High-Temperature Structural Stabilities of Ni-Based Single-Crystal Superalloys Ni–Co–Cr–Mo–W–Al–Ti–Ta with Varying Co Contents

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Abstract It has been recently pointed out that the compositions of industrial alloys are originated from cluster-plus-glue-atom structure units in solid solutions. Specifically for Ni-based superalloys, after properly grouping the alloying elements into Al, Ni-like ($\overline{\text{Ni}}$), γ' -forming Cr-like ($\overline{\text{Cr}}'$) and γ -forming Cr-like ($\overline{\text{Cr}}''$), the optimal formula for single-crystal superalloys is established $[\text{Al}-\overline{\text{Ni}}_{12}](\text{Al}_1\overline{\text{Cr}}'_{0.5}\overline{\text{Cr}}''_{1.5})$. The Co substitutions for Ni at the shell sites are conducted on the basis of the first-generation single-crystal superalloy AM3, formulated as $[\text{Al}-\text{Ni}_{12-x}\text{Co}_x](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{W}_{0.25}\text{Mo}_{0.25})$, with $x = 1.5, 1.75, 2$ and 2.5 (the corresponding weight percents of Co are 9.43, 11.0, 12.57 and 15.71, respectively). The 900 °C long-term aging follows the Lifshitz–Slyozov–Wagner theory (LSW theory), and the Co content does not have noticeable influence on the coarsening rate of γ' . The microstructure and creep behavior of the four (001) single-crystal alloys are investigated. The creep rupture lifetime is reduced as Co increases. The alloy with the lowest Co (9.43 Co) shows the longest lifetime of about 350 h at 1050 °C/120 MPa, and all the samples show N-type rafting after creep tests.

KEY WORDS: Nickel-based single-crystal superalloys; Cluster-plus-glue-atom model; Composition formula; Co

1 Introduction

Ni-based single-crystal superalloys are widely used in aero-engines and industrial gas turbines for their superior mechanical properties at elevated temperatures [1–3]. The major alloying elements of the first-generation Ni-based single-crystal superalloys, being Re-free, are mainly Al, Ti, W, Ta, Mo, Cr, Nb, V and Co. Among them, Co is a

solution-strengthening element [4] that improves the microstructural stability [5], decreases the stacking fault energy [6] and reduces the precipitation tendency of topologically close-packed (TCP) phase [7]. The formation of TCP is suppressed by promoting the diffusion process with more Co additions [5]. It was also shown that Co plays a role similar to Ru for inducing the reverse partitioning of the alloying elements and for improving the resistance to the TCP formation [8]. However, the third-generation Ni-based single-crystal superalloys, CMSX-10, with low Co concentrations, also maintain an acceptable alloy stability [9], signifying that multi-element alloying effects are quite complex as Co alloying necessarily involves composition changes of other elements. In fact, the Co alloying is not always essential for practical use because Co-containing superalloys are very sensitive to tensile orientation [10]. Due to the increasing Co content, strongly segregating elements such as Ta and Re exhibit a significant decrease in

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the segregation tendency [11]. In addition, the Co content only influences slightly the lattice constants of the γ and γ' phase [5, 12], because Co substitutes Ni [13] with similar atomic radii of 0.125 nm [14].

Apparently, the Co content should be maintained at a proper level, which is actually not easy to be optimized experimentally as many alloying elements are present and are varying at the same time. Actually, early superalloys design methods, such as electron vacancies and d-electron method [15], mainly target the microstructural stability control, including TCP and γ' phases [16]. However, there still lacks an atomic structure platform on which the Co addition can be quantified in superalloys of complex chemistry.

Recently, in order to describe the chemical short-range-order structure in solid solutions, we developed a new cluster-plus-glue-atom approach. The major merit of this model is the proposition of chemical composition formulas that interpret the composition selections of many widely used alloys [17–31]. For instance, the most widely used industrial specification cartridge Cu–30Zn is formulated as $[\text{Zn-Cu}_{12}]\text{Zn}_4$ [18, 19]. The cluster-plus-glue-atom model, which is established on the atom interactions over nearest-neighbor clusters and next outer-shell glue atoms, is the bridge linking the chemical short-range-order structure and the alloy composition. We were also able to evaluate the γ' precipitation tendency in ternary $\overline{\text{Ni}}-\overline{\text{Cr}}-\text{Al}$ system using this approach [32].

In the study, the cluster-plus-glue-atom model and the relevant composition formula for Ni-based single-crystal superalloys are first proposed. The general formula is then used as the atomic structural platform for the composition design of alloys with different Co contents. Then, the designed alloy series are fabricated into Ni-based single-crystal superalloys and are characterized for their structures, thermal stabilities and high-temperature capabilities.

2 General Composition Formula for Ni-Based Single-Crystal Superalloys

The basic idea of the cluster-plus-glue-atom model is to derive a short-range-order structural unit with high structural stability as resulted from the chemical difference between many alloying elements and the solvent Ni.

Since Ni-based single-crystal superalloys are based on face-centered cubic (FCC) lattice (including the L1_2 - γ' phase which is a superstructure based on FCC), the cluster-based model identifies a structural unit consisting of a nearest-neighbor cuboctahedron cluster and some glue atoms situated outside the cluster, expressed by a so-called cluster formula $[\text{cuboctahedron}](\text{glue atoms})_x$ [18]. Here

the cuboctahedron cluster with coordinate number of 12 is centered by the major solute Al and nearest-neighbored by solvent Ni and its alike ($\overline{\text{Ni}}$, to represent the average solvent of Ni-like, including Ni and Co in this paper), for both the FCC- γ solid solution matrix and the L1_2 - γ' precipitation phase elements.

This cluster is expressed as $[\text{Al-Ni}_{12}]$ in Fig. 1. Interpreting the γ' precipitation tendency has been attempted using a general cluster formula $[\text{Al-Ni}_{12}](\text{Al}, \overline{\text{Cr}})_3$, where $\overline{\text{Cr}}$ covers Cr-like elements such as Ti, Ta, Cr, Mo and W.

According to the enthalpies of mixing (ΔH) between solute and major solvent Ni [33], the alloying elements are classified into Al (strongly interacting with Ni, negatively large ΔH 's), Ni-like $\overline{\text{Ni}}$ series (near zero ΔH 's, such as Ni and Co), γ -forming Cr-like series ($\overline{\text{Cr}}$, negatively small ΔH 's, such as Cr, Mo and W) and γ' -forming Cr-like series ($\overline{\text{Cr}}'$, negatively large ΔH 's but minor amounts, such as Ti and Ta).

Following the principles in constructing cluster formulas, the cluster center site is only filled with Al, the nearest-neighbor shell sites are occupied by $\overline{\text{Ni}}$ series, and the glue sites are taken by extra Al, $\overline{\text{Cr}}'$ and $\overline{\text{Cr}}$. Therefore, a general formula for Ni-based single-crystal superalloys is established, $[\text{Al-Ni}_{12}](\text{Al}, \overline{\text{Cr}}', \overline{\text{Cr}})_x$. Normal first-generation Ni-based single-crystal superalloys, being Re and Ru free, can be formulated accordingly as $[\text{Al-(Ni, Co)}_{12}](\text{Al}, (\text{Ti, Ta}), (\text{Cr, Mo, W}))_x$. After an extensive analysis on the available Ni-based single-crystal superalloys using the proposed general formula, the late generations of Ni-based single-crystal superalloys, destined for withstanding high

Fig. 1 $[\text{Al-Ni}_{12}]$ cluster in L1_2 - γ' . The same cluster also applies in face-centered cubic Ni–Al solid solution γ , as γ' is an ordered superstructure of γ

temperature in aero-engines, generally converge to a particular 16-atom cluster formula $[\text{Al}-\overline{\text{Ni}}_{12}](\text{Al}_1\overline{\text{Cr}}_{0.5}'\overline{\text{Cr}}_{1.5}')$, where the three glue atoms are taken by 1 Al atom, $0.5 \overline{\text{Cr}}'$ series atom and $1.5 \overline{\text{Cr}}'$ atoms. The relevant results will be reported in another paper. In order to unveil the role of Co in controlling the structural stabilities, new first-generation single-crystal superalloys are designed based on the ideal cluster formula $[\text{Al}-\overline{\text{Ni}}_{12}](\text{Al}_1\overline{\text{Cr}}_{0.5}'\overline{\text{Cr}}_{1.5}')$ with varying Co addition, as stated below.

3 Composition Design and Experimental Procedure

3.1 Composition Design and X-Ray Fluorescence Spectrometry Measurements

Composition series $[\text{Al}-\text{Ni}_{12-x}\text{Co}_x](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{Mo}_{0.25}\text{W}_{0.25})$ are designed as listed in Table 1, where different amounts of Co replace Ni in the shell sites of the cluster ($x = 1.5, 1.75, 2$ and 2.5). Actually, these composition series are close to an existing specification AM3 [1, 34], developed by ONERA for use in ARRIEL 2 TURBOMECA engines which equipped in the helicopters [1]. Its weight percent composition Ni–8Cr–5.5Co–2.25Mo–5W–6Al–2Ti–3.5Ta is concerted to the 16-atom cluster formula as $[\text{Al}-\text{Ni}_{10.6}\text{Co}_{0.9}](\text{Al}_1)(\text{Ti}_{0.4}\text{Ta}_{0.2})(\text{Cr}_{1.4}\text{Mo}_{0.2}\text{W}_{0.3})$. The designed alloys are named after their Co

levels in weight as shown in Table 1. The parent alloys were prepared by means of vacuum induction melting and then were cut into cylindrical rods with 30 mm in diameter for X-ray fluorescence spectrometry (XRF) by using Typenr-PW 4400/40. The test face of XRF was polished, and the measured compositions are listed in Table 1.

Table 1 shows the designed compositions and the ones measured by XRF. Al has a large absolute error (*A* error, $\pm 0.2\%$) because Al is easily exhausted due to evaporation during melting. Ti shows a large relative error (*R* error, $\sim 10\%$) because of its low contents. The deviations from designed compositions are generally low for the other elements, within $\pm 0.23\%$ (*A* error) and $\pm 5\%$ (*R* error).

3.2 Experimental

The [001]-oriented single-crystal superalloys were produced by the selector technique, and the crystal orientation deviations of the samples were maintained within 10° from the [001] orientation. The standard heat treatment scheme consists of three steps: 1310°C for 2 h plus 1315°C for 4 h (solution treatment, 15°C below the measured incipient melting temperature of 1330°C); 1150°C for 4 h (first aging to precipitate γ'); and 870°C for 24 h (second aging to regulate the cuboidal morphology). After each step, the specimens were air cooling. The alloy stability was evaluated both by long-term aging (900°C for 1000 h) and by creep test on a GWT304-type creep machine (at $1050^\circ\text{C}/120\text{ MPa}$). The temperature

Table 1 Chemical composition of the test alloy (wt%)

Alloy names	$[\text{Al}-\text{Ni}_{12-x}\text{Co}_x](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{W}_{0.25}\text{Mo}_{0.25})$		Al	Co	Cr	Ti	Ta	W	Mo	Ni
9.43 Co $x = 1.5$	$[\text{Al}-\text{Ni}_{10.5}\text{Co}_{1.5}](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{W}_{0.25}\text{Mo}_{0.25})$	Nominal	5.75	9.43	5.54	1.28	4.82	4.90	2.56	Bal.
		Actual	5.57	9.36	5.43	1.17	4.68	4.91	2.64	
		<i>A</i> error	− 0.18	− 0.07	− 0.11	− 0.11	− 0.14	0.01	0.08	
		<i>R</i> error%	− 3.21	− 0.71	− 2.07	− 8.31	− 2.98	0.21	3.22	
11 Co $x = 1.75$	$[\text{Al}-\text{Ni}_{10.25}\text{Co}_{1.75}](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{W}_{0.25}\text{Mo}_{0.25})$	Nominal	5.75	11.00	5.54	1.28	4.82	4.90	2.56	
		Actual	5.54	10.92	5.41	1.17	4.70	4.89	2.64	
		<i>A</i> error	− 0.21	− 0.08	− 0.13	− 0.11	− 0.12	− 0.01	0.08	
		<i>R</i> error%	− 3.72	− 0.70	− 2.42	− 8.31	− 2.56	− 0.20	3.23	
12.57 Co $x = 2$	$[\text{Al}-\text{Ni}_{10}\text{Co}_2](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{W}_{0.25}\text{Mo}_{0.25})$	Nominal	5.75	12.57	5.54	1.28	4.82	4.90	2.56	
		Actual	5.54	12.48	5.38	1.17	4.72	4.88	2.64	
		<i>A</i> error	− 0.21	− 0.09	− 0.16	− 0.11	− 0.10	− 0.02	0.08	
		<i>R</i> error%	− 3.71	− 0.69	− 2.96	− 8.30	− 2.14	− 0.39	3.23	
15.71 Co $x = 2.5$	$[\text{Al}-\text{Ni}_{9.5}\text{Co}_{2.5}](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{W}_{0.25}\text{Mo}_{0.25})$	Nominal	5.75	15.71	5.54	1.28	4.82	4.90	2.56	
		Actual	5.52	15.68	5.34	1.08	4.77	4.72	2.72	
		<i>A</i> error	− 0.23	− 0.03	− 0.20	− 0.20	− 0.05	− 0.18	0.16	
		<i>R</i> error%	− 4.05	− 0.17	− 3.67	− 15.34	− 1.09	− 3.65	6.37	

A error absolute error = actual − nominal, *R* error relative error% = (actual − nominal)/nominal * 100%

measurement error was limited to ± 3 °C. The microstructures of the samples at different conditions (the standard heat treatment, the long-term aging and the creep test) were characterized by S-3400N scanning electron microscope (SEM), and before the SEM observation all the samples were mechanically polished and electrically etched in a solution of 12 vol% H_3PO_4 + 40 vol% HNO_3 + 48 vol% H_2SO_4 which dissolves the γ phase.

4 Results and Discussion

4.1 Structural Stability During Long-Term Aging

The microstructural morphologies after the standard heat treatments and longer term aging as observed by SEM (Fig. 2) all show typical γ' cuboidal precipitates. The mean

size (denoted by “ a ” [5]) of the γ' was measured by quantitative metallography. For the mean size of the γ' after standard heat treatment, at least 100 particles were measured and the value of the mean size a was calculated by $a = (a_1 a_2)^{0.5}$, where a_1 and a_2 are the length and width in a γ' particle, respectively.

The coarsening of the γ' particles is followed during the long-term aging at 900 °C for up to 1000 h as shown in Fig. 2, too. After 300-h aging, γ' shows smooth cuboidal shapes but rafting is not initiated as the particles are basically separated. After 500-h aging, rafting occurs already as adjacent particles start to merge. After 1000-h aging, the coalescence of adjacent γ' particles is nearly accomplished and exhibits typical raft structures.

As shown in Fig. 3, the γ' coarsening is basically controlled by diffusion following LSW theory which was first proposed by Lifshitz, Slyozov and Wagner [35, 36]:

Fig. 2 Microstructures of the alloys 9.43 Co, 11 Co, 12.57 Co and 15.71 Co after standard heat treatment and long-term aging: **a, e, i, m** for alloys 9.43 Co, 11 Co, 12.57 Co and 15.71 Co, respectively, after standard heat treatment; **b, f, j, n** for alloys 9.43 Co, 11 Co, 12.57 Co and 15.71 Co, respectively, after 900 °C/300 h; **c, g, k, o** for alloys 9.43 Co, 11 Co, 12.57 Co and 15.71 Co, respectively, after 900 °C/500 h; **d, h, l, p** for alloys 9.43 Co, 11 Co, 12.57 Co and 15.71 Co, respectively, after 900 °C/1000 h

Fig. 3 Relationships between the mean particle diameter (a_0 being the initial particle diameter and a_t being the instantaneous one) at time during aging at 900 °C

$$\left[(a_t/2)^3 - (a_0/2)^3 \right] = Kt^{1/3}, \quad (1)$$

where a_0 is the initial particle diameter, a_t is the instantaneous particle diameter and t is the coarsening time. K is a rate constant [5], which is defined as formula:

$$K = 2\gamma DC_e V_m^2 / (\rho_c^2 RT), \quad (2)$$

where γ is the interfacial free energy of the particle/matrix interface, D is diffusion coefficient of solutes in matrix, C_e is the concentration of solute (γ' -forming element) in equilibrium with a particle of infinite radius, V_m is the molar volume of precipitates, ρ_c is the numerical constant dependent on the distribution of particle size ($\rho_c = 1.5$), R is the gas constant and T is the absolute temperature.

In this paper, the T is constant (1173.25 K, 900 °C). As a result, the γ , ρ_c and R are stable. V_m is only related to the lattice constant of γ' . However, the changing of Co level shows not much change on the lattice constant [12]. In the other word, the ρ_c is also constant. Co, replacing the solvent Ni, interacts strongly with the γ' -forming elements which occupy the center sites and the glue sites. More γ' -forming elements are excluded from the matrix with increasing Co contents, as a result of reduced C_e . At the same temperature, Co is able to increase the diffusion coefficient D when Co is increased [5]. So the slopes in Fig. 3 are nearly unchanged, and the constant K is not much related to the Co alloying. As a result, the Co content does not have noticeable influence on the coarsening rate of γ' .

Fig. 4 Creep rupture lifetimes as a function of Co contents

4.2 Creep Performance

The creep rupture lifetime as illustrated in Fig. 4 shows obvious decreasing tendency as a function of the Co contents. The 9.43 Co alloy survives the longest creep rupture life (about 350 h) at 1050 °C/120 MPa while the 15.71 Co alloy has the shortest creep rupture life (about 100 h). In this paper, the level of Co is above 9 wt%, as already known [8] sufficient amounts of Co (for instance 12.5–20 wt%) depress the γ' solvus temperature, accelerate particle coarsening at high temperatures and reduce the creep resistance.

Here, the cluster-plus-glue-atom formula for single-crystal superalloys always includes 1 center atom, 12 shell atoms and 3 glue atoms, where solvent Ni is all located at the shell sites. Since Co replaces Ni, it should be considered as a solvent similar element that interacts with other solute elements such as Al and Cr (Fig. 5). The weakened creep resistance is also caused by the fact that, as compared to Ni, Co has smaller negative enthalpies with other alloying elements. The enthalpies of mixing between Co and (Al, Ti, Ta, Cr, Mo, W) are, respectively, -19 , -28 , -24 , -4 , -5 and -1 kJ/mol, and those between Ni and (Al, Ti, Ta, Cr, Mo, W) are, respectively, -22 , -35 , -29 , -7 , -7 and -3 kJ/mol [33]. The tendency of creep rupture life shows that the content of Co should not exceed 9.43 wt% Co (below 1.5 Co in the proposed formula).

Figure 6 shows the SEM micrographs of the rupture surfaces after the creep test. In the condition of 1050 °C/120 MPa, the N-type raft occurs after γ' coarsening along the direction vertical to the stress and the cuboidal γ' particles disappear.

Fig. 5 The [center-shell] interaction and the [glue-shell] interaction about 9.43 Co, the value of the interaction means the value of enthalpies of mixing (kJ/mol) [33]

Fig. 6 Surface morphologies on longitudinal cross section after creep rupture (at 1050 °C/120 MPa): **a** 9.43 Co, **b** 11 Co, **c** 12.57 Co, **d** 15.71 Co

5 Conclusion

The cluster-plus-glue-atom model has been utilized to formulate Ni-based single-crystal superalloys as $[\text{Al}-\overline{\text{Ni}}_{12}](\text{Al}_1\overline{\text{Cr}}_{0.5}'\overline{\text{Cr}}_{1.5}')$, where the alloying elements are divided into four groups, Al, Ni-like ($\overline{\text{Ni}}$), γ' -forming Cr-like ($\overline{\text{Cr}}'$) and γ -forming Cr-like ($\overline{\text{Cr}}''$). The Co substitutions for Ni at the shell sites are conducted on the basis of an alloy series of the first generation, $[\text{Al}-\text{Ni}_{12-x}\text{Co}_x](\text{Al}_1\text{Ti}_{0.25}\text{Ta}_{0.25}\text{Cr}_1\text{W}_{0.25}\text{Mo}_{0.25})$, with $x = 1.5, 1.75, 2$ and 2.5 (the corresponding weights of Co are 9.43, 11.0, 12.57 and 15.17, respectively). After 1050 °C/120 MPa creep tests, the 9.43 Co alloy shows the longest creep rupture lifetime of about 350 h and the creep rupture lifetime is reduced when Co increases. All the alloys show N-type rafting for creep samples. The result of creep rupture life suggests that the level of Co should not exceed 9.43 wt% (below 1.5 Co in the cluster-plus-glue-atom formula). The 900 °C long-term aging process is followed by LSW theory. The slope K is stable because the Co is able to increase the C_e (the concentration of solute) and reduce the D (the diffusion coefficient). Therefore, the Co content does not have noticeable influence on the coarsening rate of γ' .

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