Microstructural characterization of Haynes 282 after heat treatment and forging

C e e n a  J o s e p h

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Microstructural characterization of Haynes 282 after heat treatment and forging

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Abstract

Over the past 75 years, solid-solution strengthened superalloys have been one among the most widely used materials for long term applications at elevated temperatures. The combination of properties such as high temperature strength, resistance to oxidation and corrosion, fabricability, and creep strength make them an unusual class of materials, attracting researchers and scientists to explore its full potential. Among this group, the nickel superalloys find wide applications in aero engines and land-based gas turbines. They are still being modified in chemical composition to meet the increasing demands of aircraft and energy producing industry. One such newly developed Ni-base alloy is Haynes 282.

Haynes 282 showed sensitivity to heat treatment temperatures. The heat treatment temperatures were varied around the conventional heat treatment and within the typical tolerance limits. The microstructural development was systematically studied at intermediate stages through microscopy. To understand the influence of microstructural change on mechanical properties tensile testing was performed at room temperature. The gamma prime (γ') morphological change was observed to change from cuboidal to spherical to bimodal in three different heat treatment conditions. The carbide morphology changes from interconnected to discrete morphology. The strength of the material is affected by the size and shape of the cuboidal γ' precipitates, while the ductility at room temperature seem to be affected by interconnected morphology of the carbides at the grain boundaries.

Haynes 282 are used in different forms such as forgings, bars, sheets in component applications. The important aspect of such alloy is to understand the structure/property relations at in-service conditions. Haynes 282 in form of forgings showed ductility variations in short transverse direction (ST) from 24% to 12% as compared to its longitudinal transversal (LT) direction. The lower limit of ductility is close to the design tolerance and thus creates a need to understand the variation in ductility. In this part, the study is focused to understand ductility variation by microscopic investigations. The influence of carbide segregation and banding is seen to influence the ductility when oriented perpendicular to the tensile axis. This influence is also qualitatively captured through micromechanical modelling.

Keywords: Haynes 282, anisotropy ductility, heat treatment, microstructure, gamma prime, carbides
Preface

This licentiate thesis is based on the work performed at the Department of Materials and Manufacturing Technology at Chalmers University of Technology during the period June 2012-June 2015. During this period the work was performed within the project funded by Swedish National Aeronautical Research Program (NFFP6) under the supervision of Professor Christer Persson and Adj.Associate Professor Magnus Hörnqvist.

List of Appended papers

**Paper I:** Influence of heat treatments on the microstructure and tensile properties of Haynes 282 sheet material

C. Joseph, C. Persson, M. Hörnqvist

*In Manuscript*

**Paper II:** Influence of carbide distribution on ductility of Haynes 282 forgings

C. Joseph, M. Hörnqvist, R. Brommesson, C. Persson

*In Manuscript*

**Paper III:** Anisotropy of room temperature ductility in Haynes 282 forgings

C. Joseph, M. Hörnqvist, C. Persson

*Proceedings at the 8th International Symposium on Superalloy 718 and Derivatives Pittsburgh, USA. October 2014, pp 601-609*

Paper not appended to the thesis

3D grain structure modelling of intergranular fracture in forged Haynes 282

R. Brommesson, C. Joseph

*Submitted to international journal*
Contribution to the appended papers

My contribution to the appended papers

**Paper I**: The work was planned by me in collaboration with my supervisors. The experimental work was performed by me. Mechanical testing was done by Prof. Christer Persson. The paper was written by me in cooperation with the co-authors

**Paper II**: The work was planned together with GKN aerospace. I did the microscopy work and the modelling part was done by Rebecka Brommesson. I wrote the paper in cooperation with the co-authors.

**Paper III**: The work was planned together with GKN aerospace. I did the microscopy work and wrote the paper in cooperation with the co-authors.
1. Introduction

After the World War II, the gas turbines became an important technology for its application in land based power generation, aircraft industry and other industrial processes [1,2] The materials that were used earlier in the engine construction could not survive more than few hundred hours at high temperatures. This created the need for developing the new alloys to meet the demand for increased performance, reliability and emission in gas turbines.

