1. Introduction

In previous study\(^1\) the behavior of deoxidation particles at solidification interface has been studied as a function of oxide composition and solidification rate, and the results for the particle engulfment and pushing are discussed by considering the force acting on the particles at the solid/liquid interface using the interfacial energies. It was observed that only Al\(_2\)O\(_3\) particles are pushed to the region of final solidification with decreasing the cooling rate. This was explained by the difference between the oxide-liquid Fe interfacial energy and the oxide-solid Fe interfacial energy.\(^1\) The interaction of oxide particles with advancing solid/liquid interface is influenced by the interface active solute such as sulfur and oxygen which reduce significantly the surface energy of liquid Fe even in the presence of small amount. Hence, it is considered that the particle engulfment and pushing transition is influenced by interface active solute. However, no systematic study on this subject has been made experimentally to date.

The particle behavior at solid/liquid interface has been theoretically investigated by Stefanescu et al.\(^2\) and the critical velocity of the particle engulfment and pushing transition (EPT) has been estimated. Shibata et al.\(^3\) performed in-situ real time observation of the interaction of inclusions with the advancing solid/liquid interface by using a confocal scanning laser microscope and determined the velocity for EPT as a function of particle radius. Smaller particles at low solidification velocity tend to be pushed to liquid phase from the advancing solid/liquid interface, but under normal continuous casting condition small particles such as around 1 mm are not pushed out. It is expected, however, in the presence of interface active solute that smaller particles may be pushed even under normal continuous casting condition, since the critical velocity for EPT increases with increasing the content of interface active solute.

Mukai and Lin\(^4\) considered the force acting on a sphere moving towards a solidification front due to an interfacial energy gradient at the particle/liquid interface. Based on their model the effect of Ti,\(^5\) S and O\(^6\) contents on adhesion of inclusion to solidification shell in continuous casting of steel has been studied.

The purpose of this article is to study the effect of sulfur or oxygen on the dispersion of fine deoxidation particles. For this purpose an Fe–10mass%Ni alloy was deoxidized with Zr or Al under a given content of sulfur or oxygen and the dispersion of Zr\(_2\)O\(_3\) and Al\(_2\)O\(_3\) particles in microsegregation domain has been observed as a function of interface active solute content.

2. Experimental

2.1. Procedure

An induction furnace (100 kHz) installed a graphite susceptor with the thickness of 20 mm was used in order to eliminate the melt stirring. An Fe–10mass%Ni alloy (70 g) containing initial oxygen content of 100 to 120 mass ppm was deoxidized with an Fe–10%Zr or Fe–10%Al alloy at 1873 K (% and ppm represent mass% and mass ppm, respectively, hereinafter). Then, an Fe–10%S alloy was added...
in an appropriate proportion to control the sulfur content and a sample was cooled from 1873 to 1673 K at the average cooling rate of 5, 10 and 50 K·min⁻¹, followed by rapid quenching. The solidification velocities for these average cooling rates are estimated from the secondary dendrite arm spacing and local solidification time, which are discussed in Sec. 3.1.2. For the purpose of controlling the dissolved oxygen content, Fe₃O₄ whose amount corresponds to the oxygen content of 300 ppm was added before deoxidant addition and a sample was cooled to 1673 K at the rate of 50 K·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 the height (20 mm). The quenched sample was sliced vertically with 2–3 mm thickness and these sliced samples in the region of 7 mm away from the crucible side were used for chemical analysis and observation of inclusion. The final solidification part which was located about 3 mm from the top was not used for the chemical analysis. Detailed descriptions of experimental procedure are given in the previously cited article.⑦

2.2. Chemical Analysis
The method for soluble and insoluble M (M=Al and Zr) and that for total oxygen analysis are already explained elsewhere.⑦,⑧ The content of sulfur and carbon was analyzed by fusion-infrared absorptiometry.

