**February 5, 2003**

## Tensile Creep of Polycrystalline Near-Stoichiometric NiAl

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### ABSTRACT

Long term tensile creep studies were conducted on binary NiAl in the temperature range 700-1200 K with the objectives of characterizing and understanding the creep mechanisms. Inverse and normal primary creep curves were observed depending on stress and temperature. It is concluded that the primary creep of NiAl is limited by dislocation mobility.  The stress exponent for creep, n, decreased from 13.9 at 700 K to 5.5 at 1200 K.  The true activation energy for creep, $Q_c$, was constant and equal to about 400 kJ mol$^{-1}$ between 20 and 50 MPa but decreased to a constant value of 250 kJ mol$^{-1}$ between 50 and 110 MPa. The activation energy was observed to be stress dependent above 110 MPa. The tensile creep results reported in this investigation were compared with compression creep data reported in the literature. A detailed discussion of the probable dislocation creep mechanisms governing compressive and tensile creep of NiAl is presented. It is concluded that the non-conservative motion of jogs on screw dislocations influenced the nature of the primary creep curves, where the climb of these jogs involves either the next nearest neighbor or the six-jump cycle vacancy diffusion mechanism.  A phenomenological model discusses the nature of the atom-vacancy exchange process likely to lead to the climb of these jogs.

### Keywords

NiAl, tensile creep, high temperature deformation, activation energy, primary creep

## I. INTRODUCTION

Over the last several decades, a considerable amount of research has been conducted on developing NiAl-based alloys for use in gas-turbine aircraft engines. An attractive combination of oxidation, physical and thermal properties make NiAl[1] a potentially useful material for fabricating turbine blades to withstand temperatures above 1200 K. However, the inherent low temperature brittleness of the material combined with its poor elevated temperature creep properties has prevented its development as rotating and stationary hardware in aircraft engines.

A fundamental understanding of the creep mechanisms dominant in NiAl is important in order to develop methods to improve its high temperature creep strength. A review of the literature reveals that most of the investigations conducted on polycrystalline binary NiAl at elevated temperatures were in compression both under constant strain rate [1,2,3,4,5] and under constant load or stress [6,7,8,9,10,11,12,13,14,15,16,17] conditions. Except for one reference source [18], virtually no tensile creep data exist on polycrystalline NiAl. Even in this case, the published data were scant and no details were provided on composition, processing conditions, grain size and activation energy for creep. Although some tensile creep data have been reported on near-stoichiometric NiAl single crystals [19,20], other single crystal data have been determined in either constant strain rate [21] or constant load [22,23] compression tests. The importance of obtaining tensile creep data on polycrystalline NiAl is that they are not only useful for comparing with the compression creep data to ensure that its creep properties are independent of the macroscopic state of stress but also for establishing a database for alloy and structural design purposes.

The present study was motivated with the intention of fulfilling three objectives: First, to generate long term tensile creep data on polycrystalline NiAl over a wide range of stresses and temperatures; second, to characterize and evaluate the creep mechanisms dominant under these test conditions; and third, to compare the tensile and compression creep data to identify the similarities and differences between the two sets of results. The present paper expounds on some of the preliminary results presented in earlier publications [24,25].

## II.  EXPERIMENTAL MATERIALS AND PROCEDURES

The materials used in this study were obtained from two heats of induction melted NiAl, where a high purity charge of Al and Ni pellets were melted in an alumina crucible to produce two nominally stoichiometric NiAl ingots (~ 1000 g). The molten alloy was cast into split copper molds about 25.4 mm in diameter and 150 mm long and allowed to cool to room temperature. After cropping their hot-tops, the two ingots were vacuum-sealed in mild steel cans and extruded at 1400 K using ratios of 16:1 (L 3175) and 20:1 (L 3176). The compositions and grain sizes of the two extruded rods of material are given in Table 1, where it is evident that the choice of these two extrusion ratios did not significantly alter the final grain sizes of the extruded rods[2]. The major elements were determined by inductively-coupled plasma (ICP) analysis, while carbon was analyzed by the combustion extraction method. Nitrogen and oxygen were determined by the inert gas fusion technique. Optical microscopy observations and quantitative measurements of the longitudinal and transverse grain sizes, d, of the extruded rods revealed that the grains were equiaxed and fully recrystallized (Fig. 1). Grain size measurements were conducted by

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[1] All compositions are given in atomic percent in the paper unless otherwise specified.
[2] Although batch L 3176 had a significantly larger amount of carbon than batch L 3175, its effect could not be separated from normal experimental scatter in the creep data.

linear intercept analysis. The mean values of the grain size are reported in Table 1, where the errors in measurement represent the 95% confidence limits.

Smooth button-head tensile specimens having gage lengths of 30.5 mm and gage diameters of 3.0 mm were centerless ground from the extruded rods (Fig. 2). Specimens with ridges for attaching the creep extensometer at the gage length were also used in a few tests to determine the primary creep strain accurately. The machined surfaces were removed by electropolishing in a 10% perchloric acid-90% methanol bath prior to testing. Constant load tensile creep tests were conducted in air using lever arm machines at absolute temperatures, T, between 700 and 1200 K under initial applied stresses, $\sigma_e$, varying between 10 and 170 MPa. Most of the specimens tested in the temperature range 900-1200 K were machined from extrusion L 3176, whereas all the specimens tested at 700 and 800 K were obtained from L 3175. Three thermocouples were attached along the gage length of the specimen and the accuracy of temperature control and the temperature gradient along the gage length were ± 1 K. The specimen load train was allowed to stabilize at temperature for at least 1 h before loading. The elongation of the specimen corresponding to the design shown in Fig. 2 was measured by attaching an extensometer to the top and bottom couplings in most of the tests, where the displacement of the extensometer heads was measured either by a linear variable displacement transformer (LVDT) or a super linear variable capacitance (SLVC) transducer. The elongation and temperature data were continuously monitored by a computerized data acquisition system. Most of the specimens were tested until rupture except in the case of a few long-term tests, which were terminated prior to rupture. In order to assess the nature of the transients after a stress change, a specimen was crept at 900 K, where the engineering stress was changed between

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40 and 50 MPa until rupture. The tested specimens were longitudinally sectioned and polished parallel to the stress axis for viewing under an optical microscope. The final grain sizes, $d_f$, were determined both in the undeformed shoulder and in the deformed longitudinal gage sections in a few specimens away from the fracture zone in order to evaluate the extent of grain growth during the test.

