Materials Science and Engineering A 521–522 (2009) 102–105

Contents lists available at ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

Single crystal PWA 1483 superalloy: Dislocation rearrangement and damping phenomena

P. Deodati [a], R. Montanari [a,*], O. Tassa [b], N. Ucciardello [a]

[a] Department of Mechanical Engineering, University of Rome "Tor Vergata", Via del Politecnico 1, 00133 Rome, Italy
[b] Centro Sviluppo Materiali (C.S.M.), Via di Castel Romano 100, 00128 Rome, Italy

ARTICLE INFO

Article history:
Received 30 May 2008
Accepted 17 September 2008

Keywords:
Ni-superalloy
Internal friction
Dynamic modulus
Dislocation structures

ABSTRACT

The structural stability of the single-crystal PWA 1483 superalloy has been investigated by internal friction (IF) and dynamic modulus measurements from room temperature to 1073 K. The examined samples were in the solubilized state.

The vibrating reed technique with electrostatic excitation and frequency modulation detection of flexural vibrations has been employed. Frequency was ~350 Hz.

IF spectra recorded in successive test runs on the same samples show a Q^{-1} maximum (M1) above 623 K, whose intensity and position change from one run to another; in correspondence with M1 the modulus undergoes a slow decrease followed by a sudden increase. Sometimes another maximum (M2) has been observed at lower temperature (~523 K).

After each run the values of the modulus and of Q^{-1} change indicating that a progressive irreversible transformation occurs. Damping phenomena have been attributed to the rearrangement of dislocation structures in the disordered matrix. This rearrangement modifies the density and the average distance of pinning points. This explanation is supported by transmission electron microscopy (TEM) observations.

1. Introduction

Ni-based superalloys with biphasic structure ($\gamma+\gamma'$) are usually employed to build up hot parts of gas and aeronautical turbines. For such applications the mechanical stability of the material is of utmost importance. Anelastic behaviour of these materials has been extensively investigated by different techniques [1–8].

Hermann et al. [2] have shown that the CMSX-4 alloy exhibits two relaxation peaks: activation enthalpy $\Delta H \cong 3.10$ eV and limit relaxation time $\tau_0 \sim 10^{-16}$ s (peak 1), $\Delta H \cong 2.7$ eV and $\tau_0 \sim 10^{-13}$ s (peak 2). From the comparison of ΔH values of the two peaks with the diffusion energies of Ni and Al obtained in a Ni_3Al single-crystal by the diffusion experiments of Frank et al. [9], peak 2 was attributed to the diffusion of Ni in the sub-lattice of Ni sites. Peak 1, with higher activation enthalpy, was ascribed to the diffusion of Al atoms which jump into the nearest neighboring sites of Ni forming anti-site defects.

A peak with ΔH and τ_0 close to those of peak 1 [2] has been observed by other investigators [1,3,4] who have given alternative interpretations. The peak was attributed by Gadaud et al. [1] to Ni diffusion whereas Mourisco et al. [3,4] considered it as a Zener peak.

In alloys with a periodic structure of the ordered phase γ' Gadaud and Rivière [6] found a peak at 10 Hz, which was attributed to thermoplastic deformation induced by chemical gradients.

This paper presents the results of internal friction (IF) and dynamic modulus measurements performed in repeated test runs on the same samples to assess the microstructural stability of the single-crystal of the PWA 1483 superalloy in the solubilized state. The experimental conditions were chosen to avoid the appearance of the aforesaid peaks 1 and 2 in the examined temperature range: the resonance frequency (~350 Hz) used in present experiments involves that peaks 1 and 2 appear at higher temperatures.

Transmission electron microscopy (TEM) observations have been carried out in the samples in the as-received condition and after test runs to follow the microstructural evolution of the material.

2. Material and experimental

The examined superalloy had the following chemical composition in wt%: C 0.05–0.09, Cr 11.60–12.70, Co 8.50–9.50, Mo 1.65–2.15, W 3.50–4.10, Ti 3.90–4.25, Al 3.40–3.80, Ta 4.80–5.20, Ni to balance. As shown in Fig. 1, the material was a single crystal of γ phase hosting the ordered γ' phase, whose particles have typical square shapes of an average size of about 1 μm. The γ' phase was substantially homogeneously distributed with channels

* Corresponding author. Tel.: +39 0672597182.
E-mail address: roberto.montanari@uniroma2.it (R. Montanari).