2.3. Estimation of Particle Size and Number
The number of particles per unit area, N, and two-dimensional particle diameter, dA, for the particles in a polished cross section were measured by a SEM at a magnification of 1000. The areas from 7 and 5 mm from top and bottom of samples, respectively, were observed for inclusion characteristics. The observed area of each position was 0.25 mm² which corresponds to the 24 observation areas consisting of the continuous 6 observation areas to the horizontal direction and the continuous 4 observation areas to the vertical direction. More detailed explanation is given elsewhere.⑦ The particle section with the size above 0.5 μm can be measured by this method which is denoted by the cross section method in this article. The number of spatial particles per unit area extracted by acetylcellulose film was measured by a SEM at a magnification of 20000 using the following technique: The polished surface was slightly etched using electrolytic extraction and then particles were extracted by acetylcellulose film. In this method the particles with the size of greater than 0.01 μm can be measured and this method is denoted by the AC film method in this article. More detail is given elsewhere.⑦

2.4. Particle Dispersion in Microsegregation Domain
The behavior of particle dispersion during solidification was studied by the following method: When the solidification structure is revealed with Oberhöffer solution, the inclusion particles cannot be measured. Therefore, a mark was made by a Vickers hardness tester at four corners of 500 μm×400 μm square prior to etching. The correlation between particle dispersion and solidification microstructure was studied by superimposing the photographs taken before and after etching at the marked region of a cross section. The fraction of solid at globular crystals was determined by the method described elsewhere.① Particle lying in the microsegregation domain of dendritic and non-dendritic solidification were measured by the method described elsewhere.①

3. Results and Discussion

3.1. Models for Engulfment and Pushing of Particles
It has been well established that the particle behavior at solid/liquid interface is influenced by the interfacial energy between particle and liquid Fe, γPL. The behavior of particles arising from the variation of the γPL value with interface active solute content of sulfur or oxygen is discussed based on the previous model⑨,⑩ concerning the particle engulfment and pushing.

The surface energy of liquid Fe, γL, is expressed as a function of oxygen and sulfur contents by the following equation⑪:

\[ γ_L = (1910 - 825 \log(1 + 210\%O)) - 540 \log(1 + 185\%S) \] ...........................................(1)

The γPL value is obtained by the Young relation as

\[ γ_{PL} = γ_L - γ_p \cos θ_{PL} \] .........................(2)

where γp is the surface energy of particle and θPL is the contact angle between particle and liquid Fe.

The γL and γAl₂O₃–Fe₃O₄ values obtained from Eqs. (1) and (2), respectively, by using the parameters given in Table 1 are plotted against sulfur content in the upper diagram of Fig. 1. It is seen that as a result of the decrease in γL with

| Table 1. Values used for the estimation of critical velocity for engulfment/pushing transition. |
|---------------------------------|----------------|
| Parameter | Value |
| a₀ | 2.5·10⁻¹⁰ m |
| η | 0.0043 kg·m⁻¹·s⁻¹ |
| Kₚ | Kₚ / K₁ |
| Kₚ | 5.5 W·m⁻¹·K⁻¹ |
| K₁ | 45 W·m⁻¹·K⁻¹ |
| γL | 1910 - 825 \log(1 + 210\%O) - 540 \log(1 + 185\%S) | mJ·m⁻² |
| γp | \Al₂O₃ = 750 mJ·m⁻², ZrO₂ = 620 mJ·m⁻² |
| θPL | Contact angle |
| Al₂O₃ = 144°, ZrO₂ = 122° |
| γPL | Interfacial energy between particle and liquid Fe |
| γp - γL \cos θPL |
| γPS | Interfacial energy between particle and solid Fe |
| Al₂O₃ = 2480 mJ·m⁻², ZrO₂ = 1760 mJ·m⁻² |
increasing sulfur content, the $\gamma_{Al_2O_3-Fe(L)}$ value decreases, as is clear from Eq. (1) where it is assumed that the contact angle, $\theta_{PL}$, is independent of sulfur content.

