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Characteristics of the γ' precipitates at high temperatures in Ni-base polycrystalline superalloy IN738LC

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IN738LC is a Ni-base cast superalloy used in land-base gas turbines and aerospace applications. As in other superalloys γ' precipitates contribute to strengthening of this alloy at high temperatures. In this study, the authors investigate the characteristics and mechanisms of precipitate dissolution into the matrix solid solution. The precipitates grow in cuboidal shape up to 1130°C, above that a duplex-size precipitate microstructure sets in upon quenching from the temperature range 1140–1150°C. The duplex-size precipitate microstructure consists of two very distinct sizes of precipitates (fine and coarse). Holding for longer times in the temperature range 1140–1150°C does not coarsen the fine precipitates of the duplex microstructure. The source for the formation of the fine precipitates in the duplex microstructure is the dissolution of the newly grown smaller-sized precipitates when the agings start from fine size precipitates and the "corner dissolution" of coarse precipitates when the starting microstructure consists of the maximum-sized cuboidal precipitates. At and above 1160°C, the duplex as well as the coarse precipitate microstructures dissolve to form a single-size fine precipitate microstructure upon quenching from any temperature up to 1225°C. A single-phase solid solution with no precipitates is obtained only upon quenching from 1235°C or above. The dissolution of coarse precipitates and formation of the fine ones are found to be very fast processes in the corresponding temperature ranges. The fine precipitates are postulated to form during quenching from the temperature range 1140–1225°C and are considered to be of the "cooling" type.

1. Introduction

IN738LC is used in gas turbine engines at high temperatures. In fact, the temperature range in which this superalloy could be utilized more effectively depends mainly on the formation, morphology, and the stability of the γ' precipitate which is the strengthening phase in this superalloy.

Developing suitable processes to control and stabilize the microstructure, which would improve the mechanical properties of materials, is important in industrial applications. Second phase particle dissolution has been a topic of interest to materials scientists for a long time. Different mechanisms have been proposed for the dissolution of precipitate particles into matrix. Aaron and Kotler [1] considered the curvature and hence concentration gradient effects at the precipitate/matrix interface. A three-step model was proposed by Vermolen and Zwaag [2] for second phase particle dissolution: decomposition of the particles, crossing of the boundary, and long distance diffusion into the matrix. They reported that interface interactions are the rate controlling processes for the dissolution of compound type particles, such as Ni_3Al (γ'). The dissolution and reformation of precipitate phase is also discussed by Solorzano [3]. In practical applications such as during fusion welding, it is found that high peak temperatures cause precipitate dissolution [4].

The authors of this paper compiled their earlier results on the microstructure development in the superalloy IN738LC in two different papers [5, 6]. The precipitate growth mechanisms and the kinetics were studied in Ref. [5]. It is reported there that after the 1120°C/24h/FC aging treatment subsequent to 1200°C/4h/WQ or 1250°C/4h/WQ solution treatments, the maximum precipitate size of ∼700 nm was obtained in cuboidal shape. At the same temperature, when the specimens were aged for longer time periods of 48 and 72 hours, a duplex-size (fine + coarse) precipitate microstructure was obtained [6]. Size of the fine precipitates was approximately 50–70 nm, and no change in size of the fine precipitates was observed, whereas the coarse ones kept on growing with time. It was also reported in Ref. [6] that the alloy shows a duplex-size precipitate microstructure when quenched from the range 1140–1150°C, only the fine precipitates embedded uniformly in the matrix when quenched from the range

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TABLE I Chemical composition of the as-received IN738LC

Element	Ni	Cr	Co	Mo	W	Ta	Nb	Al	Ti	Zr	B	C
wt.%	balance	15.7- 16.3	8.0- 9.0	1.5- 2.0	2.40- 2.80	1.5- 2.0	0.60- 1.10	3.20- 3.70	3.20- 3.70	0.03- 0.08	0.007 0.012	0.09- 0.13

1160–1225°C, and a single-phase solid solution with no precipitates when quenched from ∼1235–1250°C. A first level analysis of the dissolution kinetics of coarse precipitates and enunciation of a possible mechanism for the fine γ' precipitate formation was undertaken in the earlier study [6]. Activation energy calculations showed that the precipitates were in an easy dissolution mode in the temperature range 1140–1150°C.