Gas turbine engines deliver mechanical power using liquid fuel. In this process these components are responsible for mixing the air and fuel, create combustion and produce high temperatures up to 1400-1500 °C .This temperature window makes the materials and design for these components very critical for such applications. Thus, such application requires the material to have excellent mechanical strength, resistance to thermal creep deformation and fatigue, good surface stability and resistance to corrosion and oxidation. This unusual class of material, called superalloys, are attractive to scientists and researchers [1-3]. Figure 1 shows the temperature capability of superalloys since the beginning.

Based on the strengthening mechanism superalloys are classified into three groups.

- Nickel base (solid solution strengthening)
- Nickel -iron base (precipitation strengthening)
- Cobalt based (oxide dispersion strengthening)

Among the above, the nickel alloys find wide application in components used in aircraft engines, constituting over 50 % of its weight. The most common components are turbine blades, discs, seals, rings and casings of aero engines. The development of manufacturing processes produces alloy with uniform properties, less defects and less elemental segregation gives the possibility to improve the mechanical properties to a large extent [4]. Thus, the temperature capability of nickel alloys has now been improved considerably.
Outline of the thesis

This thesis focuses on characterizing a relatively new nickel base superalloy Haynes 282, after heat treatment and forging. The organization of this work begins with a general introduction to nickel base superalloy followed by introducing Haynes 282 and the research objectives. Thereafter, the experiment and characterization tools are described briefly. This is followed by discussion about important results, a summary of appended papers and intended future work.
2. Nickel-base superalloys

Nickel base superalloys are solution/precipitation strengthened alloys containing many alloying elements. These are complex engineered materials because they involve precipitation of intermetallic phases, called the gamma prime ($\gamma'$) and gamma double prime ($\gamma''$), and carbides such as MC (rich in Ti and Mo), $M_23C_6$ (rich in Cr) and other carbides like $M_6C$ (rich in Mo) and $M_7C_3$ [5-11]. The superior strength, high resistance to oxidation and corrosion, and creep properties of these alloys are essentially derived from the presence of these micro constituent phases [5]. The $\gamma'$ phase is coherent with gamma matrix ($\gamma$) and is an important constituent that contributes to the strength, while the carbides are incoherent to the matrix and are present at grain boundaries and intragranularly in the nickel alloys [1]. The alloying elements determine composition of the superalloy while the heat treatment is important for optimizing the properties. Each of them are subsequently discussed in this section.

2.1 Role of alloying elements

The matrix consists principally of Ni, Co, Cr and refractory metal such as Mo; the relative amounts of all these are determined by other elements such as Al, Ti, C and B which react to form precipitating phases. Alloying elements and their importance in nickel base superalloys has been reported widely in literature and is summarized in Table 1. Some of the critical elements such as Al and Ti are important for fabricability [12]. Lower levels of Mo (1 or 2 %) is deleterious as it can affect the creep strength of the material, while a minimum of 15-20 % of Cr is desirable for hot corrosion properties [6].

Table 1. Role of alloying elements in nickel alloys.

<table>
<thead>
<tr>
<th>Element</th>
<th>Amount found in Ni-base/Fe-Ni base alloys (wt%)</th>
<th>Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cr</td>
<td>5-25</td>
<td>Oxidation and hot corrosion resistance; carbides; solution hardening</td>
</tr>
<tr>
<td>Mo-W</td>
<td>0-12</td>
<td>Carbides solution hardening</td>
</tr>
<tr>
<td>Al-Ti</td>
<td>0-6</td>
<td>Precipitation hardening; carbides</td>
</tr>
<tr>
<td>C</td>
<td>0.02-0.10</td>
<td>Form carbides</td>
</tr>
<tr>
<td>Co</td>
<td>0-20</td>
<td>Affects amount of precipitate, raises $\gamma'$ solvus</td>
</tr>
<tr>
<td>Ni</td>
<td>rest</td>
<td>Stabilizes austenite; forms hardening precipitates</td>
</tr>
<tr>
<td>Ta</td>
<td>0-12</td>
<td>Carbides; solution hardening; oxidation resistance</td>
</tr>
<tr>
<td>Nb</td>
<td>0-4</td>
<td>Carbides; solution hardening; precipitation hardening</td>
</tr>
</tbody>
</table>
2.2 Heat treatment of Ni-base superalloys

Microstructure is basic to attain property requirement in superalloys [13]. The relation between microstructure and resulting mechanical properties is widely studied in both wrought and cast superalloys [14-28]. To derive its high temperature strength, and properties during in-service conditions, it is essential to control the microstructure via use of precipitated phases [14].

