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Marina Fedorischeva

Citation: AIP Conference Proceedings **1909**, 020149 (2017);
View online: https://doi.org/10.1063/1.5013830
View Table of Contents: http://aip.scitation.org/toc/apc/1909/1
Published by the American Institute of Physics

Orientation and Faulted Structure of γ′-Phases in Lanthanum-Alloyed Ni-Al-Cr Superalloy

Elena Nikonenko[1,2,a)], Lyubov' Shergaeva[2,b)], Natalya Popova[2,3,c)], Nina Koneva[2,d)], Rongshan Qin[4,e)], Victor Gromov[5,f)], and Marina Fedorischeva[3,g)]

[1] *National Research Tomsk Polytechnic University, Tomsk, 634050 Russia*
[2] *Tomsk State University of Architecture and Building, Tomsk, 634003 Russia*
[3] *Institute of Strength Physics and Materials Science SB RAS, Tomsk, 634055 Russia*
[4] *School of Engineering and Innovation, Open University Walton Hall Milton Keynes MK7 6AA, United Kingdom*
[5] *Siberian State Industrial University, Novokuznetsk, 654007 Russia*

[a)] vilatomsk@mail.ru
[b)] shergaeva@bk.ru
[c)] natalya-popova-44@mail.ru
[d)] koneva@tsuab.ru
[e)] Rongshan.Qin@open.ac.uk
[f)] gromov@physics.sibsiu.ru
[g)] Corresponding author: fed_mv@mail.ru

Abstract. The paper presents the transmission and the scanning electron microscope investigations of thin foils of Ni-Al-Cr-based superalloy, which is obtained by the directional crystallization technique. This superalloy contains γ′- and γ-phases. Additionally, lanthanum is introduced in the superalloy in 0.015, 0.10 and 0.30 wt % concentrations. The superalloy specimens are then subjected to 1273 K annealing during 10 and 25 h. It is shown that γ′-phase is major. In the superalloy, lanthanides La_2Ni_3 and Al_2La are detected along with carbide La_2C_3 particles located on dislocations of the major phase. The amount of phases in the superalloy depends on its thermal treatment and lanthanum concentration. The investigations include the effect of annealing on scalar density of dislocations in γ′-phase. It is demonstrated that lanthanum alloying modifies the preferred orientation of γ′-phase. Annealing of lanthanum-alloyed superalloy causes the orientation dispersion. In γ′-phase, the correlation is observed between the degree of heterogeneity of solid solution and scalar dislocation density. It is shown that this heterogeneity results in the formation of high-density dislocations in γ′-phase.

INTRODUCTION

In superalloys containing (γ + γ′)-phases, a full orientation correlation is usually observed between these phases [1, 2]. One of the methods to obtain such superalloys is a method of direct crystallization. Such a superalloy is expected to possess a well-ordered [001] orientation [3]. However, the superalloy, which is alloyed with a great amount of various elements, including lanthanum as well as its annealing modifies its orientation [4–7]. The paper mainly focuses on this issue.

MATERAILS AND METHODS

The superalloy obtained *via* the directional crystallization technique and composed of 70.84–71.0 at % Ni, 17.0–17.08 at % Al, 5.73–5.74 at % Cr, 1.98 at % Mo, 1.7 at % Ti and 0.88 at % W was used in this experiment.

Proceedings of the International Conference on Advanced Materials with Hierarchical Structure for New Technologies and Reliable Structures 2017 (AMHS'17)
AIP Conf. Proc. 1909, 020149-1–020149-4; https://doi.org/10.1063/1.5013830
Published by AIP Publishing. 978-0-7354-1601-7/$30.00

Lanthanum was added. It was expected that each state of the superalloy would have a single-crystal structure with [001] orientation.

The structural investigations of this alloy were carried out after the directional crystallization (original state) at three La concentrations (0.015, 0.10 and 0.30 wt %) and after 1273 K annealing for 10 and 25 h.

