

# The Effect of Aging on the Strength of Newly Developed Corrosion Resistance-Resistant Incoloy Alloys 945 and 945X: from A Microstructural Perspective

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## Abstract

~~It has been found~~The results of this study show that Incoloy 945X ~~has~~ 19.36% ~~more hardness~~harder and 10.91% ~~more strength~~stronger than Incoloy 945. ~~The~~Of particular interest is the morphology of precipitates ~~morphology~~ responsible for precipitation hardening in both alloys ~~is of particular interest in current investigation.~~ To identify and understand the physical ~~reason for this,~~mechanisms that lead to these results, we ~~undertook~~ an extensive microstructural study ~~is performed wherein which~~  $\gamma'$  size, shape and elemental constitution ~~is was~~ investigated and correlated to the aging conditions. ~~The~~The main strengthening phase for both alloys is identified by transmission electron microscopy ~~studies reveal only one to be the~~ main population of  $\gamma'$  precipitates ~~sized, which are~~ between 10 and 25 nm ~~as the main strengthening phase for both alloys in size.~~ Scanning transmission electron microscopy line scans across the  $\gamma'$  precipitate ~~exhibit~~reveal more Nb and Ti deflection in Incoloy 945X than in Incoloy 945. This is further supported by the results of transmission electron microscopy ~~findings~~, in which  $\gamma'$  in 945X appear bright (because of Nb) with faceted growth, resulting in a higher value of 0.2% yield strength (1100 MPa) and hardness (430 HV5) than ~~the~~Incoloy 945. ~~Coarsening~~For each aging temperature, the coarsening kinetics of the  $\gamma'$  phase ~~were was~~ simulated for each aging temperature by using JMatPro. ~~The coarsening simulations were found to be in good agreement, and the simulation results were consistent~~ with the experimental results for the mean diameter of the  $\gamma'$  precipitates in each alloy.

Keywords: ~~945;~~ 945X; Scanning electron microscopy; Transmission electron microscopy;  $\gamma'$ ; Coarsening simulations, ~~precipitation, strengthening~~

## 1. Introduction

The high-strength Incoloy alloy 945 was developed in 2008 by the Special Metals Corporation for the oil and gas industry, ~~as it and~~ is more suitable than the traditional Inconel alloy 718 for deep-drilling applications ~~that involve deep drilling.~~ This alloy, Incoloy 945 is a precipitation-hardened and corrosion resistant Ni-Fe-Cr alloy. Although ~~its it contains~~ less nickel ~~content~~ than Inconel 718 (53 wt%), this alloy provides excellent ductility, impact strength, and resistance to stress corrosion cracking. In 2011, a higher-strength version of the alloy, called ~~the~~“Incoloy alloy 945X” (54 wt% nickel content),

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**Commented [SC ED3]:** Please mention the names of the authors who were involved in conducting this research. Please follow the below format for the names: First Author<sup>1</sup>, Second Author<sup>2\*</sup>, Third Author<sup>3</sup>

**Commented [SC ED4]:** Please provide full postal address of all the affiliations of the authors you have mentioned above. An example of the format is given below:  
<sup>1</sup>First Department, First University, Address, City, Country Name  
Also, mention the email address of each author, if available.

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**Commented [SC ED6]:** The abstract reflects the aim of the study and the methods implemented to achieve the study's objectives. However, you may consider to provide a brief introduction about the phases discussed in the section, namely,  $\gamma'$  and  $\gamma''$ , that would help in conveying the essence of the study in a clear and well-defined manner.

**Commented [EN QA7]:** Precipitation hardening is a very common strengthening mechanism approached by scientists, especially for compositionally complex alloys. This area of research has been in limelight for quite some time. Hence, your statement here needs to be more intense in its expression of what exactly is the interest area of this paper. What new is the reader going to find in this paper?

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was introduced. These Incoloy alloys can be used in high-pressure, high-temperature (HPHT) applications; and are approved for use under NACE MR0175/ISO 15156-3 up to levels V and VI [1].

