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Evolution of the microstructure and of the mechanical properties of γ/γ' NiAlTi alloys during electron irradiation

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Abstract

In an Ni–4.5at.%Al–6.5at.%Ti two-phase alloy, low dose electron irradiation has been shown to have two opposite effects, according to the operative deformation regime (precipitate shearing or precipitate by-passing). This paper compares direct observations of microstructural changes during irradiation in a high voltage electron microscope and the observed changes in mechanical properties. The displacement rates used in the microscope and in the accelerator are very different; furthermore, comparison between the observed effects suggests that the irradiation-induced decrease in flow stress when deformation occurs by shearing of γ' particles, is due to disordering of the latter. The increase in flow stress in the by-passing regime is due to the creation of point defects and their clusters in the matrix.

1. Introduction

Nickel-based superalloys are formed by a γ-matrix which is a disordered solid solution of alloying elements in nickel, with γ' ordered precipitates having the composition $Ni_3(Al, X)$ and structure $L1_2$. These alloys are interesting materials for use in an irradiation environment owing to hardening resulting from the presence of γ' precipitates. Moreover, it has been pointed out that two-phase alloys exhibit a high resistance to swelling under irradiation. In fact, in addition to compositional effects of alloying elements in reducing the swelling rate under irradiation at elevated temperatures [1], the γ–γ' interfaces act as convenient sites for defect annihilation, which may reduce the vacancy concentration within the matrix and thus inhibit void nucleation [2]. The effects of irradiation on the microstructure of nickel-based superalloys have been studied in industrial alloys and binary Ni–Al alloys, in the high fluence–high temperature range [2, 3]. However, ternary Ni–Al–Ti alloys have not been investigated.

The aim of this work was to compare the effect of electron irradiation in the low dose–moderate temperature range, on the mechanical properties of ternary NiAlTi alloys with the microstructural changes observed during irradiation in a high-voltage electron microscope (HVEM). The alloys were chosen to have a simple composition which, however, reproduces the γ/γ' microstructure of industrial alloys. The solute elements in the matrix are aluminium and titanium, and precipitates are of the composition $Ni_3(Al, Ti)$. The evolution of the microstructure of these alloys, and the resulting changes in mechanical properties during aging treatments, have been reported in previous papers [4, 5].

2. Experimental details

An alloy of composition Ni–4.5at.%Al–6.5at.%Ti and a γ' volume fraction of 11.6% was used for this investigation. Specimens for transmission electron microscopy were taken from a sheet 0.2 mm thick obtained by rolling; they were quenched from 1473 K and aged for 8 h at 1025 K before final thinning. Irradiation experiments were performed with 1 MeV electrons in the HVEM of D. Tech. CEN-Saclay at temperatures of 190–400 K and displacement rates of 0.29–12 dpa h^{-1}. Morphological changes were observed both in bright fields and dark fields. At regular intervals of time during irradiation, the electron beam was defocused and a sequence of pictures of bright field, dark field and diffraction patterns was taken. Dark field images were obtained using γ' superlattice reflections. For tensile tests, specimens 10.25 mm in length and 2 mm in width, obtained by punching from a sheet of 0.1 mm thick, were annealed at 1025 K for different times to obtain γ'-particles with different sizes [4]. They were subsequently irradiated in a Van de Graaff accelerator with 2 or 2.5 MeV electrons at 400 K and displacement rates of 32 or 8×10^{-6} dpa h^{-1}. Specimens were irradiated to doses of 9 or 7×10^{-4} dpa. For more experimental details, see refs. 5 and 6.

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3. Results and discussion

The microstructural evolution of the alloy under irradiation has been reported previously [6, 7]. We summarize below the main observations.

At an irradiation temperature of 290 K, and for all defect production rates used (0.29, 2 and 12 dpa h^{-1}), a high dislocation density nucleates in bright field images at the early stages of irradiation, owing to point defect condensation. At this temperature, only interstitials are mobile [8]; therefore, the dislocation loops should be of interstitial nature. Dark field images show a decrease in the contrast of the γ' precipitates during irradiation, probably due to disordering of the latter. In the advanced stages of irradiation, fine precipitates with $L1_2$ structure nucleate, representing a reprecipitation of γ' precipitates after partial dissolution of the pre-existing coarse particles [6].

