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## Chemical composition around $\gamma/\gamma'$ interface in single crystal nickel-based superalloys in the early stage of creep

A. RACINE and A. HAZOTTE

*Laboratoire de Science et Génie des Matériaux Métalliques, URA 159 du CNRS, Ecole des Mines de Nancy, Parc de Saurupt, 54042 Nancy cedex, France*

### ABSTRACT

The aim of this work was to examine if a chemical anisotropy around  $\gamma'$  precipitates could be induced by the applied stress at the beginning of a compressive creep test. This question was solved in terms of concentration profiles around  $\gamma'$  in order to see which alloying elements diffuse and if they do, in which direction. The results were discussed in the light of finite element simulations where the local stress states were determined in terms of equivalent Von Mises stress and hydrostatic pressure.

### INTRODUCTION

The morphology of  $\gamma'$  precipitates in Nickel-based superalloys is known to strongly evolve during the very early stage of high temperature creep tests. Nevertheless, the phenomena which govern the shape changes - in particular the role of the applied stress and strain on the diffusion mechanisms - are still not very well understood. As an attempt to highlight these questions, we submitted single crystals of AM1 and MC2 Nickel-based superalloys to a compression creep test and we characterized resulting chemical composition around the  $\gamma'$  precipitates.

### EXPERIMENTAL

#### a) Materials and heat treatment.

Two different single crystal superalloys presenting a high temperature equilibrium volume fraction of precipitated  $\gamma'$  phase close to 65% have been used in this study. These superalloys differ in their composition and were heat treated in order to obtain a standard microstructure before the creep test as shown in Table I :

Alloy	Composition (weight %)								Misfit (%) at 1050 °C	Initial heat treatment
	Ni	Cr	Co	Mo	W	Al	Ti	Ta		
AM1	bal	7.5	6.5	2	5.5	5.2	1.2	8.6	-0,3 [1]	1300°C/50h+1050°C/15h
MC2	bal	8	5	2	8	5	2	6	-0,4 [2]	1300°C/50h+1080°C/6h

#### b) Experimental conditions.

The creep test submitted the alloys to a compressive strength of 100 MPa along the [001] direction of solidification, during 5 hours at 1050 °C .

Analyses were mainly carried out using a 120 kV TEM Philips CM12 coupled with a SiLi detector (ECON IV 30 mm<sup>2</sup>). The experimental conditions are reported in Table II :

	Standard AM1	Crept AM1	MC2
Microscope	CM20		CM12
Tilt angle (°)	20		19
Accelerated voltage (kV)	160		120
Spot size (nm)	7		20
Mode	Nanoprobe		Microprobe
Resolution (eV)			156,2
Energy window (keV)	0 to 20		0 to 20
Width of channel (eV)	10		10
Acquisition time (s)	150		150 to 200

The spectra were evaluated using Econ's SUPQ program which allows the subtraction of the background modelled by the Kramer's law, expressed by  $T*(V-E)/E$  where V is the accelerated voltage, E is the energy and T is the spectrometer efficiency.

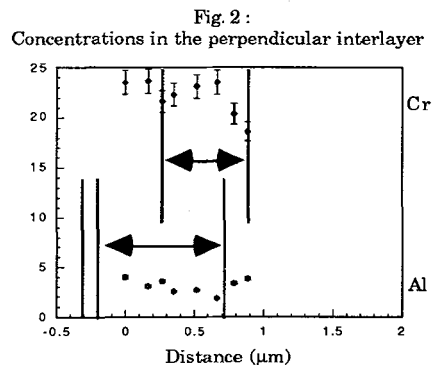
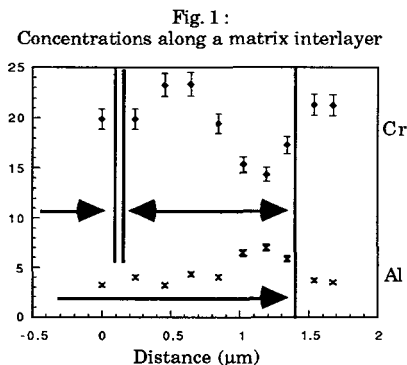
Because the detector is made of Silicon, escape peaks - resulting from the absorption of the radiations of elements by Silicon - are located 1.739 keV below the main peaks. Thus we performed escape peak correction which subtracts counts from the removed escape peaks and adds them into the main peaks where they belong.

