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Study of precipitation along a concentration gradient

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The present work is based on the microstructural characterization method proposed by T. Miyazaki [1-3], the so-called Macroscopic Composition Gradient (MCG) method. This technique allows the investigation of phase transformations in a single specimen and helps to evaluate the mechanical properties for different alloy compositions. It is based on the microstructural observation of a continuous concentration gradient, which can be generated by several methods, for instance, diffusion coupling, imperfect homogenization of coarse discontinuous precipitates, etc.

Buttons of Ni–11.5 wt. % Ti alloy and pure Ni were melted in an electric-arc furnace under an argon atmosphere using pure elements (99.9 %). An assembly consisting of the buttons was placed into an austenitic stainless steel holder with two screws, encapsulated into a quartz tube under an argon atmosphere and heat treated at 1200 ºC for 28 h to promote the diffusion and generate the concentration gradient in the diffusion couple, subsequently, the diffusion couple was isothermally aged at 850, 750 and 650 ºC for different times. Microstructural characterization was carried out by High Resolution Scanning Electron Microscopy (HR-SEM) using a JSM-7401F microscope with Energy Dispersive Spectroscope (EDS).

The diffusion process that occurs during annealing and aging treatments produces a characteristic microstructure in the diffusion couple, where the Kirkendall effect and a mixture of phases are evidenced. The Fig. 1a shows the microstructure at the interface of the Ni–13.75 Ti (at. %)/Ni diffusion couple after annealing at 1200 °C. The variation of Ti concentration as a function of distance is also shown in this figure evidencing the concentration gradient at the diffusion couple interface. A region of about 140 µm that goes from the interface to the Ti-rich side, delimited by the solvus line, exhibits the presence of voids, which are formed due to the different diffusion rates of the diffusing elements. As reported elsewhere [4], the Ni diffusion rate is higher than that of Ti. Figs. 1b and 1c, show the solvus line and the precipitation boundary of the γ' phase in samples thermal aged at 850 °C. The phases observed in these figures with cuboidal-shaped morphology correspond to γ' phase and those with plate-shaped morphology to Ni_3Ti precipitates (η-$D0_{24}$). The solvus and the precipitation boundary of the γ' phase determined experimentally by EDS were found at 9.16 and 9.92 Ti (at. %), respectively. These values are close to the corresponding values in the Ni-Ti phase diagram [5].

The variation in Vickers hardness (HVN) as function of aging time in Ni-rich Ni-Ti alloys with different Ti concentration is shown in Fig. 2. The maximum hardness observed (under all temperatures) is related with the presence of γ' precipitates. In addition, it is observed that as aging temperature decreases, the *fv* of precipitates and the HVN increase, but at concentrations less 6 at. % Ti there is not precipitation hardening at 3 aging temperatures studied.

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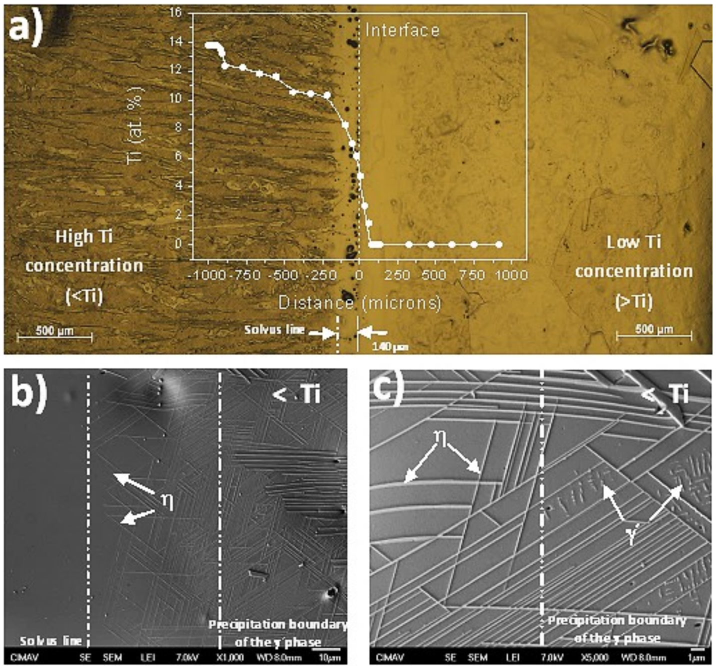

Figure 1. a) Optical micrograph of Ni–13.75 Ti (at. %)/Ni diffusion couple and Ti concentration profile, b) and c) FE-SEM images indicating the solvus line (-·-·-) and precipitation boundary of the γ' phase (-··-··-).

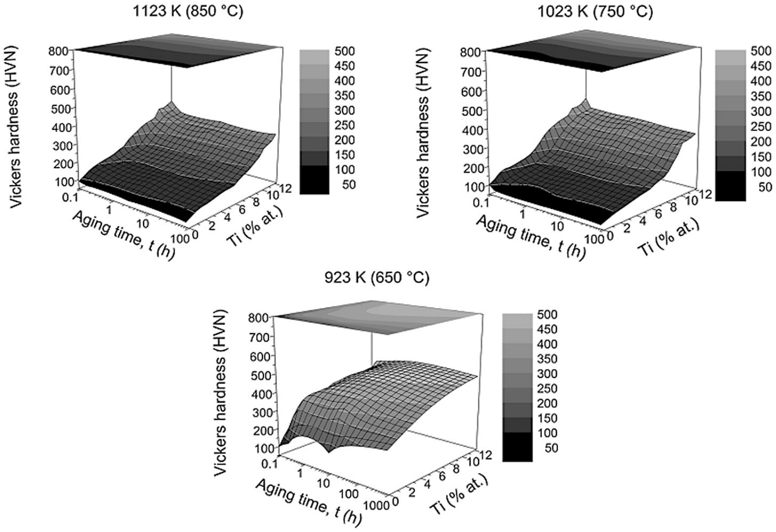

Figure 2. Age-hardening curves obtained as a function of Ti concentration at 850, 750 and 650 °C.