Atom Probe Tomography investigations of microstructural evolution in an aged Nickel superalloy for exhaust applications

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ABSTRACT

The formation and evolution of nanoscale $\gamma''$ ($\text{Ni}_3\text{(Nb, Ti, Al)}$) precipitates formed during thermal ageing in the nickel superalloy Inconel 625 has been characterised using Atom Probe Tomography (APT). The onset of $\gamma''$ precipitation has been found to occur after only 1 hour, half the ageing time reported in the current literature. Coarsening behaviour of the precipitates has been found to follow the Lifshitz-Slyozov-Wagner model. Evolution of precipitate composition and morphology during ageing has been analysed, and the potential onset of the $\gamma'' \rightarrow \delta$ ($\text{Ni}_3\text{(Nb, Mo, Cr, Fe, Ti)}$) transformation after long ageing times is discussed.

KEYWORDS: Nickel-based Superalloys, Ageing, Microstructure evolution, Atom Probe Tomography

1. INTRODUCTION

The development of nickel superalloys has been driven by the need for corrosion resistant materials with excellent high temperature performance for use in gas-turbine engines for aircraft propulsion and power generation. The first jet engines were made from steel but as engine efficiency increased, so too did engine operating temperatures. New materials were required to make turbine blades able to withstand the higher temperatures and corrosive jet engine environment. Nickel superalloys were developed to fulfil these requirements, and have been extensively used by aerospace industries since 1929 [1].

The unique combination of high temperature strength and excellent corrosion resistance means that use of nickel superalloys is not limited to the aerospace sector but is in fact applicable in a wide range of extreme environments. For example, Inconel 625 is a nickel superalloy originally designed for high strength steam piping for super critical steam power
plants [2]. However, in recent years the alloy has been used to make exhausts for Formula 1 class racing engines [3].

The excellent corrosion resistance of Inconel 625 enables exhaust tubings to withstand the corrosive environment created by the exhaust gases. Tight fuel allowances in the sport mean that in order to reach competitive speeds, an energy recovery system (ERS) must now be used in conjunction with the internal combustion engine (ICE) [4]. This increases the thermal efficiency of the power unit (ERS + ICE) to ~50%. The high temperature behaviour of Inconel 625 must as a result be well-understood to enable the exhaust system to cope with the rising heat loads.

Nickel superalloys owe their corrosion resistance and high temperature strength to the careful choice of alloying additions. For example, aluminium, chromium and molybdenum are added to improve corrosion resistance and, in the case of Inconel 625, formation of the ordered γ’’ strengthening phase is promoted by addition of niobium and aluminium [5]. This coherent γ’’ phase has a DO_{22} crystal structure and can result in a 60% increase in yield stress [6]. γ’’ (Ni_{3}(Nb, Ti, Al)) forms in preference to γ’ (Ni_{3}Al) in alloys with lower aluminium and titanium content such as Inconel 625 [7].

Ageing controls the precipitate size, volume fraction and morphology of the γ’’ precipitates, which in turn determines the mechanical properties of the alloy. Although the exhaust is initially in a solution-treated state, it operates between 600-1000°C which means that the microstructure evolves throughout service.

Despite the importance of the γ’’ phase in governing the mechanical properties of Inconel 625, the ageing time and temperature at which γ’’ precipitates first form is not well understood. Multiple time-temperature-transformation (TTT) curves have been proposed for the Inconel 625 system [8]–[10], but there is disagreement between these curves. In particular, the position of the γ’’ nose, which is the point on the TTT curve at which γ’’ first starts to form, varies between studies from 2 to 10 hours [8]–[10].

It is likely that an increase in resolution of the characterisation techniques used for phase identification (from optical microscopy [9] to Field Emission Gun SEM [10]) has led to the nose of the γ’’ curve being shifted to shorter ageing times. Microhardness measurements made by Suave et al. [11] support this hypothesis, suggesting that γ’’ precipitation may occur after ageing for as little as 0.5 hours at 650°C. This work uses the high spatial resolution and chemical sensitivity of atom probe tomography (APT) [12] to determine the onset of γ’’ precipitation to a higher degree of precision.

