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THE CHARACTERISATION OF "ALUMINISED" INCONEL

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Résumé - L'Inconel aluminisé est un produit commercial fabriqué suivant des procédés confidentiels. On caractérise sa microstructure en utilisant : la microscopie optique, les microanalyses par émission X et par émission d'électrons Auger, la diffraction des rayons X, etc...

Abstract - 'Aluminised' Inconel is made commercially by undisclosed methods. Consequently its microstructure is being characterised. This has been done using: optical microscopy, electron microprobe analysis, Auger microprobe analysis, X-ray diffraction, Auger mapping, Digimapping, and multiple quantitative electron microprobe point analyses. The results obtained are combined to show how it has been possible to characterise 'the structure' of such a coating.

I - INTRODUCTION

The UKAEA is considering the use of 'aluminised' Inconel in the hostile environment of a fast reactor where it offers the promise of mechanical robustness, and ability to withstand chemical attack by sodium and the fast neutron flux present, as well as having tribologically desirable properties.

'Aluminised' Inconel is made commercially by an undisclosed process, or processes, and is marketed by various suppliers in different countries. If the UKAEA is to use this material it must understand the nature of the 'aluminised' material and be able to distinguish and assess the material as supplied by different manufacturers. The characterisation of a single sample of this material is described here to demonstrate how a number of techniques must be used in concert to understand and characterise the nature of this material.

II - EXPERIMENTAL

The following methods have been applied to characterise 'aluminised' Inconel. They are:

(i) Optical microscopy (using Nomarski interference contrast) applied to polished transverse sections of the material. Note: before sectioning, the material was nickel plated to preserve any fine surface details during sectioning.

(ii) Electron, microprobe analysis, EPMA, was carried out for selected elements using simple line scans made perpendicular to the sample surface with a Jeol JXA-50A electron microprobe analyser.

(iii) Auger microprobe analysis, AMPA, was made with a Jeol JAMP-3 Auger microprobe. Analyses were carried out on the as-received sample and then repeated after a series of in-situ argon ion etches. In this way an elemental depth profile was established to a depth of 90 μm into the material.

(iv) X-ray powder diffraction, XRD, was carried out on the surface of the as-received material using a Philips PW 1051 X-ray diffractometer. The surface of the material was then polished away and the analysis repeated. This process was repeated until the Inconel substrate was reached.
The electron microprobe analyser was used in conjunction with a Link Systems 860 series II energy dispersive analysis system to generate Digimaps. These are maps which allow one to colour code for a particular element (or concentration). This method of mapping has the further advantage that all elements of interest in a particular picture point (pixel) are analysed for simultaneously. Therefore the maps for each element are in exact registry.

Auger maps were made for selected elements using the JAMP-3 on polished cross sections of the 'aluminised' material.

Finally the electron microprobe analyser was used to generate very many precise quantitative analyses on selected points of interest on the electron image of the polished sectioned material.

III - RESULTS

These are shown for each technique used in Figs 1 to 13 and summarised in Fig. 14.

The optical micrograph, Fig. 1, showed that the structure of the 'aluminised' coat was complex. This was confirmed by both the EPMA line scans made perpendicular to the surface, Fig. 2, and the elemental depth profiles made by AMPA, Fig. 3. Although clearly these results are consistent they gave no meaningful understanding of the material. This was achieved as described below.

The combined techniques showed that the 'aluminised' layer was superficially contaminated with $\text{AlN}$ and $\alpha$-$\text{Al}_2\text{O}_3$. The presence of nitrogen was demonstrated by AMPA, Fig. 3, and the presence of $\alpha$-$\text{Al}_2\text{O}_3$, tentatively, by quantitative EPMA. These two results were keyed together by the XRD results which were themselves unusual. This was because the $\text{AlN}$ and $\alpha$-$\text{Al}_2\text{O}_3$ were embedded in fissures in the surface, as shown by Auger mapping, Figs 4, 5 and 6. The result was that the low angle Bragg reflections were anomalously weak.