In the non loaded samples, we analyzed two  $\gamma$  interlayers perpendicular to each other ; in the crept samples, we analyzed interlayers parallel and perpendicular to the applied stress direction which was previously marked on the TEM foils.

## EXPERIMENTAL RESULTS

### a) Non loaded samples.

As we can see on Figures 1 and 2 for AM1, concentration variations are to be observed. More precisely, it seems that in a given  $\gamma$  interlayer the concentration of Cr is minimum near the middle of the precipitates, whereas the one of Al is maximum there. Moreover variations of the concentration profiles occur for all the elements between two interlayers perpendicular to each other.



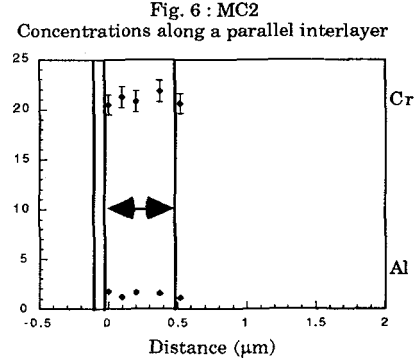
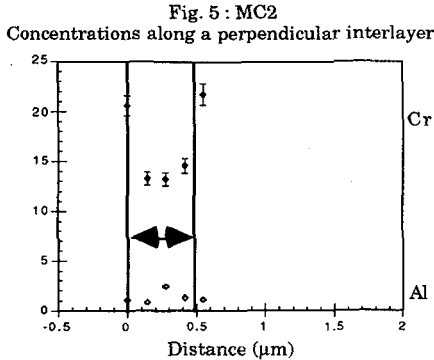
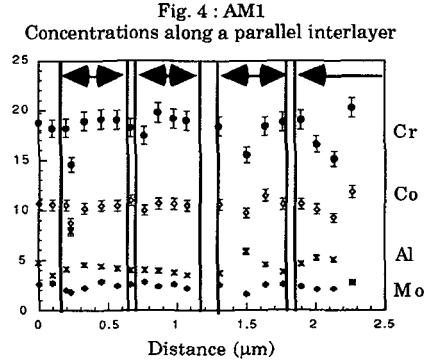
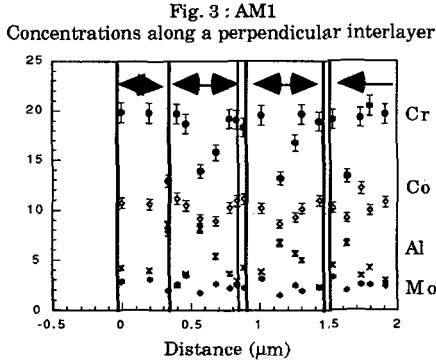
The arrows localize the  $\gamma'$  precipitates ; two arrows "at the same place" and shorter vertical lines mean that the precipitates are not well facing each other on both sides of the  $\gamma$  interlayer.

### b) Crept samples.

The concentration profiles in  $\gamma$  interlayers can be seen on the Figures 3 to 6 for both alloys. We can observe that the qualitative evolution is the same as previously, essentially for Al, Cr, Mo and Ti.

However some differences occur between the aged and the crept states. On one hand, statistical analyses allow us to determine mean concentration variations which seem to be

different in the interlayers parallel and perpendicular to the applied stress axis essentially for Cr, Co, Ta and W, the  $\gamma$ -forming elements being more present in the parallel interlayers. On the other hand, some matrix dislocations take place at the corner of the precipitates and a few of them take place along the {100} faces.