## III. RESULTS

*The shape of the creep curves*

Figures 3(a-c) show the typical tensile creep curves for NiAl, plotted as the true creep strain, $\varepsilon$, against time, t, in the temperature range 700 to 1200 K under initial applied stresses varying between 10 and 170 MPa. The corresponding plots of the true creep rate, $\dot{\varepsilon}$, against $\varepsilon$ are shown in Figs 4 (a-c), where the measured creep rates typically vary between $10^{-10}$ and $10^{-4}$ s$^{-1}$. Since the data were obtained under constant load conditions, the creep rate increases gradually with increasing strain under quasi-steady-state conditions due to the fact that the specimen cross-sectional area continues to decrease and the true stress continues to increase during deformation (Figs 4(a-c)). It is important to note that the minimum creep rates shown in Fig. 4 do not always represent steady-state values. Unlike pure metals, where steady-state deformation can occur typically after $\varepsilon > 10\%$, polycrystalline NiAl exhibits quasi-steady-state behavior when $\varepsilon > 2\%$.

At 700 K, the creep rate decreases soon after loading, where this initial decrease is steep at 100 and 120 MPa but relatively gradual at higher stresses (Fig. 4(a)). Two distinct shapes are noticeable in the primary transients depending on the magnitudes of the stress. Inverse primary creep occurs at and below 135 MPa, whereas normal primary creep transient is observed at higher stresses (Figs. 3(a) & 4(a)). Similar creep curves are also observed at 900 K, where the

transition from inverse to normal primary creep occurs above 70 MPa. At 1200 K, except for the specimen deformed at 13 MPa, which exhibits an apparent inverse transient behavior, only normal primary creep behavior is observed. Inverse and normal primary creep transients were also observed in both as-cast [6,14] and powder-metallurgy extruded NiAl specimens tested in compression [11,13]. In contrast to the inverse and normal primary creep behavior shown in Figs 3 & 4, the specimen deformed at 1200 K under an initial stress of 10 MPa underwent several stages of normal primary transient behavior over 9100 h (Fig. 5). Similar primary curves have been observed during creep of Pb and these have been attributed to dynamic recrystallization [26]. However, long term tests conducted at other stresses and temperatures, while revealing inverse or normal primary creep transients, did not show similar multiple normal primary creep transient behavior (Fig. 5). The final grain sizes of several specimens from these long term experiments are shown in Table 2 along with the testing conditions, where it is clear that there was considerable grain growth during the duration of these tests. An analysis of the data revealed that the final grain size was given by the relation

$$(d_f/d) = 17.2 \, t_r^{0.14} \sigma_e^{0.94} \exp(-46.0/RT) \quad (R_d^2 = 0.945) \tag{1}$$

where $t_r$ is either the rupture life or the time at which the test was stopped, R is the universal gas constant, $R_d^2$ is the coefficient of determination, and the pre-exponential constant and the activation energy for grain growth are in units of $h^{-0.14} MPa^{-0.94}$ and kJ mol$^{-1}$, respectively.

Figures 6(a-c) show the shape of the creep transients at 900 K after a series of engineering stress changes varying between 40 and 50 MPa. The true creep strain is plotted against time in Fig. 6(a), while the variation of the true creep rate with either time or true strain is

shown in Figs. 6(b) & (c), respectively. The magnitudes of the true stresses, $\sigma$, before and after each stress change are also shown in Figs. 6(b) & (c). Prior to the first stress change from $\sigma_e = 40$ to 50 MPa, the magnitude of the true creep strain was extremely small at 40 MPa even after 300 h and the shape of the primary creep curve could not be accurately discerned (Fig. 6(a)& (c)). Although the creep rates could not be determined with any degree of precision, it was estimated that they would be between $10^{-10}$ and $10^{-9}$ s$^{-1}$. On increasing the stress to 50 MPa, the creep rate initially increases from about 7.8 x $10^{-9}$ s$^{-1}$ at $\sigma = 40.2$ MPa to about 4.5 x $10^{-8}$ s$^{-1}$ for $\sigma = 50.2$ MPa exhibiting a trend characteristic of class A behavior in many solid solution alloys (Figs. 6(a-c)). As demonstrated in Fig. 4(b), inverse primary creep occurs at a similar value of stress and the creep transient observed in Figs. 6(a-c) is consistent with the expected trend. On decreasing the stress from 50 to 40 MPa, the transient creep behavior is now similar to the class M response reported for pure metals [27], where the creep rate first decreases to a minimum value for about 14 h after the load drop (Fig. 6(c)) before increasing with increasing strain (Fig. 6(b & c)). In this case, the true stress following the stress drop was 49.2 MPa. This observation is contrary to the class A transient response that would be normally expected under conditions when inverse primary creep is observed (Fig. 4(b)). A third stress change from 40 to 50 MPa confirms the occurrence of class M or pure metal behavior, where the creep rate continues to decrease from an initially high value after the stress increase as deformation progresses. In this case, the creep rate is higher than the extrapolated creep rate observed after the first stress increase by over a factor of five due to an increase in the magnitude of the true stress from a value of 64.6 MPa soon after the stress increase to a final value of 75.7 MPa. Noting that the true stress soon after the stress increase is 64.6 MPa, a class A transient behavior is expected to occur at this value of stress

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based on the fact that inverse primary creep is observed between 60 and 70 MPa in Fig. 4(b). However, this behavior is not observed in Fig. 6, where instead a class M transient is observed.

*Stress dependence of the secondary creep rate*

Since the creep rates increased gradually with increasing strain in the quasi-steady-state region in the $\dot{\varepsilon}$ - ε plots, it was not possible to determine a unique steady-state creep rate from these plots. Instead, the secondary creep rates were determined by linearly regressing the ε - t data over the quasi-steady-state region. Figure 7 shows the double logarithmic plots of $\dot{\varepsilon}$ against σ between 700 and 1200 K under stresses varying between 10 and 170 MPa, where the secondary creep rates vary between $10^{-10}$ and $10^{-4}$ $s^{-1}$. In this case, σ is the average value of the true stress determined over the same region of the ε - t plot which yielded the secondary creep rate. The single datum point shown at 1200 K under an initial stress of 10 MPa, which underwent dynamic recrystallization (Fig. 5), was not considered in the regression analysis since it lies close to the datum point at 1100 K under an identical initial stress. The regression lines through the data are also shown assuming the power-law relation:

$$\dot{\varepsilon} = A_1 \, \sigma^n \tag{2}$$

where $A_1$ is a microstructure and temperature dependent constant and n is the creep stress exponent. The magnitudes of $A_1$, n and $R^2_d$, are tabulated in Table 3, where it is seen that n decreases from 13.9 at 700 K to 4.9 at 1200 K. The data at 1000 K exhibit a break in the $\dot{\varepsilon}$-σ plot, where n is 5.1 below 35 MPa and 12.3 above this value of stress.

