

Effect of the Particle Size of γ' Phase on the Mechanical Properties of Ni base Superalloy

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Abstract. The effect of γ' particle size upon the mechanical properties of Ni base superalloy EP741NP obtained by powder metallurgy was investigated. The particle size of γ' phase in γ - γ' microstructure was varied by changing the cooling rate V from the temperature of the solid solution treatment at 1200 °C ($V = 80, 200$ and 400 °C \ min.). After solid solution treatment billets were subjected to aging in the standard mode. It was established that as V increases from 80 to 200 °C \ min., the average particle's size of γ' phase decreases from 0.54 microns to 0.22 microns in the aged state. This improves the characteristics of creep and low cycle fatigue at 650°C: time to rupture under load 1000 MPa increased from 132 hours to 416 hours and low cycle fatigue increased from 42,215 to 82,016 cycles.

Introduction

Ni base superalloys are among the classic precipitation hardening alloys. The alloys are strengthened by dispersion in the γ matrix of particles based on the L_{12} intermetallic compound Ni_3Al [1, 2]. The ordered structure L_{12} can be sheared by matrix dislocations traveling in pairs, thus forming a total Burgers vector $2(a/2)[110]$ twice as long as in fcc crystals. The antiphase boundary (APB) energy pulling the two similar dislocations together is of the order of $\gamma_{APB} \approx 150$ mJ/m², which is almost an order of magnitude greater than the energy of coherent interphase boundaries γ/γ' . The low energy of interphase boundaries γ/γ' provides high stability of $\gamma - \gamma'$ microstructure against the process of diffusion coarsening at elevated temperatures, and the formation of APBs in the ordered structure of γ' phase triggers one of the most effective mechanisms of hardening.

The change in yield strength τ with particle size can be presented as [1]

$$\tau = \alpha \gamma_{APB}^{3/2} f^{1/3} r^{1/2} \quad (1)$$

Here f is the volume fraction of γ' phase, r is the average size of particles.

The parabolic $r^{1/2}$ dependence indicates that larger precipitates will increase the critical stress. This is true until another mechanism (Orowan mechanism) requiring less energy becomes possible. The critical stress for Orowan by-passing by single dislocations falls off as $1/r$. Therefore, there is an optimum particle size of γ' phase in γ matrix, which ensures the maximum hardening effect.

The optimum size of γ' particles in Ni base superalloys was defined, for example, by the authors [3, 4] on the alloy CMSX-2. Alloy samples were subjected to heat treatment at the temperature of 1315°C for 30 minutes for homogenization and dilution of γ' phase. After homogenization, samples were divided into 2 groups and each group was two-step aged using the following regimes: i) 980°C - 5 hours, air cooling + 850°C - 48 hours, air cooling (T_1), ii) 1050°C - 16 hours, air cooling + 850°C - 48 hours, air cooling (T_2).

As a result of the T_1 heat treatment the particles of γ' phase had sizes in the range of 0.25 - 0.36 microns. After treatment T_2 more homogeneous $\gamma - \gamma'$ microstructure was formed with larger particle sizes of γ' phase: the average particle size was equal to 0.45 microns.

The different regimes of heat treatments had a strong influence on the creep characteristics of the samples. Thus, times to rupture were 570 and 1125 hours in the states of T_1 and T_2 , accordingly, at the temperature of 760° C under a stress of 750 MPa. Consequently, the more dispersed $\gamma - \gamma'$ microstructure in single crystals of the alloy CMSX-2 in the state of T_1 was much less resistant to the creep than the state of T_2 . Thus, the results of [3, 4] are consistent with the theoretical predictions [1]: the larger the size of the γ' particles the better is the resistance against creep.

However, it should be noted that according to [3, 4] the state of T_1 was characterized by a more regular $\gamma - \gamma'$ microstructure than T_2 . That is why we believe that the optimum size of cubic particles of the hardening γ' phase in a regular microstructure of superalloys requires further studies.

In the present work the influence of cooling rate (80, 200 and 400 °/min.) from 1200°C (the temperature of homogenization) to room temperature on the microstructure, formed during the subsequent aging, and on the mechanical properties was investigated for the Ni base superalloy EP741NP. EP741NP is a typical precipitation hardening alloy obtained by powder metallurgy [5,6,7,8, and 9].

