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► To cite this version:

P. Gadaud, A. Rivière. Thermoelastic Relaxation Due to Chemical Gradient in γ - γ' Ni₃Al Materials. Journal de Physique IV Proceedings, 1996, 06 (C8), pp.C8-867-C8-870. 10.1051/jp4:19968187 . jpa-00254624

HAL Id: jpa-00254624

<https://hal.science/jpa-00254624>

Submitted on 1 Jan 1996

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Thermoelastic Relaxation Due to Chemical Gradient in γ - γ' Ni₃Al Materials

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Résumé: Internal friction measurements performed in two types of γ - γ' Ni₃Al structures (an AM₁ superalloy with periodic distribution of 1 μ m and a non-periodic biphased material with grain size in the order of 100 μ m) bring out a high amplitude relaxation peak located at about 10 Hz, independently of the measurement temperature. The characteristics of the peak (amplitude, frequency position and temperature range observation) and experimental conditions (torsion tests) do not allow to interpretate this effect in terms of classic thermoelastic relaxation due to transverse or intercrystalline thermal currents. We propose a model of thermal relaxation under confined chemical gradient related to the elaboration of this kind of biphased materials; the chemical potential appears as a new internal variable for thermomechanical equilibrium conditions and implies local temperature gradients under stress annihilated by delayed thermal currents. The expression of the relaxation time is a function of the chemical gradient and of the thermal diffusivity.

1. INTRODUCTION

Nickel-base alloys elaborated from the Ni₃Al intermetallic compound have been developed for aeronautical industry owing to their lightness and high temperature strength. High temperature internal friction tests have been previously performed in our laboratory to analyze some aspects of the mechanical behaviour : the fragile to ductile transition of a β - γ superalloy [1], the influence of the stoichiometry on the γ' intermetallic phase in relation to the increase of yield strain [2] and dislocation climbing in γ' and AM₁ superalloy [3]. In this paper we present an original result of thermoelastic relaxation only observed in γ - γ' alloys (respectively disordered and ordered f.c.c. Ni₃Al phases). A new model is proposed to explain such result, based on the classic development of thermoelasticity [4] and taking into account the specific properties of this biphased material

2. EXPERIMENTAL CONDITIONS AND RESULTS

2.1. Specimen specification and experimental conditions

AM₁ superalloy developed for the elaboration of monocrystalline turbine blades present a very regular structure with γ' precipitates coherent in the γ matrix; chemically, this pseudo-monocrystal has a certain amount of additive elements (Co, Cr, Mo...). The periodicity of the structure (1 μ m) is obtained by growth of precipitates during the appropriate heat treatment (Figure 1). In our case, the sample has been annealed 5 hours at 1450 K.

The second structure is a polycrystal of γ and γ' grains with an average size in the order of 100 μ m ; in this case the two kinds of phases are purely constituted of Ni and Al. Figure 2 shows the contrast of chemical attack (2/3 HCl+1/3 HNO₃) between the grains of different nature

Internal friction tests were performed on 20x5x0.6 mm³ samples with a low frequency torsional pendulum [5] under high vacuum (10⁻⁵ Pa). This size of specimen is imposed by the high strength of the studied materials: they must be thin enough to be strained in the range 10⁻⁵ where the accuracy of measurement is optimum. All heat treatments have been realized in situ.

Figure 1: AM₁ superalloy structure

Figure 2: γ - γ' polycrystal structure

2.2 Experimental results

Apart from the thermally activated relaxations mentioned in the introduction, an additional effect is observed for the two structures described above.

In the case of the biphased polycrystal (Figure 3), this effect appears at 1100 K and is located at about 10 Hz; the amplitude decreases quickly after annealing at 1290 K (Figure 4). The width of this effect is not well determined because the maximum of damping is near the limit of our experimental frequency window. For the AM₁ superalloy, a similar relaxation peak is observed in the same frequency range at much lower temperatures but is very unstable during the first heating; after annealing at 1000 K it becomes more stable (Figure 5) and is not affected by further heat treatments as in the first case.

Figure 3: Damping spectra in γ - γ' polycrystal

Figure 4: Evolution of the amplitude during thermal cycling

Figure 5: Damping spectra in AM₁ superalloy

Figure 6: Ni and Al concentration profiles in AM₁

3. DISCUSSION

If we can assume that this relaxation without activation energy is comparable to a thermoelastic relaxation, its origin must be discussed. Classical thermoelastic mechanisms can not be involved: transverse thermal currents can not exist with torsion tests and intercrystalline thermal currents give rise to lower amplitude dampings. Furthermore, the very different average size of grains in the two kinds of structure would give very different resonance frequencies contrarily to our observations.