The main body of the coating was f.c.c. $\beta-2$ $\text{Al}(\text{Ni, Fe})$ termed $(\text{AlNi})''$. Its structure was shown by XRD, Fig. 7, and its lattice parameter, which varied slowly with depth into the coating, in Fig. 8. The lattice parameter and its variation corresponded to that which would have been expected from the composition of this phase as determined by a series of quantitative EPMA analyses made across the coat but avoiding any precipitates in it. The crystallographic orientation of this phase was also determined by the inverse pole figure method, Fig. 9. This showed that the $(\text{AlNi})''$ was orientated with the crystallographic 111 planes formed preferentially parallel to the surface of the sample.

XRD also showed that the outer regions of the $(\text{AlNi})''$ phase were associated with body centred cubic $\text{AlCr}$. EPMA revealed that this phase occurred as discrete precipitates within the outer layers of the coating. It also showed the presence further into the coating of niobium and molybdenum rich precipitates within the $(\text{AlNi})''$ phase - a fact not noted by XRD.

Immediately above the 'reaction layer' - a two phase region which separated the $(\text{AlNi})''$ coating and the Inconel - Digimapping, Fig. 10, found a niobium/titanium phase. This was shown to be a carbide by Auger mapping, Figs 11 and 12, a fact then confirmed by the XRD results which demonstrated, from its lattice parameter, the presence of $\text{Nb}_{0.8}\text{Ti}_{0.2}$. Quantitative EPMA data, Fig. 13, showed that in this region a phase occurred. The composition of $\alpha$ was $\text{Al}_0.02\text{Cr}_{0.67}\text{Fe}_{0.2\gamma}\text{Ni}_{0.06}\text{Mo}_{0.03}\text{Si}_{0.02}$. Beneath this region was the 'reaction zone'. Digimapping, Fig. 10, and multiple quantitative point analyses confirmed that this consisted of two phases, $\beta$ and $\gamma$. These phases had the compositions:

- $\beta$: $\text{Al}_{0.01}\text{Cr}_{0.49}\text{Fe}_{0.26}\text{Ni}_{0.16}\text{Nb}_{0.02}\text{Mo}_{0.05}\text{Si}_{0.02}$
- $\gamma$: $\text{Al}_{0.02}\text{Cr}_{0.24}\text{Fe}_{0.24}\text{Ni}_{0.19}\text{Nb}_{0.19}\text{Mo}_{0.06}\text{Si}_{0.04}\text{Ti}_{0.01}$
These phases were noted by the XRD results only as phase L, Fig. 7, but they were neither distinguished nor resolved by this method.

The EPMA data were extended to show that γ was the Ni-rich, Fe-poor member of a phase γ' which extended 40 μm into the Inconel as a grain boundary precipitate.

The quantitative EPMA results showed finally that the aluminising process had significantly perturbed the composition of the Inconel to a depth of 80 μm below the reaction zone.

All these results are summarised in Fig. 14.

IV - CONCLUSION

It is concluded that a multi-technique approach is necessary to understand the structure of 'aluminised' Inconel. It is considered that this type of approach may well be necessary to understand the near surface structure and behaviour of other technologically important materials.

V - ACKNOWLEDGEMENT

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*More exactly the composition of the A&Cr phase was:

$$ \text{A}^\text{Al}0.23 \text{Cr}0.53 \text{Fe}0.14 \text{Ni}0.06 \text{Mo}0.03 $$
Fig. 1
Optical micrograph.

Fig. 2
EPMA line scans.

Fig. 3
AMPA line scans.
Fig. 4 AMPA electron image.

Fig. 5 AMPA oxygen image.

Fig. 6 AMPA nitrogen image.

Fig. 7 XRD phase analyses and SEM image.
Fig. 8 Unit cell of (AlNi)'' as a function of depth.

Fig. 9 (AlNi)'' texture.

Fig. 10 Digimap showing Nb$_{0.8}$Ti$_{0.2}$C (white) (see note 1).

Fig. 11 AMPA niobium map.

Fig. 12 AMPA carbon map.

(1) Due to the very high price involved, we apologize for reproducing the coloured figures in black and white (The Editors).
Fig. 13 Quantitative EPMA point analyses.

Fig. 14 Summary of results.