Material and experimental technique

The study was conducted on the disk alloy EP741NP obtained by powder metallurgy. The chemistry of the EP741NP under investigation is given in Table 1 [10].

Table 1. The chemical composition of the alloy EP741NP (weight %).

Cr	W	Mo	Nb	Co	Al	Ti	Hf	Ta	Zr
8,64	5,68	3,66	2,46	15,73	5,10	1,9	0,25	0,15	0,026
Fe	Si	C	Mn	B	V	Cu	P	S	Ni
0,12	0,035	0,05	0,019	0,014	0,01	0,01	0,001	0,001	bal.

Compacted billets were quenched from the temperature of 1200°C after holding for 4 hours in a vacuum oven SEKO\WARWIK. The required rate of cooling (80, 200 and 400 °C/min.) from a temperature of 1200°C to 700°C was achieved by cooling with nitrogen and using the billets with different weights from 1 to 110 kg.

Control thermocouples were used to determine the actual cooling rate of the billets. After quenching, the billets were subjected to a double aging on the standard mode of processing the alloy EP741NP. The billets were cooled with different rates 80 (V_1), 200 (V_2) and 400 °C/min. (V_3) from the temperature of 1200°C and then subjected to the two-step aging.

Metallographic study of $\gamma - \gamma'$ microstructure of the alloy in different states was carried out using scanning electron microscopy (SEM). Analysis of images of the microstructure included the possibility of constructing the size distributions of particles of γ' phase and verify compliance of the experimental distributions and the canonical curve in line with the model of Lifshitz-Slyozov-Wagner (LSV; see, for example, [11]).

Experimental results and discussion

Figure 1 (a and b) shows the SEM micrographs with the different sizes of the γ' phase in the states of V_1 and V_2 . The γ' particles have mostly cubic shape in both states and are distributed in a regular manner in γ matrix. The experimental size distributions of γ' phase demonstrated that the mean particle size equals to 0.54 microns in the condition of V_1 , and this parameter decreases to 0.22 microns in the state of V_2 . The microstructure of the alloy in the V_3 state practically does not differ from the state of V_2 .

Significant change in particle size of γ' phase in the states of V_1 , V_2 and V_3 had no significant effect on the short-term mechanical properties at room temperature (Table 2). One can note a slight increase in $\sigma_{0,2}$, accompanied by a decrease in elongation to failure δ , as a trend.

Table 2. Results of the short-term testing of mechanical properties of Ni base superalloy EP741NP at room temperature in different states. σ_b is the ultimate tensile strength, KCU is impact toughness.

State	Mechanical properties				
	σ_b , N\mm ²	$\sigma_{0,2}$, N\mm ²	δ , %	ψ , %	KCU, Kg.m\cm ²
V_1	1512	1066	22,7	23,6	6,1
V_2	1575	1191	19,7	23,2	5,7
V_3	1550	1132	19,3	21,4	--

The difference in the size of γ' phase particles in the states of V_1 and V_2 has much stronger impact on the long-term mechanical properties. It was found that at 650°C the time-to-failure under an applied creep stress of 1000 MPa was 132 and 416 hours in the states of V_1 and V_2 , accordingly. In the state of V_3 the samples were not destroyed during the tests up to 261 hours. Low-cycle fatigue tests were carried out at 650°C with a frequency of 1 Hz and a stress range between 980 MPa $\geq \sigma \geq$ 0. Numbers of cycles to failure were 45,215 and 82,016 in the states of V_1 and V_2 , accordingly.

High values of the long-term mechanical properties in the state of V_2 were also obtained at other temperatures and stresses. For example, time-to-failure at 650°C under a stress of 1050 MPa was 122 hours, and it was 107 hours at 1100 MPa. Time-to-failure at 750°C under a stress of 700 MPa was 136 hours, and it was 182 hours at 650 MPa.

One can see the formation of the relatively regular uniform size of cuboidal shape of the γ' phase in the states of V_1 and V_2 . The main difference between microstructures of V_1 and V_2 is that the mean particle size of γ' phase in the state of V_2 is approximately two times lower compared to V_1 . Consequently, these results show that γ - γ' microstructure of Ni base superalloy EP741NP with a smaller particle size of γ' phase is characterized by higher values of the long-term mechanical properties.

It is obvious that the observed behavior of γ - γ' microstructures in the states of V_1 and V_2 is not consistent with the representations of the mechanism of cutting (formula (1)). According to the mechanism of cutting, the microstructure changes (antiphase boundaries) are accumulated in *the bulk* of γ' phase.

