Control of γ ['] Particle Size and Volume Fraction **in the High Temperature Superalloy Udimet 700**

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Experiments were conducted to determine the chemical composition, volume fraction and particle size of the γ' precipitate in Udimet 700 as a function of temperature and time. Growth of the γ' particles was found to follow $t^{1/3}$ diffusion controlled coarsening kinetics. The composition of γ' varied only slightly with temperature and was independent of time. From this information, a method was developed to estimate the volume fraction and average particle size of the γ' precipitate for any given heat treatment. It is suggested that this approach could be applied to other γ' strengthened superalloys.

NICKEL-BASE superalloys are widely used in applications requiring strength at high temperatures. Most of these alloys are precipitation hardened by a Ni₃ (Al, Ti) γ' phase which has an ordered fcc structure (Ll₂) and precipitates coherently in a nickel-rich fcc γ matrix. The strength of a given alloy is dependentupon such factors as volume fraction, particle size, coarsening rate, and composition (antiphase boundary energy) of the γ' precipitate. All of these factors can be controlled to varying degrees by heat treatment; but since most standard heat treatments have evolved empirically, little is known quantitatively about the influence of time and temperature on the γ' precipitate in commercial alloys. Information of this type would be valuable not only in the desigm of heat treatments for superalloys but also for understanding the effects of high service temperatures where coarsening and perhaps partial solution of the γ' phase occur.

There have been several contributions of interest in this area. Kriege and Baris¹ have determined the composition and volume fraction of the γ' precipitate in a large number of commercial alloys following standard heat treatment. Ardell²⁻⁵ and others^{6,7} have demonstrated that in simple binary or ternary γ - γ' (low volume fraction) systems, growth of the γ' precipitate obeys a diffusion-controlled coarsening model where the average particle radius increases linearly with time to the one-third power, $\vec{r} \propto t^{1/3}$. In addition, Mitchell⁸ has shown that the coarsening of γ' in the relatively simple, low volume fraction superalloy Nimonic 80A is also diffusion-controlled. Recently, the effect of high volume fraction and irregular distribution of second phase particles on coarsening kinetics has been evaluated mathematically.⁹ It was shown that a $t^{1/3}$ coarsening rate will be obtained for an array of spherical particles when coarsening is controlled by volume diffusion, even though the Lifshitz-Wagner theory^{10,11} of diffusion-controlled coarsening may not accurately describe the system. It has not yet been experimentally shown, however, that a $t^{1/3}$ kinetic law holds for complex commercial alloys containing large volume fractions of the γ' phase.

In this study Udimet 700 was chosen as being a rep-

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reseatative advanced high temperature nickel-base superalloy. Characterization of the alloy included determination of the volume fraction, particle size and composition of the γ' precipitate as a function of time and temperature. Precise quantitative determination of the volume fraction and composition of the γ' phase was possible using the technique of γ' separation recently refined by Kriege and coworkers.^{1,12} The results define the extent to which the γ' phase can be controlled by heat treatment. Furthermore, the particle size data demonstrate that the growth of the y' precipitate in this high volume fraction nickelbase alloy follows $t^{1/3}$ diffusion controlled coarsening kinetics.

MATERIALS AND EXPERIMENTAL PROCEDURE

Commercially available wrought Udimet 700* bar

			* Cr Co Mo Ti Al B C Mn Si Ni		
			wt pct 14.5 17.5 5.1 3.7 4.1 0.015 0.08 - - Bal.		

stock was used in this study. The microstructure of this alloy after a representative high temperature age is presented in Fig. 1. All specimens were annealed above the γ' solvus temperature for 4 hr at 2150°F followed by a fast air cool. The γ' solvus temperature was determined metallographically. The equilibrium volume fraction of γ' at various temperatures was determined for samples aged in the temperature range 1300° to 2000°F. In alloys such as Udimet 700 additional fine γ' particles usually form upon cooling after exposure at high aging temperatures. For accurate determination of γ' volume fraction at a given temperature, it is imperative that there be no precipitation of such cooling γ' . For this reason thin specimens (2.5 mm thick) were drop-quenched in iced brine directly from the heat zone of a vertical furnace after aging. Transmission electron microscopy as shown in Fig. 2 confirmed that aged specimens quenched from 2000°F or below were free of fine cooling γ' .