Furthermore, γ’’ precipitate composition is not well-defined. Work by Floreen et al. [8] reports the precipitate composition as a range, Ni_{3}(Nb>0.5 Ti<0.5 Al<0.5). However, whilst transmission electron microscopy (TEM) has sufficient spatial resolution to image such nanoscale precipitates, compositions determined using TEM-EDS are only semi-quantitative. Thus a secondary goal of this research was to use APT to provide fully quantitative γ’’ compositions, as well as studying the evolution of both size and composition of the precipitates during ageing.
2. EXPERIMENTAL METHODS

Inconel 625, originally melted by Special Metals Corporation (SMC), was provided by Good Fabrications Ltd. Additional Inconel 625 was sourced from Goodfellow Cambridge Ltd (GCL). The SMC strip was cold rolled to deep drawing quality and subsequently pickled and annealed at 1040°C in a continuous furnace, whereas the GCL sheet was annealed and tempered. The heat treatments for both materials were such that no γ” precipitates were present in the as-received material. The nominal compositions of the as-received material are reported in table 1.

Table 1: Nominal compositions of as-received Inconel 625 material.

|     | Ni  | Co  | Cr  | Fe  | Mo  | Nb  | Al  | Ti  | Mn  | Si  | C   | S   |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| SMC | >58.0 | <1.0 | 24.0 | <5.3 | 6.0 | 2.0 | <0.9 | <0.5 | <0.5 | <1.1 | <0.5 | <0.03 |
| GCL | Balance (Ni+Co) | 23.6 | <5.1 | 5.3 | 2.2 | 0.5 | 0.3 | 0.3 | 1.0 | <4.7 | 0.03 |

The Inconel 625 strip was cut into matchsticks using a Buehler Isomet 4000 saw. The matchsticks were ∼20 mm in length, with a square cross section of side length ∼0.7 mm. Samples were then aged in a vacuum tube furnace for 1 hour at 650/700/750°C to investigate the onset of γ” precipitation, and for 10/100/1000 hours at 650°C to investigate the evolution of γ” precipitates during ageing. Matchsticks were heated to the respective ageing temperatures at a rate of 1200°C per hour to minimise uncertainty in ageing time, and a vacuum of $10^{-6}$ mbar was maintained throughout.

Specimens were prepared for analysis by APT via electrochemical polishing [12], using a solution of 10% perchloric acid in solution in 90% acetic acid and an applied voltage of 25V. A second stage of polishing was carried out to refine the tip shape, using a solution of 2% perchloric acid dissolved in 98% butoxyethanol, with an applied voltage of 20-25V.

Specimens were analysed using a Cameca LEAP 3000X HR system or a Cameca LEAP 5000XR (Department of Materials, University of Oxford.) The LEAP 3000X HR and LEAP 5000XR have detector efficiencies of ~37% and ~52%, and laser wavelengths of 532 nm and 355 nm, respectively. During APT experiments, all specimens were cryogenically cooled to 50-55 K, within a UHV analysis chamber pumped to $<4 \times 10^{-11}$ Torr. Specimens were run in laser-mode at a pulse frequency of 200 kHz and at a target evaporation rate between 0.4% - 1%. A laser-pulse energy between 0.3-0.5 nJ and 0.03-0.05 nJ was used on the LEAP 3000 and LEAP 5000, respectively. The laser powers were selected to ensure power density remained comparable between the two instruments.

Hardness tests were performed using a Wilson Wolpert microhardness indenter and processed using the analysis software Minuteman. The dwell time was 10 seconds and the applied load was 0.5 kg. A minimum of ten measurements were made across the length of
the sample, keeping away from the specimen edge, to ensure representative sampling. Each test was performed sufficiently far from the previous to prevent interaction of plastic zones.

3. RESULTS

Onset of precipitation

Figure 1 compares atom maps of solution-treated and aged Inconel 625 (1 hour at 650°C, 700°C and 750°C.) The atom maps display only nickel, titanium and niobium ions for visual clarity. The precipitates are highlighted using a 4.5 at% Nb+Ti isoconcentration surface and are identified as γ” since they are enriched in nickel, niobium and titanium. It can be seen from Figure 1 that nanoscale (∼5nm) precipitates have formed after ageing for only 1 hour at 700°C and 750°C, whereas the alloying elements remain in complete solid solution in the sample aged for 1 hour at 650°C. This is supported by the nearest neighbour distributions [12] for this sample, shown in Figure 2 (b).