The current study presents some results complementary to those in Ref. [6]. The formation of fine precipitates in the duplex as well as in the single-size, fine precipitate morphologies is studied further in this paper.

2. Experimental

Howmet Corporation, Whitehall, Michigan provided the polycrystalline IN738LC cast superalloy in the form of rods, 15 mm in diameter and 110 mm in length. The as-received stock had already been “hipped” (hot isostatically pressed) and heat-treated after casting as indicated in the literature (1120°C/2h/AC followed by an aging treatment at 850°C/24h/AC). The chemical composition of the as-received material is given in Table I, and its microstructure is shown in Fig. 1.

All the heat treatments discussed in this paper were carried out under vacuum by sealing the specimens in silica tubes. After agings, the specimens were ground and polished using regular metallographic techniques and etched with a solution of composition 33% $HNO_3 + 33\%$ acetic acid $+ 1\%$ HF $+ 33\%$ H_2O. A Hitachi S-2460N type scanning electron microscope (SEM) was used to characterize the microstructure and morphology of the γ' precipitates. X-ray analysis of different precipitate microstructures was carried out using 10-mm diameter and 2-mm thick round specimens to reveal the crystal structures of the different size precipitates. Further experimental details can be found in Refs. [5, 6].

3. Results

Two different solution treatments were applied to the specimens. The one at 1200°C/4h/WQ yielded a fine size (∼70 nm) precipitate microstructure as seen in Fig. 2, while the other at 1250°C/4h/WQ produced the single-phase solid solution matrix with no precipitates. Results of X-ray analysis of the specimens also supported these findings [7]. After the solution treatment, four different types of aging treatments were applied to the specimens to study the mechanisms of formation of the fine and duplex-size (coarse + fine) precipitate microstructures.

Two of the four aging treatments are discussed in detail in Ref. [6]. The first one, called Type-I treatment, consisted of holding (aging) the specimens at 1140°C for various time periods after the solution treatment of 1200°C/4h/WQ (starting microstructure for Type-I aging treatments consisted of only fine precipitates, ∼70 nm in size, evenly distributed in the matrix). The duplex-size precipitate microstructure was first observed only after an aging for 30 minutes at this temperature in this type of treatment. Very fine precipitates of the duplex microstructure, barely visible after 30 minutes, could be seen clearly after the 2 and 4-hour aging treatments.

In the second procedure, after solutionizing at 1200°C/4h/WQ, the specimens were first aged at 1120°C/24h/FC to obtain the single-size coarse unimodal-cuboidal precipitates with about 700 nm size, as seen in Fig. 3. Then, these specimens were aged at 1140 and 1200°C for 1, 3, 5, and 30 minutes and at 1160°C for 15, 30 and 60 seconds to observe the dissolution of the coarse unimodal-cuboidal precipitates and the formation and growth of the finer ones. This procedure was designated Type-II (starting microstructure for the agings consisted only of maximum size unimodal-cuboidal precipitates, ∼700 nm in size). The duplex-size precipitate microstructure was found

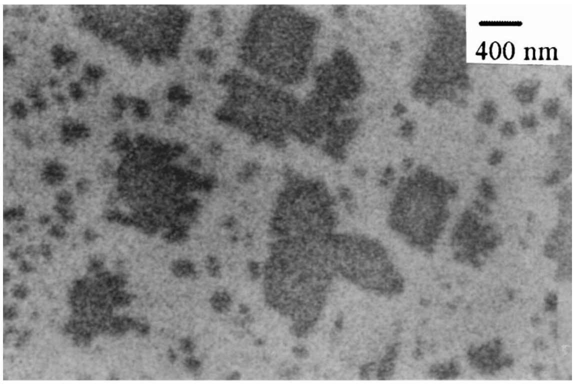

Figure 1 Duplex-size microstructure of the as-received IN738LC. The dark regions are the γ' precipitates. (Magnification: 15kX).

