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IN-713C Characteristic properties optimized through different heat treatments

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The strength of a nickel-based superalloy hardened through precipitation is related to the volume fraction, particle size and distribution of the precipitated phase, γ'. These parameters may vary as a result of heat treatment, or high-temperatures service. The information obtained, describing the influence of time and temperature on the precipitated phase, γ', is of special importance owing to its technological application at high temperatures. Dissolution or precipitation kinetics are such that the volume-fraction balance of the γ' phase is quickly established at an ageing temperature given by successive changes due only to the particle growth. The results of the present study describe heat-treatment effects on size and distribution of the γ' phase, and precipitated carbides ageing of a nickel-based superalloy (IN-713C). These on ageing studies at a certain temperature show that the kinetic growth of γ' particles by controlled diffusion follows that $r \propto t^{1/3}$ law.

1. Introduction

Nickel-based superalloys are widely used in experimental and technological applications requiring resistance to high temperatures. Most of these alloys are hardened by precipitates of a γ' phase which has an arranged f c c (Ll_2) structure, and precipitates coherent by in a γ' matrix (f c c) rich in nickel, obtaining a strong γ/γ' interaction, which controls the dislocations behaviour thus allowing a high resistance to creep at high temperatures. Carbides, depending on the alloy composition, and acting usually in the grain boundaries, cooperate to reinforce and minimize their sliding.

The purpose of this work was to study the growth and morphological development of γ' particles in a vacuum induction melting alloy. Micro-structural characteristics of this material depend, in the first place, on the following parameters: (a) overheating temperature of the liquid metal; (b) casting temperature; (c) ceramic mould temperature; (d) metal–mould balance temperature; (e) the time required to reach the metal–mould balance temperature; (f) cooling speed; (g) heat flow. All these parameters have a strong impact on microstructural characteristics such as grain size and direction, dendritic spacing, λ, and coalescence and distribution of γ', all of them governing the physical and mechanic properties of the alloy.

The resistance and structural stability of these alloys at high temperatures depend on such factors as volume fraction, f, particle size, r, distribution and composition of γ' phase. The morphology of the coherent γ' phase, depends also on the composition and the lattice disregistry parameter between matrix and precipitate. The molybdenum content, as well as the relation between titanium and aluminium, allows different kinds of γ' morphology to be obtained, being it spheroid, globular, or cubed, with an increase in the lattice disregistry parameter, δ [1]. At high temperatures, a low coherent distortion is desired to minimize the surface of energy γ–γ' and so minimize the stability of the phase.

All these factors can be controlled by varying the heat parameters [2]; however, even the most common treatments, have mostly been empirically developed. Quantitative knowledge about the influence of time and temperature on the γ' precipitate in commercial alloys is not enough. Information of this kind could be evaluated not only for the design of superalloy heat treatments, but also to understand changes in the microstructure as a consequence of work at high service temperatures where the growth in volume and possible partial solubility of γ' phase may take place.

The theory of the controlled growth of particles through diffusion has been formulated by Lifshitz and Slyozov [3] and by Wagner [4], known as the LSW theory. In this case, the driving force for the precipitate growth is the decrease of the precipitate superficial energy [5]. The theory predicts that the average radius, r, of a particle would increase with time, t, according to the equation:

$$(r^3 - r_0^3)^{1/3} = Kt^{1/3} \quad (1)$$

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where r_0 is the particle size before growth starts. The speed constant, K, is given by

$$K = \frac{2\gamma D C_e V_m^2}{\rho_c^2 RT} \quad (2)$$

where γ is the interfacial energy between precipitate and matrix, D is the solute diffusion coefficient in the matrix, C_e is the concentration of solute balanced with a precipitate of radius ∞, V_m is the molar volume of precipitate, ρ_c is a numerical constant related to the precipitate distribution size, R the gas constant and T the temperature.

