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Gas-phase oxidation processes

SOLID-PHASE OXIDATION PROCESSES – HIGH TEMPERATURE OXIDATION RESISTANCE OF Ni-BASED ALLOYS

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ABSTRACT

Nickel-based superalloys are group of alloys dedicated for energy saving at high temperature applications. Oxidation has been considered as the most significant degradation mode of the alloy when they are used at high temperatures. Aluminum and chromium have been widely involved in the alloys to provide protective oxides of Al_2O_3 and Cr_2O_3 as they grow relatively slow. However, spallation of the protective scales might occur when the components experience thermal cyclic. Increasing the oxidation resistance of Ni-based alloys has been done by doping of yttrium and hafnium. However, investigations of the effect of germanium and zirconium on the oxidation behaviour of nickel-based alloys are quite few and the mechanisms have not been well explained. This study investigated the cyclic oxidation behaviour of Ge and Zr additions on Ni–9.6Cr–5.6Al–2.4Ti alloy at 900, 1000 and 1100°C. It was found that while Zr improved the cyclic oxidation resistance of the alloy it gave higher oxidation rates. Meanwhile, Ge addition improved the oxidation resistance of the alloys by decreasing the oxidation rate as well as improving the spalling resistance.

Keywords: nickel-based superalloys, cyclic oxidation, high temperature oxidation, effect of Ge and Zr.

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AIMS AND BACKGROUND

Components used at high temperatures, such as those found in boiler as well as gas turbine engines, require materials with combination of mechanical properties and surface stability at relatively high temperatures[1]. Development of high temperature alloys is mostly driven by the requirement to increase energy efficiency of such engines. Ni-based superalloy is a group of materials that are designed to have good fracture toughness, creep and fatigue properties, and resistance to high-temperature oxidation and hot corrosion[2,3]. The development of Ni-based superalloys has been based primarily on the need to have alloys with high-temperature strength. The addition of relatively small amounts of Al and Ti in a binary Ni-Cr alloys increased the strength of the materials at high temperatures owing to the formation of coherent precipitates of the ordered intermetallic γ'-Ni_3 (Al, Ti) (Ref. 4). This phase has unique mechanical properties in which the strength increases with temperature up to about 900°C. To provide materials with a high strength level at high temperatures, it has been found that the volume fraction of the γ' precipitates needs to be increased to a value of up to 70% (Ref. 5), and this is done by increasing the amount of Al and Ti in the alloys.

Cr, which is mostly dissolved in the γ-Ni matrix of nickel-based alloys, provides resistance to high-temperature oxidation and hot corrosion due to formation of a protective scale consisting relatively slow-growing oxide of Cr_2O_3 (Refs 6 and 7). The addition of Al in the alloys can also improve high-temperature oxidation resistance, since this element forms other protective scale of Al_2O_3, which is thermodynamically more stable than Cr_2O_3 (Ref. 8). However, the primary reason for the addition of Al in nickel alloys is to provide strength. Moreover, the maximum chemical content of Al necessary to optimise strength in these alloys is considered too low to produce the protective scale, Al_2O_3.

In high temperature applications involving cyclic stresses, the scales might easily crack and spall, resulting in shifting of the kinetic types of oxidation from parabolic type of oxidation behaviour to linear type indicating higher rate of oxidation. To improve the spalling resistance during cyclic oxidation of Ni-based superalloys, elements such as Y, Hf, Ce and Zr have been involved in relatively few amounts and the mechanisms of adherence of the oxides on the alloy surface has been proposed[9–11]. Slight amount addition of Zr increased the adherence of Al_2O_3 and improves the oxidation resistance of the alloy[12]. Nevertheless, relatively large amount of Zr eliminate the protective effect of alumina and consequently reduce the oxidation resistance. Addition of Zr up to 0.9 wt.% in iron aluminide alloys improved the oxidation behaviour of the alloy[12].

