-temperature applications. Although extensive literature exists on the strain rate–controlled compressive and tensile behaviors of RCCAs as reviewed and compiled by Miracle et al. [18], experimental investigations of their tensile creep properties remain scarce [19-21]. Specifically, the HfNbTaTiZr system is the only RCCA that has been systematically studied in tensile creep. It was initially selected as a model alloy because of its nominally single-phase BCC structure stable up to 1523 K and its demonstrated tensile ductility, which enables reliable tensile creep testing. Stresses were varied from 5 MPa to 100 MPa from temperatures between 1253 K and 1523 K, and this revealed a stress exponent ranging from 2.0 to 2.8 and an activation energy of 273 ± 15 kJ/mol [19], [20]. However, HfNbTaTiZr was found to be 25 times weaker than the Ni-based superalloy CMSX-4 at 1253 K and 70 times weaker at 1373 K in terms of the stress required to achieve a secondary creep rate of 10^{-7} s^{-1} due to the lack of secondary phase strengthening and fast diffusion [19]. That is, HfNbTaTiZr did not display any additional, secondary phase differing from the BCC solid solution. In addition, HfNbTaTiZr is weaker than the single-phase FCC matrix of CMSX-4, indicating an inferior solid-solution strengthening effect in this BCC RCCA compared to its FCC counterpart [19]. In a separate study, Sahragard-Monfared et al. studied tensile creep properties of $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$ [21], another nominally single phase BCC RCCA, which displays a stress exponent of 4.1 and activation energy of 253 kJ/mol when crept under 50 MPa to 300 MPa at temperatures from 1123 K to 1223 K. When extrapolated to

1253 K, the secondary creep rate of $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$ is approximately one order of magnitude lower than that of HfNbTaTiZr , primarily due to its higher melting point. Despite a slightly better creep resistance, the secondary creep rate of $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$ extrapolated to 1253 K is still higher than that of CMSX-4 for at least two orders of magnitude, and the creep strength to achieve a secondary creep rate of 10^{-7} s^{-1} is 5 times lower. This shortfall unfortunately will position HfNbTaTiZr and its subsystems uncompetitive compared to Ni-based superalloys.

2.1.3 Creep Mechanisms of RCCAs

Currently, it has been observed that tensile creep deformation in the above RCCAs is controlled by screw dislocations. In the HfNbTaTiZr system, screw dislocations were identified via g-dot-b analysis [20]. In the $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$ system, post-creep microstructures displayed long, straight dislocations that showed signs of bowing, pinning, and debris, features characteristic of screw-dislocation-controlled deformation involving cross-kink unpinning and jog-dragging mechanisms [21-24]. Recently, Rao et al. [25] employed a modified Suzuki model [23] to rationalize the measured secondary creep rates based on the aforementioned screw dislocation glide mechanisms. Screw-controlled deformation leads to high strength in RCCAs at lower temperatures [26], but these improvements to creep properties are largely eliminated at high temperatures when diffusion becomes active [23]. In order to design RCCAs with better performance at increasingly elevated temperatures, different deformation mechanisms must be considered. One such mechanism may be present in the composition of interest in this work, NbTaTiV . As mentioned above, it is observed that edge dislocations control elevated-temperature deformation due to the presence of V [14]. The large lattice distortion caused by V forces dislocations to move across high energy barriers, which requires high thermal or stress activation, especially for edge dislocations that are less temperature-sensitive [12], [27]. It has been established that the edge-strengthening effect of

V contributes to high yield strength up to 1173 K. However, whether this mechanism also enhances creep resistance remains to be investigated in detail.

2.1.4 Scope of this work

The present work aims to establish a mechanistic understanding of tensile creep deformation in NbTaTi-based ductile RCCAs by systematically comparing the high-temperature creep behavior of NbTaTi, Nb₄₅Ta₂₅Ti₁₅Hf₁₅, and NbTaTiV alloys. These three compositions were strategically selected to isolate the effects of alloying additions: Hf, which tends to increase atomic size mismatch and promote screw-dislocation-dominated deformation, and V, which introduces strong lattice distortion and is known to favor edge-dislocation activity. All three compositions are previously shown to possess stable or metastable BCC single-phase structures and can be processed and recrystallized to coarse-grained structures [11], [12], [28]. Tensile creep data and microstructure of Nb₄₅Ta₂₅Ti₁₅Hf₁₅ are available through a previous study [21].

High-temperature tensile creep experiments were performed across several stresses and temperatures to quantify the stress exponents, activation energies, and steady-state creep rates of these alloys. Complementary scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analyses of post-creep microstructures were performed to characterize the long-term phase stability of the alloys under stress and directly identify the dominant dislocation characters and deformation mechanisms operative under each condition. By correlating the creep response with the underlying dislocation mechanisms, this study seeks to determine whether edge dislocation-controlled deformation, as induced by V alloying, can provide improved creep resistance compared to conventional screw dislocation-controlled mechanisms prevalent in most RCCAs. The insights gained from this comparison will provide design principles for developing next-generation refractory alloys with enhanced high-temperature strength and creep resistance.

Other critical factors for structural applications at elevated temperatures, such as oxidation resistance, are beyond the scope of the current paper.

2.2 Experimental

60 g buttons of equiatomic NbTaTi and NbTaTiV RCCAs were produced via vacuum arc melting of high purity elemental metals. The arc melter was evacuated to a vacuum better than 3×10^{-3} Pa before Ar gas back filling. The buttons were flipped and remelted at least five times to ensure homogeneity of the alloy. These circular buttons were approximately 35 mm in diameter and 8 mm thick. The arc melted material was cold rolled to 90% thickness reduction with a final thickness of approximately 0.8 mm. Tensile creep specimens were then cut from the rolled sheets via electrical discharge machining, with geometry matching that reported in Ref. [29]. The creep specimens were vacuum annealed (7×10^{-3} Pa) in a sliding tube furnace under different conditions to yield comparable grain sizes: NbTaTi specimens were annealed at 1473 K for 5 hours followed by air cooling (the tube was slid away from the heating elements while maintaining vacuum, yielding a cooling rate of ~ 100 K/min), whereas NbTaTiV specimens were annealed at 1473 K for 2 hours followed by furnace cooling (~ 10 K/min). The difference in the cooling scheme has significant implications for the phase stability of the RCCAs, which is elaborated in the next section. Complementary calculation of phase diagram (CALPHAD) was performed using the CompuTherm PanRHEA database from room temperature to 3000 K. Two calculations were conducted for each composition: one with only the refractory elements, and one with the addition of 5000 molar ppm of oxygen and 1000 molar ppm of nitrogen to understand the effect of interstitial impurities on phase stability. The amount of oxygen and nitrogen was selected based on inert gas fusion results from our recent study that uses the same materials [30], and the values

are consistent with the sum of the oxygen and nitrogen levels in the stock material provided by the manufacturer. Due to the high vacuum used for all of the processing steps at elevated temperatures, the oxygen and nitrogen uptake during arc melting and annealing is considered to be small.

Constant stress tensile creep tests were performed at 1173 K at stresses of 50, 80, 100, and 130 MPa for NbTaTi, and 50, 100, 250, and 300 MPa for NbTaTiV under a high vacuum better than 7×10^{-4} Pa. All creep specimen surfaces were ground to 600 grit using SiC grinding paper prior to testing in order to mitigate surface effects [29], [31]. One sample was used for one applied stress, and the creep tests were either performed to failure or interrupted at steady-state creep for detailed microstructure characterization. For interrupted tests, the samples were cooled under load to preserve the deformation microstructure against annealing. The creep machine was equipped with an Andrade-Chalmers cam lever to maintain a constant true stress during the test [32]. Additional creep tests were performed at 1123 K and 1223 K at 100 MPa for NbTaTi and 250 MPa for NbTaTiV to yield the activation energy of creep for each material. Temperatures around 1173 K were of interest because these temperatures sit near the end of the yield strength plateaus of NbTaTi and NbTaTiV, at temperatures above which their yield stresses begin to decrease [12], [26]. Understanding the fundamental deformation mechanism near this temperature is critical to design new RCCAs that can retain strength to still higher temperatures.

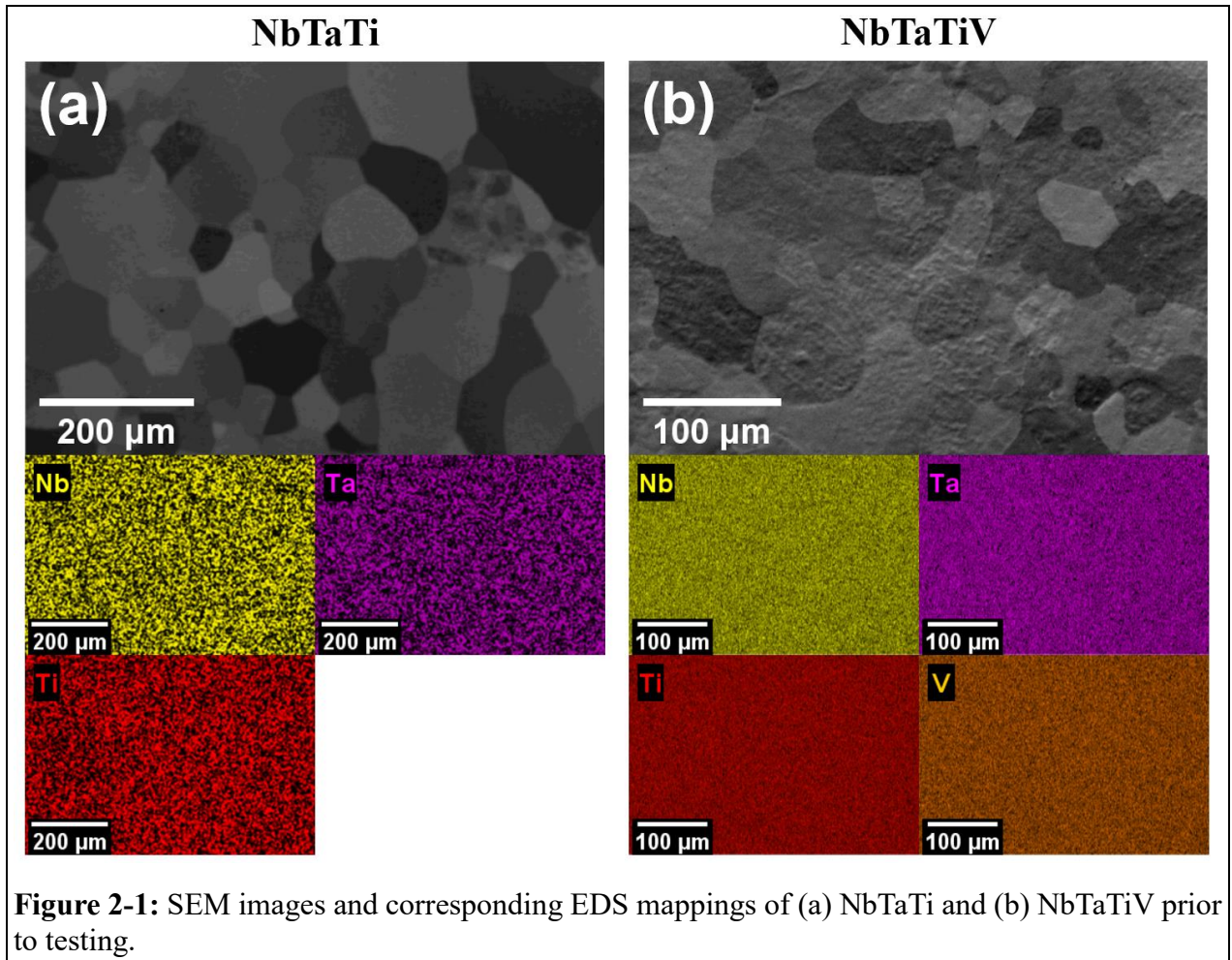
Analyses of microstructures prior to and after creep testing were performed via scanning electron microscopy (SEM) with secondary electron (SE), backscattered electron (BSE), and energy dispersive x-ray spectroscopy (EDS) techniques on a ThermoFisher Quattro S Environmental SEM and a ThermoFisher Scios DualBeam FIBSEM. Transmission electron microscopy (TEM) was performed on a ThermoFisher F20 UT Tecnai at the National Center for Electron Microscopy (NCEM), Lawrence Berkeley National Laboratory to further image dislocation microstructures

after creep testing. Samples for SEM characterization were prepared via mechanical polishing down to 1200 grit sandpaper followed by electropolishing using an electrolyte of 10 vol% H₂SO₄ in methanol under a current of 80 mA and a temperature of -35 °C. TEM specimens were prepared by mechanical thinning to approximately 80 µm followed by twin-jet electropolishing using the same parameters.

2.3 Results and Discussion

2.3.1 Initial microstructure and phase stability

Fig. 2-1 displays the microstructures of NbTaTi and NbTaTiV prior to testing. Both appear to be homogeneous single-phase materials, as the EDS scans in **Fig. 2-1** show no obvious elemental segregation for either composition. The different annealing parameters for the two compositions were important to avoid the formation of secondary phases. **Fig. 2-2** displays an early annealing attempt for NbTaTi using slow furnace cooling. These annealing conditions yielded small Ti-rich secondary phases also enriched with nitrogen at grain boundaries, which is consistent with the (Ti, N)-rich phase in NbTaTi previously reported by Senkov et al. [33].