0921-5093/$ – see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.msea.2008.09.107

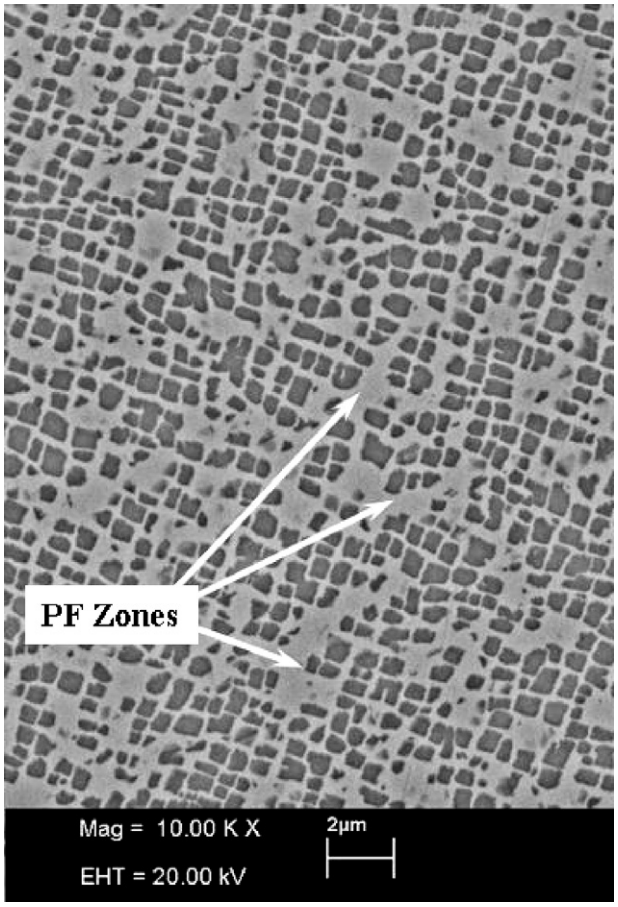

Fig. 1. Structure of the superalloy.

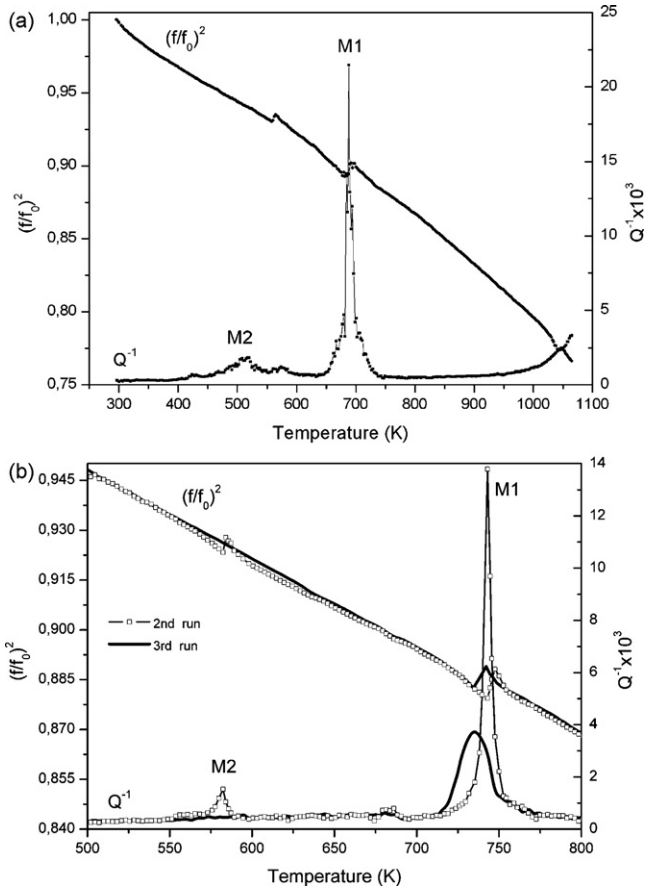

Fig. 2. (a and b) Q^{-1} and $(f/f_0)^2$ vs. temperature in the first test run (a). The trends in the successive runs with the same sample are displayed in (b); f_0 is the resonance frequency measured at room temperature before each test run.

of ~100 nm between the particles; some particle-free (PF) zones of the matrix with size of 1–2 μm were also observed.

IF and dynamic modulus measurements have been carried out with bar-shaped samples (50 mm × 6 mm × 0.7 mm) using the method of frequency modulation. The VRA 1604 apparatus employed in the experiments was previously described in detail [10]. Q^{-1} values have been determined from the logarithmic decay of flexural vibrations. The resonance frequency was ~350 Hz. The samples have been heated from room temperature to 1073 K at a rate of 1.7×10^{-2} K s^{-1}. The strain amplitude was kept lower than 1×10^{-5}. Repeated test runs have been performed with the same samples to assess the microstructural stability of the material.

