



HAL
open science

ATOM-PROBE INVESTIGATION OF THE EARLY STAGES OF DECOMPOSITION IN A NICKEL-BASE SUPERALLOY

Sylvain Chambreland, D. Blavette, Monique Bouet

► **To cite this version:**

Sylvain Chambreland, D. Blavette, Monique Bouet. ATOM-PROBE INVESTIGATION OF THE EARLY STAGES OF DECOMPOSITION IN A NICKEL-BASE SUPERALLOY. *Journal de Physique Colloques*, 1987, 48 (C6), pp.C6-361-C6-366. 10.1051/jphyscol:1987659 . jpa-00226867

HAL Id: jpa-00226867

<https://hal.science/jpa-00226867>

Submitted on 4 Feb 2008

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

ATOM-PROBE INVESTIGATION OF THE EARLY STAGES OF DECOMPOSITION
IN A NICKEL-BASE SUPERALLOY

S. Chambréland, D. Blavette and M. Bouet

UA CNRS 808, Laboratoire de Microscopie Ionique, Faculté des Sciences
de Rouen, B.P. 67, 76130 Mont-Saint-Aignan, France

Résumé : Une sonde atomique à temps de vol a été utilisée pour étudier les premiers stades de précipitation de la phase γ' dans un superalliage à base nickel. Les paramètres de microstructure, déduits des profils de concentration, ont été étudiés en fonction du temps de vieillissement à 650°C. Pour des temps de vieillissement inférieurs à quelques heures, la composition des précipités varie dans une large mesure alors que la taille des particules reste sensiblement constante. En revanche, dans le stade de coalescence les précipités γ' atteignent leur composition d'équilibre après 25 heures.

Abstract : A conventional time of flight atom-probe has been used for the investigation of the early stages of precipitation of γ' phase in a nickel base superalloy. The microstructure parameters, as derived from concentration profiles, have been studied as a function of the aging time at 650°C. For aging times smaller than a few hours, the precipitate composition varies in a large extent while the particle size remains nearly constant. In contrast, in the coarsening stage γ' precipitates reach their equilibrium composition after the first 25 hours.

I - INTRODUCTION

Due to their great interest for aerospace applications a number of nickel base superalloys were developed in the past. For instance, high temperature single crystal blades were recently designed for aircraft engines [1]. These materials derive their excellent creep properties from the presence of γ' ordered precipitates finely dispersed in a FCC solid solution. The composition of particles is based on Ni_3Al while the matrix is a chromium enriched phase.

Various nickel base alloys were studied in the past. For instance, Wendt et al [2] recently investigated the decomposition process in a binary Ni Al alloy. They showed, in contrast to Hill and Ralph results[3], that this material obeys classical laws. This paper is aimed at reporting some atom-probe investigations we made on the early stages of formation of γ' precipitates in a simplified superalloy [4]. The microstructure parameters as well as the phase composition were followed as a function of the aging time. The isothermal heat treatment was carried out at $T = 650^\circ C$.

II - EXPERIMENTAL

The simplified superalloys we studied were supplied by the Office National d'Etudes et de Recherches Aérospatiales (ONERA). The materials were fabricated by powder metallurgy then, solution treated 1 hour at 1100°C. This treatment was immediately followed by a water

quench in order to avoid cluster formation already during cooling. The kinetics of precipitation was followed for aging times varying from 15mn to 500h at $T = 650^{\circ}\text{C}$. Every experiments were conducted using a conventional atom-probe operating at $T \approx 40\text{K}$ in a vacuum of a few 10^{-10} Torr.

Nickel base alloys are known to be very sensitive to preferential evaporation effects [4],[5]. A low specimen temperature (40K) as well as a high pulse fraction (20 %) were used in order to prevent such effects from interfering. In addition, the averaged number of pulses per collected ion was kept greater than 20 so that to minimize pile-up artifacts.

III - RESULTS AND DISCUSSION

A typical mass spectrum of the as-quenched material is given figure 1. The overall composition of the superalloy as measured by the atom-probe is compared in table 1 to the nominal composition given by the ONERA. The very good agreement between both data sets shows that the experimental conditions are optimum.

at %	Al	Ti	Cr	Ni + Co _w
Atom-probe data (46000 ions)	4.4 ± 0.2	1.3 ± 0.11	11.1 ± 0.3	83.2 ± 0.35
Nominal composition	4.26	1.41	10.86	83.46

Table 1

Statistical tests as well as autocorrelation analyses were performed in order to ensure that no precipitation occurs in the as-quenched state. No Al rich nucleus was actually detected.