NbTaTi annealed at 1473 K for 3 hours, furnace cooled

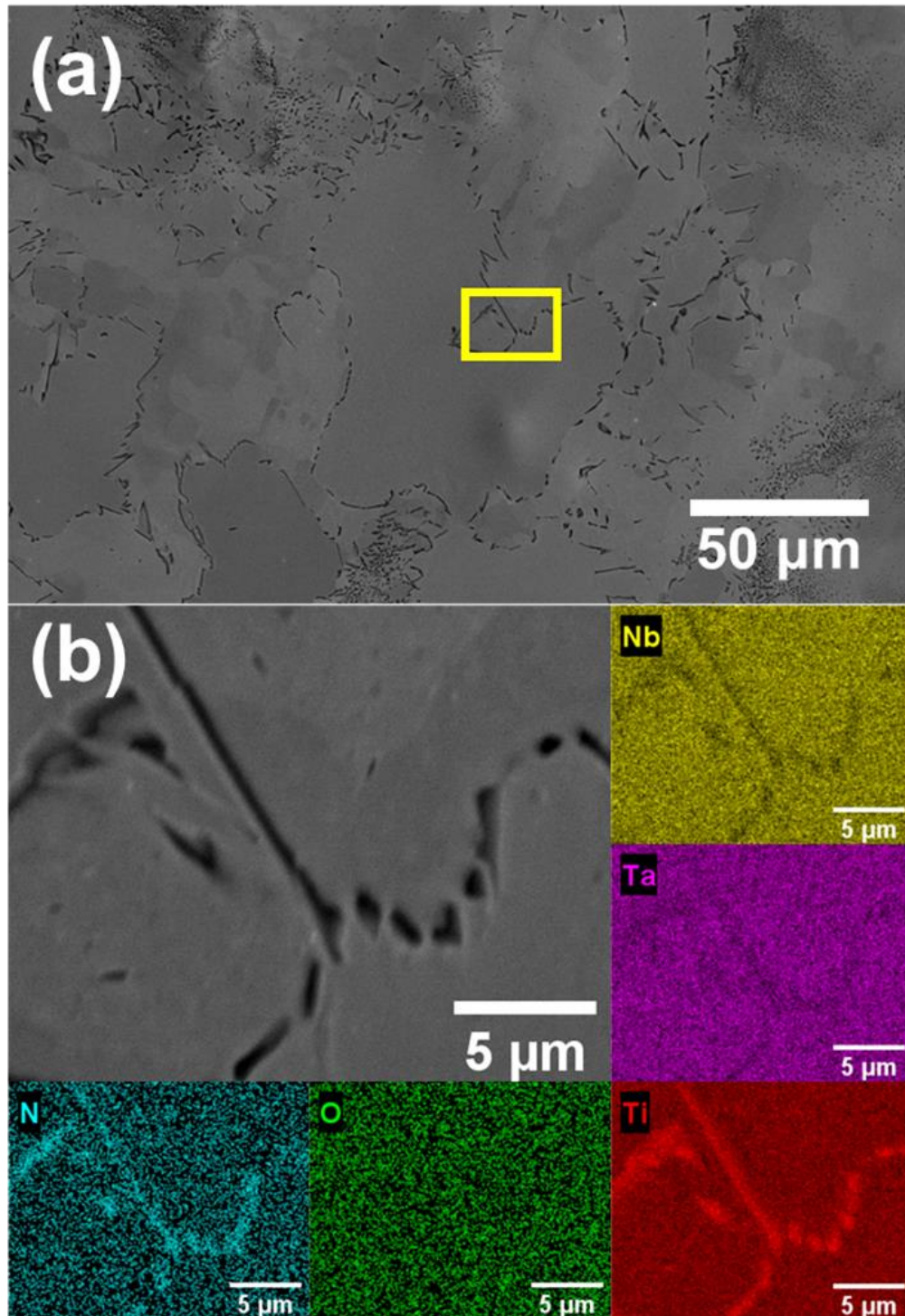


Figure 2-2: (a) SEM image of NbTaTi after annealing at 1473 K for 3 hours and slow cooling. (b) shows an expanded view of a phase segregated area with corresponding EDS maps that display segregation of Ti and N.

The important revelation of potential phase separation due to oxygen and nitrogen interstitial impurities is also supported by the CALPHAD computation, as shown in **Fig. 2-3**. **Fig. 2-3a** and **Fig. 2-3c** give the phase predictions of the pristine RCCAs, where the BCC single phase remains stable under the recrystallization annealing temperature of 1473 K and the subsequent creep testing temperature of 1173 K. However, even when minute concentrations of oxygen and nitrogen are considered, the temperature range of the thermodynamic stability of the Ti-rich HCP phase significantly increases to above 1700 K, which covers both the recrystallization annealing and the creep testing temperature. In addition, CALPHAD also predicts a new Ti_2N phase in NbTaTi and NbTaTiV that is stable at low temperatures, which appears to support the observed secondary phase decomposition for NbTaTi. Although CALPHAD predicts only minor fractions of undesirable secondary phases under bulk equilibrium conditions, actual phase decomposition can proceed rapidly at grain boundaries due to fast diffusion kinetics and strong local segregation. Consequently, the compositions and structures of the secondary phases may deviate significantly from equilibrium predictions. For instance, the Ti_2N phase predicted to adopt a rutile structure does not match the experimentally observed (Ti, N)-rich FCC phase reported in NbTaTi by Senkov et al. [33]. Given the large variability in interstitial impurity contents among stock materials and processing routes, a detailed determination of the crystal structure and composition of the secondary phases in this work is not expected to be universally representative and is therefore beyond the scope of the present study. Nevertheless, the presence of such phases can cause severe grain boundary embrittlement and markedly reduce the tensile ductility of the RCCAs [5], [6], [10], [21], [34].

The undesired secondary phase in NbTaTi was kinetically suppressed by faster air cooling after annealing, manifesting the inhibition of nucleation and growth of the low-temperature secondary phase, which led to the single-phase microstructure in **Fig. 2-1a**. NbTaTiV exhibited better phase stability and showed no signs of this elemental segregation under slow furnace cooling. This led to the single-phase microstructure in **Fig. 2-1b**. This indicates that the kinetics for secondary phase decomposition in NbTaTiV is slower than that of NbTaTi. However, for both NbTaTi and NbTaTiV, these CALPHAD data reveal that the presence of oxygen and nitrogen thermodynamically stabilize the formation of a (Ti, N)-rich phase at lower temperatures. The annealing times were controlled such that the two compositions have comparable grain sizes to allow for direct comparison of mechanical performance. Grain sizes and both nominal and observed compositions for each material are listed in **Table 2-1**. With our careful control in processing, the creep testing of NbTaTi and NbTaTiV began with two homogeneous BCC single-phase solid solutions with similar grain sizes.

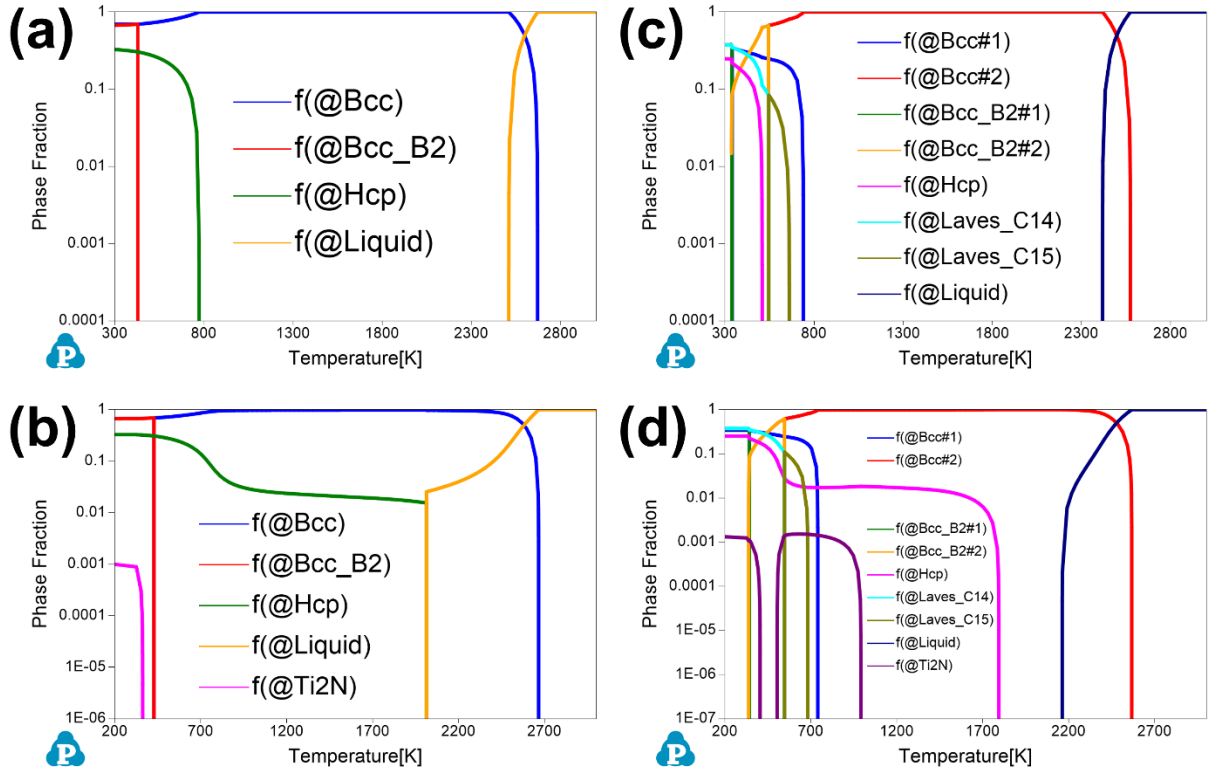


Figure 2-3: CALPHAD plots for NbTaTi (a) without O and N and (b) with 5000 molar ppm of O and 1000 molar ppm of N added. CALPHAD plots for NbTaTiV (c) without O and N and (d) with 5000 molar ppm O and 1000 molar ppm N added.

Table 2-1: Average grain sizes (μm) and EDS measured elemental compositions (atomic percent) of NbTaTi and NbTaTiV prior to testing.

Composition	Grain Size (μm)	Nb (%)	Ta (%)	Ti (%)	V (%)
NbTaTi	40.0 ± 7.6	34.3	33.4	32.3	-
NbTaTiV	52.3 ± 22.9	26.6	28.9	23.3	21.2

2.3.2 Creep results and microstructure evolution

Examples of the creep curves from NbTaTi and NbTaTiV are shown in **Fig. 2-4**. Well-defined primary creep characterized by a decreasing strain rate transitioning into secondary creep is

observed across all stress levels, which is indicative of strain hardening resulting from dislocation multiplication [35]. Creep tests under lower stresses typically spanned multiple weeks to months, and the tests were terminated within the long secondary creep regime. The high-stress creep curves in **Fig. 2-4b** show more typical creep behavior of primary, secondary, and tertiary creep. These tests spanned much shorter times, and thus they were terminated immediately when tertiary creep was observed to preserve the microstructure during secondary creep. In all tests, a clearly defined steady-state creep regime was observed, during which the creep rate remained constant under a constant true stress. The steady-state creep rate, $\dot{\epsilon}_{ss}$, can therefore be related to the applied stress σ by the power-law equation with an Arrhenius relation with temperature T [36]:

$$\dot{\epsilon}_{ss} = A\sigma^n \exp\left(-\frac{Q_c}{RT}\right), \quad (1)$$

where A is a material constant that includes dislocation density and elastic properties, n is the stress exponent, Q_c is the apparent activation energy of creep, and R is the ideal gas constant.

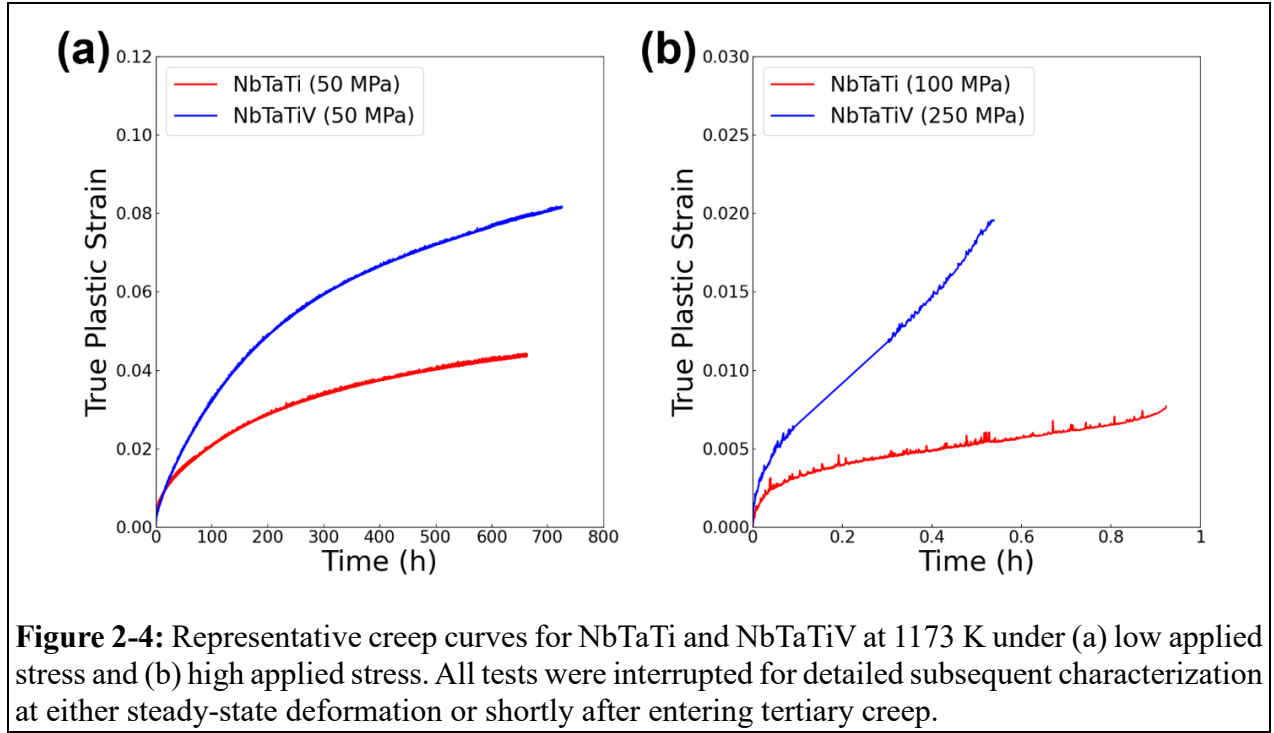


Fig. 2-5 displays a summary of the steady-state creep rates of NbTaTi, NbTaTiV, and a comparison with previously investigated $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$ as functions of applied stress and testing temperatures. NbTaTiV displays superior creep resistance compared to both NbTaTi and $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$, indicated by its lower steady-state creep rate at all stresses other than 50 MPa in **Fig. 2-5a**. It is observed that both NbTaTiV and $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$ follow the power-law relationship, although the data point at the lowest applied stress locates slightly below the overall trend. However, NbTaTi shows significant deviation from the power-law relation, where the steady-state creep rates at lower stresses decrease significantly. This behavior suggests a possible transition in the dominant deformation mechanism at lower stresses, likely associated with microstructural evolution induced by secondary phase formation during creep. Similarly, under low stresses and prolonged creep durations, a Hf-rich phase in $\text{Nb}_{45}\text{Ta}_{25}\text{Ti}_{15}\text{Hf}_{15}$ was previously reported to form along grain boundaries within the sample interior, while HfO_2 developed at grain

boundaries near the surface, even when testing was conducted under high vacuum [21]. However, these features were not observed in samples tested under higher stresses and shorter durations. Given the phase decomposition observed earlier during slow cooling of NbTaTi prior to testing and the predicted Ti-rich secondary phase formation tendency in both NbTaTi and NbTaTiV from CALPHAD calculations (**Fig. 2-3**), it is highly likely that secondary phase formation occurred during creep. Consequently, the steady-state creep rates, particularly under long testing durations, appear reduced due to potential precipitation strengthening. Therefore, the interpretation of the stress exponents is not straightforward if all data points are considered. For NbTaTi, NbTaTiV, and Nb₄₅Ta₂₅Ti₁₅Hf₁₅, the data point at the lowest stress of 50 MPa was excluded from the linear fit to determine the stress exponent.

