EFFECTS OF Si ADDITION ON THE HIGH TEMPERATURE STRENGTH AND OXIDATION BEHAVIOR OF γ' -BEARING Co-BASED SUPERALLOYS

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Abstract

This article aims to present the effect of Si addition on the microstructure, elemental partitioning, high temperature hardness, and oxidation behavior for γ' -bearing Co-based superalloy. The fraction of γ' phase can be increased with the addition of Si, rendering an improvement on hardness at elevated temperatures. Furthermore, Si partitions preferentially toward γ' phase and affects partitioning behavior of W. Isothermal oxidation tests conducted at 900 °C, 1000 °C, and 1150 °C have indicated that Si additions can improve oxidation resistance. Furthermore, the content of W can have a profound impact on the oxidation behavior; low content of W can reder the formation of protective Al_2O_3 at temperatures at and above 1000 °C, while high W content can slow down oxidation at 900 °C.

Introduction

The thermal efficiency of gas turbine engines depends on the temperature capability of materials used in the turbine section [1]. Most of the turbine section components are made of Ni-based superalloys due to their ability to precipitate high temperature stable γ' or γ'' [2, 3]. In 2006, a new type of Co-based superalloy containing γ' phase with a chemical formula of $\text{Co}_3(\text{Al}, \text{W})$ was reported [4]; it contains L_{12} γ' microstructure and offers potential to replace conventional Ni-based superalloys. Many studies have focused on alloy design to improve mechanical properties [4-8] and to raise γ' solvus temperature [7, 9-11]. With respect of the surface stability, studies have shown that Cr addition could provide protection by forming Cr_2O_3 at 800 °C, however, it also destabilizes the cuboidal morphology of γ' and lead to degradation in mechanical properties [12]. Since Cr_2O_3 becomes less protective at temperatures above 1000 °C [2, 13]. The formation of Al_2O_3 is needed for higher temperature applications [14]. Previous studies have indicated that Si additions can promote the formation of Al_2O_3 and improve the adhesion between Al_2O_3 and the substrate of Ni-based superalloys [15-18]. Similar concepts have been exploited by Klein et al. [5] on some other Ni-bearing Co-based superalloys; although oxidation resistance can be improved, the formation of Al_2O_3 does not seem to be dense and continuous in previous studies.

Although it has been reported that Si additions can improve the oxidation resistance of Co-based superalloys by promoting formation of Al_2O_3 , very little is known about its effect on elemental partitioning and lattice misfit that can have a profound impact the mechanical properties at elevated temperatures. Since Si-bearing Co-based superalloys appear to be a promising direction for future alloy design, the purpose of this research is to uncover the effects of Si on elemental partitioning, microstructure and hardness, as well as oxidation behavior.

Experimental procedure

Compositions of interests are shown in Table 1; alloys are based on the Co-Al-W-Ni-Si alloy systems containing 0 – 2.0 at% Si; FC-2.0 and CS-4 both contain 2.0 at% Si, and the W content is reduced by half to 5.0 at% in CS-4. A total of 40 g of raw materials were prepared by vacuum arc melting for each alloy; ingots were melted four times to ensure uniform compositions. γ' solvus was analyzed by differential scanning calorimetry (NETZSCH DSC 404) at a heating rate of 10 °C.min⁻¹ and then cooled at a cooling rate of 20 °C.min⁻¹. As-cast ingots were sealed in quartz tubes with vacuum atmosphere, and then were subjected to solution heat treatment at 1250 °C for 20 h, followed by ageing below the γ' solvus at 900 °C for 168 h. Samples were ground, polished and etched with 100 ml distilled water, 100 ml HCl and 0.5 g $\text{K}_2\text{S}_2\text{O}_5$ for 40 s. Microstructures were observed by scanning electron microscope (Hitachi SU8010) at a voltage of 15 kV. The compositions of phases were determined by the electron probe microanalyzer (EPMA, JEOL JXA-8500) coupled with wavelength dispersive spectroscopy (WDS) operated at a voltage of 12 kV. γ' particles were coarsened for reliable composition measurements, and details of the experimental procedure is described in previous work [15]. Phase identification and lattice misfits were determined by X-ray diffractometer (XRD, Shimadzu XRD-6000) under radiation conditions of 30 kV, 20 mA, Cu target ($\text{K}\alpha$ radiation, 1.54056 Å) with scanning speed of 2 °.min⁻¹. The lattice parameters of γ and γ' phases were determined by deconvoluting (200) diffraction peak into γ and γ' sub-peaks to calculate the lattice misfit $\delta = 2(a_{\gamma'} - a_{\gamma})/(a_{\gamma'} + a_{\gamma})$, where $a_{\gamma'}$ and a_{γ} are lattice parameters of γ' and γ , respectively. Hardness of samples were measured by a high temperature Vickers hardness tester (Model AVK-HF, Akashi) equipped with a vacuum chamber and a heating unit. All specimens were tested from room temperature up to 1027 °C under the argon. A load of 49 N and a dwell time of 15 s was used. The heating rate of the sample was 10 °C.min⁻¹ and 10 indentations were made to get an average value. The indentor material was diamond, and indentation positions were all within individual grains. All specimen surfaces were ground by 1200-grit sand paper and then ultrasonically cleaned in ethanol and dried before experiments. Isothermal oxidation tests were carried out at 900 °C, 1000 °C and 1150 °C for 5, 48, 96, 146 h in a box furnace. At each time interval, samples were removed from the furnace and the weight of oxidized samples were measured by electronic weighting balance with sensitivity down to 10⁻⁵ g, and weight gains were determined by subtracting the weight of the original sample from the weight of the oxidized sample.