The concentration profiles shown in figure 2 illustrate the fluctuations which are observed for an aging time of 1h at 650°C . The γ' precipitates appear as (Al, Ti) enriched and Cr depleted zones. Because of the large concentration fluctuations, the interpretation of profiles is somewhat difficult. It is sometimes delicate to decide whether a concentration peak is a particle or not.

In spite of these problems, it has been possible to derive the microstructure parameters of the material as well as the phase composition from the concentration signal features. Table 2 gives the atom-probe data obtained. For the alloy aged for 1h the particle size, the volume fraction and the number density have been calculated by using the method we described last year [6]. The averaged lateral resolution was close to 15 Å.

It is noteworthy to notice the particle diameter ϕ as deduced from the apparent size $\bar{\ell}$ of enriched regions [7] was found in good agreement with the value of the first minimum k_{ϕ} in the autocorrelrogram. In addition the volume fraction F_v as calculated from the linear fraction F_{ℓ} [7] is also compatible with the measured phase composition (table 3). The overall composition C_n as deduced from the lever rule ($F_v = 7.5 \%$) is close to that given in table 1.

apparent size \bar{l} of enriched regions	Linear fraction F_l	Mean distance between particles \bar{d}_p
36 Å	13.7 %	260 Å
Particle diameter ϕ	Volume fraction F_v	Number density N_v
50 ± 16 Å	$7.5\% \pm 0.4\%$	$1.2 \cdot 10^{18}$ part/cm ³

Table 2

at %	Matrix composition C_m	Precipitate Composition C_p	Overall Composition $C_n = F_v C_p + (1 - F_v) C_m$
Al	3.78 ± 0.15	13.55 ± 1.62	4.51 ± 0.5
Ti	1.12 ± 0.08	2.53 ± 0.74	1.22 ± 0.2
Cr	12.02 ± 0.25	6.28 ± 1.15	11.6 ± 0.5
Ni+Co	83.08 ± 0.29	77.64 ± 1.97	82.7 ± 2.1
Number of collected ions	67500	1800	69300

Table 3

The evolution of the particle size ϕ and the number density N_v as a function of the aging time is exhibited in figures 3 and 4. However, the results suggest that the precipitation process is composed of two distinct regimes. For aging time smaller than one hour the particle size is nearly constant while the number density N_v is observed to increase with time. In contrast, for larger aging times, the zone radius increases approximately as $t^{1/3}$ while N_v decreases proportionally to t^{-1} . These two time ranges are thought to be associated with the nucleation-growth of nuclei and the subsequent coarsening of particles. The observed growth law as well as the time dependence of the density are, besides, in a reasonable agreement with the well known theory of diffusion controlled coarsening developed by Lifshitz, Slyozov and Wagner [8], [9]. Such laws have been observed in number of cases especially for nickel base alloys [2]. These materials are actually good candidates because important hypotheses of the theory are satisfied (small interfacial energy, spherical precipitates).

The Al+Ti and Cr concentrations in particles are given in figure 5 as a function of the aging time. The results show that the precipitate composition changes in a large extent in the early stages of transformation, that is for the nucleation stage. However, taking into account the statistical fluctuations, one can see that precipitates reach their steady-state concentration after the first 25 hours. In addition, experimental data may be interpreted as the substitution of chromium on aluminium sites of the ordered $Ni_3(Al+Ti)$ phase.

The figure 6 shows the evolution of the matrix concentration during the decomposition process. As expected, the Al supersaturation decreases slowly while the chromium level increases. The volume fraction as deduced from phase composition varies from 8 % to 14 % in the coarsening stage.

As a conclusion, the atom-probe investigations we performed show that γ' particles form via nuclei the Al concentration of which increases with aging time while the chromium level decreases. In spite of this, no periodically dispersed enriched zones were observed. In addition, the results may be interpreted in the scope of the classical LSW theory.

ACKNOWLEDGMENTS

The authors wish to thank Drs Walder and Marty from the ONERA who supplied the samples for fruitful discussions. This work have been financially supported by the Direction des Recherches Etudes et Techniques [DRET contrat N°84/1230 and 85/1424] we would like to acknowledge.