The stress exponents for NbTaTi, NbTaTiV, and Nb₄₅Ta₂₅Ti₁₅Hf₁₅ are $n = 3.2$, $n = 3.0$, and $n = 3.6$, respectively. All three of these stress exponents best represent the solute-drag creep mechanism [37], [38], in which the glide of dislocations is controlled by the diffusion of solute atoms segregated at dislocation cores. In complex concentrated alloys, especially those with equiatomic compositions, the distinction between “solute” and “solvent” atoms within the solid solution becomes ambiguous. Consequently, a stress exponent near three is typically interpreted as evidence of thermally activated glide- or viscous glide-controlled creep [39-42], where the rate-limiting deformation mechanism involves dislocation-vacancy interactions controlled by the concentrated solid solution. Including the 50 MPa data points increases the apparent stress exponents for all compositions, yielding $n = 6.2$, 3.9 , and 4.1 for NbTaTi, NbTaTiV, and Nb₄₅Ta₂₅Ti₁₅Hf₁₅, respectively. These values are nominally consistent with dislocation climb-controlled creep [43]. However, inclusion of the 50 MPa data points is likely inappropriate, as

phase decomposition over long time, high temperature exposure at low stress may artificially elevate the measured stress exponent.

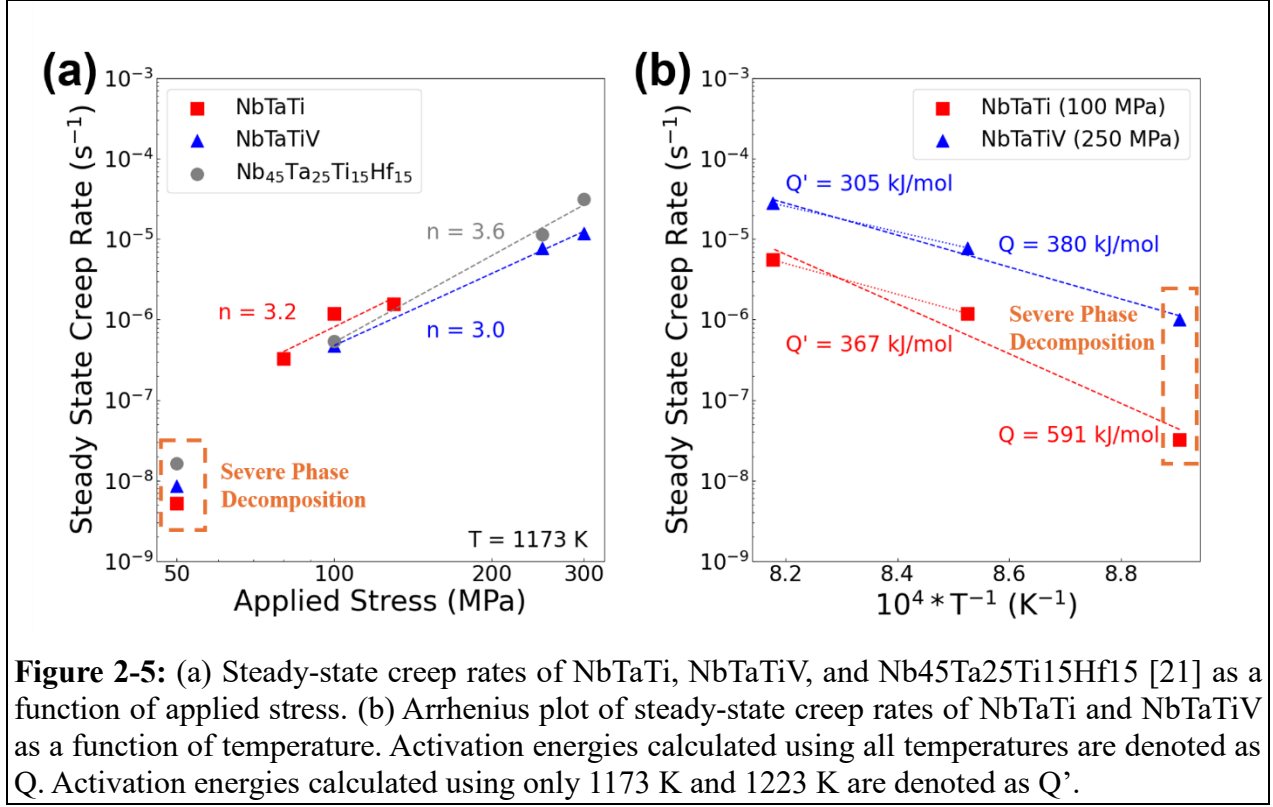
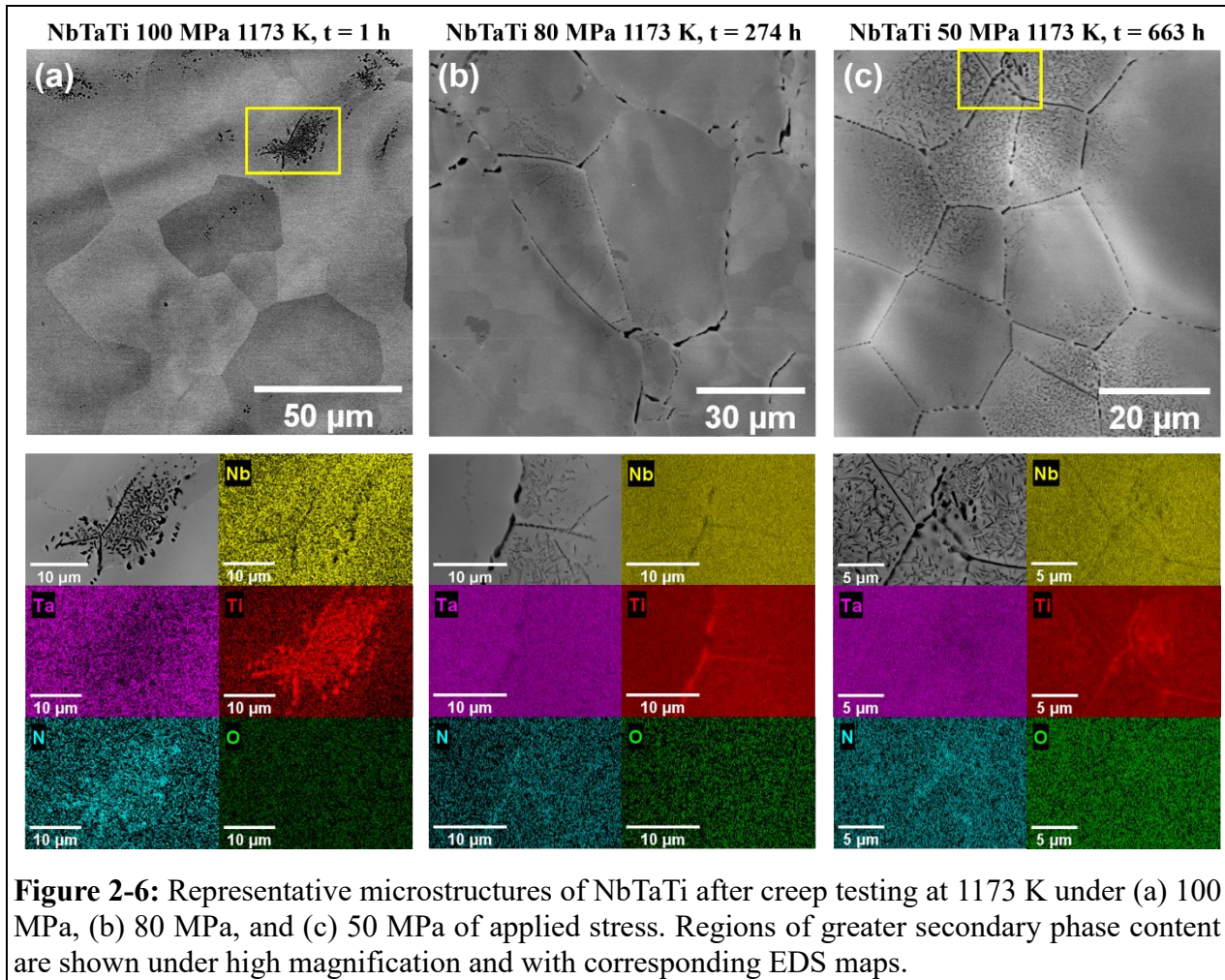


Fig. 2-5b reveals that the creep activation energies for NbTaTi and NbTaTiV are 591 kJ/mol and 380 kJ/mol, respectively. Again, the high apparent activation energy of 591 kJ/mol for NbTaTi is difficult to rationalize since it significantly exceeds all of its individual components' vacancy diffusion activation energies; the highest of which is Ta with a vacancy diffusion activation energy of 413 kJ/mol [44]. The reported activation energy of NbTaTi comes from fitting the Arrhenius relation with the three steady-state creep rates corresponding to the three tested temperatures: 1123 K, 1173 K, and 1223 K. One important observation not revealed by **Fig. 2-5b** is that the duration of these three creep tests varies greatly. Steady-state creep was reached at 1173 K and 1223 K under 100 MPa within one hour, whereas at 1123 K it took 189 hours. A likely secondary phase

formation can also occur at 1123 K, which could be strengthening the material under extended time. Eliminating the 1123 K data point and fitting the activation energy yields $Q' = 367$ kJ/mol for NbTaTi, displayed in **Fig. 2-5b**. This is a much more reasonable activation energy for this composition, as it lies near the atomic concentration-weighted rule-of-mixture average vacancy diffusion activation energy of its components in pure metal form: 322 kJ/mol [44]. A similar observation can be made for NbTaTiV, but much less pronounced than for NbTaTi. Both the 1173 K and 1223 K creep tests for NbTaTiV spanned less than one hour to reach steady state under 250 MPa, whereas the duration of the 1123 K creep test was 22 hours. The creep rate at 1123 K does appear to be lower than the trend from the higher temperature data. Thus, it can be argued that even for NbTaTiV there could be some strengthening at these creep testing temperatures that increases as a function of time; and this could be attributed to secondary phase decomposition and strengthening, which will be discussed in an upcoming section. Like with NbTaTi, the creep activation energy can be fitted with just the 1173 K and 1223 K data, and this yields $Q' = 305$ kJ/mol. This is similar to the rule-of-mixtures vacancy diffusion activation of its components: 319 kJ/mol. As the measurements of diffusion coefficients and activation energies in RCCAs are largely absent to date, these rule-of-mixture activation energies serve as a first approximation to evaluate how the activation energies measured during creep can be correlated with those for vacancy diffusion. It can be concluded that a good agreement between these activation energies is observed if the solid solution can largely remain in a single phase.

The creep results above show a critical need to characterize the evolution of microstructure, especially on the potential phase decomposition that causes strengthening during creep. **Fig. 2-6** shows that there is a strong presence of a secondary phase in NbTaTi. This secondary phase is a Ti-rich phase that is also rich in N, which is similar to the FCC (Ti, N)-rich phase observed by

Senkov et al. [33]. This secondary phase is even present at short creep durations such as $t = 1$ hour in **Fig. 2-6a**. This indicates that this secondary phase could already be forming even as the sample is being heated up to the desired temperature of 1173 K. Progressing from **Fig. 2-6a** to **Fig. 2-6b** to **Fig. 2-6c**, it is also clear that the concentration of the secondary phase increases as the creep test duration increases. In **Fig. 2-6a**, the secondary phase appears grouped and sparsely dispersed across the bulk. **Fig. 2-6b** shows a greater spread of the secondary phase, as it appears concentrated at the grain boundaries, as well as having some presence within grains. The longest creep testing time of $t = 663$ hours reveals that the secondary phase is present strongly at the grain boundaries, forming a continuous network, as shown in **Fig. 2-6c**. A large fraction of the bulk grain area also contains this secondary phase. This implies that the conditions of the creep testing environment do provide a driving force for this phase decomposition, and the kinetics are slow enough to observe the time dependence of this decomposition. The presence of N seems to drive the segregation of Ti, and the source of N is likely from trace amounts of N present in the stock material since the creep tests were conducted under high vacuum. It is possible that this phase decomposition may be enhanced due to the thin geometry of the sample, as thinner creep specimens have shown more prominent phase decomposition, especially for low stresses and creep test durations exceeding 100 hours [45], [46]. The confirmation of significant secondary phase formation can rationalize the improvement of the creep properties of NbTaTi at longer times (or lower stresses).



Although NbTaTiV displayed sufficient phase stability in the initial processing steps prior to creep testing, **Fig. 2-7** reveals that secondary phase decomposition occurs for NbTaTiV under creep testing conditions. NbTaTiV also shows a secondary phase that is Ti and N rich, similar to NbTaTi [33]. However, the EDS also reveals that O is also concentrated at some parts of these secondary phases. As displayed in the evolution of NbTaTi over time, NbTaTiV also displays a general increase of secondary phase content as the creep test duration increases. Even at the shortest creep test duration of $t = 0.6$ hours, phase decomposition already occurred as discontinuous spots of this secondary phase are present at the grain boundaries, seen in **Fig. 2-7a**. At an intermediate creep test duration, this secondary phase is no longer contained only at the grain boundaries, but it is also

spread in clusters across multiple grains, displayed in **Fig. 2-7b**. **Fig. 2-7c** reveals significant phase decomposition in NbTaTiV, with the secondary phase being present both strongly at grain boundaries and within the majority of grains.