*Temperature dependence of the secondary creep rate*

Figure 8 shows Arrhenius plots of $\dot{\varepsilon}$T/E against 1/T, where E is the Young's modulus, for different $\sigma$ varying between 15 and 150 MPa corresponding approximately to normalized stresses, $\sigma$/E, varying between 8.9 x 10$^{-5}$ and 7.6 x 10$^{-4}$. Since $\dot{\varepsilon}$ for the specimen deformed at 1200 K under an applied stress of 10 MPa is closer to the 1100 K data in Fig. 7, it was not possible to accurately determine $Q_c$ at stresses lower than 15 MPa. The creep rates were normalized by the factor (T/E) in accordance with the semi-empirical universal power-law creep equation proposed by Mukherjee *et al*. [28], where the temperature dependence of E was determined from the equation

$$E = 2.37 \text{ x } 10^5 - 51T \quad \text{(MPa)} \tag{3}$$

obtained from a linear regression fit to experimental data, where T is K [29]. The data shown in Fig. 8 were primarily determined from multiple specimen tests under identical stresses. However, in some instances, additional temperatures were included in order to improve the statistical estimate of $Q_c$, where these data were estimated from the regression constants given in Table 3.

The magnitudes of the true activation energies for creep, $Q_c$, determined from the slopes of the Arrhenius plots shown in Fig. 8 are given in Table 4 at different values of the applied stress and plotted in Fig. 9. The broken horizontal line represents the activation energy for lattice self-diffusion of Ni, $Q^*_{Ni}$, in near-stoichiometric NiAl based on tracer diffusion measurements, which have been determined to be about 305 [30] and 290 kJ mol$^{-1}$ [31]. Four deformation regimes are observed in Fig. 9, where the boundaries between regimes I and II, and II and III, are

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arbitrarily marked as the stresses at which $Q_c = 290$ kJ mol$^{-1}$. In regime I, the activation energy is less than 290 kJ mol$^{-1}$, presumably tending towards a constant value of 100 kJ mol$^{-1}$ as reported earlier for powder-metallurgy (PM) extruded NiAl tested in compression creep [11,13]. In this case, a corresponding value of $n \sim 2$ was observed and it was concluded that a grain boundary sliding mechanism was dominant in this regime. The activation energy for creep is approximately independent of stress in regime II with an average value of about 400 kJ mol$^{-1}$ determined from the two datum points for which $Q_c > 385$ kJ mol$^{-1}$. This value is much higher than that reported for PM-extruded NiAl tested in constant load compression for which $Q_c \approx 290$ kJ mol$^{-1}$ [13] and for cast and extruded NiAl, where $Q_c \approx 300$ kJ mol$^{-1}$ [14]. It is also higher than $Q^*_{Ni}$ as well as the values of the activation energies for interdiffusion in stoichiometric NiAl, which have been measured to be about 195 kJ mol$^{-1}$ [32] and about 355 kJ mol$^{-1}$ [33]. For comparison, an activation energy of about 360 kJ mol$^{-1}$ was determined from interdiffusion measurements on Ni-48(at.%)Al [34]. The activation energy for creep in regime III decreases to a constant value of about 250 kJ mol$^{-1}$, which is about 0.86 $Q^*_{Ni}$ and about 0.63 $Q_c$ observed in regime II.

The activation energy for creep decreases linearly with increasing stress in regime IV. A similar decrease in activation energy with increasing stress [1,7] or normalized stress [13] was reported in other investigations on the compressive creep of polycrystalline NiAl. A linear regression analysis through three datum points suggested that

$$Q_c = 442.2 - 1.75\, \sigma \quad (R^2_d = 0.952) \tag{4}$$

where $Q_c$ and $\sigma$ are in kJ mol$^{-1}$ and MPa, respectively.

When the stress dependence of the entropy term in the rate equation is negligible, the apparent activation volume for creep, $V^*$, can be estimated from eqn. (4) using [35,36],

$$V^* = -2 \ (\partial Q_c / \partial \sigma)_T \tag{5}$$

assuming that $\sigma = 2\tau$, where $\tau$ is the shear stress. Thus, $V^* \approx 245 \ b^3$, where b is the Burgers vector ($\mathbf{b} = 0.2876$ nm). This value is within the same order of magnitude as other values of 75-350 $b^3$ reported in the literature [1,13]. Similarly, Xu and Arsenault [5] reported values of $V^*$ varying between 50 and 250 $b^3$ for binary NiAl determined from constant engineering strain rate tests.

Alternatively, instead of the linear fit to the three data points described by eqn. (4), other regression fits assuming a $\sigma^{1/2}$ and $\sigma^{-1}$ dependence gave the following regression equations:

$$Q_c = 655.1 - 38.7 \ \sigma^{1/2} \quad (R^2_d = 0.939) \tag{6(a)}$$

$$Q_c = 14.2 + 25,511.7 \ \sigma^{-1} \quad (R^2_d = 0.891) \tag{6(b)}$$

where once again $Q_c$ and $\sigma$ are in kJ mol$^{-1}$ and MPa, respectively. It is important to note that some of the models formulated to describe the cross-slip of screw components of dislocations predict the stress dependence given by eqns. (4), 6(a) and (b) at low stresses [37], while eqn.(4) also represents the general formulation for other thermally activated mechanisms [35,36]. The corresponding values of $V^*$ for equations 6(a) & (b) are $38.7/\sigma^{1/2}$ and $51,023.4/\sigma^{-2}$ in the appropriate units of volume. The difference in the magnitudes of $R^2_d$ between eqns. (4) & (6(a)) is not significant enough to choose between the two types of stress dependencies given the scatter in the data.

The values of n and $Q_c$ were also determined by multiple regression analysis of the data shown in Fig. 7. In this case, creep data obtained below 40 MPa were separately regressed from those determined at and above 40 MPa, where this transition stress was chosen based on the point of transition in the low and high stress exponents observed at 1000 K (Fig. 7). Referring to Fig. 9, the data lying below 40 MPa correspond to regimes I and II, while most of the data lying above this value of stress lie within regimes III and IV. Thus, the regressed results are expected to be influenced to some extent by these transitions. The regressed equations are given below:

$$\varepsilon = 5.3 \times 10^4 \sigma^{5.9} \exp(-410.2 \pm 14.5/RT) \quad (R^2_d = 0.946) \qquad (\sigma < 40 \text{ MPa}) \qquad (7)(a)$$

$$\varepsilon = 7.6 \times 10^{-12} \sigma^{10.1} \exp(-230.9 \pm 8.6/RT) \quad (R^2_d = 0.908) \qquad (\sigma \geq 40 \text{ MPa}) \qquad (7)(b)$$

where $\varepsilon$, $\sigma$ and $Q_c$ are in $s^{-1}$, MPa and kJ mol$^{-1}$, respectively. Clearly, these values of $Q_c$ are generally consistent with those determined from Fig. 8 and shown in Fig. 9.