The first growth theories were developed by Greenwood [6] for very small γ' volume fractions. The particle growth LSW theory is also applicable when the volume fraction is small. When this fraction is large, the diffusion distances become shorter. The effect of the volume fraction on the precipitate growth was treated theoretically by Ardell [7] proving that particle kinetic growth controlled through diffusion followed this law. In the present work, we noticed that the larger the volume fraction of the precipitate, the bigger is the particle. Lifshitz, Slyozov and Warner, proved the particle growth theory versatility by applying it to alloys with volume fractions of γ' varying between 9% and 60%. The kinetic growth, $t^{1/3}$, has been applied to the γ' particles in a great variety of commercial alloys [8–10].

Later works [11–14] studied γ' phase growth for volume fractions of about 45%.

2. Experimental procedure

An IN-713C ingot (composition C = 0.12, Cr = 12.5, Mo = 4.2, Nb = 2.0, Ti = 0.8, Al = 6.1, Ni = bal.) vacuum induction melted, of 66 mm diameter, was cut into slices 15 mm thick. Each of them was divided across in small test tubes for the study of later heat treatments.

Thermal analysis shows, for alloy IN-713C, that the liquidus temperature is 1288 °C and its melting range is 60 °C [15]. The solvus temperature of γ' is the top limit for γ' phase precipitation, and this temperature varies for each superalloy [16]. To determine this temperature metallographically more precisely, the test tube was first aged at 1050 °C to obtain thick γ', then it was heated to a temperature above 1160 °C for 1 h and quickly cooled in water. The dissolution kinetics of γ' at that temperature is very quick and the volume fraction in equilibrium is reached within a few seconds. In this way, a heterogeneously distributed γ' phase is obtained; when later heated at 1170 °C, γ' is no longer seen in the matrix, but is found to have migrated to the grain edge. A later heating at 1180 °C totally dissolved γ'. This result indicated that the solvus temperature is 1175 ± 5 °C. After that, test tubes were thermally treated in order to obtain γ' phase.

Initially, a solution treatment was carried out (1176 °C/2 AC h) and then precipitation (926 °C/16 AC h). This treatment, T_s, is one of the most usual ones for this kind of alloy, and was taken as reference.

Because IN-713C alloy is within the range of alloys containing > 45% γ' phase [2, 17, 18], and because it was designed to be used at high temperatures for long times, two solution treatments (1176 °C/2 AC h + 1080 °C/2 AC h) and two precipitation treatments (925 °C/16 AC h + 760 °C/16 AC h) were carried out, with the purpose of obtaining a greater volume fraction of γ' phase and a smaller particle size, this being the test heat treatment, T_T. In each case the microhardness in the precipitated γ' phase was measured. Fig. 1 shows microhardness, H_v, volume percentage of γ' phase and the γ' particle size, all of these being functions of the different experimental precipitation temperatures, for each heat treatment, for 16 h. All treatments were carried out in an argon atmosphere.

Test tubes were first polished mechanically to 2 μm diamond paste and then electrolytically etched (for 15 s at 20 V), with a reactive composed of chromium oxide (25 g), phosphoric acid (250 ml) and sulphuric acid (20 ml). The microstructures were studied by optical and scanning electronic microscopies (SEM). The phase volume percentage and the γ' particle size were directly measured from pictures obtained by SEM. The morphological development and the kinetic growth of γ', the size of which can be controlled as a function of time and temperature, was studied for the standard heat treatment, T_s, (1176 °C/2 AC h + 925 °C/16 AC h).and for the heat treatment, T_T, proposed earlier [14]. Test tubes were prepared for each of the treatments (in an argon atmosphere) and different ageing temperatures (800, 875 and 950 °C) were determined, to obtain the γ' growth. Test tubes

