1. Introduction

The intragranular ferrite/pearlite nucleation on the inclusions and precipitates in austenite (fcc γ) has been used to refine the microstructure of steels for structural use to improve the toughness in low carbon steels when the deformation of austenite matrix before transformation is hardly applicable such as the heat affected zone formed in welding of structural steels. Recently, the intragranular ferrite transformation promoted by inclusions or precipitates dispersed in the matrix has been extensively studied both for Widmanstätten type intragranular ferrite (named ‘acicular ferrite’) and idiomorphic ferrite. It is generally accepted that proeutectoid ferrite can nucleate at the surface of such inclusions/precipitates within austenite grain. Although the inclusions and precipitates which are effective nucleation sites for intragranular transformation are different depending upon alloys, the following three mechanisms have been proposed, as summarized in a review paper to explain the effect of inclusions: (1) the existence of local variation of composition near the austenite/inclusion interface; (2) the generation of strain–stress fields around the inclusions due to the difference of thermal expansion coefficient between austenite and inclusion; (3) the improvement of lattice matching through the replacement of austenite/inclusion interface by ferrite/inclusion interface.

It is currently thought that a (MnS+V(C,N)) complex precipitate, each of which has a B1(NaCl) structure, is one of the most superior nucleation sites of intragranular ferrite idiomorphs in medium carbon steels without heat treatment after hot forging. Since the intragranular ferrite transformation occurs by the nucleation of new phase at austenite/precipitate/austenite interphase boundary, the energy balance of ferrite/precipitate/austenite interphase boundaries affects greatly on the nucleation kinetics of intragranular ferrite. Therefore, the crystallographic relationships between these phases are of great importance. It is believed that the most important role of incoherent MnS formed at casting stage is to provide nucleation sites for V(C,N) precipitation. V(C,N) nucleated on incoherent MnS has no specific orientation relationship (OR) with respect to the austenite matrix. Ishikawa et al. reported that the presence of low energy ferrite/V(C,N) interface with the Baker–Nutting orientation relationship ([001]//[001]_{V(C,N)}, [110]_{f}/[100]_{V(C,N)}) promotes intragranular ferrite transformation. In the case of acicular ferrite, Yang and Bhadeshia showed that there is near K–S orientation relationship between ferrite and austenite (transformed to martensite during quenching). Recently, some of the present authors found that intragranular ferrite idiomorphs formed in low supersaturation do not hold rational orientation relationships with their austenite matrices although acicular ferrite
formed in high supersaturation holds near K−S relationship in the case that ferrite nucleates on an incoherent MnS + V(C, N) complex precipitate.

Similar to the case of proeutectoid ferrite, inclusions or precipitates dispersed within matrix should be useful for the refinement of pearlite structure in high-carbon alloys. However, there is no study made on this subject. For crystallography of pearlite transformation, the earlier indirect X-ray study by Smith and Mehli\(^{23}\) indicated that intergranular pearlite does not have a specific orientation relationship with respect to its austenite matrix. However, Dippenaar and Honeycombe\(^{22}\) provided, through transmission electron microscopy investigation, the direct experimental evidence showing that ferrite or cementite in intergranular pearlite holds the near-rational orientation relationships with one of the adjacent austenite matrix in its nucleation stage. The present authors\(^{23}\) recently studied the crystallography of intergranular pearlite in Fe−12Mn−0.8C (almost identical in composition to the alloy studied by Dippenaar and Honeycombe) and Fe−12Mn−0.8C−0.3V alloys both of which austenite matrix can be retained even after quenching, by electron backscatters diffraction pattern (EBSP) analysis. It was revealed that the proportion of specific orientation relationship between the ferrite in intergranular pearlite and the austenite decreases significantly by V addition although majority of pearlite in the V-free alloy hold near-K−S relationship with respect to one of the austenite grains. This is due to that pearlite nucleates on incoherent MnS + VC at austenite grain boundaries in the V-added alloy. This result implies that pearlite/austenite crystallography is disturbed when it nucleates on incoherent inclusions. However, there is no systematic study made on the crystallography of intragranular pearlite transformation.

The present study aims to reveal the effect of MnS and VC on the kinetics and crystallography of intragranular pearlite transformation in Fe−12Mn−0.8C and Fe−12Mn−0.8C−0.3V hypereutectoid steels. Special attention is paid to the three-phase orientation relationships among the intragranular pearlite, the incoherent MnS + V(C, N) complex precipitate and their austenite matrix.