As is normal with any nickel-based superalloy, the matrix consists of a face-centred cubic (FCC) structure called gamma ( $\gamma$ ). The solid solution strengthening elements of gamma are chromium, iron, molybdenum, titanium, aluminium, and cobalt. The main strengthening precipitates,  $\gamma'$  ( $\text{Ni}_3\text{Al}$ , Ti, Nb) and  $\gamma''$  ( $\text{Ni}_3\text{Nb}$ ) having  $\text{L1}_2$  and  $\text{DO}_{22}$  crystal structures, respectively, and have been the subject of numerous investigations [2-7]. As reported by Hagel and Beattie [8], the  $\gamma'$  phase is coherent with the gamma matrix, with lattice ( $\gamma$ ) misfits of 0.1 %, 0.5%–1.0% and greater than 1.25 % associated with the spherical particles, cubes, and plates, respectively. The  $\gamma'$  phase is an  $\text{A}_3\text{B}$  phase, in which the element substituting A is Ni, while Co and B are replaced by Ti, Al, and Nb. Hume-Rothery [9, 36, 37-11] stated that  $\text{Ni}_3\text{Al}$  is the most stable  $\gamma'$  phase, followed by  $\text{Ni}_3\text{Ti}$ ,  $\text{Ni}_3\text{Nb}$ , and  $\text{Ni}_3\text{Ta}$ , in the order of decreasing stability.

A nickel content above 42 wt.% is typically required for resistance to aqueous stress corrosion cracking [4012]. Molybdenum and chromium in the alloy provide resistance to pitting corrosion in reducing acid and alkali solutions [413, 4214]. Niobium, titanium, and aluminium are the primary precipitation-strengthening components [4315]. Although titanium increases the strength and hardness of the alloy, an excessive amount of titanium is known to degrade the microstructural stability [4416]. Aluminium increases the volume fraction of the  $\gamma'$  phase [4517], which is expected to further increase hardness; however, a high aluminium content reduces the lattice mismatch and coherency strain, resulting in a net decrease in hardness. The metastable  $\gamma''$  phase [5], [7], [4618-4820] has a body-centred tetragonal structure (BCT), and usually precipitates at temperatures less than 650 °C. The nickel alloys normally exhibit  $\gamma''$  precipitation under high coherency strains [4315]. These are fine nano-precipitates, like  $\gamma'$ , with high hardening and strength, having a length approximately six times greater than their width. This metastable phase tends to be replaced by a stable delta  $\text{Ni}_3\text{Nb}$  ( $\delta$ ) phase when exposed for extended periods to temperatures above 650 °C for extended periods [3521]. The presence of intergranular  $\delta$  platelets [4922] with an orthorhombic crystal structure results in a loss of hardness and ductility [2023].

This study seeks to verify whether Incoloy alloys 945 and 945X are known to resist the formation of intergranular phases, and this study seeks to verify this as is suspected. This resistance could be due to a lower Nb content of Nb in the actual alloy composition of the alloys, as compared to with the Nb content in the parent alloy, Inconel 718. Typically, the nickel-based alloys have low carbon content is low in nickel-based alloys. Carbon tends to react with refractory elements to form MC-type carbides that reside at the grain boundaries and within grains, with the 'M' mostly most often being Nb. Furthermore, Ti helps to form TiN; both the MC carbides and TiN are beneficial for help

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W. Hume-Rothery, G. W. Mabbott, and K. M. Channel-Evans, "The freezing points, melting points, and solid solubility limits of the alloys of silver and copper with the elements of the B Sub-Groups", Philos. Trans. R. Soc. London, Ser. A. 233, 1934, 1-97.