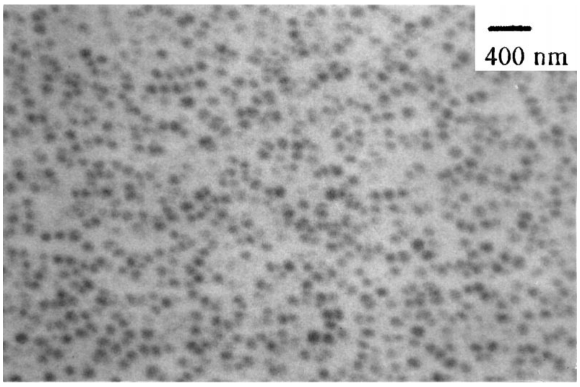

Figure 2 Microstructure obtained after 1200°C/4h/WQ solution treatment (starting microstructure for Type-I treatments). (Magnification: 15kX).

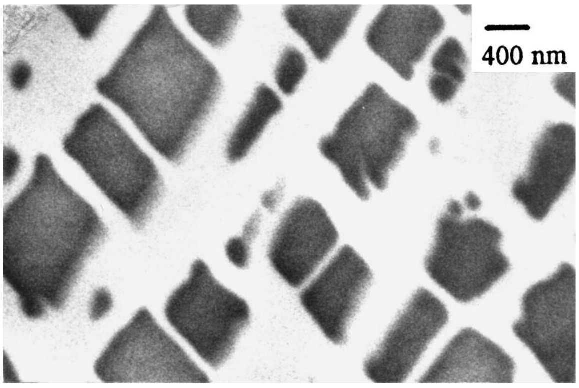

Figure 3 Microstructure obtained after 1120°C/24h/FC subsequent to the solution treatment, showing the maximum size (single size coarse) of the γ' precipitates (starting microstructure for Type-II treatments). (Magnification: 15kX).

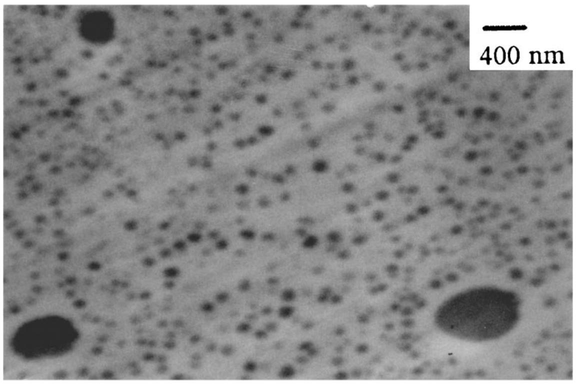

Figure 4 The partially fine precipitate microstructure obtained through Type-II heat treatment- 1200°C/4h/WQ + 1120°C/24h/FC + 1160°C/t/WQ. $t = 15$, 30, and 60 s. (Magnification: 15kX).

after holding the specimens for 5 minutes at 1140°C and subsequent water quenching. However, a fine-size precipitate microstructure, as shown in Fig. 2, could be obtained after holding for 1 minute at 1200°C and subsequent quenching to room temperature. In Type-II treatment, after application of very short aging times (15, 30, and 60 seconds) at 1160°C, fine-size precipitates could be observed even after the first 15 seconds of aging, along with a few coarser ones at their final stages of dissoution (leading to the partially fine microstructure). This is illustrated in Fig. 4.

In the third procedure, Type-III, specimens were solution-treated at 1250°C/4h to obtain the single-phase solid solution matrix and then directly transferred to another furnace maintained at 1140°C. The specimens were aged at this temperature for 1, 3, 5, and 30 minutes. This procedure was set up to observe the evolution of the precipitates from the supersaturated solid solution without allowing any nucleation that might happen during quenching to room temperature via spinodal decomposition [8] or reheating of the specimens for aging purposes after quenching to room temperature. The microstructures obtained, given in Fig. 5, are quite similar to those obtained after Type-I heat treatments at 1140°C [6]. It was indicated earlier that the duplex microstructure was found to have developed after about 30 min. of holding time at 1140°C in Type-I treatment involving fine precipitates to start with. Similar behavior was also observed in Type-III treatment where no precipitate should have been present to start with.

