# Precipitation and Residual Stress Relaxation Kinetics in Shot-Peened Inconel 718

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(Submitted February 12, 2006)

Mechanical surface treatment by shot peening followed by aging at 700 and 740 °C was performed on Inconel 718. A previously proposed XRD method (Ref 10) for the quantitative phase analysis of Inconel 718 allowed for the determination of the precipitation kinetics of the  $\gamma''$  phase in the shot-peened layer and the matrix, respectively. The residual compressive stress field induced by shot peening and its relaxation behavior during aging were also determined. The relaxation process can be described by the Zener-Wert-Avrami function. The precipitation rate in the  $\gamma''$  phase in the shot-peened layer is greatly accelerated, which causes differences in the  $\gamma''$  phase amounts between the skin and the core during aging, especially during the initial stage. The high precipitation rate of the  $\gamma''$  phase in the shot-peened layer can be interpreted by the nonequilibrium segregation of niobium.

Keywords	Inconel 718, precipitation, residual stress relaxation,
	shot peening

## 1. Introduction

Inconel 718, a precipitation-strengthening Ni-base superalloy, has been widely used in aviation, space flight, energy, and chemical engineering industries for its combination of high strength, corrosion resistance, good processing-ability, and welding performance. The intermetallic compounds  $\gamma''$  and  $\gamma'$ are the main strengthening phases, and the  $\delta$  phase is the equilibrium phase of  $\gamma''$ . It has been found that precipitation of the  $\delta$  phase is always preceded by precipitation of  $\gamma'$  and  $\gamma''$  in the low-temperature range (below 900 °C). Precipitation of δ phase occurs in the approximate temperature range 750-1050 °C. It precipitates directly from the supersaturated  $\gamma$  matrix if treatments are carried out in the range 900-1000 °C. Contrary to the  $\gamma'$  and  $\gamma''$  behaviors,  $\delta$  phase seems to have a beneficial effect on stress rupture ductility (Ref 1-3). Cold working has pronounced effects on precipitation of the  $\gamma'$ ,  $\gamma''$ , and  $\delta$  phases. Cold working promotes the precipitation of  $\delta$  and  $\gamma''$  phases at different temperatures (Ref 4). At 910 °C, cold working promotes precipitation of  $\delta$  phase. At temperatures below 900 °C, Singh et al. (Ref 5) found that precipitation of  $\gamma''$  phase was more prevalent than that of  $\delta$  phase for 30% cold-rolled material, and precipitation of  $\delta$  phase was more preferable than that of  $\gamma''$  phase for 50% cold-rolled materials.

Shot peening is an often-used surface treatment process applied to improve the fatigue strength and fatigue life of cyclically loaded components. Shot peening can induce compressive residual stress fields and work-hardened microstructure in the surface layer (Ref 6), which then influences the precipitation process in Inconel 718. During aging, different micro-

structures may then form between the shot-peened layer and the matrix. A similar graded structure produced through shot peening and after aging in Timetal 21 has been reported (Ref 7).

The aim of this work is to study the precipitation as well as the residual stress relaxation behaviors of solution-treated and then shot-peened specimens of Inconel 718 during aging.

## 2. Materials and Methods

### 2.1 Materials and Shot Peening Processes

Hot-rolled bars of Inconel 718 were used in this work; its chemical composition is presented in Table 1. Solid solution treatment was conducted at 970 °C for 1 h followed by aircooling. The shot-peening treatments were then performed using an air blast machine. To obtain a sufficient shot peening layer and a satisfactory surface quality, the shot-peening process proceeded in two sequential steps. In the first step, a steel shot with a diameter of 0.8 mm was selected, and the shot-peening intensity was 0.35A(arc height on strip A). In the second step, the specimens were shot peened again by the glass shots with a diameter of 0.2 mm, and the intensity was 0.15A(arc height on strip A). After that, aging treatments were carried out at 700 and 740 °C, respectively, for different periods of time.

# 2.2 X-ray Diffraction Quantitative Phase Analysis and Determination of Residual Stress

According to the phase transformation characteristics of Inconel 718, the  $\gamma'$ ,  $\gamma''$ ,  $\gamma$ , and  $\delta$  phases and NbC are the five main phases after solution and aging treatment. Our previous study (Ref 8) proposed a simple x-ray diffraction (XRD) quantitative phase analysis method for Inconel 718 that was based on the relationship between the lattice constant of the  $\gamma$  phase and the content of the precipitates. The precipitation behavior in the shotpeened layer and the matrix of Inconel 718 during the aging process were analyzed using the XRD method mentioned above (Ref 8), and the ratio of  $W\gamma''/W\gamma'$  was found to be 3 (Ref 9).