## IV DISCUSSION

*Primary tensile creep behavior*

An examination of the shape of the tensile creep curves between 700 and 1200 K generated under initial stresses between 10 and 170 MPa reveals three types of primary creep behavior after the initial steep drop in the creep rate soon after loading (Figs. 3-5). Normal primary creep is observed at high stresses and high temperatures, where the creep rate continues to decrease towards a quasi-steady-state value with increasing strain. At low stresses and low temperatures, inverse primary creep transients are observed as deformation continues beyond the

minimum creep rate, and the creep rate increases with increasing creep strain towards a quasi-steady-state behavior. A third type of creep behavior involving a series of multiple normal primary creep transients was also observed at 1200 K under an initial applied stress of 10 MPa (Fig. 5). This type of primary creep behavior is normally attributed to dynamic recrystallization in the literature [26].

A consistent picture emerges when the above observations are mapped out in $\sigma_e$-T space as shown in Fig. 10, where open, solid and half-solid circles represent inverse (I), normal (N) and multiple (M) normal primary transients, respectively[3]. Figure 10 clearly reveals that the observed primary transient behavior is acutely dependent on the magnitudes of stress and temperature. Inverse primary creep is observed at stresses and temperatures corresponding to the shaded region. Normal primary creep occurs at other stresses and temperatures above and below this area. The position of the lower boundary of the shaded region is ill defined due to an absence of low stress data below 1100 K and this is shown as a broken curve.  For example, under an initial stress of 40 MPa, there is a transition from inverse to normal primary creep above 1050 K in the temperature.  Similarly, at 1100 K, the present observations suggest a transition from normal to inverse and back to normal primary creep transient with increasing stress. Likewise, both Cu-30%Zn [38] as well as Fe-1.5 (at.%)Mo [39] also exhibit similar trends in the nature of the primary creep transients.

The occurrence of inverse primary creep is due to a low initial density of mobile dislocations at the start of the creep test. This situation is likely to occur when the density of

active dislocation sources is low or the mobility of dislocations is impeded in some way by such factors as high Peierls forces [40,41,42], jogs on screw dislocations [43,44,45], solute atoms [40], short range ordering [40,46], long range ordering [46] or the nucleation of sessile dislocations resulting from the decomposition of a perfect dislocation core [47]. Many solid solution alloys, such as Al-5(at.%) Mg, generally show transitions from $n \approx 5$ to $n \approx 3$ and back to $n \approx 5$ with increasing stress and decreasing temperature, where inverse and normal primary creep transients are observed under conditions when $n \approx 3$ and 5, respectively [48]. In this case, it has been suggested that the solute atoms create drag forces on the mobile dislocations in the $n \sim 3$ region so that dislocation glide is impeded and becomes slower than dislocation climb [49]. In the regions corresponding to $n \sim 5$, dislocation climb becomes the rate–controlling mechanism since it becomes slower than the viscous glide process.

Although the observations shown in Fig. 10 are consistent with the expected behavior for a solid solution alloy, such as Al-5%Mg, a close examination of Fig. 7 fails to reveal the expected transition in the stress exponent from 5 to 3 to 5. It is important to note that some solid solution alloys, such as Cu-30%Zn [38], also fail to exhibit this transition although other characteristics of class A behavior have been observed in stress change tests. However, the nature of the creep transient behavior observed after repeated stress changes from 40 to 50 MPa and back at 900 K (Fig. 6) reveals that NiAl exhibits the characteristics of class A behavior only under low deformation strains and class M behavior at large values of strain. As noted earlier, the uninterrupted tests under similar stress and temperature conditions result in inverse primary creep. These observations indicate that the inverse primary creep behavior, while due to an initial

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[3] It should be noted that although the M-type curves may be due to microstructural instability, there is no other

low mobile dislocation density, is not indicative of the typical class A response observed during steady-state creep of Al-5% Mg and other similar solid solution alloys involving solute locking of mobile dislocations in the n ~ 3 region.

*Comparisons of primary creep behavior under compression and tension loading*

Figure 11 superimposes the regions of the $\sigma_e$-T space, where inverse and normal primary creep occur under compression and tension loading for as-cast and extruded polycrystalline NiAl with similar grain sizes. In this case, the compression curves were generated on as-cast and extruded near-stoichiometric NiAl with a grain size of about 35 μm for which the з - σ data are reported elsewhere[4] [14]. The region where inverse primary creep occurs under each type of loading is depicted by the shaded area in each case. The role of cracks and cavitation on the tensile creep behavior was judged to be small for two reasons: (a) the microstructures of longitudinal sections did not generally show any significant damage outside the immediate vicinity of the fractured area [50], and (b) the observations shown in Fig. 11 relate to the initial stages of creep, where damage accumulation is expected to be negligible. An examination of Fig. 11 reveals that inverse primary creep occurs at higher stresses and higher temperatures in compression than under tension loading. This interesting and important observation suggests that the factor(s) influencing dislocation mobility in NiAl is asymmetric in nature.

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distinction between them and the single normal primary N-type curves. Hence, these two curves are treated as a single entity in this discussion.

[4] It is noted that frictional effects are expected to be negligible in these tests in part because boron nitride and Ta foils were used as a lubricant and as compliant layers, respectively, and in part because $\varepsilon < 0.05$ to reach quasi-steady-state conditions in most instances. The datum point shown at 800 K under an initial stress of 250 MPa in Fig. 11 was obtained from a constant load creep test. It is not depicted in з - σ data reported in [14].

*Examination of the role of impurities on dislocation mobility*

Two characteristics of the transition from inverse to normal primary creep behavior should be noted. First, as shown in Figs 4 and 10, the nature of this transition is stress dependent at a constant temperature. The effect of increasing the stress is to activate a larger number of dislocation sources, increase the density of mobile and immobile dislocations as well as lower the activation energy for thermally-activated dislocation motion. Second, the fact that the inverse primary creep behavior is only observed in an intermediate stress and temperature regime (Fig. 10) indicates that the process is thermally activated and inconsistent with the operation of an athermal process, such as dislocation nucleation. A close examination of Figs. 4 reveals that there is a sharp drop in the creep rate soon after loading due to rapid work hardening with no significant recovery. This observation indicates that most of the dislocations that were generated at the commencement of creep were immobilized within a short period of time at these stresses and temperatures. Creep proceeds only when the mobility of the dislocations increases with time as recovery mechanisms become more predominant. As a result, it must be concluded that the observation of inverse primary creep is due to factors influencing dislocation mobility rather than those affecting dislocation generation.