Ge has been employed in Si–Ge alloy that intensively used as semiconductor in electronic devices[13]. Oxidation behaviour in this system has been investigated and it is normally accepted that germanium increases the oxidation rate in this system. For example, silicon-germanium alloys with 28 and 36% of Ge oxidised at 700°C showed that mixed of GeO_2 and SiO_2 in the scales formed with the higher rate in higher Ge

content[13]. However, there have been reported that addition of Ge, in relatively small amount, in some alloy systems could reduce the oxidation rate and therefore improve oxidation resistance of the alloys[14,15]. Addition of small amount Ge in silicide coatings improved isothermal and cyclic oxidation of Ti alloys through retarding the cracking and spalling of protective oxides[14]. Moreover, it was also found that addition of Ge decreased the oxidation resistance of near-α Ti alloy due to the formation of Ge rich layer on the interface of substrate-oxide layer[14]. The formation of Ge rich layer was believed due to the low solubility of Ge in TiO_2. Increasing the oxidation resistance obtained by small amount addition of Ge has also been found in the NiTi alloys oxidised at 800, 900 and 1000°C. In this study it was revealed that the highest oxidation resistance was found in alloys with 1.5% Ge addition[15]. In Pb free Sn – 0.7Cu solder, trace element of Ge was also reported improved the oxidation resistance of the alloy[16]. Furthermore, other study reported that in a nuclear reactor fuel cladding material of Zr–Nb–Mo–Ge alloy, the addition of Ge was found can increase the oxidation resistance of the material[17]. In Cr-based high temperature alloys of Cr–Cr_3Si system, addition of up to 2% Ge element has showed affect the spalling resistance of the scales at 1350°C (Ref. 18).

Among the rare elements, the effect of small amount addition of Ge on the oxidation behaviour of Ni-based superalloys has not been studied. This paper reports the investigations results on the effect of Ge and Zr addition on the cyclic oxidation behaviour of Ni–9.6Cr–5.6Al–2.4Ti model alloy at temperatures 900, 1000 and 1100°C. These results provide scientific consideration when Zr and Ge are involved to in nickel-based superalloys to improve the oxidation resistance at high temperature applications, such as in turbine blades of land based as well as aircraft gas turbine engines.

EXPERIMENTAL

Three Ni–9.6Cr–5.6Al–2.4Ti model alloys with nominal chemical compositions shown in Table 1 were melted in a single-arc furnace purged with high purity argon using pure element materials obtained from Sigma Aldrich. Prior to melting, the furnace chamber was purged with argon for 20 min and kept flowing during the melting. To ensure homogeneity, each alloy was melted four times. The alloy buttons were then homogenised at 1150°C for 100 h in a tube furnace in which argon was introduced to avoid from oxidation. After quenching in water, the alloys were then aged at 750°C for 2 h followed by air cooling. The as-aged model alloy buttons were sectioned to obtain 2.0 mm thick coupons with a single-side surface area of approximately 6 × 8 mm^2. Prior to the cyclic oxidation tests, the samples were ground to a 1200-grit finish using silicon carbide papers, and then ultrasonically cleaned in acetone followed by weighing of the clean samples. Polished samples of 1 μm finish were etched using chromic acid solution containing 50 ml H_2O, 150 ml HCl and 25 g CrO_3 for 10 s.

The microstructures of the model alloys were then observed under scanning electron microscope (SEM).

Table 1. Chemical composition of the alloys

Elements	Chemical composition (wt.%)			
	alloy 1	alloy 2	alloy 3	alloy 4
Ni	82.35	81.35	81.35	80.35
Al	5.60	5.60	5.60	5.60
Cr	9.60	9.60	9.60	9.60
Ti	2.40	2.40	2.40	2.40
C	0.05	0.05	0.05	0.05
Zr	0	1	0	1
Ge	0	0	1	1

Cyclic oxidation tests were conducted by heating the samples in a vertical tube furnace at three different temperatures of 900, 1000 and 1100°C up to 20 cycles. Each cycle represents heating the samples for 50 h followed by cooling to room temperature for 30 min. The weight change for each sample at each cycle was measured and plotted in curves of $\Delta W/A_0$ versus cycle number, where ΔW and A_0, respectively, represent weight change and original total surface area of each sample. X-ray diffraction (XRD) analysis was used to identify the oxides existed on the samples after experienced 20 cycles of oxidation tests. The microstructures of the oxidised samples were observed under a scanning electron microscope (SEM), in which an energy dispersive spectroscopy (EDX) facility was attached. Chemical mappings were conducted to distinguish the elemental distribution in the scales.