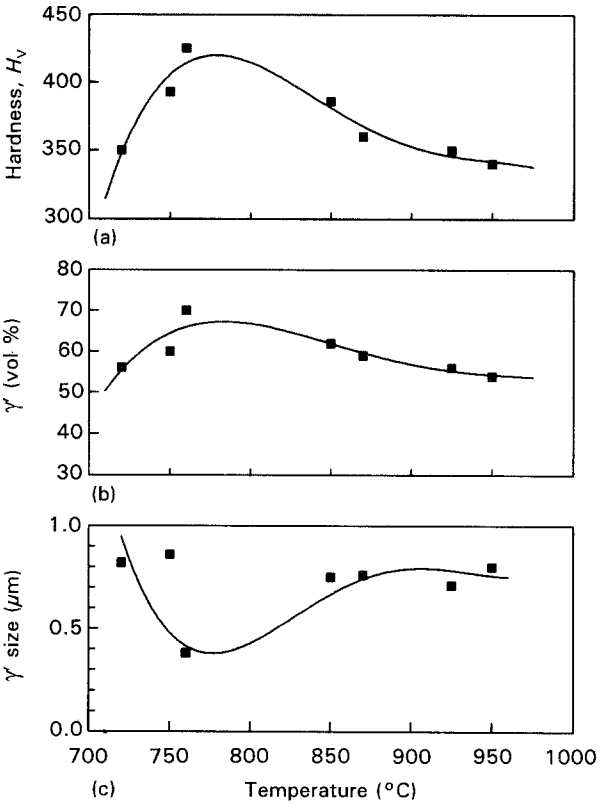

Figure 1 Variation of (a) hardness, H_v, (b) total γ' precipitate fraction, (c) relative γ' size (μm), versus different temperatures of precipitation.

were taken out of the furnace at different time intervals and subjected to air cooling.

The particle average size measured from pictures obtained through SEM, was plotted as $a/2$ versus ageing time ($t^{1/3}$), $a/2$ being half of the cube edge. Tables I–III show data on the particle size as a function of ageing time. Columns T_s and T_T correspond to the experimental values obtained, and columns T_{SN} and T_{TN} to normalized values. Figs 2 and 3 show graphs of these normalized values. From the previous figures we obtained the values of K (growth speed constant) for each ageing temperature (800, 875 and 950 °C), and for each of the heat treatments considered in this work. In Table IV, values of K_S and K_T are specified.

TABLE I

800 °C		$\bar{a}/2$ (nm)			
t (h)	$t^{1/3}$ ($h^{1/3}$)	T_T	T_S	T_{TN}	T_{SN}
51	3.708	431.5	390.5	97.4	87.8
267	6.439	441.3	454.0	107.2	151.3
360	7.113	440.0	470.3	105.9	167.6
456	7.697	498.0	483.7	163.9	181.0
600	8.434	515.6	501.2	181.5	198.5
696	8.862	514.9	511.3	180.8	208.5
792	9.252	539.0	520.6	204.9	217.9
888	9.611	529.5	530.5	195.4	227.8

TABLE II

875 °C		$\bar{a}/2$ (nm)			
t (h)	$t^{1/3}$ ($h^{1/3}$)	T_T	T_S	T_{TN}	T_{SN}
144	5.241	409.5	439.5	121.1	173.3
240	6.214	431.6	474.9	143.2	208.7
336	6.952	451.6	492.0	163.2	225.8
432	7.559	465.0	517.0	176.6	250.8
528	8.082	473.7	536.0	185.3	269.8
648	8.653	486.5	552.0	198.1	285.8
720	8.962	498.3	563.0	209.9	296.8

TABLE III

950 °C		$\bar{a}/2$ (nm)			
t (h)	$t^{1/3}$ ($h^{1/3}$)	T_T	T_S	T_{TN}	T_{SN}
168	5.517	362.0	396.0	111.6	189.3
240	6.214	409.0	426.2	158.6	219.5
312	6.782	418.1	459.9	167.7	253.2
408	7.416	436.1	467.0	185.7	260.3
504	7.958	441.2	493.2	190.8	286.5
600	8.434	453.2	530.2	202.8	314.4
816	9.344	460.5	562.1	210.1	320.6

TABLE IV

Temperature (°C)	K ($nm\,h^{-1/3}$)	
	K_P	K_U
800	20.23	23.56
875	23.18	33.13
950	23.76	35.70