Apparently, the creep behavior of the Ni base superalloy under consideration can be associated with another mechanism of strengthening. One can expect the change of several structure factors when the size of γ' phase particles reduces: a) increase of the number of γ' particles, b) increase of the interphase boundaries γ/γ' in the specific area and c) decrease of the width of the layers of γ matrix between the particles of γ' phase. Each of these factors can trigger the specific mechanism of creep resistance, thereby increasing the creep life of the alloy.

During the creep tests in our case the mechanism of coarsening of the γ' phase particles in γ matrix appears to be inconsistent with the LSV model behavior. Indeed, in the initial state, after full heat treatment, the experimental histograms are in a good agreement with the LSV model particle size distribution of the γ' phase, Fig. 2 (a). However, after testing at 650°C under load of 1000 MPa during 416 hours there is a noticeable discrepancy between the experimental histogram and the model curve in Fig. 2 (b).

One can suppose that there is a special mechanism of diffusion coalescence of the isolated particles of γ' phase, which begins to operate in conditions of the creep, when the dislocation density in γ matrix increases and a non-equilibrium state of the γ/γ' interfaces can be formed. It is interesting to note that 650°C (temperature of tests on creep) is a particular temperature from the point of view of the diffusion permeability of the interfaces that interact with lattice dislocations [12, 13, 14, and 15].

Conclusions

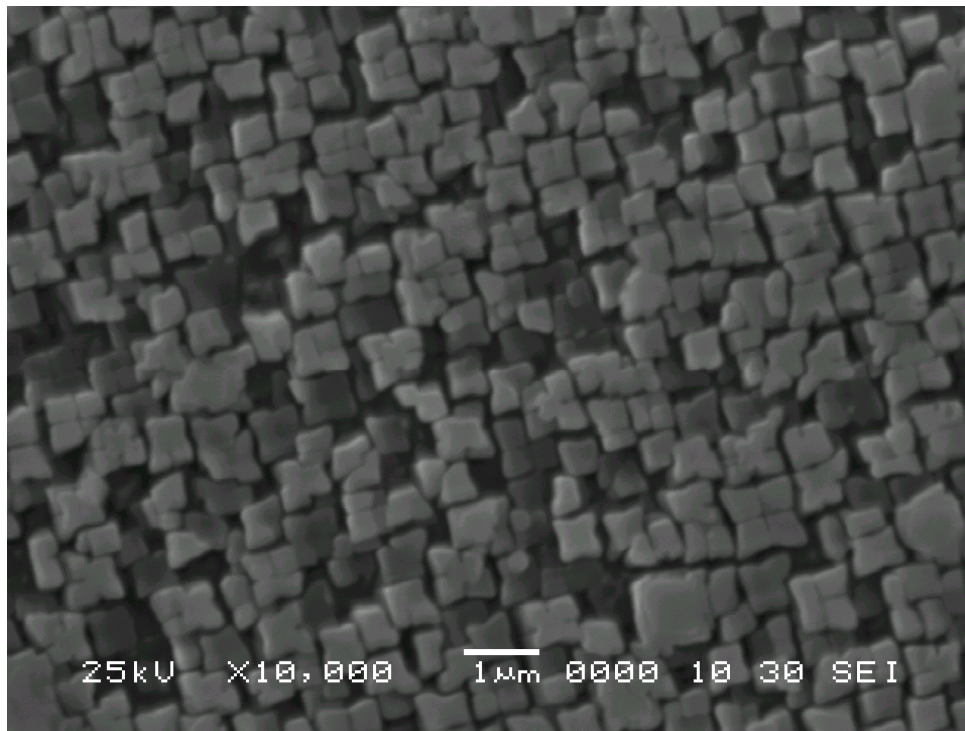
The decrease in the size of regular γ' phase in the V_2 state improves the characteristics of heat resistance at 650° C, compared to V_1 : time to rupture under a stress of 1000 MPa increased from 132 to 416 hours, the number of cycles to failure (low cycle fatigue) increased from 45,215 to 82,016 cycles.

