



## Full length article

## On the temperature dependent strengthening of nickel by transition metal solutes

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## ABSTRACT

Fundamental insights are presented into the factors influencing the temperature-dependent strengthening of nickel by transition metal solutes. Strain rate jump tests are used using the alloying elements Ta, W and Re which lie beside one another in the d-block. At 800 °C, the hardening induced by Ta is significantly greater than that by W and Re. At  $T \geq 1000$  °C, Re becomes more potent than Ta at slow strain rates; at 1200 °C it is the most effective at all the strain rates employed. The results are interpreted using theory for temperature-dependent strengthening that emphasises solute–dislocation interaction. It is confirmed that at low temperatures the solute strengthening is controlled by paraelastic interaction – the solute with the largest size difference with the host Ni is the most potent. At higher temperatures, the evidence indicates that solutes collect on dislocations such that the slowest diffusing solutes confer maximal resistance to dislocation glide and climb; thus Re is particularly potent. The findings elucidate the role of transition metal solutes in strengthening. Moreover, they provide the necessary quantitative data for ongoing alloy design efforts.

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## 1. Introduction

What is the optimum combination of alloying elements needed to impart superior mechanical properties to nickel, so that the very best superalloys can be designed? At present, there exists no unequivocal answer to this question, probably due to the complexity of these alloys [1–3]. This difficulty is aggravated by the multiple effects which each element can induce, contributions to both solid-solution strengthening and precipitation hardening, which are difficult to de-convolute [3–8]. In this paper, systematic experiments are carried out to elucidate the role played by the transition metal solutes rhenium (Re), tungsten (W) and tantalum (Ta) in the hardening of nickel (Ni). To simplify matters, precipitate hardening is avoided by eliminating the critical elements causing this effect – specifically aluminium and titanium. Moreover, any effect of grain boundaries is circumvented by growing single crystal specimens

and testing them along the technologically-relevant  $\langle 001 \rangle$  crystallographic direction. In this way, unequivocal, fundamental insights are gleaned into the physical phenomena responsible for the temperature-dependent hardening of nickel alloys in the service-relevant regime.

## 2. Background

The accepted view is that the degree of strengthening in substitutional solid solutions depends upon the size difference  $\delta$  but also the modulus mismatch  $\eta$  between solute and solvent [9–12], with the solutes being assumed to cause hardening by locking dislocations or else by exerting drag forces on them. At ambient temperatures the dislocation locking mechanism can be described by Fleischer's work [9] in which solute atoms are assumed to act as point defects which resist dislocation motion. Labusch's theory [10], on the other hand, considers the solute atom to exert a drag-type force on the dislocations; a stronger pinning of dislocations by the solutes then results. Either way, the contribution of a solute to solid solution strengthening (SSH) –  $\Delta\sigma_{\text{SSH}}$  – can be estimated by:

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$$\Delta\sigma_{ssh} = k_i c^{a'} \quad (1)$$

where  $k_i$  is a solid solution strengthening coefficient,  $c$  is the solute concentration and  $a'$  depends upon the solute–dislocation interaction, and equal to 1/2 for weak pinning [9] and 3/2 for strong pinning of dislocations [9]. For Labusch's theory, the strengthening parameter  $k_i$  can be estimated using:

$$k_i = 3G\varepsilon_i^{4/3}Z; \quad \varepsilon_i = \left(\eta_i^2 + \alpha_d^2\delta^2\right)^{1/2} \quad (2)$$

where

$$\delta = \frac{1}{a} \frac{da}{dc}; \quad \eta_i = \frac{\eta_i}{1 + 0.5|\eta_i|}; \quad \eta_i = \frac{1}{G} \frac{dG}{dc} \quad (3)$$

Here,  $a$  is the lattice parameter and  $G$  is the shear modulus. The terms  $\delta$  and  $\eta'$  are used to quantify the paraelastic and modulus interaction respectively. The term  $\alpha_d$  depends on the nature of dislocation considered; it is taken as 16 in the present work, to account for strong pinning of dislocations by the solutes. The term  $Z$  is a fitting parameter [12,13].

Comparison of theory with the available experimental data is vital. Pelloux & Grant [14] studied SSH in binary nickel based alloys (Ni-Cr, Ni-W & Ni-Mo) at room temperature (RT) and suggested that for strengthening of nickel – in addition to the size difference – the solute-solvent electronic interactions also play an important role. Later Akhtar & Techtsoonian showed for Ni-Cr alloys that Labusch's theory can describe SSH for Cr contents below 9 wt.%. Suzuki et al. [15] investigated a series of binary nickel solid solution alloys and showed that the transition metal solutes (Ta & W) make an extra contribution to the SSH of nickel which cannot be totally explained by the modulus and size mismatch of solutes using Fleischer's theory [9]. Solute-dislocation interaction energies for different solutes in nickel have been studied and it has been suggested that the transition metal solutes provide this extra strengthening effect due to their electronic interaction with the host nickel [16]. However, more recent work [12,17,18] suggests that this stronger strengthening of nickel at ambient temperatures can be explained using Labusch's theory. Interestingly, Gan et al. [19] have tested binary nickel-based solid solutions and have used Labusch theory to fit their data, but conclude that at RT, the dominant mechanism for strengthening of Ni is not the size difference between the solute and solvent but instead the electronic structure and the resultant change in stacking fault energy,  $\gamma_{SFE}$ . From the above, it is clear that conflicting conclusions have been drawn to explain the hardening of nickel in the low temperature athermal regime.