At a higher temperature (400 K), the alloy exhibited a similar evolution. However, this was only detected for a defect production rate of 12 dpa h^{-1}. No changes were observed for the two lower rates.

Figure 1 shows a set of diffraction patterns taken for increasing irradiation doses at 290 K. It shows that the intensity of the γ'-superlattice spot (arrowed on the picture) decreases as the irradiation dose increases.

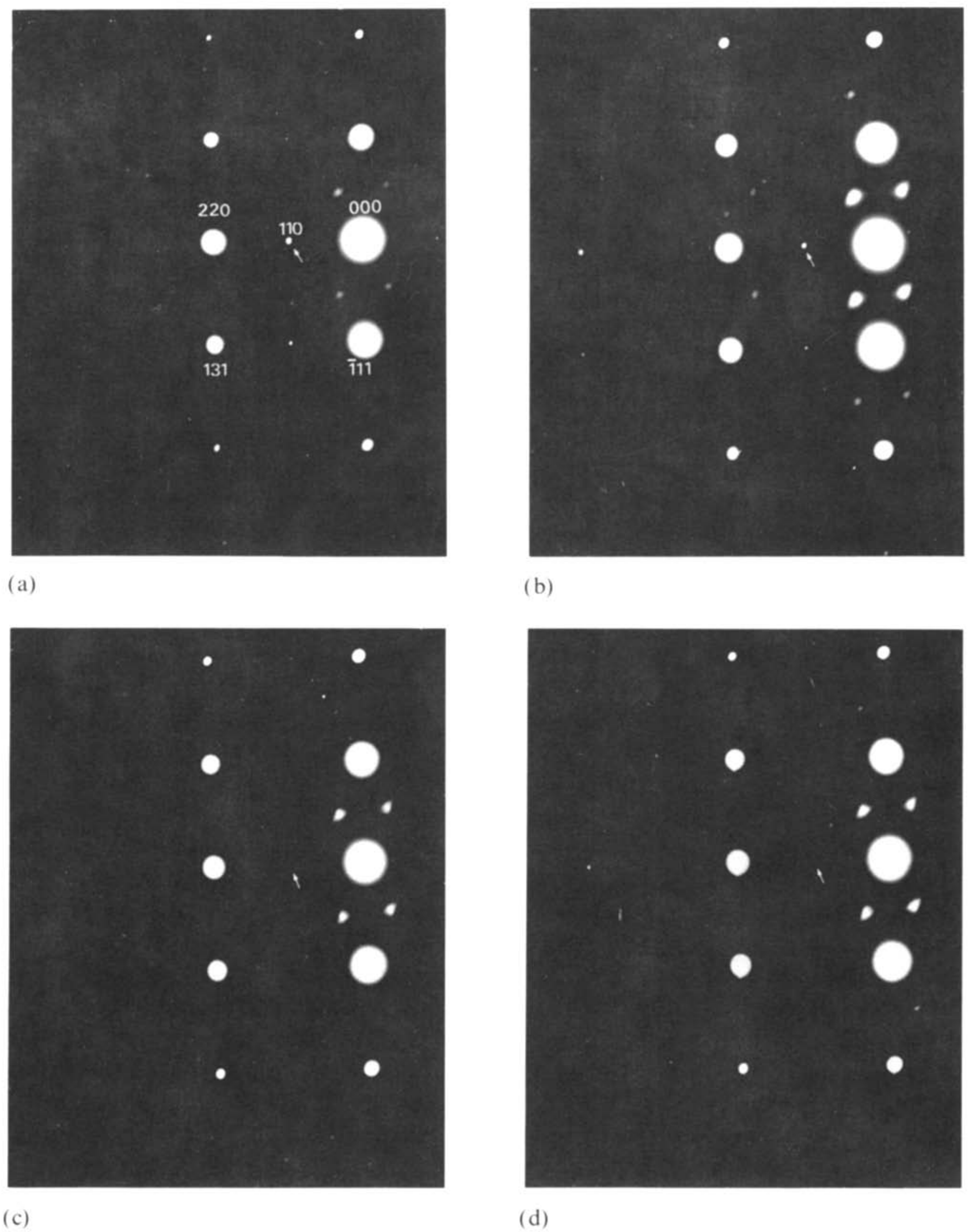

Fig. 1. (112) diffraction patterns recorded for increasing doses during 1 MeV electron irradiation with displacement rate of 2 dpa h^{-1} at 290 K: (a) 0 dpa, (b) 0.41 dpa, (c) 1.7 dpa, (d) 2 dpa. Note the evolution of the intensity of the 110 superlattice spot.