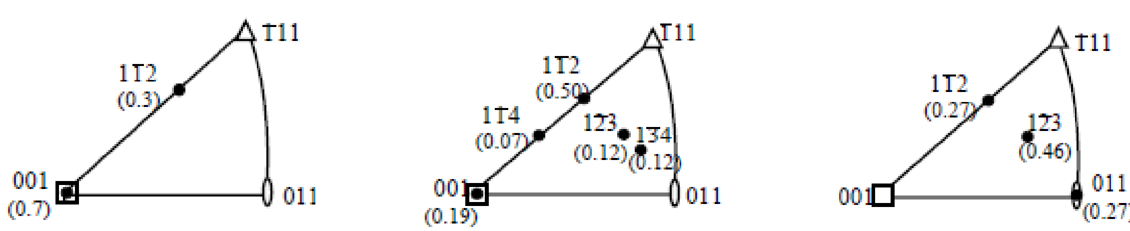
The scanning electron microscopy (SEM) observation and transmission electron microscopy (TEM) investigations of thin foils were used in the experiment. Observations of thin foils were carried out on an EM-125 transmission electron microscope with 125 kV accelerating voltage and 10000–50000 magnification capacity. X-ray phase (XRD) analysis was carried out by DRON-7 diffractometer, using copper radiation (K_α). Specifications for the DRON-7 included 30 kV voltage and 30 mA current. The XRD analysis included measurements of the alloy phase composition, parameters of crystal lattices, crystal constants mismatch between γ- and γ′-phases and heterogeneity of concentration, medium-size and volume fractions of structural elements, density and localization of dislocation defects. Isothermal sections of Ni-Al-Me ternary phase diagrams were widely used for the XRD analysis of phase composition [8].

The XRD analysis was based on the research results of SEM investigations, interpretation of electron diffraction patterns, observations in bright and dark fields. Structural investigations were performed on specimens that are normally cut to the crystal's growing axis. The obtained diffraction patterns allowed us to detect stereographic components for each state of the alloy. With respect to the specific weight of orientations, their distribution was plotted on a standard stereographic triangle, and orientations were then analyzed. Medium sizes of the structural elements were detected on micrographs using the secant method [9].

RESULTS AND DISCUSSION

Generally, Ni-Al-Cr-based superalloy consists of six phases. The major phase is γ′-phase. Its morphology represents quasi-cuboids with rather a clear cut. Quasi-cuboids in γ′-phase are surrounded with relatively thin layers of γ-phase, whose crystal system is a face-centered cubic (FCC) disordered solid solution [7, 10–13]. In addition to γ- and γ′-phases, there are lanthanides La_2Ni_3 and Al_2La and one more denoted as α_2-phase [12, 14]. Besides these phases, the superalloy consists of a small amount of La_2C_3 carbide particles deposited on dislocations in γ′-phase [12, 15]. The content of these phases depends on the lanthanum concentration and thermal treatment of the superalloy, however, they are present in each of its states. As it concerns γ-phase, it is observed in each state, excepting a case after the extreme thermal treatment, i.e. 25-hour annealing.

Let us consider the preferred orientation of γ′-phase more exactly the alloy orientation. The analysis of diffraction patterns obtained for different parts of the structure shows that the introduction of lanthanum in the superalloy composition results in a considerable orientation dispersion. The quantity of [001] orientation quickly reduces with the increase in the lanthanum concentration. The process of annealing has a strong effect on its orientation. Thus, in the original state (before annealing), the quantity of [001] orientation is 0.7 already at a small lanthanum concentration (0.015 wt %). The other part of 0.3 refers to [112] orientation as shown in Fig. 1a. The increase in the lanthanum concentration up to 0.1 wt % results in the orientation dispersion as presented in Fig. 1b. The quantity of [001] orientation reduces down to ~0.2. The bulk of orientations (~0.6) accounts for side [001]–[011] of the stereographic triangle and its centre (~0.25). The further increase in the lanthanum concentration leads to the elimination of [001] orientation in the superalloy (Fig. 1c). This orientation is replaced by those along the bisector of the stereographic triangle, namely along [112]–[011] side. At the same time, 50% of orientations falls in the centre of the stereographic triangle. It is quite obvious that the introduction of lanthanum in the superalloy terminates the formation of the cubic orientation.