2. Experimental Procedures

The alloys listed in Table 1 were used in the present study. Fe−12Mn−0.8C (hereafter V-free alloy) was chosen because pearlite transformation occurs approximately below 923 K in this alloy\(^{23}\) and austenite matrix is metastable at room temperature so that as-transformed microstructure can be retained by quenching from transformation temperatures. Furthermore, this alloy contains MnS as inclusions in austenite. In Fe−12Mn−0.8C−0.3V (hereafter V-added alloy), VC precipitates from austenite so that this alloy is suitable to study the effect of VC or MnS + VC on the pearlite formation. Solution temperature of MnS in austenite in the V-free alloy is 1 815 K calculated with the equation of solubility product proposed by Turkdogan.\(^{24}\) This temperature is higher than the melting temperature of austenite (about 1 700 K). Thus, it is considered that MnS is formed in liquid state and embedded as incoherent inclusion in austenite by solidification. It should be mentioned that the molar volume of MnS (10.7 cm\(^3\)/mol) is larger than that of austenite (7.3 cm\(^3\)/mol) by nearly 50%. Thus, the interphase boundaries between these two phases should be incoherent unless some specific orientation relationship such as the cube-on-edge relationship previously reported\(^{25}\) is satisfied. In the V-added alloy, VC is expected to precipitate below 1 293 K from the equation of solubility product by Taylor\(^{26}\).

To eliminate Mn segregation, the specimens of 20 mm\(^3\) × 30 mm × 15 mm\(^3\) were hot rolled by 60% at 1 473 K, encapsulated in quartz tube under vacuum of approximately 10\(^{-3}\) Pa and homogenized at 1 473 K for 86.4 ks and furnace cooled to room temperature. Homogenized specimens were cold rolled by 70% and solution treated at 1 473 K for 0.6 ks and then water quenched to obtain austenite single-phase specimens. After this treatment, MnS becomes incoherent with respect to austenite matrix because of the austenite recrystallization. For the V-added alloy, VC precipitation treatment was performed at 1 173 K for various periods after the same solution treatment as the V-free alloy. These specimens were isothermally treated at 873 K or 773 K for various periods to promote pearlite transformation. Microstructures of the transformed specimens were studied by means of optical microscopy (OM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). For OM and SEM observations, specimens were mechanically polished and electropolished and etched with the solution of 100 g Cr\(_2\)O\(_3\) + 500 ml CH\(_3\)COOH + 20 ml H\(_2\)O at 20 V for 120 s. SEM-EBSD (Electron Back-Scatters Diffraction) examinations were performed with a Hitachi S-3100H equipped with TSL OIM (Orientation Imaging Microscopy) system. Thin discs, 3 mm in diameter and 0.2 mm in thickness were first thinned to 0.1 mm in thickness by chemically polishing with 5 ml HF + 80 ml H\(_2\)O\(_2\) + 15 ml H\(_2\)O and subsequently electropolished with 25 ml HClO\(_4\) + 500 ml CH\(_3\)COOH at 50 V by a twin-jet method. Final polishing of thin foils was performed by ion milling. TEM observation was performed by using Philips CM200 operated at 200 kV.

It should be noted that since the EBSD pattern from cementite in pearlite was weak, only the OR between pearlitic ferrite and austenite was examined. Two methods were used to express the OR between ferrite in pearlite and austenite matrix; one is the stereographic analysis and the other is the matrix calculation. Both were based on the Euler angles obtained from EBSD measurement. The former method provides convenience to check any possible specific OR between bcc (ferrite in pearlite) and fcc (austenite matrix) structures, while the latter method is useful for the statistical investigation of misorientation. In the matrix calcula-

| Alloy          | C   | Mn | V  | Si | S  | P  | O  | N  | Fe |
|---------------|-----|----|----|----|----|----|----|----|----|
| Fe-12Mn-0.8C  | 0.79| 11.69|<0.005|0.005|0.004|<0.005|0.0005|0.0011|bal.|
| Fe-12Mn-0.8C-0.3V | 0.79| 11.81|0.30|0.005|0.004|<0.005|0.0004|0.0011|bal.|

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tion, rotation matrices for ferrite in pearlite \( (B) \) and austenite \( (F) \) are both expressed in terms of Euler angles. Column vectors of rotation matrix are the base vectors of rotated orthogonal coordinate system with respect to unrotated system. The misorientation matrix between ferrite and austenite can be expressed as \( M = F B^{-1} \). Since both ferrite and austenite are high symmetric structures, all the 24 possible variants were considered. When the standard K–S OR is selected as reference, the misorientation angles between the close packed planes and between the close packed directions of two phases can be deduced, respectively.

