Refinement of Solidification Microstructure and Austenite Grain by Fine Inclusion Particles

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The effect of deoxidation products of Ce_2O_3 , ZrO_2 and MgO particles on solidification microstructure has been studied in Fe–10mass%Ni, Fe–0.20mass%C–0.02mass%P and Fe–0.50mass%C–1mass%Mn alloys. The degree of the equiaxed crystallization is explained by the lattice misfit parameter between γ (or δ)-Fe and oxide. The single-phase solidification microstructure of Fe–10mass%Ni and Fe–0.50mass%C–1mass%Mn alloys is well related to austenite grain boundaries under the inhibition of grain growth by pinning. The correspondence between solidification structure and initial austenite grain has been studied in two-phases solidification of Fe–0.15 (or 0.30)mass%C–1mass%Mn–1mass%Ni alloy. The γ -grain size decreases with decreasing the lattice misfit parameter between γ -Fe and oxide and increases with decreasing the Zener pinning force. The number of γ -grains to that of primary δ -grains per unit area in a cross section increases with decreasing the aforementioned lattice misfit parameter, indicating that more than one nucleation event per δ -grain occurs at δ -ferrite grain boundary during δ to γ transformation.

KEY WORDS: solidification; δ - γ transformation; Zener pinning force; grain growth; deoxidation; inclusion.

1. Introduction

In low alloy steel weld metals the austenite grains are usually finer than the δ -ferrite columnar grain boundaries during δ to γ transformation.¹⁾ However, if the austenite is bounded by an orientation relationship with the δ -ferrite, the austenite cannot grow across the primary δ -ferrite solidification boundaries. It is reported²⁾ that in the presence of Al_2O_3 inclusions, nucleation of austenite phase becomes energetically more favorable and thus steel solidifies directly as austenite in interdendritic liquid.

In this review the refinement of solidification microstructure and the equiaxed crystallization by using various deoxidation products are first discussed as a function of inclusion number and composition, and solute content in an Fe–10mass%Ni or Fe–0.50mass%C. Secondly, the correspondence between solidification microstructure and initial austenite grain is demonstrated for a single-phase (Fe–10mass%Ni and Fe–0.50mass%C) and two-phases (Fe–0.15 and 0.30mass%C) solidification alloys, focusing on the effects of oxide particles on the heterogeneous nucleation of δ to γ transformation and the austenite grain growth inhibition.

2. Experimental

An Fe–10mass%Ni alloy (70 g) containing the initial oxygen content of 80 to 120 mass ppm was deoxidized with an Fe–10mass%M ($M=\text{Ti}$, Zr and Ce) and Ni–10mass%Mg alloys at 1600°C in an Al_2O_3 crucible using an induction furnace (100 kHz). (% and ppm represent mass% and mass

ppm, respectively, hereinafter.) A melt was cooled to 1400°C at $50\text{ K}\cdot\text{min}^{-1}$, followed by rapid quenching. The solidification structure was observed after etching in an Oberhoffer solution. The austenite grain size was measured after etching in a saturated picric acid solution. More details are given elsewhere.^{3,4)}

An Fe–C (0.15 to 0.50%) alloy (70 g) containing 1% Mn whose initial oxygen content was 80 to 100 ppm was deoxidized with an Fe–10%M ($M=\text{Ti}$, Al , Zr and Ce) and Ni–10%M ($M=\text{Ca}$ and Mg) alloys at 1600°C in an Al_2O_3 crucible using an induction furnace (100 kHz). A melt was cooled to the respective temperatures at $50\text{ K}\cdot\text{min}^{-1}$, followed by rapid quenching. The shape of a quenched sample was the frustum with the top (28 mm) and bottom diameters (16 mm) and height (20 mm). The vertically sliced sample was used for the observation of solidification structure and austenite grain. The solidification structure was observed after etching in an Oberhoffer solution and/or a saturated picric acid and CuCl_2 solution. The austenite grain size was measured after etching in 2%-Nital or a saturated picric acid and CuCl_2 solution. The mean primary arm length, $\bar{l}_1$, mean primary arm spacing, $\bar{\lambda}_1$, and density of dendrite were measured from solidification structure etched in an Oberhoffer solution and/or a saturated picric acid and CuCl_2 solution. The observation area was about 200 mm^2 , which corresponded to the half of the cross section area of vertically sliced sample. The observation number of dendrite was about 20 to 50. More details are given elsewhere.^{5,6)} The method of the particle size measurement is described elsewhere.⁷⁾

Table 1. Chemical composition and particle characteristics in Fe-0.15~0.50%C and Fe-10%Ni alloys.