The agreement between the expected distribution for a random solid solution (shown in red on figure 2(b)) and the measured distribution of titanium and niobium atoms (shown in black on figure 2(b)) suggests there is no clustering in the sample aged for 1 hour at 650°C. However, deviation of the measured distribution from the randomized distribution can be seen in the plots in figure 2 for all other ageing conditions, suggesting clustering of niobium and titanium atoms is occurring at longer ageing times and higher ageing temperatures.

Evolution of precipitates as a function of ageing time

Samples of Inconel 625 were aged at 650°C for 10, 100 and 1000 hours. Figure 3 (a) shows the atom maps corresponding to these treatments, with precipitates apparent at all ageing times, as highlighted using Nb+Ti isoconcentration surfaces. The values chosen for these isoconcentration surfaces (see figure caption) were chosen using the proxigrams in Figure 6. The isoconcentration value was adjusted until the position of the interface, shown by the dotted line in Figure 6, coincided with the point of inflection of the niobium concentration profile on the proxigram. A separate set of isoconcentration surfaces (not shown) were used to select the core of precipitates for compositional analysis, thus avoiding the inclusion of precipitate-matrix interfacial regions. Precipitates initially start off spherical, as seen in Figure 3 (a)(i) and then become elongated with increasing ageing time. After long ageing times (1000 hours) precipitates become aligned along two distinct directions, with an angle of approximately 80 degrees between them, as can be seen in Figure 3(a)(iii). This alignment is consistent with SEM images of Inconel 625 aged for 1000 hours at 650°C in the literature [10]. Figure 3(b) shows the evolution of the precipitate size distribution with ageing time. An equivalent precipitate radius was calculated by approximating the APT-determined precipitate volume as a sphere of equivalent volume [13]. This also assumes a uniform precipitate density regardless of size, which was confirmed in the data. It can be seen that as ageing time increases, the number of precipitates decreases. The peak equivalent precipitate radius increases from 2.4 nm to 3.9 nm to 9.6 nm as ageing progresses. During the APT experiment atoms from the precipitates require application of a higher electric field.
to be evaporated in comparison to those in the matrix, resulting in a deviation of the ion that can impact the APT image reconstruction [12]. This ultimately means the equivalent precipitate radii reported here should be considered in a qualitative manner only.

Closer analysis of the atom probe data taken from the long-term aged samples reveals the partitioning preference of the alloying elements in Inconel 625. APT analysis determined the average concentration of precipitates in this 1000 hour aged sample to be (in at. %) 71 Ni, 15 Nb, 5.1 Mo, 4.3 Cr, 2.7 Ti, 1.0 Al, 0.5 Fe. It can be seen from Figure 4 that niobium, titanium and nickel preferentially partition to the precipitates whereas chromium, iron and molybdenum partition to the matrix. Consequently the precipitates are identified as γ”.

The plots in Figure 5, created using the Lever rule [14], also provide information about the partitioning preferences of the alloying elements. The Lever rule is as follows:

$$C_n = V_{\gamma''} C_{\gamma''} + (1 - V_{\gamma''}) C_{\gamma}$$  \hspace{1cm} (1)

where $C_n$ is the composition of the whole dataset, $C_{\gamma}$ and $C_{\gamma''}$ are the compositions of the matrix and γ” precipitates respectively and $V_{\gamma''}$ is the volume fraction of the γ” precipitates. For each element, $C_n - C_{\gamma}$ has been plotted against $C_{\gamma''} - C_{\gamma}$, creating a linear plot with gradient equal to the γ” volume fraction.

Elements plotted in the negative-x, negative-y quadrant partition to the matrix whereas elements plotted in the positive-x, positive-y quadrant partition to the precipitates. Figure 5 (d) compares the Lever rule plots for all three ageing conditions and confirms that nickel, titanium and niobium partition to the precipitates, whilst the remaining elements partition to the matrix. It can be seen from figure 5 (d) that molybdenum has no strong partitioning preference as ageing progresses, although uniquely after 1000 hours of ageing at 650°C it appears to switch preference to the precipitates instead of the matrix. The arrows on figure 5 (d) indicate how the precipitate composition evolves with ageing time.