3.1.1. Wetting/Interfacial Energy

According to the Wetting/Interfacial energy model proposed by Wu and Nakae, the contact angle, $\theta_{PS}$, between solid Fe/liquid Fe and particle/solid Fe interfaces is given by

$$\cos\theta_{PS} = \frac{\gamma_{PL} - \gamma_{PS}}{\gamma_{SL}} \tag{3}$$

where $\gamma_{PL}$ and $\gamma_{PS}$ are the interfacial energies between particle and liquid Fe and between particle and solid Fe, respectively, and $\gamma_{SL}$ is the interfacial energy between solid Fe and liquid Fe. When $\gamma_{PL}$ is greater than $\gamma_{PS}$, that is, $\theta_{PL} < 90^\circ$ in Eq. (3), particles are easily engulfed to solid Fe. This is because particles tend to combine with solid Fe. Similarly, when $\gamma_{PL}$ is smaller than $\gamma_{PS}$ which means $\theta_{PL} > 90^\circ$, particles are pushed to liquid Fe. The $\theta_{PS}$ values were estimated from Eq. (3) by using the $\gamma_{PL}$, $\gamma_{PS}$ and $\gamma_{SL}$ for $Al_2O_3$ and $ZrO_2$ particles given in Table 1 and the results are shown in the lower diagram of Fig. 1. The calculated results shown in the lower diagram suggest that particles are not completely wetted in the range of $\cos\theta_{PS} < -1$. It is obvious that the $Al_2O_3$ and $ZrO_2$ particles are pushed even in the range of a very low sulfur content and these particles tend to be pushed more strongly with an increase in sulfur content.

3.1.2. Critical Velocity for Particle Engagement and Pushing Transition

The critical velocity for particle engulfment and pushing transition has been theoretically derived by Stefanescu and Catalina as follows;

$$V_{cr} = \left( \frac{\Delta\gamma_o \cdot d_o^2}{3 \cdot \eta \cdot k \cdot R} \right)^{1/2} \tag{4}$$

with $\Delta\gamma_o$ as

$$\Delta\gamma_o = \gamma_{PS} - \gamma_{PL} \tag{5}$$

where $k$ is the ratio of $k_p/k_i$ in which $k_p$ and $k_i$ are the thermal conductivities of oxide particle and liquid Fe, respectively, $\eta$ is the viscosity of liquid Fe, $d_o$ is the atomic distance and $R$ is the particle radius.

On substituting the respective parameters given in Table 1 into Eq. (4), the critical velocity, $V_{cr}$, at $R = 0.5$ and 5 $\mu m$ for $ZrO_2$ particles and at that $R = 0.5$, 5 and 50 $\mu m$ for $Al_2O_3$ particles are estimated as a function of sulfur content and the results are demonstrated in Fig. 2, along with the experimental data explained in Sec. 3.2. The solidification velocities at $T = 5$, 10 and 50 $K/min$ in the present study were estimated from the relation: $V = \lambda_2/\gamma_o$, where $\gamma$ and $\lambda_2$ are the local solidification time and the secondary dendrite arm spacing, as 1.60, 2.88 and 5.50 $\mu m/min$, respectively. It can be seen that the experimental results are in good agreement with the calculated line at $R = 0.5 \mu m$, as shown in the lower diagram. In the case of $ZrO_2$ particles, however, the experimental results are not in good agreement with the calculated line. Although the reason for this is not certain, the effect of sulfur enrichment due to the microsegregation during solidification should be taken into consideration.

The $V_{cr}$ value for $Al_2O_3$ particles was calculated from Eq. (4) as a function of particle diameter at sulfur contents of 0.01 and 1% and oxygen content of 0.01% by using the $\gamma_{GL}$ and $\gamma_{PL}$ values in Eqs. (1) and (2), together with the parameters given in Table 1. In this calculation the effect of sulfur enrichment due to the microsegregation during solidification should be taken into consideration.

The $V_{cr}$ value is relatively small in the range of particle diameter, $d > 2–3 \mu m$, but its effect is markedly in-
creased in the range of \( d<1 \mu m \).

### 3.2. Effect of Sulfur on Engulfment and Pushing of ZrO\(_2\) and Al\(_2\)O\(_3\) Particles

For the purpose of studying the effect of interface active solutes on particle EPT, an Fe–10%Ni alloy was deoxidized with Al or Zr at 1873 K, and cooled to 1673 K at 5, 10, and 50 K·min\(^{-1}\), followed by rapid quenching. Chemical compositions of metal phase are summarized in Table 2. The number of particles per unit area, \( N_A \), and mean particle size, \( d_bar \), were measured in a polished cross section. In this cross section method the particle diameter greater than 0.5 \( \mu m \) can be measured. By using the method described in Sec. 2.4, the particle dispersion in microsegregation domain was also observed. The number of particles per unit area of acetylcellulose film whose particle diameter greater than 0.01 \( \mu m \) was measured by the acetylcellulose film extraction technique, as described in Sec. 2.4. Similarly, the particle dispersion in microsegregation domain was also observed by this AC film method.

