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# THE IRRADIATION-INDUCED MICROSTRUCTURAL DEVELOPMENT IN Ni-AI ALLOYS

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The microstructural development for Ni-13.1 at % Al during irradiation was investigated. Different metallurgical conditions were obtained of this alloy, and  $\gamma'$ -precipitates were produced. Specimens were bombarded at 300° under proton irradiation from cyclotron. It was observed that the irradiation changed the precipitate structure and with increasing dose a splitting of the pre-existing precipitates into smaller fragments was observed. The irradiation induced growth of  $\gamma'$ -precipitates in quenched specimens by a displacement rate of 2.10<sup>-6</sup> dpa/sec. at 300° was comparable to the growth under thermal aging at 570°.

Key words: Irradiation, Microstructural development, Ni - Al alloy.

#### Introduction

Several precipitations-hardening nickel and iron base alloys are candidate materials for use in fast breeder and further fusion reactors in which precipitation is an important metallurgical factor [1,2]. The swelling resistance of precipitation hardening alloys in such irradiation environments may be explained by the fact that the precipitate-matrix interfaces can act as sites for enhanced recombination and/or serve as sinks for point defects [3] in particular, the misfit between  $\gamma'$ precipitates and matrix and the precipitate instability will influence the characteristics of materials containing  $\gamma'$ -precipitates. Therefore, it is important to study the irradiationinduced microstructural development in order to understand the behaviour of pre-existing precipitates which may be dessolved.

In this investigation, Ni-13.1 at % Al was chosen for study because nickel alloys containing aluminium forming  $\gamma^{\circ}$  (Ni<sub>3</sub>Al)-precipitates are relatively simple and well-known potential alloys. The following metallurgical conditions were produced of this alloy:

Quenched into cold water	(SQ)
Quenched and aged at 750°:5 hours	(QA)
Quenched, 20% C.W. and aged at 625°:100 hrs	(CA)

#### Exprimental

The specimens used in this investigation were Ni-Al alloy. Strips were cold rolled in steps with intermediate annealing at  $1100^{\circ}$  for 1 hr. Then these specimens were quickly quenched into liquid nitrogen and cold water. The fast quenching of Ni-Al specimens in liquid nitrogen and water is described elsewhere [4]. Some of these quenched samples were then aged at 750° for 1 hr. while others were cold rolled to 20% C.W. and then aged for 100 hrs at 625° in order to produce a \* Institute Für Festkörperforschung der KFA Jülich, W. Germany.

uniform precipitate distribution. Following these heat-treatments, specimens were then irradiated at 300° under 7 MeV proton from the cyclotron. The microstructural changes under irradiation were examined in a Phillips EM 400 transmission electron microscope operating at an accelerating voltage of 120 KV. In order to facilitate thé measurement of particle size, the micrographs were taken in dark field using the super lattice reflections from the ordered  $\gamma$ '-precipitates. All precautions were taken to ensure that the images were aberation free by employing gun-tilt method. Specimens for transmission electron microscopy were prepared by the jet polishing technique.

## **Results and Discussion**

Figure 1 shows the precipitate structure of solution quenched (SQ) Ni-13.1 at % Al specimens after irradiation to a dose of 0.13 dpa at 300°. The dark field micrographs (Fig. 1a) shows a fine uniform distribution of small  $\gamma$ '-precipitates except at regions where the dislocation loops are formed. The dislocation loops are well imaged in a bright field micrograph (Fig. 1b) for the same foil area.

Thermal aging of solution quenched Ni-13.1 at%Al specimens for 5 hrs at 750° (QA) gave a fine distribution of cube shaped  $\gamma'$ -precipitates as shown in Fig. 2a. The average cube edge length is comparable with the literature data [5-7]. Some alignment of the precipitates is also observed. The  $\gamma'$ -precipitates seem to be fully coherent indicating a small strain field which is clear from the bright field image of Fig.2b. The aged specimens irradiation to a dose of 0.14 dpa at 300° results in a fragmentation of these cube precipitates and formation of a new  $\gamma'$  precipitates distributions shown in the micrograph of Fig.3a. The fragmentation of the pre-existing precipitates occurs by the nucleation of dislocation loops within and around  $\gamma'$ -precipitates which are visible in the bright field

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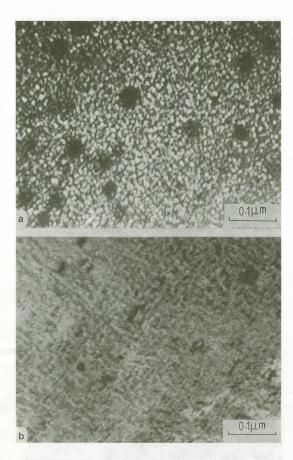


Fig.1.(a) Dark and (b) bright fields electron micrographs of solutionquenched (SQ) Ni-13.1 at%Al alloys after irrdiation to a dose of 0.13 dpa at 300°.