As discussed earlier, dislocation mobility can be inhibited by the presence of interstitial or substitutional atoms. It is evident from Table 1 that the carbon content of the extrusion L 3176 was three times more than that of L 3175. The amounts of substitutional impurities were determined to be insignificant in these two batches of material to be of relevance in influencing creep behavior. The nitrogen, oxygen and sulfur contents of the two extrusions were similar, and therefore, the role of these elements cannot be unambiguously separated. As mentioned earlier,

all the specimens tested at 700 and 800 K were obtained from L 3175, whereas those deformed at 900K and above were mainly machined from L 3176. However, in the few cases where specimens from both extrusions were crept at identical temperatures with a relatively small variation in the magnitude of the applied stress, no significant difference was observed other than the fact that the nature of the primary creep behavior was essentially dependent on stress and temperature. Also, Prakash and Pool [51] did not observe inverse primary creep in carbon-doped NiAl containing up to 0.2 at.% of C in the temperature range 973 to 1173 K under an initial compressive stress of 74.5 MPa. Instead, only normal primary creep was observed in all instances. Indirect evidence that carbon does not influence the shape of the primary creep curve is evident in other reports. For example, a comparison of the compressive creep curve for cast and extruded NiAl (d ≈ 35 μm) with that for powder metallurgy (PM) and extruded NiAl (d ≈ 23 μm) crept at 1100 K under an initial stress of 40 MPa revealed, that while the PM material exhibited inverse primary creep transient, the as-cast alloy exhibited normal primary creep [14]. In this case, the carbon contents of the PM and the as-cast alloys were 110 and 350 at. ppm, respectively. Based on these arguments, it appears highly unlikely that inverse primary creep in NiAl is caused by impurity or substitutional atoms.

*Examination of other factors inhibiting dislocation mobility*

Factors other than impurity and solute atoms can influence dislocation mobility. These include the existence of high Peierls forces [40,41,42], locking of dislocation sources due to the presence of sessile decomposed edge dislocation components [47], dislocation jogs on screw dislocations [43,44,45] and dislocation interaction with "extrinsic obstacles" [43,44,45]. Recent microstructural observations suggest that the Peierls forces are small in NiAl above 143 K

[43,44,45], so that dislocation mobility is unlikely to be limited by these forces in this alloy. Alternatively, it has been suggested that inverse primary creep in NiAl is due to the formation of two <010> sessile edge dislocations from the decomposition of the core of perfect <011> edge oriented dislocations inhibits dislocation glide mobility and locks the dislocations [47]. The unlocking mechanism involves the cooperative climb of the two <010> edge segments at high temperatures with the climb of one segment leading to vacancy generation while the climb of the other leading to vacancy annihilation.

However, this coupled climb of the two <010> edge dislocations and its sequential nature presents a serious difficulty in rationalizing the present experimental observations. First, since this mechanism would result in the two <010> dislocations climbing away from each other as creep proceeds with a corresponding increase in the vacancy diffusion distance, it is expected that the creep rate would continue to decrease with increasing strain or time and finally become immeasurable. In reality, neither NiAl single [20] nor polycrystals (Fig. 4) show this predicted creep hardening behavior thereby suggesting that this is not a viable unlocking mechanism. Second, the above climb-controlled unlocking mechanism is likely to be independent of whether the applied stress was compressive or tensile in nature owing to the coupled nature of the process. As demonstrated in Fig. 11, the creep behavior of NiAl appears to be asymmetrical in nature and extremely sensitive to the nature of the stress state. Similarly, the observed magnitudes of $Q_c$ are close to 300 and 400 kJ mol$^{-1}$ in regime II in compression and tension tests, respectively (Fig. 12). Clearly, the coupled climb mechanism would lead to a unique value of $Q_c$ depending on the slowest diffusion species. If instead, it is assumed that the climb of these edge dislocations are decoupled, and hence independent processes, the observed differences in creep

behavior in compression and tension tests can be qualitatively rationalized. In this case, the climb of each <010> dislocation can occur with the aid of Al vacancies, $V_{Al}$, and Ni vacancies, $V_{Ni}$, diffusing to the dislocation core thereby providing a means of unlocking the dislocations.

An alternative locking mechanism proposed by Caillard *et al*. [43,44,45] based on microstructural observations in NiAl involves the locking of screw dislocations by jogs formed as segments of the same dislocation cross slips on different slip planes. At high temperatures, the climb of these jogs is likely to unlock the dislocations. This model is consistent with both the experimental observations reported here and in the literature on the high temperature creep of near-stoichiometric NiAl especially under conditions corresponding to regime III in Fig. 9 [1,7,13]. First, the experimental values of V* varying between 50 and 350 $b^3$ [1,13,5] fall within the expected range of values for the cross-slip of jogged screw dislocations [35,36]. Second, the stress dependence of the activation energy described by equation (6(a)) is consistent with models describing the cross-slip screw dislocations [37]. Third, TEM observations of crept NiAl specimens have confirmed the occurrence of cross-slip of screw dislocations [7,19] along with the observations of jogs, dipoles and prismatic loops [7,8,11,12,19]. Therefore, it is concluded that the non-conservative motion of jogs on screw dislocations proposed by Caillard *et al*. [43,44,45] provides a qualitative rationale for the occurrence of inverse primary creep in NiAl.

*Compression and tension creep asymmetry*

As shown in Fig. 11, the primary creep response of NiAl is asymmetrical in compression and tension creep. This asymmetry is also observed when the experimental values of $Q_c$ determined in the compression and tension tests in regime II are compared with each other (Fig.

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12). In this case, the horizontal line represents the value of $Q^*_{Ni} \approx 290$ kJ mol$^{-1}$ based on experimental measurements of the activation energy for tracer diffusion of Ni in stoichiometric NiAl [31]. It is clear from Fig. 12 that the values of $Q_c$ obtained in compression in regime II are similar to the experimental values of $Q^*_{Ni}$ [30,31] irrespective of the method used to process the extrusions, whereas the magnitude of $Q_c$ of about 400 kJ mol$^{-1}$ determined from the tension tests is about 100 kJ mol$^{-1}$ higher than that observed in the compression tests. However, the magnitudes of $Q_c$ vary between 250 and 275 kJ mol$^{-1}$ in regime III irrespective of loading and processing conditions although it is likely that the boundary between regimes II and III may have moved to higher stresses for the cast and extruded material crept in compression.

