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# The oxidation of four oxide dispersion strengthened Ni-5Al-xSi-0.2Y<sub>2</sub>O<sub>3</sub> (x = 1, 3, 5, 7 wt.%) alloys in air at 1100 °C



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#### ABSTRACT

The cyclic oxidation behavior of oxide dispersion strengthened Ni-xSi-5Al-0.2Y $_2$ O $_3$  (with x=1,3,5,7 wt.%) superalloys was investigated at 1100 °C for 100 h. The alloys contained 5 wt.% silicon possessed the best oxidation resistance with the formation of continuous external  $\alpha$ -alumina scales developed on the surface without any noticeable internal oxides. Therefore, the addition of silicon decreased the oxidation rate significantly and exerted the third-element effect by reducing the critical Al content needed for the transition from its internal to external oxidation. However, the excessive silicon content (7 wt.%) reduced the spallation resistance of oxide scales.

#### 1. Introduction

With an increasing demand for a promotion of the thermal efficiency of power plants, superalloys have been required to possess extraordinary oxidation resistance at higher temperatures [1,2]. Hightemperature oxidation resistance depends on a continuous, dense, slowgrowing and adherent scale that can significantly slow oxidation rate and prolong service life [3]. However, a protective scale relies on the selective oxidation of Al, Cr, Si and so on [4]. In addition, due to the volatility of chromia scale over 900 °C, alumina and silica scales have been considered more protective oxide scales [5–8]. Alumina with its chemically inert, dense, slowly growing rate and low defect concentration properties, in particular, is thermodynamically the most stable oxide and is able to promote the plasticity and adhesion strength of oxide scales [1,3,9]. However, an external and continuous alumina scale on surface demands a high aluminum concentration that leads to a decrease of mechanical properties [10].

Nevertheless, silicon is known to promote selective oxidation of aluminum and avoid internal alumina [11,12], there is a more general principle, called the third-element effect (TEE), in which an addition of a third element with an oxygen affinity between that of the components of a given binary system can reduce the level of the most reactive element needed to form its external oxide scales [13,14]. Therefore, the principle of TEE is of great significance of design, processing and application of superalloys. Beside of all that mentioned above, the formation of crystalline silica or highly protective amorphous silica as diffusion barrier has been mentioned by many different researchers in

Furthermore, due to an extremely high thermodynamic stability of oxide particles (i.e., Y2O3, ThO2, Al2O3, etc.), oxide dispersion strengthening (ODS) is extensively used to develop high-temperature Ni-based superalloys for industrial gas turbines, aerospace applications, power industries and so on [8,17,18]. It is well-known that ordered  $\gamma$ ' precipitation strengthening is stable only under intermediate temperature due to the dissolution of precipitation at high temperatures. Compared with ODS-free Ni-based superalloys, the control of the inert oxide dispersion is an effective way to improve the high temperature mechanical strength and long-term creep resistance by hindering the dislocation motions [19-22]. On the other hand, the reactive element oxides (RE<sub>x</sub>O<sub>y</sub>) including Y<sub>2</sub>O<sub>3</sub> can generally improve the spallation resistance of oxide scales, which has been often referred to as "reactive element effect (REE)" [23-25]. On the contrary, earlier studies in our group demonstrated that excessive Y2O3 (> 0.6 wt.%) induced segregation had decreased both the oxidation resistance and mechanical properties of alloys [26]. Therefore, 0.2 wt.%  $Y_2O_3$  was introduced into the designed Ni-based superalloys in this study.

Although the system of Ni-Si-Al-Y $_2O_3$  superalloys is now commercially less important, it is still of great importance to understand the oxidation mechanisms and behavior of alloys and has extensive reference value and guidance significance, instead of the complexity of commercial materials. Moreover, it has been imperative to promote the operating temperature of high-temperature materials, and oxidation is the major contributor to the failure of turbine blades when service temperature reaches up to  $1100\,^{\circ}$ C [27]. In addition, the oxidation

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other papers [15,16].

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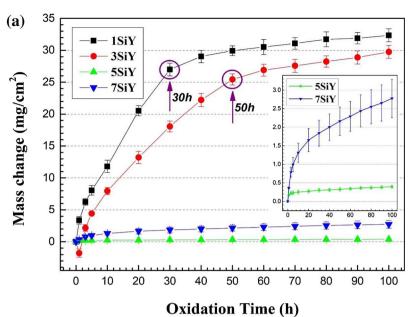
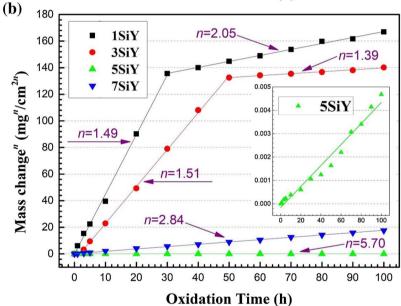


Fig. 1. Oxidation kinetics curves (a) and the corresponding fitted power function curves (b) of 1SiY, 3SiY, 5SiY and 7SiY alloys at 1100 °C in air (mass change was defined as the weight change of only samples without spalled oxides).



behavior of Ni-based superalloys above 1100 °C has been examined less extensively. Hence, in this work, the cyclic oxidation behavior at 1100 °C of four ODS Ni-based superalloys with different silicon content were investigated. The main objective of the present paper is to study the role of silicon on the morphology, composition and formation process of oxide scales during cyclic oxidation.

### 2. Experimental procedure

In this experiment, the four alloy compositions of Ni-xSi-5Al-0.2Y<sub>2</sub>O<sub>3</sub> (with x=1, 3, 5, 7 wt.%, all compositions in wt.%, if not otherwise specified) were respectively named as 1SiY, 3SiY, 5SiY and 7SiY, which were prepared by powder metallurgy technology. The raw materials include nickel powder (diameter 38  $\mu$ m, and purity 99.95 wt.%), silicon powder (diameter 25  $\mu$ m, and purity 99.99 wt.%), aluminum powder (diameter 38  $\mu$ m, and purity 99.95 wt.%) and yttrium oxide particles (diameter 75  $\mu$ m, and purity 99.99 wt.%). And the weight of each powder was measured by electronic balance (0.01 mg precision, Sartorius BT 25 S, German). XQM-2L planetary ball mill grinding was used to achieve mixing for mechanical alloying with stainless steel

containers and stainless steel balls. Ball milling parameters are as follow: rotation speed was 150 r/min, milling time was 10 h, and the mass ratio of stainless balls to powders was 9.8:1. In addition, the containers were filled with high purity argon without any controlling reagent (i.e., ethanol) during the ball milling. After that, the mixed powders were pressed with hydraulic machines and modes, in which the pressure was 550 MPa and the press time was 1 min. Each alloy component has three parallel specimens to be tested for the average weight change and oxidation behavior of alloys. Thereafter, all specimens were sintered in vacuum carbon tube furnace, in which the sintering temperature was 1200 °C and holding time was 120 min. After sintering, relative density of 1SiY, 3SiY, 5SiY and 7SiY measured by an electronic densimeter (GH-128E, China) with the sensitivity of 0.1 mg/ cm<sup>3</sup> were 91.7%, 92.7%, 96.3% and 95.5% respectively. Then, all samples were firstly ground with SiC abrasive papers from No.100 to 2000 in order to obtain the same surface roughness, and cleaned by an ultrasonic cleaner in ethanol before cyclic oxidation test.

Cyclic oxidation test was conducted at  $1100\,^{\circ}\text{C}$  in air by a muffle furnace, each specimen was individually placed in a crucible. Before the test, all crucibles were heated to a constant weight at  $1200\,^{\circ}\text{C}$ , as the

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