Microstructural Characterization Of Intermetallic Precipitates In Inconel 718 Alloy By DC Electrical Resistivity Measurements

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Abstract

Inconel 718 is a Ni-Fe base high strength superalloy; which can be age hardened by precipitation hardening. Inconel 718 in wrought condition contains non uniform distribution of \(\gamma', \gamma'', \delta\) and carbides in an austenite matrix \(\gamma\). The aim of current investigation is to characterize different microstructural features, evolved though various heat treatments, using DC electrical resistivity measurements and correlate it with the variation in hardness. Precipitation can cause variation in resistivity due to the various scattering caused during the rearrangement, while formation of the precipitates. Thus this study in effect can be used as a potential non destructive tool for evaluation of the microstructures on ageing.

Introduction

Superalloys are a group of alloys that exhibit high strength at high temperatures. Precipitation strengthened alloys containing substantial quantities of both nickel and iron form a distinct class of superalloys, known as Ni-Fe base superalloys; hardened by intermetallic or grain boundary precipitates mostly in the form of \(\gamma', \gamma'', \eta, \delta\) and MC. Precipitation hardening is produced by solution treating and quenching an alloy in which a second phase is in solid solution at the elevated temperature but precipitates on quenching and ageing at a lower temperature. The degree of strengthening resulting from the second phase particles depends on the distribution of particles in the matrix. \(\gamma'\) phase is an intermetallic phase mostly containing Al and Ti. In \(\gamma''\) phase, Ni and Nb combine in the presence of Fe as a catalyst to form Body Centered Tetragonal (BCT) \(\text{Ni}_3\text{Nb}\) which is consistent with \(\gamma\) phase. This phase provides high strength at low and intermediate temperature, but it is unstable above 650°C.

IN-718 is strengthened by \(\gamma''(\text{Ni}_3\text{Nb})\). IN-718 also contains smaller quantities of Al and Ti which leads to the formation of \(\gamma'\) \(\text{Ni}_3(\text{Al},\text{Ti})\) phase. Along with these two phases; during the extended service exposure at higher temperature in the range of 650°C-750°C, there is the possibility of stable orthorhombic delta \(\delta\) phase formation. Carbides with MC (M=Ti, Mo, V, Cb, Ta, Zr) type may also precipitate at the grain boundaries of IN-718 during processing, heat treatment or in-service [1,2].

IN-718 is an alloy with many desirable mechanical properties such as high yield and ultimate tensile strength; good creep and rupture strength; and high resistance to fatigue. It also possesses long time strength and toughness at higher temperature along with confinement of corrosion and oxidation resistance up to elevated temperature. It has lower thermal conductivity or higher
electrical resistivity. It is most commonly used in gas turbine applications and can also be used in various other applications where high temperature and severe operation conditions result in limit of choice of material.

Depending upon temperature and time of exposure along with the amount of alloying elements; there will be change in the intermetallic and grain boundary precipitation behavior; leading to change in various properties of the alloy. So it is necessary to characterize various intermetallic phases using NDE method.

Measuring material's conductivity or resistivity non destructively helps in identifying various metals and alloys along with the detection of damage that gives rise to a change in conductivity [3,4]. Electrical resistivity of naturally aged alloy increases during precipitation hardening due to the formation of small solute clusters which uses notable scattering of the conduction electron.[5] Four point direct current potential drop technique is well suited for accurate non destructive measurement of material resistivity. The potential drop method of evaluation is based on the principle that the electrical resistance of an alloy changes due to the presence of structurla inhomegeneties.

The aim of present investigation was to study the precipitation behavior in Ni-Fe base superalloy IN 718 by DC electrical resistivity measurement and validate the same with hardness and microstructural features.