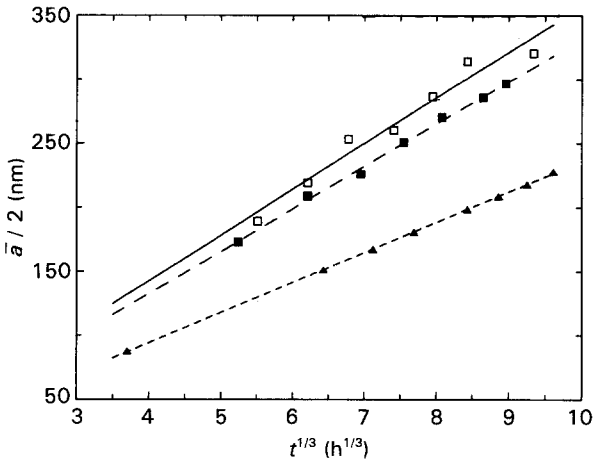

Figure 2 Plot of $(\bar{a}/2)$ versus $t^{1/3}$ ageing at different temperatures for standard treatment. (□) 950 °C, (■) 875 °C, (▲) 800 °C. K_{950} = 35.74 nm $h^{1/3}$, K_{875} = 33.13 nm $h^{1/3}$, K_{800} = 23.56 nm $h^{1/3}$.

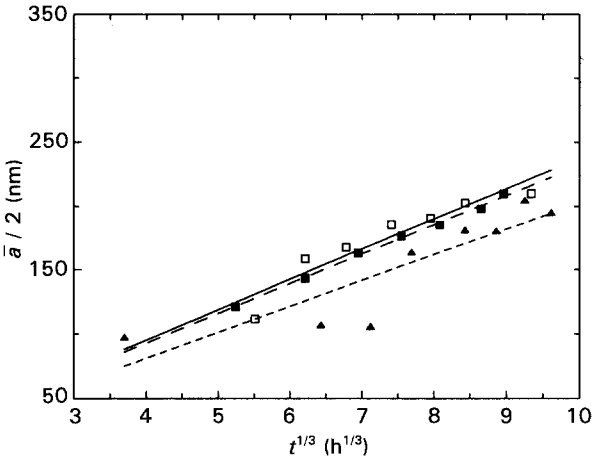

Figure 3 Plot of $(\bar{a}/2)$ versus $t^{1/3}$ ageing at different temperatures for the test treatment. For key, see Fig. 2. K_{950} = 23.76 nm $h^{1/3}$, K_{875} = 23.18 nm $h^{1/3}$, K_{800} = 20.23 nm $h^{1/3}$.

3. Discussion

The need for materials resistant to high temperatures has been the main promoter of the development and study of special alloys. The influence of heat treatment on the properties of these materials is deeply related to microstructural changes. Some superalloys, with a high volume content of γ', are used in the as-cast condition and microstructures are strongly influenced by the castings parameters previously specified.

To obtain a desired microstructure for a specific purpose, the heat treatment usually consists of two main steps:

(a) a solubilization heat treatment, consisting of annealing at a temperature high enough to homogenize the elements that make up γ' in solid solution, and to dissolve the $M_{23}C_6$-type carbides;

(b) a precipitation heat treatment in the γ/γ' phase region at temperatures lower than those of the previous step, with the aim of obtaining a determined size, morphology and distribution of γ'.

In a schematic phase diagram, such as that of Fig. 4, the sum of elements of the solid solution and the sum of elements forming γ' are plotted against temperature. The different heat-treatment steps according to the phase volume γ' characteristic for each alloy may be observed. In our case, in which the alloy has a high percentage of γ' (60%), it may be used in its as-cast condition, optimized by a standard treatment or improved by the proposed treatment, T_T. We investigated, according to Fig. 4, a treatment consisting of four steps:

1. solution annealing at temperatures higher than 1100 °C;
2. a first ageing annealing at temperatures higher than 1080 °C;
3. a second ageing annealing to obtain a precipitate of thin γ', stabilization of the γ' precipitated and carbides precipitation;
4. a third ageing annealing to increase the amount of thin γ' precipitated.