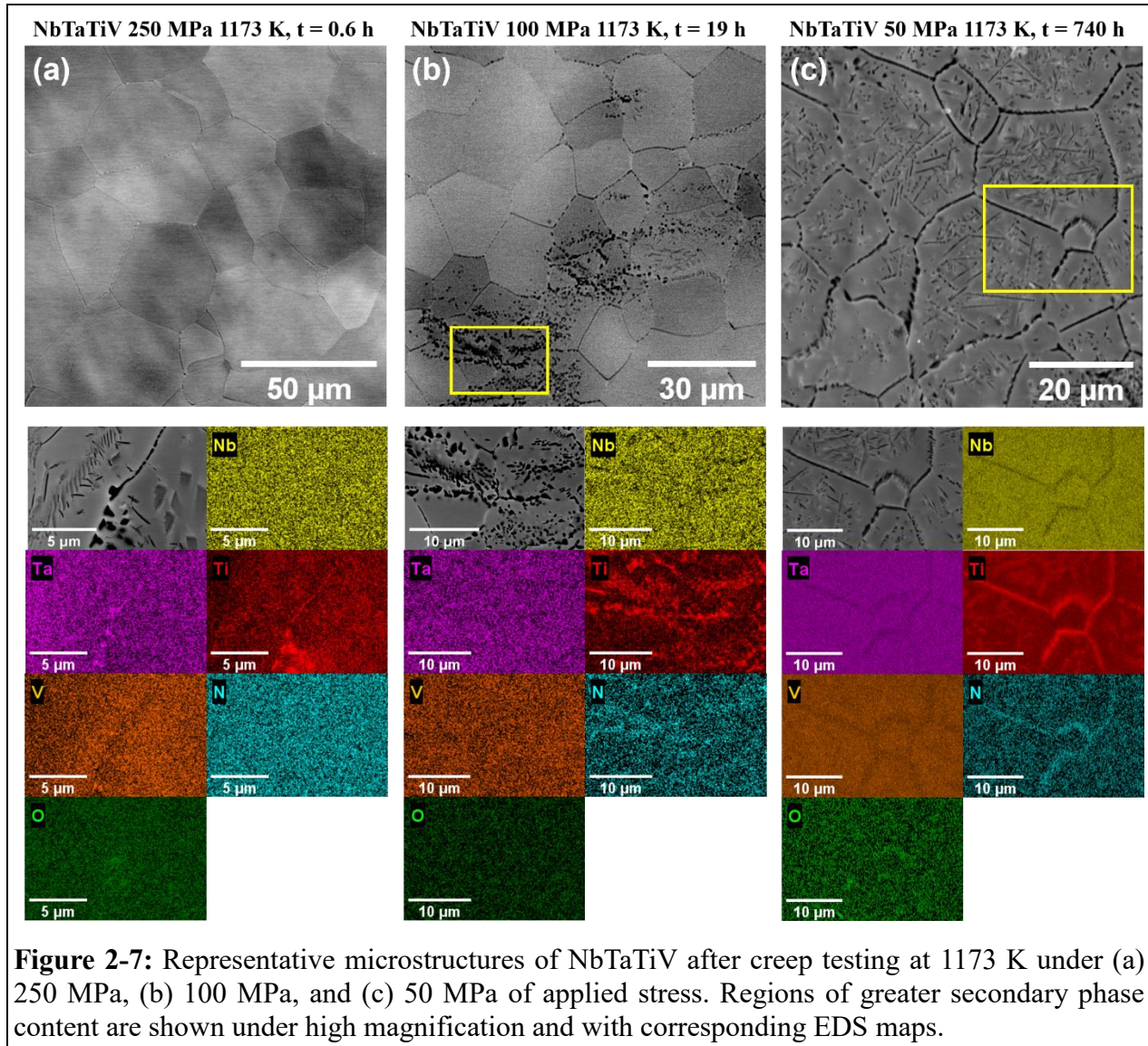
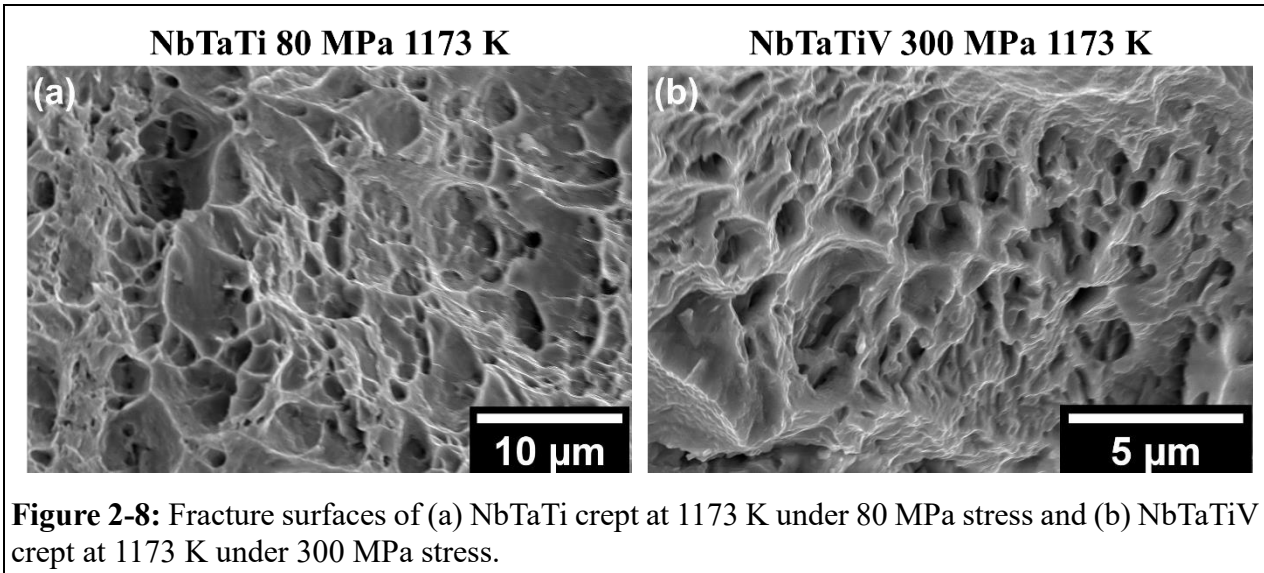


Fig. 2-6 and **Fig. 2-7** indicate that both NbTaTi and NbTaTiV have phase instability, as Ti segregates from the BCC solid solution with interstitial impurities. Additionally, this phase segregation is exacerbated by increasing the time that these materials are held at 1173 K. However,

the formation of these secondary phases appears to enhance the creep resistance of the alloys, suggesting that deliberate incorporation of precipitation strengthening mechanisms could be beneficial in the design of nominally single-phase RCCAs.

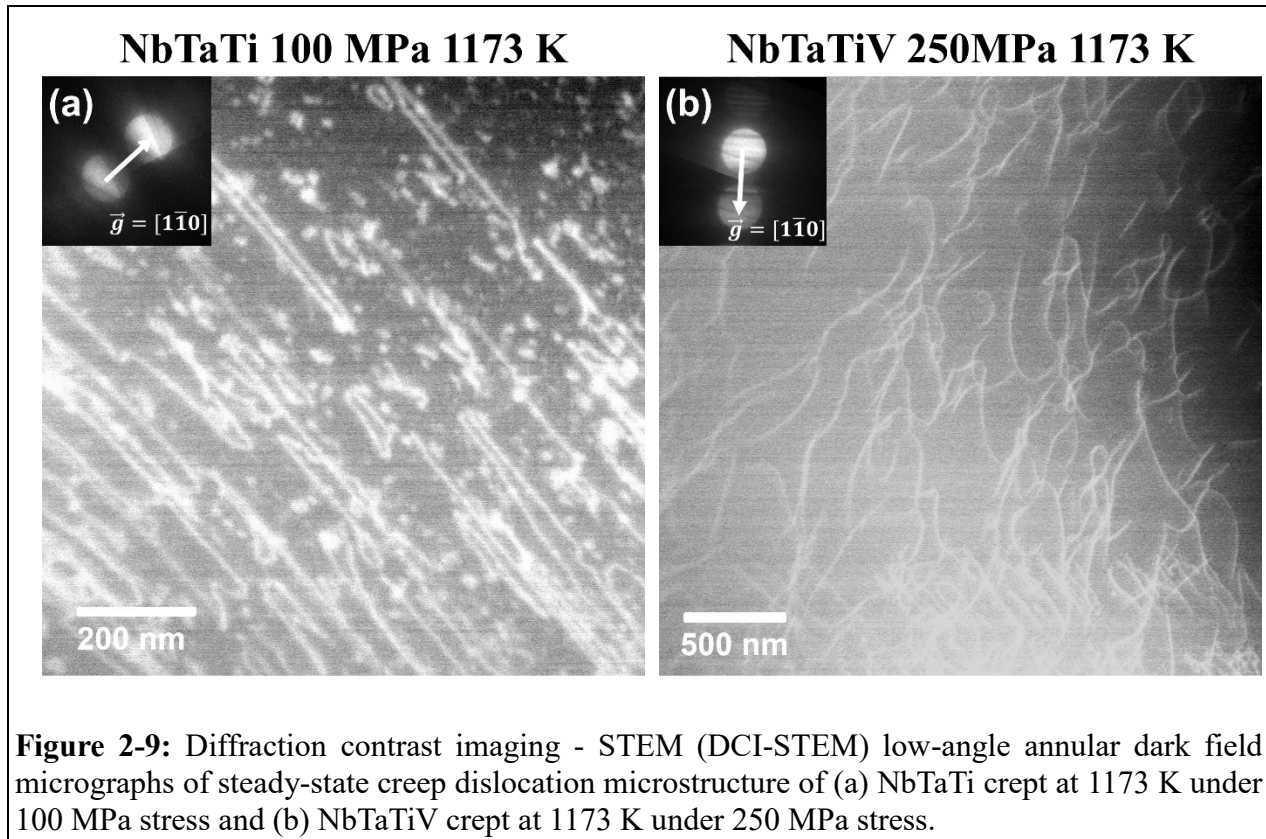
Despite the appearance of Ti-rich phase decomposition, both NbTaTi and NbTaTiV displayed ductile fracture for samples crept to failure. Representative fracture surfaces, shown in **Fig. 2-8**, show dimples throughout. This indicates that the quantity of the secondary Ti-rich phase may not be significant enough to cause NbTaTi and NbTaTiV to become embrittled by either intergranular fracture or cleavage. Additionally, the similarity between these NbTaTi and NbTaTiV fracture surfaces demonstrate that the addition of V also does not embrittle the material.



2.3.3 Creep mechanisms

The creep mechanisms in NbTaTi and NbTaTiV were further investigated through TEM characterization, revealing behaviors that extend beyond a simple interpretation based on thermally activated dislocation glide, as inferred from their apparent stress exponents (excluding

the 50 MPa data point affected by severe phase decomposition). **Fig. 2-9** clearly reveals distinct rate-controlling mechanisms in NbTaTi and NbTaTiV. In NbTaTi, the presence of long, straight screw dislocations accompanied by a high density of debris (interstitial or vacancy loops) indicates that deformation is governed by screw-dislocation glide through cross-kink unpinning. An identical dislocation substructure has been previously reported in creep-deformed Nb₄₅Ta₂₅Ti₁₅Hf₁₅ samples [21], where detailed g-dot-b analysis was performed. Therefore, this procedure is not repeated here. On the other hand, NbTaTiV exhibits a notable absence of debris and features curvy, interacting dislocations, indicative of strong dislocation–solute interactions. These curved dislocations naturally adopt mixed characters as their sense vectors vary while maintaining constant Burgers vectors, emphasizing an equally important role of edge components in controlling creep deformation compared screw components. The dislocation substructure in NbTaTiV exhibits comparable mobility of screw and edge dislocations, a behavior reminiscent of FCC CCAs. Consequently, the post-creep dislocation substructures in NbTaTiV closely resemble those observed in recrystallized CrMnFeCoNi [42], [47] and CrCoNi [29]. The TEM analysis here evidently demonstrates that the mechanisms governing the yield strengths of NbTaTi and NbTaTiV can also be extended to their creep deformation behavior at lower strain rates, which can rationalize the higher creep resistance of NbTaTiV compared to NbTaTi and Nb₄₅Ta₂₅Ti₁₅Hf₁₅.



A final discussion is presented here, following a comprehensive analysis of the creep behaviors and mechanisms in NbTaTi and NbTaTiV. NbTaTiV has the most superior creep properties of NbTaTi, NbTaTiV, and Nb₄₅Ta₂₅Ti₁₅Hf₁₅, since it has the lowest creep rate for the majority of the applied stresses in this work (**Fig. 2-5**). It also holds the greatest yield strength of these three materials. **Fig. 2-10a** compares the tensile yield strengths of the materials in this work with the selected single-phase BCC RCCAs and single-crystal Ni-based superalloy CMSX-4 up to 1573 K [10], [11], [30], [48]. It is observed that NbTaTiV maintains a superior yield strength compared to HfNbTaTiZr above 1000 K and exceeds that of CMSX-4 above 1253 K. This represents a promising advancement in the strength of RCCAs due to the effect of edge strengthening and underscores their potential for applications where they surpass the strength of Ni-based superalloys below the latter's maximum operating temperature. However, the creep properties are strongly

lacking. This is made clear in **Fig. 2-10b**, where NbTaTiV displays only slightly superior creep properties to other RCCAs, but its creep resistance is many orders of magnitude worse than that of CMSX-4 at the same temperature. More importantly, at slower strain rates, the pronounced edge-strengthening advantage of NbTaTiV, which gives it a much higher yield strength than NbTaTi, Nb₄₅Ta₂₅Ti₁₅Hf₁₅, and HfNbTaTiZr, appears to diminish. Additionally, the stress exponent of CMSX-4 is $n = 12.5$. Along with its already low creep rates at stresses over 200 MPa, this stress exponent indicates that Ni-based superalloys can display vanishingly small creep rates under low stress applications at 1253 K. This high apparent stress exponent is related to the back stresses applied on the mobile dislocations via a high volume fraction of particles [49], [50]. Incorporating secondary phases is essential to improve the creep strength of RCCAs, as some strong single-phase BCC RCCAs in yield such as NbTaTiV cannot compete with Ni-based superalloys under creep. This work shows that this is likely the case, as the 50 MPa creep tests for NbTaTi and NbTaTiV with unintentional precipitation showed a sharp decrease in steady-state creep rate compared to the higher stress tests. This is attributed to the large fraction of secondary phases that formed within these materials over long creep durations, demonstrating that secondary phases can improve the creep strength for BCC RCCAs and increase their stress exponents.