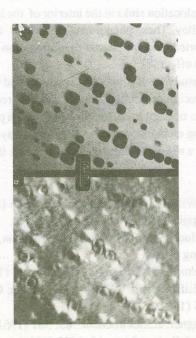


Fig.2.(a) Dark and (b) bright fields micrographs of quenched and aged at 750° for 5 hrs (QA) specimens of Ni-13.1 at% Al before irradiation.

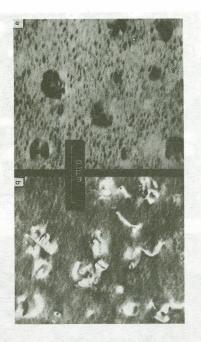
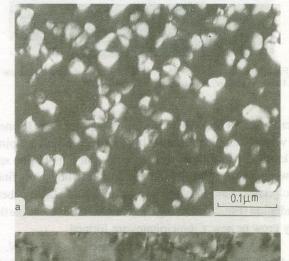


Fig.3.(a) Dark and (b) bright fields micrographs of specimens of Fig.2 after irradiation to a dose of 0.14 dpa at 300°.



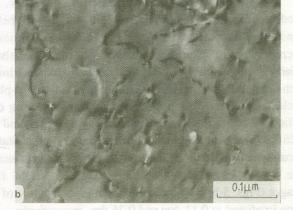


Fig.4.Electron micrographs of quenched, cold worked and aged Ni-13.1 at%Al alloys before irradiation, (a) Dark and (b) bright field images.

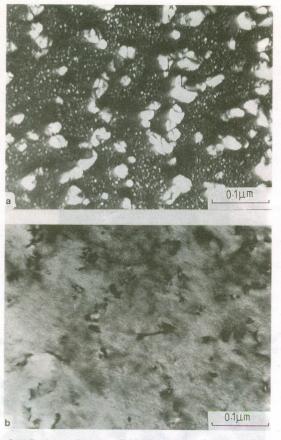


Fig.5.(a) Dark and (b) bright field micrographs of same specimens as in Fig.4 after irradiation to a dose of 0.11 dpa at 300°.

micrograph of Fig. 3b. Dislocation loops are also formed in the way of precipitates where the line dislocations are pinned by loops or precipitates as can be seen in Fig.3b. The space between large  $\gamma'$ -precipitates is density filled with fine  $\gamma'$ precipitates. Fine  $\gamma'$ -phase was precipitated already before irradiation (Fig.2a). During irradiation at 300° these particles may grow or new  $\gamma'$ -precipitates are formed.

The precipitate structure of quenched, 20% C.W. and aged at 625° for 100 hrs (CA) specimens at 300° before irradiation (at zero dose) is given in Fig.4. The dark field image micrograph (Fig.4a) made by using  $\gamma'$ -phase spot shows clearly  $\gamma'$ -precipitates. Correlation of the dark field image with bright field image (Fig.4b) allows to reveal that  $\gamma'$ -phase nucleates preferentially at dislocations formed during cold rolling. After irradiation to a dose of 0.11 dpa (Fig.5) and to a 0.26 dpa (Fig.6) the fragmentation of the large  $\gamma'$ -precipitation occurs which increases by increasing the dose. In the structure fine  $\gamma'$ -particles are also visible (Figs. 5a, 6a). Bright field images (Figs. 5-b, 6-b) show the precipitate structure of the alloy irradiated to 0.11 dpa and 0.26 dpa, respectively.

Irradiation of Ni-Al alloys resulted in resolution of preaged  $\gamma'$  into small fragmentation and reprecipitation at sur-

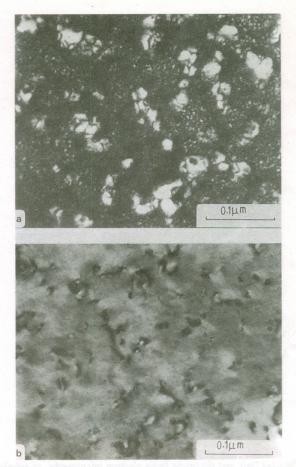


Fig.6.Same specimens as in Fig.4 to a dose of 0.26 dpa at 300° (a) Dark and (b) bright fields micrographs.

faces or dislocation sinks at the interior of the irradiated area in Ni-Al alloy. These phenomena are similar to those previously reported, and are caused by segregation depending on solute size effect [8].

The instability of  $\gamma$ ' may also be associated with the solute size and precipitate misfit effect, although the recoil resolution will be also operative, i.e, for Ni-Al alloy with positive misfit, the alloying elements could be diffusing away from the precipitate as a results of vacancy flow towards the interfaces.

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