Results and Discussion

Figure 1 shows the as-cast, solution heat treated and aged microstructures of FC-0.0, FC-1.0, FC-2.0 and CS-4 alloys. For Si

contents higher than 2.0 at%, μ phase has been identified and cannot be re-solutioned after heat-treatment [19]. The as-cast microstructure contains dendrites, and residual segregation can be eliminated after solution heat treatment. Ordered γ' precipitates are present in FC alloys and CS-4 after solution and ageing heat treatment, and they appear to be homogeneously distributed in γ matrix.

Table I. Nominal compositions (at%)

Element	Co	Ni	Si	Al	W
FC-0.0	Bal.	30	0.0	10	10
FC-0.5	Bal.	30	0.5	10	10
FC-1.0	Bal.	30	1.0	10	10
FC-1.5	Bal.	30	1.5	10	10
FC-2.0	Bal.	30	2.0	10	10
CS-4	Bal.	30	2.0	10	5.0

To understand the effect of Si on microstructures, the elemental partitioning coefficients, K , are obtained. $K = C_{\gamma'}/C_{\gamma}$, where $C_{\gamma'}$ is the concentration of element in γ' , and C_{γ} is the concentration of element in γ . Element with $K > 1$ corresponds to partitioning toward γ' phase, whereas $K < 1$ represents partitioning to γ phase. Particle-coarsening method was employed, Figure 2, and results are summarized in Table 2 and Figure 3; it indicates that Ni, Al, W and Si partition preferentially to γ' phase; Co partitions preferentially to γ phase. Since Si is a γ' forming element, it is likely that Si prefers Al sites than Co sites in the L1₂. The partition coefficient of W is decreased and that of Al is increased with increasing Si content, so higher Al content and lower W content in γ' can result higher solvus temperature, possibly due to less disordering of the L1₂. The addition of Si can modify the size and volume fraction of γ' significantly, Table II. Comparing to FC-0.0, with 2.0 at% Si addition, the γ' volume fraction of γ' have been increased by 11 %; Si seems to be a strong γ' forming element in the Co-Al-W-Ni system. And the reduction in W has decreased the volume fraction of γ' in CS-4 significantly from 82.6 % to 39.8 %.

Figure 1. As-cast, solution-heat-treated condition (SHT), and SHT+ageing condition for FC-0.0, FC-1.0, FC-2.0, and CS-4.

Fig 2. Microstructure of cellular γ/γ' with composition analyzed by EPMA, (a) FC-0.0 (b) FC-1.0 (c) FC-2.0 (d) CS-4

Table II. Elemental partitioning coefficients, γ' vol% and lattice misfits

	Co	Al	W	Ni	Si	γ' vol.%	δ (%)
FC-0.0	0.78	1.88	1.2	1.14		74.3	0.272
FC-1.0	0.77	1.89	1.14	1.17	1.03	76.8	0.263
FC-2.0	0.77	1.96	1.12	1.17	1.04	82.6	0.246
CS-4	0.8	1.44	1.78	1.17	1.001	39.8	-

Figure 3. Partitioning behaviors of elements in FC-0.0, FC-1.0, FC-2.0 and CS-4.

The lattice misfits between γ and γ' have been decreased from 0.272 % to 0.246 % from FC-0.0 to FC-2.0, Table II. And, it is possible that increase in γ' volume fraction is a result of both increasing Si content and higher K value of Al. The partition behavior of W may also explain the decrease in lattice misfit from FC-0.0 to FC-2.0, since there are more W content in the γ phase with higher content of Si, the lattice parameter of γ can expand more to match that of γ' . For advanced Ni-based superalloys, negative lattice misfit is preferred for the formation of N-raft microstructure, which can hinder dislocation climb effectively.