3. Results

In successive test runs with the same samples a Q^{-1} maximum (M1) has been always observed at temperatures above 623 K. Its position does not depend on the resonance frequency and randomly changes from run to run while its intensity tends to decrease with increasing the number of thermal cycles. Sometimes M1 was accompanied by another maximum (M2) at lower temperature (~523 K) and of lower intensity. Both the maxima have an irregular shape. After some test runs the maxima disappear; these results are illustrated in Fig. 2(a and b).

In correspondence of M1, the modulus shows a slow decrease followed by a sharp increase. In the case of M2, which is quite broad in the first run (Fig. 2a), the modulus change seems shifted to a higher temperature with respect to the central position of the maximum. In the second run (Fig. 2b) M2 is narrower and the correspondence is good.

At the end of each test run the modulus value at room temperature is higher than the original one while that of Q^{-1} is lower. The variations of E and Q^{-1} occur mainly in the first runs, then negligible changes are observed (Fig. 3); after stabilization the Young's modulus has increased ~4% with respect to the original value.

To understand the nature of such behaviour, TEM observations have been carried out in the as-received material and after successive test runs.

Fig. 4(a and b) shows the material before IF tests. Micrographs display the different structures observed in the PF zones (a) and in the zones with homogeneous distribution of γ′ phase (b).

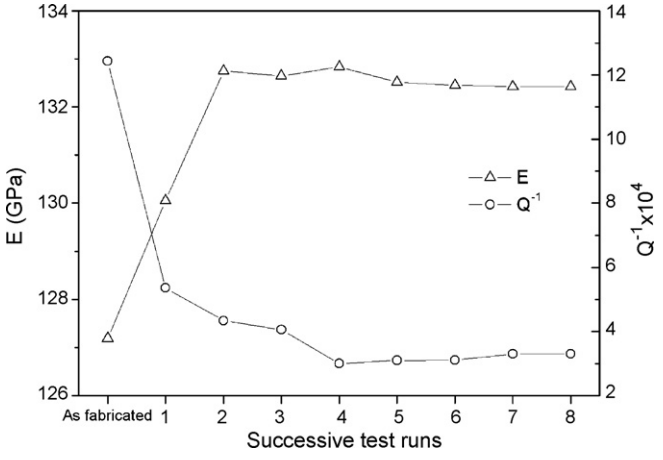

Fig. 3. Q^{-1} and modulus values at room temperature after successive test runs.

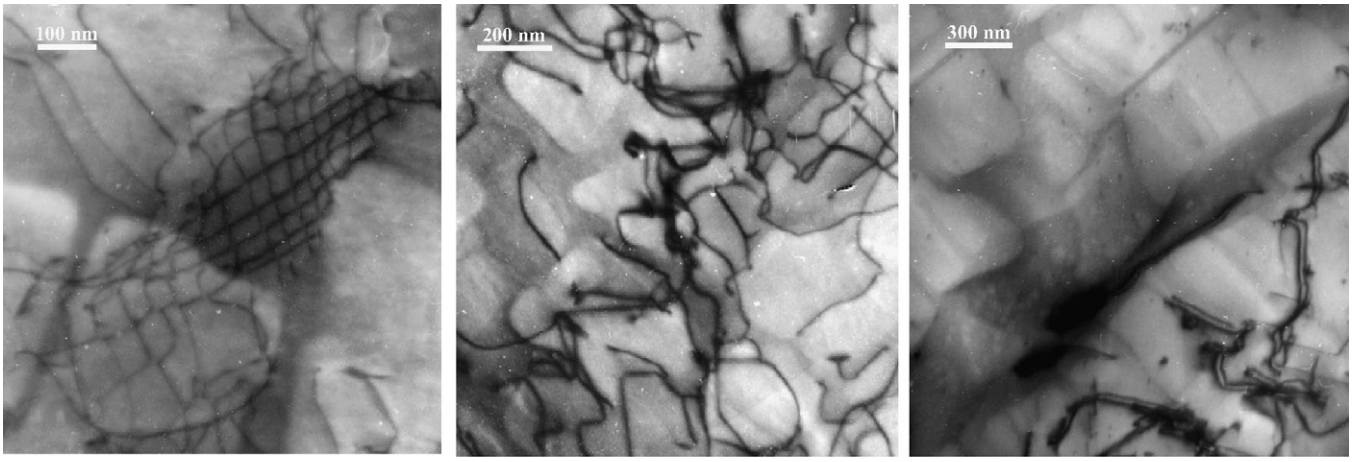

Fig. 4. Zone without (a) and with (b) particles of γ′ phase before IF test runs. Zone with homogeneous distribution of γ′ particles after eight test runs (c).