The results obtained in Fig. 1, size, phase percentage and microhardness of γ', are a function of precipitation temperature (the last step of the treatment) for 16 h. Different precipitate temperatures were investigated for the test treatment, T_p (720, 760, 850, 870 and 950 °C), 926 °C corresponding to the standard treatment, T_S. The best results were obtained for 760 °C. Comparing both T_S and T_T treatments, we can see in Fig. 1 that microhardness increased from 350 Hv to 450 Hv, γ' phase percentage increased from 60% to 70% and the particle size decreased from 0.70 μm to 0.38 μm. In this way we see that test treatment T_T has considerably improved γ' phase with respect to the standard treatment, T_S, obtaining a greater volume fraction, a higher microhardness value and a smaller particle size.

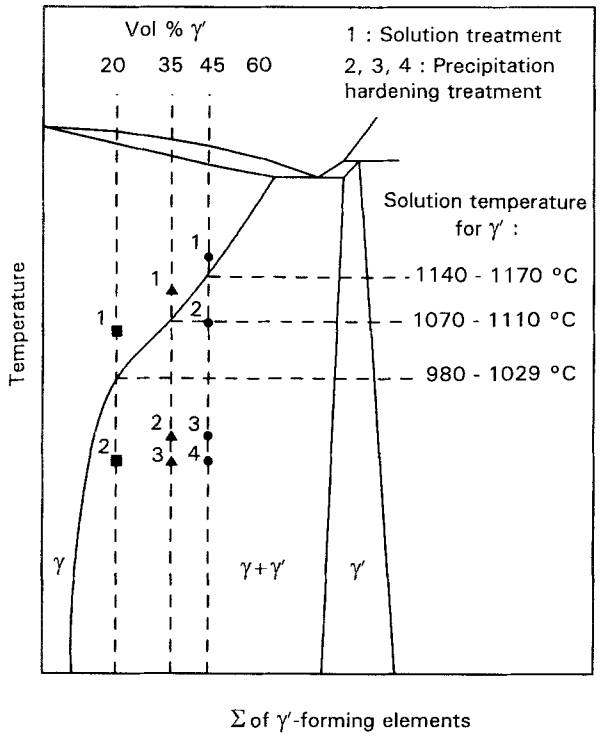

Figure 4 Schematic illustration of heat treatment for γ' hardened superalloys [2].

Analysis of the results obtained up to now finds agreement with those expressed by other authors [2, 18], in that there is considerable evidence suggesting that the volume fraction, f, is the most significant variable controlling the fluency tension and the resistance to creep, together with other factors such as radius and particle size (which basically depends on the cooling rate of the solid after completing solidification), strengthening in a solid solution of γ–γ' and the presence of hyper-thin γ'. The kinetic growth of γ' shows a change in particle average size which depends on the heat treatment carried out. In Figs 2 and 3 we observe that the slope of the straight line T_S, as well as the slope of the straight line T_T, is in each case a constant, K, which gives the speed of volumetric growth for each ageing temperature and for each treatment.

From the analysis of the graphs and from values in Table IV we observe that the slopes of the test treatment, K_T, are lower than the slopes of the standard treatment, K_S, for each ageing temperature, which implies a lower growth speed of γ'. This shows that the test heat treatment, T_T, improves alloy life time with regard to T_S. Data are consistent with the lineal relation. This proves that the growth of γ' in a superalloy of high volume fraction, such as IN-713C, also follows standard kinetics $t^{1/3}$ of particle growth by controlled diffusion, after 800 h ageing at a determined temperature [15]. In this way the γ' particle size can be predicted (or controlled) by extrapolation. The structure of the precipitated phase appears initially directed at random, and as ageing proceeds, cubic γ' particles start to line up along the [1 0 0] directions. The degree of alignment begins to be more significant from a determined particle size and volume fraction. In Figs 5–7 we observe the sequence of particle growth obtained through SEM. Fig. 5 shows particles in their as-cast condition. In Fig. 6, γ' phase precipitate appears after being subjected to the test heat