RESULTS AND DISCUSSION

It was found that all model alloys had the same microstructures, as addition of either Zr or Ge of only 1% is predicted did not affect the microstructures of different model alloys. Figure 1 shows the microstructure of typically model alloys, consisting of γ-solid solution matrix and ordered intermetallic coherent precipitate of γ′-Ni_3 (Al, Ti). Two types of γ′, namely primary γ′ and secondary γ′ were found in all model alloys. This microstructure is essentially similar with that normally obtained in commercial nickel-based superalloys. However, no carbides were found in these model alloys as no carbon was added in the model alloys. The size of primary γ′ was always larger than that of secondary γ′ as the primary γ′ formed at higher temperatures that provide higher activation energy for elements to diffuse much faster during nucleation and growth of the primary γ′.

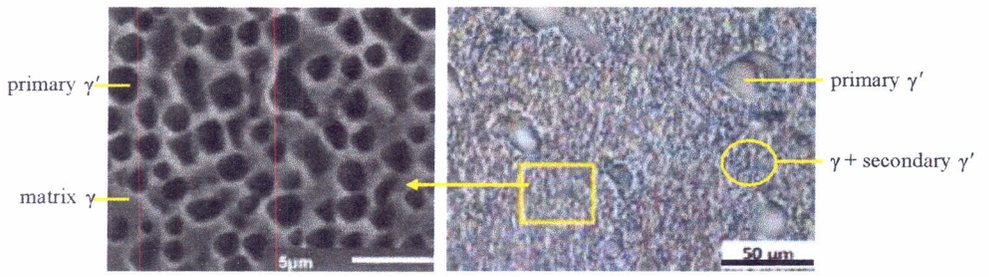

Fig. 1. Scanning and optical micrographs of typical microstructures of the model alloys

Figures 2, 3 and 4 provide the data of weight change during oxidation of model alloys for cycles up to 20 cycles at different temperatures. It is seen that most weight change is positive, indicating the dominance of oxide growth if compared with spalling of the scale. The characterisation results of the scales left on the surface of the samples after cyclic oxidation test for 20 cycles at 1100°C using XRD analysis, depicted in Figs 5–8, show various oxides. As higher temperature application is usually concerned, only XRD spectra of the sample oxidised at 1100°C are presented in the paper. Protective oxide of Al_2O_3 is mostly observed. However, unprotective TiO_2 occurred in addition of Cr_2O_3 and spinnels $NiCr_2O_4$.

Mappings of elements on the cross-sections of samples have been conducted to identify the distribution of oxides in the scales as well as in sub-scales. Most attention is addressed on samples of alloys 2, 3 and 4 after oxidised cyclically at 1100°C, where Ge and Zr are involved. The X-ray mapping results of these three alloys were depicted in Figs 9, 10 and 11, respectively. It is seen that in alloy 2, the outer part of the scale consists spinel of $NiCr_2O_4$. TiO_2 located underneath of this spinel, while Al_2O_3 found on the interface between scale and substrate as well as distributed as particles in the outer part of the scale. Zirconium was preferably distributed as ZrO_2 along the grain boundary of substrate that connected with the scale. Nevertheless, some particles of ZrO_2 were also found distributed in outer part of the scale.

In alloy model 3, the outer part of the scale is dominated by TiO_2, below which chromium oxide as Cr_2O_3, was found as compact layer. However, Al_2O_3 mostly formed as internal oxides distributed underneath of the scale. Germanium was found distributed homogeneously in the scale as well as substrate. In alloy model 4, where Ge and Zr were added, the outer part of the scale was dominated by Cr_2O_3 and $NiCr_2O_4$. A layer consisting of Al_2O_3 and TiO_2 found underneath of this Cr rich scale. Similar with that occurred in alloy model 3, Ge distributed homogeneously in the scale and substrate. However, higher distribution of Zr was found in the scale indicated the formation of Zr_2O_3 in the scale.