The volume fraction of γ' in the aged samples was measured by an extraction method. Extraction of the γ' was performed by anodic dissolution of the matrix using an electrolyte composed of 1 pet ammonium sulfate and 1 pct citric acid in water.¹ Since carbides are insoluble in this electrolyte, a correction for carbide contamination was necessary. Therefore, a second anodic dissolution which separated only carbides was

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Fig. 1-Udimet 700 aged for 4 hr at 2000°F. Magnification $11\bar{9}0$ times.

Fig. 2-Absence of fine cooling γ' in Udimet 700 quenched from 2000° F. The coarse particles were present before quenching.

performed and the amount of γ' was obtained by weight difference measurements. (The correction turned out to be minor since the amount of carbides was slight, \sim 1 wt pct.) Conversion of weight fraction γ' to volume fraction γ' was simplified because calculation showed that the two were essentially equivalent. When possible, the volume fraction determinations were checked by replica microscopy. Such results were in agreement, but the large scatter in the data obtained from replicas indicated that the extraction method was much more reliable. The composition of the extracted γ' was determined by an atomic absorption method.¹³

Particle size measurements were made on the extracted γ' particles. Since the particles were not *in situ,* great care was taken to obtain a representative distribution of particle sizes. A small quantity of well-mixed particles was deposited on a carbon film supported by a copper grid. Photographs of the particles were taken in a Hitachi HU-11A electron microscope, at least fifty particles per specimen being photographed. The size of the generally cuboidal particles

Fig. 3-Extracted γ' used for particle size measurements after heat treatment of 1900°F for 27 hr.

was determined by taking the average of the particle width and length from enlarged prints such as that shown in Fig. 3. The accuracy of this method was confirmed by comparison to *in situ* particle sizes measured by dark field transmission electron microscopy, for example, Fig. 2.

RESULTS

Solvus Temperature of γ'

The γ' solvus temperature is the upper limit for precipitation of the γ' phase. To determine this temperature, specimens were first aged at 1900° F to obtain coarse γ' , then heated to a temperature above 2050° F, held 1 hr, and rapidly cooled. The kinetics of γ' dissolution at these temperatures are very rapid and the equilibrium volume fraction is attained in a matter of seconds. As shown in Fig. $4, \gamma'$ is distributed heterogeneously in the sample exposed at 2055° F; at 2065° F, it is seen only at grain boundaries; and at 2075° F, it is absent. These results indicate that the solvus temperature is $2070^{\circ} \pm 5^{\circ}$ F.

Volume Fraction of γ'

The equilibrium volume fraction of γ' was determined for temperatures from 1300"F to the solvus. A plot of this data is presented in Fig. 5. Since experimental results discussed below showed that there was no measurable increase in volume fraction of γ' after longer periods, a standard aging time of 16 hr was used. The maximum amount of γ' present in the alloy is 38 vol pct and this amount is precipitated by aging at 1650° F or lower. Above 1650° F, the equilibrium volume fraction of γ' decreases gradually but at a continuously increasing rate with temperature. However, significant amounts of γ' persist to high temperatures. For example, 14 vol pct is present at 2000° F, only 70° F below the solvus.

The effect of aging time on the volume fraction of γ' precipitated is indicated in Table I. Within experimental error, the amount of γ' is essentially independent of aging time. This result is due to the extremely rapid kinetics of γ' precipitation. Previously reported work by Kriege and Sullivan¹² on Udimet 700 demonstrated

(b}

Fig. $4-\gamma$ ' solvus determination. Magnification 935 times. (a) Heterogeneous γ' at 2055°F; (b) grain boundary γ' at 2065°F; (c) no γ' at 2075°F

that oil quenching $1\frac{1}{4}$ in. by $\frac{5}{8}$ in. diam rod samples from temperatures up to 2000° F was too slow to prevent precipitation of γ' during cooling. The γ' volume fraction m all their samples was about 36 pct, which was probably the maximum volume fraction in that heat of material. Their results indicate that about 22 vol pct of γ' formed during the cooling of a sample aged at 2000° F.