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strengthen the grain ~~boundary strengthening, inhibition of boundaries, inhibit~~ grain growth and ~~reduction of~~ reduce grain sliding, despite their detrimental properties of reduced ductility and toughness. Few studies [24,225] have ~~been carried out~~ focussed on 945 and the relatively new 945X alloys. The relationship between the microstructure and mechanical properties for these alloys ~~has~~ not been characterised ~~as fully~~, as ~~compared to~~ has that of other ~~Ni~~nickel-based alloys. The aim of the current study is to investigate the microstructure of the 945 and 945X alloys under isothermal solution treatment and aging, and analyse ~~their effects on~~ how it affects the mechanical strength. ~~The reason for which of the alloy.~~

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## 2. Experimental Methods

### 2.1. Experiment Materials

Incoloy alloys 945 (140 × 110 × 24 mm<sup>3</sup>) and 945X (250 × 250 × 360 ~~mm~~), ~~in its as received condition, mm<sup>3</sup>~~ were supplied by Titanium Engineers, Inc and C. H. Robinson Europe BV, respectively, ~~and used as received~~. The ~~as received~~ alloys ~~presented~~ had the ~~microstructures of the type of microstructure~~ typically used in oil and gas applications. ~~The alloys in the as received condition and~~ were ~~termed called~~ 'as ~~As Received~~ received' (AR) alloys ~~and details~~. Details of the AR heat treatment [416] ~~are~~ appear in Table 21. ~~The Table 42 shows the results of the optical-~~ emission spectroscopy (OES) ~~studies carried out on~~ applied to thin off-cuts from each alloy, ~~are shown in Table 1.~~

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Specimen	Alloy	Dimensions	Heat Treatment
ST	945/945X	14 <del>×</del> × 10 mm <sup>2</sup>	1038 <sup>o</sup> °C 1 h WQ
STA	945/945X	14 <del>×</del> × 10 mm <sup>2</sup>	1038 °C 1 h WQ then 718 °C 8 h FC + 621 °C 8 h AC 1010-1066 °C for 0.5-4 h, WQ to RT
AR	945/945X	14 <del>×</del> × 10 mm <sup>2</sup>	704-732 °C for 6-8 h, FC at 0.433-0.933 °C/min to 607-635 °C, then hold for 6-8 h, AC to RT
NS	945X	14 <del>×</del> × 10 mm <sup>2</sup>	1250 °C 1 h WQ then 718 °C 8 h FC + 621 °C 8 h AC

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~~The 945 and 945X alloys were subjected to thermal heat treatments under the conditions described in Table 2. The samples were ST in a preheated furnace to 1038 °C for 1 h and then quenched with water to RT 20 °C at a cooling rate of 50 °C/min. The ST samples were prepared for microstructural investigations, while the remaining samples were STA in a preheated furnace to 718 °C and aged at this temperature for 8 h.~~

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**Note:** Air cooling (AC); ~~Water~~ quenched (WQ); ~~Furnace Cooling~~furnace cooling (FC); ~~Room Temperature~~room temperature (RT); ~~solution treated (ST)~~~~Solution Treated~~; ~~solution treated and aged (STA)~~~~Solution Treated and Aged~~; ~~Non~~; non-standard (NS)

**Table 21.** Solution annealing and aging temperature of ~~the~~ 945 and 945X specimens.

Alloy	wt. % C	Si	Mn	Cr	Mo	Fe	Al	Cu	Nb	Ti	Ni
945	0.01	0.1	0.07	<b>20.5</b>	3.2	<b>22.2</b>	0.17	1.95	<b>3.11</b>	1.55	<b>47.04</b>
945X	0.01	0.08	0.04	<b>20.2</b>	3.2	<b>14.4</b>	0.15	2.09	<b>4.14</b>	1.57	<b>53.9</b>

**Table 22.** Elemental composition of as-received 945 and 945X alloys.

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### 2.2. Microstructural studies

Metallographic samples for ~~high~~optical microscopy (~~LM~~) and scanning electron microscopy (SEM); XL30 ESEM with Oxford ISIS 310 EDX facilities ~~is~~ operated at 10 kV; spot size 5; working distance 5; ~~Backscatter Electron~~, backscatter electron mode) were prepared by using standard mechanical polishing procedures. The specimens ~~to be observed~~ were ground by using silicon carbide papers with P240–P2500 grit sizes and polished by using ~~the~~ abrasive cloths in a descending order of grit size from 6 ~~µm~~ to ~0.25 µm. Kalling’s reagent etchant (100 ml HCl, 100 ml ethanol, 5 g CuCl<sub>2</sub>) and colloidal silica (0.06 µm) were used to reveal the microstructure (i.e. the grain boundaries and metal carbides). Transmission electron microscopy ~~studies were carried out was done~~ with ~~the~~ JEOL 2100 operated at an acceleration voltage of 200 kV (beam current ~102 µA) with a spot size of 1 and an alpha angle of 2°. Thin foils prepared from each sample were mechanically ground ~~down to a thickness~~ less than 100 µm ~~thick by~~ using ~~the~~ silicon carbide papers (240–1200 grit). ~~3 mm~~