Exp. No.	Deoxidant	Q.T. (°C)	$\bar{D}_A$ (μm)	$\bar{d}_A$ (μm)	N_A (mm ⁻²)	f_V × 10 ⁻⁴	insol. M. (ppm)	sol. M. (ppm)	M
Fe-0.15%C-1%Mn-1%Ni alloy									
1	without Ce	1470	1800	3.28	41	0.30	8	4	Al
2		1470	530	0.79	525	3.68	278	18	Ce
3	Mg	1500	—	—	—	1.43	40	35	Mg
4	Mg	1470	934	1.10	494	1.43	40	59	Mg
5	Ti/Mg	1470	701	0.83	1306	2.44	68	35	Mg
6	Ti	1500	—	—	—	0.29	12	217	Ti
7	Ti	1470	260	0.64	185	1.62	63	248	Ti
8	Zr	1500	—	—	—	3.85	221	24	Zr
9	Zr	1470	790	0.53	792	4.32	250	24	Zr
10	Ca	1500	—	—	—	1.46	23	17	Ca
11	Ca	1470	2000	0.90	535	1.00	32	4	Ca
Fe-0.20%C-0.02%P alloy									
12	Mg	1400	541	1.30	321	1.35	32	2	Mg
13	Mg	1400	1051	1.02	634	1.78	50	50	Mg
14	Mg	1400	1181	0.60	530	1.06	30	275	Mg
15	Zr	1400	225	2.38	248	3.94	205	23	Zr
16	Zr	1400	316	1.49	212	2.19	115	1	Zr
17	Zr	1400	356	1.94	238	5.26	305	2	Zr
18	Zr	1400	1038	2.33	1435	125	7251	81	Zr
19	Ce	1400	208	2.20	156	1.60	120	711	Ce
20	Ce	1400	323	2.08	81	0.84	63	244	Ce
21	Ce	1400	558	2.05	47	0.44	33	163	Ce
22	Ce	1400	275	2.13	135	1.06	80	536	Ce
23	Ce	1400	393	2.11	95	0.92	69	227	Ce
24	Ce	1400	820	2.18	57	0.61	46	142	Ce
Fe-0.30%C-1%Mn-1%Ni alloy									
25	Ce	1460	353	0.98	720	2.15	163	11	Ce
26	Mg	1460	915	1.14	514	1.54	43	10	Mg
27	Ti	1460	576	0.97	268	1.22	48	35	Ti
28	Zr	1460	—	0.80	206	3.40	197	7	Zr
Fe-0.50%C-1%Mn alloy									
29	without	1200	885	0.62	82	0.47	13	<1	Al
30	Zr	1200	330	0.90	967	2.61	150	2	Zr
31	Ce	1200	720	0.94	1039	0.51	39	114	Ce
32	Mg	1200	1800	1.08	905	1.49	42	91	Mg
Fe-10%Ni alloy									
33	Ce	1400	182	1.26	311	7.08	535	391	Ce
34*	Ce	1400	172	1.05	375	2.64	199	490	Ce
35**	Ce	1400	202	1.59	203	2.26	171	229	Ce
36	Ce	1400	433	1.35	73	4.30	325	2	Ce
37	Mg	1400	903	0.47	658	4.26	119	105	Mg
38	Mg	1400	1230	0.51	738	5.08	142	280	Mg
39*	Mg	1400	688	0.83	432	2.76	77	31	Mg
40*	Mg	1400	801	1.20	265	2.61	73	<1	Mg
41***	Mg	1400	552	1.58	124	1.68	47	1	Mg
42*	Mg	1400	753	0.65	738	1.86	52	53	Mg
43**	Mg	1400	477	0.74	335	1.86	52	56	Mg
44	Zr	1400	153	1.05	144	4.73	272	263	Zr
45**	Zr	1400	318	1.25	134	9.14	525	148	Zr
46***	Zr	1400	382	1.37	109	3.34	192	118	Zr
47	Zr	1400	110	1.12	342	16.37	941	299	Zr
48	Zr	1400	117	1.12	481	8.53	490	570	Zr
49	Zr	1400	93	0.97	492	11.24	646	1080	Zr

Q.T.: Temperature in water quenching

*, **, ***: 5, 30 and 60 min holding times at 1600 °C

—: not analyzed

Fig. 1. Solidification microstructure in Fe-10%Ni alloy deoxidized with Ce, Zr or Mg at 1873 K.