The \( d_bar \) and \( N_A \) values for ZrO\(_2\) and Al\(_2\)O\(_3\) particles observed by the cross section method are plotted against dissolved sulfur content in Figs. 4 and 5, respectively, for three different cooling rates. It can be recognized that total sulfur is equal to dissolved sulfur because of no formation of sulfide in the present experiments. As shown in the upper diagrams of Figs. 4 and 5, the \( d_bar \) and \( N_A \) values for ZrO\(_2\) particles at 50 K·min\(^{-1}\) become larger and smaller, respectively, in the range of [ppm S] > 300–400. As explained in detail in Sec. 3.3, it was found that the ZrO\(_2\) particles are pushed to the region of final solidification at [ppm S] > 300–400. The \( d_bar \) and \( N_A \) values for Al\(_2\)O\(_3\) particles at 50 K·min\(^{-1}\) become smaller and larger, respectively, in the range of [ppm S] > 100, as shown in the lower diagrams of Figs. 4 and 5. It is demonstrated experimentally that the sulfur content at EPT tends to increase with an increase in the cooling rate.

The reduction of the \( N_A \) values with decreasing cooling rate, as shown in Fig. 5, can be interpreted by the fact that the time for particles to grow by Ostwald ripening becomes longer, thus resulting in the decrease in \( N_A \) and the increase in \( d_bar \). As explained later in this section, the 80% of the total volume of particles is pushed to the region of the final solidification at [ppm S] > 300–400 for ZrO\(_2\) particles and [ppm S] > about 100 for Al\(_2\)O\(_3\) particles. This difference of

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**Table 2. Chemical compositions of metal phase in the experiments of Fe–10mass%Ni alloy.**

| Cooling rate (K·min\(^{-1}\)) | [T. S.] (Mass ppm) | [T.O] (Mass ppm) | Sol. | Insol. |
|-----------------------------|-------------------|------------------|------|-------|
| ZrO\(_2\) (M = Zr)          |                   |                  |      |       |
| 50                          | 6                 | 69               | 92.2 | 301   |
| 50                          | 98                | 107              | 91.3 | 342   |
| 50                          | 210               | 95               | 91.9 | 313   |
| 50                          | 435               | 160              | 80.5 | 305   |
| 10                          | 5                 | 110              | 87.2 | 315   |
| 10                          | 105               | 117              | 81.9 | 324   |
| 10                          | 390               | 138              | 91.9 | 319   |
| 5                           | 6                 | 82               | 95.5 | 273   |
| 5                           | 106               | 112              | 99.2 | 392   |
| 5                           | 296               | 106              | 83.8 | 238   |
| 5                           | 404               | 169              | 88.9 | 389   |
| *50                         | 9                 | 378              | 1.7  | 117   |

| Al\(_2\)O\(_3\) (M = Al)    |                   |                  |      |       |
|-----------------------------|-------------------|------------------|------|-------|
| 50                          | 8                 | 78               | 261  | 106   |
| 50                          | 102               | 74               | 201  | 97.0  |
| 50                          | 189               | 65               | 273  | 111   |
| 50                          | 362               | 79               | 255  | 93.6  |
| 10                          | 6                 | 41               | 276  | 76.0  |
| 10                          | 107               | 33               | 274  | 34.6  |
| 10                          | 209               | 23               | 255  | 28.6  |
| *50                         | 9                 | 233              | 30.1 | 155   |
| *50                         | 2                 | 266              | 55.2 | 145   |

* : Fe\(_3\)O addition

**Fig. 3.** Relationship between critical velocity and diameter of Al\(_2\)O\(_3\) particle as a function of sulfur and oxygen contents. Shadow area represents the present experimental condition.

