Simulation of Microstructure Formation Process
in Fe-23Cr-6Ni-3Mo-0.1N Alloy Using Multiphase Field Method

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The microstructure formation process in duplex stainless steel was simulated using a multiphase field (MPF) method. The dendrite solidification of a primary δ phase and the growth of an acicular γ phase by solid-state transformation were simulated, and the calculation results were found to nearly correspond to those of the experimental microstructure obtained using a liquid-tin quenching method. The MPF method is considered to be highly effective in designing duplex stainless steels and in evaluating the cooling-rate dependence of the microstructure morphology in welding solidification.

Key Words: Duplex Stainless Steel, Solidification, Solid-State Transformation, Microstructure, Multiphase Field Method

1. INTRODUCTION

Duplex stainless steels with high nitrogen content offer high strength and excellent corrosion resistance and have been used for a wide variety of applications, such as in chemical tankers and desalination plants. Although the material properties of duplex stainless steel depend on the ferrite/austenite phase balance 1), the phase proportion is affected not only by the chemical composition, but also by the cooling rate. Therefore, kinetic analyses of precipitation for the austenite (γ) phase, which has higher nitrogen solubility than ferrite, have previously been conducted 2,3). However, this requires the acquisition of substantial amounts of experimental data for each alloy, and a statistical analysis of the data must also be performed. In addition, the composition profile could not be predicted in previous studies, although the nitrogen content in the ferrite phase significantly affects the pitting corrosion resistance in duplex stainless steels 4).

The authors investigated the microstructure formation process of duplex stainless steel using a liquid-tin quenching method and revealed dendrite growth in the δ-phase and γ-phase formation by solid-state transformation 5,6). Based on these results, the microstructure formation process was simulated in duplex stainless steel using a multiphase field (MPF) method. The simulation results obtained are presented in this paper. The MPF method 7,8) was found to be capable of simulating the microstructure formation process in multiphase and polycrystalline systems, as well as analyzing complex microstructure morphology and composition distribution, such as those of dendrites during solidification. The purpose of this study is to simulate the microstructure formation process using the MPF method and to estimate the effect of cooling rate on the formation of γ phases.

This study used the MICRESS software (version 6.4)7) for the calculation of the microstructure formation. The analysis performed by the MPF method is described in detail, for example, in Fukumoto and Nomoto 9). In this method, rectangular lattices with a certain lattice width are analyzed two-dimensionally by a finite difference method. In the calculation, the size of the lattice used was 0.20 μ and the interface thickness was four lattices. MICRESS also has a feature that allows the user to select the thermodynamic database and diffusion database to be used for the calculation of the free energies and mobilities, respectively, in conjunction with the CALPHAD-based software Thermo-Calc 9). The thermodynamic database used in the simulation was FE_DATA version 6 10), and the diffusion database used was MOB2. The calculation components were Fe-23%Cr-5.8%Ni-3.0%Mo-0.11%N; the five-component system was selected by considering the precision of the thermodynamic database.

An outline of the calculation is shown in Figure 1. Two points were set as the position of the dendrite in the primary δ phase, and these growth directions were tilted by 1° with respect to the center to make stable grain boundaries in the midsection. The primary dendrite arm spacing λ1 and the cooling rate for the calculation were set according to the experimental results obtained by Iwasaki et al 9). The solidification cooling rate U1 was 100 K/s, and the temperature gradient G1 was 600 K/m; therefore, the solidification velocity at the steady state was U1/G1 = 1.67 mm/s. For the initial temperature of the formation of the primary δ phase at the bottom, the temperature at the tip of the dendrite (at a solidification rate of 1.67 mm/s) calculated by the Kurz-Giovanola-Trivedi model 11,12) was used and set to reach the steady state in a short time. The calculation consisted of the following four steps: (1) stable growth of two dendrites in the primary δ phase using a moving frame.

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(\(U_l = 100\) K/s during solidification); (2) single \(\delta\) phase solidification in the region; (3) homogenization of the components in the single \(\delta\) phase \((U_s = 50\) K/s after the solidification); and (4) growth of the \(\gamma\) phase from the grain boundaries and the grains. The \(\gamma\)-phase growth was assumed to be under paraequilibrium, which considers the diffusion control of \(N\), and its morphology was assumed to be an acicular growth\(^{13}\). The direction of 45° was selected for stable growth. Moreover, the interfacial energy of the liquid/\(\delta\) phase and that between the \(\delta\) and \(\gamma\) phase were set to 0.1 and 0.2 J/m\(^2\), respectively, and the interfacial mobility was set to automatic\(^{14}\). It is noted that the interfacial mobility of the liquid/\(\delta\) phase was approximately 0.07 cm\(^4\)/J/s and that of the \(\delta/\gamma\) phase (at 1373 K) was approximately 5×10\(^{-5}\) cm\(^4\)/J/s.

