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# A study of growth kinetics and distribution of χ precipitates in an induction-aged nickel-base superalloy

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This paper focuses on age hardening behavior of a Ni-based superalloy, during induction and conventional resistance aging. Particular emphasis is placed on the characterization of γ' precipitates, using light laser scattering (LLS), scanning electron microscopy (SEM) and image analysis. The results indicate that the growth of χ' precipitates in the induction-aged samples is considerably faster than that in the conventionally treated ones. This could imply the existence of an additional effect by induction on the growth kinetics of χ' precipitates in this alloy.

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*Keywords:* Ni-based superalloy, Induction aging, Precipitation, Size distribution

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## 1. Introduction

Ni-based superalloys are the most complex and the most widely used material for manufacturing of critical gas turbine components. It is well known that these materials have a high temperature strength because of the fine particles of LI$_2$ type ordered γ' phase precipitated coherently with the matrix [1].

The mechanical properties of a given alloy depend on such factors as volume fraction, particle size, nucleation, growth and coarsening rates and composition of γ' phase [2]. For instance, the existence of finer γ' precipitates with constant volume fraction in the microstructure, increases the hardness of the sample [3]. Moreover, studies show that the nucleation and growth of γ' precipitates in Ni-based superalloys is diffusion controlled [4-6]. Therefore, the rate of nucleation and growth are dependent upon the time and temperature of the aging process [1].

A research by Razavi and coworkers [7] showed that induction aging on IN738LC leads to a higher rate of nucleation and growth in comparison with conventional resistance and salt bath aging in similar time and temperature. Induction field with medium frequency applies an electronic flux in the sample which leads to a sharp increase in temperature [7].

The aim of this study is to explore further the effect of induction on the rate of nucleation and growth of γ' precipitates in Ni-based superalloys. The alloy Udimet 500 has been selected for this purpose. Two types of age hardening, namely induction aging and resistance tube furnace aging were conducted under different processing conditions to investigate the additional effect of induction on precipitation in this alloy.

## 2. Experimental procedure

The alloy used in this study was a cast superalloy Udimet 500 with the chemical composition (in wt%) of 19.1Cr, 16.20Co, 3.70Al, 3.05Ti, 4.20Mo, 0.40Fe, 0.02C, 0.02Si and balance nickel. Cylindrical samples with the height and the diameter of 20 and 8 mm, respectively, were machined. A hole was drilled on one side of each sample with the depth and inner diameter of 5 and 1 mm, respectively, to accommodate thermocouple. Samples were solution treated in the selected condition at 1180 $^{o}$C for 4 hrs and then quenched in iced-brine. The samples were aged in two different furnaces, namely induction and conventional resistance tube furnace. In each furnace, two samples were aged at 850$^{o}$C for 60 and 120min followed by iced-brine quenching. The heating rate in both furnaces was 141$^{o}$C/min.

The effect of induction aging time was investigated by aging the samples at 850$^{o}$C for 15, 30, 45, 60 and 120 min in induction furnace followed by quenching in iced-brine. Temperature and the heating rate of the samples in induction furnace were programmed and controlled by an electronic programmable device.

The hardness of the samples was measured by using the HV method. The samples were then electroetched in 5% $H_2CrO_4$. Microstructures were examined by scanning electron microscope (SEM) and characterized further by an image analyzer. In this stage, the size and volume fraction of γ' precipitates were measured for all samples. The weight percent of γ' precipitates of the samples were measured using an electrolytic extraction method. In order to dissolve the matrix, samples were immersed in 20% $H_3PO_4$ at room temperature for 1 hr. The obtained solutions were subsequently filtered and centrifuged. The remaining precipitates were characterized by X-ray diffractometery. Furthermore, unfiltered electrolytic solutions were analysed by light laser scattering (LLS) technique to obtain the size distribution of the γ' precipitates.

## 3. Results and discussion

### 3.1. Hardness

The hardness versus time curve for induction samples at the aging temperature of 850 $^{o}$C is shown in Fig. 1. It can be seen that the hardness of induction-aged samples increases with increasing the aging time up to 2 hrs. This implies that the nucleation and growth of γ' precipitates are the predominant processes during 2 hrs aging without any evidence of coarsening.

