doi:10.1017/S1431927617004160
Microsc. Microanal. 23 (Suppl 1), 2017

Atom Probe Characterization of Oxide Layers Formed on Polycrystalline Nickel Based Superalloys

M.T. Lapington[1], D.J. Crudden[1], R.C. Reed[1], M.P. Moody[1] and P.A.J. Bagot[1]

[1.] Department of Materials, Parks Road, Oxford, United Kingdom

Nickel based superalloys are a class of high-temperature materials used extensively throughout the high pressure sections of jet turbine engines. Two characteristics that make them particularly suited to this application are the ability to maintain strength at high temperatures, combined with the formation of a protective oxide scale which prevents further oxidation damage. These characteristics are prime motivations during the alloy design process.

Care must be taken when manipulating alloy composition however, as elements which enhance certain material properties can be detrimental to others. This kind of property trade-off is highlighted in the case of titanium additions, which are vital to maintaining strength at high temperatures but have previously been shown to reduce oxidation resistance [1]. One possible approach to managing this tradeoff is to replace Ti with another element that maintains strength and partitions to the same phase. Previous thermodynamic modelling and density functional theory calculations predict that Nb should provide the same strengthening effect as Ti but to a lesser extent [2]. Additionally, Nb has been used previously in superalloys without dramatically increasing oxidation rates [3].

In order to investigate this strength-oxidation tradeoff, a series of nickel superalloys has been created which are identical in regard to manufacturing technique and composition, with the exception of substituting titanium (4.0 to 2.8at.%) with niobium (0 to 1.2at.%) in a 1:1 ratio. The susceptibility to oxidation for these alloys was measured by performing Thermo-Gravimetric Analysis (TGA) using a Netzsch Jupiter F1. Figure 1 displays the specific mass gain over time for each alloy, indicating a correlation between the Ti:Nb ratio and observed oxidation rates.

The oxide layers that formed on the TGA specimens were prepared for further characterization using a Zeiss Nvision40 FIB-SEM Focused Ion Beam instrument. Trenches were cut perpendicular to the surface through the oxide layer in order to observe the cross-section using SEM (Fig. 2). The structure of the various oxide layers can be seen, damage depth has been evaluated and other features such as oxidized grain boundaries have been identified. Similar cross-sections through oxide layers have also been analyzed using Energy-Dispersive X-ray spectroscopy (EDX), which have enabled chemistry to be identified through the creation of a quantitative composition map. Much higher resolution and mass sensitivity are available through the use of Atom Probe Tomography (APT), which also provides a 3D representation of the structures present. This allows visualisation of structural features such as grain boundaries and defects, along with extremely localized changes in composition resulting from segregation, which can play a large role in corrosion processes.

Atom probe specimens were prepared in the Nvision40 FIB from specific sites in the chromium oxide layer. The needle-shaped specimens were carefully milled and constantly imaged to make sure that the final samples would represent selected depths through the chromia layer. Samples were run on a Cameca LEAP5000 using laser-mode to aid ion evaporation. The atom maps shown below in (Fig. 3) come from the high-Ti alloy (4at.% Ti, 0% Nb), with all atoms displayed in the left-hand atom map and only Ti, TiO and TiO_2 species shown on the right. Segregation of Ti-containing species to this planar feature is being investigated as a possible causal link to oxidation kinetics. 1D concentration profiles taken across these interfaces found many other species, including a number of other oxides not present through the rest of the chromia.

References:

[1] H. Nagai, M. Okabayashi. Transactions of the Japan Institute of Metals **22** (1981) p. 691.
[2] D.J. Crudden *et al*, Acta Materialia **75** (2014) p. 356.
[3] F. Weng *et al*, Surface Interface Analysis **47** (2014) p. 362.

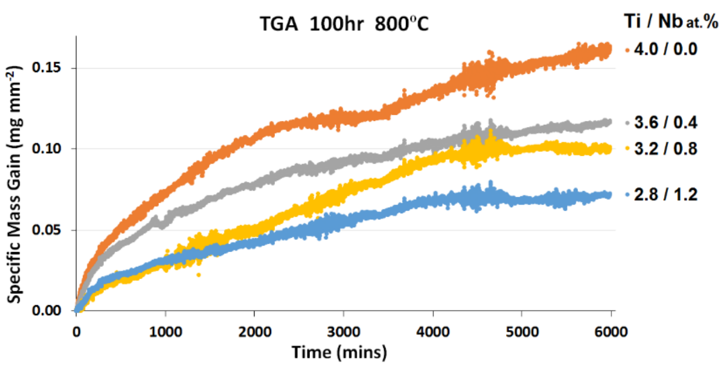

Figure 1. Specific mass gain against time during TGA of alloys with specified Ti/Nb compositions. Performed at 800°C over 100hrs.

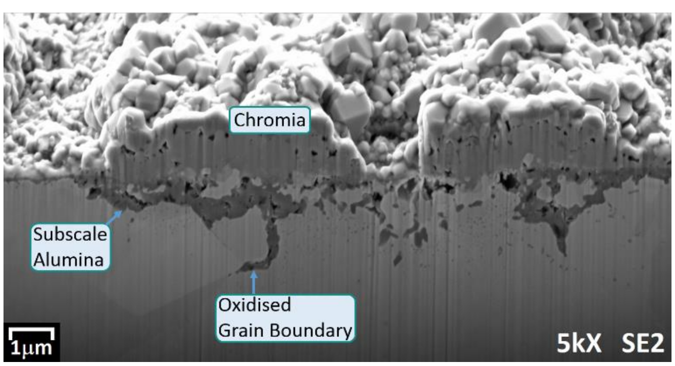

Figure 2. Oxide cross-section imaged during FIB sample preparation, showing structure within and below oxide scale.

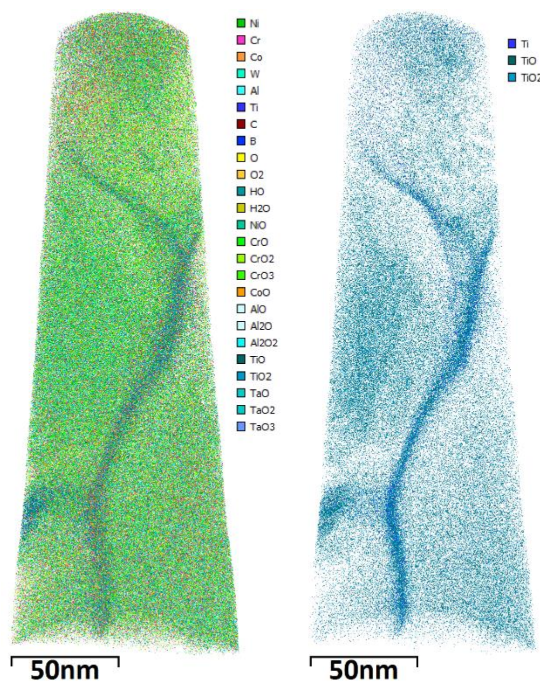

Figure 3. Reconstructed atom maps of high-Ti superalloy (4at.%Ti, 0%Nb), showing all detected ions on left, and Ti-containing species only on right.

https://doi.org/10.1017/S1431927617004160 Published online by Cambridge University Press