3. Results

3.1. Effect of Complex Precipitates on the Pearlite Microstructure and Transformation Kinetics

Figure 1 shows optical microstructures of V-free and V-added alloys transformed at 873 K. Pearlite transformation starts from austenite grain boundaries and all the grain boundary area seems to be occupied by pearlite in the V-free alloy (Fig. 1(a)). As the isothermal holding time gets longer, grain boundary pearlite grows and intragranular pearlite also forms as shown in Fig. 1(b). In V-added alloys, the numbers of intragranular transformation increases (Figs. 1(c) and 1(d)). Figure 2 shows the SEM micrograph of the intergranular pearlite formed at 873 K in the V-free alloy. It is clearly seen that each pearlite nodule grows into one of the adjacent matrix grain.

The morphology of transformation product is often characterized by optical or electron microscopy. It is rather difficult to discriminate whether an ‘apparent’ intragranular pearlite nucleates truly within the grain from the observation on a single plane of sectioning. For this reason, the serial sectioning observation by optical microscopy similar to that made by Kral and Spanos was carried out to verify the morphological characteristics of intragranular pearlite nucleated at \((\text{MnS} + \text{VC})\) complex precipitates. An example of the morphological change of intragranular pearlite in an austenite grain of the V-added alloy by the serial sectioning is shown in Fig. 3. Two kinds of pearlite are visible in each micrograph; one is at austenite grain boundary and the other is within a grain. The former can be definitely determined as intergranular pearlite nucleated at grain boundary. On the other hand, for the latter case, most of them can be classified as intragranular pearlite nucleated within austenite grains but not always. For example, \( p_1 \) within a grain in Fig. 3(b) contacts with austenite grain boundary in fact when the previous plane of specimen is observed (Fig. 3(a)). This kind of pearlite should be regarded as intergranular pearlite. A complete morphological evolution from appearance to disappearance of pearlite during sectioning was observed for

![Fig. 1. Optical micrographs of Fe–12Mn–0.8C (V-free alloy) transformed at 873 K for (a) 18 ks and (b) 86.4 ks and of Fe–12Mn–0.8C–0.3V (V-added alloy) transformed at 873 K for (c) 18 ks and (d) 86.4 ks.](image1)

![Fig. 2. SEM micrograph of intergranular pearlite in the V-free alloy transformed at 873 K for 18.0 ks.](image2)
the pearlite \( p_2 \) in Fig. 3. The morphological features of this kind of pearlite are as follows: (1) They exist within austenite grain entirely in three-dimensional directions; (2) A particle is visible in each pearlite in some planes sectioned, usually near the center of pearlite; (3) Pearlite which nucleates at the surface of particle grows into austenite grain in three-dimensional directions.

**Figure 4** (a) shows the SEM micrograph of the intragranular pearlite in the deeply etched specimens of the V-added alloy transformed for a short time after aging at 1 173 K. A small particle indicated by the arrow ⃞ was identified as VC (Fig. 4(d)). This VC forms at the surface of a large particle. The particle pointed by the arrow ⃜ was identified as MnS (see Fig. 4(c)). The spectrum of Fig. 4(b) implies that this MnS nucleated on the oxide particle containing Al, Cr and Mg pointed out by the arrow ①. It is clear that the pearlite in Fig. 4(a) nucleates at VC but not at MnS, indicating that VC is more effective than MnS as the nucleation site for intragranular pearlite transformation. These features verify that the formation of (MnS+VC) complex precipitates promote intragranular pearlite transformation.

**Figure 5** shows the change in the volume fraction of pearlite for the V-added alloy aged at 1 173 K for various periods with holding time at transformation temperature. As a reference, the volume fractions of pearlite in the V-free alloy which contains only MnS are also plotted. As transformation proceeds, total volume fraction of pearlite increases. At 873 K (Figs. 5(a) and 5(b)), VC precipitation by aging accelerates pearlite transformation mainly due to the promotion of intragranular pearlite transformation. In the specimens aged at 1 173 K for 360 ks, initial transformation is suppressed but the transformation accelerates in the later stage of transformation. Contrarily, total transformed fraction at 773 K is lower in the V-added alloy although intragranular pearlite transformation becomes more active in the aged specimens than at 873 K. These results indicate that MnS+V(C, N) complex precipitate, which may be formed in the later stage, is more potent than MnS as the nucleation site of intragranular pearlite.