Oxidation of all model alloys resulted in the weight change due to two main contributions, i.e. pick-up of oxygen during oxidation which give weight gain, and oxide spalling which give weight loss. In total, the weight change (ΔW) in mg represents the different between sample weight after certain cycle and original weight of the sample

before oxidation was performed. Meanwhile, A_0 is the surface area of the original sample measured in cm^2. Figures 2–4 show the relation between cycle number and $\Delta W/A_0$ at three different temperatures for 4 model alloys. Negative values of $\Delta W/A_0$ indicate loss of protective scale due to spallation.

As shown in Fig. 2, at 900°C, only Zr-modified alloy that had no negative values. All three non-modified, Ge and Ge + Zr modified alloys had negative values of $\Delta W/A_0$ at relatively early cycles up to 6 cycles. At 1000°C, however, the non-modified alloy experienced spallation of its protective scale at early cycles of up to 6 cycles followed by build-up the scale, even though continued spallation occurred at cycle 10. The other alloys principally had higher spalling resistance. Among these three alloys, Zr-modified alloys had higher weight gain and this indicates higher oxidation rate. At 1100°C, all alloys had positive weight gains. However, the Ge-modified alloys showed minimum weight gain and indicated slight spallation. Therefore, it is seen that at all temperatures the weight gain curves of alloy 2 always in highest position, and this indicates that the scale of alloy 2 has highest spallation resistance but it has highest oxidation rate. Addition of Ge in alloy 3 reduced the rate of oxidation as shown that the weight gain curve of alloy 3 is lower compared with that of alloy 1.

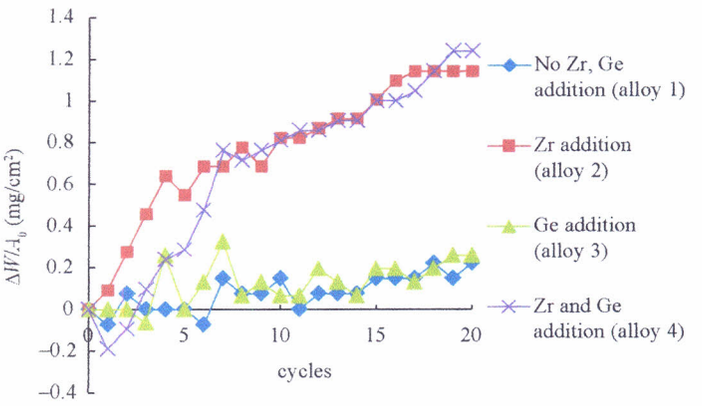

Fig. 2. Weight change of different alloy samples oxidised at 900°C for 20 cycles

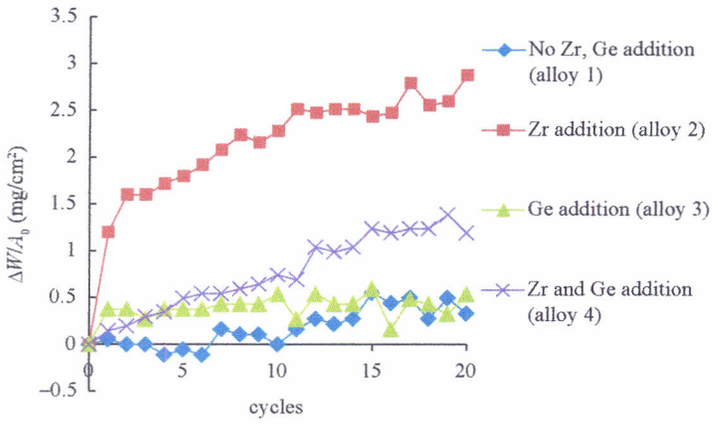

Fig. 3. Weight change of different alloy samples oxidised at 1000 °C for 20 cycles