Ni-base superalloys are normally supplied in solution treated condition because they have optimum combination of properties for room temperature fabrication and elevated temperature service. However, by heat treatment it is possible to achieve

- Precipitation hardening
- Desired precipitation of carbide
- Optimum grain size through grain growth (in wrought and cast alloys), and through recrystallization and grain growth along with mechanical deformation (forging)

Figure 2 shows a schematic sketch for heat treatment in general for superalloys.

![Figure 2. Schematic sketch of heat treatment steps in superalloys](image)

In case of wrought Ni-base superalloy, solution treatment is performed to dissolve nearly all γ′ and carbides other than the stable MC carbides. Typical solution treatments are in the range of 1050 to 1200 °C followed by either air cooling or water quenching. On quenching super saturated solid solution is formed. The following two-step aging is done to precipitate γ′; the first often being carbide stabilization while the second for completing the precipitation of γ′.

In order to achieve desired properties, the heat treatment process has to be optimized. Factors such as cooling rate [15], aging temperatures and time [18], and solutionizing temperatures [25] are some of the heat treatment parameters that can alter the morphology of precipitated phases and thereby affect the properties of these alloys.
2.2.1 Carbide precipitation

One of the basic mechanisms for strengthening of wrought Ni-base alloy is carbide precipitation. The type of carbides formed depends on the alloying elements, while its morphology and distribution is affected by heat treatment. The temperatures and time for carbide precipitation are often carefully considered because these carbides undergo complex reactions, which can generate detrimental and beneficial effects by either changing to different forms of carbides or by changing their morphology. Carbides present at the grain boundaries in a desired morphology exhibit good creep strength and ductility, by inhibiting grain boundary sliding [5, 29].

Since carbide precipitation and distribution is very important in Ni-base alloy, the influence of carbide morphologies, type and distribution to high temperature properties has been studied and reported in the literature [28-49]. The three main forms of carbides reported in literature for Ni-base alloys are MC, M\textsubscript{23}C\textsubscript{6} and M\textsubscript{6}C. (Where M stands for metallic element) Table 2 shows the different morphologies of carbides formed in Ni-base alloys.

<table>
<thead>
<tr>
<th>Carbides</th>
<th>Morphology</th>
</tr>
</thead>
<tbody>
<tr>
<td>MC (Ti and Mo rich)</td>
<td>Blocky, discrete, script</td>
</tr>
<tr>
<td>M\textsubscript{6}C(Mo rich)</td>
<td>Discrete, acicular , platelets</td>
</tr>
<tr>
<td>M\textsubscript{23}C\textsubscript{6}(Cr rich)</td>
<td>Film, blocky, cellular, zipper</td>
</tr>
</tbody>
</table>

MC carbides are formed at higher temperatures during melting. They are generally insoluble carbides which precipitate in irregular shape and are believed not to influence the properties unless they are segregated [38, 48]. On thermal exposure, the carbides can undergo decomposition to form different stabilized states.

\[ MC + \gamma \rightarrow M\textsubscript{6}C + \gamma' \]

\[ MC + \gamma \rightarrow M\textsubscript{23}C\textsubscript{6} + \gamma' \]

M\textsubscript{6}C form of carbides is formed in the intermediate temperature range and enhance the property of the material only if present in desired discrete morphology. They are generally preferred instead of M\textsubscript{23}C\textsubscript{6} form of carbides, because of their stability at higher temperatures. [37]

M\textsubscript{23}C\textsubscript{6} usually precipitates at the grain boundaries in chromium rich alloys as irregular and discontinuous particles at temperatures between 760-980 °C. Continuous films of carbides can affect the ductility and, stress rupture of the material are avoided in this form [20, 31].

2.2.2 Gamma prime precipitation

Gamma prime precipitate is the principal strengthening phase in Ni-base alloys. The \( \gamma' \) precipitation is generally formed during cooling from the first aging step or during the second aging [29]. Temperature, time and cooling rates on heat treatment can affect \( \gamma' \) precipitation [50-53]. The morphology, distribution and volume fraction of \( \gamma' \) plays a very important role not only for strength but also for determining its fabricability [5, 12]. They are seen as cuboidal and spherical morphologies in different Ni-base alloys. The principal alloying elements that form these precipitates are Al and Ti. The \( \gamma' \) strengthened alloys have increased need for easy fabricability so that it can be readily welded and formed into various shapes. Hence, their addition is carefully balanced in order to get better fabricability and optimum hardening [29].
3. Haynes 282- A new fabricable superalloy

3.1 Introduction to Haynes 282

Haynes 282, is a relatively new wrought γ strengthened alloy which has been attracting interests for various applications due to a combination of properties like creep strength, thermal stability and fabricability [12,54]. The chemical composition of this alloy is as shown in Table 3.