3.1 Chemical potential

To understand the existence of this effect observed in such different structures, we must find a same microscopic parameter providing thermoelasticity. As regards to the great difference of structure, the only common parameter is the coexistence of the defined intermetallic compound with its coherent disordered phase; at the atomic scale, continuity between the two phases is chemically impossible and so implies a chemical gradient in the disordered phase where the stoichiometry can vary. Under stress, the equation for the differential of the Gibbs free energy per unit volume becomes:

$$dg = -\epsilon d\sigma - (\mu/T) dT \quad (1)$$

where μ is the local chemical potential. To go further, we have to determine μ . If we consider a chemical gradient $c(x)$ along the x direction in the γ phase, the expression of the atomic flux is given by the second equation of Fick:

$$J_{at}(x) = -\sigma_i(x) a^2 (D/kT) c(x) - D [\delta c(x)/\delta x] \quad (2)$$

where D is the coefficient of diffusion, a the lattice parameter and σ_i an internal stress derived directly from the chemical potential, by the equation:

$$\mu(x) = - \int \sigma_i(x) a^2 dx \quad (3)$$

During the elaboration, to respect the stoichiometry of the γ' phase especially during the growth of precipitates, a flux of atoms (Al or Ni) is necessary between the two phases; the chemical gradient created by this flow remains stable after annealing because of the confinement in the case of the superalloy. Figure 6 represents the schematic periodic profile of Al and Ni concentrations, L being the average distance between precipitates ($\approx 0.4 \mu m$). The case of the other structure will be discussed later.

At thermodynamical equilibrium ($J_{at}(x)=0$), we obtain:

$$\sigma_i(x) c(x) a^2 = -kT \delta c(x)/\delta x \quad (4)$$

and by integration:

$$\mu(x) = kT \ln[\gamma c(x)] \quad (5)$$

where γ is a constant equal to the chemical activity. If we consider that Al is dilute, the Al potential represents the whole potential in the γ phase [6] which allows us to consider only Al atoms. If we take the more general solution of the equation of Fick, the symmetry of the Al concentration profile implies a solution of the form:

$$c(x) = A \cosh [B (L-2x)] \quad (6)$$

where A and B are constants. To determine them, we must consider the interface conditions $c(0) = c(L) \approx C'$ and the conservation of stoichiometry in the γ phase (where C' is the concentration of Al in the γ' phase and C the average concentration of Al in the γ one with the condition $C < C'$):

$$L C = \int_L c(x) dx \quad (7)$$

We obtain the two implicit relations:

$$C' = A \cosh (BL) \quad (8)$$

$$C B L = A \sinh (BL) \quad (9)$$

Considering that C and C' are not very different, BL is small and the expressions (8) and (9) can be simplified:

$$(B L)^2 = 3 [1 - (C/C')] \quad (10)$$

$$A [1 + 3C/2C'] = C' \quad (11)$$

3.2 Thermoelastic relaxation under chemical potential

Under applied stress σ , the local deformation can be written in the form:

$$\varepsilon = (\delta\varepsilon/\delta\sigma)_T \sigma + (\delta\varepsilon/\delta T)_\sigma \Delta T = J \sigma + (\delta\varepsilon/\delta T)_\sigma \Delta T \quad (12)$$

The second term of the equation represents the local inhomogeneity of deformation. The exact differential equation of Gibbs (1) gives:

$$T (\delta\varepsilon/\delta T)_\sigma = (\delta\mu/\delta\sigma)_T \quad (13)$$

It is the fundamental assumption of this model: the chemical gradient (by way of the internal stress derived from the chemical potential) does not allow an homogeneous strain at the atomic scale. It implies a local temperature gradient which can be relaxed by heat transport as in classic models [4]:

$$\delta(\Delta\mu(x,t))/\delta t = D_{th} \delta^2(\Delta\mu(x,t))/\delta x^2 \quad (14)$$

where D_{th} is the thermal diffusivity. If we take the expression of the instantaneous variation of potential which must be then relaxed:

$$\Delta\mu(x) = \delta\mu(x)/\delta\sigma)_T \sigma = \delta\mu(x)/\delta L)_\sigma \delta L/\delta\sigma)_T \sigma \quad (15)$$

$$= -\varepsilon kT B L \tanh B(L-2x) \quad (16)$$

and by double differentiation:

$$\delta^2(\Delta\mu(x))/\delta x^2 = -8B^2 \Delta\mu(x) [1 - \tanh^2 B(L-2x)] \quad (17)$$

In the hypothesis where the C/C' ratio is almost equal to unity, we obtain:

$$\delta^2(\Delta\mu(x))/\delta x^2 = -8B^2 \Delta\mu(x) = -24 (1 - C/C') \Delta\mu(x) / L^2 \quad (18)$$

If we consider a unique relaxation time τ for heat transport, the solution of equation (14) must have the form:

$$\Delta\mu(x,t) = \Delta\mu(x) \exp(-t/\tau) \quad (19)$$

We obtain the expression of the relaxation time:

$$\tau = L^2 / 24 D_{th} (1 - C/C') \quad (20)$$

Although the periodic parameter L is very different for the two structures, the fact that we find nearly the same resonance frequency means that the quantity $1 - C/C'$ is also very different, even if the C/C' ratio is almost the same; this is unfortunately impossible to verify experimentally. Other tests performed on superalloy samples without or with lower heat treatments did not reveal the relaxation. We can suppose at first, that the effect is shifted out our experimental frequency range, but more reasonably that the phase of precipitate growth is necessary to confine the chemical gradient. The last point is the presence of the relaxation in the polycrystal; we can suppose that the elaboration introduces an important chemical gradient and so the relaxation intensity is strong during the first heating; but this chemical gradient can be annihilated by atomic diffusion towards higher temperatures, as much as the notion of confinement is very smooth here ($L \approx 100 \mu\text{m}$).

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