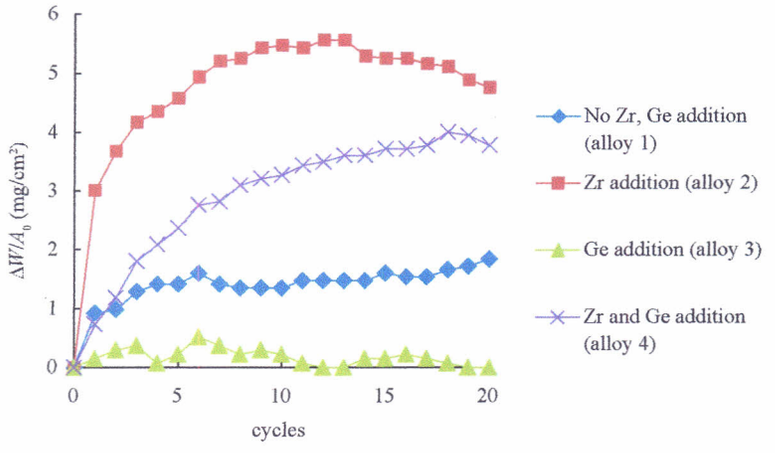

Fig. 4. Weight change of different alloy samples oxidised at 1100°C for 20 cycles

In this paper only XRD results of highest oxidation temperature, i.e. 1100°C, are presented, as shown in Figs 5–8. The scales of all four samples possessed different oxides. Alloy 1 had Al_2O_3, $NiAl_2O_4$ and Cr_2O_3, while alloy 2 contained Al_2O_3, Cr_2O_3, $NiCr_2O_4$ and NiO. Moreover, alloy 3 produced Al_2O_3, $NiAl_2O_4$, Cr_2O_3 and $NiCr_2O_4$, while alloy 4 consisted Al_2O_3, $NiAl_2O_4$, Cr_2O_3, $NiCr_2O_4$, TiO_2, NiO and ZrO_2. The XRD patterns confirm the occurrence of Al_2O_3 at the surface of the scales of all samples. However, two types of spinels $NiAl_2O_4$ and $NiCr_2O_4$ were observed as the results of reactions between NiO and Al_2O_3, and NiO and Cr_2O_3, respectively. Meanwhile, NiO has reacted with Cr_2O_3 to form $NiCr_2O_4$ spinel.