<table>
<thead>
<tr>
<th>Ni</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>Fe</th>
<th>Mn</th>
<th>Si</th>
<th>C</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bal</td>
<td>19.44</td>
<td>10.22</td>
<td>9.42</td>
<td>2.15</td>
<td>1.44</td>
<td>0.92</td>
<td>0.06</td>
<td>0.07</td>
<td>0.067</td>
<td>0.004</td>
</tr>
</tbody>
</table>

The conventional heat treatment of this alloy is 1010 °C/2h/aircooled (AC) and 788 °C/8h/AC. As shown in Figure 3, the conventionally heat treated alloy has spherical γ precipitates and discrete carbide morphology at the grain boundaries.

![Figure 3. Scanning electron microscope (SEM) images of conventional heat treated Haynes 282 alloy showing (a) carbides (b) γ']

Haynes 282, since introduction into the market for high temperature applications, has been explored for its full potential. The studies on Haynes 282 include the oxidation and corrosion behavior, weldability, creep and fatigue on conventionally heat treated condition [54-57]. However, the effect of heat treatment parameters such as temperature, time and cooling rate is under research.

3.2 Basis for current research

3.2.1 Issues with heat treatment

In the interest to explore high temperature fatigue life of this new material, first part of the project was aimed at studying the low cycle fatigue behavior. However, in this process it was found that Haynes 282 had lower yield strength and shorter fatigue life at higher temperature than expected. Characterization of the tested specimen showed differences in carbide morphology at the grain boundaries. As shown in
Figure 3 (a), the alternative heat treatment, shows presence of grain boundary carbides as films, while in the conventional heat treatment it appears as blocky (brick wall) structure.

Figure 4. SEM images showing (a) Grain boundary carbides with film morphology, tested for fatigue at higher temperature (alternative heat treatment) (b) Grain boundary carbides with blocky morphology (conventional heat treatment)

Additionally, we see bimodal $\gamma$ precipitates intragranularly, and $\gamma'$ precipitates and discrete carbides at the grain boundary. As shown in Figure 5 (a), alternative heat treatment shows presence of bimodal $\gamma'$ precipitates and $\gamma$ at the grain boundaries. While, in the conventional heat treatment, as shown in Figure 5(b), we see uniform size of $\gamma'$ precipitates intragranularly and grain boundary with discrete carbides.

Figure 5. SEM image showing presence of (a) bimodal precipitation in alternative heat treatment (b) Spherical $\gamma'$ prime precipitates.

Due to lack of enough literature on heat treatment and indications that the material is sensitive to heat treatment parameters, the first part of this project was aimed to understand the microstructural development of Haynes 282. The important aspect of this study was aimed to correlate the microstructural changes to mechanical properties like tensile strength and ductility at room temperature.

### 3.2.2 Anisotropic ductility

Second part of the work focuses in understanding the ductility variations in Haynes 282 forgings after heat treatment. In this study we had the possibility to see microstructures of Haynes 282 forgings, bars and sheets. The grain size, and distribution of carbides in different forms are shown in Figure 6. In Figure 6(a), the forgings indicate presence of banded structure, with bimodal distribution of grain size and carbide stringers. In case of bar, the grain size is uniform with carbides uniformly distributed, see Figure 6(b). While, as shown in Figure 6(c), sheets show presence of carbide stringers in the rolling direction with uniformity in grain size. The mechanical test results on forging shows ductility variation
from 12 % to 24 % in the short transversal direction, while in the longitudinal direction it was unaffected. While, in case of sheets and bars, the ductility shows variations with heat treatment adopted.

![Figure 6. Optical microstructures of Haynes 282 (a) forging (b) bar (c) sheet](image)

3.3 Research Objectives

1. To understand the microstructural development in Haynes 282 and its sensitivity to heat treatment affecting the room temperature tensile properties.