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### 2.3. Mechanical Studies

To investigate the strength of the  $\gamma'$  precipitates, each sample was subjected to Vickers micro-hardness (HV) measurements ~~were carried out for each sample, using done with a~~ Rockwell Indentation Hardness Tester by ~~(Tecquipment ltd, to obtain insights on the strength of the  $\gamma'$  precipitates, Ltd.)~~. These measurements were ~~performed done by~~ using a diamond pyramid indenter with a force of 5 kgf (HV5). ~~The set of~~ values were recorded while moving from one end of the sample to the other (~~unit: units of~~ mm). ~~The To prevent overlap of the strain fields, the distance between each indentation was approximately  $\geq 3d$ , where  $d$  is the indent diameter (in either ~~in the~~ x or y direction), to prevent overlap~~

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~~of the strain fields.~~ Tensile specimens from each alloy were machined ~~as per to~~ the following dimensions: gage length ~~of~~ 22.5 mm, diameter ~~of~~ 6.35 mm, and width of the grip section ~~of~~ 12.5 mm, with M8 threaded ends. The specimens were tested ~~by~~ using an Instron tensile machine at a standard displacement rate (SDR) of  $2.22 \times 10^{-4} \text{ s}^{-1}$ . SEM ~~with energy dispersive x ray spectrometry (EDS)~~ was ~~conducted used~~ to determine the compositions of the various phases. ~~Scanning transmission electron microscopy (and STEM)~~ was used to identify ~~and obtain~~ the chemical constituents of the ~~nanoprecipitates~~ ~~nanoprecipitates~~. To simulate the precipitation kinetics of the  $\gamma'$  precipitate in the 945 and 945X alloys, ~~we used the JMatPro~~ thermo-kinetic software package ~~known as JMatPro~~ (version 8.04) ~~was used and compared~~ the results from ~~the simulation and experiments were then compared. The with~~ those from experiment. These simulations ~~helped provide~~ provided insights ~~on into~~ the size of the  $\gamma'$  precipitates ~~for~~, which ~~could affect~~ the mechanical properties of the alloy ~~could be affected~~.

## 2.3 Results and Discussion

### 3.1. Grain size after heat treatment

~~Light~~ Optical microscopy images were obtained for the ST, STA, and AR samples of each alloy. Fig-ure 1-(a) ~~is shows a typical image of alloys 945 and 945X, containing which contain~~ a  $\gamma$  matrix with grains and grain boundaries. ~~The nano scale~~ In the subsequent sections, the nanoscale  $\gamma'$  phase in the  $\gamma$  matrix is examined for each alloy sample ~~in the subsequent sections~~. Initially, an uneven microstructure was obtained by swabbing the samples with a cotton bud containing ~~Kalling's~~ Kalling's reagent (concentrated and dilute solutions); some grains were etched ~~while whereas~~ others remained under-etched with no clear distinction between ~~the~~ grain boundaries. By using colloidal silica (0.04  $\mu\text{m}$  in size) as the final polishing suspension, the grains were not only polished, but also slightly etched, ~~thus thereby~~ revealing the grain boundaries. Grain sizes were evaluated ~~by~~ using the intercept method, ~~by and~~ considering 10 ~~light optical~~ micrographs of each sample. ~~No significant variation in the grain size was observed, and an average grain size of 110  $\mu\text{m}$  was recorded for appeared, and each standard alloy specimen had an average grain size of 110  $\mu\text{m}$ .~~ However, due to ST at 1250  $^{\circ}\text{C}$ , the ~~grain size in the 945X NS sample had an increased grain size in the range of to~~ 140–150  $\mu\text{m}$ , and exhibited an intergranular phase after double aging, ~~as shown in [see Fig. 1-(b)-].~~