REFERENCES

- [1] D. BLAVETTE and A. BOSTEL Acta Met 32 (1984) 811
- [2] H. WENDT and P. HAASEN, Acta Met. 31 (1983) 1649
- [3] S.A. HILL and B. RALPH, Proc. of Conf. Phase Transf., York, Institution of Met. Conf., vol. 2, ser. 3, 11 (1979) 44
- [4] D. BLAVETTE, A. BOSTEL, J. M. SARRAU Met. Trans 16 A (1985) 1703
- [5] K. M. DELARGY and G. D. W. SMITH, Met. Trans. 14 A (1983) 1771
- [6] D. BLAVETTE and S. CHAMBRELAND J. de Phys. C7-11 t47 (1986) 281
- [7] D. BLAVETTE, A. MENAND and A. BOSTEL, This symposium
- [8] I. M. LIFSHITZ, V. V. SLYOZOV, J. Phys.Chem.Solids 19 (1961) 35
- [9] C. WAGNER, Z. Elektrochemie 65 (1961) 581

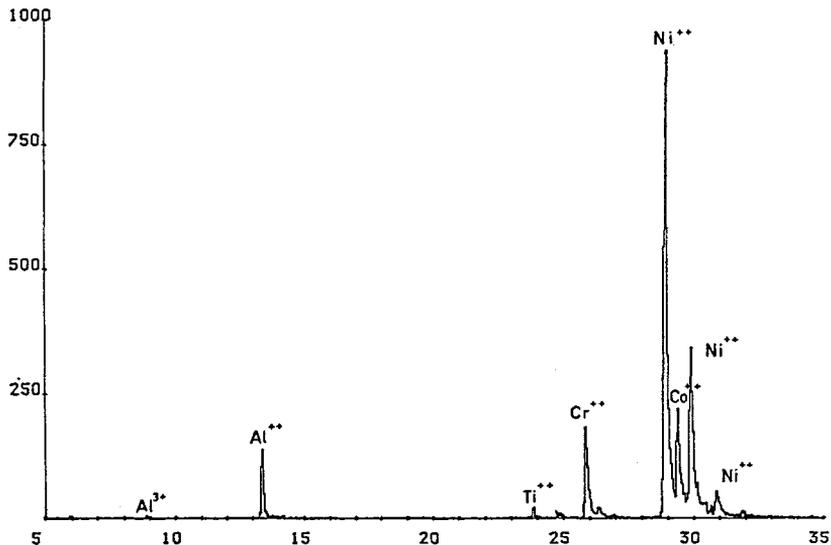


Figure 1 : Mass spectrum of the as-quenched Nickel Base Superalloy

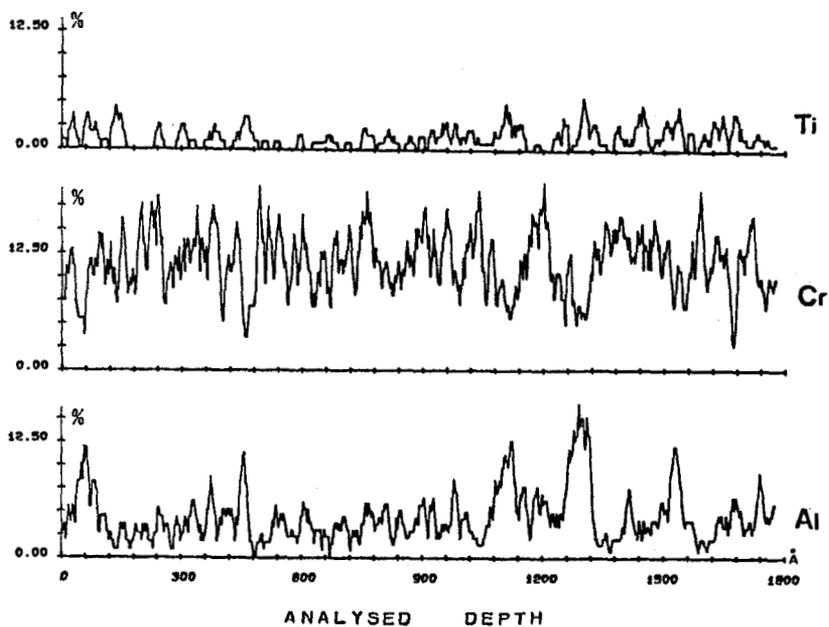


Figure 2 : Concentration profiles obtained for the material aged for 1h at 650°C. γ' particles appear as (Al, Ti) enriched regions.