The above applies predominantly to deformation at ambient conditions; in comparison, the influence of solutes at high temperatures – at which a time-dependent mode of creep hardening is expected – is not so well understood. Important questions include: what is the role of the temperature and/or strain rate at which the deformation is no longer athermal? And which elements cause the most potent hardening? For nickel alloys which are devoid of elements such as Al and Ti which promote precipitation hardening via the  $Ni_3(Al, Ti)$  phase known as  $\gamma'$ , the steady-state creep strain rate  $\dot{\varepsilon}$  is given by

$$\dot{\varepsilon} = A \left( \frac{DGb}{k_b T} \right) \left( \frac{\sigma}{G} \right)^n \quad (4)$$

where  $D$  is a diffusion coefficient,  $G$  is the shear modulus,  $k_b$  is the Boltzmann constant,  $\sigma$  is the applied stress,  $T$  is the temperature,  $A$  is a material dependent constant and  $n$  is the stress exponent. In the

literature, the creep of any given solid solution alloy has been assumed to fall into one of two classes [20,21]. In *class-II* alloys, the solutes have a large size difference from the solvent which favours elastic interactions between solutes and dislocations; creep mechanisms in these alloys are similar to pure metals for which diffusion-controlled climb controls the creep rate. Furthermore, the creep rate is thought to depend strongly upon  $\gamma_{SFE}$ . On the other hand, the creep behaviour of *class-I* alloys is interpreted differently. The creep rate is controlled by viscous glide of dislocations with no profound influence of the stacking fault energy. At high temperatures, the solute atoms have a high mobility; they are attracted to the dislocations as it reduces their elastic stress field. This results in the formation of either a Cottrell-type solute environment around the dislocation or the collection of the solute atoms on the dislocation line; a drag force on the moving dislocation results so that the creep rate in class-I solid solutions is controlled by this solute drag [20,22,23]. It follows that – depending upon the applied stresses and temperature – a transition from climb-controlled creep to viscous glide-controlled creep might occur [24]. A careful analysis of the deformation conditions and the alloy microstructure is required to determine the rate controlling process. One can refer to [20,21,24–28] for a detailed critique of the creep behaviour of solid solutions. Some progress has been made recently by Fleischmann et al. [1,29] along the lines of what is needed; single crystal nickel alloys were prepared of composition identical to that of the  $\gamma$  matrix of a commercially-relevant  $\gamma'$ -strengthened superalloy. The idea to test the properties of a single phase material is a good one which is taken up in the present paper.

What is clear from the above is that systematic, well-designed experimentation is needed to elucidate the athermal and thermal contributions to strengthening. The strain-rate dependence of deformation also needs careful clarification. Any findings need to be placed in the context of theory presented thus far. The present work was initiated with these points in mind.

### 3. Experimental methods

An industrial scale investment casting facility at the University of Birmingham, UK was used to prepare specimens in single crystal specimens in rod form. Both pure Ni and solid solution alloys with 2 at.% X (X = Ta, W & Re) were cast. These dilute concentrations were selected to ensure complete solubility of the solutes in Ni at all the temperature ranges needed for testing. The cast rods had a diameter of 15 mm and a length of 150 mm; they were macro-etched to ensure that no casting defects such as freckles were present. Further details about the casting process employed can be found elsewhere [30]. Any segregation in the as-cast state was homogenized using a multi-step solution heat treatment which involved heating at 1235 °C for 0.5 h followed by 1260 °C for 0.5 h, 1288 °C for 0.5 h and 1312 °C for 6 h. The samples were then cooled from the homogenisation temperature using gas fan cooling. Details about the use of gas quenching technique can be found in Ref. [31]. These rods were then orientated by Laue back reflection method in the  $\langle 100 \rangle$  orientation. Cylindrical specimens for mechanical testing were spark eroded with a height of 4.5 mm and 3.5 mm diameter, corresponding to an aspect ratio ( $l/\sqrt{A}$ ) of 1.5, where  $A$  is the area of cross-section and  $l$  is the length. The samples were polished plane parallel to an accuracy of better than 10  $\mu$ m.

A uniaxial compression testing machine (Instron 4505) was used to perform strain rate jump tests at 500 °C and in the service-relevant temperature range 800–1200 °C. These tests were performed by controlling the applied initial strain rate, which was varied in the range from  $10^{-3}s^{-1}$  to  $10^{-5}s^{-1}$ . An example of the strain rate jump from the strain rate of  $10^{-4}s^{-1}$  to  $10^{-5}s^{-1}$  is shown in Fig. 1. The instantaneous change in stress by varying the strain

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