3. Results

The results for an Fe-10%Ni alloy deoxidized with Zr, Mg and Ce which are reported in previous study^{3,4)} are used in this study. The additional experimental results are given in **Table 1**, where quenching temperature, Q.T., average austenite grain size, $\bar{D}_A$, particle diameter in cross section, $\bar{d}_A$, particle number per unit volume, N_A , and volume fraction of particle, f_V , are summarized. The previous results^{5,6)} for an Fe-0.20%C-0.02%P alloy deoxidized with Ce are used in the present discussion. The f_V values are obtained from the chemically analyzed values for insol. M by using the method described elsewhere.⁷⁾ The experimental results for Fe-0.15, 0.20, 0.30 and 0.50%C alloy are summarized in Table 1.

4. Discussion

4.1. Refinement of Solidification Microstructure

4.1.1. Primary γ -phase Solidification

The effects of the number of primary deoxidation particles on area fraction of equiaxed crystals and their size have been studied in the experiments where an Fe-10mass%Ni alloy with γ -phase solidification was deoxidized with Ce, Zr or Mg at 1600°C and cooled to 1400°C at 50 K·min⁻¹, followed by rapid quenching. The solidification microstructure in this alloy consists of columnar dendrite and globular cell.^{3,8)} In **Fig. 1**, the area fraction of globular cell and its size are plotted against the number of oxide particles per unit area, N_A , in a cross section. It is seen from the upper diagram that the area fraction of globular cell for a given N_A increases in the order MgO, Ce₂O₃ and ZrO₂ which is in good agreement with that for the lattice misfit between γ -Fe and oxide,⁹⁾ as shown in the upper diagram of **Fig. 2**. The finding that the area fraction of globular cell for Mg deoxidation decreases with an increase of the number of MgO particles can be explained by the fact that dissolved Mg affects the development of columnar dendrite.

The result shown in the lower diagram of **Fig. 1** indicates

Fig. 2. Lattice misfit parameter between γ -Fe and oxide (upper diagram) and that between δ -Fe and oxide (lower diagram).

that the size of globular cell is about $150\ \mu\text{m}$, independent of N_A in the range of $N_A > 100\ \text{mm}^{-2}$ and irrespective of kind of oxides. It was found in the experiments where an Fe–10%Ni alloy was cooled to 1400°C that the size of globular cell is strongly dependent on cooling rate. From the results shown in Fig. 1, it follows that the area fraction of globular cell is influenced by the chemical composition of oxide, but the size of globular cell is independent of the number of oxide and its chemical composition. The particle to particle spacing, λ , for $N_A = 100\ \text{mm}^{-2}$ are estimated as $50\ \mu\text{m}$ from the relationship of $\lambda = 1/2N_A^{-1/2}$. When this result is compared with the mean size ($150\ \mu\text{m}$) of globular cell, it can be concluded that most of the particles distributed uniformly are not used for the heterogeneous nucleation.

An Fe–0.50%C–1%Mn alloy was deoxidized with Zr, Ce or Mg at 1600°C and cooled to 1400°C with $50\ \text{K}\cdot\text{min}^{-1}$, followed by rapid quenching, as shown in the lower diagram of Fig. 3. The $\bar{l}_1/\bar{\lambda}_1$ ratios (mean primary arm length/mean primary arm spacing) for ZrO₂ and Ce₂O₃ are 2 and 4, respectively, but that for MgO is considerably high (10), indicating the development of the columnar dendrite. The reason that the columnar dendrite develops with an increase of dissolved Mg is not clear at present. One of the possible reason for this is that the Mg vaporization at crucible wall causes the steep thermal gradient.

The number of columnar dendrite was measured in a cross section of the ingot and then the number of columnar dendrite per unit area is plotted against the lattice misfit parameter between γ -Fe and oxide in the upper diagram of Fig. 3. It can be seen that the number of dendrite per unit area increases with decreasing the misfit parameter, suggesting that the order of heterogeneous nucleation potency for γ -phase solidification is explained by the lattice misfit parameter.