Acknowledgements

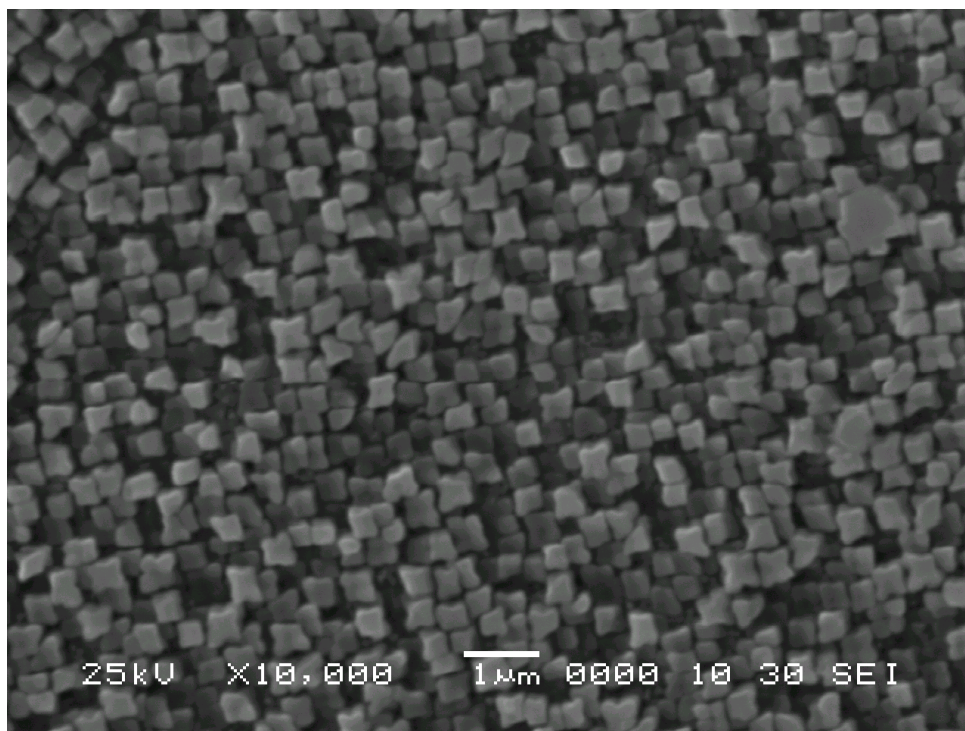
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References

1. J.-L. Strudel, in: Physical metallurgy, eds. by R.W. Cahn and P. Haasen, vol. III, the Netherlands, Elsevier Science (1996) p. 2147
2. B.A. Kolachev, V.I. Elagin and V.I. Livanov. Metallovedenie i termicheskaya obrabotka cvetnyh metallov i splavov. Moscow: MISiS (2005) 328 p.
3. P. Caron, T. Khan, Mat. Sci. Eng. 61 (1983) p. 173
4. P. Caron, T. Khan, Aerosp. Sci. Technol. 3 (1999) p. 513
5. A.I. Logacheva, A.G. Beresnev, A.V. Logunov, T.G. Bogdanova, A.V. Logachev. Aviakosmicheskaya tekhnika & tekhnologiya 2 (2008) p. 35
6. J. Radavich, D. Furrer, T. Carneiro, J. Lemsky, in: Superalloys 2008, eds. by R.C. Reed et. al., TMS (The Minerals, Metals & Materials Society), 2008, p. 63
7. J. Radavich, D. Furrer, in: Superalloys 2004, eds. by K.A. Green et. al. TMS (The Minerals, Metals & Materials Society), 2004, p. 381
8. G.S. Garibov, A.V. Vostrikov, in: Proc. of the 2005 International Conference on Hot Isostatic Pressing. Paris, May 22-25, 2005, p. 86
9. G.S. Garibov. Tekhnologiya legkikh splavov № 5-6 (2001) p. 138
10. Y.G. Bykov, I.M. Razumovskii. Perspektivnye Materialy 1 (2010) p. 10
11. R.D. Doherty, in: Physical metallurgy, eds. by R.W. Cahn and P. Haasen, vol. III, the Netherlands, Elsevier Science (1996) p. 1438
12. L.G. Kornelyuk, A.Y. Lozovoi, I.M. Razumovskii. Phil. Mag. 77 (1998) p. 465
13. S.Z. Bokstein, N.V. Migunova, I.M. Razumovskii. Fizika metallov i metallovedenie 9 (1990) p. 191
14. R. Valiev, L. Kornelyuk, I. Razumovskii. Dokl. Akad. Nauk SSSR 320 (1991) p. 322
15. L. Kornelyuk, R. Valiev, V. Sergeev, I. Razumovskii. Mater. Sci. Eng. A. 167 (1993) p. 123