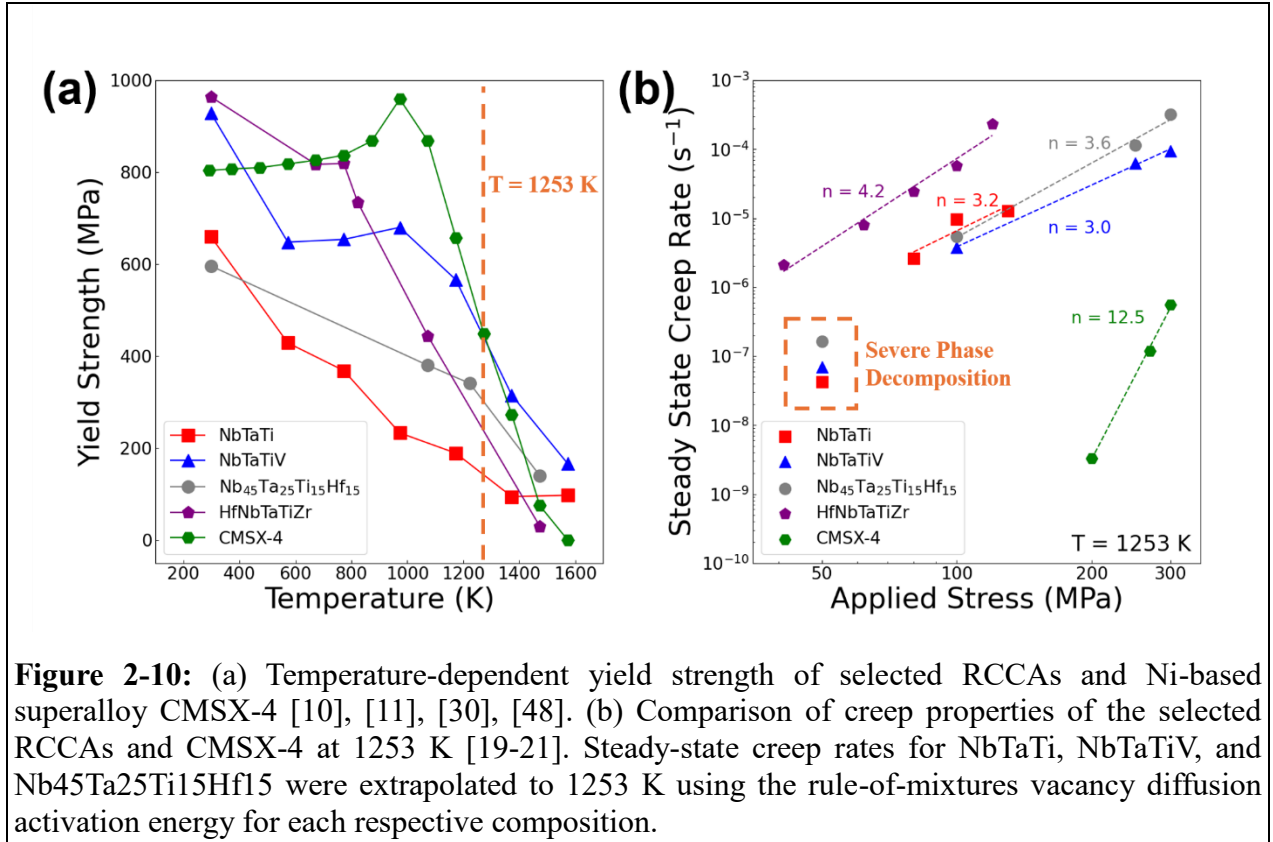


Figure 2-10: (a) Temperature-dependent yield strength of selected RCCAs and Ni-based superalloy CMSX-4 [10], [11], [30], [48]. (b) Comparison of creep properties of the selected RCCAs and CMSX-4 at 1253 K [19-21]. Steady-state creep rates for NbTaTi, NbTaTiV, and Nb₄₅Ta₂₅Ti₁₅Hf₁₅ were extrapolated to 1253 K using the rule-of-mixtures vacancy diffusion activation energy for each respective composition.

2.4 Conclusions

The main results of the paper can be summarized as follows:

1. NbTaTiV exhibits the highest yield strength and superior creep resistance among NbTaTi, Nb₄₅Ta₂₅Ti₁₅Hf₁₅ and HfNbTaTiZr, with edge dislocation glide-controlled deformation contributing significantly to its mechanical performance.
2. Tensile creep of NbTaTi and NbTaTiV follows a thermally activated dislocation glide-controlled mechanism with stress exponents near three, while activation energies are consistent with a rule-of-mixtures estimation of vacancy diffusion in their constituent elements.
3. Both NbTaTi and NbTaTiV undergo phase decomposition during creep, forming Ti- and N-rich secondary phases. This secondary phase formation, though unintentional, enhances creep

resistance, suggesting that controlled precipitation strengthening is a viable strategy for improving BCC RCCA performance.

4. Despite NbTaTiV's high yield strength, single-phase BCC RCCAs remain insufficient to compete with Ni-based superalloys in creep under engineering-relevant low-stress, long-duration conditions.

5. These findings underscore that deliberate secondary phase design, in conjunction with strategic alloying (e.g., V for edge-dislocation strengthening), is essential for developing next-generation RCCAs with both high strength and creep resistance at elevated temperatures.

2.5 Acknowledgements

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Chapter 3. The Effect of Lattice Distortion on Tensile and Creep Properties of the NbV Alloy

3.1 Introduction

3.1.1 Complex concentrated alloys

Complex concentrated alloys (CCAs) are an emerging class of materials that display exceptional strength while challenging traditional alloy design [1], [2]. CCAs are typically made up of at least three different elements at high concentrations, often at equiatomic levels [1]. These materials may be single-phase or multi-phase, and they have a wide range of desirable properties depending on their compositions. One example of a set of desirable properties includes retaining high strength at elevated temperatures [3], [4] along with acceptable tensile ductility [5], [6] which is important for realistic applications. In light of the variance in the mechanical properties of CCAs, the high complexity of having many component elements at high concentrations makes it difficult to predict exactly how slight compositional changes will affect the material response. One approach to mitigating this high variability this would be simplifying the CCA space to sets of simpler binary or tertiary compositions in order to isolate certain structure-property relationships. Using smaller composition spaces to build models supported by extensive theory and strong experimental data can help inform CCA design to match specific engineering requirements and constraints.

3.1.2 Strengthening in CCAs

The main strengthening effect present in CCAs is solid solution strengthening; in many cases, over half of the yield strength of CCAs can be attributed to solid solution strengthening [7], [8], [9]. Thus, it is important to understand this fundamental mechanism behind the impressive mechanical

properties of CCAs in order to inform further development of superior designs. One way to approach this is through predictive modelling of solid solution strengthening in CCAs. This has been done by the Toda-Caraballo model, which uses a foundation of solid solution strengthening models for dilute binary systems and several refinements to apply to the CCA space [10], [11], [12], [13]. In this model, two misfit parameters make up the strengthening term: modulus misfit and atomic size misfit. This atomic size misfit is the main focus of this work. The atomic size misfit is directly tied to lattice distortion [14], which is readily quantified by methods introduced by Lee, et al. [15]. The effects of lattice distortion and solid solution strengthening is best observed in single-phase CCAs, as this eliminates strengthening from other factors such as secondary phase formation, which is quite common in many CCA studies [16], [17], [18]. The Nb-Ta-Ti-V system displays a single phase microstructure [6], [15], [19], which makes it a prime system for this study.

3.1.3 Scope of this work

The aim of this work is to observe the effects on the change of the mechanical properties from the Nb-Ta-Ti-V system by maximizing the lattice distortion effect. Knowing that lattice distortion is mainly controlled by the V content in this system [15], the target alloy in this investigation is equiatomic NbV. This composition is important for two main reasons: the content of V is made identical to that of Nb to ensure the greatest degree of atomic size misfit, and the elimination of Ta and Ti to simplify the system. Removing Ta and Ti from the system isolates lattice distortion, promoting mostly atomic size misfit instead of modulus misfit, while avoiding possible secondary phase effects seen with compositions containing these elements [20]. Both tensile testing and tensile creep testing were planned to identify whether the lattice distortion effect is the main controlling factor of yield strength and creep resistance in these single phase BCC CCAs. Tensile

testing was planned over a wide temperature range to understand at which temperatures lattice distortion controls the yield strength. Extensive characterization via scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were planned for after-testing specimens in order to connect deformation microstructures with the mechanical response and the effect of maximizing the lattice distortion.

3.2 Experimental

60 g circular buttons of 50 at% Nb and 50 at% V alloy were produced by vacuum arc melting using high purity elements. The arc melter was pumped below 3×10^{-3} Pa and backfilled with high purity Ar gas. Each button was flipped and remelted at least five times to ensure thorough mixing of the constituent elements. These buttons were then cold rolled to two different thicknesses, both exceeding 80% reduction. Buttons were rolled to a final thickness of approximately 0.8 mm for tensile creep specimens, and approximately 1.5 mm for tensile testing specimens. All specimens were cut directly from the rolled sheets via electrical discharge machining. All specimens were annealed in a sliding tube furnace under vacuum of 7×10^{-3} Pa at 1573 K for 5 hours and cooled in the furnace at a rate of 10 K per minute. These annealing parameters were necessary for recrystallization and control of the grain size prior to testing such that they match the initial microstructures of the Nb-Ta-Ti-V alloys used in Refs. [19], [20], [21], allowing for direct comparison of mechanical properties.

Constant stress tensile creep tests were performed under vacuum of 7×10^{-4} Pa at 1173 K using stresses of 100, 250, and 300 MPa. An additional creep test at 130 MPa at 1173K is planned. The gauge sections of the creep specimens were mechanically ground to 600 grit using SiC sandpaper to minimize surface effects [22]. Creep tests were performed either to failure or interrupted during

steady-state creep. These interrupted tests were necessary for further characterization of deformation microstructures via TEM. Additional creep tests are planned at 250 MPa at temperatures of 1123 K and 1223 K in order to determine the apparent activation energy of creep.

Tensile tests were performed at 298 K and 1173 K. One test at 298 K was performed to failure, and a repeat test will be interrupted at yielding for further characterization of deformation microstructure via TEM. Further tests are planned for the following temperatures: 573 K, 773 K, 973 K, 1373 K, and 1573 K to allow for direct comparison of tensile yield strengths of other Nb-Ta-Ti-V system alloys reported in Refs. [19], [21]. These tests are also planned to be interrupted at yielding for extensive characterization. All tensile tests were performed at a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$.

Microstructure characterization was performed via SEM on a ThermoFisher Quattro S Environmental SEM equipped with backscattered electron (BSE) and energy-dispersive x-ray spectroscopy (EDS) techniques. SEM sample preparation began with mechanical polishing using SiC sandpaper down to 1200 grit. Then, samples were electropolished using 50 mA of current in 10 vol% H_2SO_4 in methanol at 238 K.

3.3 Results and Discussion

3.3.1 Initial microstructure

The microstructure of NbV prior to mechanical testing is presented in **Fig. 3-1**. The microstructure displays equiaxed grains following the recrystallization. Additionally, NbV also appears to be homogeneous, as there are no obvious signs of secondary phase formation under BSE imaging or elemental segregation in the EDS maps for the two constituent elements. This is consistent with

other NbV works, which also report that NbV displays a single phase BCC microstructure [23], [24]. The average grain size and the elemental composition as reported by EDS appear in **Table 3-1**. This grain size and single phase microstructure is comparable with that used in Refs. [19], [20], [21]. Thus, mechanical testing began with samples with this initial microstructure.

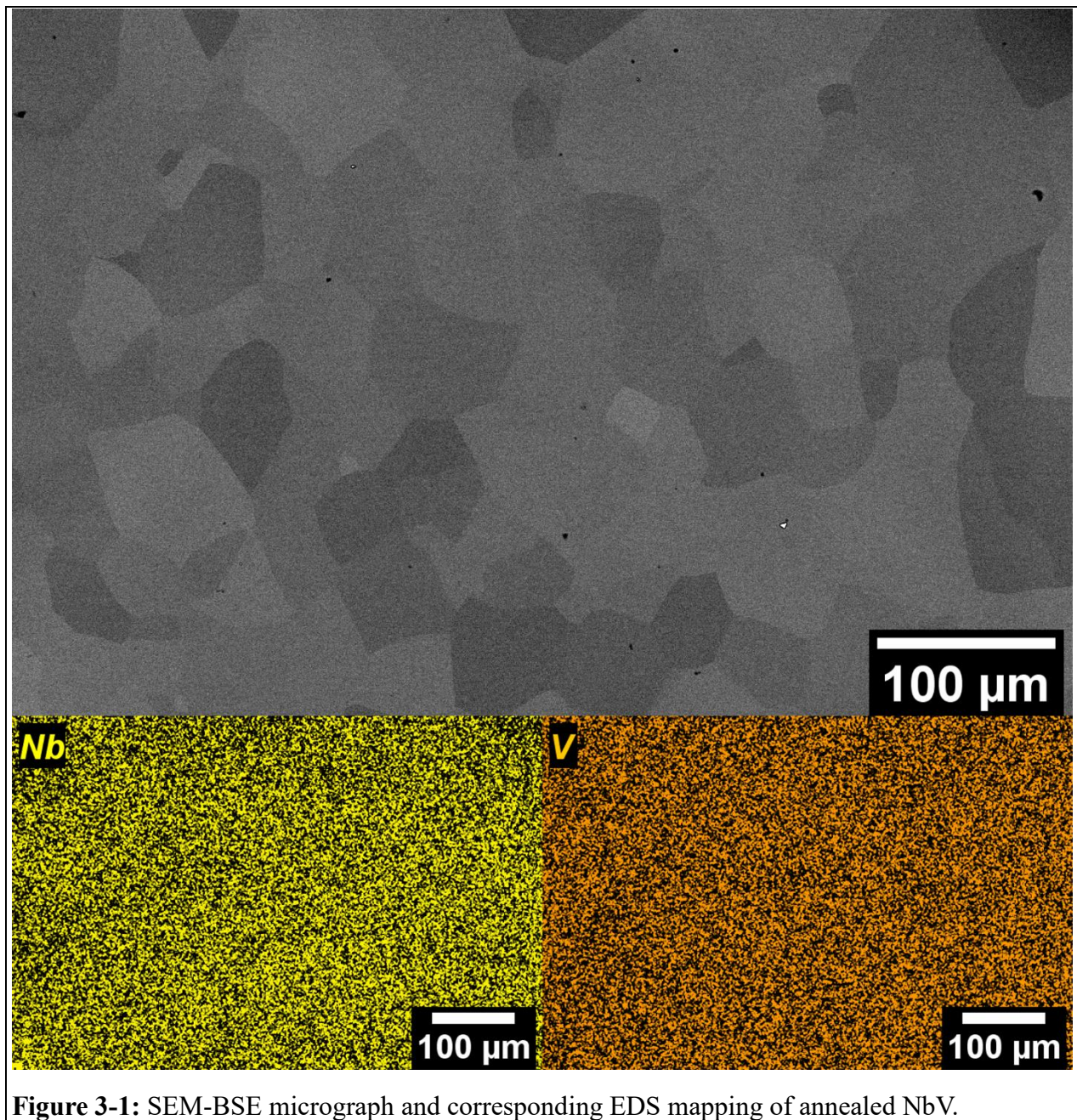


Figure 3-1: SEM-BSE micrograph and corresponding EDS mapping of annealed NbV.

