Original Paper

Formation of Austenite Grains and Grain Boundary Segregation in Heating Process of Steel

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Abstract

The transforming process was traced on 1%Cr-0.5%Mo steel in the conditions of as-hot-rolled, quenched and quenched-and-tempered. The rapid heating process was employed from the viewpoint of weld thermal cycle. In as-hot-rolled specimen, austenite grain nucleates at ferrite/pearlite boundary, and grows into massive form. In quenched and quenched-and-tempered specimens, two types of austenite grains were observed. One nucleates at prior-austenite grain boundary, and grows into massive form as same as hot-rolled specimen. Another one nucleates at the inside of prior-austenite grain along the lath structure of martensite. The starting and finishing temperatures of transformation rises with increasing heating rate. The rise of temperature is larger for as-hot-rolled specimen than for quenched specimen. The size of austenite grain decreases with increasing heating rate, however, the condition of specimen before transformation does not affect the final size at the end of transformation. The segregation of phosphorus was discussed on the basis of the moving speed of transforming interface, which was determined by the experiments.

Key Words: transformation, ferrite, austenite, heating rate, segregation of phosphorus, nucleation of grain, grain growth

1.Introduction

The ferrite/austenite transformation in the heating process is very im-portant, especially for the case of welding, by the following reasons. (1) Size and shape of austenite grains obtained by several ultimate temperatures and heating rates bring different influences on the transformation characteristics in the succeeding cooling process, therefore, the properties of transformation

products, (2) Segregation of impurity elements causes several embrittling phenomena in heat affected zone of welded joint. This segregation occurs originally in the course of ferrite/austenite transformation [1]. The degree of segregation depends on the speed of transformation, especially the moving speed of transforming interface [2]. Therefore, the data of this transformation propose a useful information for clarifying the mechanism of segregation. In this investigation, the transforming process was traced on a ferritic heat resisting steel of 1%Cr-0.5%Mo with the heating rate up to 200 K/s.

2.Experiments

The chemical composition of 1%Cr-0.5%Mo heat resisting steel (JIS SCMV2) is listed in Table 1. The steel was received in as-hot rolled condition, ; its microstructure consisted of 82 % ferrite and 18% pearlite, as shown in Fig.1(a).

The quenched specimen was made by heating the as-recieved steel at the rate of 50 K/s up to 1273 K, and keeping for one hour followed by water-quenching. The quenched-and-tempered specimen was made by tempering the quenched steel at 923 K for one hour. Fig.1(b) and (c) show the microstructures of quenched and quenched-and-tempered specimens, respectively. These three specimens are called briefly as AHR, Q and QT specimens.

Three specimens were transformed by the following two heating procedures. (1) Heating up to a given temperature above Ae1 by the rate of 50 K/s, and keeping for one hour followed by water-quenching. The specimen obtained by this procedure was named the specimen in equilibrium state assuming that its microstructure is same as that in the equilibrium state.
(2) Heating up to a given temperature above Ae1 by a series of heating rates (0.05 to 200 K/s) followed immediately by water-quenching.

Austenite grains, which were transformed into martensite or bainite grains at room temperature, were observed on polished and etched surfaces of the specimens. In cases of Q and QT specimens, the etchant of saturated aqueous solution with picric acid was used to reveal the ferrite/austenite interface. In case of AHR specimen, martensite was very hardly distinguished from pearlite by the etchant of picric acid , as shown in Fig.2(a). Then, the etchant of 3 % nital was used. Martensite and pearlite were colored light gray and dark gray, respectively by nital, as shown in Fig.2(b).