In the fourth heat treatment procedure, Type-IV, first the duplex-size precipitate microstructure, with coarese-cuboidal precipitates of 450 nm and fine precipitates of 50 nm size, was developed in the specimens [6]. Then, they were aged at 1160°C for 15, 30, and 60 seconds to observe the change in the duplex microstructure when aged at the fine size precipitate formation temperature. Gradual reduction in the size of coarse precipitates in the duplex-size microstructure was observed with increasing time, as illustrated in Fig. 6.

4. Discussion

IN738LC prefers the duplex-size γ' precipitate microstructure when held at and quenched from the temperature range of 1140–1150°C, size of precipitates depending on the aging time and temperature, as already mentioned.

Two possible mechanisms could be deduced for the dissolution of coarser precipitates at temperatures above about 1130°C. One is the corner dissolution of coarse precipitates [6] which incorporates solute into the matrix solid solution and leads to the evolution of the fine-size precipitates of the duplex microstructure at the aging temperature or more possibly during quenching to room temperature. A second possible mechanism is a stepwise splitting of the coarser precipitates into smaller ones for the purpose of easier dissolution. Coarse precipitates usually split into two fractions and dissolution of the split portions occurs starting at the split plane. Splitting of coarse precipitates is evident in Fig. 6c. In Fig. 6c some of the precipitates are found to have split possibly parallel to {111} planes. Fig. 6d shows some precipitates thinned down along the side edges, pointing to the dissolution progressing along the {100} planes, as well.

Some precipitates, being distinctly smaller in size (newly grown precipitates), migh directly dissolve into the matrix due to curvature effects, giving rise to reformation of evenly distributed fine precipitates in the matrix during quenching.

After the early stages of the aging treatments, very fine precipitates form, apparently during quenching, in all of the three experimental procedures (Type-I, Type-II, and Type-III). The size of the very fine precipitates formed at the initial stages in the duplex microstructure is smaller than that of the precipitates in the 1200°C solution-treated and quenched microstructure. This is probably because there is not enough solute supply for the fine precipitate formation and growth [6]. The same explanation applies also for the Type-II heat treatments at 1140°C, since there is not enough “corner dissolution” to enable the growth of the very fine precipitates. However, it is clear that the size of the very fine precipitates should slowly rise to that of 1200°C solution-treated and quenched microstructure after all of the coarse precipitates dissolve with increasing temperature. It is also obvious that the formation of the very fine precipitates is a relatively very rapid process, for they