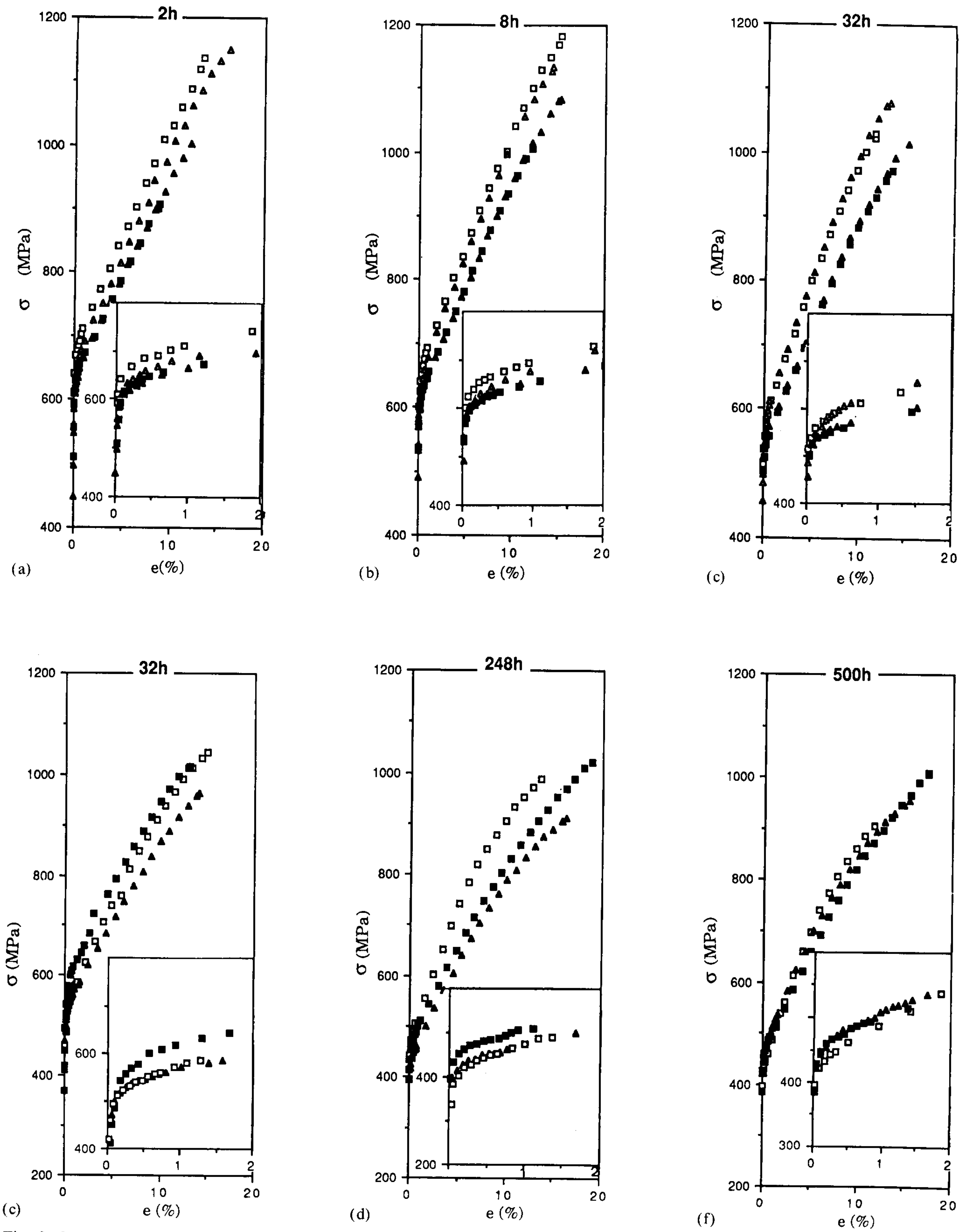

Fig. 2. Stress–strain curves of the alloy before and after irradiation at 400 K: (a)–(c) 2.5 MeV electrons, 6.7×10^{-4} dpa; (d)–(f) 2.0 MeV electrons, 9.4×10^{-4} dpa. Aging times at 1025 K are indicated on the plots.

Owing to the fact that for kinematical conditions of diffraction, the intensity is linear in S^2 (S is the long-range order parameter) [7], the decrease in superlattice spot intensity should be attributed to a disordering of γ' precipitates. The kinetics of this disordering is discussed in refs. 6 and 9. For all temperatures, the S parameter decays rapidly for the shorter irradiation times to reach (except at 190 K where we have observed full disordering of the γ' phase), a steady-state value for the longer times. The steady-state value depends on irradiation conditions and is the result of competition between irradiation-disordering and thermally activated reordering [7].

A set of stress–strain curves showing the effects of low-dose irradiation on the tensile properties of the alloy are shown in Fig. 2. Aging times of specimens at 1025 K are indicated on the plot. These times were chosen based on previous work, to define γ'-particle distances and hence the deformation mechanism of the alloy [5].

In the shorter aging-time specimens (2–32 h), a slight decrease in the yield stress is observed, whereas in the long aging-time alloys (32–500 h), we observe a slight increase in the yield stress. The work-hardening coefficient is lower in the irradiated samples. In the specimens with small γ' particles (2–32 h), the flow stress after irradiation remains lower for all elongations, whereas in the specimens with large γ' particles (32–500 h) a crossover occurs and the initially irradiation-hardened specimens exhibit a lower flow stress at large strains.