The scanning electron microscope was employed to observe fine austenite grains (martensite at room temperature) produced by rapid heating. The polished surface was etched by a selective potentiostatic etching by electrolytic dissolution (SPEED method) [3]. Fig.3 shows the relation between electrolytic current and potential for the martensitic and ferritic specimens containing carbide particles. The threshold values of potential were -250 and -300 mV, respectively. The broken line shows the ratio of both current values, C_f/C_m. The ratio became maximum at the potential of -150 to -100 mV. Therefore, -125 mV
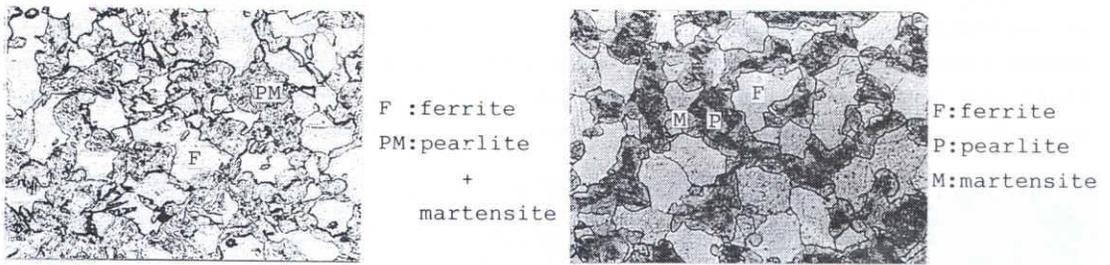
Table 1 Chemical compositions of the steel used (wt%)

C	Si	Mn	P	S	Cr	Mo
0.15	0.28	0.57	0.011	0.0088	1.09	0.55
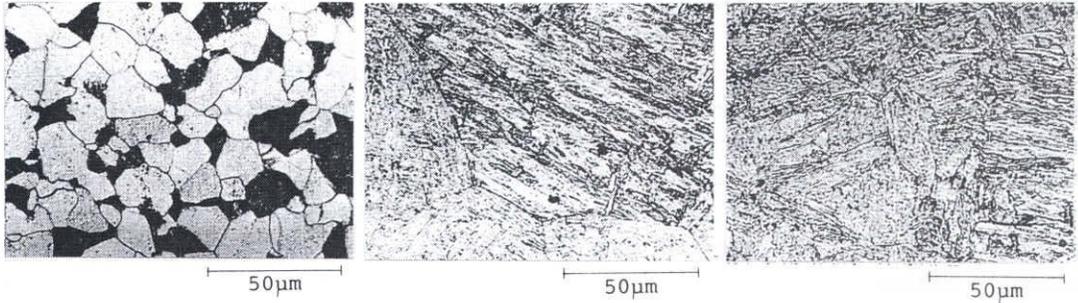
(a)As-hot-rolled   (b)Quenched   (c)Quenched-and-tempered

Fig.1   Microstructures of the specimens before transforming process

F :ferrite
PM:pearlite
+
martensite

F:ferrite
P:pearlite
M:martensite

(a) saturated solution with   (b) nital
    picric acid

Fig.2   Identification of martensite by optical microscope on as-hot-rolled
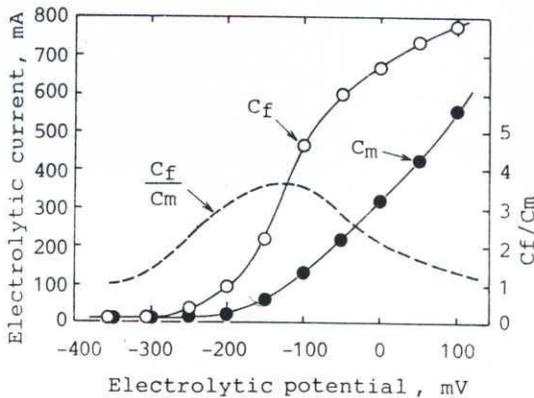   specimen