The contact angle, θ , determined by the interfacial energy balance between oxide, γ -Fe and liquid Fe phases is represented by the following relation:

$$\cos \theta = (\gamma_{\text{MO/Fe(l)}} - \gamma_{\text{MO/Fe(}\gamma)}) / \gamma_{\text{Fe(l)/Fe(}\gamma)} \dots \dots \dots (1)$$

Although the results shown in Fig. 3 can be explained by the lattice misfit parameter between oxide (MO) and γ -Fe,

Fig. 3. Number of columnar dendrite and $\bar{l}_1/\bar{\lambda}_1$ ratio plotted against lattice misfit parameter between γ -Fe and oxide.

Fig. 4. Effect of dissolved Ce, Zr of Mg content on solidification microstructure in Fe–0.20%C–0.02%P alloy.

these results should be properly interpreted from the values of contact angle. However, the $\gamma_{\text{MO/Fe(l)}}$ and $\gamma_{\text{MO/Fe(}\gamma)}$ values in the present study are not available and the $\gamma_{\text{Fe(l)/Fe(}\gamma)}$ value is supposed to be dependent on the content of deoxidant.

4.1.2. Primary δ -phase solidification

The effect of dissolved solute M ($M = \text{Ce, Zr and Mg}$) on the solidification microstructure has been studied in the experiments⁵⁾ where an Fe–0.20%C–0.02%P alloy with primary δ -phase solidification was deoxidized with Ce, Zr or Mg at 1600°C and cooled to 1400°C at $50\ \text{K}\cdot\text{min}^{-1}$, followed by rapid quenching. The relationship between the $\bar{l}_1/\bar{\lambda}_1$ ratio and the content of dissolved M is shown in Fig. 4. This ratio for Ce deoxidation is 2 independent of the dissolved Ce content and that for Zr deoxidation is 3 to 4. However, that for Mg deoxidation increases with increasing the dissolved Mg content.

In a previous study,⁵⁾ it was observed that the mean primary and secondary dendrite arm spacings decrease with an increase in the dissolved Ce content. The present results coupled with previous one⁵⁾ suggest that the dendrite arm length also decreases with the dissolved Ce content. In a

Fig. 5. $\bar{l}_1/\bar{\lambda}_1$ ratio and $\bar{l}_1$ values plotted against lattice misfit parameter between δ -Fe and oxide in Fe-0.15%C, 0.30%C-1%Mn-1%Ni alloy.

previous study,⁵⁾ it was found that the area fraction of equiaxed crystals increases with an increase of Ce_2O_3 or CeS particles. The lattice misfit parameter between δ -Fe and Ce_2O_3 shown in the lower diagram of Fig. 2 is small and nearly the same as that between δ -Fe and TiN in which the heterogeneous nucleation of δ -Fe is experimentally confirmed.¹⁰⁾

An Fe-0.15 or 0.30%C-1%Mn-1%Ni alloy was deoxidized at 1600°C with Ti, Ce, Mg or Zr and cooled to 1470°C ($C=0.15\%$) and 1460°C ($C=0.30\%$) at $50\text{ K}\cdot\text{min}^{-1}$, followed by rapid quenching. These quenching temperatures determined by the cooling curves correspond to the start of the single γ -phase, namely, the temperatures in the single γ -phase just below the $\delta+\gamma$ and $\gamma+\text{liquid}$ two phases, respectively. For the purpose of revealing the solidification microstructure clearly, one % Ni was added in this study.

The $\bar{l}_1/\bar{\lambda}_1$ ratio and $\bar{l}_1$ values are plotted against the lattice misfit parameter between δ -Fe and oxide in Fig. 5, where $\bar{l}_1$ and $\bar{\lambda}_1$ represent the mean columnar dendrite arm length and its spacing, respectively. It can be seen that the $\bar{l}_1/\bar{\lambda}_1$ ratio decreases with decreasing the lattice misfit parameter, indicating that the equiaxed crystallization for Ce_2O_3 and MgO is more favorable than that for ZrO_2 . The reason that the data for Ti_2O_3 deviated from the line may be explained by the fact that number of primary deoxidation particles of TiO_x is considerably small and TiO_x particles together with TiC particles are observed in the interdendritic region.