The effect of Si on the partitioning behavior of W indicates its influence on lattice misfit and role to in-directly alter the mechanical properties of Co-based superalloys. Figure 4 shows the hardness results. Among three FC alloys, FC-0.0 has the highest hardness at room temperature, whereas alloys with higher Si contents are softer, and the decrease of K value of W with higher Si content may be the underlying cause. In other words, as the Si content increases, the concentration of W in γ' would be reduced. As a result, FC-2.0 can be softer than FC-0.0 due to its γ' containing lower amounts of W, which is known to provide solid solution strengthening and may increase the anti-phase-boundary energy. However, FC-2.0 is harder than FC-0.0 and FC-1.0 at higher temperature (1027 °C) owing to higher γ' fraction. For comparisons, data of IN718 have been included (subjected to standard 720 °C 8h + 620 °C 8h ageing). FC alloys exhibit higher hardness throughout all temperatures, especially at elevated temperatures owing to high temperature stable γ' phase. IN718 is strengthened by Ni_3Nb γ'' (DO_{22}), which is stronger than L_{12} γ' , however γ'' is only stable until 650 °C, beyond it, γ'' would transform to δ phase leading to degradation in mechanical property. The reduction in W clearly reduces the strength of CS-4 significantly due to lower volume fraction of γ' . The addition of 2.0 at% Si can increase the volume

fraction and solvus temperature of γ' , so Si addition offers potential to increase strength of advanced Co-base superalloys at elevated temperatures. The effect of Si addition on elemental partitioning behavior such as W may be utilized to design more negative lattice misfit alloys in the future.

The size and volume fractions of γ' for FC-2.0 and CS-4 are 800 nm and 300 nm, 80 % and 30 %, respectively, and the only compositional difference between CS-4 and FC-2.0 is the reduced W content. Results of isothermal oxidation tests conducted at 900 °C, 1000 °C and 1150 °C for all alloys are illustrated in Figure 5(a), 5(b), and 5(c), respectively. At 900 °C, oxidation weight gains for FC alloys decrease with increasing Si content. However, the effect of Si is only observed when Si content is increased from zero to 0.5 at%, and from 0.5 at% to 1.0 at%. Although both CS-4 with FC-2.0 contain 2.0 at% Si, it appears that FC-2.0 exhibits better resistance against oxidation at 900 °C due to higher W content. Interestingly, CS-4 alloy has outperformed those of FC alloys at 1000 °C, and FC alloys exhibit the same trend of oxidation behavior as that shown at 900 °C. The oxidation resistance of CS-4 is significantly better than those of FC alloys at 1150 °C, furthermore, more pronounced effect of Si addition can be seen for FC alloys, as the oxidation resistance does increase with more Si content. Since this oxidation weight gain behavior appears to follow the parabolic-rate law equation [20-22] which is defined by Pilling and Bedworth [23]:

$$\left(\frac{\Delta m}{A}\right)^2 = K_p t + C$$

where Δm is weight gain after a certain time t , A is the surface area of metal, K_p is parabolic rate constant in the unit of $\text{g}^2\text{cm}^{-4}\text{sec}^{-1}$, and C is a constant. The K_p values and weight gain after 146 h are determined and listed in Table IV.

Figure 4. Hardness vs. temperature for FC series alloys, CS-4, and IN718

Higher K_p value implies faster oxidation rate, thus lower resistance against oxidation. At 1150 °C, CS-4 possesses a K_p value, which is several orders of magnitude lower than those of FC alloys; after 146 h, its oxidation weight gain is 6.79 mg.cm⁻² comparing to 110.1 mg.cm⁻² of FC-2.0. After 146 h at 1000 °C, oxidation weight gain of CS-4 and FC-2.0 are 11.7 mg.cm⁻² and 27.5 mg.cm⁻², respectively. At 900 °C, the oxidation weight gain for CS-4 and FC-2.0 are 13.3 mg.cm⁻² and 8.0 mg.cm⁻² after 146 h, respectively. A clear trend has emerged from Figure 5 and Table IV, the effect of Si on oxidation behavior appears to be more pronounced at higher temperatures, and reduced W content coupled with Si addition (CS-4 vs FC-2.0) seems to yield much better resistance against oxidation at higher temperature, Figure 5(c). While higher W content can be beneficial for lower temperature condition, Figure 5(a). Cross-sectional micrographs of oxidized FC-0.0, FC-1.0, FC-2.0 and CS-4 alloys at 1150 °C for 146 h are shown in Figure 6, and the XRD analysis for oxidized CS-4 is shown in Figure 7. The extent of oxidation at 1150 °C can be seen from the thickness of external cobalt oxide, after 146 h, and it is clear that higher Si content can provide better oxidation resistance for FC alloys; FC-2.0 contains a total thickness of 800 µm oxidized layer consisting of external CoO, middle layer of CoAl₂O₄, internal CoWO₄ and discontinuous Al₂O₃. By contrast, the oxide thickness of CS-4 is less than 20 µm, and it possesses a dense and continuous Al₂O₃, which is shown by the EDS analysis and can be detected by XRD; no Si segregation can be observed. The oxide constituents for FC alloys and CS-4 at 1000 °C and 900 °C are the same as that of 1150 °C. Figure 6 shows that at 1000 °C, FC alloys contain external CoO, middle layer of CoAl₂O₄, internal CoWO₄ and discontinuous Al₂O₃,