Fig.3   The relations between current and
        potential for ferritic and
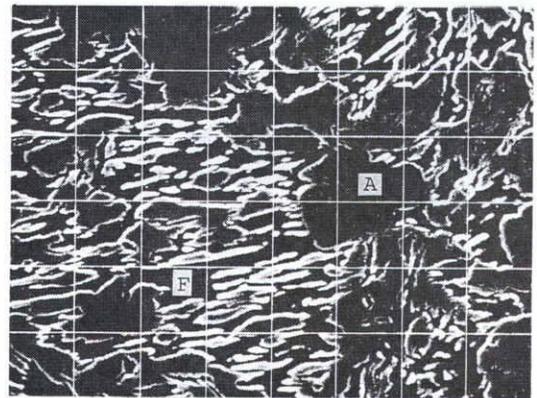   martensitic specimen

Fig.4   Example of microstructure
        of electrolytically
        etched surface

was employed for the electrolytic etching. Fig.4 shows the SEM microphotograph
on the surface etched electrolytically. Ferrite portion was dissolved uniform-
ly remaining martensite portion undissolved.

The area- and line methods were employed for quantifying the transformed
volume, and the number of austenite grains was counted directly on the surface
area of 0.3 mm^2.

3.Nucleation and growth of austenite grain

In AHR specimen, austenite grains nucleate mainly at the grain boundaries
of ferrite/pearlite and ferrite/ferrite (Fig.5(a)). The transforming interface
moves toward inside of ferrite and pearlite grains, and each austenite grain
grows massive form. The nucleation never occurs inside of ferrite or pearlite
grains, except for a rapid heating, such as 50 or 200 K/s. At the final stage
of transformation, all ferrite and pearlite grains transformed into massive
austenite grains.

In Q and QT specimens, austenite nucleates at the prior-austenite grain
boundaries when the transformation begins (Fig.5(b) and (c)). Austenite grains
become massive and larger with rising temperature.

Fig.6 shows the microstructures of three specimens, of which the fraction
of austenite grains reaches about 60 %. In case of AHR specimen, martensite
grains (gray portion in Fig.6(a)) grow surrounding ferrite grains (bright por-
tion) and pearlite grains (dark portion). Martensite grows in the region bet-
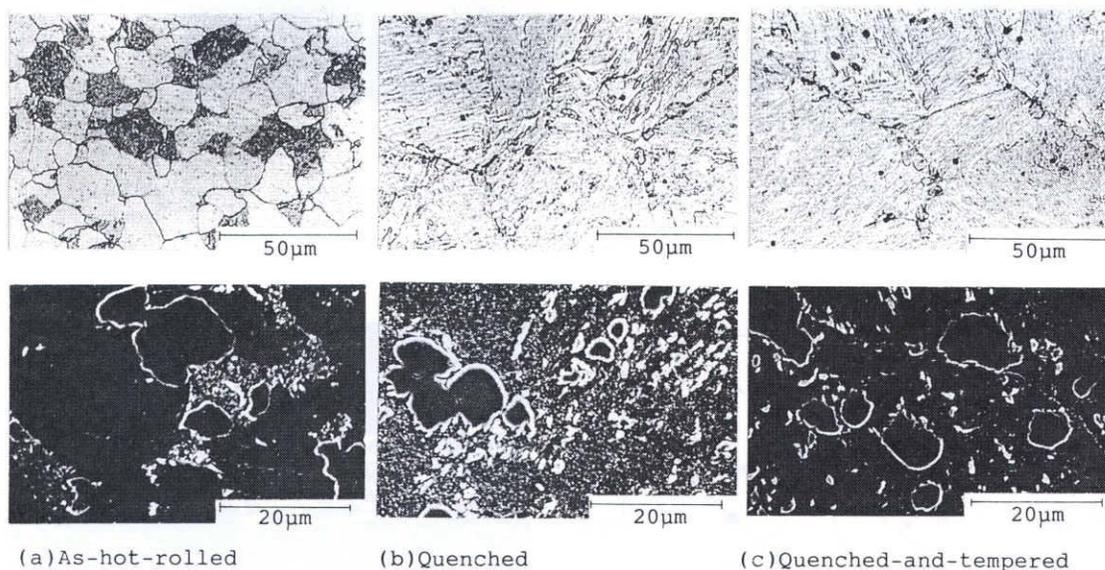ween ferrite and pearlite, and the center portions of ferrite and pearlite