2. To identify the cause for variation in ductility of Haynes 282 forgings and sheets.
4. Experimental details

4.1 Material

As discussed in Chapter 3, the material used in this study is Haynes 282. The heat treatments were done on mill annealed sheets of 3 mm thickness, and with grain size ASTM 3.5. In the course of anisotropic study we also had the possibility to check on other forms such as forgings and bars of Haynes 282.

4.2 Heat treatment

The Haynes 282 sheet, as received, was heat treated in air in a chamber furnace. The sheet was cut into small pieces before subjecting it to heat treatment schedules mentioned in Table 4.

<table>
<thead>
<tr>
<th>Referred as</th>
<th>Solution treatment</th>
<th>Aging step 1</th>
<th>Aging step 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solution treatment + aging (ST+A)</td>
<td>1120 C/2hr (WQ*)</td>
<td>1010 C/2hr (FC**)</td>
<td>788 C/8hr (FC)</td>
</tr>
<tr>
<td>Mill annealed + aging (MA+A)</td>
<td>-</td>
<td>1010 C/2hr (FC)</td>
<td>788 C/8hr (FC)</td>
</tr>
<tr>
<td>Mill annealed + low temperature aging</td>
<td>-</td>
<td>996 C/2hr (FC)</td>
<td>788 C/8hr (FC)</td>
</tr>
<tr>
<td>(MA+LTA)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*WQ- water quenched ** FC – furnace cooled

While, the anisotropy ductility study was done on forged specimens, heat treated according to AMS4951.

4.3 Test methods

4.3.1 Mechanical Testing

Tensile specimens were cut by water jet cutting and the room temperature testing was done in MTS servo hydraulic machine as per the ASTM standard E8

Forgings: The tensile testing was done by GKN aerospace as per the ASTM standard E8

4.3.2 Hardness

Hardness measurement were performed to determine the aging of material. The Vickers macrohardness test was done at 10kg load as per the ASTM standard E92, and the reported values in thesis are an average of 10 indentations.
4.3.3 Microscopy

A Leitz DMRX light optical microcopy equipped with Axio-vision software was used to study the microstructure on polished and etched samples. Additionally, SEM LEO 1550 was used for fractographic analysis and to observe the γ precipitates and carbide morphology under different heat treated condition.
5. Results

5.1 Heat treatment on Haynes 282 sheet

In order to study the sensitivity of Haynes 282 to heat treatment temperatures and to understand its subsequent impact on room temperature properties, the heat treatment schedules as shown in Table 4 were done on mill annealed specimens.

After heat treatment, the carbide morphologies at the grain boundaries changed. Figure 7 shows the different morphologies of grain boundary carbides. Figure 7(a) shows occasional presence of discrete grain boundary carbides in as received condition. On additional solutionizing followed by conventional heat treatment (ST+A), grain boundary shows interconnected morphology (Figure 7(b)). While, in MA+A condition, grain boundary carbides have discrete morphology, see Figure 7(c). However, as compared to ST+A and MA+A conditions, MA+LTA shows discrete grain boundary carbides and $\gamma'$ precipitates (cf. Figure 7(d)).

Additionally, $\gamma$ etching also showed morphological changes, as seen in Figure 8. Figure 8(a) shows no presence of $\gamma'$ in as received condition. In ST+A i.e. additional solutionizing followed by conventional
heat treatment $\gamma'$ precipitates are seen with cuboidal morphology (Figure 8(b)). While, MA+A shows presence of fine spherical $\gamma'$, see Figure 7(c). However, as compared to other conditions, MA+LTA shows bimodal $\gamma'$ precipitates (cf. Figure 7(d)), spherical and cuboidal morphology.

The morphological changes in grain boundary carbides and $\gamma'$ precipitate, were tested for its impact on room temperature tensile properties and hardness. The tensile test results along with hardness and morphological changes in $\gamma'$ is summarized in Table 5.

![Figure 8](image)

**Figure 8.** SEM images showing difference in carbide morphologies at the grain boundaries in 4 different conditions (a) As received condition: No $\gamma'$ seen (b) ST+A condition: cuboidal $\gamma'$ (c)MA+A condition: spherical $\gamma'$(d) MA+LTA condition: bimodal $\gamma'$ precipitates (small-spherical and coarse-cuboidal).