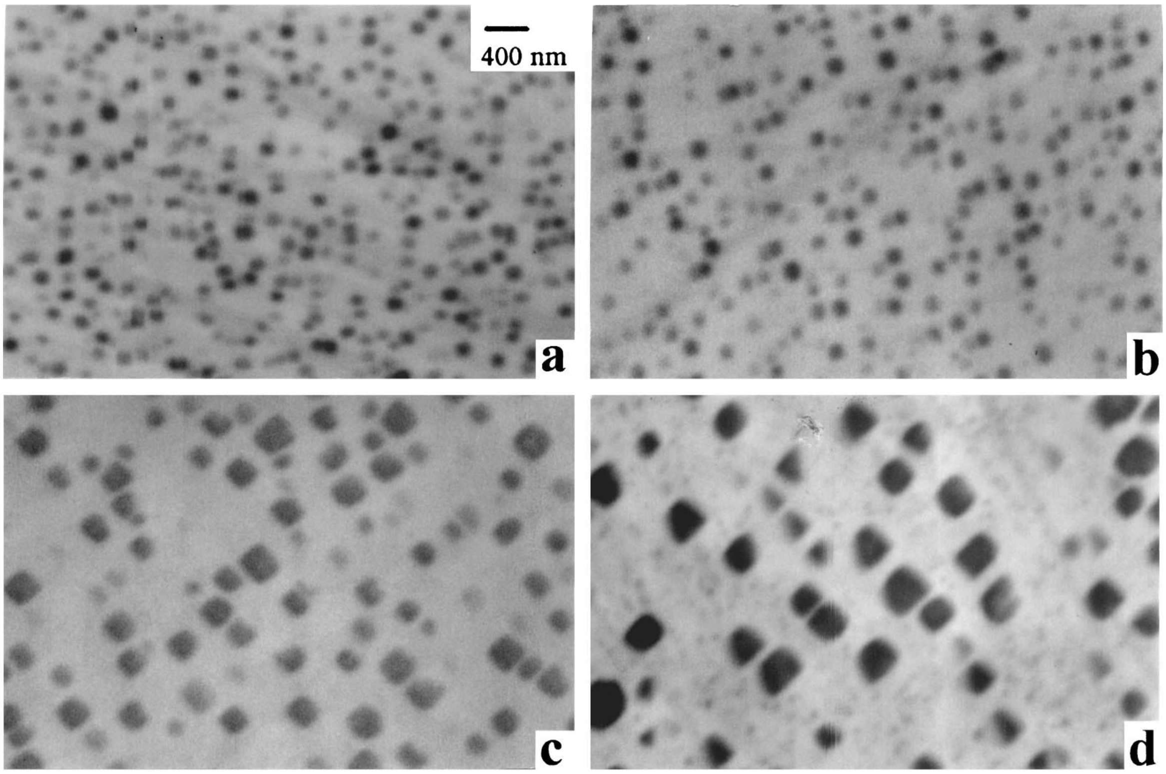

Figure 5 The duplex-size γ' precipitate microstructure obtained through Type-III heat treatment- 1250°C/4h + 1140°C/t/WQ. (Magnification: 15kX). a) $t = 1$ min. b) $t = 3$ min. c) $t = 5$ min. d) $t = 30$ min.