(a)As-hot-rolled   (b)Quenched   (c)Quenched-and-tempered
Fig.5   Microstructures of the specimens at the beginning of transformation
        Top:optical microphotograph; Bottom: SEM microphotograph

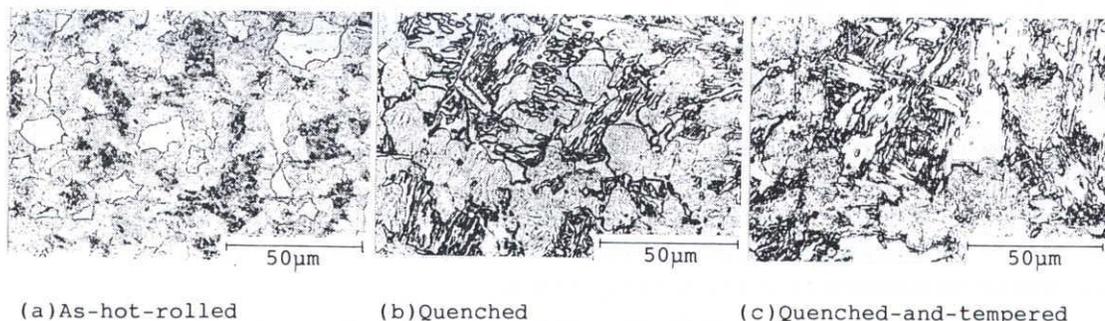(a)As-hot-rolled   (b)Quenched   (c)Quenched-and-tempered
Fig.6   Microstructures of the specimen at the middle stage of transformation
        Fraction of austenite is about 60 %.

grains transform at finally. In Q and QT specimens, massive and rod-like aus-
tenite are observed, as shown in Fig.6(b) and (c).

