

## Chemical and Morphological Changes at $\text{Al}_2\text{O}_3/\text{NiAl}$ Interfaces and Their Relationship to Scale Adhesion

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The segregation of indigenous sulfur impurity from an alloy to the  $\text{Al}_2\text{O}_3$  scale/alloy interface during high temperature oxidation is often considered as the major cause that weakens the interface<sup>1,2</sup>. Systematic studies of the chemical changes at  $\text{Al}_2\text{O}_3$ /alloy interfaces have in recent years been carried out for  $\text{FeCrAl}$ <sup>3</sup>,  $\text{Fe}_3\text{Al}$  and  $\text{FeAl}$ <sup>4</sup>. Although sulfur was the major segregant in all cases, the segregation behavior varied significantly with different alloys and differed from surface segregation, showing a possible dependence on interface microstructure. Whether similar behavior on  $\text{FeAl}$  can be expected for  $\text{NiAl}$  is unknown. Despite its practical importance, the compositions at growing  $\text{Al}_2\text{O}_3/\text{NiAl}$  alloys have never been studied. The purpose of this work is to study the chemical changes at  $\text{Al}_2\text{O}_3/\text{NiAl}$  interfaces as oxidation proceeds, and compare that to the interface morphology and interfacial strength. This paper summarizes initial results on these studies performed on several different  $\text{NiAl}$  alloys with slightly different purity.

$\text{Ni-(40-50)at\%Al}$  alloys with different C and S contents were oxidized at  $1000^\circ\text{C}$  or  $1150^\circ\text{C}$  for various times in oxygen. To study the interface chemistry, the oxidized specimen was placed in the ultra high vacuum of a scanning Auger microscope. The surface of the specimen was scratched with a diamond stylus to cause spalling of the scale adjacent to the scratch. This exposes the underlying alloy surface, which can be examined using an Auger probe. The underside of the oxide was also studied on scale pieces that flipped over during the spalling process. Images from secondary electrons were used to distinguish features at the alloy surfaces. These usually include faceted voids and scale imprinted areas. Several different areas covering more than one alloy grains were often examined. Surveys were performed on many similar features in order to attain a statistical analysis of the results. Spallation resistance of the scale during cooling and scratching were used to evaluate the relative interfacial strength.

Results show that unlike  $\text{FeAl}$ ,  $\text{FeCrAl}$  or  $\text{NiCrAl}$ , sulfur did not segregate to any features at the  $\text{Al}_2\text{O}_3/\text{NiAl}$  interface until extended oxidation times. This is because the amount of S impurity in  $\text{NiAl}$  is much lower than in the Fe-based alloys or in  $\text{NiCrAl}$ . Given enough time, the small amount of S

in the alloy will segregate, but only to void faces, not oxide/alloy interfaces where the scale was in contact with the alloy. The time for S to saturate at the void surfaces calculated from the S concentration in the alloy and approximate S diffusivities in Ni-based alloys agree with the results. Figure 1(a) shows the appearance of a typical interfacial area with faceted voids. Fig. 1(b) is the corresponding Auger S map of this area. The amount of S on void faces varied, showing strong segregation anisotropy. Even though S was abundant on void faces, none could be detected at the interfaces between the voids. Very small amounts of P and Cl were sometimes found on these interfaces.

Before sulfur from the alloy segregated to the void surface, carbon was the major segregant. It covered the void faces the same ways sulfur did, but was also absent from the interface. With time, S replaced the carbon. One of the voids shown in Fig. 1 (marked by X) is an example where the carbon content was still high after S had segregated to most of the void faces.

These segregation results found on  $\text{NiAl}$  are compared with earlier ones obtained for  $\text{FeAl}$ . Spallation resistance is discussed in light of the chemical and morphological changes at these interfaces.

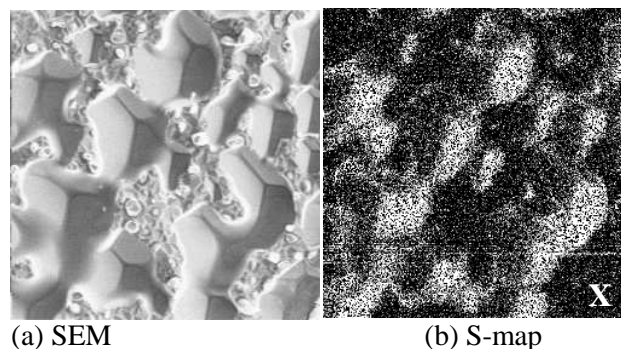


Figure 1: (a) SEM image of a  $\text{NiAl}$  surface after removal of the scale that was formed at  $1150^\circ\text{C}$  for 100 hours. (b) Corresponding Auger map of sulfur. The area marked "X" is rich in carbon.

<sup>1</sup> A. W. Funkenbusch, J. G. Smeggil, and N. S. Bornstein, *Metall. Trans. A*, **16A**, 1164 (1985).

<sup>2</sup> J. L. Smialek, *Metall. Trans.* **22A**, 739 (1991).

<sup>3</sup> P. Y. Hou, *Mater. and Corr.*, **51**, 329 (2000).

<sup>4</sup> P. Y. Hou, *Mater. Sci. Forum*, **369-372**, 23 (2001).