The number of primary δ -grains which correspond to the columnar dendrite was measured in a cross section of an Fe-0.15%C-1%Mn-1%Ni alloy. This sample was obtained by quenching at 1500°C which is the liquid and δ -Fe two phases region. The number density of δ -grains is plotted against the lattice misfit parameter between δ -Fe and oxide in the lower diagram of Fig. 6. It can be seen that the number of density of δ -grains tends to increase with decreasing the misfit parameter, suggesting that the nucleation rate for

Fig. 6. Number of δ -grains plotted against number of oxide particles (upper diagram) and misfit parameter between δ -Fe and oxide (lower diagram) in Fe-0.15%C-1%Mn-1%Ni alloy.

solidification become greater with increasing the nucleation potency represented by the misfit parameter. As shown in the upper diagram, the density of δ -grains is independent of the number of oxide particles per unit area.

As mentioned previously in the explanation of the results in Fig. 3, the results in Fig. 6 are also explained by the following relation with respect to the contact angle between oxide, $\text{Fe}(\delta)$ and $\text{Fe}(\text{l})$ three phases.

$$\cos \theta = (\gamma_{\text{MO}/\text{Fe}(\text{l})} - \gamma_{\text{MO}/\text{Fe}(\delta)}) / \gamma_{\text{Fe}(\text{l})/\text{Fe}(\delta)} \dots \dots \dots (2)$$

In order to have the high value of $\cos \theta$ in which the rate of heterogeneous nucleation is high, not only low value of $\gamma_{\text{MO}/\text{Fe}(\delta)}$ which corresponds to the lattice misfit parameter between MO and δ -Fe, but also high value of $\gamma_{\text{MO}/\text{Fe}(\text{l})}$ is desirable.

4.2. Correspondence between Solidification Microstructure and Initial Austenite Grain

4.2.1. Single-phase Solidification

It is well known in a single-phase solidification that the columnar dendrites with their major axes aligned in the same direction correspond to the initial austenite or δ -ferrite grain, if the grain growth is completely prohibited by pinning and/or solute drag. The correspondence between single-phase solidification microstructure and initial austenite grain is discussed in the following.

Fe-10%Ni alloy: An Fe-10%Ni alloy was deoxidized with Ce or Zr at 1600°C and cooled to 1400°C at $50\text{ K}\cdot\text{min}^{-1}$, followed by rapid quenching. Solidification microstructure of globular cell ($\bar{d}_A$) and the austenite grain size ($\bar{D}_A$) were measured as a function of N_A and dissolved M ($M=\text{Ce}$ and Zr) contents. The results are shown in Fig. 7, indicating that the $\bar{D}_A/\bar{d}_A$ ratio is nearly unity in the range of $N_A > 150\text{ mm}^{-2}$ and $[\text{sol. M}] > 0.02\%$. Under the present cooling condition it is said that the austenite grain growth is completely prevented by pinning and solute drag effects, resulting in keeping the size of solidification microstructure.

Fe-0.50%C alloy: In the upper diagram of Fig. 8, the $\bar{D}_A$

Fig. 7. Correspondence between solidification microstructure and austenite grain in Fe-10%Ni alloy of single-phase solidification.

Fig. 8. $\bar{D}_A$ and Number of γ -grains/number of δ -grains ratio plotted against lattice misfit parameter between γ -Fe and oxide in Fe-0.50%C-1%Mn alloy.

values are plotted against the lattice misfit parameter between γ -Fe and oxide in an Fe-0.50%C-1%Mn alloy, which was cooled to 1200°C at 50 K·min⁻¹. It is seen that the $\bar{D}_A$ values decrease with decreasing the misfit parameter. The reason that the $\bar{D}_A$ value for MgO greater than that obtained without deoxidation particles (a melt was held for 30 min after Al addition) can be explained by the effect of dissolved Mg on the development of columnar dendrite as mentioned previously. If it is assumed that the austenite grain growth is completely prohibited by particle pinning, this finding can be explained by the fact that in single-phase solidification the solidification microstructure which is influenced by the degree of the lattice misfit parameter between γ -Fe and oxide corresponds to the initial austenite grain.

This was confirmed by the plot shown in the lower diagram of Fig. 8, in which the ratio of the number of γ -grains

Fig. 9. Relationships between $\bar{D}_A$ and $\bar{\lambda}_2$ (upper diagram) and that between $\bar{D}_A$ and $\bar{\lambda}_1$ (lower diagram) in Fe-0.15, 0.30%C-1%Mn-1%Ni alloys.

to the number of δ -grains per unit area is plotted against the lattice misfit parameter. These ratios obtained by Zr, Ce and Mg deoxidations are nearly unity and independent of the misfit parameter. It can be concluded that in single phase solidification the solidification microstructure is influenced by the chemical composition of oxide with different nucleation potency, but the solidification microstructure corresponds to the austenite grains under the complete inhibition of grain growth.