Fig.7 shows the sequence of transforming process of Q specimen with the
heating rate of 50 K/s. At the beginning of transformation, austenite nucleates
at prior-austenite grain boundary (Fig.(a) and (a)'). At the same time, fine
particles are also produced inside of prior-austenite grain (Fig.(a')). At 1063
K, massive austenite at grain boundary grows, the number of fine particles
inside of a grain increases (Fig.(b)). These particles are also austenite as
shown in Fig.(b)'. This type of austenite is called as "lath austenite" because
it is produced in each lath of martensite succeeding the lath-structure. As the
transformation proceeds (Fig.(c) and (d)), lath austenite grows into rod-like
shape. At final stage of transformation, lath austenite grains join together to
form one massive grain, as shown in Fig.(e)'. The grain marked by the arrow was
originally lath austenite, because the profile of rod-like shape was observed.
The massive grain produced by joining the lath can not be distinguished from
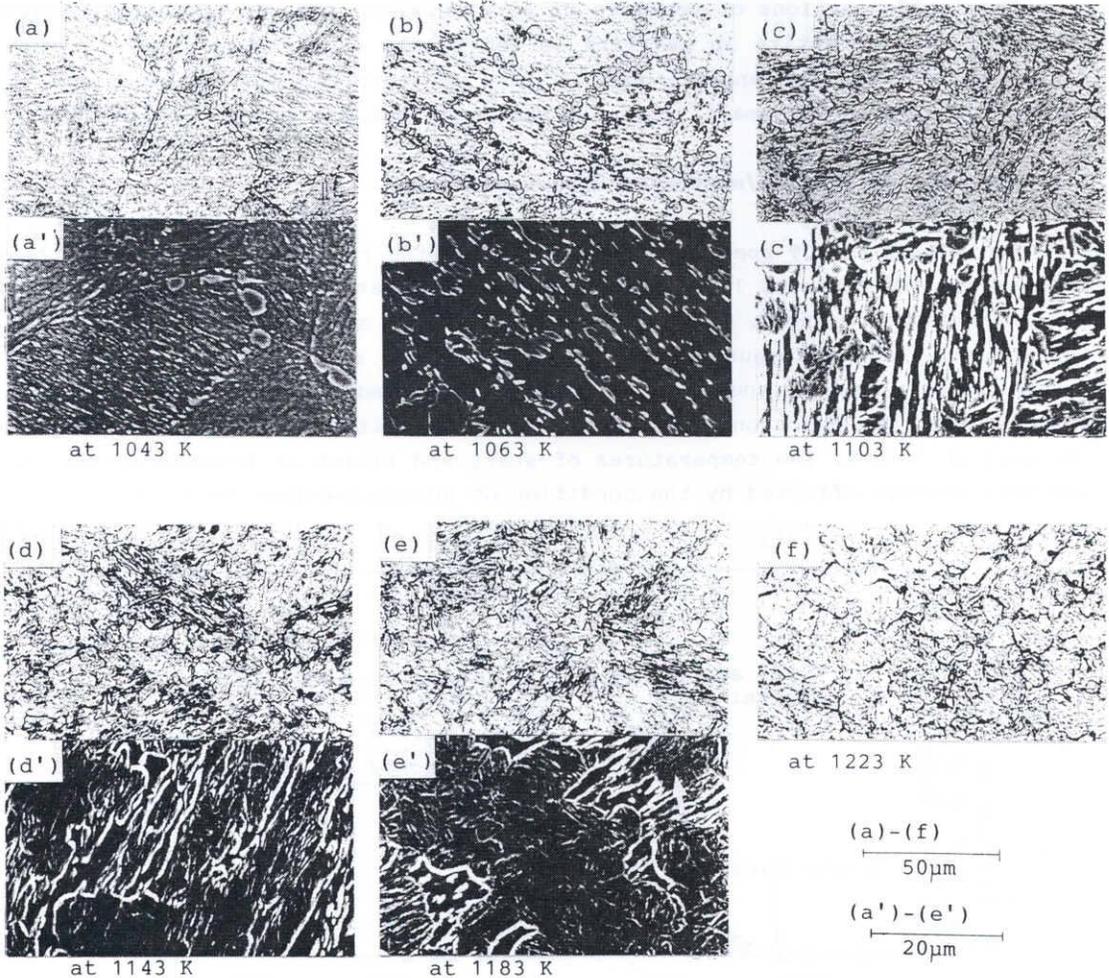

Fig.7   The sequence of transforming process in quenched specimen with the
        heating rate of 50 K/s
        (a)-(f): optical microphotograph; (a')-(e'): SEM microphotograph

the original massive grain at the final stage of transformation.

Although lath austenite was recognized both in Q and QT specimens, it was larger and clearer in appearance in Q specimen than in QT specimen.

At the end of transformation, massive grains are observed as shown in Fig.(f).

Above experimental results will be explained from the viewpoint of the energy change of the system as follows. The nucleation of austenite occurs at the sites where following two conditions are attainable. (1) Atoms can be rearranged into f.c.c. structure with a minimum activation energy, (2) 0.8% of carbon can be supplied with a local diffusion. The grain boundaries of ferrite/ferrite and ferrite/pearlite is one of the suitable site, because in the grain boundary the b.c.c. structure of ferrite is in the incomplete stage, and carbon is enriched there inherently. The martensite-lath is another suitable site, because martensite-lath and austenite hold a crystallographic coherency in each other, and carbide phase is in the state of fine particles which can be dissolved into iron-matrix with minimum energy.

The inside portions of pearlite or ferrite grain are not suitable, because the plate-like cementite in pearlite is too stable to dissolve into   iron-matrix with a smaller energy, and in case of ferrite, the concentration of carbon (0.02 %) is too small to supply the enough carbon to austenite (0.8 %).