Figure 13 compares the $\dot{\varepsilon}$-$\sigma$ plots for as-cast and extruded near-stoichiometric NiAl crept at identical temperatures in compression [15] and tension, where the specimens had similar grain sizes. The broken and solid lines represent the regression lines through the compression and tension data, respectively. A close examination of Fig. 13 suggests two major differences in these two sets of data. First, the material creeps at significantly faster rates in tension creep than in compression creep especially at the lower temperatures, where the creep rates often vary by several orders of magnitude for T < 1000 K and $\sigma$ > 40 MPa. Some degree of convergence of the two sets of data is evident at 1000 and 1100 K when the stresses are low.  Second, n $\approx$ 5 for the compression data between 900 and 1200 K for $\sigma$ >10 MPa and only increases to about 13 at 800 K. In contrast, as noted earlier, n $\approx$ 5-6 when T $\geq$ 1000 K for $\sigma$ < 35 MPa and n > 8.5 when T $\leq$ 1000 K for $\sigma$ > 35 MPa (Table 3).

*Comparison of the activation energy for creep in regime II with diffusion results*

The magnitude of $Q_c \approx 300$ kJ mol$^{-1}$ observed in regime II during compression creep (Fig. 12) can be reasonably identified with that associated with $Q^*_{Ni}$ in NiAl [30,31]. In contrast, since the atomic volumes of Al and Ni are 1.7 x 10$^{-29}$ and 1.1 x 10$^{-29}$ m$^3$ with Al being the larger atom [52], it is reasonable to expect that the higher value of $Q_c \approx 400$ kJ mol$^{-1}$ observed in regime II during tension creep corresponds to the activation energy for lattice diffusion of Al in NiAl. However, direct measurements of the tracer diffusivity of Al, $D^*_{Al}$, in NiAl have not been reported due to the absence of a suitable radioactive tracer. Recently, Nakamura *et al.* [34] estimated the magnitude of $D^*_{Al}$ in Ni-43.4Al from the Darken-Manning relation [53,54] using Ni tracer diffusion literature data, $D^*_{Ni}$, and the measured interdiffusion coefficient, $\widetilde{D}$, in the alloy. Attempts to estimate the activation energy for tracer diffusion of Al, $Q^*_{Al}$, from published data [31,33] for near-stoichiometric NiAl in a similar manner resulted in values of $Q^*_{Al}$ varying between 355 and 430 kJ mol$^{-1}$ for $\Phi S = 0.01$ to $0.25^5$, where $\Phi$ is the thermodynamic factor and S is the vacancy wind factor. However, these calculations were unreliable in part due to uncertainties in the magnitudes of $\Phi$ and in part due to discrepancies in using experimental diffusion data from different investigations. Alternatively, $D^*_{Al}$ can be estimated directly from the relationship $D^*_{Al} = D^*_{Ni}(D_{Al}/ D_{Ni})$, where $D_{Al}$ and $D_{Ni}$ are the intrinsic diffusion coefficients of Al and Ni, respectively. Using an experimental value of $D_{Al}/ D_{Ni} \sim 0.3$ [32], and assuming it to be independent of temperature, the estimated values of $Q^*_{Al}$ were 290 and 307 kJ mol$^{-1}$ for values of $D^*_{Ni}$ reported by Frank *et al.* [31] and Hancock and McDonnell [30], respectively. These estimates of $Q^*_{Al}$ are much lower than the experimental value of $Q_c \approx 400$ kJ mol$^{-1}$, and once again, they are sensitive to variations in the diffusion data published in the literature. It is

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[5] It should be noted that values of $\Phi S > 0.25$ resulted in negative values of $D^*_{Al}$.

also worth noting that Kim and Chang [33] have suggested that the Darken-Manning equation cannot be applied to highly ordered intermetallic compounds like NiAl.

In the absence of a reliable way of estimating or measuring $Q^*_{Al}$, it is necessary to compare the creep activation energy data with theoretical estimates of the activation energies for the lattice diffusion of Al and Ni vacancies, $Q_{Al}$ and $Q_{Ni}$, respectively, in NiAl [55,56]. The experimental values of $Q_c$ observed in regimes II and III during compression [11,13,15] and tension creep are compared in Table 5 with the calculated values of activation energy for self-diffusion involving the diffusion of Al and Ni vacancies for several probable diffusion mechanisms [31,55,56]. Details of these diffusion mechanisms are discussed elsewhere [31,55,56]. An examination of Table 5 shows that the four-ring (4R) mechanism predicts very high activation energies for diffusion, which is not observed in both diffusion [30,31,32,33,34] as well as in creep experiments. Therefore, Mishin and Farkas [55] concluded that this mechanism is unimportant in diffusion in NiAl. Similarly, its importance in governing the creep of NiAl can be ruled out. The anti-structure bridge (ASB) mechanism is expected to be important only in the Ni-rich NiAl when the Ni anti-structure defects exceed 5% [31]. Thus, its importance in transferring Ni vacancies through the near-stoichiometric NiAl lattice to the dislocation core in order to aid dislocation climb during creep is expected to be insignificant. The next nearest neighbor (NNN) mechanism predicts $Q_{Al} \approx 285$ kJ mol$^{-1}$ [55,56], which is in reasonable agreement with the value of $Q_c \approx 300$ kJ mol$^{-1}$ observed for the compression creep data in regime II (Fig. 12). However, the value of $Q_{Ni} \approx 265$ kJ mol$^{-1}$ predicted for Ni diffusion by the NNN mechanism deviates from the experimental value of $Q_c \approx 300$ kJ mol$^{-1}$ by over 10%. The triple defect (TD) and the six jump cycle (6JC($V_{Ni}$)) mechanisms involving the diffusion of Ni

vacancies, which predict $Q_{Ni} \approx 300$ kJ mol$^{-1}$, also agree with $Q_c \approx 300$ kJ mol$^{-1}$ observed in compression creep tests (Fig. 12). Only the 6JC(V$_{Al}$) mechanism involving the diffusion of Al vacancies predicts a value of $Q_{Al} \approx 365$ kJ mol$^{-1}$, which is within 10% of the experimental value of $Q_c \approx 400$ kJ mol$^{-1}$ observed in regime II during tensile creep (Fig. 12). Theoretical calculations suggest that the activation volumes for diffusion are much larger for the two 6JC mechanisms than for ASB, which implies that they are extremely sensitive to hydrostatic stresses than the ASB mechanism [56,57]. Although the magnitudes of the creep activation energies as well as their sensitivity to the nature of the stress state are in reasonable agreement with the predicted diffusion mechanisms (Table 5), it is noted that a hydrostatic stress state exists only in the vicinity of the dislocation core in specimens deformed under uniaxial loading.

