Quantification of precipitates and their effects on the response of nickel-base superalloy to shot peening

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Abstract
This paper reports on a microstructural study of a nickel-base superalloy, Inconel 718, with a focus on quantifying precipitate density and their effects on conductivity variations. The study is motivated by eddy current (EC) characterization of residual stresses, where observed EC signals are attempted to correlate with stress profiles of shot peened superalloy surfaces. It has been observed that the correlation is less universal than anticipated, and in fact strongly influenced by the material hardness, or the aging conditions. For example, the soft sample surface exhibits significantly stronger EC signals than the fully hardened sample when both are shot peened at the same Almen intensity. Thus, the objective of the present study is to examine this complex material response against aging and shot peening treatments at the microstructure scale, by the use of techniques such as transmission electron microscopy (TEM) and scanning electron microscopy (SEM). We will describe preparations of a series of Inconel 718 samples that was aged and shot peened at various conditions, and present microstructural data obtained from SEM and TEM images such as precipitate densities, correlated with bulk properties such as the hardness and conductivity.

Keywords
hardness, internal stresses, nickel alloys, precipitation hardening, scanning electron microscopy, shot peening, superalloys, transmission electron microscopy, nondestructive evaluation, QNDE, Materials Science and Engineering

Disciplines
Materials Science and Engineering

Comments
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QUANTIFICATION OF PRECIPITATES AND THEIR EFFECTS ON THE RESPONSE OF NICKEL-BASE SUPERALLOY TO SHOT PEENING

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ABSTRACT. This paper reports on a microstructural study of a nickel-base superalloy, Inconel 718, with a focus on quantifying precipitate density and their effects on conductivity variations. The study is motivated by eddy current (EC) characterization of residual stresses, where observed EC signals are attempted to correlate with stress profiles of shot peened superalloy surfaces. It has been observed that the correlation is less universal than anticipated, and in fact strongly influenced by the material hardness, or the aging conditions. For example, the soft sample surface exhibits significantly stronger EC signals than the fully hardened sample when both are shot peened at the same Almen intensity. Thus, the objective of the present study is to examine this complex material response against aging and shot peening treatments at the microstructure scale, by the use of techniques such as transmission electron microscopy (TEM) and scanning electron microscopy (SEM). We will describe preparations of a series of Inconel 718 samples that was aged and shot peened at various conditions, and present microstructural data obtained from SEM and TEM images such as precipitate densities, correlated with bulk properties such as the hardness and conductivity.

Keywords: Superalloy, Conductivity, Precipitate Density, Shot Peening

INTRODUCTION

Shot peening is a widely used surface treatment to improve fatigue resistance of the components in aircrafts engines, by introducing near-surface, protective compressive residual stresses [1]. Eddy current (EC) method has been studied regarding its potential for measuring subsurface and surface residual stress based on the empirical piezoresistivity effect [2-6]. It has been reported that shot peening-induced change in conductivity measured using the EC method is affected by the microstructure of the material [5, 7-8]. The objective of this study is to understand how the microstructure of the samples affect the eddy current measurements. This paper talks about the quantification of size and concentration of the secondary phase precipitates (γ', γ'', δ and metal carbides) and their effects on the conductivity of Inconel 718. This study is approached by preparing a series of heat treated
samples with different microstructures. Heat treated samples were shot peened at multiple Almen intensities. Swept frequency eddy current (SFEC) measurements were performed on the heat treated samples before and after the shot peening. Sample microstructures were studied from SEM and TEM images. Bulk conductivities and hardness values of the samples were also measured.

**EXPERIMENTAL PROCEDURE**

A series of Inconel 718 samples were heat treated according to the Time-Temperature-Transformation (TTT diagram) (Fig. 1) to produce various microstructures with different amounts of secondary phase precipitates. The heat treatment conditions are shown in Table I.

Sample I was kept in the as-received condition, while the others were first solutionized at 1024°C for 0.5 hour and then quenched. Sample II was kept in the solutionized condition. The others were aged under different conditions as given in Table I.

**MICROSTRUCTURAL CHARACTERIZATION**

Strips were cut out of each heat treated sample to make coupons, which were then polished and etched for microstructure characterization using an FEI Quanta-250 field-emission scanning electron microscope (SEM). Thin foils were prepared by dimpling, followed by jet polishing into perforation, and examined by a Tecnai G2 F20 and a Phillips CM30 transmission electron microscopes (TEM).

![Time-Temperature-Transformation diagram for Inconel 718](image)

**FIGURE 1.** Time-Temperature-Transformation diagram for Inconel 718 [9]. [Reprinted with permission of ASM International. All rights reserved. www.asminternational.org]
TABLE I. Seven heat-treatment conditions of the Inconel 718 samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heat Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>As-received(solutionized)</td>
</tr>
<tr>
<td>II</td>
<td>Solutionized at 1024°C/0.5hr</td>
</tr>
<tr>
<td>III</td>
<td>Solutionized, aged at 620°C/10hrs</td>
</tr>
<tr>
<td>IV</td>
<td>Solutionized, aged at 680°C/50hrs</td>
</tr>
<tr>
<td>V</td>
<td>Solutionized, aged at 718°C/8hrs, furnace-cooled to 621°C, aged at 621°C/8hrs</td>
</tr>
<tr>
<td>VI</td>
<td>Solutionized, aged at 850°C/10hrs</td>
</tr>
<tr>
<td>VII</td>
<td>Solutionized, aged at 900°C/20hrs</td>
</tr>
</tbody>
</table>

The secondary precipitates in Inconel 718 are γ’, γ’’ and δ precipitates, of which γ’’ is considered to be the primary strengthening precipitate. The expected microstructures from each condition are listed in Table II. In Inconel 718, γ’ is spherical in shape having an ordered fcc structure, and γ’’ is disc in shape having a bct structure, while δ has an orthorhombic structure [9]. A subset of the seven sample conditions was studied and reported here, i.e. the solutionized, peak-aged and over-aged conditions. Figure 2 shows the microstructure obtained from few conditions, which correlates with expected microstructure given by Table II.