<table>
<thead>
<tr>
<th>Heat treatment condition</th>
<th>Room temperature</th>
<th>Hardness (HV)</th>
<th>$\gamma'$ morphology</th>
</tr>
</thead>
<tbody>
<tr>
<td>As received</td>
<td></td>
<td>212 ± 4</td>
<td></td>
</tr>
<tr>
<td>ST+A</td>
<td>650</td>
<td>1100±10</td>
<td>16±1 14</td>
</tr>
<tr>
<td>MA+A</td>
<td>760±8</td>
<td>1245±10</td>
<td>32±1 33</td>
</tr>
<tr>
<td>MA+LTA</td>
<td>765±8</td>
<td>1255±10</td>
<td>32±1 34</td>
</tr>
</tbody>
</table>
As seen in Table 5, the strength in ST+A condition is lower as compared to other heat treatments. From literature it is evident that $\gamma'$ precipitation gives strength to the material. A change in size of $\gamma'$ precipitates to coarse cuboidal morphology of 120 nm is affecting its room temperature YS and UTS. However, in MA+A- and MA+LTA- conditions, strength levels are similar.

The elongation is affected in ST+A condition and can be considered to be the effect of interconnected morphology of carbides at grain boundaries. The discrete carbide morphology does not affect the tensile ductility in MA+A and MA+LTA conditions, which is consistent with observations reported in literature. Hardness values are high for MA+A condition with fine spherical $\gamma'$ as compared to the coarse cuboidal precipitates in ST+A condition. However, the bimodal precipitate morphology for MA+LTA shows hardness in between the two conditions.

### 5.2. Ductility in forgings

In the ductility of forgings study, mechanical test on specimens from Haynes 282 forgings, shows similar values for YS, while their ductility and to some extent UTS changed. In this section, observations from representative samples are discussed. The fractography of tensile test specimens show intergranular failure as seen in Figure 9. Figure 9(a), shows a sample with intergranular failure and Figure 9(b) with presence of cracked MC carbide at grain boundaries, and presence of dimpled features on fractured surface indicating ductile matrix.

![Fractographs of tensile specimen showing (a) Intergranular failure (b) Presence of dimpled features, intergranular failure and cracked MC carbide at the grain boundary.](image)

Figure 9. Fractographs of tensile specimen showing (a) Intergranular failure (b) Presence of dimpled features, intergranular failure and cracked MC carbide at the grain boundary.
The fracture surface also showed presence of segregated M₆C and MC carbides as shown in Figure 10. In order to understand the segregation of carbides and their distribution, longitudinal sections of specimens were cut, polished and etched for microscopy. Figure 11(a) shows the longitudinal section of a sample just below the fracture surface, indicating cracks along a segregated carbide region. A region with segregated carbides, shows presence of M₆C, MC carbides and carbo nitrides, as seen in Figure 9(b). These stringers were observed to be either 90°, inclined or along the tensile axis direction in investigated specimens.

Figure 11(c) shows carbides of size almost similar to the size of smaller grains. As shown in Figure 12, γ' precipitates are seen to be distributed uniformly in matrix. Figure 12(a) shows presence of bimodal distribution of γ'; coarse- intragranularly, and fine close to grain boundaries. It also shows presence of cracked MC carbide at the grain boundary. While, in one of the forgings heat treated conventionally, γ' is uniformly distributed in size and shape intragranularly and near grain boundaries, see Figure 12(b).
Figure 12. SEM images showing (a) Uniform distribution of spherical γ' and crack in MC carbide at the grain boundary in forging (heat treated according to AMS 5951) (b) Distribution of very fine γ' near grain boundary and intragranularly (conventional heat treatment).

Figure 13. Optical microscopic images showing carbide stringer and bimodal distribution of grains in specimens from short transversal (ST) direction (a) Specimen with 12 % elongation: showing carbides perpendicular (white arrow) to tensile axis direction (black arrow) (b) Specimen with 16 % elongation: showing carbides along (white arrow) the tensile axis direction (black arrow)

The optical microscopy of specimen shows presence of smaller grains in regions where carbide segregations are observed while regions outside of carbide segregation are coarse grains, see Figure 13. Figure 13(a) is an optical image from a specimen in ST direction from forging, and measured ductility of 12 %. The carbide distribution were seen 90° to the tensile axis (black). In Figure 13(b), a specimen with 16 % elongation shows presence of carbide stringers along the tensile axis direction. While specimens from LT direction had uniformly distributed carbides along the tensile axis direction, see Figure 14.
5.3 Summary of appended papers

This section is aimed to summarize the results in appended papers. Paper 1 deals with the heat treatment on Haynes 282 sheet material. Paper 2 and Paper 3 are about forgings studied for their anisotropic ductility through microscopic investigation.