Based on the above discussion, it is reasonable to rule out the role of the ASB, NNN and the TD mechanisms in governing tensile creep results observed in regime II. Only the 6JC(V$_{Al}$) mechanism predicts a value of $Q_{Al}$ consistent with the present observations. However, the value of $Q_{Al}$ predicted by the NNN process as well as the calculated values of $Q_{Ni}$ for the TD and 6JC(V$_{Ni}$) mechanism are all in reasonable with the compression creep data observed in regime II, and it is not possible to unambiguously identify which of these three diffusion mechanisms is important during compression creep of near stoichiometric NiAl.

*Phenomenological interpretation of regime III*

Figure 9 suggests that $Q_c \approx 250$ kJ mol$^{-1}$, which is about 0.63 of that measured in regime II, for the tension data in regime III. As discussed in the last section, the rate controlling mechanism in regime II presumably involves the diffusion of Al vacancies through the lattice.

Therefore, it is reasonable to conclude that regime III is controlled by dislocation core diffusion of Al vacancies. Noting that $Q_c = 365$ kJ mol$^{-1}$ for the 6JC($V_{Al}$) lattice diffusion mechanism [56] (Table 5), the predicted value for dislocation core diffusion of Al vacancies is about 230 kJ mol$^{-1}$ assuming that its magnitude is 0.63 of that for lattice diffusion. This value is reasonably close to the experimental results shown in eqn. 7(b) and Fig. 9. In contrast, the interpretation of the compression results in regime III in a similar manner is unsatisfactory since the predicted value for dislocation core diffusion of Ni vacancies is about 190 kJ mol$^{-1}$ assuming that the lattice diffusion of Ni vacancies is by the 6JC($V_{Ni}$) mechanism.

However, a consistent picture emerges if it is assumed that the vacancy flow to the dislocations also occurs through the lattice as in regime II but by a different mechanism. A comparison of the predicted activation energies for diffusion with the experimental data (Table 5) suggests that the Al or Ni vacancy flow occur by the 6JC mechanism in regime II and by the NNN process in regime III under tensile or compressive creep, respectively. The predicted activation volume for the NNN mechanism is less than that for the 6JC process [56] so that $Q_c$ is expected to be less dependent on the nature of the stress state, which is consistent with the observations shown in Fig. 9.

*Mechanistic of dislocation climb in near-stoichiometric NiAl*

A simple atom-vacancy exchange process facilitating dislocation climb of a jog on a screw dislocation in NiAl can be envisioned if the NNN mechanism is operative. For the dislocation core structure shown in Fig. 15(a) [58], it can be envisioned that atoms 1 and 2 exchange positions with vacancies A and B, respectively, residing on the NNN site (Fig. 15(a)).

Next, atom 4 exchanges positions with vacancy A and atom 5 with vacancy B. The final configuration is shown in Fig. 15(b) after atom 6 exchanges position with vacancy A assuming that there is no lattice relaxation.

## SUMMARY AND CONCLUSIONS

Long term tensile creep tests were conducted on near-stoichiometric NiAl between 700 and 1200 K and their behavior were compared with previously published compression creep data on material with a similar grain size and processing history [15,24,25]. Inverse and normal primary creep occurs in both modes of deformation, where the former type of transient behavior was observed at higher stresses and higher temperatures under compression than in tension loading. Several possibilities limiting dislocation mobility in NiAl were examined and it was concluded that models postulating the non-conservative motion of jogs on screw dislocations are the most consistent with the present observations.

The stress exponents for tensile creep varied between 4.9 and 13.9 with the values of n ~ 5-6 varying between 1000 and 1200 K and n > 8.5 between 700 and 1000 K. Four deformation regimes were identified. The true activation energy was observed to be less than that for lattice diffusion of Ni tracers in regime I. The true activation energy for tensile creep in regime II was determined to be about 400 kJ mol$^{-1}$ for $20 \leq \sigma \leq 50$ MPa and 250 kJ mol$^{-1}$ in regime III for $50 \leq \sigma \leq 110$ MPa. The activation energy exhibited an inverse stress dependence in accordance with both obstacle glide and compact cross-slip controlled creep mechanisms in regime IV. The values of 400 and 250 kJ mol$^{-1}$ observed in regime II and III, respectively, were attributed to the lattice diffusion of Al vacancies based on values predicted by diffusion models for NiAl. In

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comparison, the measured activation energies for compressive creep of 300 kJ mol$^{-1}$ in regime II is in reasonable agreement with the experimental values reported for lattice self diffusion of Ni tracers in NiAl as well as with the calculated values for the lattice diffusion of Ni vacancies in NiAl. Finally, the mechanistic of dislocation climb in NiAl is discussed based on the next nearest neighbor atom-vacancy exchange mechanisms occurring within the core of jog on a screw dislocation.

## ACKNOWLEDGEMENTS

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**Table 1.:** Chemical composition (at.% ) and initial grain sizes of the two extruded NiAl rods.

| Extrusion I.D. | Extrusion conditions | Al | C | N | Ni | O | d ($\mu$m) |
|---|---|---|---|---|---|---|---|
| L 3175 | 1400 K; 16:1 | 49.8 | 0.0090 | 0.0005 | 50.2 | 0.0110 | 38.7 ± 2.5 |
| L 3176 | 1400 K; 20:1 | 50.6 | 0.0270 | 0.0010 | 49.4 | 0.0120 | 38.6 ± 2.3 |

**Table 2:** Test conditions and final grain sizes for several specimens after long term testing.

| Region | T (K) | σ (MPa) | ε (%) | $t_r$ (h) | $d_f$ (μm) | Sample size |
|---|---|---|---|---|---|---|
| shoulder | 700 | 100 | 0.0 | 6529.4 | 65.7 ± 5.3 | 210 |
| gage | 700 | 100 | 8.4 | 6529.4 | 67.0 ± 3.9 | 749 |
| shoulder | 700 | 120 | 0.0 | 2413.6 | 62.9 ± 7.9 | 266 |
| gage | 700 | 120 | 53.4 | 2413.6 | 54.2 ± 4.2 | 888 |
| shoulder | 1100 | 10 | 0.0 | 6693.5 | 98.0 ± 18.8 | 114 |
| gage | 1100 | 10 | 4.0 | 6693.5 | 150.6 ± 9.6 | 533 |
| shoulder | 1200 | 10 | 0.0 | 9114.2 | 195.4 ± 31.1 | 86 |
| gage | 1200 | 10 | 4.3 | 9114.2 | 199.6 ± 15.0 | 367 |
| shoulder | 1200 | 13 | 0.0 | 326.6 | 162.6 ± 11.7 | 106 |
| gage | 1200 | 13 | 55.8 | 326.6 | 168.2 ± 25.4 | 264 |