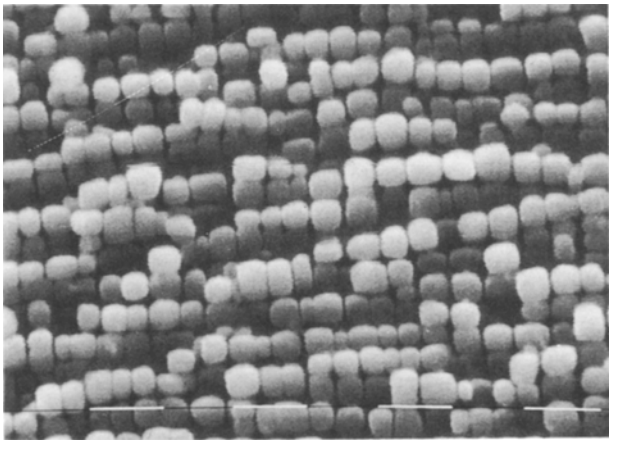

Figure 5 γ', as-cast. × 6400

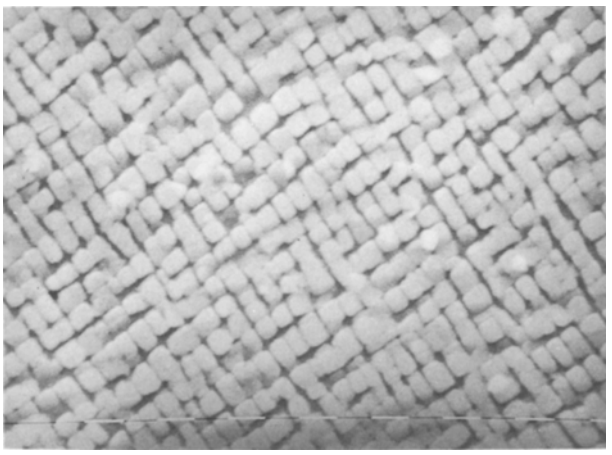

Figure 6 γ', precipitated. × 6400

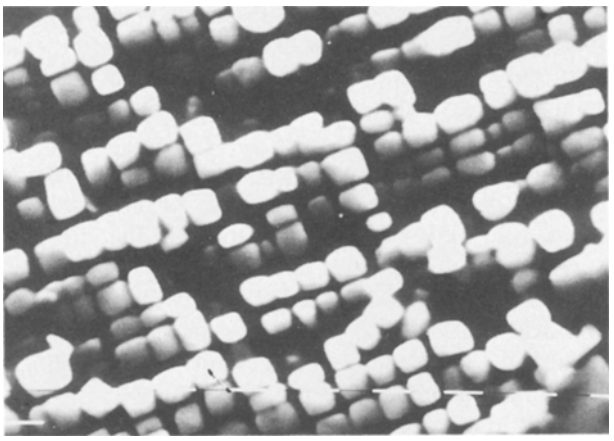

Figure 7 γ', aged for 800 h. × 7680

treatment (two solution and two precipitation). Fig. 7 is the same as Fig. 6 but after 800 h ageing.

4. Conclusions

The results of the present study describe the comparison between a standard heat treatment (solution and precipitation), and a test heat treatment (two solution and two precipitation).

1. The solvus temperature for γ' in an IN-713C superalloy is $1175 \pm 5\,^{\circ}C$.

2. The test heat treatment, with regard to the original one, improves the γ' phase volume precipitated by 16% (60% → 70%), the microhardness value increases by 20% (350 → 425 H_v) and the particle size decreases by 45% (0.70 → 0.38 μm).

3. γ' particle growth follows standard kinetics $r \propto t^{1/3}$ by controlled diffusion for both heat treatments.

4. The γ' phase growth speed is lower for K_T than for K_S.

5. Particles become aligned in the [1 0 0] direction; this is more marked as ageing progresses.

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