Composition of the γ' Precipitate

The composition of the γ' phase is remarkably insensitive to aging temperature and time. The chemical

Fig. 5-Volume fraction of γ' as a function of temperature.

analyses of γ' extracted from samples aged 16 hr at 1600° to 2000° F are indicated in Fig. 6. Only slight changes are evident. As the aging temperature is increased, the precipitate becomes somewhat leaner in molybdenum and aluminum and correspondingly richer in cobalt and chromium. The titanium content of the γ' decreases slightly up to 1700° F and then begins to increase; the nickel content varies in the opposite manner. The effect of aging time at 1800° F on the composition of the γ' phase is shown in Table II. Here there is no significant change; the differences axe well within experimental error $(\pm 5 \text{ pct})$. It would appear that heat treatment cannot be used for the purpose of altering γ' composition.

The representative formula for γ' in this heat of Udimet 700 may be written $(Ni_{0.88}C_{0.10}Cr_{0.02})_3$ $(Al_{0.51} Ti_{0.37} Mo_{0.05} Cr_{0.07})$. This formula coupled with the determination of volume fraction of precipitate as a function of temperature, Fig. 5, allowed computation of a pseudobinary γ - γ' phase diagram. The resultant phase diagram is shown in Fig. 7 where the phase

Table I. Effect of Aging Time on the Volume Fraction of γ'

Aging	Volume Fraction of γ' after Aging for						
Temperature. [°] F	1 hr	4 _{hr}	16 hr	27 _{hr}			
1800	31.4	31.8	32.9	31.7			
1900	25.0	25.0	26.0	25.0			
2000	12.9	14.4	13.5	13.5			

Fig. 6-Composition of γ' as a function of temperature after aging for 16 hr.

boundaries are plotted vs the sum of the aluminum and titanium concentration. The solubility of aluminum and titanium in Udimet 700 is in close agreement with the $Ni-Cr-Al-Ti$ quarternary system, 14 but is considerably less than that seen in the binary Ni-A1 system. At 1900°F for example, the solubility of aluminum in nickel is 14.5 at. pct¹⁵ compared to only 9.5 at. pct Al and Ti in the γ matrix of Udimet 700.

Particle Coarsening

GROWTH KINETICS

Measurements of particle coarsening kinetics were made at 1800° , 1900° , and 2000° F. If coarsening is diffusion-controlled, the average particle size should increase according to the relationship

$$
\left[\widetilde{r}^3-\widetilde{r}_0^3\right]^{1/3}=kt^{1/3}\tag{1}
$$

where \bar{r} is the average particle radius at time t, \bar{r}_0 is the average particle radius at $t = 0$, and k is a temperature dependent rate constant. Following Ardell,³ for cuboidal particles \vec{r} is replaced by $\vec{a}/2$ where \vec{a} is the average length of a cube edge. The particle size data are therefore presented as a plot of $[(\bar{a}/2)^3 - (\bar{a}_0/2)^3]$ vs $t^{1/3}$ in Fig. 8. The data are consistent with a linear relationship. This fact demonstrates that the coarsening of γ' in a complex, high volume fraction superalloy such as Udimet 700 also follows the standard $t^{1/3}$ kinetics of diffusion-controlled particle growth. The effect of time and temperature upon particle size can then be predicted.

The initial particle size $\bar{a}_0/2$ used above was esti-

Composition, wt pet Aging Time, hr Cr Co Mo AI Ti Ni

Table Ih Effect of Aging Time at 1800~ F on the Composition of the 7" Phase

mated to be 150 to 300A depending upon the aging temperature. The presence of fine γ' particles at $t = 0$ resulted from the precipitation of γ' during cooling from the solution temperature and the growth that occurred during heating to the aging temperature. The correction due to $\bar{a}_0/2$ is small and could easily be neglected so that $\overline{a}/2 \approx kt^{1/3}$.

ACTIVATION ENERGY

The slope of each line in Fig. 8 is a temperature dependent rate constant which is defined as³

$$
h = \left[\frac{2\gamma \, DC_e \, V_m^2}{\rho_c^2 \, RT}\right]^{1/3}
$$
 [2]

*On occasion certain investigators have defined $K = k³$ as the rate constant.

$$
\quad \text{where} \quad
$$

- $y =$ interfacial free energy of the particle/matrix interface
- $D =$ composite coefficient of diffusion of several atom species
- C_e = concentration of solute (γ' forming elements) in equilibrium with a particle of infinite radius

 V_m = molar volume of precipitate

Fig. 8-The $t^{1/3}$ dependence of average particle size (a/2 vs $t^{1/3}$).

