

Microstructural Study of Internal Oxidation of Dilute Ni-Al Alloys

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Internal oxidation occurs when a component of an alloy is selectively oxidized so fast that it can't reach the surface to form an oxide scale. Then it results in a subsurface formation of oxide precipitates that embrittle and expand the alloy subsurface, causing break off and failure of structural components [1]. Particularly, Aluminium depleted γ Ni-Al solid solutions are susceptible to internal oxidation [2,3]. Most of the research performed on the internal oxidation of dilute NiAl alloys has been done at 1, 5, and 10 h [1,2,3]. Here 20 h of internal oxidation is presented to complement current research.

NiAl dilute alloys of composition 1.0, 2.5 and 4 wt. % Al (namely A, B and C respectively) were prepared using pure elements (≥ 99.9 wt % purity) and clean melting with non-consumable tungsten electrode in a copper water-cooled crucible. The cast alloys were heat treated in vacuum at 1200 °C during 24 h for homogenization. Electropolishing was used to obtain a clean surface on the samples prior to oxidation. Rhines Pack method [4] at 1100°C for 1, 10 and 20 hours were used for internal oxidation (isothermal). The cast alloy, heat treated, oxidized and internal oxidation products, were characterized using X-Ray diffraction (XRD), Scanning Electron Microscopy and Energy Dispersive X-Ray spectroscopy SEM/EDS analyses. For the analysis of exposed internal oxide precipitates, metal from the alloy surface and from the internal oxidation zone (IOZ) was removed with deep electrochemical etching.

BSE images of the cross-section in the alloys after internal oxidation (Figure 1), alloys B and C (2.5 and 4 wt. % Al respectively) show non-uniform oxidation front; the alloy B presented a lateral discontinuous development of Al_2O_3 layer with dark grey contrast at the oxidation front. The alloy C presented the shortest IOZ including at grain boundaries where the precipitation front was deeper. It also presented a coarse Al_2O_3 inner layer which clearly indicated the transition from internal to external oxidation. SE images of the exposed oxide precipitates after deep electrochemical etching and X-Ray elemental maps in figure 2 show morphology and their elemental composition. In alloys A and B, the precipitates formed as a network of rod-like oxide particles grown from the oxidation front to the alloy surface while in alloy C rod-like precipitates were attached to a compact Al_2O_3 layer formed in the oxidation front. XRD diffractograms of the alloy B in all studied conditions shown in figure 3a suggested the presence of Ni_{ss} for cast and heat-treated conditions, Ni_{ss} and NiAl_2O_4 phase after all internal oxidation times. The insert image in figure 3a also shows the XRD data of deeply etched A, B and C alloys and suggested the presence of Ni_{ss} and NiAl_2O_4 , α - Al_2O_3 phases. Elemental line scans of a cross-section of the etched alloy B in figure 3b suggests Al_2O_3 at the oxidation front and NiAl_2O_4 near the surface of the alloy [5].

References:

- [1] F.H Stott *et al*, Solid State Ionics **12** (1984), p. 365.
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- [3] D. J. Young in "High Temperature Oxidation and Corrosion of Metals", ed. Tim Burstein, (Elsevier, Oxford), p. 247.

[4] F.N. Rhines, W. Johnson, W. Anderson. *Trans. Am. Inst. Min. Metall. Engrs.* **147**(1942), p. 205.

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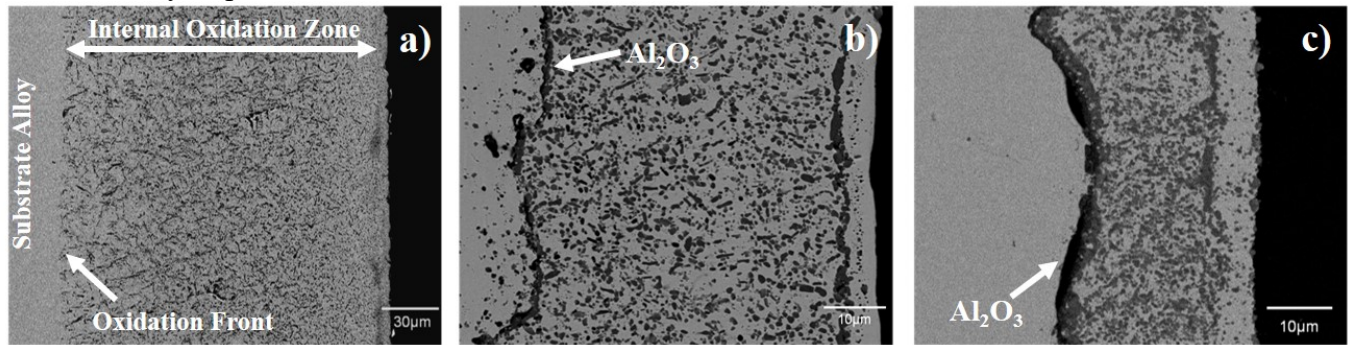


Figure 1. a) BSE images of typical microstructure of the internally oxidised alloys for 20 h a) 1 wt. % Al, b) 2.5 wt. % and c) 4wt. % Al.