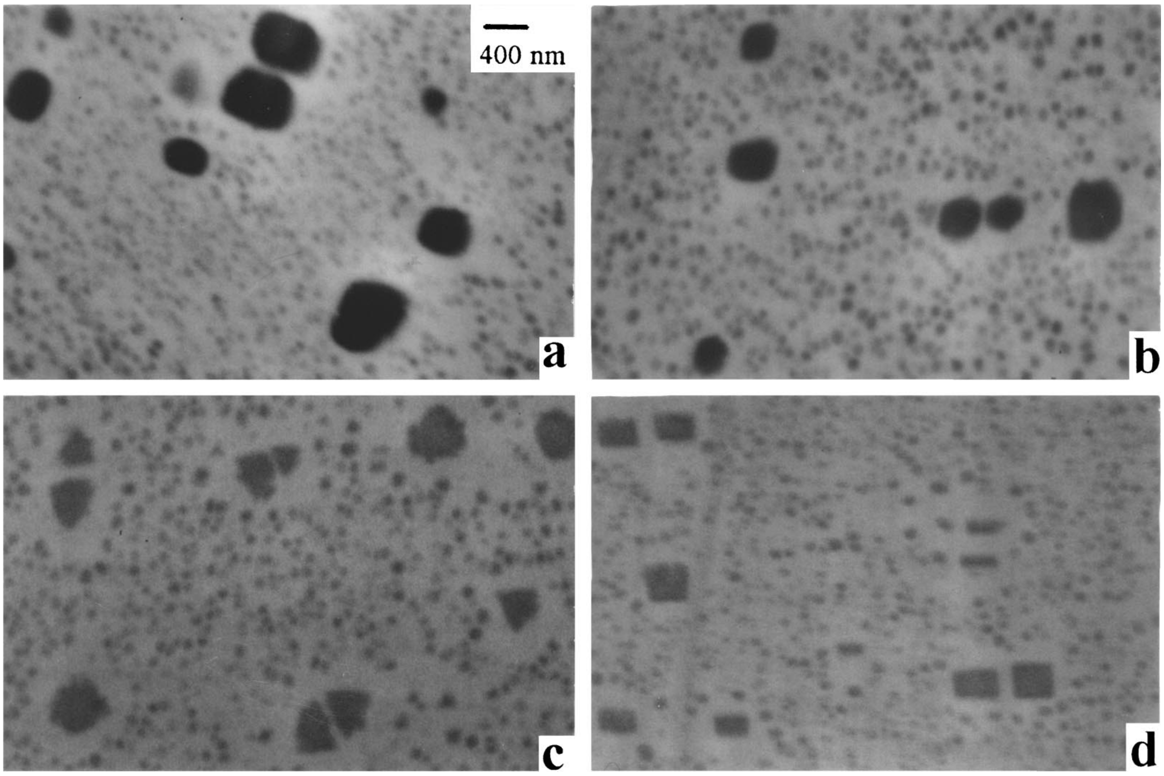

Figure 6 The microstructure obtained through Type-IV heat treatment- 1200°C/4h/WQ + 1140°C/4h/WQ + 1160°C/t/WQ. (Magnification: 15kX) a) $t = 15$ s b) $t = 30$ s c) $t = 60$ s d) same specimen as 'c' showing different area.

are most probably produced during quenching. Likewise, the dissolution of most of the coarse precipitates at 1160°C is a very fast process, for it yields the partially fine precipitate microstructure in as little as 15 seconds, Fig. 4. One important feature of the micrograph in Fig. 4 is that the size of the fine precipitates is already large, comparable to the size of the fine precipitates obtained after the 1200°C/4h/WQ solution-treatment, shown in Fig. 2. Further, the very fine precipitates of the starting duplex microstructure seem to have grown also in such a short time, Fig. 6. The dissolution is aided by the splitting of the coarse precipitates, and this might continue until the coarse precipitates dissolve completely and form the fine ones.

Because of the fact that the materials prefer the lowest possible total interface-related energy, it appears that the coarse γ' precipitates dissolve at higher than a critical temperature and/or size, due possibly to the need for a sharp reduction in interfacial (coherency-related) misfit strain energy. As already mentioned above, precipitates grow in a unimodal-cuboidal shape upto 1130°C/24h/FC. The duplex-size microstructure, obtained at the lower temperature of 1120°C after the 48 and 72-hour aging treatments [6], is an indication of the precipitate size and misfit strain energy effects for the initiation of dissolution and subsequent formation of the duplex size microstructure. In addition, the apparent formation of the duplex-size microstructure in a shorter span of time via Type-II treatment (5 min.) than Type-I and Type-III treatments (30 min.) at 1140°C is an evidence for the size effect (the need to reduce the interfacial, coherency-related misfit strain energy) on the formation of duplex-size γ' microstructure. Moreover, splitting and dissolution of the maximum sized coarse precipitates and formation of partially fine size precipitate microstructure at 1160°C occurs in as little as 15 seconds. Also, comparing Figs 4 and 6 in which the specimens were finally aged at 1160°C, the number of the coarse precipitates is higher in Fig. 6. The starting size of the coarse precipitates in Fig. 6 was much smaller than the initial size of the coarse ones in Fig. 4, 450 nm vs. 700 nm, respectively. This initially smaller size of the coarse precipitates in Fig. 6 due to dissolution might have reduced the misfit-strain energy and enabled their slower further dissolution at 1160°C. The coarser the precipitates, the faster they seem to dissolve. These experimental results may be used to conclude that the dissolution of coarse ones and the eventual reformation of the fine-size precipitate microstructure after holding at the highest possible temperature are determined perhaps by the need to reduce the interfacial coherency-related misfit strain energy, as explained above.

5. Conclusions

The following can be concluded based on the findings of this study:

1. "Corner dissolution" of coarse precipitates is one of the modes for the precipitate dissolution and formation of the fine precipitates of the duplex-size microstructure. The other one is the dissolution of the newly grown smaller precipitates in the temperature range 1140–1150°C. Also, precipitate splitting aids in the dissolution of coarse precipitates and the eventual reformation of fine ones.
2. The fine precipitate microstructure could be obtained after holding for fifteen seconds in the range 1160–1225°C and quenching. These precipitates are formed either at the aging temperature or most probably during water quenching to room temperature. Very coarse precipitates almost completely dissolve and the fine ones get formed in a matter of few seconds.
3. It is proposed that the need for a large reduction in coherency-related interfacial misfit strain energy is perhaps one of the reasons for the formation of duplex-size microstructure and dissolution of the coarsest precipitates and development of the refined fine precipitate microstructure.

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