### 3.2. Crystallography of Intragranular Pearlite Nucleated at (MnS+VC) Complex Precipitate

A typical SEM micrograph of intragranular pearlite is shown in **Fig. 6(a)**. The (MnS+VC) complex precipitate is located near the center of pearlite and different colonies or blocks develop from the precipitate. The ORs between pearlitic ferrite in five colonies and austenite matrix were measured with EBSD. Figures. 6(b) and 6(c) are examples of EBSD patterns taken from austenite (fcc) and from ferrite (bcc) in this intragranular pearlite, respectively. The ORs observed are expressed on the stereographic projection in Fig. 6(d). It is to be noted that the pearlitic ferrites show no specific OR such as K–S or N–W with respect to austenite. Furthermore, a relatively large scatter can be seen.
among these ORs.

The low energy OR between austenite (fcc) and ferrite (bcc) is K–S or N–W OR. \(\{111\}_g\) is parallel to \(\{110\}_a\) in both ORs and the rotation of 5.26° around \((110)_g/(111)_a\) axis separates the K–S and the N–W ORs. The statistical measurement on the OR between pearlitic ferrite and
austenite was carried out. Figure 7 shows the deviation angles from K–S OR for the observed OR between ferrite in intragranular pearlite and austenite. Measurement was made for 22 colonies in 10 different intragranular pearlites nucleated at (MnS/VC) complex precipitates. Each of the solid triangles, solid cubes and the open cubes represent the colonies formed at the identical (MnS/VC) complex precipitate, respectively. This result confirms the followings: (1) there is no specific OR between pearlitic ferrite and austenite, and the deviation angle from the K–S OR can be as much as 30° both for close packed planes and close packed directions of the two phases; (2) the multiple colonies are formed at each (MnS/VC) complex precipitate surface, suggesting that there are many potential nucleation sites of intragranular pearlite on a complex precipitate.

The previous studies showed that the possible ORs between ferrite and austenite in intergranular pearlite are: Bagaryatskii ([100]a/[110]γ, [010]a/[111]γ and [001]a/[112]γ), Isaichev ([010]a/[111]γ and [101]a/[112]γ) and Pitsch-Petch ([100]a 2.6° from [311]γ, [010]a 2.6° from [131]γ and [001]a/[(112)γ]. The OR between cementite and ferrite in the intragranular pearlite was determined by TEM observation. Figure 8 shows the lamellar structure of intragranular pearlite (a), a set of Kikuchi patterns taken from ferrite and cementite ((b) and (c)) and the corresponding diffraction pattern of cementite (d). The stereographic projection in Fig. 8(e) shows that ferrite and cementite have approximate Pitsch-Petch OR, but neither Bagaryatskii nor Isaichev. The angle between [100]a and [131]γ is about 2.7° and that between [010]a and [113]γ is about 2.5°.

4. Discussion

As shown in Fig. 5, the volume fraction of intragranular pearlite in the V-added alloy is several times higher than in the V-free alloy after the same pearlite transformation treatment. This suggests that the precipitation of VC on MnS plays an important role to promote intragranular pearlite transformation. The effect of V addition on the acceleration of intragranular pearlite transformation should be explained based on the above crystallographic results.

When a particle is embedded in the matrix, the stress–strain field should be produced due to the differences in thermal expansion coefficient and in elastic constant between the particle and matrix during cooling. The elastic and thermal properties of austenite, VC and MnS are shown in Table 2, where £ is thermal expansion coefficient, E is Young’s modulus, v is Poisson’s ratio. It can be seen that the £ values of MnS and austenite are nearly the same, indicating that the stress–strain field caused by incoherent MnS is relatively small. On the other hand, there are large differences of thermal expansion and elastic properties between VC and austenite. Although accurate calculation was not attempted, compressive stress–strain field in austenite matrix around (MnS+VC) surface might be caused by
presence of the VC.