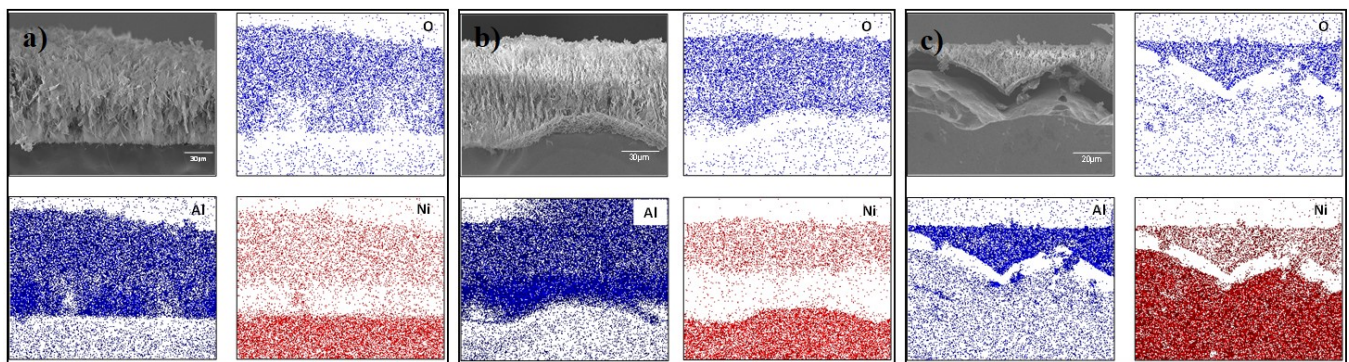


Figure 2. X-ray elemental maps and SE images from cross section of deeply etched alloys after internal oxidation at 1100 °C for 20 h, a) Alloy A , b) Alloy B and c) Alloy C.

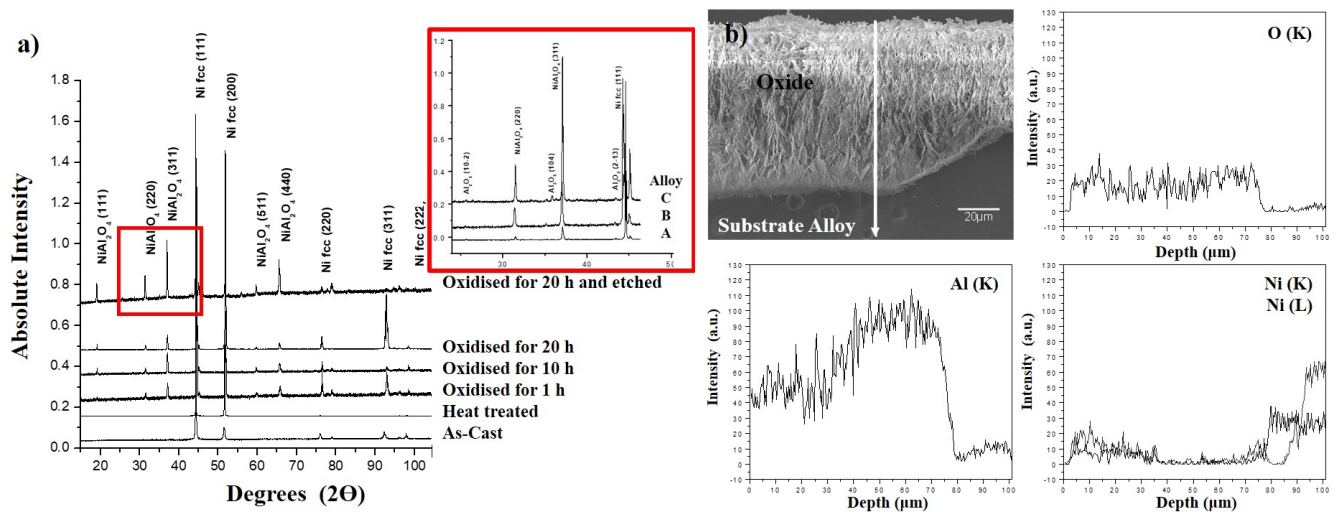


Figure 3. a) XRD diffractograms of the alloy B in the as-cast and heat treated conditions, oxidised for 1, 10 and 20 h and deeply etched after oxidation for 20 h, insert image show Al_2O_3 peaks in the deeply etched alloys after internal oxidation for 20 h, b) Elemental line scans of a cross section of the deeply etched alloy B after internal oxidation at 1100 °C for 20h.