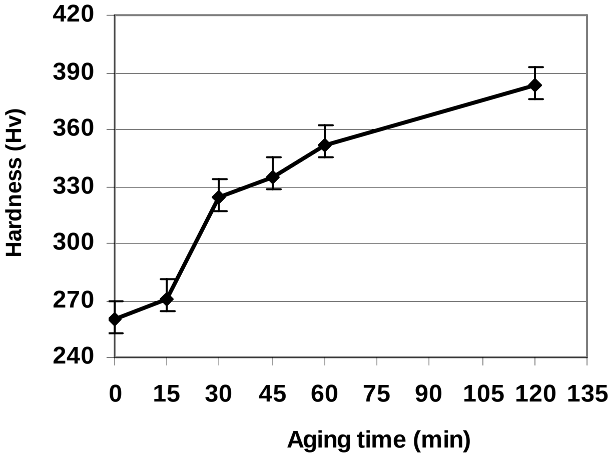

*Fig. 1. Variation in hardness with aging time for induction aging temperature of 850 $^{o}$C.*

Fig. 2 illustrates a comparison between the hardness results of induction and conventional methods. It indicates that the values of hardness of induction-aged samples are lower than those aged in tube furnace for similar conditions. This result can be interpreted in light of the difference in: a) the wt%, and b) the size distribution of γ' precipitates in the two samples. These two factors will be discussed in more detail in the following sections.

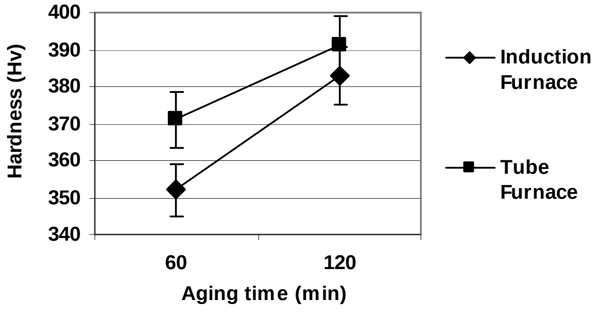

*Fig. 2. Variation in hardness with aging time for two furnaces at aging temperature of 850 $^{o}$C.*

### 3.2. SEM and image analyzing tests

The typical SEM micrographs of samples after aging in two different furnaces are shown in Fig. 3. Fig. 4 presents the mean size and volume fraction of γ' precipitates versus induction aging time. Two bar charts for comparing the volume fractions and mean sizes of γ' precipitates resulted by aging in two furnaces are also illustrated in Figs. 5 and 6, respectively.

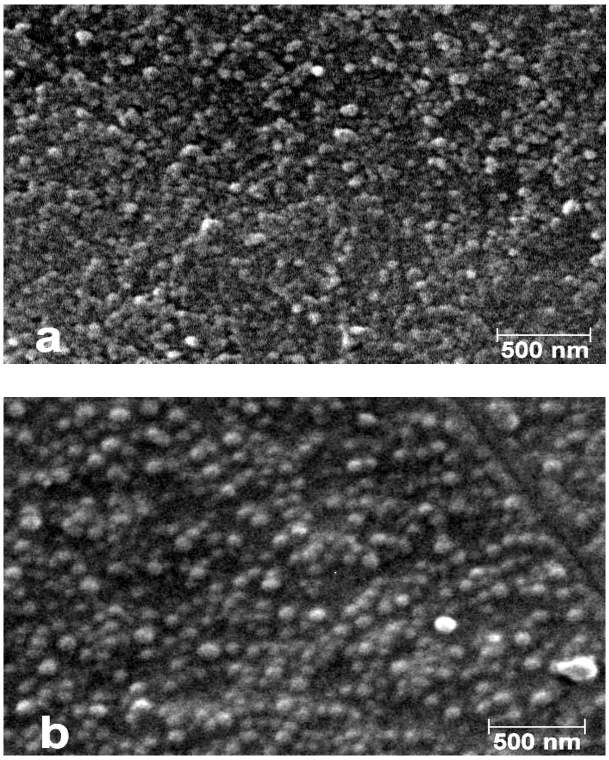

*Fig. 3. SEM images of the samples aged at 850 $^{o}$C for 2 hours in a) resistance tube furnace, and b) induction furnace.*

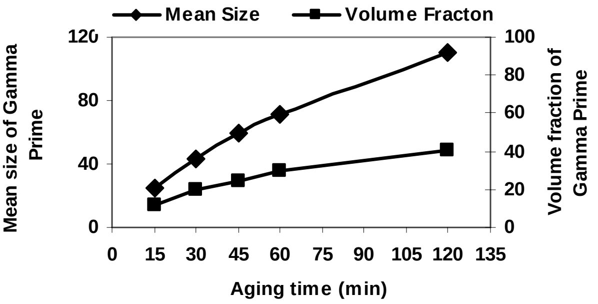

*Fig. 4. The mean size and volume fraction of γ' precipitates versus aging time for induction samples.*