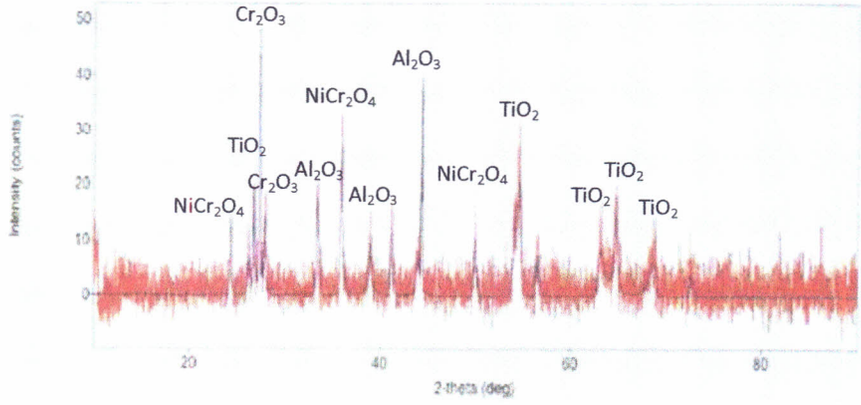

Fig. 5. Difractogram of alloy 1 after heated cyclically at 1100 °C for 20 cycles

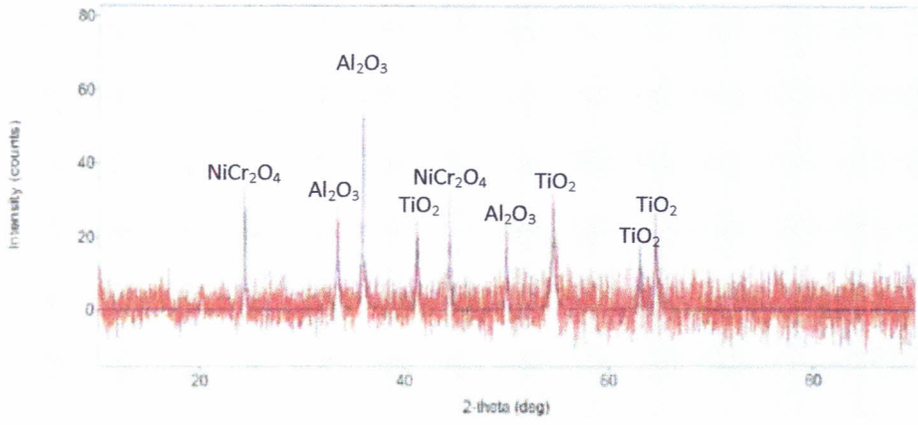

Fig. 6. Difractogram of alloy 2 after heated cyclically at 1100 °C for 20 cycles

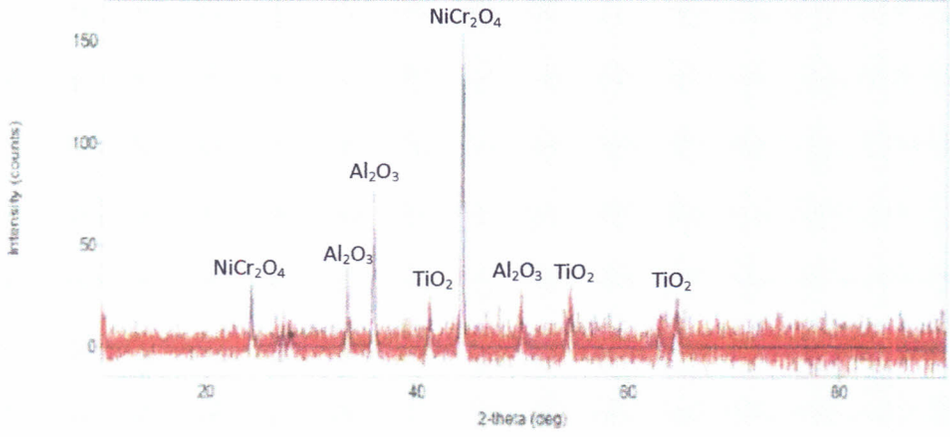

Fig. 7. Difractogram of alloy 3 after heated cyclically at 1100 °C for 20 cycles

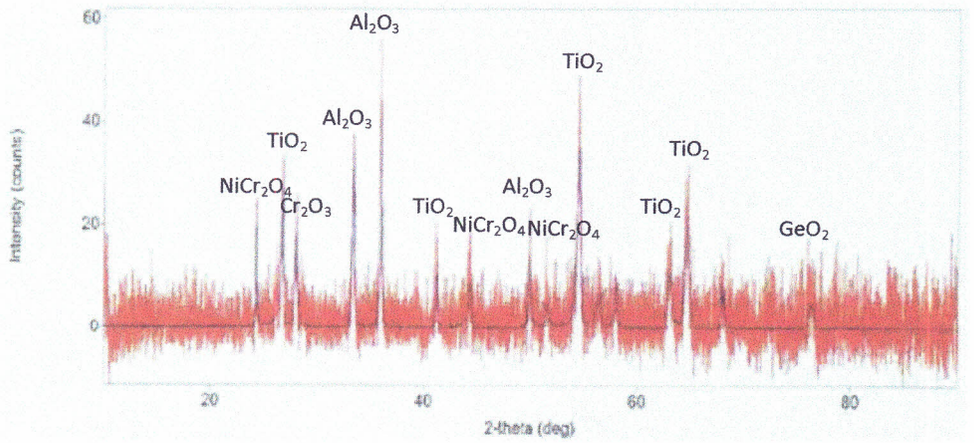

Fig. 8. Difractogram of alloy 4 after heated cyclically at 1100 °C for 20 cycles

The X-ray mapping analysis was conducted to evaluate the distribution of elements in the cross section of the scales, from which distribution of oxides could be expected, as shown in Figs 9, 10 and 11. Only X-ray mapping of alloy 2, 3 and 4 are