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The electron diffraction pattern (Fig. 6, left) for the 945 ST sample is more associated with a fcc  $\gamma$  lattice; however, due to the presence of small-sized  $\gamma'$  precipitates, they are more coherent with the matrix and not easily distinguishable. In contrast, for the 945X ST sample (Fig. 6, right), upon higher magnification (~~(Figs. 6(b) and 6(c)-)~~), the  $\gamma'$  precipitates ~~can be are~~ clearly distinguished from the  $\gamma$  matrix. This is further ~~evident from~~ supported by the electron diffraction spots ~~off from~~ the  $\gamma$  matrix

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and  $\gamma'$  precipitates. These very small precipitates give rise to secondary diffraction spots in the selected-area diffraction patterns, and are primarily associated with the presence of the  $\gamma'$  phase in the matrix.

**Fig. 4.** TEM images of 945 (left) and 945X (right) AR sample. Left: (a) Dark-field imaging—: Overview of  $\gamma'$  precipitates (appearing bright— which spots) that are distributed homogeneously throughout the  $\gamma$  matrix with selected-area diffraction pattern (SADP) corresponding to  $(\bar{1}10)$   $\gamma'$  phase. (b) Bright-field mode showing contrast image of  $\gamma'$  and  $\gamma$  matrix with a faint line around precipitates distinguishing  $\gamma'$  precipitates from  $\gamma$  phase and (c) at a higher magnification, clearly showing  $\gamma'$  appearing black in a spherical shape at bound 4–8 nm in diameter and in a compact— disc shaped ranging between shape about 10–20 nm in diameter. Right: 945X AR: (a) shows  $(0\bar{1}0)$   $\gamma'$  precipitates as grey precipitates in dark-field mode with the  $\gamma$  matrix appearing as dark grey. Panels (b) and (c) correspond to higher magnification, clearly showing  $\gamma'$  with spheroid shape (10–15 nm) and coalescence of  $\gamma'$  into aggregates 10–25 nm in size.

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Figure 7 shows the size distribution of  $\gamma'$  in the AR and STA samples— are presented in Fig. 7. The graph clearly indicates a peak position where the maximum number of  $\gamma'$  precipitates are found in any sample. The distribution is not a normal distribution, as there are because some secondary peaks appear at 0–5 nm, 10–15 nm, and 15–20 nm for each alloy. The error bars associated with the distribution data is  $\pm 0.5$  nm for each reading. The graph for the 945 AR and STA samples shows that the mean size range of  $\gamma'$  is between ranges from 6– to 8 nm, while whereas that for the 945X AR and STA samples shows that the mean  $\gamma'$  size range increases to ranges from 10– to 12 nm. It is important to note Note that, for each alloy, the curves for the AR samples nearly overlap with the curves for the STA samples, for each alloy. It can be assumed that the AR samples of each alloy have undergone the same heat treatment as the STA samples. A mean size difference of  $\sim 4$  nm between the 945 STA and 945X STA samples was recorded is found for  $\gamma'$ . It is important to consider here Note that it this is not the mean, but the average  $\gamma'$  size of each alloy, which allows us to predict their cutting or bowing phenomena under service stress and cathodic protection.

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As discussed earlier, the amount of Nb that is present in 945 and 945X is at bound 3.11 wt% and 4.14 wt%, as compared to which differs from its parent alloys Inconel 718 (5.06 wt%) and Incoloy 925 (0 wt%), respectively. This reduction in the Nb content aids in resisting helps to resist the

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precipitation of metastable  $\gamma''$  ( $\text{Ni}_3\text{Nb}$ ) and the stable  $\delta$  phase. This change in chemistry results in the precipitation of only the  $\gamma'$  phase at the ~~nano-scale, nanoscale~~ and is evident from the TEM images in Fig. 4-1.6 (see section 3.3.1). ~~This conclusion~~ is further supported by the findings of Slama and Abdellaoui [2428], who reported that  $\gamma'$  appears before  $\gamma''$  during isothermal aging at 680 °C. ~~The authors~~