The behaviour of molybdenum was investigated further by taking proximity histograms [12] to measure the chemistry across the precipitate-matrix interfaces, which are shown in Figure 6. Comparison of the three proxigrams reveals segregation of molybdenum to the precipitate-matrix interface in the 1000 hour aged sample, whereas the molybdenum concentration increases smoothly across the interface (from matrix to precipitate) for shorter ageing times.
Table 2: γ” precipitate and matrix compositions as a function of ageing time.

| Ageing time (hours) | Precipitate composition (at. %) | Matrix composition (at. %) |
|---------------------|---------------------------------|---------------------------|
|                     | Ni    | Cr   | Fe   | Mo   | Nb   | Al  | Ti  |
| 10                  | 67.0  | 8.1  | 0.43 | 6.3  | 14.0 | 1.0 | 2.8 |
| 100                 | 69.0  | 4.4  | 0.36 | 5.3  | 16.0 | 1.1 | 3.1 |
| 1000                | 72.0  | 4.0  | 0.28 | 5.1  | 15.0 | 1.0 | 2.6 |

The full evolution of precipitate and matrix compositions with ageing time is presented in Table 2. As ageing progresses, the concentration of nickel and niobium in the matrix becomes slightly depleted to form γ”. This is reflected in the movement of nickel and niobium in the positive x direction with increasing ageing time on the Lever diagrams in Figure 5(d). The concentration of γ stabilisers in the precipitates, including chromium and iron, decreases with ageing time, as these elements are rejected into the matrix and the precipitates approach a stoichiometry of Ni₃(Nb, Ti, Al). This corresponds to a shift in the negative x direction of chromium and iron in Figure 5(d) as ageing progresses.

The volume fractions of obtained from the Lever rule plots are shown in Figure 7. It can be seen that volume fraction increases with ageing time. Microhardness measurements were also taken for all samples aged at 650°C, and for a solution treated sample. These are also shown in Figure 7, revealing a strong correlation of hardness with γ” volume fraction, confirming a strong link between the APT-measurements and bulk mechanical properties.

4. DISCUSSION

Onset of precipitation

The APT results of Figure 1 show no apparent precipitates in samples aged for 1 hour at 650°C, however, in comparison precipitation of γ” was observed after only 1 hour of ageing at 700°C. This is half the ageing time previously reported for the onset of precipitation in the literature at this temperature [10]. This information has been used to produce an updated time-temperature-transformation (TTT) curve for Inconel 625, which can be seen in Figure 8.

Kinetics of γ” precipitation

The change in precipitate size with ageing time can be used to determine γ” growth kinetics. γ” coarsening behaviour has previously been studied using the Lifshitz-Slyozov-Wagner (LSW) theory of Ostwald ripening [15], [16], which is also used here to calculate the activation energy for γ” coarsening. LSW theory describes diffusion-driven growth of larger precipitates at the expense of smaller precipitates, as defined by equation (2):

$$R^3 - R_0^3 = K \times t$$

(2)
where $R_0$ is the initial precipitate radius, $K$ is the coarsening rate and $R$ is the precipitate radius after ageing time, $t$. The relationship between the cube of the equivalent precipitate radius ($R^3$), determined from Figure 3 (b), and ageing time ($t$) was found to be linear, with all points lying within 95% confidence bounds. This confirms that the γ’’ precipitates in this study grow according to LSW theory via a diffusion-controlled mechanism.

The precipitate coarsening rate ($K$) is the constant of proportionality in equation 2, and is defined in equation 3. This expression for $K$ can be used to calculate the activation energy for coarsening, $Q$:

$$K = \frac{128\Gamma C_e V_m^2 D_o}{9q\pi R T} \times \exp\left(-\frac{Q}{RT}\right)$$

where $q$ is the aspect ratio of the γ’’ precipitates, $\Gamma$ is the particle-matrix interfacial energy, $C_e$ is the concentration of the niobium atoms in equilibrium with a matrix of infinite radius, $V_m$ is the molar volume of precipitates, $D_o$ is the diffusion coefficient of niobium, $R$ is the molar gas constant and $T$ is the absolute temperature [10].