In PF zones the dislocations are partially arranged to form networks with cells dimensions of about 50 nm. These structures are not present in the zones containing the γ′ particles. Here dislocations are mainly concentrated in the channels between the particles.

After some test runs the dislocation networks disappear and only some cells of large size can be observed. As shown in Fig. 4c), in the zones with homogeneous γ′ distribution the dislocation density decreases. Several of the remaining dislocations form pairs (superdislocations) in the channels, mostly at the γ–γ′ phase boundaries.

4. Discussion

The main characteristics of the aforesaid anelastic phenomena can be summarized as follows:

1. the positions of maxima M1 and M2 (when present) do not depend on the resonance frequency and randomly change in each test run;
2. modulus changes in correspondence of Q^{-1} maxima;
3. after each run the original values of the resonance frequency f_0 and of Q^{-1} at room temperature are modified.

These results are compatible with an irreversible microstructural transformation taking place in successive steps giving rise to Q^{-1} maxima. When the transformation has been completed the structure is stable and the maxima are no more observed in the IF spectra. The occurrence of an irreversible transformation is in agreement also with the trend of Q^{-1} and f_0 measured at room temperature after successive test runs (Fig. 3). They progressively change and finally tend to constant values when transformation has been completed.

The explanation is based on the evolution of dislocation structures observed by TEM. For simplicity, it is assumed that the only contribution to dislocation damping comes from free dislocations. At low frequencies, i.e. in the kilohertz range, the Granato-Lücke string model for dislocation damping [11] predicts the following relationships:

$$Q^{-1} \propto \rho l^4 \omega \quad (1)$$

$$\frac{\Delta G}{G} \cong -\beta \rho l^2 \quad (2)$$

where β is a constant, ρ the dislocation density, l the average length between pinning points, $\omega/2\pi$ the vibration frequency. Although Eq. (2) refers to the shear modulus G, the same effect occurs also for the Young's modulus. According to Eqs. ((1) and (2)), a decrease of ρ and l leads to higher values of modulus and lower values of Q^{-1}.

Dislocation networks are no more observed after some cycles of heating and cooling. The growth of cells occurs by coalescence, namely two or more cells of smaller size form a cell of larger size. The process requires that the walls separating the cells, which merge together, disappear. When a wall breaks up the dislocations forming the wall become free and are able to give their contribution to the damping, thus ρ increases determining the initial modulus decrease and the ascending part of a IF maximum (see Eqs. (1) and (2)). When dislocations rearrange in the walls of the new cell of larger size they cannot contribute to damping so modulus increases and Q^{-1} decreases (descending part of maximum). Cell growth ends when the cells reach a size comparable to that of the corresponding PF zone. At that point the material has a stable microstructure which cannot be further modified by successive heat cycling in the temperature range examined here.

5. Conclusions

From experimental results it is possible to conclude that the microstructure of the PWA 1483 superalloy in as-fabricated condition is not stable. Following heating the cells observed in γ′ particle-free (PF) zones tend to grow forming cells of larger size. The irreversible process is not continuous but proceeds by steps occurring in successive test runs and each transformation step gives rise to an IF maximum. The microstructural stability is reached when the cell growth leads to a size comparable with that of the corresponding PF zone.

References

[1] P. Gadaud, K. Chakib, Mater. Sci. F 119–121 (1993) 397–400.
[2] W. Hermann, Th.V. Ort, H.G. Sockel, J. Phys. III 6 (1996) C8–223.
[3] A. Mourisco, N. Baluc, J. Bonneville, R. Schaller, J. Phys. III 6 (1996), C8-235-238.
[4] A. Mourisco, N. Baluc, J. Bonneville, R. Schaller, Mater. Sci. Eng. A 239–240 (1997) 281.
[5] L. di Masso, B. Coluzzi, F.M. Mazzolai, J. Phys. III 6 (1996), C8-247-250.
[6] P. Gadaud, A. Rivière, J. Phys. III 6 (1996), C8-867-870.

[7] E. Bonetti, E.G. Campari, A. Casagrande, L. Pasquini, E. Sampaolesi, J. Alloys Compd. 310 (2000) 351–355.
[8] M. Weller, M. Hirscher, E. Schweitzer, H. Kronmüller, J. Phys. III 6 (1996), C8-231-234.
[9] S. Frank, U. Södervall, Ch. Herzig, Phys. Stat. Sol. (b) 191 (1995) 45–55.
[10] S. Amadori, E.G. Campari, A.L. Fiorini, R. Montanari, L. Pasquini, L. Savini, E. Bonetti, Mater. Sci. Eng. A 442 (2006) 543–546.
[11] A.S. Nowick, B.S. Berry, Anelastic Relaxation in Crystalline Solids, Academic Press, New York and London, 1972, p. 415.