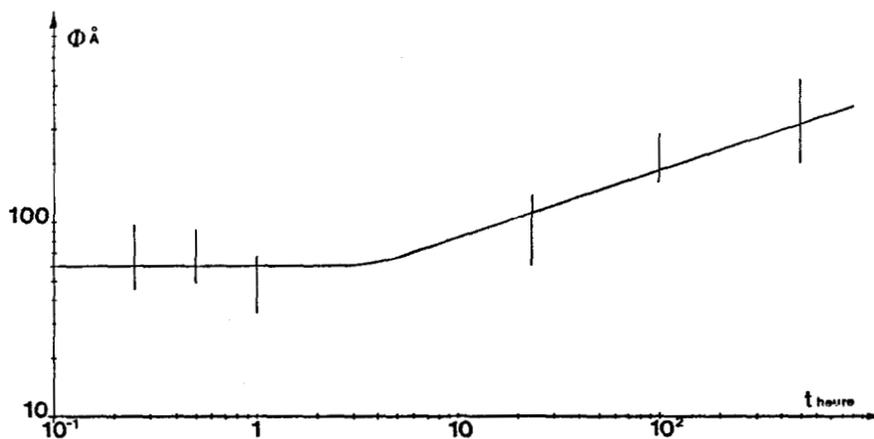


Figure 3 : The particle size as a function of the aging time at 650°C

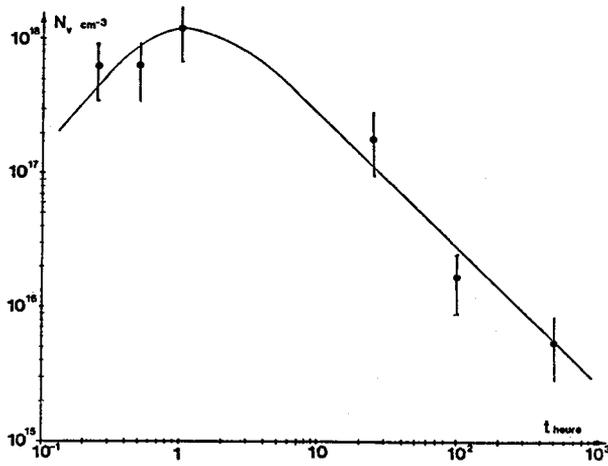


Figure 4 : The number density versus the aging time at 650°C

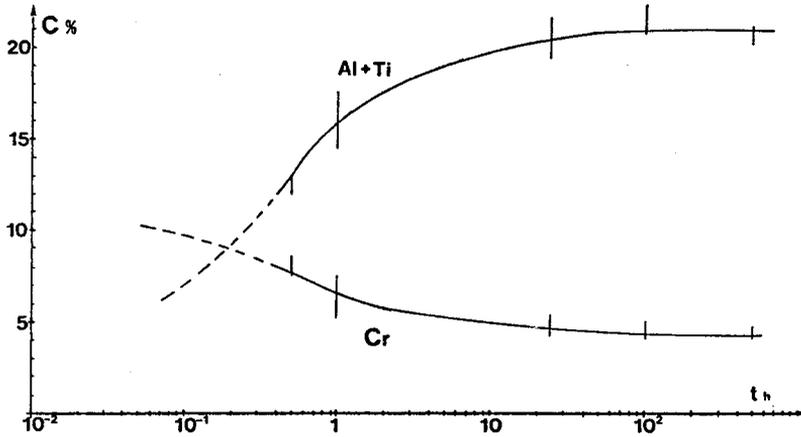


Figure 5 : Measured concentrations in γ' particles as a function of the aging time at 650°C

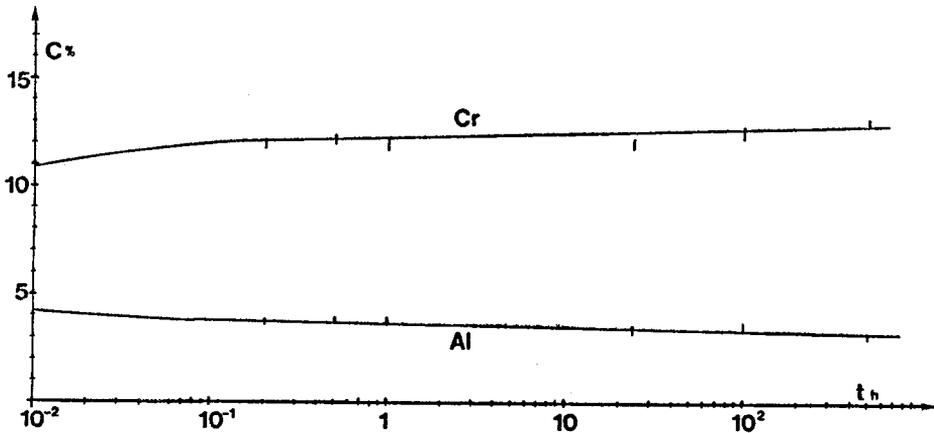


Figure 6 : Matrix composition versus the aging time at 650°C