## FINITE ELEMENT SIMULATION - DISCUSSION - CONCLUSION

Finite Element calculations have been performed, using the F.E. code SYSWELD (Framasoft-C.S.I.), in order to estimate the internal stress states corresponding to the different thermomechanical treatments used in the present study. Details about these calculations can be found in ref. [3]. The 3-D elementary cell used for the calculations was an unit cube representing an eighth of a  $\gamma'$  precipitate with the embedding  $\gamma$  matrix. Symmetry was introduced by constraining three faces of the cube to deform along their plane while periodicity was obtained by constraining the three others to remain plane. The mechanical behaviour of both phases was assumed to be elastic and anisotropic. Elastic properties of  $\gamma$  and  $\gamma'$  phases were taken constant and deduced from a literature review. Lattice mismatch was introduced as an initial deformation :  $\delta$  value was taken equal to -0.003 or -0.004, which is coherent with the values measured at 1050°C for AM1 and MC2, respectively. Calculations were performed for both non loaded and loaded cases. Figure 7 illustrates the type of results which can be obtained in terms of equivalent Von Mises stress distribution. Table 3 gives the values of equivalent Von Mises stress and hydrostatic pressure calculated at different points of the geometry shown in Figure 7.

		Point 1	Point 2	Point 3
Equivalent Von Mises stress (MPa)	Non loaded/Crept AM1	306/208	306/360	158/145
	Non loaded/Crept MC2	407/311	407/459	211/182
Hydrostatic pressure (MPa)	Non loaded/Crept AM1	-149/-187	-149/-178	-120/-153
	Non loaded/Crept MC2	-198/-236	-198/-228	-159/-193

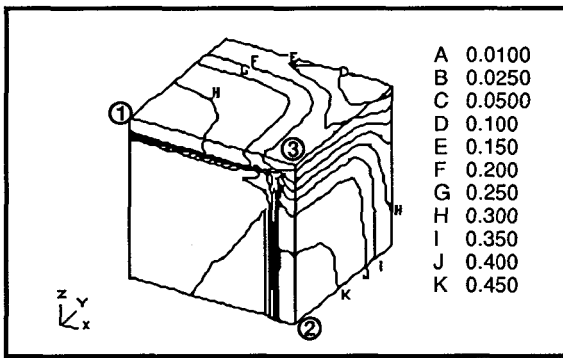


Fig 7 : Isovalues of equivalent Von Mises stress reported here for the crept MC2. The values are expressed in GPa.

In the non loaded samples, the calculated local internal stress state is the same for the two kinds of  $\gamma$  interlayers. Consequently the variations of the concentration profiles in the two interlayers which we have pointed out can not be explained by the results of the simulation. Nevertheless it is to be noted that the precipitates are not so cubic and so regularly arranged as the cubes are supposed to in the simulation. Therefore the diffusion fluxes are not perfectly symmetric and lead to non symmetric concentration variations. However the simulation pointed out strong stress variations in a given matrix interlayer. This can explain why the concentration varies along a given  $\gamma$  interlayer.

In the crept samples, the equivalent Von Mises stresses present a large difference between the two kinds of  $\gamma$  interlayers for the two alloys. However we can note that the hydrostatic pressures (compressive) are nearly of the same order in the middle of the two interlayers in our test conditions. Consequently, an explanation of the local chemical segregation only based on the diffusion of the bigger alloying elements ( $\gamma'$ -forming) from the more compressed areas to the less compressed one due to the discrepancy of hydrostatic pressure in the  $\gamma$  interlayers [1] seems not to be completely satisfying. Here again the precipitates are not so cubic and so regularly arranged as the cubes are supposed to be in the simulations, which lead to non symmetric concentrations variations.

In both cases, we can note that the  $\gamma$ -forming elements segregate always where the  $\gamma'$ -forming elements do not and vice-versa.

Works are still in progress in order to confirm these first results. Moreover, tension creep tests will be done at different temperatures and stresses in order to relate more precisely the concentration profiles in the two kinds of  $\gamma$  interlayers to the local stress state, by examining the different couples equivalent Von Mises stress/hydrostatic pressure corresponding to the different couples temperature/stress.

## AKNOWLEDGMENTS

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