The value of $K$ at 650°C is given by the gradient of a plot of $R^3$ vs. $t$ for the precipitates in the samples aged at 650°C for 10, 100 and 1000 hours. This value of $K$, along with constants reported in Table 3, can be used to calculate the activation energy for coarsening as 364-372 kJmol$^{-1}$.

Table 3: Constants required to calculate precipitate coarsening activation energy, $Q$.

| Parameter | Value                  |
|-----------|------------------------|
| $\Gamma$  | 9.25E-2 Jm$^{-2}$ [17] |
| $V_m$     | 2.92E-05 [17]          |
| $q$       | 0.2-0.72               |
| $D_o$     | 8.80E-05 m$^2$s$^{-1}$ [18] |
| $C_e$     | 1345-1794 molm$^{-3}$  |
| $T$       | 923 K                  |

The range of values is due to the variation of $q$ and $C_e$ with ageing time, and is higher than the apparent activation energy of 358 kJ mol$^{-1}$ calculated by Suave et al. [10]. However, it has previously been reported that, of the parameters in Table 3, the concentration of niobium atoms in the matrix, $C_e$, has the largest associated error [17]. When calculating the precipitate coarsening rate, $K$, Devaux et al. [17] determined $C_e$ used energy dispersive spectroscopy (EDS) analysis, but this only gives semi-quantitative composition measurements. In this study, $C_e$ was determined using APT, which combines high chemical sensitivity and spatial resolution to provide quantitative compositional data. Consequently the activation energy reported here is believed to be a more accurate value than proposed in earlier studies.

γ’’ → δ transformation

The composition of the γ’’ precipitates that form during ageing treatment evolves over time, as shown in Table 2. In the current literature, the composition of γ’’ is reported as a range: Ni$_3$(Nb>0.5 Ti<0.5 Al<0.5), which corresponds to (all in at.%) 75 Ni, >12.5 Nb, <12.5 Ti, <12.5
This study has narrowed down these ranges to 67-72 Ni, 14-16 Nb, 1.0-1.1 Al and 2.6-3.1 Ti. In particular, the values for titanium and aluminium have been significantly refined compared to the overestimated values reported by Floreen et al. [8]. Molybdenum (∼6 at.%), chromium (∼6 at.% ) and iron (∼0.4 at. %) are also present in the precipitates; further work is needed to confirm their particular site preferences.

The evolution in precipitate chemistry with ageing highlights the metastable nature of the precipitates, and could indicate possible transformation to a further phase. This hypothesis is supported by proxigram analysis across the precipitate-matrix interface in the 1000 hour aged samples, showing Mo is segregating to the interface. This suggests a loss of precipitate coherency, possibly indicating the start of the γ'' → δ transformation. The orthorhombic intermetallic δ phase has the stoichiometry Ni₃(Nb,Mo) and can be distinguished from γ'' via composition, morphology or crystal structure.

The morphology of the precipitates also evolves with ageing time, changing from spherical to lenticular. This is also in agreement with the mechanism for γ'' → δ transformation proposed by Xing et al. [19]. In this, lattice distortion, arising from γ'' phase formation, leads to creation of stacking faults within γ'', which act as nucleation points for the δ phase. The δ phase then develops along the three closed packed directions within the γ'' structure.

The TTT curve produced by Suave et al. [10], and displayed in Figure 8, indicates that samples aged for 1000 hours at 650°C are on the boundary of the δ precipitation domain. Further work is needed to investigate the mechanism underpinning the γ'' → δ transformation. For example, using TEM to determine the crystal structure of the precipitates formed over longer ageing durations, which would distinguish between the orthorhombic crystal structure of the δ phase and the DO₂₂ crystal structure of γ''. Accompanying APT of the precipitates could be used to monitor any compositional changes that occur as a result of the phase transformation.

It can be seen from Figure 7 that hardness and volume fraction of γ'' both increase with ageing time. This is in agreement with the literature, where increasing hardness was attributed to increased γ'' volume fraction [10], [20], and as mentioned demonstrates the ability of nanoscale APT data to inform about the mechanical properties of an alloy.