(a) (b) (c)

FIGURE 1. Orientation at different lanthanum concentrations in Ni-Al-Cr superalloy: 0.015 wt % La (a); 0.1 wt % La (b); 0.3 wt % La (c). Volume fractions of orientations are given in brackets

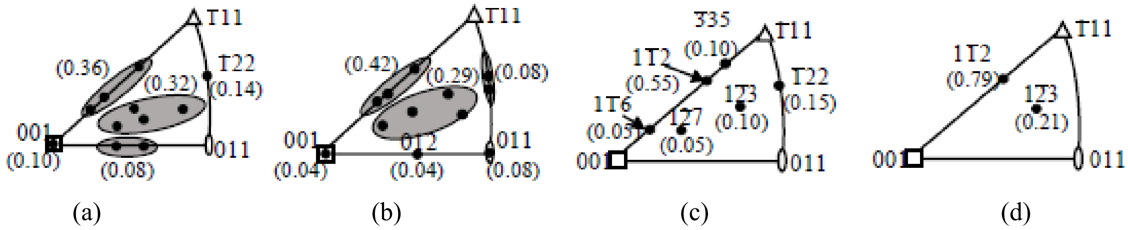


FIGURE 2. Orientation at different lanthanum concentrations in Ni-Al-Cr superalloy annealed during 10 (a, c) and 25 h (b, d); a, b—0.1 wt % La; c, d—0.3 wt % La. Volume fractions of orientations are given in brackets

The superalloy annealing at 1273 K during 10 and 25 h leads to a more pronounced orientation dispersion. Figure 2 contains the stereographic triangles with the indication of orientations in the superalloy and their volume fractions for annealed alloys containing lanthanum in the amount of 0.1 wt % (Figs. 2a, 2b) and 0.3 wt % (Figs. 2c, 2d). As can be seen from Fig. 2a, 10 h annealing of the alloy with 0.1 wt % La results in a pronounced orientation dispersion (compare to Fig. 1b). A large number of new orientations is observed along all the sides of the stereographic triangle. The quantitative analysis of the specific weight of observed orientations (Fig. 2a) shows that 10 h annealing results in almost uniform dispersion of orientation, both along the sides and in the center of the stereographic triangle. At the same time, the specific weight of [001] orientation decreases. In case of 25 h annealing, the orientation dispersion continues (Fig. 2b), but the amount of [001] orientation further decreases. In this case, the number of orientations in the center of the stereographic triangle is practically unchanged.

Annealing of the superalloy with 0.3 wt % La content also modifies its orientation. The comparison of Figs. 2c, 2d and Fig. 1c shows that 10 h annealing results in the orientation dispersion. In this case, firstly, the specific weight of orientations located in the centre of the stereographic triangle decreases, while those located on [001]–[111] side intensively grows. Secondly, the amount of [112] orientation is twice increased. Annealing enhanced up to 25 h leads to a further modification of the superalloy orientation as shown in Fig. 2d. This figure illustrates the stereographic triangle with just two orientations, namely in the centre and on [001]–[111] side. The specific weight of the central orientation [123] is 0.21, while that of orientation [112] is the highest (~0.8). Thus, it may be affirmed that annealing of the superalloy with 0.3 wt % La, results in a gradual transformation of [001] orientation to [112] orientation.

The value of scalar dislocations density is determined for γ′-phase depending on La concentration and some annealing parameters. TEM images of the dislocation structure in γ′-phase are presented in Fig. 3. It is found that the process of annealing has a low effect on scalar dislocation density of this phase, which is higher as before. Measurements show that the average value of scalar dislocation density is close to the value of its dispersion (Fig. 4a). According to the XRD analysis, the bulk of γ′-phase dislocations piles up against the barriers, which are usually Kear-Wilsdorf locks and typical for the FCC crystal system. Figure 4 contains the plots of scalar dislocation density ρ in γ′-phase. The effect of La concentration on ρ value is significant only in the original state of the superalloy (Fig. 4b, curve *1*). In the annealed superalloy, La concentration has no a serious effect on the scalar dislocation density ρ (Fig. 4b, curves *2* and *3*). Therefore, lanthanum complicates particularly the growth of perfect crystals during the superalloy crystallization. Figure 5 presents the comparison of values of the scalar dislocation density ρ and heterogeneity of solid solution described by Δ*d* for γ′-phase.

