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# Deposition of Cr-modified silicide coatings on Nb–Si system intermetallics

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#### Abstract

In order to improve the oxidation resistance of Nb–Si system intermetallics, silicide was deposited on the substrate by molten salt or by pack cementation, and Cr was deposited by pack cementation. Phase and microstructure were observed by scanning electro microscope (SEM), X-ray diffraction (XRD) and energy diffusion spectrum (EDS). It was found that phases of NbSi<sub>2</sub>, CrSi<sub>2</sub>, Cr<sub>2</sub>Nb were formed. The result showed that the high temperature oxidation resistance of the Nb–Si system intermetallics will be improved by applying the Cr doped Si coatings. Maybe it was attributed by the formation of SiO<sub>2</sub> and Cr<sub>2</sub>O<sub>3</sub> which could prevent the penetration of oxygen into the inner coatings and the substrate. © 2006 Elsevier Ltd. All rights reserved.

Keywords: B. Diffusion; B. Oxidation; C. Coatings, intermetallic and otherwise; C. Vapour deposition; D. Microstructure

### 1. Introduction

Nb–Si system intermetallics have great potential as high temperature structural materials because of their low density, high melting points and high strength at high temperatures. Nb–Si system intermetallics must be the better choice than most of the alloys. However, the oxidation resistance of Nb–Si system intermetallics is extremely poor at high temperatures [1-3]. In order to use this kind of intermetallics in air at high temperatures, they are modified by elements such as Hf, Cr and so on. Even though, the oxidation resistance was not so satisfied as hoped. Then there must be coatings on them.

To achieve better oxidation resistance, silicon was filtered onto the substrate by molten salt, by pack cementation and by other methods. So there is NbSi<sub>2</sub> coating at the surface [4]. NbSi<sub>2</sub> is a kind of intermetallics. When it was heated in the open air at high temperatures, the protective coating SiO<sub>2</sub> was formed. SiO<sub>2</sub> is a glassy substance and it will stop the oxygen coming further into the substrate. And the oxidation

resistance was really enhanced, proved by the oxidation experiment. Unfortunately,  $SiO_2$  layers were consumed heavily when heated in the open air for a long time. The reasons of which may be, first of all,  $SiO_2$  layers were volatilized in the very high temperature region; second, silicon was diffused into the substrate when heated for a long time. So chromium was doped into the NbSi<sub>2</sub> coatings by pack cementation [5]. It was suspected that after the deposition of Cr and Si, both NbSi<sub>2</sub> and Cr<sub>2</sub>Nb will be made. When it was tested in the oxidation experiment SiO<sub>2</sub> and Cr<sub>2</sub>O<sub>3</sub> will be formed. Both of them will protect the diffusion of the oxygen into the inner coating and the substrate.

#### 2. Experimental procedures

The alloys included high purity niobium, titanium, silicon, chromium, alumina, and hafnium and were prepared by arc melting. The specimens with dimension of  $\Phi$  13 mm × 2 mm were spark-cut from ingots, then ground and cleaned ultrasonically in acetone. The nominal composition of the alloy is 24Ti-16Si-6Cr-6Al-2Hf.

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Fig. 1. Cross-sectional SEM image of the niobium alloy with Cr, Si co-deposition coating at 1100 °C heated for (a) 8 h and (b) 16 h under the argon field.

The halide pack cementation was used to deposit the chromium onto the substrate and NbSi<sub>2</sub> coating. The components for the pack cementation process consisting of Cr or Si powder, halide activator (AlF<sub>3</sub> or NH<sub>4</sub>Cl), and inert filter (Al<sub>2</sub>O<sub>3</sub> powder) were weighed, mixed and ground smaller than 75  $\mu$ m. The process is listed in Table 1. The samples and the mixtures were all put into the Al<sub>2</sub>O<sub>3</sub> crucibles and heated under the argon shield [6].

After diffusion, the samples were cleaned ultrasonically and the packing inert masses were removed gently from the surface of the samples. The samples were examined by scanning electron microscopy (SEM), X-ray diffraction (XRD) and energy dispersive spectrum (EDS).

# 3. Results and discussion

## 3.1. Cr, Si co-deposition

Fig. 1 shows the cross-sectional SEM images of the Cr, Si co-deposition coating formed on Nb–Si system intermetallics

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