- ρ_c = numerical constant dependent on the distribution of particle sizes ($\rho_c = 3/2$ according to Lifshitz-Wagner theory)
- $R = \text{gas constant}$
- $T =$ absolute temperature

In this equation the temperature dependent terms are C_e , D, and obviously T. The diffusion coefficient D is given by $D = D_0 \exp(-Q/R)$. D_0 is a frequency factor and Q is an activation energy which is dependent primarily upon the activation energy for diffusion of aluminum and titanium in the matrix but is also influenced by the interrelated diffusion of Co, Cr, and Mo away from a growing γ' particle. The relationship defining k can be reformulated to give

$$
\ln [k^3 (T/C_e)] = \text{const.} - (Q/RT) \tag{3}
$$

The activation energy can be determined from the slope of a plot of log $[k^3(T/C_e)]$ vs $(1/T)$. In the past variation of the quantity C_e/T with temperature has usually been neglected because there was little change in the temperature ranges studied. However, this factor must be considered in the present analysis since C_e (Al + Ti) increases significantly at temperatures above 1650°F. The necessary values of C_e can be determined, see Fig. 7, since the temperature dependence of both volume fraction and chemical composition of the γ' phase are known.

The activation energy was calculated from the plot of log $[k^{\circ}(T/C_e)]$ vs $(1/T)$ given in Fig. 9. The values of log $\lfloor k^3 (T/C_{\varrho}) \rfloor$ do indeed vary linearly with $(1/T)$. The values of k for 1800[°], 1900[°], and 2000[°]F were obtained by measuring the slope of the particle coarsening curves in Fig. 8. The other k values were calculated from the 16 hr particle size measurements by assuming that the t^{++} relationship held at all tempera tures. Both the values of k and C_e for the different temperatures are listed in Table III. The data for all temperatures fit very well establishing that the standard $l^{1/3}$ rate of coarsening probably holds for the whole range of temperatures studied. The small amount of scatter in the data and the range of temperatures included give reasonable confidence in the

Fig. 9--Determination of the activation energy for coarsening. The cube of the rate constant has been compensated by the change in concentration of solute with temperature and is plotted vs the reciprocal of the absolute temperature.

measured activation energy of 64.5 kcal per mole. Despite the complexity of this alloy, the activation energy correlates well to that for diffusion of aluminum or titanium in nickel (64.4 and 61.4 kcal per mole¹⁶ respectively) and to the coarsening of γ' in binary Nt-A1 and Nt-Ti alloys (64.4, Ref. 2, and 67.5 kcal per mole, Ref. 5, respectively). The temperature dependence of k can be given as

$$
k = A \left(\frac{C_e}{T}\right)^{1/3} \exp. (-64,500/3RT) \tag{4}
$$

where $A = 12.3 \times 10^6 (\text{\AA}^{\circ} \text{K}/\text{hr}^{1/3})$ for concentration C_e of aluminum plus titanium in at. pct.

DISCUSSION

The results illustrate the extent to which the distribution and composition of the γ' phase in a typical superalloy can be controlled by heat treatment. Surprisingly, the chemical composition of the γ' precip-

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Fig. 10-Average γ' particle size $(\overline{a}/2)$ vs time for temperatures from 1400° to 2000° F (volume fraction of γ' from 38 to 14 pct).

itate is nearly independent of aging time or temperature. It has sometimes been assumed¹⁷ that γ' initially forms containing an excess of certain solute elements, particularly the refractory alloying additions. In such an event, the γ' composition should then adjust toward equilibrium during continued exposure at temperature and this could result in a variation of mechanical properties. Similarly a change in γ' composition with temperature has been suggested. However, with the possible exception of heavy elements such as tantalum and tungsten which are not present in Udimet 700, it appears that the variation of γ' composition with heat treatment or thermal exposure is negligible.