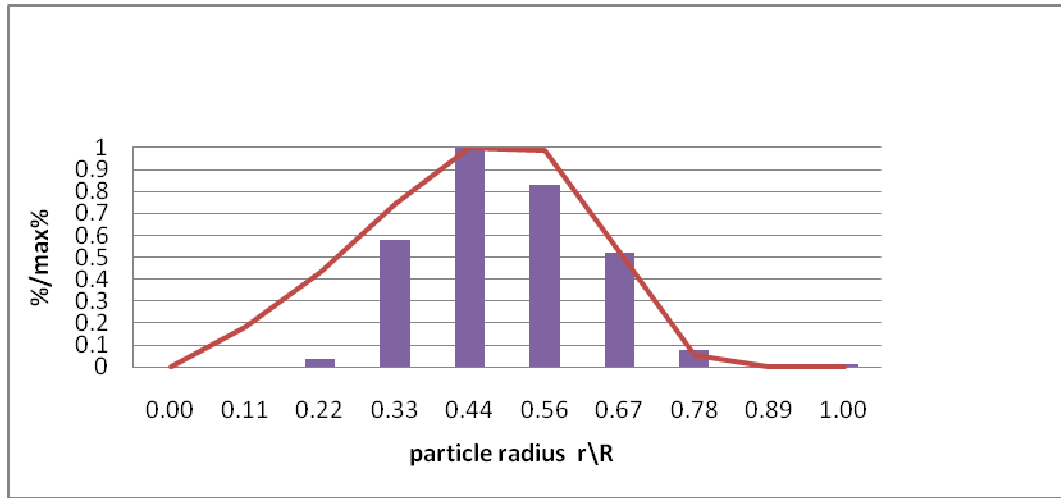


(a)

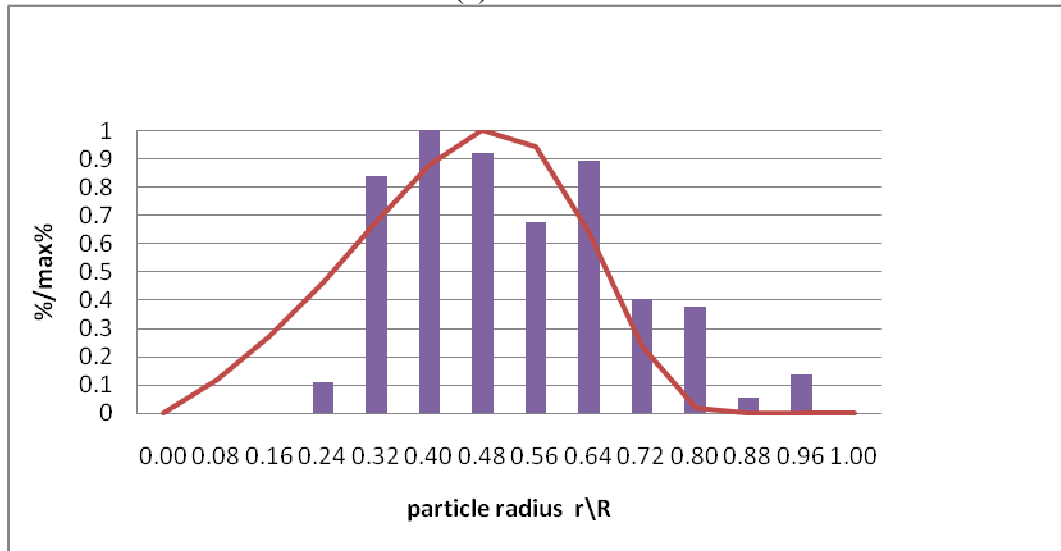


(b)

Fig. 1. SEM micrographs showing the morphology and size of the γ' phase in the states of V_1 (a) and V_2 (b).



(a)



(b)

Fig. 2. The distributions of particles of γ' phase according to the size, the state of V_2 : (a) the initial state (after a full cycle of heat treatment), (b) - after testing $650^\circ\text{C}/1000\text{ MPa}/416\text{ hours}$. The experimental data are presented by histograms; the curves correspond to the LSV model distributions.