The most significant effect of V addition may be the formation of C-depleted zone at local area surrounding (MnS+VC) complex precipitates. In the present alloys, both MnS and VC can result in the composition depleted zones, which are Mn-depleted and C-depleted zones respectively. Mn depletion around MnS was recently detected by nano-probe analysis in a low carbon steel.\(^6\)\(^7\) Such depletion shifts the eutectoid composition to the higher carbon side in the Fe–C diagram, resulting in that ferrite can act as active nucleus for intragranular pearlite transformation in the present alloys. However, this effect seems to be not significant because intragranular pearlite transformation was not so effectively promoted in the V-free alloy. C depletion caused by the formation of VC may be pivotal effect to stimulate ferrite to act as active nucleus for intragranular pearlite nucleation at surface of (MnS+VC) complex precipitate. The SEM micrographs of Figs. 9(a) and 9(b) show the intergranular pearlite in the early stage of transformation in the V-added and the V-free alloys, respectively. The particles at austenite grain boundaries, with brighter contrast in Fig. 9(a), were determined as VC precipitates while the particle in the etched hole was identified as a (MnS+VC) complex precipitate by EPMA analysis. It is seen that three pearlite colonies nucleate at austenite grain boundary. There is no proeutectoid cementite film along the austenite grain boundary. The pearlite \(p_1\) directly nucleates at a (MnS+VC) complex precipitate, whereas \(p_2\) nucleates at a VC precipitate. In contrast, proeutectoid cementite film covers the austenite grain boundary in the V-free alloy (Fig. 9(b)). Pearlite colonies develop from the proeutectoid cementite. The formation of VC might result in the significant depletion of carbon at local area, favoring the ferrite to act as active nucleus for intragranular pearlite transformation thermodynamically even though the alloy has a hypereutectoid composition. This is similar to the formation mechanism of abnormal intergranular ferrite in the hypereutectoid steels as proposed by Han et al.\(^{35}\) and Edmonds and Chaiuangsi.\(^{36}\)

Another factor which may make ferrite as active nucleus for intragranular pearlite transformation is that there can be a low energy Baker–Nutting OR \([(001)]_a//[(001)]_{VC}, [110]_a//[100]_{VC}\) between ferrite and VC, as reported for intragranular ferrite transformation.\(^{13}\) The theoretical calculation on the structure component of interface energy shows that the ferrite/V(C,N) interphase boundary energy with Baker–Nutting OR is smaller than that of austenite/V(C,N) with
cube–cube OR for the (001)_{VC,N} interface while ferrite/MnS interphase boundary energy (with cube–cube OR) is larger than that of austenite/MnS (cube-on-edge OR) OR for the (001)_{MnS} interface.\(^{37}\) Therefore, it is deduced that VC in the complex precipitate acts as favorable nucleation site for intragranular pearlite transformation. This deduction is in good agreement with the results observed in Figs. 4 and 9 showing that pearlite nucleates directly at VC but not at MnS. In addition, relatively large deviation of ferrite/austenite OR from K–S OR for the intragranular pearlite (Figs. 6 and 7) might also indicate that there is a low-energy orientation relationship between VC and ferrite since there is no specific orientation between VC on MnS and its austenite matrix.\(^{15,18}\) The presence of multiple colonies with different ferrite orientations implies that many VC precipitates nucleated at the surface of a MnS particle. Figure 10 is the SEM micrograph showing the MnS+VC complex precipitate after prolonged aging at 1173 K. There are many VC precipitates (small particles with bright contrast) nucleating on a large MnS particle elongated by hot-rolling made prior to homogenization at 1473 K.

Based on the above discussion, the crystallography of intragranular pearlite at the (MnS+VC) complex precipitate is schematically described in Fig. 11. There is no specific OR between VC and MnS and between VC and austenite when VC precipitates at incoherent MnS surface.\(^{15,16}\) The formation of VC might result in the C-depleted zone at local area in austenite surrounding the (MnS+VC) complex precipitate. Such C depletion stimulates ferrite to become the active nucleus for intragranular pearlite transformation even though the present alloy has a hypereutectoid composition. It is possible that a low energy interface can be formed between ferrite and VC when pearlite nucleates at VC surface similar to the case of intragranular ferrite transformation on MnS+V(C,N) complex precipitate in low carbon alloys.\(^{13,15,20}\) Lack of specific OR with respect to austenite for both of ferrite and cementite ensures good cooperative growth necessary for pearlite with a low-energy ferrite/cementite OR (Pitsch-Petch OR). In addition, since many VC are formed at the surface of MnS, pearlites which nucleate at different VC precipitates should have non-specific OR each other, as in the case of P\(_2\) and P\(_3\) in Fig. 11.

5. Conclusion

The kinetics and crystallography of intragranular pearlite nucleated at (MnS+VC) complex precipitate were investigated in the hypereutectoid Fe–Mn–C alloys with V addition. The following conclusions were reached:

(1) Intragranular pearlite transformation is promoted by the addition of vanadium. Intragranular pearlite nucleates preferentially at the VC formed on incoherent MnS dispersed in austenite.

(2) There is no specific OR between intragranular pearlitic ferrite and austenite although there is a specific OR (Pitsch-Petch OR) between cementite and ferrite in pearlite.

Acknowledgement

The financial supports of the Iron and Steel Institute of Japan and the Ministry of Education, Science and Culture through a Grand-in-Aid for Scientific Research (A) No. 10305050 are gratefully acknowledged.

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