Experimental

Table 1, shows the chemical composition of the IN-718 used in present study. Time Temperature Transformation (TTT) diagram for IN-718 [6] is shown in Fig. 1. The heat treatment cycle used for IN-718 was solution annealing (SA) at 980°C for 1 h and then water quench to room temperature. [7] Subsequently the solutionised samples were aged at different temperatures and duration of time. With reference to the TTT diagram (Fig.1) of IN-718 solution annealed specimens of dimensions 25mm x 29mmx 10mm were aged for microstructural variations.

For resistivity measurement, four probe Van der Pauw method has been used. In this method, electrical DC currents are injected into a conducting specimen though two probes; while second set of two probes was used for measuring the voltage drop across the area of contact; as shown in Fig. 2. After an accurate measurement of the distance between the voltage probes, the resistivity ($\rho$) of the sample is measured using Eq. 1.

$$\rho = \frac{RA}{L}$$

where $R$=resistance or voltage drop when a constant current is passed.

$A$=cross sectional area of contact of the sample.

$L$=distance between the voltage probes.

For better accuracy and speed of measurement, a sample holder has been used; in which four contacts are made by pressure contacts with gold tips. Keithley 2400 Sourcemeter is used as constant current source; while Keithley 2182A Voltmeter is used for measurement of the voltage drop. Sample holder for the same is shown in Fig. 3.

Hardness measurements were carried out using Indentation method at a load of 700N. An average of thee readings is reported here. A maximum change of $\pm 5$ BHN was obtained in hardness measurements in any specimen. Image analyzer and Scanning Electron Microscopy were carried out to study the precipitation behavior, and validate the resistivity and hardness measurement.
Table 1-Chemical composition of Inconel 718

<table>
<thead>
<tr>
<th>Element</th>
<th>Ni</th>
<th>Fe</th>
<th>Cr</th>
<th>Ti</th>
<th>Al</th>
<th>Nb</th>
<th>Mo</th>
<th>Co</th>
<th>C</th>
<th>Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt%</td>
<td>53.30</td>
<td>17.66</td>
<td>19.21</td>
<td>1.03</td>
<td>0.4</td>
<td>5.06</td>
<td>2.92</td>
<td>0.01</td>
<td>0.04</td>
<td>&lt;0.10</td>
</tr>
</tbody>
</table>

Results and Discussions:

A set of specimens are aged as per the schedule given in Table-2. Table 2 and Fig. 4 to Fig. 7 show the variations in the hardness and resistivity with different ageing cycle. The SA specimen exhibited lowest hardness (165 BHN) and it is increased with increase in ageing temperature.
from 600°C to 750°C; while there is decrease in hardness on further increase of temperature from 750°C to 900°C. On ageing at 650°C; hardness was found to continuously increase with increase in time of exposure (10, 25, 50, 75, and 100). While on ageing at 750°C, and 800°C; hardness was initially found to increase with increase in time of exposure and further exposure decreases the hardness values. On the other hand for ageing at 900°C it is decreasing continuously. This is in agreement with the expected phase diagram [6]. The increase in strength by ageing is due to the presence of fine and uniformly distributed metastable phase γ" in the matrix. While at higher temperature above 750°C there is decrease in the strength due to rapid coarsening of γ". At 800°C it is expected from the TTT diagram that for longer time of exposure; nucleation of stable orthorhombic δ phase will start from γ". δ phase degrade the strength of the alloy by consuming the precipitation strengthening elements. And this is one of the reasons for decrease in hardness at 800°C and 900°C for longer exposure time. Along with the formation of delta at the higher temperature there will be the formation of MC type grain boundary carbides which will also affect the hardness of the alloy.

