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Irradiation-induced defects in solid solution and intermetallic compounds of the Ni-Al system: the effects of a third element (Ti or Ta)

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Abstract:

The effects of low-temperature electron irradiation experiments and of subsequent annealing treatments were investigated by means of residual resistivity measurements in model alloys based on the Ni-Al system. The materials, representing the two constituent phases of technological Ni-based superalloys, were a Ni(Al,Ti) solid solution and three intermetallics Ni₃Al, Ni₃(Al,Ti) and Ni₃(Al,Ta). The presence of the additional elements Ti or Ta had an important influence both in the solid solution and in the intermetallic compounds. These elements were found to interact strongly with the irradiation-induced defects, reducing significantly their mobility via a trapping effect. Such a result could explain, at least in part, the self-diffusion retardation found in the same materials by means of ordering kinetics studies.

I-INTRODUCTION

Ni-based superalloys, which are formed by an L1₂ long-range ordered (LRO) Ni₃Al-based intermetallic phase (the strengthening constituent), dispersed in an FCC short range ordered (SRO) Ni(Al)-based solid solution, may be used in a radiation environment. Two processes are investigated in the present work by means of residual resistivity measurements: the creation of point defects (Frenkel-pairs) during electron irradiation and the annihilation of these defects during isochronal anneals. The purpose of the present work is to determine the specific effects of the additional elements Ti or Ta on the properties and reactions of the defects. The present work, which gives information about the migration characteristics of point defects (including vacancies) complements the investigation of ordering kinetics in the same materials, which relies on equilibrium self-diffusion and which involves both vacancy migration and vacancy formation (T. Tarfa et al., these Proceedings)

II- EXPERIMENTAL PROCEDURE

Four compositions of single phase alloys were investigated: a ternary solid solution Ni-4at%Al-2at%Ti and three intermetallic compounds Ni-25at%Al, Ni-21at%Al-4at%X (X=Ti, Ta). The material preparation and the preliminary thermal treatments were the same as for the investigation of order changes (T. Tarfa et al., these Proceedings).

For the solid solution, a sheet 50 µm in thickness was obtained by cold-rolling then was spark-shaped. Because of their intergranular brittleness, the intermetallic samples were cut with a diamond saw, then mechanically polished down to a thickness of about 80 µm. Four samples, one of each composition, were simultaneously irradiated with 2.5 MeV electrons in the liquid hydrogen cryostat (21K) of the Van de Graff accelerator of LESI (Ecole polytechnique, Palaiseau, France). The damage process and the

recovery of the irradiated materials during thermal treatments were investigated by means of low-temperature electrical resistivity measurements.

III- RESULTS AND DISCUSSION

Frenkel-pair creation

Under electron irradiation at low temperature, point defects (Frenkel pairs) are created. Their concentration increases linearly at the beginning of the irradiation, but tends to saturate at higher fluences because of the spontaneous defect recombination. Defect production is characterised, in figure 1, by the irradiation-induced resistivity changes for both types of materials. A qualitatively similar behaviour is observed for all investigated alloys. However, the evolution amplitudes for the solid solutions are quantitatively comparable to that of pure nickel, whereas they are much larger in the intermetallics. Assuming a negligible effect of disordering on the resistivity variations, the Frenkel-pair resistivities ρ_F and the spontaneous recombination volumes V_0 were obtained (Table 1) by analysing the evolution of the resistivity damage rate as a function of the resistivity increment.

Materials	Ni	solid solutions Ni ₉₄ Al ₆	Ni ₉₄ Al ₄ Ti ₂	Intermetallics Ni ₇₅ Al ₂₅	Ni ₇₅ Al ₂₁ Ti ₄	Ni ₇₅ Al ₂₁ Ta ₄
ρ_F ($\mu\Omega \cdot \text{cm}/\%$)	6.7*	6.2 ± 0.6	6.7 ± 0.5	109 ± 11	115 ± 14	103 ± 13
V_0 (at.vol)	$220 \pm 40^{**}$	120 ± 70	172 ± 19	325 ± 43	161 ± 23	111 ± 19
Φ ($10^{18} \text{ e}/\text{cm}^2$)	8.0**	8.0	9.3	9.3	9.3	9.3
E_e (MeV)	2.95**	2.95	2.5	2.5	2.5	2.5
References	* [3], ** [1,2]	[1,2]	This work	This work	This work	This work

Table 1. Frenkel pair resistivities ρ_F and recombination volumes V_0 . Φ is the total fluence and E_e the electron energy.