Table 3-1: Average grain size and composition of NbV alloy from SEM and EDS results.

Grain Size (μm)	Nb (at%)	V (at%)
53.8 ± 10.9	51.0	49.0

3.3.2 Mechanical properties

The steady state creep rates of NbV at 1173 K are displayed as a function of applied stress alongside RCCAs of similar elements in **Fig. 3-2a**. NbV displays superior creep performance up to a full order of magnitude compared to NbTaTiV at 100 MPa and 300 MPa. However, unlike the other Nb-based CCAs, the stress exponent for NbV is approximately $n = 1.4$, compared to the others at $n \approx 3$. This indicates that there may be a fundamentally different creep mechanism at play for NbV. A stress exponent of $n = 3$ is associated with the solute-drag mechanism where dislocation motion is rate limited by the diffusion of solute atoms at the dislocation cores [25]. However, a stress exponent between $n = 1$ and $n = 2$ is associated with lattice self-diffusion or grain boundary diffusion [25]. This stress exponent appears questionable, especially since fitting only the two highest stress tests yields a stress exponent of $n = 3.8$, which is expected of Nb-based CCAs. Whether this change in stress exponent is only an artifact of preliminary data at this stage or truly indicates a shift in creep mechanism at lower stresses is yet to be seen through more testing and extensive post-creep characterization. Regardless, it is of key significance that the NbV alloy outperforms previous Nb-based CCAs under creep at 1173 K for the tested stresses, which may be attributed to its intentionally high lattice distortion composition.

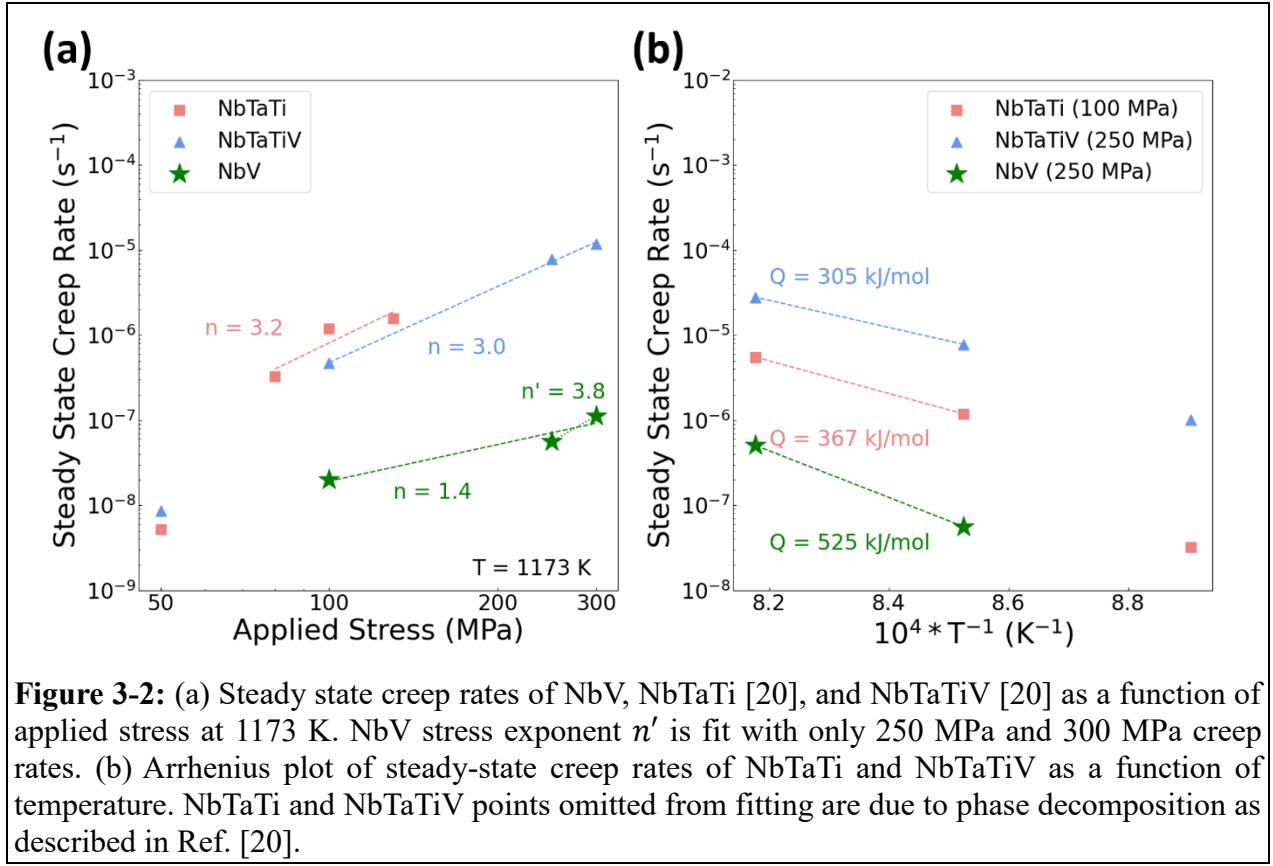


Fig. 3-2b reveals that the apparent creep activation energy of NbV is 525 kJ/mol. This activation energy exceeds either of the two components' vacancy diffusion activation energies (401 kJ/mol for Nb and 308 kJ/mol for V) [26]. The increase in activation energy could be attributed to sluggish diffusion, which is a hypothesized mechanism where the potential energy landscape caused by lattice distortion creates large barriers to diffusion, typically associated with CCAs [27], [28]. Although NbV by definition is not a CCA since it only has two components, the proposed high lattice distortion could contribute to the sluggish diffusion effect.

The tensile yield strengths of NbV with respect to temperature are plotted alongside similar RCCAs in **Fig. 3-3**. NbV displayed a yield strength of 926 MPa, which is about the same as the reported yield strength of 928 MPa from Ref. [19]. Interestingly, this indicates that increasing the

V content may only contribute to the strengthening to a certain limit. The high strength of the NbTaTiV alloy may also be attributed to an increased oxygen content, where oxygen diffusion into the matrix could cause further strengthening. This cannot yet be confirmed, as the oxygen content of the NbV alloy has not been explored in this work. The yield strength of NbV at 1173 K was 539 MPa, which again is comparable to the yield strength of NbTaTiV at 566.2 MPa from Ref. [19]. The overall trend of increasing the concentration of V in the Nb-Ta-Ti-V alloys seems to be associated with increased strengthening at 298 K and 1173K, but this effect appears diminished with the extreme V concentration of NbV. The quantitative connection between the V content and lattice distortion will be covered in the following section.

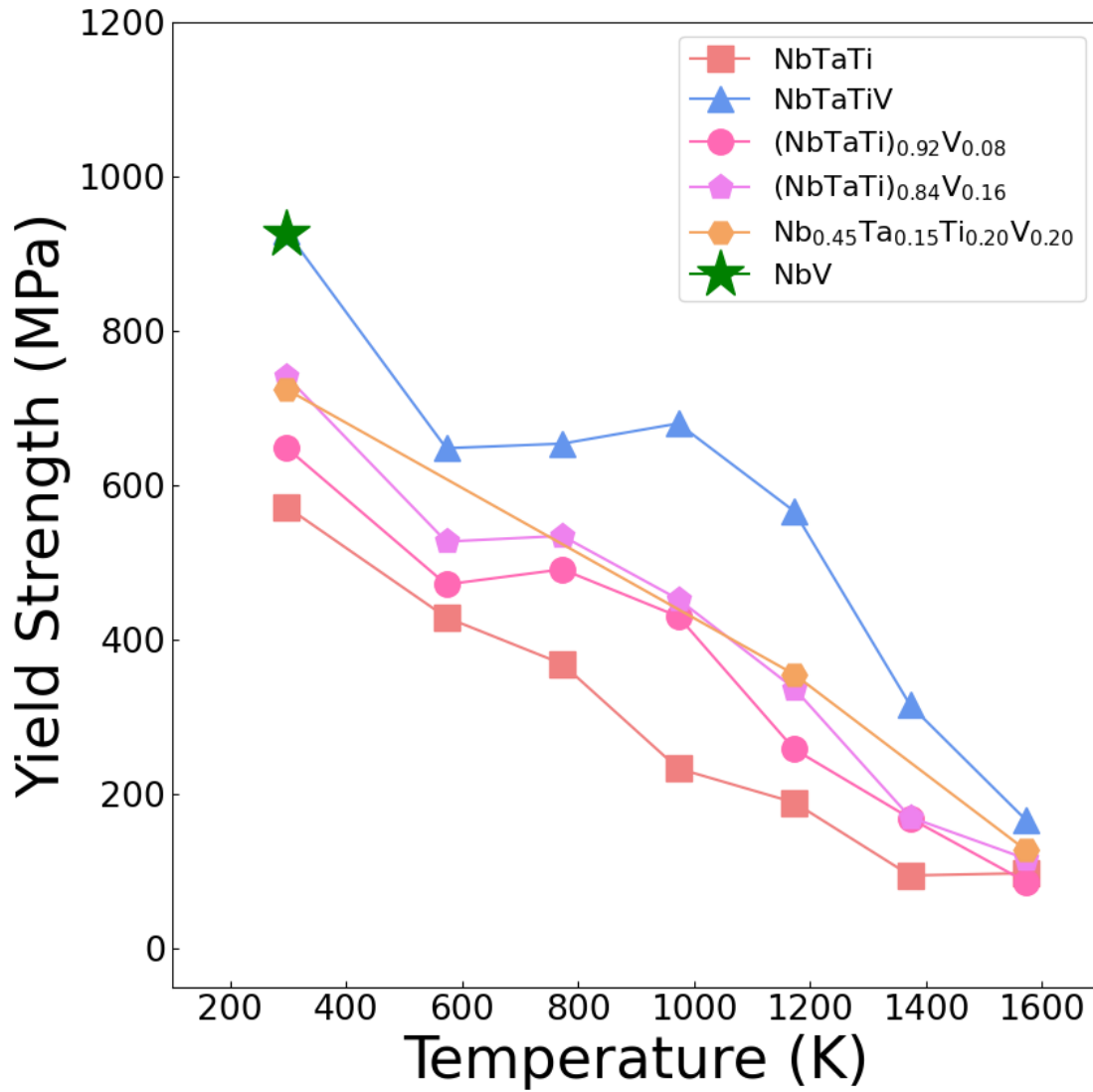


Figure 3-3: Yield strength as a function of temperature for various Nb-Ta-Ti-V system alloys [19] [21].

3.3.3 Lattice distortion and solid solution strengthening

The origin of the superior strength and creep resistance of NbV compared to its RCCA counterparts can be attributed to its greater lattice distortion. Knowing that in the Nb-Ta-Ti-V system, Nb, Ta, and Ti all share a similar atomic size whereas V is significantly smaller [29], it follows that the

lattice distortion is controlled by the concentration of V due to its atomic size misfit. In the case of NbV, it is likely that the lattice distortion is maximized due to the equal concentrations of Nb and V. This can be quantified via the lattice distortion model reported in Ref. [15]. Given the first nearest neighbor interatomic distances and atomic volumes of each constituent element in the Nb-Ta-Ti-V system, the lattice distortion $\overline{u^D}$ can be calculated by **Eq. 3-1**:

$$\overline{u^D} = \sqrt{\sum_i^n f_i (d_i^{eff} - \bar{d})^2} \quad (3-1)$$

where f_i is the atomic fraction of element i , d_i^{eff} is the effective first nearest neighbor interatomic distance of element i , and $\bar{d}$ is the average first nearest neighbor interatomic distance of the n -element alloy. In this work, $\bar{d}$ is calculated as the atomic fraction weighted rule of mixtures for each of the constituent elements' first nearest neighbor distance. The inclusion of f_i is actually a modification of the equation provided in Ref. [15], as this factor is necessary to take into account non-equiatomic compositions and their components' weighted contributions to lattice distortion. This step is also necessary to ensure that a pure element would yield a lattice distortion of 0 when modelling with this equation. The values for d_i^{eff} from **Eq. 3-1** are calculated by **Eq. 3-2**:

$$d_i^{eff} = \sum_j^n f_j \left(\frac{\Delta V_{ij}}{V_i} \right)^{1/3} d_i \quad (3-2)$$

where f_j is the atomic fraction of element j , ΔV_{ij} is the change in atomic volume of element i dissolved in element j , V_i is the atomic volume of element i , and d_i is the first nearest neighbor interatomic distance of element i . For this model, data for ΔV_{ij} and V_i were calculated from the special quasi-random-structure (SQS) method [30] and taken from Ref. [15].

This model is used in **Fig. 3-4**, which shows the lattice distortion as a function of V content in at%. The plotted theoretical curve uses the same SQS data and interatomic distances from Ref. [15] to model how the lattice distortion changes in the pseudo-binary $(\text{NbTaTi})_{1-x}\text{V}_x$. As expected, this model predicts that lattice distortion increases as a function of V content until it attains its maximum value at approximately 50 at% V. The experimental data in **Fig. 3-4** is calculated using interatomic distances from neutron diffraction data [15]. Since this data pertains directly to the $(\text{NbTaTi})_{1-x}\text{V}_x$ system, the pseudo-binary experimental data falls almost exactly on the curve. Additionally, the lattice distortion of the composition $\text{Nb}_{0.45}\text{Ta}_{0.15}\text{Ti}_{0.20}\text{V}_{0.20}$ falls close to the curve due to it containing all 4 elements. However, it is clear in **Fig. 3-4** that the lattice distortion of NbV actually exceeds that of the theoretical lattice distortion of the pseudo-binary with 50 at% V. This result goes against what may be expected of CCA systems, where increasing the number of elements intuitively should increase the diversity in component element atomic sizes and thus increase lattice distortion further.