presented in this paper as these represents addition of Zr and Ge. X-ray mapping for alloy 2 containing Zr after cyclic oxidation at 1100°C for 20 cycles. As shown in Fig. 9, titanium oxide existed at the outer layer of the sample, but at relatively thin of about 5 μ. Chromium oxide, with relatively thick of about 20 μm, formed underneath of the titanium oxide layer. Aluminum oxide occurred as separate particles below chromium oxide layer as the result of internal oxidation.

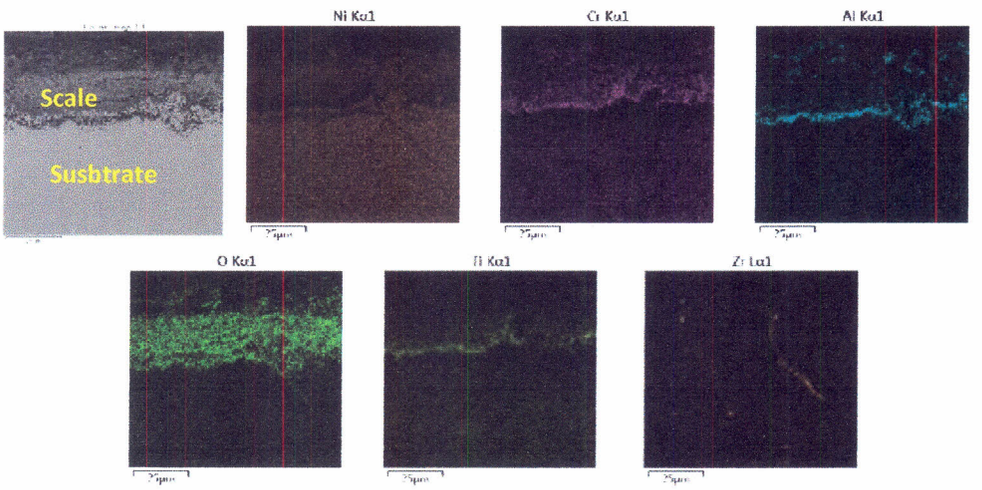

Fig. 9. X-rays mapping of model alloy 2 after cyclic oxidation at 1100°C for 20 cycles

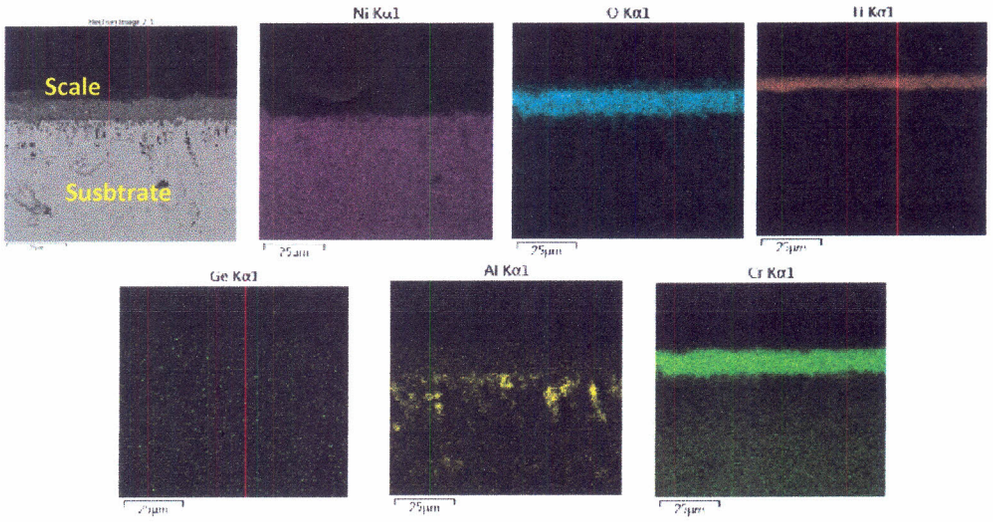

Fig. 10. X-rays mapping of model alloy 3 after cyclic oxidation at 1100°C for 20 cycles