Analysis of microstructure of the induction samples shows that γ' precipitates are fine, dispersed and almost spherical and are randomly distributed in the microstructure. Two types of γ' precipitates in aging times of more than 45 min were identified: a) large precipitates with more than 100 nm diameter as secondary γ', and b) the fine precipitates with less than 100 nm diameter as tertiary $\gamma'$.

The results of electron image analysis indicate that in the induction aging process, the increase of time at 850 $^{o}$C results in the increase in the size and the volume fraction of $\gamma'$ precipitates. As can be seen in Fig. 6, the volume fraction of $\gamma'$ precipitates in induction samples are more than that of tube furnace samples. It is initially expected from this result that the hardness in induction samples should be higher. However, this statement is in contradiction with the hardness results shown in Fig. 2. Consequently, the amount of volume fraction of precipitates does not seem to play a predominant role. The observed differences in hardness, Fig. 2, may thus be attributed to the differences in size distribution of $\gamma'$ precipitates.

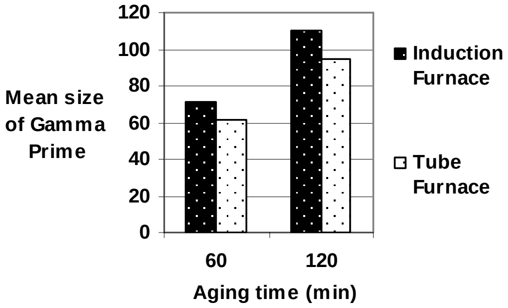

*Fig. 5. The mean size of $\gamma'$ precipitates versus aging time for the samples aged at two different furnaces.*

## 3.3. Electrolytic extraction

### 3.3.1. XRD

The existence of $\gamma'$ precipitates without any evidence of carbide precipitates or other phases on the filters was confirmed by X-ray diffractometery. A very good agreement was observed between the volume fractions as obtained by electron image analysis of the bulk sampls and that by weighing the extracted precipitates. These observations confirm that the solution made for the study of $\gamma'$ precipitates is appropriate.

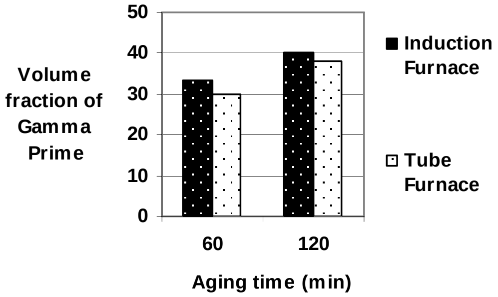

*Fig. 6. The volume fraction of $\gamma'$ precipitates versus aging time for the samples aged at two different furnaces.*

### 3.3.2. Size distributions

The electrolytic suspensions were investigated by the LLS analyzer to obtain the size distribution of $\gamma'$ precipitates. The curves obtained by this method show the size distribution of precipitates in all parts of the surface of the sample. The size distribution curves for different induction aging times are shown in Fig. 7. To compare the size distributions of $\gamma'$ precipitates for two furnaces, the related curves are illustrated in Fig. 8. As can be seen in Fig. 7, with the increase of the aging time, the size of $\gamma'$ precipitates increases and in contrast to the width of peaks, the height of the peaks decrease to keep the surrounded area constant. Fig. 8 shows that the size distribution of $\gamma'$ precipitates for induction samples is larger than that of tube furnace samples. Moreover, a new peak with the mean diameter less than 100 nm in the samples of both groups can be observed. This results in changing the size distribution curve to a bi-modal shape. These new peaks can be the result of nucleation of tertiary $\gamma'$ precipitates after 45 min aging.