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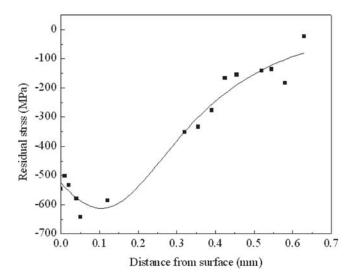


Fig. 1 Depth distribution of residual stress in shot-peened Inconel 718

Table 1Chemical composition of Inconel 718 (wt.%)

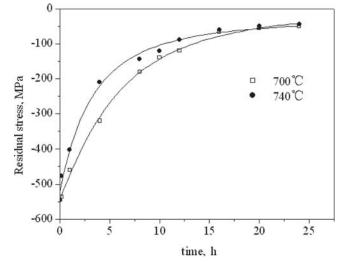


Fig. 2 Residual stress relaxation of shot-peened Inconel 718 at 700 and 740  $^{\circ}\mathrm{C}$ 

С	Cr	Ti	Ni	Мо	Nb+Ta	Al	В	Fe	Mn	Si	S	Р
0.032	18.8	1.05	53.28	2.97	5.12	0.58	0.002	bal	0.13	0.09	0.002	0.005

Residual stress in the shot-peened layer of Inconel 718 was measured by XRD according to the  $\sin^2 \Psi$  method. Measurements were carried out on the {220} planes of the  $\gamma$  phase in a diffractometer using Cu K $\alpha$  radiation. Successive removal of material to determine stress depth distribution was achieved by electropolishing. Electropolishing was only performed on the shot-peened surface.

## 3. Results and Discussion

### 3.1 Distribution of Residual Stress in Shot-Peened Layer

The compressive residual stress distributions were measured over the hardened layer below the surface of Inconel 718 after shot peening. The residual stress depth distribution is shown in Fig. 1. The results show the residual stress at the surface is compressive, and the residual stress value increases with depth to a peak value and then decreases. The compressive residual stress field (CRSF) of shot-peened metals is influenced by the metal properties and the peening parameters (Ref 10). Because the CRSF has direct effects on the following aging process, it is necessary to quantitatively describe the CRSF. It can be seen that the compressive residual stress at the surface is about -530 MPa, and the maximum value of the compressive residual stress is -620 MPa. The maximum value of the compressive residual stress is -620 MPa. The maximum value of the surface, and the total penetration reaches 0.65 mm.

#### 3.2 Relaxation of Residual Stress during Aging Process

The shot-peening treatment of Inconel 718 was applied before the aging process in this work, so the residual stress induced by shot peening will partially or fully relax during the subsequent aging at high temperatures. Thermal relaxation effects on the residual stress after shot peening are shown in Fig. 2 for temperatures of 700 and 740 °C. As aging time increases, the residual stress gradually decreases and almost disappears after 24 h. The relaxation rate of residual stress at 740 °C is a little higher than that at 700 °C. After 12 h, the ratios of relaxation at 700 and 740 °C are 71.0 and 83.7%, respectively.

Time and temperature influences on the residual stress relaxation during aging are controlled by a thermally activated process (Ref 7) and can be described by a Zener-Wert-Avrami function as:

$$\sigma^{\rm RS} / \sigma_0^{\rm RS} = \exp[-(At)^m] \tag{Eq 1}$$

where  $\sigma^{\text{RS}}$  is the residual stress at the surface after *t* hours of aging;  $\sigma_0^{\text{RS}}$  is the residual stress at the surface before the aging treatment, *m* is a parameter that depends on the dominating relaxation mechanism, and *A* is a function of the material and the temperature:

$$A = B \exp[-Q/(RT)]$$
(Eq 2)

*B* is a constant and *Q* is the activation energy. As a consequence of Eq 1, a plot of  $\log[\ln(\sigma_0^{\text{RS}}/\sigma^{\text{RS}})]$  as a function of log *t* for a given temperature will yield a straight line of slope *m*. The experimental verification of this linearity is apparent in Fig. 3. The different value for the *m* exponent of the Zener-Wert-Avrami function may indicate some interaction between precipitation of the  $\gamma'$ ,  $\gamma''$ , and  $\delta$  phases and relaxation of residual stress.

## 3.3 Precipitation Kinetics in the Shot-Peened Layer and the Matrix

Precipitation kinetics of the  $\gamma''$  and  $\delta$  phases in the shotpeened layer and in the matrix of Inconel 718 at 700 and 740 °C are plotted in Fig. 4 and 5, respectively.

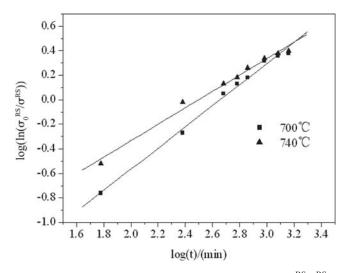


Fig. 3 Influence of aging time on residual stress in  $\log[\ln(\sigma_0^{RS}/\sigma^{RS})]$  –  $\log t$  diagram

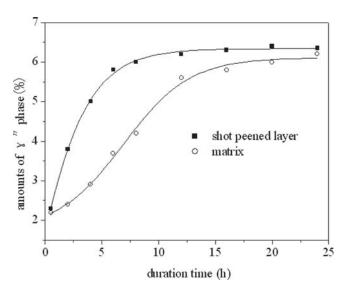


Fig. 4 Precipitation kinetics of  $\gamma''$  phase in shot-peened Inconel 718 during aging at 700 °C