**Fig. 4.** Effect of cooling rate on the relation between \( d_bar \) and dissolved S for ZrO\(_2\) (upper diagram) and Al\(_2\)O\(_3\) (lower diagram) particles observed in cross section.
sulfur level for EPT can be explained by the difference in the interfacial energy between ZrO\textsubscript{2} and liquid Fe (1 630 mJ·m\textsuperscript{-2}) and that between Al\textsubscript{2}O\textsubscript{3} and liquid Fe (2 290 mJ·m\textsuperscript{-2}). That is, the sulfur level at EPT for Al\textsubscript{2}O\textsubscript{3} particles is lower than that for ZrO\textsubscript{2} particles due to the higher interfacial energy between Al\textsubscript{2}O\textsubscript{3} and liquid Fe.

The numbers of ZrO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} particles per unit area of acetylcellulose film are plotted against dissolved sulfur content in Fig. 6 for different cooling rates. The number of particles above 0.01 μm becomes smaller in the range of [ppm S]=200–300 for ZrO\textsubscript{2} particles and [ppm S]>about 50 for Al\textsubscript{2}O\textsubscript{3} particles. In the estimation of \(N_A\) by the AC film method, the particles not only on surface of the specimen, but also the particles located at the distance of 10 to 20 μm below the surface are extracted by the acetylcellulose film. For this reason, the \(N_A\) values obtained by the AC film method tend to be always overestimated and if the depth which is determined by etching during the electrolytic extraction is accurately measured, the \(N_V\) values can be obtained from this AC film method.

For the purpose of studying the particle EPT, the particle size distributions and the dispersion in microsegregation domain obtained at high sulfur content are compared with those obtained at very low sulfur content in the following.

3.2.1. Particle Size Distribution

The size distributions of ZrO\textsubscript{2} particles obtained at low ([ppm S]=6) and high sulfur contents ([ppm S]=435) are shown in Fig. 7. The increase of larger particle size of \(d_A\) to 5 μm and the decrease of smaller particle size of \(d_A<2 \mu m\) are observed at high sulfur content, as shown in the upper diagram obtained by the cross section method. These results suggest that particles are pushed to the region of final solidification, followed by the coagulation of particles by collision. It can be seen from the lower diagram obtained by the AC film method that the modal values of \(d_A\) at low and high sulfur contents are nearly the same and the number of these small particles becomes smaller at high sulfur level. The size distributions of Al\textsubscript{2}O\textsubscript{3} particles obtained by both methods are shown in Fig. 8, in which a similar trend observed for ZrO\textsubscript{2} particles is obtained.

3.2.2. Dispersion in Microsegregation Domain

The effect of sulfur on the dispersion of ZrO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} particles in microsegregation domain has been studied by plotting the \(N_A\) values obtained by the cross section and AC film methods against fraction of solid, \(f_S\), in Figs. 9 and 10, respectively. The method for the determination of \(f_S\) is de-
scribed in detail elsewhere.\textsuperscript{1)} As shown in the upper diagrams of Figs. 9 and 10 which were obtained by the cross section method, the ZrO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} particles are pushed to the region of final solidification at high sulfur level. The fine particles of ZrO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} obtained by the AC film method shown in the lower diagrams are dispersed uniformly in the microsegregation domain. However, the number of particles at high sulfur level is smaller than that at low sulfur level, thus indicating the occurrence of particle pushing at high sulfur level. The reason that the $N_A$ values for fine particles at high sulfur level are independent of $f_S$ is explained by the fact that the fine particles are also pushed to the region of final solidification at high sulfur level where the particle coagulation takes place. However, such coagulated large particles are difficult to be measured by the AC film method, since the observed area is limited due to high magnification in this method.

### 3.3. Effect of Oxygen on Engulfment and Pushing of ZrO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} Particles

In order to study the effect of oxygen on engulfment and pushing of particles, an Fe–10\%Ni alloy was deoxidized with Zr or Al at 1873 K and then Fe\textsubscript{2}O\textsubscript{3} was added to control the dissolved oxygen content, followed by cooling to 1673 K at 50 K·min\textsuperscript{-1}. The numbers of ZrO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} particles obtained by the cross section and AC film methods are plotted against total oxygen content in the upper and lower diagrams of Fig. 11, respectively. The data points above 200 ppm oxygen correspond to the results obtained by the addition of Fe\textsubscript{2}O\textsubscript{3}. The $N_A$ values for Zr or Al deoxidation observed by the cross section and AC film methods become smaller in the range of [ppm total O]$>100$–200. The compositions of particles after solidification in the experiments of Fe\textsubscript{2}O\textsubscript{3} addition are not pure ZrO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3}, but Fe\textsubscript{2}O-containing complex oxides, which are different from the results obtained in sulfur addition experiments explained in Sec. 3.2. It was observed that spherical particles are present at the region of final solidification.

