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High temperature oxidation protection of titanium and nickel alloys by single-step aluminization plus fluorination

Alexander Donchev, Ali Soleimani-Dorcheh, Mathias Galetz | donchev@dechema.de | AiF | 1st December 2015 – 31st March 2018

Introduction

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- Ti alloys: No use at temperatures > 600°C due to fast growing TiO_2 scale [1]
- Ni alloys: Chromia evaporation at temperatures above 1000°C and even below in the presence of water vapour [2]
- Solution: Protective alumina layer [3]
- Intermetallic aluminide diffusion layer with fluorine reservoir
- Alumina formation via the fluorine effect [4]

Experimental

2

- Powder pack with fluorine containing activator (AlF_3 or NH_5F_2)
- Oxidation testing (isothermal and cyclic)
- Post experimental investigations (X-ray diffraction (XRD), metallographic preparation, light optical microscopy (LOM), scanning electron microscopy with coupled energy dispersive X-ray spectroscopy (SEM/EDX) and electron probe micro analysis (EPMA)

Results - 1

3

Powder Pack

1. Ti alloys:
   - activator AlF_3
   - Temperature 600 - 900°C ($T_p < T_\beta < T_p$) T_β = β-transus
   - Variation Al/F content (1/1 – 20/1)
   - Alloys α-Ti, Ti6242 (α+β), β-21S, Ti-15Mo (β)
   - $TiAl_3$ layer (< 5 µm) with F
2. Ni alloys
   - activator NH_5F_2
   - Temperature 800 - 900°C
   - Variation Al/F content (1/1 – 20/1)
   - IN 600, IN 617, IN 625
   - Uniform Ni_2Al_3 layer (ca. 10 µm) but only minor F

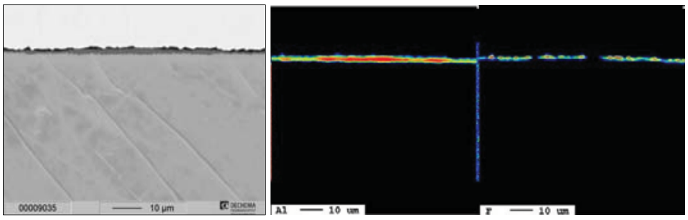

Figure 1a-c: EPMA/BSE image (a) and elemental distribution images of Al (b) and F (c) of a α-Ti sample after pack

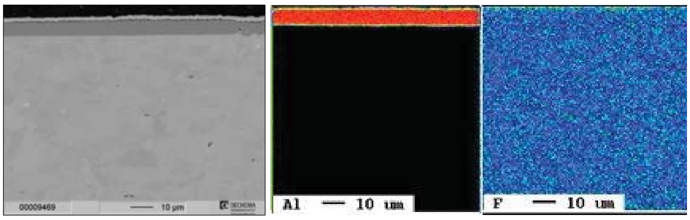

Figure 2a-c: EPMA/BSE image (a) and elemental distribution images Al (b) and F (c) of a IN 625 sample after pack

Results - 2

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Oxidation

1. Ti alloys
   - Protective alumina formation
   - Aluminide diffusion zone intact ($TiAl_3$)
   - Fluorine reservoir underneath alumina
   - Interdiffusion of Al ca. 7 µm
2. Ni alloys
   - Alumina formation
   - Transformation to Ni_3Al layer (ca. 15 µm)
   - No detectable fluorine reservoir
   - Formation of intermetallic Cr-containing phases
   - Interdiffusion of Al into the substrate (ca. 30 µm)

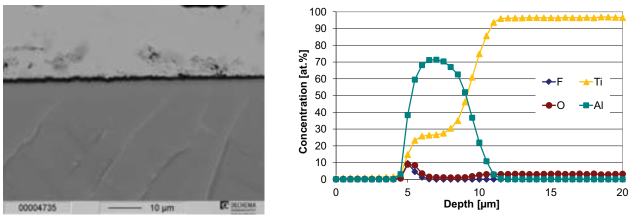

Figure 3a, b: EPMA/BSE image (a) and depth profiles (b) of Al, Ti, O and F on an α-Ti sample after oxidation (600°C/120h/air)

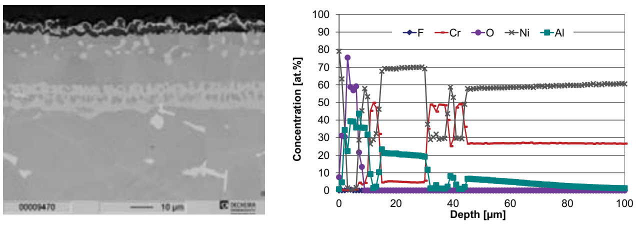

Figure 4a, b: EPMA/BSE image (a) and depth profiles (b) of Al, Cr, Ni, O and F on an IN 625 sample after oxidation (1000°C/100h/air)

Conclusions

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1. Ti
   - Aluminide diffusion layer with F on α-Ti
   - Protective α-alumina after oxidation
2. Ni
   - Aluminide layer but no F detectable
   - Alumina after oxidation

Outlook

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1. Investigations of the technical Ti alloys
2. Optimization of the pack for Ni alloys to ensure the fluorine effect
3. Cyclic oxidation testing for treated Ti- and Ni-specimens

Literature

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[1] F.H. Froes “Titanium” ASM International, Materials Park 2015

[2] H. Asteman et al., Ox. Metals 57(2002)193

[3] P. Kofstad, Mat. Science Forum 154(1994)99

[4] A. Donchev et al, Intermetallics 11(2003)387

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