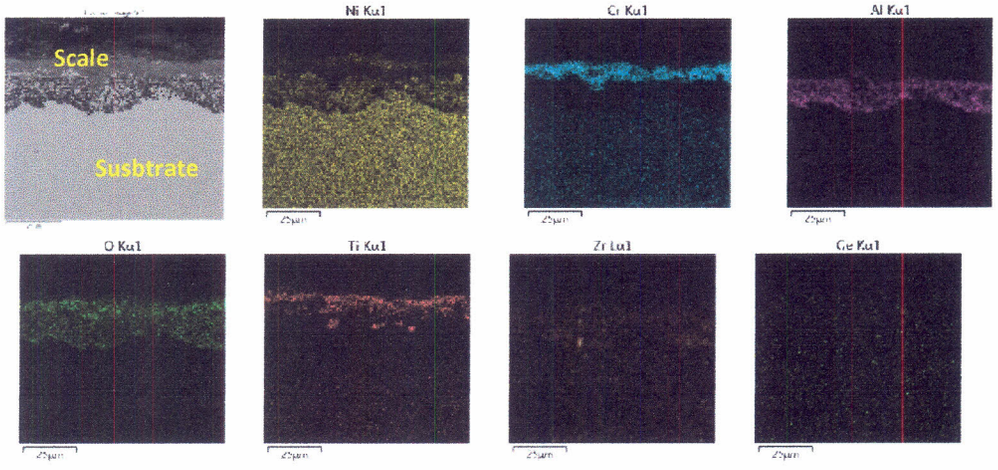

Fig. 11. X-rays mapping of model alloy 4 after cyclic oxidation at 1100°C for 20 cycles

Addition of Zr in model alloy 2 is believed to provide the driving force for formation of aluminium oxide underneath the spinnel of $NiCr_2O_4$. Figures 10 and 11 clearly show that different with that occur in model alloy 2, in model alloy 3 the Al_2O_3 formed as internal oxides. However, relatively compact Al_2O_3 layer developed at lower part of the scale. From the X-ray mapping it is seen that Zr distributed as ZrO_2 in several parts of the scale. Meanwhile, relatively homogeneous distribution of Ge, expected as GeO_2, was found in alloys 2 and 4.

The X-ray mapping on alloy 4 after cyclic oxidation at 1100°C for 20 cycles, depicted in Fig. 11, shows that chromium oxide formed at the outer part of the scale,

while mixture of aluminium and titanium oxides occurred below the chromium oxides layer. However, germanium oxide was detected by XRD on the sample of alloy 3 and was not detected in sample of alloy 4. Thermodynamically, GeO_2 should be formed at 1100°C. However, in the Ellingham-Richardson diagram the position of GeO_2 formation line is slightly above the line for Cr_2O_3 (Ref. 19). While germanium atoms were distributed relatively homogeneously in the scales, zirconium atoms concentrated in certain spots indicated larger particles of ZrO_2 compared with GeO_2.

CONCLUSIONS

Cyclic oxidation of Ni–9.6Cr–5.6Al–2.4Ti model alloys at 900, 1000 and 1100°C have been conducted in atmospheric condition. The scales containing mostly $NiCr_2O_4$ Al_2O_3 and TiO_2 in which ZrO_2 and GeO_2 dispersed inside of the scale. TiO_2 formed on the outer part of the scale, while Al_2O_3 formed underneath of the scale. The study showed that increasing the spalling resistance was obtained when 1% Zr was added. However, higher consumption of metals was found in the alloy containing Zr. It is concluded that alloys with addition of 1% Ge provided relatively better spalling resistance compared with that no Ge addition. However, further experiments are still needed with involvement of Ge and Zr in different chemical composition variations.

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