For heat treatment or service temperatures above 1650°F, the volume fraction of γ' is temperature dependent, decreasing continuously from \sim 38 vol pct at 1650° F to zero at the solvus temperature 2070 $^{\circ}$ F. The particle size of the γ' precipitate can be predicted (or controlled) in a straightforward manner since particle growth obeys $t^{1/3}$ coarsening kinetics despite the complexity of the alloy and the high volume fraction of γ' formed. For Udimet 700 the rate of coarsening is given by

$$
\begin{aligned}\n\left[\left(\frac{\overline{a}}{2}\right)^3 - \left(\frac{\overline{a}_0}{2}\right)^3\right]^{1/3} \\
= A t^{1/3} \left(\frac{C_e}{T}\right)^{1/3} \exp. \left(-64,500/3RT\right)\n\end{aligned} \tag{5}
$$

A series of particle size vs time plots are presented in Fig. 10 for temperatures from 1400° to 2000° F. The volume fraction of γ' at each temperature is also indicated. Such a chart can be used to predict the effect of thermal exposure upon the volume fraction and particle size of the γ' precipitate or as a guide to heat treatment. For example, one could produce a distribution of γ' particles 1800Å on a cube edge ($\bar{\alpha}/2$ = 900Å) where the amount of γ' varied from 14 vol pct (1.2 hr at 2000° F) to 38 vol pct (65 hr at 1650[°]F).

While strict application of Fig. 10 is limited to the prediction of γ' particle size after a single-step aging treatment or long time thermal exposure at a single temperature, this data can also be useful in approximating what will occur during multiple step heat treatments or thermal cycling. For example, a standard

Fig. 11-Fully heat treated Udimet 700. 2150° F/4 hr + 1975° F/4 hr + 1550° F/24 hr + 1400° F/16 hr.

heat treatment of Udimet 700 includes a solution anneal followed by a three-step aging sequence: 1975 F for 4 hr + 1550°F for 24 hr + 1400°F for 16 hr. Taking the volume fraction data, Fig. 5, and interpolating from Fig. 10, it follows that the 4 hr age at 1975° F produces 18 vol pct of γ' particles 2560Å on a cube edge. Aging at 1550° F increases the equilibrium volume fraction of γ' by 20 pct. A small amount of the supersaturation is relieved by growth of the coarse γ' already present; however, diffusion distances are limited and precipitation occurs rapidly such that nearly 20 vol pct of additional fine precipitate particles are formed. If the coarsening of these smaller particles were unaffected by the large γ' present, Fig. 10 predicts the average small particle size will be 780Å after the 1550° F heat treatment. Since the maximum volume percent of γ' is present at 1550°F, no additional γ' can precipitate either on cooling from 1550° F or on subsequent heat treatment at 1400~F. Thus there should only be two basic sizes of γ' rather than three. The final age at 1400° F should produce only very slight coarsening of the existing γ' (1400°F for 16 hr is approximately equivalent to $1550\textdegree F$ for 1 hr). For comparison, the microstructure of fully heat treated Udimet 700 is shown in Fig. 11. Only two basic sizes of γ' are present as predicted. The large particles average 2930A on a cube edge, 15 pct larger than predicted while the small particles are 590Å, nearly 25 pct smaller than predicted. But the deviations are in the direction expected for two reasons. First, as stated previously relief of the supersaturation at 1550°F occurs in part by growth of the large γ' already present. And secondly, recent work by Weins³ has shown that the coarsening of a particle is strongly dependent on the size of the particles which surround it. Therefore the large particles will coarsen more rapidly than predicted when surrounded by small particles and similarly, the coarsening of the small particles will be retarded due to the presence of the large particles.

It is felt that the rates of γ' coarsening and the activation energy for diffusion found for Udimet 700 should hold well for most other γ' containing nickelbase superalloys. It also seems reasonable to assume that the shape of the volume fraction γ' vs temperature curve should be roughly the same so that such a

curve could be approximated for other alloys simply by measuring the maximum amount of γ' formed and the solvus temperature. Finally, since the chemical composition of the γ' phase appears to be invariant for a particular heat of material, one determination of γ' composition (typical γ' compositions for many superalloys have been reported¹) coupled with the temperature dependence of volume fraction γ' would permit an estimation of the temperature dependence of C_e . If γ' compositions could not be obtained, the values of C_e shown in Fig. 7 might be suitable. In this way the effects of thermal exposure and heat treatment upon γ' could be predicted for other high temperature nickelbase superalloys by constructing graphs similar to Fig. 10.