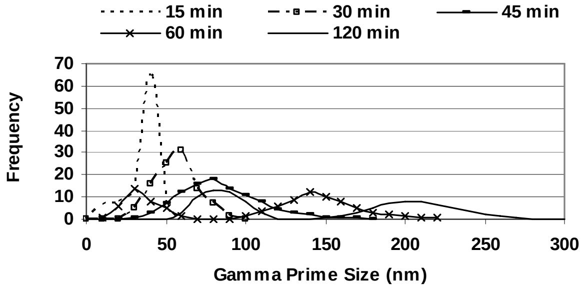

*Fig. 7. Particle size histograms for different induction aging times.*

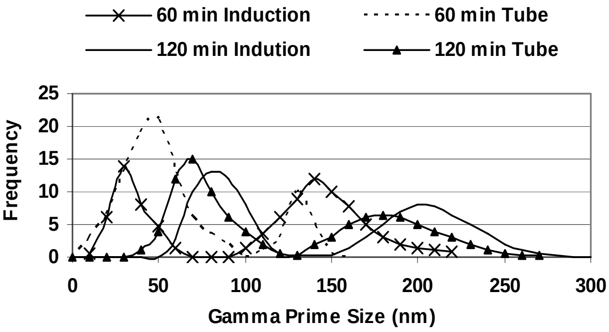

*Fig. 8. Particle size histograms for the samples aged at two different furnaces.*

The frequency percentages of tertiary $\gamma'$ precipitates were obtained by calculating the area under the tertiary $\gamma'$ peaks are illustrated in Fig. 9. The results show that the amount of tertiary $\gamma'$ precipitates for induction samples is lower than that of tube furnace samples. On the other hand, the mean size of tertiary $\gamma'$ precipitates for induction-aged samples is higher than that for tube furnace samples.

The wt% of γ' precipitates in tube furnace samples is lower than that in induction samples. The higher hardness in the former may therefore be attributed to the presence of higher amount of tertiary γ' precipitates with smaller mean diameter.

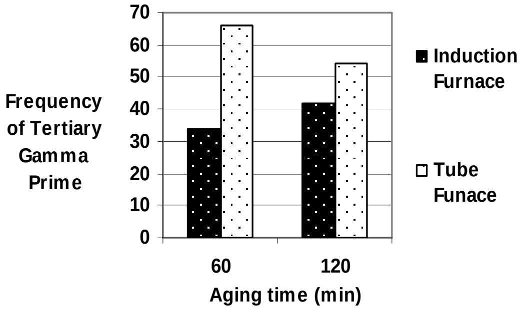

*Fig. 9. Particle size bar chart of tertiary γ' precipitates with less than 100 nm diameter for the samples aged at two furnaces.*

Formation of fine γ' precipitates can be due to a gradual decrease in the rate of growth and an increase in the rate of nucleation. At the beginning of the aging process, particles of secondary γ' nucleate in supersaturated matrix of γ. As the time passes further, growth of secondary γ' occurs with partitioning the solutes into the surrounding matrix. Furthermore, with a decrease in the amount of γ' forming elements in γ and an increase in the range of diffusion, the diffusion rates of solute decreases. So, the γ' precipitates cannot grow fast enough to decrease the supersaturation of the matrix. Eventually, at a critical aging time, the driving force is sufficient to allow the nucleation of tertiary γ' to occur. In this case, the condition for diffusion is more simple in induction samples and the growth mechanism is more active. Therefore, in induction samples, secondary γ' mean diameter is larger and its relative frequency is higher than thoes of the tube furnace samples. So, the induction samples have lower hardness due to lower amount of tertiary γ' precipitates. These results indicate that induction aging can lead to the higher rate of growth. This observation can be attributed to an increase of diffusion rate due to induction aging. Applied external field may affect the vibrational frequency of forming elements atoms in the matrix which lead to an increase in their jumps.

## 4. Conclusions

In the present study, two types of heating, namely induction aging and resistance tube furnace aging were conducted on a chosen Ni-based cast superalloy, Udimet500 and the results were analyzed and compared. The results can be summarized as follows:

1. The hardness of induction samples are less than that of tube furnace samples in the same aging times.
2. The volume fraction of γ' precipitates in induction samples are more than that of tube furnace samples. However, the size distribution curves of γ' precipitates showed that the amount of tertiary γ' precipitates finer than 100 nm in induction samples are less than tube furnace samples. This can explain lower hardness results.
3. The size distribution curves for the samples of both furnaces show a bi-modal distribution of γ' precipitates for aging more than 1 hr.
4. After induction aging at 850°C for 2 hrs, nucleation and growth of γ' precipitates take place without any evidence of coarsening process.
5. The rate of growth in induction aging is higher than that of conventional resistance furnace aging. It can be attributed to an increase in the rate of diffusion of γ' constitutional elements during induction aging.

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