Furthermore, the effect of the cooling rate on the formation of \(\gamma\) phases was analyzed. The solidification cooling rates \(U_l\) were 50, 100, and 200 K/s, and the cooling rate in the solid state \(U_s\) was assumed to be halved. The limited range of the cooling rate was selected to avoid changes in the microstructural morphology. Based on the Kurz-Fisher model\(^{15}\), the primary dendrite arm spacing \(\lambda_1\) was varied with the solidification velocity (= cooling rate/temperature gradient). Following Iwasaki et al\(^\text{5}\) and Iwasaki and Fukumoto\(^\text{6}\), the initial nucleation temperatures for each cooling rates were set to 1483 (undercooling: 100 K), 1478, and 1473 K in the grain boundaries and to 1368, 1328, and 1318 K in the grains. Thus, the greater the cooling rate, the higher the degree of undercooling.

3. RESULT AND DISCUSSION

Figure 2 shows a comparison of the dendrite microstructure determined using the calculation with that obtained using the liquid-tin quenching method. The anisotropy of the interface mobility had a four-fold symmetry, and by adjusting the strength of anisotropy \((\epsilon_z = 0.0133)\), we obtained, through simulation, a microstructure that was close to that obtained experimentally. In the component system, the interfacial energy of the liquid/\(\delta\) phase was halved from the conventional analysis\(^{8}\) to simulate a fine structure. Additionally, as shown in Figure 3, the MPF method also enabled calculation of the distribution of solute during solidification. In the single \(\delta\) phase after solidification, microsegregation was homogenized by diffusion; however, the behaviors of \(N\) and \(Ni\) were found to be different owing to the difference in their diffusion rates.

| Temperature at the bottom | Phase | N (wt%) | Ni (wt%) |
|--------------------------|-------|---------|----------|
| 1738 K                   |       |         |          |
| 1500 K                   |       |         |          |

Fig.3 Calculated phase and composition by using the MPF method.

The \(\gamma\) phase was developed from the grain boundary at the middle portion between the two dendrites at 1478–1428 K and was also developed in the grain at 1328–1278 K. These values were set according to the experimental results obtained by Iwasaki et al\(^\text{5}\). The \(\gamma\) phase was then grown acicular 45° upward with decreasing temperature. Figure 4 compares the calculation results of the behavior of the \(\gamma\) phase grown with the microstructure obtained through the experiment using the liquid-tin quenching method. The amount of \(\gamma\) phase calculated was 25.1%, which was comparable to the experimental result (24.1% as obtained
by image analysis), while the morphology of the γ phase also appeared to be nearly similar to that obtained by the experiment. Because N was concentrated at the γ phase, the average concentrations of N in the γ and δ phases were 0.45% and 0.014%, respectively. Figure 5 shows the solute distribution measured by electron probe micro-analysis (EPMA). The γ phase is considered to grow under paraequilibrium, which considers the diffusion control of N, as described in the previous findings\(^1\). The calculated N concentration in the γ phase, shown in Fig. 4, was relatively comparable to the experimental N concentration in the γ phase in the grains; however, it was larger than the experimental N concentration in the γ phase in the immediate vicinities of the grain boundaries. The reason for this could be attributed to the analytical deviation of EPMA that occurs due to the minute γ phase in the immediate vicinities of the grain boundaries; thus, the accuracy of the thermodynamic database (e.g., partition of nitrogen between δ and γ, diffusion coefficient, etc.) may also need to be verified.

Subsequently, the γ phase formation was analyzed with varying cooling rates. The solidification cooling rates \(U_l\) were 50, 100, and 200 K/s. Furthermore, the initial nucleation temperatures were varied both in the grain boundaries and in the grains. Figure 6 shows the effect of cooling rate on the amount of γ phase formed; as the cooling rate increased, the amount of γ phase tended to decrease.

The change in growth behavior around 1350 K corresponds to the γ phase formation in the grains. This result nearly corresponds to that of the previous study\(^4\), in which the cooling rates during laser welding were changed. In addition, the calculated average concentration of N in the δ phase decreased with an increase in the cooling rate.

**Fig. 4** Calculated phase and N content by using the MPF method.

| Calculation | Microstructure |
|-------------|----------------|
| Phase at 1473 K | [Image]
| at 1373 K | [Image]
| at 1273 K | [Image]
| N (wt%) at 1273 K | [Image]

**Fig. 5** Result of the EPMA in the liquid-tin quenched microstructure.

| Microstructure | N (wt%) | Ni (wt%) |
|----------------|---------|----------|
| [Image] | [Image] | [Image] |

The MPF method can analyze complex microstructure morphology as well as predict the composition profiles...
affecting the microstructure formation process and the effect of
the cooling rate. It is thought that the nucleation
temperature significantly affects the amount of $\gamma$ phase. The
degree of undercooling for intergranular $\gamma$ precipitation was
varied in steel grades\textsuperscript{5).} Quantification of the nucleation and
improvement of the thermodynamic database will enable
highly accurate calculations.

4. CONCLUSIONS

The microstructure formation process in duplex stainless
steel was simulated using the MPF method, and the
following results were obtained. Adjusting the interfacial
energy and strength of anisotropy of the interfacial mobility
and the growth morphology of the $\gamma$ phase yielded a
microstructure morphology that was relatively similar to
that obtained by the experiment. Our finding that the amount
of $\gamma$ phase decreased with the increase in the cooling rate
nearly corresponds to the previous findings, thus verifying
the effectiveness of the MPF method.

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