5.3.1 Paper 1

In this study, Haynes 282 shows sensitivity to heat treatment temperatures by forming different morphologies of grain boundary carbides and γ' precipitates as seen in Figure 7 and Figure 8 respectively. The conventional heat treatment for Haynes 282 (1010 °C/2h/AC) and (788 °C/8h/AC) produces a microstructure with fine γ' precipitates as shown in Figure 3.

For the standard heat treatment (i.e. MA+A condition), the γ’ precipitates were fine and of spherical morphology. Additional solutionizing (i.e. ST+A) leads to an increase in size of γ’ precipitates, and an associated change to cuboidal shape, which lowers the YS and UTS of the material. The change to cuboidal morphology, could be due to the fact that longer solutionizing time at 1120 °C, reduced the density of dislocations after recovery process which has led to fewer nucleation points and coarser precipitates. By reducing the temperature to 996 °C during the first aging step (i.e. MA+LTA), a bimodal distribution of γ’ with two different morphologies was observed – small spherical and large cuboidal. This is because 996 °C is below the γ’ solvus temperature for Haynes 282, and hence nucleates γ’ precipitates at this temperature and on further cooling they grow to cuboidal shape. On subsequent aging at lower temperatures further nucleation of smaller γ’ with spherical morphology occurs.

When adding the solutionizing step, the grain boundary carbide changes from discrete particulate morphology seen after standard treatment, to an interconnected structure, which led to a significant drop in room temperature tensile ductility. The secondary carbides are brittle and being interconnected makes it even more detrimental. On cracking of grain boundary carbides, its interconnected morphology further accelerates the crack propagation thereby leading to premature failure. On lower ageing temperature, produced a mix of carbide and γ’ at the grain boundaries, but the secondary carbides still seen with their discrete morphology and therefore does not affect the room temperature tensile properties. Thus this
study shows that slight variations in aging temperature influence mechanical properties due to microstructural changes.

5.3.2 Paper 2 and 3

In this study, tensile test results show that ductility changes from 12 % to 24 %. The fractography shows intergranular failure and presence of segregated MC and $M_6C$ carbides which are brittle, see Figure 9 and 10. Metallographic investigations show presence carbide stringers and bimodal grain size distribution. Figures 13 and 14 show preferential orientation of the carbide bands with respect of tensile axis direction.

The $\gamma'$ precipitates are uniformly distributed in size and morphology, so the heat treatment adopted gives similar YS. While, UTS also shows anisotropy to some extent. The formation of carbide stringers during forging and subsequent heat treatment is due to local elemental segregation in the ingot. The MC and $M_6C$ carbides are brittle. These brittle carbides initiates and propagates crack under loading conditions. They also pin the grain boundaries and gives bimodal grain size distribution. The preferential alignment of carbide stringers and bimodal distribution are the microstructural inhomogeneity, which influences the measured tensile ductility. Measured ductility is thus anisotropic and inhomogeneous due to the preferential orientation of carbide stringers, which is also qualitatively confirmed in the modelling attempt, where the orientation of carbides $45^\circ$ to the tensile axis shows maximum ductility as compared to that inclined at $90^\circ$. 
6. Suggestions for future work

From the heat treatment and forging study we see that Haynes 282 is sensitive to heat treatment temperatures, which changes both carbide morphologies and $\gamma'$ precipitation. The microstructural changes are also seen to affect the room temperature properties. Therefore, we aim to develop a better understanding on the microstructural development by varying few heat treatment parameters such as:

- Solutionizing temperature, time and cooling rate
- Aging temperature, time and cooling rate

And also to study their effects on room and high temperature mechanical properties like strength, ductility both at room temperature and high temperature.

We also aim to understand the secondary carbides formed at grain boundaries and their influence on ductility.
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“Sometimes the Bad Things that happen in our lives put us directly on the path to the Best things that will ever happen to us” ☺
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