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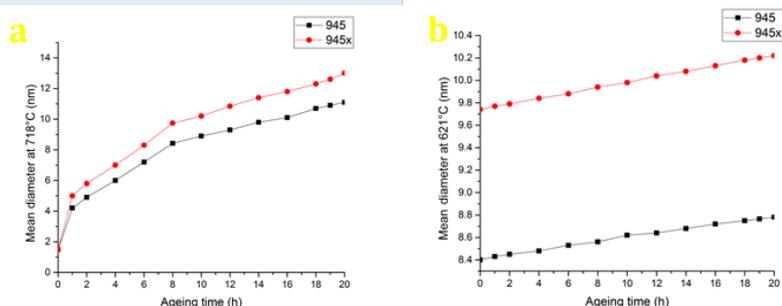
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~~This~~ increases further with the increase in ~~the~~ size of  $\gamma'$ . ~~In~~For the 945X samples, the  $\gamma'$  precipitates appear bright (Figs. 4 and 1.5), which could be attributed to the presence of higher-atomic-number elements ~~like~~such as Nb, which diffuse at the edges of the  $\gamma'$  precipitates to minimise the elastic strain caused by the lattice misfit between the  $\gamma'$  phase and  $\gamma$  matrixes [2832,2933].

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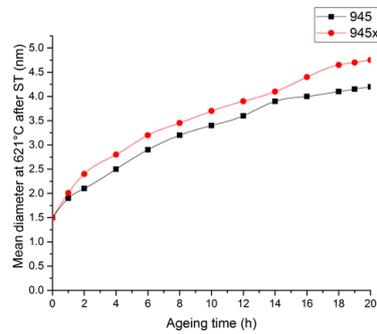
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The hardness values for the 945 and 945X ST samples are not surprising, ~~as~~since incomplete chemical ordering within the  $\gamma'$  precipitates and a small size contributes partially to the strength (see Fig. 10-; 330 MPa and 380 MPa, respectively) and hardness (140-HV5 and 155 HV5, respectively) of the alloys. The solvus temperature of the  $\gamma'$  precipitates is less than the solution temperature of 1038 °C [2326], resulting in dissolution back into the  $\gamma$  matrix. The average hardness value for each sample ~~obtained~~ is mostly due to the presence of small-sized  $\gamma'$  precipitates, which ~~are~~ precipitated/precipitate during ~~water-quenching~~WQ, as shown in Fig. 4, and MC-type carbides and nitrides ( $\text{NbC}$  and  $\text{TiN}$ ) ~~that are precipitated~~, which precipitate randomly throughout the structure, some of which dissolve into the  $\gamma$  matrix during solution treatment.



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**Fig. 10.** Mean  $\gamma'$  coarsening simulation of 945 (in black) and 945X (in red) STA samples ~~at~~for two -step aging ~~of~~at (a) 718 °C and (b) 621 °C. The simulation (c) shows the effect of aging at 621 °C on mean  $\gamma'$  coarsening after solution treatment.

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The secondary aging at 621 °C helps to achieve clear edge definition by fractionally increasing the  $\gamma'$  size, which enhances the hardness and 0.2% strength of the alloys. The effect of single and double aging on the morphology of  $\gamma'$  in ATI 718Plus ~~has been~~was studied by using TEM ~~and reported in [2832]. It would be~~An interesting experiment would be to ~~carry out~~apply TEM again ~~onto~~ both alloys, ~~and to~~ see the individual effects of each aging temperature after solution treatment. The  $\gamma'$  coarsening simulation shown in Fig. 11-(c) ~~demonstrates~~indicates the effect of individual aging at 621 °C for 8 h, showing an increase in ~~the~~mean size of ~~the~~ $\gamma'$  particles to ~3 nm, which is contrary to the fractional increase of ~0.20 nm after consecutive double aging. The ~~results of the~~ theoretical simulations are close to ~~the~~ experimental ~~observations~~results (TEM) and could be used as a benchmark for comparison.

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