The  $\gamma''$  and  $\gamma'$  phases can precipitate directly from the supersaturated  $\gamma$  matrix and then proceed to precipitation of  $\delta$  phases at 700 and 740 °C. Figure 4 and 5 show the high degree of cold work introduced by shot peening always accelerates precipitation of the  $\gamma''$  phase. During the beginning stage of aging, the amount of  $\gamma''$  in the shot-peened layer is almost the same as that in the matrix. As aging proceeds, the difference in the amounts of the  $\gamma''$  phase between the skin and the core increases remarkably. A near-equilibrium state of the precipitation process then tends to be reached, first for the skin. Meanwhile the amount of the  $\gamma''$  phase in the core increases slowly. For both temperatures, the precipitation curves for the skin and core are similar.

It is well known that the shot-peened layer is characterized as microstructure of deformation with high-density dislocation. In our previous work (Ref 4), the influence of cold deformation on the precipitation kinetics of  $\gamma''$  phase was systemically analyzed. It was found that the precipitation process of the  $\gamma''$ phase was greatly promoted at different temperatures and was

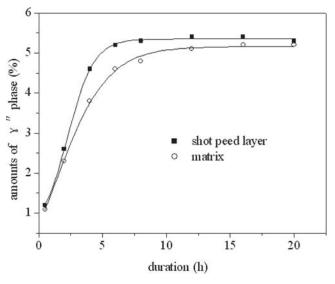


Fig. 5 Precipitation kinetics of  $\gamma''$  phase in shot-peened Inconel 718 during aging at 740  $^{\circ}\mathrm{C}$ 

interpreted to be caused by the nonequilibrium segregation of niobium. The high precipitation rate of  $\gamma''$  phase in the shotpeened layer can also be explained in the same manner.

Figure 6 and 7 show the diffraction patterns of the shotpeened layer of Inconel 718 after aging at 700 and 740 °C. Diffraction peaks of  $\gamma$  phase (111), (200), (220), (311), (222), NbC (111), and (220) and of  $\delta$  phase (002), (201), (020), (012), (211), (221), and (032) can be observed. On the lower angle side of the  $\gamma$  phase (200), (220), (311), and (222) diffraction peaks, some weak and diffuse profiles of sidebands are observed (marked by arrows). Separated by the computer, the overlapping peaks can be calculated and the result of aging at 700 °C is shown in Table 2. With the help of the method introduced in the literature (Ref 11), it was determined the peaks belong to the niobium-rich region. Niobium is likely to segregate at excess vacancies, dislocations, and extrinsic stacking faults in the  $\gamma$  matrix. This phenomenon seems to favor precipitation of the  $\gamma''$  phase.

## 4. Conclusions

- The CRSF induced by shot peening in Inconel 718 will relax during the subsequent high-temperature heat treatment. This relaxation process can be described by a Zener-Wert-Avrami function. The different *m* values for the exponent of the Zener-Wert-Avrami function may indicate some interaction between precipitation of the  $\gamma', \gamma''$ , and  $\delta$ phases and relaxation of residual stress.
- The precipitation rate of the  $\gamma''$  phase in the shot-peened layer can be greatly promoted. Different amounts of the  $\gamma''$ phase exist in the skin and the core during the aging process. In addition, the precipitation curves for the skin and core are similar. The high precipitation rate of the  $\gamma''$  phase in the shot-peened layer can be interpreted as a nonequilibrium segregation of niobium.

## Acknowledgments

This work was supported by the National Nature Science Fund of China under contract No. 59971039. The authors thank

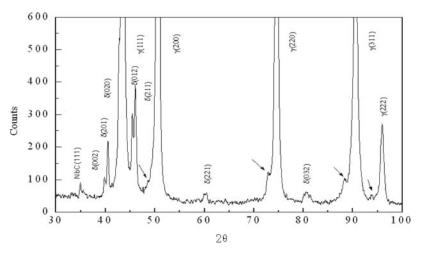


Fig. 6 XRD patterns of the shot-peened layer of Inconel 718 after aging at 700 °C for 10 h

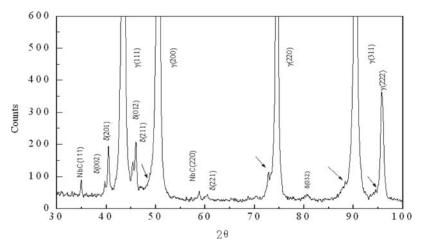


Fig. 7 XRD patterns of the shot-peened layer of Inconel 718 after aging at 740 °C for 6 h

Table 2	Diffraction	angle $(2\theta)$	) of the γ	matrix and
Nb-rich	region, with	aging at 7	'00 °C	

	Diffractio	Diffraction angle 20, deg			
Diffraction plane	γ matrix	Nb-rich region			
(111)	43.48				
(200)	50.57	48.92			
(220)	74.54	72.72			
(311)	90.46	87.74			
(222)	95.85	93.98			

Prof. J. Clumpner and Ms. K. Clumpner for their devoted reading of our manuscript.

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