TABLE II. Expected microstructure from each heat treatment condition.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Expected Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>Homogeneous microstructure free of γ’, γ’’ and δ precipitates</td>
</tr>
<tr>
<td>II</td>
<td>Homogeneous microstructure free of γ’, γ’’ and δ precipitates</td>
</tr>
<tr>
<td>III</td>
<td>Small amount of γ’ and γ’’ precipitates</td>
</tr>
<tr>
<td>IV</td>
<td>Large amount of γ’ and γ’’ precipitates</td>
</tr>
<tr>
<td>V</td>
<td>Homogeneous mixture of γ’ and γ’’ throughout the matrix</td>
</tr>
<tr>
<td>VI</td>
<td>γ’’ precipitates will coarsen and gets converted into δ precipitates</td>
</tr>
<tr>
<td>VII</td>
<td>Predominate δ precipitates</td>
</tr>
</tbody>
</table>
FIGURE 2. Micrographs of heat treated Inconel 718 samples. (a) An SEM image of Sample II. (b) A dark field TEM image showing γ” precipitates. c) An SEM image of Sample VI showing an overaged microstructure. (d) An SEM image of Sample VII showing copious δ platelets and γ” in grain interior.

QUANTIFICATION OF PRECIPITATES

The micrographs taken by SEM and TEM were processed by the commercial software packages (e.g. ImagePro). The contrast and brightness of the images was enhanced and then the secondary precipitates alone were highlighted (Fig. 3). The apparent sizes (i.e. diameter for γ’, diameter and disc thickness for γ”) and volume fraction of the secondary precipitates were calculated [10] for each images and average apparent sizes and average volume fraction of the secondary precipitates were calculated. Figure 3a shows an unprocessed SEM micrograph of sample VII. Figure 3b shows the processed image using ImagePro software.
FIGURE 3. (a) An SEM micrograph with high contrast and brightness. (b) Image processed using ImagePro software.

TABLE III. The sample hardness (in Rockwell C scale), bulk electrical conductivity(%IACS) and volume fraction of all these secondary phase precipitates(%). (100%IACS = 5.8×10^7 S/m)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Hardness HRC</th>
<th>Bulk electrical conductivity (%IACS)</th>
<th>Volume fraction(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>II</td>
<td>8.0*</td>
<td>1.381 ± 0.001</td>
<td>NA</td>
</tr>
<tr>
<td>V</td>
<td>42.9</td>
<td>1.483 ± 0.001</td>
<td>7.53 ± 2.36</td>
</tr>
<tr>
<td>VI</td>
<td>26.1</td>
<td>1.464 ± 0.001</td>
<td>17.26 ± 5.19</td>
</tr>
<tr>
<td>VII</td>
<td>20.4*</td>
<td>1.428 ± 0.003</td>
<td>17.09 ± 9.94</td>
</tr>
</tbody>
</table>

*Hardness is measured using Rockwell B-scale and converted into Rockwell C-scale.

RESULTS AND DISCUSSION

The bulk electrical conductivities of the samples were measured using a conductivity gage operated at 60 kHz. Hardness of the samples was measured and reported in Rockwell C-scale. Both the bulk electrical conductivities and hardness of the samples are tabulated in Table III.

The relation between the hardness and bulk electrical conductivities of the samples is shown in Fig. 4. The observed correlation between the conductivity and hardness can be interpreted in terms of the formation and growth of the secondary phase precipitates. During the formation of the secondary phase precipitates (γ’, γ” and δ), solute elements are removed from the matrix, resulting in reduced electron scattering by the impurity atoms in the solid solution.
The precipitates also hinder the dislocation motion and thus increase the hardness and yield strength of the material. Such effect is indicated in Fig. 5, which shows that as the volume fraction of the secondary phase precipitates increases, the bulk conductivity of the samples increases.

FIGURE 4. Bulk conductivity (% IACS) plotted against hardness in Rockwell C scale.

FIGURE 5. Bulk conductivity plotted against the volume fraction (%) of the secondary phase precipitates.
Figure 6 relates the average size of the precipitates and hardness of the samples. The increase in yield strength of the material $\Delta \sigma$ is related to the size and volume fraction of the precipitates by [11],

$$
\Delta \sigma \propto F^{0.5} r^{0.5} \text{ dislocations shear precipitates, or}
$$

$$
\Delta \sigma \propto F^{0.5} r^{-1} \text{ dislocations by-pass precipitates,}
$$

where $F$ is volume fraction of the secondary phase precipitates and $r$ is the precipitate size.

Dependence of the hardness on the precipitate size is examined by plotting $\Delta \text{hardness} / \sqrt{F}$, where $\Delta \text{hardness}$ is the relative change of the hardness from that of Sample II, against $F$ in Fig. 7. The hardness of the material appears to increase with $1/r$ due to increased strengthening effects of fine precipitates.
CONCLUSIONS

A systematic study of microstructure effects on SFEC signals of Inconel 718 alloy has been carried out. The size and volume fractions of the secondary precipitates were quantified using techniques including SEM and TEM. The peak-aged sample has smaller diameter secondary phase precipitates with a high density while the over-aged sample has larger diameter secondary phase precipitates with a low density. The peak-aged sample also shows higher conductivity while the over-aged and solutionized samples show lower conductivities. Thus, conductivity increases with the precipitate size and volume fraction of the secondary phase precipitates. Further study is being conducted to find out any shot peening induced change in the depth profile of the precipitate contents of the samples.

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REFERENCES