**Table 3.:** Values of A, n and $R^2_d$ observed in tension creep of NiAl at different

temperatures.

| T (K) | $\sigma$ (MPa) | n | $A_1$ (MPa$^{-n}$ s$^{-1}$) | $R^2_d$ |
|---|---|---|---|---|
| 700 | 100-170 | 13.9 | $3.3 \times 10^{-37}$ | 0.946 |
| 800 | 75-145 | 8.7 | $3.1 \times 10^{-24}$ | 0.992 |
| 900 | 40-80 | 13.2 | $7.4 \times 10^{-31}$ | 0.894 |
| 1000 | 25-35 | 5.1 | $7.4 \times 10^{-16}$ | 0.664 |
| 1000 | 35-65 | 12.3 | $9.3 \times 10^{-28}$ | 0.968 |
| 1100 | 10-40 | 6.1 | $1.0 \times 10^{-15}$ | 0.891 |
| 1200 | 10-25 | 4.9 | $7.7 \times 10^{-13}$ | 0.902 |

**Table 4.:** Activation energies for creep at different values of true stress.

| σ (MPa) | $Q_c$ (kJ mol$^{-1}$) | $R^2_d$ |
|---------|----------------------|---------|
| | | |
| 15.9 | 302.5 | - |
| 21.3 | 237.9 | - |
| 25.7 | 387.9 ± 20.8 | 0.997 |
| 40.3 | 414.0 ± 39.7 | 0.993 |
| 50.2 | 341.0 ± 29.4 | 0.994 |
| 60.9 | 231.7 ± 14.2 | 0.992 |
| 76.2 | 259.4 ± 4.8 | 0.999 |
| 100.0 | 260.7 | - |
| 120.0 | 244.0 | - |
| 150.0 | 175.7 | - |

**Table 5**. Comparison of the true activation energies for compression and tensile creep of NiAl with those predicted for lattice self-diffusion of Al and Ni vacancies for several mechanisms.

| $Q_C$ (kJ mol$^{-1}$) Compression | $Q_C$ (kJ mol$^{-1}$) Tension | Diffusion Mechanism | $Q_{Al}$ (kJ mol$^{-1}$) | $Q_{Ni}$ (kJ mol$^{-1}$) |
|---|---|---|---|---|
| 300 [11,13,15] | 400 (20 ≤ σ ≤ 50 MPa); <br><br> 250 (50 ≤ σ ≤ 110 MPa) (present study) | | | |
| | | Triple Defect (TD) | - | 300 [31] |
| | | Four Ring (4R) | 835 [55] | 835 [55] |
| | | Next Nearest Neighbor (NNN) | 285 [55] | 265 [55,56] |
| | | Six Jump Cycle (6JC) | 365 [56] | 300 [56] |
| | | Anti-structure Bridge (ASB) | - | 255 [56] |

**FIGURE CAPTIONS**

Fig. 1: Optical micrographs of the (a) longitudinal and (b) transverse sections of the as-extruded rods.

Fig. 2: Tensile creep specimen design.

Fig. 3: Tensile true strain vs. time curves for near-stoichiometric NiAl at (a) 700 K, (b) 900 K and (c) 1200 K under initial applied stresses varying from 10 to 170 MPa showing inverse and normal primary creep behavior. The y-axis is offset in (a), (b) & (c) and the x-axis is offset in (c) to allow the curves to be resolved.

Fig. 4: Tensile true creep rate vs. true strain curves for near-stoichiometric NiAl at (a) 700 K, (b) 900 K and (c) 1200 K under initial applied stresses varying from 10 to 170 MPa showing inverse and normal primary creep behavior.

Fig. 5: Long term tensile creep curves observed between 700 and 1200 K under initial applied stresses varying from 10 to 100 MPa showing inverse, multiple and normal primary creep behavior.

Fig. 6: The shapes of the creep transients observed in a stress change experiment conducted at 900 K between engineering stresses of 40 and 50 MPa under conditions where inverse primary creep is observed: (a) Tensile true strain vs. time curves; (b)

tensile true creep rate vs. true strain curves; (c) tensile true creep rate vs. time curves. The true stresses before and after each stress change are indicated in (b) & (c).

Fig. 7: Tensile true creep rate vs. true stress plots for near-stoichiometric NiAl between 700 and 1200 K determined in the secondary creep region.

Fig. 8: Arrhenius plots for near-stoichiometric NiAl for true stresses varying between 15.9 MPa ($\sigma/E \sim 8.9 \times 10^{-5}$) and 150 MPa ($\sigma/E \sim 7.6 \times 10^{-4}$). The creep rate has been normalized by the absolute temperature and the Young's modulus to take into account the temperature dependence of the pre-exponential term in the Mukherjee-Bird-Dorn creep equation [28].

Fig. 9: Stress dependence of the true activation energy for tensile creep showing four deformation regimes (I, II, III and IV). The activation energy for the lattice diffusion of Ni tracers, depicted by the upper horizontal broken line, is based on the data of Frank *et al*. [31].

Fig. 10: Map of the stress-temperature space depicting the conditions under which inverse (I), multiple (M) and normal (N) primary creep curves were observed during tension creep of near-stoichiometric NiAl.

Fig. 11: Stress-temperature space where inverse (I) and normal (N) primary creep curves were observed during creep of near-stoichiometric NiAl showing a shift to higher stresses

and higher temperatures for the compression creep data [15,24,25] compared to the tension creep results.

Fig. 12: Comparison of the stress dependence of the true activation energy for creep of near-stoichiometric NiAl determined in compression [13,24,25] and tension creep experiments. The activation energy for the lattice diffusion of Ni tracers, depicted by the upper horizontal broken line, is based on the data of Frank *et al*. [31].

Fig. 13: Comparison of the true creep rate vs. true stress for as-cast and extruded, near-stoichiometric NiAl tested in compression [15,24,25] and tension between 800 and 1200 K. The average grain size for the compression specimens was 35 μm.

Fig. 14(a-b): Schematic showing the probable sequence of atom-vacancy exchanges occurring within the core of a <100> {010} edge dislocation during climb by one atomic step assuming a next nearest neighbor jump cycle. The dislocation core structure is based on atomic simulation studies [58]. The Al and Ni atoms are represented by the solid and open circles, respectively, while the vacancy is represented by the square. The positions of atoms 1 to 6 and Al and Ni vacancies denoted as A and B, respectively, are indicated in (a) the initial and (b) the final configuration, where the arrow denotes the direction of motion of the Al and Ni atoms. Lattice relaxation is ignored.