4.2.2. Two-phases Solidification

In this section the effects of oxide particles on the heterogeneous nucleation site for the δ to γ transformation and the austenite grain growth are studied in an Fe-0.15% or 0.30%C-1%Mn-1%Ni alloy. The deoxidation particles dispersed uniformly in microsegregation domain are used for the γ -phase nucleation in δ -phase matrix in an Fe-C (<0.1%) alloy. In the case of Fe-C (0.15 or 0.30%) alloy, however, the particles located at δ /liquid interface where the peritectic reaction occurs are only used for the heterogeneous nucleation site. The place at which the peritectic reaction occurs approaches low fraction of solid with decreasing carbon content. It should be noted that the correspondence between the interdendritic region and austenite grain boundaries is different at hypo and hyper peritectic regions.

The mean austenite grain size, $\bar{D}_A$, obtained in an Fe-0.15 or 0.30%C-1%Mn-1%Ni alloy which was quenched at 1470°C (0.15% C) and 1460°C (0.30% C) is plotted against the mean values for the secondary dendrite arm spacing, $\bar{\lambda}_2$, and the primary dendrite arm spacing, $\bar{\lambda}_1$, in the upper and lower diagrams of Fig. 9, respectively. It can be seen that the $\bar{D}_A$ values vary considerably from 200 to 1000 μ m, but the $\bar{\lambda}_1$ and $\bar{\lambda}_2$ values are nearly constant.

The aspect ratio of austenite grain and the geometric standard deviation in log-normal distribution of grain size, $\ln \sigma$, are plotted against the $\bar{l}_1/\bar{\lambda}_1$ ratio in the upper and lower diagrams of Fig. 10, respectively. It can be seen from

Fig. 10. Aspect ratio of grain and $\ln \sigma$ plotted against $\bar{l}_1/\bar{\lambda}_1$ in Fe-0.15, 0.30%C-1%Mn-1%Ni alloy.

the upper diagram that the morphology of austenite grain is more or less equiaxed, while that of the δ -grains is columnar. This is clear from the comparison between the aspect of grain and $\bar{l}_1/\bar{\lambda}_1$ ratio. The $\bar{l}_1/\bar{\lambda}_1$ ratio in the Zr deoxidation is significantly higher than those in other deoxidations. This is probably due to the fact that the morphology of δ -grains is strongly influenced by the content of dissolved deoxidant, in particular, the dissolved Zr content. In the Mg deoxidation experiment dissolved Mg content was minimized by holding at 1600°C for 10 min to vaporize. It is seen from the lower diagram that the spread of grain size distribution is greater in the Mg deoxidation. Although the reason for this is not certain, it was experimentally observed that the $\ln \sigma$ value in the Mg deoxidation increases with an increase of dissolved Mg content.

The grain size distributions observed in an Fe-0.15%C-1%Mn-1%Ni alloy are shown in Fig. 11. It is seen that the grain size distribution in the Mg deoxidation is bi-modal and the peak observed in larger grain size is due to the effect of dissolved Mg. If dissolved Mg is removed by evaporation and its content approaches zero, no bi-modal distribution was observed. The mode value of the grain size distribution observed in the Ti deoxidation is smaller compared with those observed in other deoxidations. It was found in the Ti deoxidation experiments by using the microprobe analysis that TiO_x and TiC particles are precipitated in the interdendritic space in an Fe-0.15 or 0.30%C alloy, not in an Fe-0.05%C alloy. It is considered that these precipitates effectively pin the austenite grain growth and/or become the candidate for the heterogeneous nucleation site for the δ to γ transformation.

The mean austenite grain sizes observed in the Ti, Zr, Ce, Mg and Ti/Mg deoxidations are plotted against the lattice misfit parameter between γ -Fe and oxide in Fig. 12, together with the results obtained in the Ca deoxidation and without deoxidation. It is clear that the $\bar{D}_A$ values decrease with a decrease in the misfit parameter. The results shown in Fig. 12 were obtained in the interrupted experiment where a sample was quenched immediately at the highest temperature of the austenite single phase. Therefore, it is reasonable to assume that the austenite grain growth is

Fig. 11. Grain size distribution in Fe-0.15%C-1%Mn-1%Ni alloy deoxidized with various deoxidants.

