

## 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.

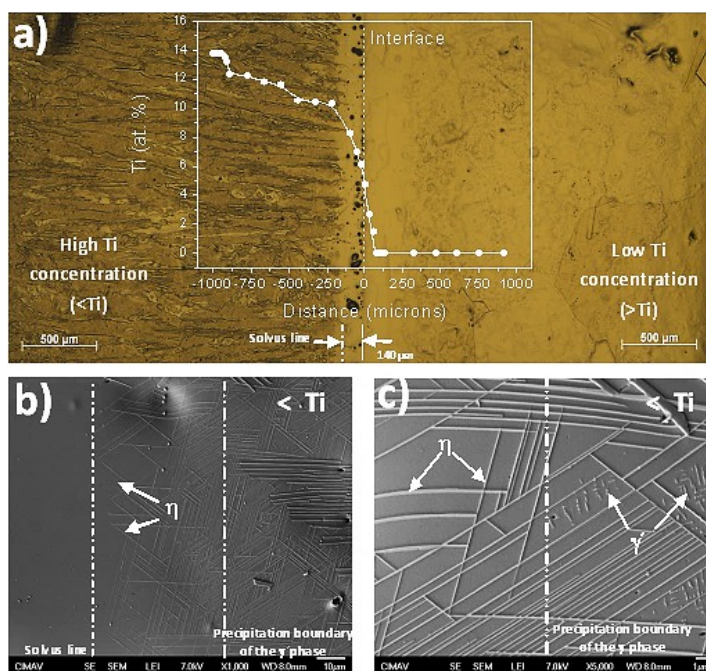
Buttons of Ni–11.5 wt. % Ti alloy and pure Ni were melted in an electric-arc furnace under an argon atmosphere using pure elements (99.9 %). An assembly consisting of the buttons was placed into an austenitic stainless steel holder with two screws, encapsulated into a quartz tube under an argon atmosphere and heat treated at 1200 °C 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 °C for different times. Microstructural characterization was carried out by High Resolution Scanning Electron Microscopy (HR-SEM) using a JSM-7401F microscope with Energy Dispersive Spectroscopy (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  $\gamma'$  phase in samples thermal aged at 850 °C. The phases observed in these figures with cuboidal-shaped morphology correspond to  $\gamma'$  phase and those with plate-shaped morphology to  $\text{Ni}_3\text{Ti}$  precipitates ( $\eta\text{-D0}_{24}$ ). The solvus and the precipitation boundary of the  $\gamma'$  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].

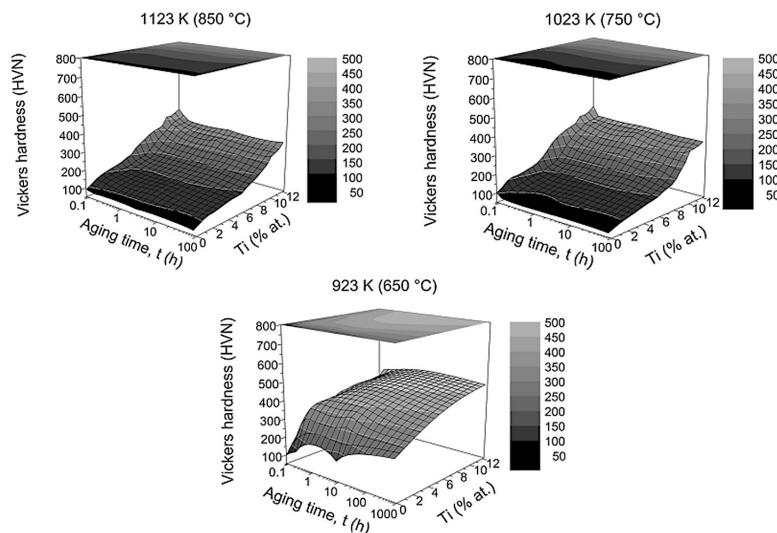
The variation in Vickers hardness (HVN) as function of aging time in Ni-rich Ni-Ti alloys with different Ti concentration is shown in Fig. 2. The maximum hardness observed (under all temperatures) is related with the presence of  $\gamma'$  precipitates. In addition, it is observed that as aging temperature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

## References

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 [4] S. Hinotani, Y Ohmori, *J Jpn Inst Metals* 29 (1988), p. 116-124.  
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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  $\gamma'$  phase (-·-·-).



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