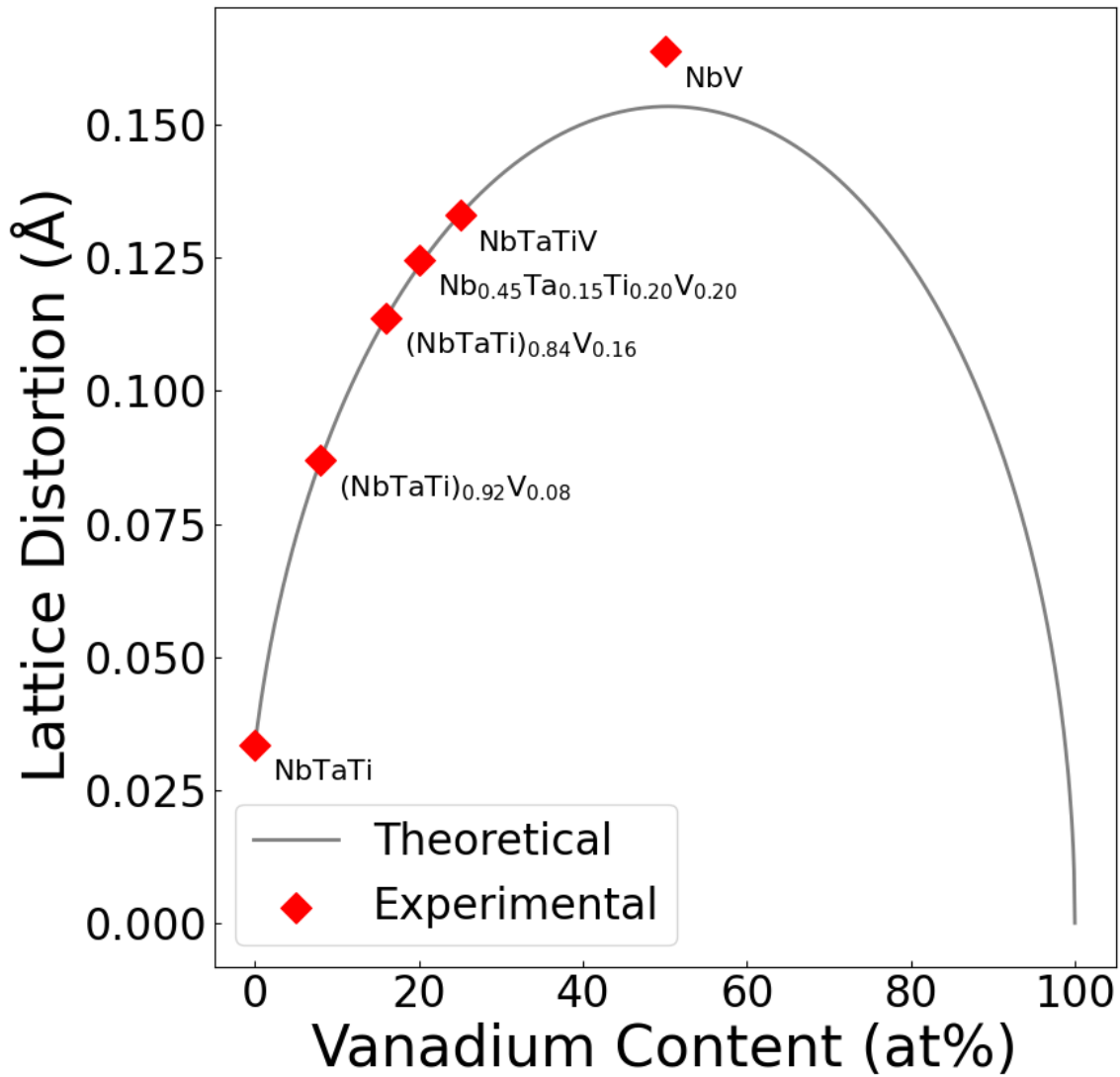


Figure 3-4: Lattice distortion plotted as a function of vanadium content. The theoretical curve is calculated according to Eq. 3-1 and Eq. 3-2 using the pseudo-binary system (NbTaTi)_{1-x}V_x.

An increase in lattice distortion is associated with an increase in strength since this indicates increasing atomic size misfit between elements in the alloy. Atomic size misfit is directly related to solid solution strengthening according to the Fleischer model [10]. Through modifications and refinements of this model to apply to concentrated, multi-component systems [11], [12], the solid

solution hardening in CCAs can be modeled using the approach described by the Toda-Caraballo model [13]. The steps used in this model and its application to the Nb-Ta-Ti-V system are presented in the next section.

The Toda-Caraballo model is a development of the Gypen and Deruyttere approach for modelling solid solution strengthening of multi-component systems [12], given by **Eq. 3-3**:

$$\Delta\sigma_{ss} = \left(\sum_i B_i^{\frac{2}{3}} X_i \right)^{\frac{2}{3}} \quad (3-3)$$

where $\Delta\sigma_{ss}$ is the solid solution strengthening, X_i is the atomic fraction of element i , and B_i is a temperature dependent term relating to the shear modulus μ and mismatch properties of solute atoms and is described in **Eq. 3-4**:

$$B_i = 3\mu\epsilon_i^{\frac{4}{3}}Z_0e^{-\frac{mk_bT}{W_0}} \quad (3-4)$$

The temperature dependent exponential term contains the temperature T as well as the Boltzmann constant k_b , a constant $m = 25$, and the binding energy of an edge dislocation segment with solute atoms W_0 . W_0 is reported to have a value of around 1×10^{-19} J for many binary systems [13] which was used for calculations in this work. The fitting constant Z_0 was fit using experimental data from this work and Refs. [19], [21] and applied as the same constant for all materials as a first approximation. The mismatch parameter ϵ_i for element i is defined by **Eq. 3-5**:

$$\epsilon_i = \sqrt{\eta_i'^2 + \alpha^2 \delta_i^2} \quad (3-5)$$

where α is a constant that changes from 3 to 16 depending on the dislocation character. This work used $\alpha = 9$ when applying this model. The misfit terms η'_i (elastic misfit) and δ_i (atomic size misfit) are defined in **Eq. 3-6** and **Eq. 3-7**:

$$\eta'_i = \frac{\eta_i}{1 + |\eta_i|}; \eta_i = \frac{\mu_i - \mu_{CCA}}{\mu_i + \mu_{CCA}} \quad (3-6)$$

$$\delta_i = \frac{ds}{dX_i} \frac{1}{s} \quad (3-7)$$

The shear modulus for CCAs has been shown to vary linearly with composition, so μ_{CCA} was calculated using the atomic fraction rule of mixtures. However, the average interatomic distance s cannot be determined this simply due to the fact that it is difficult to differentiate between a “solute” and a “solvent” in a CCA system. Instead, s can be calculated using data given interatomic distances for each distinct element pair in the CCA, shown in **Eq. 3-8**:

$$s = \sum_i \sum_j s_{ij} X_i X_j = [X_1 \quad \dots \quad X_n] \begin{bmatrix} s_{11} & \cdots & s_{1n} \\ \vdots & \ddots & \vdots \\ s_{n1} & \cdots & s_{nn} \end{bmatrix} \begin{bmatrix} X_1 \\ \vdots \\ X_n \end{bmatrix} \quad (3-8)$$

where X_i and X_j are the atomic fractions of elements i and j , and the matrix s_{ij} contains each interatomic distance between element pairs i and j . The Toda-Caraballo model also finds that the change in s with respect to X for each component is approximately linear when modelling a pseudo-binary CCA system. Because of this, ds/dX_i can be simplified to the difference between the average interatomic distance of the pure element s_{ii} and the average interatomic distance of the CCA that excludes element i . This interatomic distance follows the same calculation as **Eq. 3-8**, but the entries in the atomic fraction and interatomic distance matrices that contain element i are omitted, and the atomic fractions are normalized by $1 - X_i$. An example of this calculation is provided in **Eq. 3-9** for element $i = 1$:

$$\frac{ds}{dX_1} = s_{11} - \frac{X_{2:n}}{1 - X_1} \begin{bmatrix} s_{22} & \cdots & s_{2n} \\ \vdots & \ddots & \vdots \\ s_{n2} & \cdots & s_{nn} \end{bmatrix} \frac{X'_{2:n}}{1 - X_1} \quad (3-9)$$

where $X_{2:n} = [X_2 \quad \cdots \quad X_n]$ and $X'_{2:n}$ is its transpose.

With all of these calculated terms, the solid solution strengthening of compositions in the Nb-Ta-Ti-V system can be modelled by **Eq. 3-3** using the shear moduli of BCC pure elemental components and interatomic distances reported in Ref. [15]. **Fig. 3-5** compares experimental yield strengths from various Nb-Ta-Ti-V system compositions [19], [21] with the calculated solid solution strengthening using the Toda-Caraballo model, all at 298 K. This model seems to overestimate the strength for the pseudo-binary $(\text{NbTaTi})_{1-x}\text{V}_x$ at 16 at% V and below, whereas the strength is underestimated for compositions with greater than 20 at% V. Notably, the computed strength for $\text{Nb}_{0.45}\text{Ta}_{0.15}\text{Ti}_{0.20}\text{V}_{0.20}$ seems significantly underestimated. This is possibly because of the relatively low fraction of Ta. Since Ta has a much higher shear modulus than the other three components, lower Ta composition likely decreases the modulus misfit term in the Toda-Caraballo model. Surprisingly, the yield strength for NbV is slightly overestimated by the model, in this case, but this can be attributed to the fitting parameter Z_0 essentially averaging the error across all six experimental data points. This means that the higher than expected strength of $\text{Nb}_{0.45}\text{Ta}_{0.15}\text{Ti}_{0.20}\text{V}_{0.20}$ and NbTaTiV in experimental could be modifying this fit. Again, this could possibly be caused by strengthening by oxygen, but this cannot be confirmed as oxygen levels for the NbV alloy have not been explored in this work. Overall, this model still provides a solid prediction for the contribution of lattice distortion and modulus misfit in the Nb-Ta-Ti-V system.

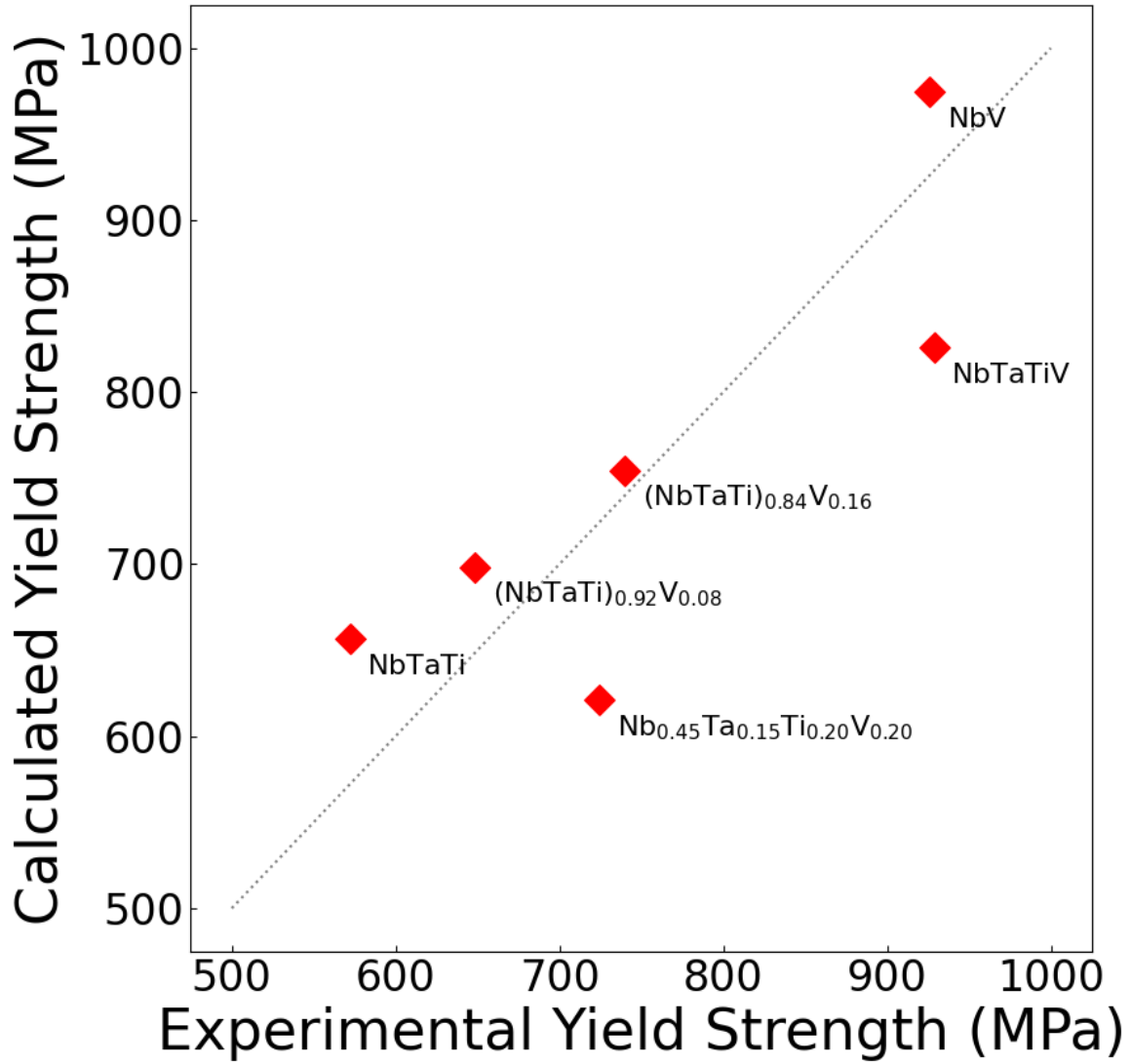


Figure 3-5: Comparison between calculated strengthening from Toda-Caraballo model and experimental yield strength of various Nb-Ta-Ti-V system alloys at 298 K [19] [21].