5. CONCLUSIONS

This study has characterised the formation and evolution of γ'' precipitates during ageing in the nickel superalloy Inconel 625. The following conclusions can be drawn:

- Precipitation of γ'' occurs after only 1 hour of ageing at 700°C, which is half the ageing time reported in the current literature [10].
- The coarsening behaviour of the γ'' precipitates follows Lifshitz-Slyovoz-Wagner model. The activation energy for coarsening of γ'' has been better refined using this model.
- The evolution of the precipitates as ageing progresses may indicate γ'' → δ transformation, supported by several pieces of information:
1. The precipitate composition changes as ageing progresses, highlighting the metastable nature of the precipitates, suggesting thermodynamic equilibrium may not be fully present in the γ’’.

2. There appears to be segregation of molybdenum to the precipitate-matrix interface, which could indicate a loss of γ’’ precipitate coherency, and thus indicate the start of a γ’’ → δ transformation.

3. The morphology of the γ’’ changes from spherical to lenticular during ageing, in agreement with the mechanism for the γ’’ → δ transformation proposed by Xing et al. [19].

- The formation and growth of γ’’ precipitates increases the material hardness by approximately 40% for an as-received sample compared to a sample aged for 1000 hours at 650°C.
- Niobium and titanium, elements which promote protective oxide formation [22], are being depleted from the matrix to form γ’’ precipitates at shorter ageing times than previously reported. This could negatively impact the corrosion resistance of this material in an exhaust environment.

6. ACKNOWLEDGEMENTS

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Fig. 1 - APT atom maps for the solution-treated, as-received sample (a), and the samples aged for 1 hour at 650°C (b), 1 hour at 700°C (c) and 1 hour at 750°C (d). In all atom maps, only nickel, niobium, aluminium and titanium atoms have been displayed for clarity.
Fig. 2- 10th nearest neighbour (10NN) distance distribution for Nb and Ti atoms for the (a) solution treated Inconel 625 and the samples aged for (b) 1 hour at 650°C, (c) 1 hour at 700°C, (d) 1 hour at 750°C, (e) 10 hours at 650°C, (f) 100 hours at 650°C, (g) 1000 hours at 650°C. The red line shows the distribution of 10NN distances that would be expected in a solution of randomly distributed atoms. The black line shows the distribution of 10NN distances measured within the APT dataset.

Fig. 3- Atom maps for Inconel 625 samples aged for 10 hours, 100 hours and 1000 hours at 650°C are shown in (a)(i), (a)(ii) and (a)(iii), respectively. In (a)(i) and (a)(ii), precipitates are highlighted using 4.5 at. % Nb+Ti isoconcentration surfaces whereas in (a)(iii), a 4 at. % Nb+Ti isoconcentration surface was used. In each atom map, 20% of the Al and Nb atoms, and 100% of the Ti atoms are displayed for clarity. Precipitate size distributions for the samples aged for 10 hours, 100 hours and 1000 hours at 650°C are shown in (b)(i), (b)(ii) and (b)(iii), respectively.
Fig. 4- APT atom maps for the sample aged for 1000 hours at 650°C. (a) displays all the ions in the dataset, (b)-(g) displays individual elements separately. Angle between precipitates ranges from 70-90°, which is in agreement with SEM images taken by Suave et al [10].
Fig. 5- Lever rule plots for samples aged for 10 hours (a), 100 hours (b) and 1000 hours (c) at 650°C. In (d) the points for the three ageing times are plotted on the same graph to allow comparison. A linear fit of the data is shown in red and 95% confidence bounds are shown in green on plots (a)-(c).
Fig. 6- Proxigram analyses highlighting change in composition across the matrix-$\gamma''$ precipitate interface for the 10 hour (a), 100 hour (b) and 1000 hour (c) samples, aged at 650°C. The dotted line indicates the position of the interface defined by the isoconcentration surface. The proxigrams in (a) and (b) were created using a 4.5 at. % Nb+Ti isoconcentration surface whereas the proxigram in (c) was created using a 4 at. % isoconcentration surface.
Fig. 7- Plot of volume fraction and hardness of γ'' precipitates as a function of ageing time at 650°C. Error bars show the standard deviation in the respective measurements.
Fig. 8- TTT curve showing points and fit determined by Suave et. al. [10] (black), and points determined in this study (red).

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