Fig. 12. $\bar{D}_A$ plotted against lattice misfit parameter between γ -Fe and oxide.

pinned during the cooling by the presence of coexisting δ -phase in the δ and γ two phases region. If the number density of γ -grains during the two phases is constant, it is considered that the $\bar{D}_A$ values shown in Fig. 12 correspond to the initial number density of γ -grains after δ/γ transformation. In this study the heterogeneous nucleation rate of γ -phase for different oxides are represented by the lattice misfit parameter between γ -Fe and oxide.

In order to study the effect of oxide particles on the grain growth inhibition, the $\bar{D}_A$ values are plotted against the Zener pinning force, Z_p , which is given by

$$Z_p = 3 \sigma \cdot V \cdot f_v / \bar{d} \dots \dots \dots (3)$$

where σ is the grain boundary energy ($6 \times 10^{-7} \text{ J} \cdot \text{mm}^{-2}$), V is the molar volume of Fe ($7 \times 10^3 \text{ mm}^3 \cdot \text{mol}^{-1}$), and f_v and $\bar{d}$ are the volume fraction and the mean particle diameter, respectively.

It can be seen from Fig. 13 that the $\bar{D}_A$ values decrease with an increase in the Z_p value, indicating that the order of the $\bar{D}_A$ values observed in different deoxidations can be explained by the different degree of the pinning force.

Fig. 13. $\bar{D}_A$ plotted against Zener pinning force in Fe-0.15%C-1%Mn-1%Ni.

Therefore, the observed behavior of the grain size distribution shown in Fig. 11 can be interpreted by both nucleation rate and grain growth inhibition.

The number of columnar dendrite per unit area in a cross section of an Fe-0.15%C-1%Mn-1%Ni alloy was measured in the experiments quenched at 1500°C and that of the austenite grain per unit area was measured in the experiments quenched at 1470°C. The ratios of the number of γ -grains to that of δ -grains per unit area are plotted against the lattice misfit parameter between γ -Fe and oxide in the upper diagram of Fig. 14. The finding that this ratio increases with a decrease in the misfit parameter indicates that the nucleation rate is higher with increasing the nucleation potency. In other words, more than one nucleation event per one δ -grain occurs at the δ /liquid interface.

The results in the lower diagram of Fig. 14 should be explained by the following relation with respect to the contact angle between oxide, Fe(δ) and Fe(γ) three phases.

$$\cos \theta = (\gamma_{\text{MO/Fe}(\delta)} - \gamma_{\text{MO/Fe}(\gamma)}) / \gamma_{\text{Fe}(\delta)/\text{Fe}(\gamma)} \dots \dots \dots (4)$$

In order to have the high rate of γ -phase nucleation at δ /liquid interface, namely, high value of $\cos \theta$, not only low value of $\gamma_{\text{MO/Fe}(\gamma)}$ which corresponds to the lattice misfit parameter between MO and γ -Fe, but also high value of $\gamma_{\text{MO/Fe}(\delta)}$ is necessary.

It can be seen from the upper diagram that this ratio does not increase with an increase in the number of particles per unit area, N_A , indicating that the nucleation rate of γ -phase is not influenced by the number of nucleation site.

5. Conclusions

The effect of deoxidation particles on the γ -or δ -phase solidification microstructure has been studied in Fe-10%Ni and Fe-0.50%-1%Mn alloys. It was found that solidifica-

Fig. 14. Number of γ -grains to number of δ -grains per unit area plotted against N_A (upper diagram) and lattice misfit parameter between γ -Fe and oxide (lower diagram).

tion microstructure for a given cooling rate is influenced by the chemical composition of oxide and the degrees of equiaxed crystallization in γ - and δ -phase solidifications are well related to the lattice misfit parameter between γ -Fe and oxide and that between δ -Fe and oxide, respectively.

The correspondence between solidification microstructure and initial austenite grains has been studied by using the interrupted solidification method coupled with the grain growth inhibition by pinning. As a result, under the condition that the grain growth is completely retarded by pinning and/or solute drag, it was confirmed that initial austenite grains correspond to the solidification microstructure in γ -phase single solidification. In two-phases solidification of an Fe-0.15 or 0.30%C alloy, the correspondence between solidification microstructure and initial austenite grains in the presence of oxide particles is explained by the nucleation potency for the δ to γ transformation and/or the grain growth inhibition.

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