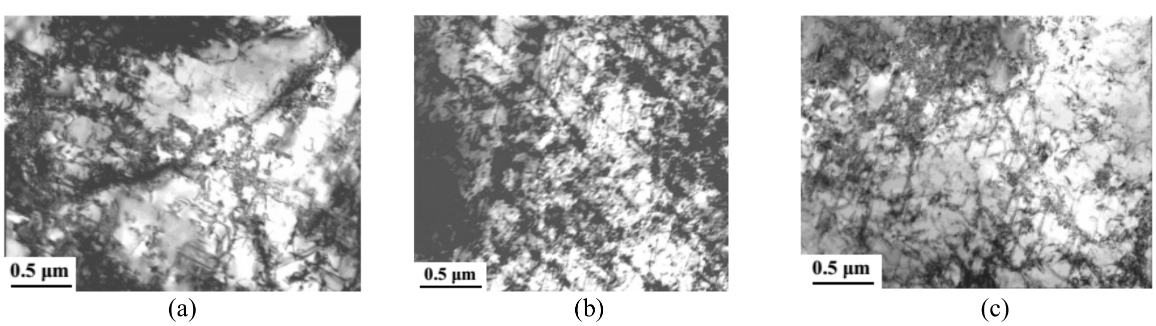
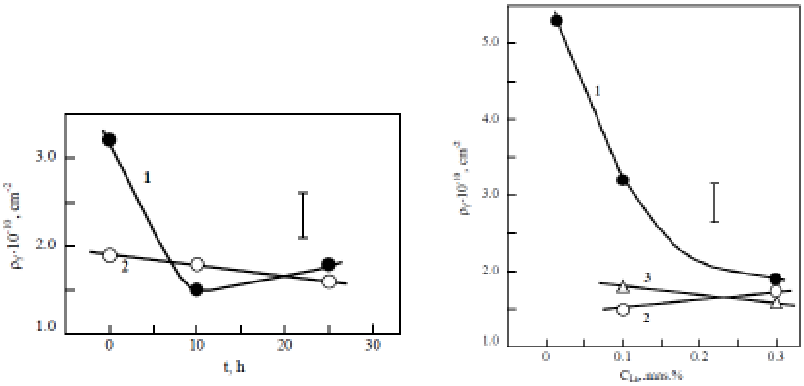


FIGURE 3. TEM images of dislocation structure of superalloy γ′-phase: a—0.1 wt % La; b, c—0.3 wt % La at 1027 K annealing for 10 (a, b) and 25 h (c)



(a) (b)

FIGURE 4. Scalar dislocation density in γ′-phase of superalloy annealed at 1273 K: a—annealing time (La concentration 0.1 (*1*) and 0.3 wt % (*2*)); b—C_{La} concentration (annealing time 0 (*1*), 10 (*2*), 25 h (*3*))

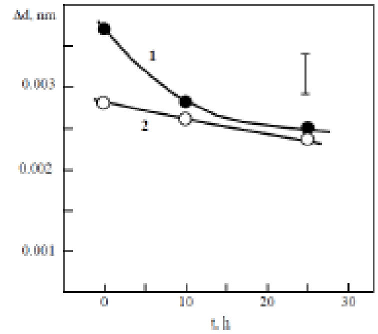


FIGURE 5. Dependence between line (111) half-width and annealing time *t* in superalloy with different La concentration 0.1 (*1*) and 0.3 wt % (*2*)

CONCLUSIONS

Summing up the results obtained with TEM and SEM investigations and the XRD analysis, it can be concluded that: (1) addition of La in the superalloy terminates the formation of the cubic orientation and the superalloy annealing has the similar effect; (2) a significant heterogeneity is observed in γ′-phase; (3) a correlation is observed between the degree of heterogeneity of a solid solution and the scalar dislocation density in γ′-phase; (4) heterogeneity of the solid solution results in the high dislocation density in γ′-phase.

ACKNOWLEDGMENTS

The research is supported with the funding from the Government Order No. 3.8320.2017/BCh.

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