Regarding the mismatch terms from the Toda-Caraballo model, the relative contribution of the modulus misfit η and atomic size misfit δ terms can be qualitatively compared by setting one of the terms to 0. **Table 3-2** displays the relative contribution of each of these misfit terms normalized by the calculated solid solution strengthening for each of the modeled compositions at 298 K.

Setting $\eta = 0$ gives the solid solution strengthening if there was only contribution from atomic size misfit. Since atomic size misfit is a defining factor in lattice distortion, this strengthening can be attributed to the effects of lattice distortion. Setting $\delta = 0$ gives the solid solution strengthening if there was only contribution from the elastic modulus misfit. Comparing the strengthening from each misfit term is only qualitative and relative since **Eq. 3-5** is nonlinear, so the sum of the two terms does not necessarily equal 1. NbTaTi shows the lowest relative strengthening from lattice distortion at 0.0448, which is expected due to it lacking V. In this case, all of the constituent elements have roughly equal atomic sizes, providing little lattice distortion. Thus, most of the strengthening comes from the modulus misfit in NbTaTi. On the other extreme, NbV displays the greatest relative strengthening from lattice distortion, at 0.9524. This result is also expected, as this composition essentially maximizes the lattice distortion as shown in **Fig. 3-4**. In general, these strengthening contributions do show a trend. Increasing the concentration of V generally increases the relative strengthening attributed to lattice distortion while decreasing the relative strengthening from the modulus misfit. Deviation from the trend occurs with Nb_{0.45}Ta_{0.15}Ti_{0.20}V_{0.20} as it has a lower V concentration than NbTaTiV, but its relative strengthening from lattice distortion is higher. This is likely caused by the nominal value of the modulus misfit strengthening being much lower, as well as the overall strengthening predicted by the model. As mentioned before, the relatively low concentration of high shear modulus Ta may be greatly hindering the calculated total solid solution strengthening of Nb_{0.45}Ta_{0.15}Ti_{0.20}V_{0.20} as shown by its abnormally low quantity in **Table 3-5**. Overall, the Toda-Caraballo model provides a reasonable prediction of the strength of these Nb-Ta-Ti-V alloys, but these results emphasize the importance to consider other factors beyond modulus misfit and lattice distortion.

Table 3-2: Solid solution strengthening calculated by the Toda-Caraballo model when either modulus misfit or atomic size misfit are set to 0, normalized by total calculated solid solution strengthening. Calculated values of solid solution strengthening are also listed.

Composition	$\Delta\sigma_{ss}(\eta = 0)/\Delta\sigma_{ss}$	$\Delta\sigma_{ss}(\delta = 0)/\Delta\sigma_{ss}$	$\Delta\sigma_{ss}$ (MPa)
NbTaTi	0.0448	0.9937	656.9
(NbTaTi) _{0.92} V _{0.08}	0.3208	0.87486	698.2
(NbTaTi) _{0.84} V _{0.16}	0.4924	0.7538	754.3
NbTaTiV	0.6295	0.6303	826.2
Nb _{0.45} Ta _{0.15} Ti _{0.20} V _{0.20}	0.6538	0.6056	620.9
NbV	0.9524	0.1707	974.5

3.3.4 Lattice distortion and creep properties

Although the increased lattice distortion of NbV is correlated with a decrease in the steady state creep rate compared to NbTaTi and NbTaTiV, the magnitude of this decrease is surprising. From **Fig. 3-2a**, the most conservative case of the decrease in creep rate from NbTaTiV to NbV is at 100 MPa, from 4.7×10^{-7} to $2.0 \times 10^{-8} \text{ s}^{-1}$, which is more than a full order of magnitude lower. From **Fig. 3-4**, the lattice distortion increases from ~ 0.13 to $\sim 0.16 \text{ \AA}$ for NbTaTiV to NbV, which is about a 23% increase. On the other hand, the increase in lattice distortion from NbTaTi to NbTaTiV is from ~ 0.03 to $\sim 0.13 \text{ \AA}$, which is about a 300% increase. If lattice distortion were the controlling factor of the creep rate, then the decrease in creep rate from NbTaTi to NbTaTiV should be substantial, even larger than that from NbTaTiV to NbV. However, this is clearly not the case. From **Fig. 3-2a**, the creep rate for NbTaTi and NbTaTiV at 100 MPa decreases from 1.2×10^{-6} to $4.7 \times 10^{-7} \text{ s}^{-1}$, which is a decrease less than a full order of magnitude. This indicates that lattice distortion is likely not the factor dominating the creep rate at 1173 K.

One of the possible factors that could be dominating the creep rate is the concentration of the group IV elements (Ti, Zr, Hf). At elevated temperatures, the BCC phases of these elements are

associated with exceptionally fast diffusion [31]. As mentioned previously, the stress exponents of the BCC CCAs in this report are all $n \approx 3$, which is associated with solute drag creep. This is a diffusion rate limited mechanism, so it is possible that the fastest diffusing species could be controlling the creep rate. **Fig 3-6** displays the steady state creep rates of various CCAs at 1173 K and 100 MPa of applied stress as a function of the atomic fraction of group IV elements in their compositions. The sizes of the data points are relatively scaled by their compositions' δ parameters, which is associated with lattice distortion [32]. The δ parameter is a simpler measure of the atomic size misfit compared to **Eq. 3-1** and **Eq. 3-7**, and it was used in this case due to minimal data surrounding interatomic distances of the unlike atom pairs at 1173 K. The δ parameter is calculated by **Eq. 3-10**:

$$\delta = \sqrt{\sum_{i=1}^N X_i \left(1 - \frac{a_i}{\bar{a}}\right)^2} \quad (3-10)$$

where X_i is the atomic fraction of the element i , a_i is the lattice parameter of element i , and $\bar{a}$ is the atomic fraction weighted rule-of-mixtures average lattice parameter of the alloy [32]. **Fig. 3-6** shows a trend where increasing the group IV content increases the creep rate. Simultaneously, it also demonstrates that the lattice distortion is not the controlling factor for creep rate, since the sizes of the points shows less correlation with the creep rate. Thus, it can be inferred that the group IV content is a more dominant factor contributing to the creep rate of CCAs compared to lattice distortion.

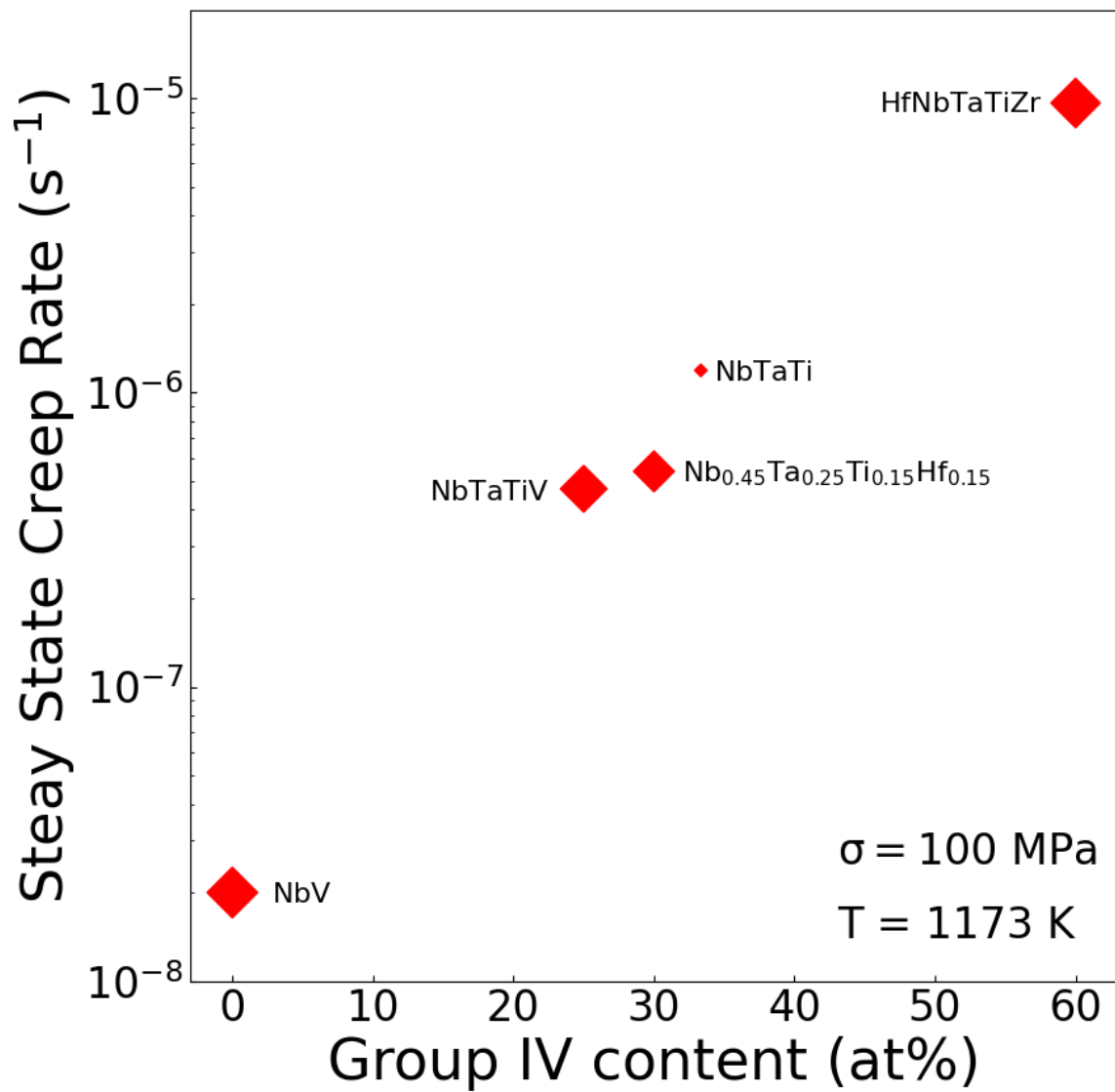


Figure 3-6: Steady state creep rates of various CCAs at 1173 K and applied stress of 100 MPa [20], [33], [34]. Steady state creep rate of HfNbTaTiZr was extrapolated to 1173 K using creep activation energy from Ref. [33]. The size of each data point are scaled by each composition's relative δ parameter as a representation of lattice distortion, calculated by Eq. 3-10 [32].

3.4 Conclusions

At the current stage of this work, the following conclusions can be drawn:

1. The increased lattice distortion of the NbV alloy is associated with a decrease in minimum creep rate at 1173 K. From NbTaTiV to NbV, the lattice distortion increases by $\sim 23\%$, but the creep rate decreases by one to two orders of magnitude. On the other hand, from NbTaTi to NbTaTiV, the lattice distortion increases by $\sim 300\%$, but the creep rate minimally decreases. The lattice distortion effect does not appear to dominate the creep rate. Instead, creep rates are better correlated with group IV content, where higher fractions of group IV elements is correlated with higher creep rates.
2. The experimental yield strength of NbV does not exceed that of NbTaTiV despite a greater lattice distortion. However, the Toda-Caraballo model provides a reasonable prediction for the yield strength of the Nb-Ta-Ti-V system at 298 K, but overestimates strength for V content at and below 16 at% and underestimates strength for V content at and above 20 at%. The exception to this is NbV, which is overestimated for this 50 at% V composition.

3.5 References

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Chapter 4. Summary and Future Work

Both of these works indicate that strong lattice distortion is a major factor contributing to both the creep properties and strength of RCCAs in the Nb-Ta-Ti-V system. Lattice distortion induced by V is correlated with a reduction in steady state creep rate, up to a whole order of magnitude when maximized, as shown by NbV. However, these results also show that lattice distortion may not be the dominant factor controlling the creep rate for these RCCAs. These creep results are also associated with a transition in dislocation behavior, where the V-free alloy displays strictly screw dislocation control while the V-containing alloy displays a network of curvy mixed dislocations. Additionally, the unintentional secondary phase formation found with NbTaTi and NbTaTiV under creep conditions actually yielded an additional improvement to the creep resistance of these alloys. This indicates that secondary phase strengthening may be a necessary consideration when designing creep resistant RCCAs. Lattice distortion also greatly affects the solid solution strengthening effect in the Nb-Ta-Ti-V system. Even when isolated to just the NbV binary, the lattice distortion plays a major role in its overall strength at room temperature via the Toda-Caraballo model. Despite the theoretical strengthening from high lattice distortion, the experimental results have shown no improvement in yield strength from NbTaTiV to NbV. The results of these works help contribute to knowledge surrounding strengthening and creep properties of RCCAs, thus helping inform design of RCCAs that can outperform current Ni based superalloys.

The immediate next steps for this work include completing experimental creep and tensile tests for the NbV alloy. These data will build towards a clearer picture of how lattice distortion affects the tensile and creep properties of RCCAs. One interesting phenomenon that is yet to be seen is the yield strength plateau of the NbV alloy, and this can be identified with a more extensive tensile

testing temperature range. The effect of lattice distortion on this plateau is of interest for higher temperature applications of RCCAs. The high temperature tensile tests can also reveal at what temperature range that lattice distortion is a dominant strengthening factor, and at what range other factors may take over. Additionally, more in depth characterization of the deformation microstructures for NbV under creep and tensile testing is required. With SEM and TEM techniques, further analysis can be performed on the tested samples in order to evaluate phase stability and potentially correlate lattice distortion with edge dislocation controlled behavior. This planned work will contribute to a more thorough investigation of the lattice distortion effect in the NbV alloy.

Potential future avenues for focused research in the RCCA field includes other strengthening mechanisms. The works in this thesis mainly investigated how lattice distortion affects the creep resistance and tensile strength of RCCAs, but other factors such as modulus misfit and secondary phase strengthening can be of great interest. As noted in the solid solution strengthening models, the shear modulus and the modulus misfit term also greatly affect the strength of RCCAs. Like NbV, a binary alloy composition that maximizes the modulus misfit while decreasing the effect of lattice distortion, such as NbTa, could be investigated to determine whether the modulus controls yield strength or creep resistance. Alternatively, the effects of secondary phase strengthening can be explored. Although unintentional in this work, the Ti- and N- rich secondary phase found in NbTaTi and NbTaTiV did contribute to improved creep resistance. Thus, it is reasonable to consider intentionally designing RCCAs with high temperature secondary phases in mind to make their creep rates more comparable or even exceed that of Ni-based superalloys.