while CS-4 possesses continuous Al₂O₃ after 146 h and the overall oxide thickness is thinner than those of FC alloys.

The cross sectional micrographs at 900 °C reveal the same type of oxides present in FC alloys after 146 h. The oxidized layer of CS-4 has a continuous Al₂O₃, however, its overall thickness of the oxidized layer is similar to that of FC alloys (Figure 6(g), 6(h)). Initial stage of oxidation for CS-4 at 900 °C and 1000 °C are shown in Figure 6; no protective Al₂O₃ could be observed after 5 h at 900 °C, (Figure 6(c), 6(d)). High temperature alloys usually rely on the formation of Al₂O₃ and Cr₂O₃ for surface protection at elevated temperatures [2]. Protective Cr₂O₃ for alloys is common for application temperatures below 1000 °C, however, for the materials that are expected to be used at higher temperatures, formation of Al₂O₃ would be necessary due to its greater thermal stability and low oxygen permeability. In the previously reported studies [12, 14], the Co-Cr-Al-W systems could form internal continuous Cr₂O₃ or Al₂O₃ layers, however its Co₃(Al, W) is not stable, unless Ni is added in the system in significant quantities, hence the Ni-bearing Co-based systems have been introduced. However, so far there has been no reported Ni-bearing Co-based alloy systems that actually grow continuous Al₂O₃ at temperatures above 1000 °C [12, 14]. To the best of our knowledge, the CS-4 is the first alloy to show continuous Al₂O₃ formation in Ni-bearing Co-based alloys (Co-Al-W-Ni-Si system). Since Si additions can promote Al₂O₃ formation in Ni-based superalloys, it is likely that Si addition can also enhance the Al activity in Co-based superalloys [15-18], promoting more rapid formation of Al₂O₃ to decrease the rate of oxidation, and this effect appears to be more pronounced with increasing temperature from 900 °C to 1150 °C for FC alloys, Figure 5.

Table III. Thermal properties of FC series alloys and CS-4 [24]

°C	FC-0.0	FC-0.5	FC-1.5	FC-2.0	CS-4
Solidus	1443	1430	1400	1387	1363
γ' Solvus	1057	1075	1104	1113	998
HTW	386	355	296	279	366

Table IV. Oxidation rate constant and weight gain after 146 h of investigated alloys [24]

Alloy	900°C		1000°C		1150°C	
	k_p ($g^2cm^{-4}sec^{-1}$)	Weight gain after 146 h (mg/cm ²)	k_p ($g^2cm^{-4}sec^{-1}$)	Weight gain after 146 h (mg/cm ²)	k_p ($g^2cm^{-4}sec^{-1}$)	Weight gain after 146 h (mg/cm ²)
FC-0.0	5.42×10^{-9}	21.7	3.02×10^{-8}	51.8	5.64×10^{-8}	171.6
FC-0.5	2.36×10^{-9}	14.1	1.39×10^{-8}	38.7	5.23×10^{-8}	165.2
FC-1.0	8.43×10^{-10}	7.8	5.69×10^{-9}	23.6	2.99×10^{-8}	125.0
FC-1.5	7.23×10^{-10}	7.6	2.67×10^{-9}	24.9	2.81×10^{-8}	121.4
FC-2.0	7.89×10^{-10}	8.0	6.48×10^{-9}	27.5	2.30×10^{-8}	110.1
CS-4	2.1×10^{-9}	13.3	2.56×10^{-10}	11.7	9.16×10^{-11}	6.79

Figure 5. Weight gain of alloys after (a) 900°C, (b) 1000°C, (c) 1150°C isothermal oxidation test [24].