It is of value to discuss the coarsening results in relationship to the diffusion-controlled coarsening theory of Lifshitz and Wagner. 10,11 The $t^{1/3}$ relationship is consistent with the Lifshitz-Wagner theory but it is also consistent with other coarsening theories.⁹ The theory was developed for a low volume fraction and high interparticle spacing system and it assumes that each particle is in an identical environment with respect to composition. As the volume fraction or the irregularity of distribution of the second phase particles increases, the conditions for application of the theory are less well satisfied due to overlapping diffusion fields. The average particle size will still be proportional to $t^{1/3}$ but the distribution of particle sizes will no longer be consistent with theory.⁹ For instance, the Lifshitz-Wagner theory predicts that no particle will be larger than 1.5 times the average particle size. In our study, however, the largest particle measured in each specimen was generally larger than 1.5 times the average and was occasionally larger than twice the average. Therefore because of the irregularity of distribution of γ' particles, the coarsening of γ' in Udimet 700 cannot be completely described by the Lifshitz-Wagner theory.

CONCLUDING REMARKS

The strength of a precipitation-hardened alloy is related to the volume fraction, particle size, and distribution of the strengthening precipitate phase. These parameters can vary markedly as a result of heat treatment or service at elevated temperatures. Quantitative information describing the influence of time and temperature upon the γ' precipitate in nickel-base superalloys is of special significance due to their application at high temperatures. The kinetics of γ' precipitation or dissolution are such that the equilibrium volume fraction of γ' phase is rapidly established at a given aging temperature with subsequent changes due solely to particle coarsening. The results of the present study describe the effect of heat treatment or thermal exposure upon the γ' precipitate in Udimet 700.

It is suggested that similar information can be obtained for other alloys. The essential data required for this purpose include:

1) Aging studies at one (preferably several) temperature(s) to establish that $t^{1/3}$ diffusion controlled coarsening kinetics are followed, as is the case for γ' particles in Udimet 700. Particle sizes are measured most accurately by thin foil or extraction techniques. For larger particle sizes replicas may be adequate.

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2) Determination of the volume fraction of precipitate phase vs temperature. For example, in Udimet 700 the volume fraction of γ' decreases in a continuous manner from a maximum of 38 vol pct at 1650°F to zero at the solvus temperature of 2070"F. Metallographic techniques can be used to determine volume fraction; but extraction of the precipitate phase is preferred.

3) Chemical analysis of the precipitate formed at various temperatures. The volume fraction and chemistry data permit calculation of the equilibrium solute concentration C_e . For Udimet 700 examined in this study, the γ' chemistry is independent of temperature and can be represented as $(Ni_{0.88}Co_{0.10}Cr_{0.02})_3$ $(Al_{0.51}Ti_{0.37}Mo_{0.05}Cr_{0.07})$. If the composition of γ' is independent of temperature in other superalloys, then extensive data is already available for this class of materials.

4) Evaluation of the rate constant k at several temperatures. Some values of k will be available from part 1. Others can be obtained by measuring particle sizes after aging and assuming $\dot{\vec{r}} \approx kt^{1/3}$. The activation energy Q is then calculated from a plot of $\log [k^3 (T/C_e)]$ vs $(1/T)$.

This information is sufficient for evaluating the parameters in the coarsening equation

 $(\bar{r}^3 - \bar{r}_0^3)^{1/3} = A t^{1/3} (C_e/T)^{1/3}$ exp. $(-Q/3RT)$ [6] as well as giving the volume fraction of precipitate at that temperature. In Udimet 700, \bar{r} is replaced by $\bar{a}/2$ where \bar{a} is the edge length of the average cuboidal γ' particle; A is $12.3 \times 10^8 \text{ (Å }^{\circ} \text{K/hr}^{1/3}$ for concentration *Ce* of aluminum plus titanium in at. pet solute; and the activation energy for coarsening is 64.5 kcal per mole.

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