For the precipitation hardening [8,9] to occur, the second phase must be soluble at an elevated temperature but must exhibit decreasing solubility with changing temperature. There is atomic matching or coherency present between the lattices of the precipitates and the matrix. After quenching from solution annealing temperature, the alloy will contain uniform solid solution of γ matrix. On further ageing solute segregation will take place in the alloy resulting in increase of the hardness. With additional ageing the hardness was increased further by ordering of larger clumps of Nb atoms on the planes of the γ matrix. This structure is known as γ" having definite precipitate platelets of Ni₃Nb, which are coherent with the matrix, leading to increase strain field in the matrix. This strain field extends over a distance more than the size of the precipitate and will thus be responsible for increase in the hardness as it interferes with dislocation movement. With still further ageing the stable orthorhombic phase δ was formed from the metastable phase γ"; which is no longer in coherency with the matrix and therefore the hardness was decreased. Continued ageing beyond this stage for longer time duration resulted in particle growth and further decrease in the hardness. It is common for coherent precipitate to form and then loose the coherency when the particle grows to a critical size. In addition to shape, the precipitates can be described by specifying the volume fraction, average particle diameter and mean interparticle spacing.

Resistivity is the ability of electrons to move freely in the alloy. Resistivity increases with respect to increase in the rate of precipitation. As mentioned early, additional ageing will lead to the formation of ordered clusters and precipitates; which will result in significant scattering of conductive electrons as they vibrate around equilibrium positions at higher temperature [10]. From an empirical relationship, it is known that changes in the resistivity of the metals are depended by the changes to the mean time between the conduction electron collisions (τ) which will further depend on the solute content and precipitate density formed. The resistivity is thus affected by increase in conduction electron scattering due to formation of fine precipitates, by increasing the resistivity to maximum during initial nucleation of the precipitation phase (due to decrease in conduction electron mean free path) and also affected by decrease in conduction electron scattering due to dissolution of the precipitates from the matrix (due to increase in conduction electron mean free path). Thus resistivity is sensitive to initial nucleation of the precipitates, their growth and coarsening, and finally their dissolution in the matrix.

Based on the experimental work, it was observed that resistivity is affected by the cluster formation; which is again dependent on the ageing time and temperature. Resistivity in short varies as a result of combined effects from solution depletion, intermetallic precipitates, high temperature solute enrichment due to the dissolution of the clusters formed during ageing and
also refining of the intermetallic precipitates. Change in resistivity at higher temperature will also be there due to the presence of preexisting intermetallic precipitates, as they affect the rate of electron scattering and also by forming coarse grain structure which will slower down the kinetics of the precipitation by reducing nucleation rate [11]. Due to the increasing precipitate to the equilibrium volume fraction at lower temperature, the resistivity shows similar peak positions in the maximum increased resistivity. Bulk resistivity decreases on increase of ordering of the material. There is an inverted C-curve type response that would be expected for a precipitation reaction. In effect, it is seen that in the present alloy IN-718, also the formation of GP zones (θ' and θ'') is observed from a study of the DC Electrical resistivity.

Fig. 8A-C show the microstructures corresponding to the SA specimens thermally aged at 650°C (25 h) + 620°C (8 h) + AC, 750°C (75 h) + 620°C (8 h) and 900°C (100 h). It can be clearly seen from micrograph (Fig. 8A) that there is uniform distribution of precipitates in the matrix at 650°C (25 h). Whereas at 750°C (75 h) it is found that there is extensive precipitation of γ' and γ" intermetallic phases. A scanning electron micrograph of the SA specimen thermally aged at 900°C for 100 h exhibits the presence of orthorhombic δ-phase, with nearby locations free from γ" phase. This clearly indicates that the precipitation of δ-phase occurs at the expense of the γ" phase as the γ" phase is in metastable state while δ-phase is in stable state and also the chemical composition of both the phases are almost similar.