The dissolved oxygen content can be estimated based on the contents of insoluble M (M=Al, Zr and Fe) and total oxygen content, but these values correspond to the dissolved oxygen after solidification, not before solidification. Accordingly, the dissolved oxygen content was estimated as follows: The dissolved oxygen content before deoxidant addition was about 400 ppm which is the sum of initial dissolved oxygen (100 ppm) and oxygen (300 ppm) from Fe\textsubscript{2}O\textsubscript{3} addition. It is assumed that this dissolved oxygen is completely consumed for the deoxidation of Zr (0.05\%) or Al.
(0.05%), thus leading to the oxygen content (200 to 250 ppm) as a oxide. The dissolved oxygen content before solidification is estimated as 150 to 200 ppm.

The size distribution of ZrO$_2$- and Al$_2$O$_3$-containing particles obtained in the experiments with and without Fe$_3$O addition are shown in Figs. 12 and 13, respectively. As shown in the upper diagrams obtained by the cross section method, the larger particles with the size of 3 to 5 μm are observed in the experiments with Fe$_3$O addition, while the smaller particles with the size of 0.5 to 1.5 μm are observed in the experiments without Fe$_3$O addition. The dissolved oxygen (sol.O) content calculated from the values for insoluble M (Zr, Al and Fe) are shown in the figures. From the results shown in the lower diagrams obtained by the AC film method, it can be seen that the number of fine particles decreases in the experiments with Fe$_3$O addition.

Figures 14 and 15 show a plot of the numbers of ZrO$_2$-containing particles measured in cross section and that on acetylcellulose (AC) film in microsegregation domain at T.O levels of 69 and 378 ppm.
served in the experiments without FeO addition are independent of fraction of solid. These results indicate that the particles in microsegregation domain are pushed to the region of final solidification in the range of dissolved oxygen above a certain level. From the results shown in the lower diagrams obtained by the AC film method, the particle dispersion in microsegregation domain is not uniform in the experiments with FeO addition, as is found in the case of dissolved sulfur (Figs. 9 and 10).

In the experiments with FeO addition which enhances the content of dissolved oxygen, the crystallization of FeO starts to occur around the primary deoxidation particles such as ZrO2 or Al2O3 during solidification and these FeO-containing complex oxides are pushed to the region of final solidification. In the case of high dissolved oxygen the oxygen level increases due to microsegregation with increasing fraction of solid. As explained in Figs. 4 to 6, the initial sulfur content in which particle pushing occurs can be experimentally determined. However, the critical level of oxygen for particle pushing could not be determined experimentally in this study. It should be emphasized that the particle pushing becomes favorable with increasing fraction of solid due to the microsegregation of sulfur and/or oxygen, in particular, the enrichment of these solutes is very marked in the range of high fraction of solid.

4. Conclusion

The effect of sulfur or oxygen on engulfment and pushing of deoxidation particles of ZrO2 and Al2O3 during solidification of an Fe–10%Ni alloy has been studied and the results obtained are summarized as follows:

(1) The ZrO2 and Al2O3 particles are pushed to the interglobular region where the particle coagulation takes place with an increase in sulfur content. The sulfur contents at the engulfment and pushing transition for ZrO2 and Al2O3 particles are 300 to 400 and about 100 ppm, respectively.

(2) The ZrO2 and Al2O3 particles are pushed to the interglobular region with an increase in oxygen content, but the critical oxygen contents in which particle pushing occur could not be determined.

(3) The effect of sulfur or oxygen on engulfment and pushing of particles has been discussed by using the interfacial energy/wetting model and the critical velocity for the engulfment and pushing transition.

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