°C	h	FC-0.0	FC-1.0	FC-2.0	CS-4
900	5
	146
1000	146
	146

Figure 6. Cross-section of oxidized FC-0.0, FC-1.0, FC-2.0, and CS-4 at 900°C, 1000°C, and 1150°C isothermal oxidation test [24].

Figure 7. XRD analysis of CS-4 after oxidation at 1150 °C for 146 h [24].

However, none of the FC alloys formed continuous Al_2O_3 , Figure 6. With a reduction in W content, it is clear that CS-4 has the ability to form continuous Al_2O_3 at each temperature. The continuous Al_2O_3 layer did not form during the early stages of oxidation at 900 °C (Figure 6(d)) in CS-4, possibly due to insufficient Al activity at this temperature. Therefore, without the protection from Al_2O_3 layer, the oxide thickness of CS-4 is actually thicker than that of FC-2.0, Figure 6(h). Since lower W content would increase the rate of outward diffusion, and Al_2O_3 could not form initially in CS-4, the oxidation weight gain for CS-4 can be higher than that of FC-2.0. However, continuous Al_2O_3 does form on CS-4 eventually after 146 h, so better oxidation protection for CS-4 can be expected for longer exposure time. At 1000 °C, continuous Al_2O_3 can be detected on CS-4 only after 5 h exposure. Consequently, the continuous Al_2O_3 can be an effective diffusion barrier, the weight gain curve of CS-4 is lower than those of FC alloys from the start (Figure 5(b)). Furthermore, the thickness of oxidized layer is thinner than that of the 900 °C condition, even it is oxidized at 1000 °C after 146 h, Figure 6(h) and Figure 6(l).

At 1150 °C, the oxidation kinetics of CS-4 clearly shows a tremendous difference in comparison with those of FC alloys. Although FC alloys can also differentiate themselves in terms of oxidation behavior with higher Si content, FC alloys all show rapid weight gain during the initial stage of oxidation, while CS-4 can be protected more rapidly at around 50 h. FC alloys exhibit oxidized thickness layers greater than 200 μm , by contrast, the overall oxide thickness of CS-4 is only about 11 μm . So, CS-4 is an Al_2O_3 former, its ability to resist oxidation at temperatures above 1000 °C is better than that of its counterpart FC-2.0. The reduction in W content has allowed Al_2O_3 to form continuously very rapidly at higher temperatures, either by increasing the diffusivity of alloy system or increasing the Al activity in the alloy system. During the oxidation process, CoO and spinels can form at the very beginning stage. Since CoO and spinels are not protective, the rate of oxidation are relatively fast at first, followed by internal oxidation of Al_2O_3 formation. The behavior of Al_2O_3 formation has determined the difference between FC alloys and CS-4. As long as the continuous Al_2O_3 can be formed, it can inhibit the interdiffusion and growth of oxide layers effectively, hence the spinels are much thinner in CS-4. Partial pressure of oxygen for Al_2O_3 formation is very low, so the formation of Al_2O_3 should be a result of inward diffusion of oxygen more than the outward diffusion of Al, hence Al_2O_3 are formed at the interface between substrate and the top oxides such as CoO and spinels. In our previous study, increasing in the

content of Si in the Co-Al-W-Ni systems beyond 2 at% can promote the precipitation of μ phase [19]. The μ phase is thermodynamically stable at high temperature, and it can be deleterious to the high temperature mechanical properties, hence an upper limit of 2 at% Si is the preferred content. W is the major γ' forming element in the alloy system, lower W contents can lead to lower volume fractions of γ' precipitates (Figure 1) and lower strength (Figure 4), although it has a positive effect on promoting the formation of continuous Al_2O_3 . So there is a trade-off between oxidation resistance and mechanical strength. Finally, the γ' solvus temperature of CS-4 needs be higher, since its solvus temperature is below 1000 °C (Table III). Future alloy design should consider further increasing the γ' solvus of CS-4 by adding Ta or Nb, while maintaining its Al activity for formation of protective Al_2O_3 .

Conclusions

The followings conclusions can provide for future alloy design advanced Co-based superalloys. Addition of Si in Co-Al-W-Ni alloy system can increase the γ' volume fraction and the γ' solvus temperature, which is consistent with the Si partitioning tendency toward γ' phase, promoting an increase of K value of Al, and affecting the partitioning behavior of W. 2.0 at% Si can decrease lattice misfit and strengthen the alloy against plastic deformation at elevated temperatures. Si addition can improve oxidation resistance. W has a profound impact on the oxidation behavior; low content of W allows the formation of continuous Al_2O_3 at temperatures above 1000 °C, while high W content can slow down oxidation at 900 °C.

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