In the particle-shearing regime of dislocation glide (small particles, formed at short aging-times), alloy hardness is controlled mainly by the precipitate properties [5, 10]; the irradiation-softening of the alloy is due to the disordering of γ' particles leading to a decrease in their anti-phase boundary energy and yield stress.

When deformation occurs by by-passing of precipitates (coarse particles, resulting from long aging-times), dislocation glide is mainly affected by matrix properties [11]. The irradiation-created point defect and their clusters within the matrix act as pinning points for dislocations. Another explanation of the irradiation-hardening for this range of particle size is a possible local ordering of the matrix due to irradiation-enhanced diffusion at the irradiation temperature of 400 K. Such an effect has been demonstrated in the high nickel content f.c.c. alloys of the Fe–Cr–Ni system [12].

Regardless of the deformation mechanisms, low-dose irradiation has a weak effect on the flow stress (20–25 MPa) because of the small doses used. Observations in the HVEM at the same temperature did not show any microstructural change during irradiation at displacement rates up to 2 dpa h^{-1}. The main reason for this observation is probably the fact that at 400 K vacancies become mobile, thus enhancing point defect recombination and reducing their stationary concentration. The processes resulting in the decrease in yield stress in the shearing regime and in its increase in the by-passing regime are then so reduced that they cannot be detected by direct observations of microstructural changes in the microscope. In fact, the slight decrease in 0.2% flow-stress should result from a decrease by a few per cent of the long-range order parameter (4%–5%); such variation cannot be detected by measuring the variation of the γ'-superlattice spot intensity on the diffraction pattern.

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