Within each class of materials, no significant effect of composition on the Frenkel-pair resistivities is apparent. The recombination volumes, however, tend to decrease in the solid solutions as compared to pure nickel, and in the substituted intermetallics as compared to Ni₃Al. This effect might be related to a defect trapping by the alloying elements (Al and/or Ti) in nickel, and by the third element (Ti or Ta) in the intermetallics.

Figure 1a. Resistivity variation during 2.5 MeV-electron irradiation of Ni₉₄Al₄Ti₂ solid solution. Ni₉₄Al₆ and Ni were 2.95 MeV-irradiated [1].

Figure 1b. Resistivity variation during 2.5 MeV-electron irradiation of the intermetallics.

Recovery of radiation damage

During isochronal anneals, the irradiation-induced point defects migrate by thermally activated jumps and disappear at sinks, annihilate with antagonist defects or form clusters. Residual resistivity was used in order to examine these processes. Based on the results obtained in pure nickel [4,5], a recovery model of the binary solid solution has been proposed [1,2]. Its main features are (figure2):

Stage I (5 --> 110K). This stage corresponds to the recombination of close Frenkel pairs and to the migration of free interstitials. The resistivity decreases and about 50% of the initial resistivity excess is recovered.

Stage II (110 --> 330K). A slight increase of resistivity is observed, resulting from a competition between the annihilation of interstitial defects which decreases the resistivity and an ordering process induced by their migration which increases the resistivity.

Stage III (330 --> 470K). The large resistivity increase indicates an important ordering promoted by the long-range migration of vacancies. This interpretation is deduced from the positron annihilation data in Ni₉₄Al₆ [2].

Stage IV (> 470K). After a plateau, the resistivity decreases strongly and tends to merge with the equilibrium resistivity curves obtained in the unirradiated materials [2]. Thus the irradiation effects disappear in this stage.

Fig. 2. Isochronal resistivity recovery of Ni₉₄Al₄Ti₂ 2.5 MeV-irradiated to a fluence of 9.3×10^{18} e/cm² and of Ni₉₄Al₆ [1] (2.95 MeV, 8×10^{18} e/cm²).

(a) Direct and (b) derivative curves.

Fig. 3. Isochronal resistivity recovery of intermetallics irradiated to a fluence of 9.3×10^{18} e/cm². (a) Direct and (b) derivative curves.

A strong interaction between Ti and lattice defects was observed when comparing ternary to binary solid solutions. The main effects due to the substitution of Al with Ti are:

- 1- an opposite evolution in stage II which leads to a progressive resistivity recovery reaching 85% of the initial increment, at 300K
- 2- stage III, corresponding to vacancy migration, is split and shifted to higher temperatures (by more than 20K)

Figure 3 displays the resistivity variations during the isochronal anneals for the three intermetallics. The behaviour of the binary intermetallic can be analyzed as follows [1]: stage I (50 to 130K) and stage III (290 to 475K) are comparable respectively to stages I and III of the the solid solutions described previously. Between these two stages, stage II consists in at least two substages attributed to the migration or dissociation of interstitial clusters with the subsequent elimination of the free interstitials at sinks. Stage III', the origine of which is not definitely established, might be related to the migration of trapped vacancies or vacancy clusters which is activated at higher temperatures. These stages have been previously observed in stoichiometric and off-stoichiometric Ni_3Al [1]. Like in the solid solutions, the presence of Ti or Ta significantly changed the recovery stages. The effects can be summarized in:

- 1- A suppression of stage I and a promotion of new peaks in satage II which can be attributed to the trapping and subsequent release of interstitial defects.
- 2- A shift of stage III to higher temperatures (by 15 to 20 K), indicating a retardation of vacancy migration.
- 3- A suppression of stage III', which might be due to a stabilization of vacancy clusters.

IV. CONCLUSION

The behaviour of Ni(Al)-based solid solutions and intermetallic compounds during electron irradiation and during subsequent anneals was investigated by means of residual resistivity measurements. Characteristic properties of point defects such as Frenkel-pair resistivities and recombination volumes were determined. Only the latter appear to be affected by the presence of Ti or Ta, suggesting a stabilization of the irradiation-induced defects. The effects of these elements on the recovery processes were very significant in both types of phases. In the intermetallic compounds, their presence suppressed the recovery corresponding to interstitial defect migration (stage I) and promoted the growth of substages at higher temperatures (in stage II). The vacancy migration stages were shifted to higher temperatures both in the solid solution and in the intermetallic compounds, indicating vacancy trapping by Ti or Ta. It is proposed that such a decrease in vacancy mobility may explain (at least in part) the self-diffusion retardation deduced by the present authors (these Proceedings) from thermal experiments performed on the same materials. Measurements of activation enthalpies in the various recovery stages are in progress.

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