<table>
<thead>
<tr>
<th>Ageing Cycle</th>
<th>Sample Designation</th>
<th>Expected Phase</th>
<th>Hardness [BHN]</th>
<th>Resistivity [Ωm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>980°C for 1h (SA)</td>
<td>R1</td>
<td>γ</td>
<td>165</td>
<td>1.2467*10^-6</td>
</tr>
<tr>
<td>SA + 650°C 10h + 620°C 8h</td>
<td>R2</td>
<td>γ</td>
<td>260</td>
<td>1.2539*10^-6</td>
</tr>
<tr>
<td>SA + 650°C 25h + 620°C 8h</td>
<td>R3</td>
<td>γ</td>
<td>298</td>
<td>1.1476*10^-6</td>
</tr>
<tr>
<td>SA + 650°C 50h + 620°C 8h</td>
<td>R4</td>
<td>γ</td>
<td>328</td>
<td>1.1236*10^-6</td>
</tr>
<tr>
<td>SA + 650°C 75h + 620°C 8h</td>
<td>R5</td>
<td>γ</td>
<td>360</td>
<td>1.0634*10^-6</td>
</tr>
<tr>
<td>SA + 650°C 100h + 620°C 8h</td>
<td>R6</td>
<td>γ' + γ&quot;</td>
<td>377</td>
<td>1.0764*10^-6</td>
</tr>
<tr>
<td>SA + 750°C 10h + 620°C 8h</td>
<td>R7</td>
<td>γ' + γ&quot;</td>
<td>385</td>
<td>1.0462*10^-6</td>
</tr>
<tr>
<td>SA + 750°C 25h + 620°C 8h</td>
<td>R8</td>
<td>γ' + γ&quot;</td>
<td>378</td>
<td>0.9913*10^-6</td>
</tr>
<tr>
<td>SA + 750°C 50h + 620°C 8h</td>
<td>R9</td>
<td>γ' + γ&quot;</td>
<td>358</td>
<td>0.9672*10^-6</td>
</tr>
<tr>
<td>SA + 750°C 75h + 620°C 8h</td>
<td>R10</td>
<td>γ' + γ&quot; + δ</td>
<td>349</td>
<td>1.0131*10^-6</td>
</tr>
<tr>
<td>SA + 800°C 25h + 620°C 8h</td>
<td>R11</td>
<td>γ' + γ&quot; + δ</td>
<td>328</td>
<td>1.0007*10^-6</td>
</tr>
<tr>
<td>Temperature</td>
<td>Heat Treatment</td>
<td>Phase</td>
<td>Hardness</td>
<td>Resistivity (*10^-6) (ohm m)</td>
</tr>
<tr>
<td>-------------</td>
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<td>-------</td>
<td>----------</td>
<td>-----------------------------</td>
</tr>
<tr>
<td>SA + 800°C</td>
<td>50h + 620°C 8h</td>
<td>R12</td>
<td>$\gamma' + \gamma'' + \delta$</td>
<td>325</td>
</tr>
<tr>
<td>SA + 800°C</td>
<td>75h + 620°C 8h</td>
<td>R13</td>
<td>$\gamma' + \gamma'' + \delta$</td>
<td>309</td>
</tr>
<tr>
<td>SA + 900°C</td>
<td>75h</td>
<td>R14</td>
<td>$\delta$</td>
<td>208</td>
</tr>
<tr>
<td>SA + 900°C</td>
<td>100h</td>
<td>R15</td>
<td>$\delta$</td>
<td>206</td>
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</table>

Fig. 4 Variation in Resistivity and hardness with SA + 650°C

Fig. 5 Variation in Resistivity and hardness with SA + 750°C

Fig. 6 Variation in Resistivity and hardness with SA + 800°C

Fig. 7 Variation in Resistivity and hardness with SA + 900°C
Conclusion:

The precipitation behavior in IN-718 has been studied using resistivity and hardness measurements. The influence of precipitation on hardness can be felt only after precipitates reach to critical size which can affect the dislocation movement. The resistivity increases with the formation of $\delta$-phase at higher temperature and for longer time of exposure as $\delta$-phase is much softer than that of $\gamma$ matrix. The present also shows that the resistivity study is sensitive to the structural variations that occur on ageing. The hardness variation on ageing occurs much later the resistivity variation consistent with the critical size of the precipitate needed as a barrier for dislocation movement.

References:


