Effect of Interactions Among Elements on Diffusion Process Associated with γ' Coarsening in a Ni-Based Single-Crystal Superalloy

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Abstract

Coarsening of cuboidal γ' precipitates and relevant diffusion process in Ni-based single-crystal superalloy CMSX-4 were investigated at 1000, 1020 and 1040 °C for specific times. The γ' coarsening kinetics followed a cubic rate law with time and was presumably controlled by bulk diffusion of elements in γ matrix. The associated diffusion activation energy was experimentally determined to be about 300 kJ/mol when it is considered the temperature-dependent thermo-physical parameters in modified Lifshitz–Slyozov–Wagner theory. The influence of temperature on γ/γ' microstructure is briefly discussed based on pseudo-binary [Ni]–[Al] phase diagram. Interactions among elements can effectively raise the local vacancy formation and vacancy–atom exchange barriers close to γ - and γ' -partitioning elements, respectively. Thus, it can significantly reduce the inter-coupling migrations of atoms during the macroscopic cross-diffusion process associated with γ' coarsening of Ni-based superalloys.

Keywords Ni-based superalloy · Coarsening kinetics · Diffusion activation energy · Elemental interaction

1 Introduction

Nickel-based single-crystal (SC) superalloys are widely used to fabricate turbine blades and vanes in advanced aeronautic and industrial gas engines due to their excellent mechanical performances under service [1, 2]. In principle, the superior properties are inherited from the unique γ/γ' microstructure with exceptional stability at elevated temperatures above 700 °C. In the γ/γ' microstructure, cuboidal γ' precipitates with a high volume fraction (always 60–70%) are uniformly distributed in γ matrix and the order temperature of γ' phase with a $L1_2$ structure is as high as more than 1250 °C. In addition, the γ/γ' interfaces always keep a coherent state, but with

a small negative lattice misfit, i.e., the lattice parameter of γ' phase is slightly smaller than that of γ phase [1, 2].

An Ostwald ripening process of γ' particles, big ones grow at the expense of small ones, takes place spontaneously during thermal exposure. The size of γ' precipitates has been found to have a close link with the mechanical behavior during service [3–8]. A thermally activated coarsening of γ' precipitates usually deteriorates the mechanical strength [4, 5, 7, 8]. Thus, an accurate information about γ' coarsening is very crucial to the microstructure evaluation of a Ni-based SC superalloy [9, 10].

Mass migration in γ matrix is one important controlling factor of γ' coarsening. It can be assessed by the associated diffusion activation energy (Q_{eff}), which can be obtained through both experiments and numerical calculations. However, the influence of temperature on γ/γ' microstructure and the role of elemental interactions in diffusion process were always neglected in the previous literature. When the γ/γ' microstructure was thought to be independent of temperature, the Q_{eff} values determined by experiments were usually in a range of 270–280 kJ/mol in alloys with various compositions [11–17]. An accepted consensus is that an addition of refractory elements like W, Mo, Ta and Re can effectively decrease the overall diffusivity, resulting in a larger Q_{eff} [12, 18]. In addition,

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these experimental Q_{eff} values seem to be confusing when a relationship of mechanical behavior with deformation microstructure is tried to be established. For example, the creep life of CMSX-4 (containing 3 wt% Re) alloy is about five times as long as that of CMSX-2 (Re free) alloy under 950 °C/240 MPa where a major rate-controlling factor is the diffusion of vacancies and elements [6, 19]. The reported Q_{eff} (272.40 kJ/mol) of CMSX-4 alloy was just the same as that (271.11 kJ/mol) of CMSX-2 alloy [14, 16]. Furthermore, using a simple linear equation method in which the complicated interactions among elements were neglected, the predicted Q_{eff} values tended to be large. In the case of CMSX-4 alloy, a calculated Q_{eff} value (324.8 kJ/mol) was larger than an experimental result (272.40 kJ/mol) [12, 16, 17]. More efforts are needed to address these crucial points mentioned above in γ' coarsening and associated mass migration.

Recently, the significant influences of elemental interactions on defect–solute complexes have been highlighted in some simple systems and model alloys [20–24]. Compared with the strain part induced by the difference in modulus and size misfits, the electronic part resulted from d – d electronic hybridization is much more efficient in interactions between dislocations as well as vacancies and alloying solutes, especially at high temperature [20, 21]. It is consistent with the experimental observation [22]. The migration of an atom depends on not only itself electronic structure but also its neighboring electronic atmosphere [21, 23]. In other words, the mobility of an atom can be noticeably influenced by neighboring atoms when their interactions are strong [21]. More importantly, the attractive electronic interactions between refractory element (Re, Ru, W or Ta) and Cr can significantly influence the diffusional flux of each other during microscopic cross-diffusion process [24]. These results obtained above are quite helpful to understand the function of elemental interactions on diffusion process associated with γ' coarsening in such a multicomponent system as Ni-based SC superalloys.

Based on the above considerations, this study focuses on the Q_{eff} of mass transport in γ matrix during γ' coarsening in CMSX-4 alloy, in which temperature-dependent γ/γ' microstructure was thoughtfully considered. Furthermore, the fundamental role of elemental interactions in migrations of γ - and γ' -partitioning elements during macroscopic cross-diffusion process is discussed qualitatively, with the help of the novel work in Ref. [21].

2 Material and Experiments

2.1 Material

Chemical composition of CMSX-4 alloy is following (wt%): 6.5 Cr, 9.6 Co, 0.6 Mo; 6.4 W, 6.5 Ta, 5.6 Al, 1.0

Table 1 Isothermal exposure experiments of CMSX-4 alloy

Tem- perature (°C)	Time (h)				
1000	260	284	304	352	
1020	210	234	260	304	
1040	170	194	210	260	

Ti, 3.0 Re, 0.1 Hf and balances Ni. A [001]-oriented SC cast rod with Φ 16 mm was received. A specific solution heat treatment was conducted on this rod at 1315 °C for 50 h, followed by air cooling (AC), to fully dissolve γ – γ' eutectics and eliminate dendritic segregation. In order to minimize the scatter of γ/γ' microstructure induced by various cooling rates, the homogenized rod was then cut into small plates with a thickness of 3 mm normal to the longitudinal axis by spark erosion. These plates packaged into a quart glass were homogenized again at 1315 °C for 2 h, AC and precipitation aged at 1140 °C for 2 h, AC and 870 °C for 20 h, AC. The obtained γ/γ' microstructure was uniform on a dendritic scale. The average size and volume fraction of cuboidal γ' particles were measured to be 0.348 (± 0.032) μm and 69.58 (± 1.13)%, respectively.

2.2 Thermal Exposure Experiments

A seemingly similar paper was published by Lapin [16]. The effect of temperature on γ/γ' microstructure was neglected and the role of elemental interactions in atomic migrations is absent in the paper. So we study further on this topic. On the basis of Lapin's work at 850, 900, 925 and 950 °C, we further conducted isothermal exposure experiments at 1000, 1020 and 1040 °C and make a relatively deep thinking about the influence of elemental interactions on mass transport during cross-diffusion process. The precise measurement of γ' volume is the first key step to experimentally determine the γ' coarsening behavior and the related Q_{eff} . An equivalent volume of a cuboidal γ' particle can be easily measured from its area through geometric projection. On the other hand, lots of interfacial dislocations can be thermally generated to relieve γ/γ' constraint strain, especially for long-term aging [25, 26]. In order to weaken the influence of massive interfacial dislocations on γ' coarsening, the aging times in this study were selected to avoid producing abundant interfacial dislocations. Small plates were isothermally exposed at 1000, 1020 and 1040 °C for definite times, as shown in Table 1, in a muffle furnace, followed by water quenching (WQ). Also each plate was encapsulated into a quart glass to reduce oxidation.

2.3 Electron Microscope Characterization

Characterizations of γ/γ' microstructure were conducted by scanning electron microscopy (SEM). SEM specimens were mechanically ground and polished and then etched in a reagent of 4 g $\text{Cu}_2\text{SO}_4 + 20$ ml $\text{HCl} + 20$ ml H_2O for 30 s at room temperature. The γ/γ' microstructure was observed using a field emission SEM (FE-SEM, Apero) with a good spatial resolution at a low voltage (10 kV).

2.4 Micrograph Statistics

Evolutions of mean size and volume fraction of γ' precipitates with temperature and time were digitalized in SEM micrographs using a computerized image analyzer. The equivalent size of each γ' precipitate was calculated as the square root of its measured area. More than 2000 γ' precipitates were chosen to count the average size of γ' particles. The equivalent volume fraction of cuboidal γ' precipitates was determined through geometric projection by the following formula:

$$V_{\gamma'} = (A_{\gamma'})^{1.5}, \quad (1)$$

where $V_{\gamma'}$ and $A_{\gamma'}$ are the volume fraction and area fraction of γ' particles, respectively. The mean γ' volume fraction was determined from three SEM micrographs at the same magnification (30000 $\times$).

3 Results

3.1 Evolution of γ/γ' Microstructure

SEM micrographs showing the γ/γ' microstructure after experiments are presented in Fig. 1. As expected, γ' precipitates still keep cubic. There occurs a few coalescences of neighboring γ' particles (i.e., L-shaped γ' particles), marked by blue arrows, in Fig. 1. This occurrence is negligible. According to the method proposed by Epishin et al. [27, 28], a small number of γ/γ' interfacial dislocations are found after through observations. The influence of interfacial dislocations on γ' coarsening and related mass migration is not taken into account here.

Counted volume fractions and sizes of γ' precipitates under different conditions are given in Fig. 2. The volume fraction of γ' precipitates remains constant at each temperature, and it decreases with increasing temperature. These experimental values are close to the predictable ones [29]. The average size of γ' precipitates, as shown in Fig. 2b, increases with time at each temperature. Based on these

observations, it is reasonably concluded that an Ostwald ripening of γ' precipitates has occurred during thermal exposure.

3.2 Coarsening Kinetics of γ' Precipitates

In order to better determine the rate-limiting factor of γ' coarsening, plots of a^3 versus t as well as a^2 versus t and corresponding fitting parameters are shown in Fig. 3 and Table 2, respectively. It clearly shows that the γ' coarsening kinetics is better in line with a cubic rate law. It indicates the mass migration in γ matrix is the rate-controlling factor according to LSW theory [11, 30, 31]. It agrees well with the previous findings [14, 16, 32–34]. The γ' coarsening process is accelerated with increasing temperature.

The corresponding k in modified LSW theory is then determined by [11]:

$$k = 8A(V_{\gamma'})\sigma D_{\text{eff}}N_{\alpha}V_{\text{m}}/(9RT), \quad (2)$$

where $A(V_{\gamma'})$ is a dimensionless factor, σ is the γ/γ' interfacial energy, D_{eff} is the effective diffusion coefficient, N_{α} is the equilibrium mole fraction of solutes in γ matrix, V_{m} is the molar volume of γ' phase, R is the gas constant and T is the aging temperature. Normally, the effective D_{eff} can be expressed as:

$$D_{\text{eff}} = D_0 \exp(-Q_{\text{eff}}/RT), \quad (3)$$

where D_0 is the pre-exponential factor. Using Eqs. (2) and (3), the expression for determining diffusion activation energy can be written as follows:

$$T \ln((9TRk)/(8A(V_{\gamma'})\sigma N_{\alpha}V_{\text{m}})) = -Q_{\text{eff}}/R. \quad (4)$$

To obtain a Q_{eff} , the temperature-dependent thermo-physical parameters such as $A(V_{\gamma'})$, σ , N_{α} and V_{m} in γ/γ' microstructure are better to be refined.

Both theoretical analysis and experimental results show that $A(V_{\gamma'})$ sensitive to the morphology of γ' precipitates has a positive correlation with $V_{\gamma'}$ [35]. In the SEM investigation, the γ' precipitates still remain cubic, and thus, $A(V_{\gamma'})$ can be achieved by the fitted polynomial [17]:

$$A(V_{\gamma'}) = 2.22 + 25.55V_{\gamma'} - 1.6V_{\gamma'}^2 + 0.65V_{\gamma'}^3. \quad (5)$$

In addition, σ plays an important role in k . In fact, σ depends on many factors, such as compositions of γ and γ' phases, $V_{\gamma'}$ and γ/γ' lattice misfit [36–38]. The σ value can be calculated according to the following relation [39, 40]:

$$\sigma = (1.12832\Delta H_{\text{m}})/[N_0(a_{\gamma} + a_{\gamma'})^2], \quad (6)$$

where ΔH_{m} is the enthalpy of 1 mol γ' -forming solutions in γ phase, N_0 is the Avogadro's constant and a_{γ} and $a_{\gamma'}$ are

Fig. 1 SEM micrographs of γ/γ' microstructure: **a, d, g, j** 1000 °C; **b, e, h, k** 1020 °C; **c, f, i, l** 1040 °C

Fig. 2 Evolutions of γ' precipitates with temperature and time: volume fraction **a** and average size **b**

Fig. 3 Comparisons between square **a** and cubic **b** rate laws of γ' coarsening

Table 2 Fitting parameters of cubic and square rate laws

Temperature (°C)	Square rate law	Cube rate law
1000	0.9639	0.9739
1020	0.9645	0.9807
1040	0.9887	0.9970

Table 3 $A(V_\gamma')$, σ , N_α and V_m at various temperatures [17, 29, 39, 40]

Temperature (°C)	$A(V_\gamma')$	σ (mJ/m ²)	N_α	V_m (cm ³ /mol)
850	19.42	84.51	0.4555	7.21
900	18.96	82.84	0.4460	7.23
925	18.67	82.00	0.4415	7.25
950	18.35	81.19	0.4370	7.26
1000	17.28*	79.51	0.4274	7.28
1020	16.85*	78.83	0.4237	7.29
1040	16.20*	78.16	0.4199	7.30

*Experimental value in this study

the lattice parameters of γ and γ' phases, respectively. Using thermo-physical properties module in “single-crystal” database of JMatPro 7.0, the calculated values of σ , also the predictable N_α and V_m values, are listed in Table 3 [17, 29, 39, 40]. $A(V_\gamma')$, σ and N_α decrease, while V_m increases as temperature raises. It clearly shows that the thermo-physical parameters are obviously influenced by temperature.

Figure 4 shows the values of D , Q_{eff} and D_0 finely determined from the experimental data in this study. Besides, the re-determined results originating from the data in Ref. [16] are shown in Fig. 4. The obvious influence of temperature on thermo-physical parameters is considered here, while it was absent in Ref. [16]. Clearly, the predictable D values have a good exponent relationship with temperature. More importantly, these D values are self-consistent, as shown in Fig. 4a. The predicted Q_{eff} and D_0 values are compared

with those fine estimated ones from Ref. [16] in Fig. 4b–d. When the temperature-dependent thermo-physical parameters were taken into account, there is a good agreement between the obtained results. The D_0 values are found to be in the same order of magnitude ($\times 10^{-4}$ m²/s). The Q_{eff} values calculated from this study and Ref. [16] are close to each other, 296.17 kJ/mol and 300.87 kJ/mol. In addition, the Q_{eff} value is found to be roughly independent of the initial γ/γ' microstructure [14]. Therefore, a Q_{eff} of mass migration in γ matrix, closely connected with γ' coarsening, is obtained to be about 300 kJ/mol in CMSX-4 alloy.

Big discrepancies are found between D , Q_{eff} and D_0 values determined here and the ones in simple systems. Values of Goldschmidt atom radius (r), Q and D_0 of solute (X) in Ni are listed in Table 4 [41–43]. The D is higher than that of Re, one solute with the slowest diffusivity in Ni, at each temperature and their difference becomes bigger with increasing temperature. By comparison, the D_0 value ($\times 10^{-4}$ m²/s) is close to that in Ni– X (Al, Ti, Hf, Mo, Co and Cr) system [41–43]. This suggests the D_0 might be closely linked with the inside elastic distortion strain. In addition, the determined Q_{eff} value (~ 300 kJ/mol) is larger than that of solute in Ni, but lower than the linear calculated value (324.8 kJ/mol) [12, 41, 43]. Based on these findings, it indicates that the interaction among elements plays a fundamental role in mass migration and its influence is significant at elevated temperature [20, 21].

4 Discussion

4.1 Temperature-Dependent γ/γ' Microstructure

Obvious modifications in γ composition and γ/γ' lattice misfit with temperature play a major role in the thermo-physical parameters in γ/γ' microstructure. A pseudo-binary [Ni]–[Al] phase diagram, analogical to γ/γ' microstructure, is illustrated in Fig. 5. Supposing one alloy has an overall

Fig. 4 Comparison of D **a**, D_0 **b**, Q_{eff} **c** values finely estimated from this paper and Ref. [16], D values of Re in Ni included in **d** [41]

Table 4 Values of r , D_0 and Q of solute in Ni [40–42]

Solute	r (nm)	D_0 (m ² /s)	Q (kJ/mol)
Cr	0.128	2.42×10^{-4} **	280**
Co	0.126	3.30×10^{-4} **	294**
Al	0.143	1.69×10^{-4} **	257**
Ti	0.146	1.10×10^{-4} **	259**
Ta	0.147	2.19×10^{-5} *, 7.05×10^{-5} **	251*, 267**
Hf	0.159	1.91×10^{-4} **	252**
Mo	0.140	2.01×10^{-4} **	282**
W	0.141	8.0×10^{-6} *, 6.77×10^{-5} **	264*, 288**
Re	0.138	8.2×10^{-7} *, 8.67×10^{-6} **	255*, 285**
Ni	0.125	5.64×10^{-8} **	265**

*Experimental values in Ref. [41]

**Calculated values in Ref. [43]

composition indicated by “a,” in equilibrium at a given temperature, individual compositions of γ and γ' phases can be indicated by “b” and “c,” respectively. The composition of γ phase is varied with temperature because of its intrinsic solid solution characterization, while that of γ' phase changes minor owing to its inborn stoichiometric nature. This tendency is noticeable at elevated temperature above 800 °C [29, 44]. In other words, the γ' phase dissolves increasingly with raising temperature. In addition, due to a higher thermal expansion coefficient of γ phase, the γ/γ' lattice misfit

Fig. 5 Schematic drawing of pseudo-binary [Ni]–[Al] phase diagram

gradually becomes more negative when the temperature is above 650 °C [45]. So the thermo-physical parameters should be fined at each temperature when a mass migration Q_{eff} associated with γ' coarsening is tried to be obtained.

4.2 Vacancy-Assisted Migrations of γ - and γ' -Partitioning Elements During Macroscopic Cross-Diffusion Process

Interactions among elements have been receiving more and more attentions in the microstructure evolution that is mainly controlled by mass transport. It is Ni-based SC superalloys

Fig. 6 Schematic drawing of microscopically directional migration of Al **a, b** and Re **c, d** atoms in terms of probability

that contain at least, more than 35 at%, nine kinds of solutes such as Ni, Co, Cr, Al, Ta, Ti, W, Mo and Re. Furthermore, the γ matrix can be enriched by as much as about 50 at% of solutes due to a significant partitioning behavior of solutes between γ and γ' phases. As a result of the differences in electronic structure, their electronic interactions are found to be outstanding. The influence of (electronic) interactions among elements on mass migrations is discussed qualitatively below.

Elemental interactions among elements play a fundamental role in their migration. Different kinds of atoms with specific electronic characters can interact with each other to form a local complex neighborhood, and even covalent-like directional bonds can be formed [46]. On an atomic scale, the vacancy formation and the atom–vacancy exchange, two substantial steps of atomic migration, are both principally controlled by not only solute itself but also so-called ternary interactions. Generally, the vacancy formation energy in Ni is minor sensitive to solute(s), while its atom–vacancy exchange energy is largely influenced by local environment [21, 23]. Typically, the atomic migration barriers of Al and Re are both influenced by other solutes as a result of the strong electronic interactions. From another perspective, the migration energies of Al are lower than its vacancy formation energies, whereas the situation for Re is on the contrary. It suggests that the mass transport of γ' -partitioning solutes (like Al) tends to be controlled by the formation of adjacent vacancies, while that of γ -partitioning solutes (like Re) is more likely to be controlled by the exchanges of atoms with neighboring vacancies.

Not only that the microscopic cross-diffusion flux of elements is another essential factor affecting the correlation effects on atomic migration. In CMSX-4 alloy, Co, Cr, Re,

Mo and W mainly segregate into γ phase, whereas Al, Ti, Ta and Hf are enriched in γ' phase [47]. These partitioning tendencies of elements can be kept up to the γ' solvus temperature, although the corresponding magnitudes gradually narrow with increasing temperature. As a result, microscopically directional diffusion fluxes of excessive γ -partitioning elements (Co, Cr, Re, Mo and W) and γ' -partitioning elements (Al, Ti, Ta, Hf and Ni) take place during γ' coarsening. In other words, γ' -partitioning solutes diffuse from a shrinking γ' particle to an expanding one due to the capillarity effect; on the contrary, it is the opposite situation for γ -partitioning elements. In fact, every atom movement is not completely independent, but largely relies on the local environment, which is called correlation effect. Also as a typical example, schematic examples of Al and Re migrations on two-dimensional stacking faults are illustrated in Fig. 6. For simplicity, Al and Re atoms move statistically along opposite directions. As shown in Fig. 6a and b, an Al atom successfully exchanges with an adjacent vacancy under thermal activation. Its front is enriched with γ -partitioning elements in terms of probability. The probability of neighboring vacancy formation is decreased, and thus, the migration of Al atom can be decreased. Alternatively, as shown in Fig. 6c and d, when a Re atom statistically encounters a local atmosphere of γ' -partitioning elements in front, its exchange with a neighboring vacancy becomes more difficult, and consequently, the movement of Re also becomes slowed.

5 Conclusions

1. The γ' coarsening following a cubic rate law is controlled by the mass transport in γ matrix according to LSW theory.

2. The diffusion activation energy of mass transport in γ matrix during γ' coarsening is experimentally determined to be about 300 kJ/mol.
3. Temperature-dependent thermo-physical parameters are closely linked with the dissolution of γ' phase and the magnitude of γ/γ' lattice misfit with temperature.
4. Elemental interaction plays a key role in atomic diffusion, which is highlighted during macroscopic cross-diffusion process, in a multicomponent system.
5. The influence of interaction between elements becomes more and more significant with increasing temperature.

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