4.Temperature of ferrite/austenite transformation

The AHR, Q and QT specimens were heated at the rate of 50 K/s up to a test temperature and kept for 1 hour. The fraction of austenite at each test temperature are shown in Fig.8. Open makes show the total amount of austenite including massive- and lath-austenite, and solid makes show the fraction of lath austenite in Q and QT specimens. Austenite is found in every specimen heated above 1010 K. The fraction of austenite increases with temperature, and reaches the unit at 1130 K. The temperatures of start and finish of transformation, Trs and Trf, are not affected by the condition of specimen before heating.
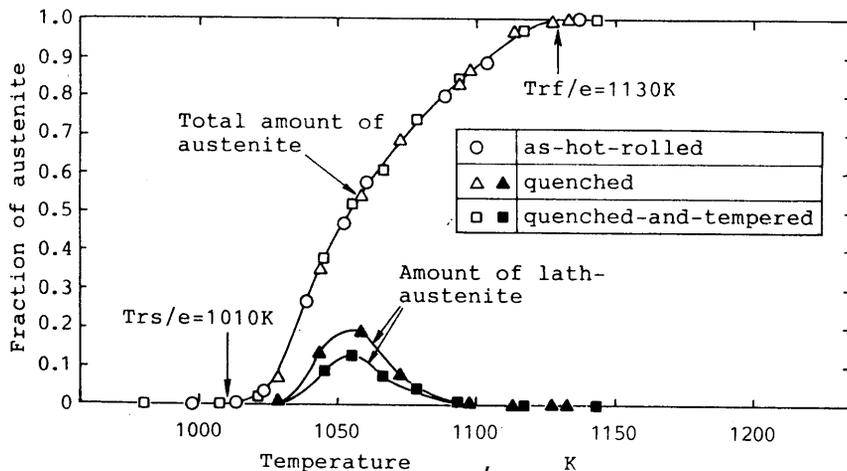

Fig.8   Fraction of transformation at each temperature for the specimen of
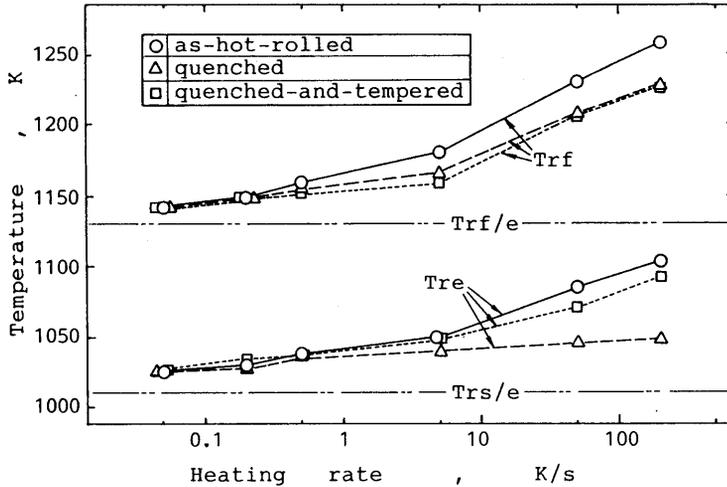   equilibrium state.

Fig.9   Influence of heating rate on the starting and finishing
        temperatures of transformation

     Fig.9 shows the influence of heating rate on the Trs and Trf temperatures
of ferrite to austenite transformation.   In case of continuous heating process,
Trs and Trf temperature are defined as followings.
     Trs : starting temperature of ferrite to austenite phase change
     Trf : finishing temperature of above phase change
Therefore, Trs and Trf temperatures have different meaning from A1 and A3 tem-
perature in the process of equilibrium state.
     These temperatures are raised above Trs/e and Trf/e only by 10 to 20 K
with slow heating. The Trs temperature rises along Trs curve with increasing
heating rate, especially for AHR and QT specimens with the heating rate above
20 K/s. However, the Trs temperature of Q specimen rises only 40 K even though
the heating rate is as large as 200 K/s. The Trs temperature of QT specimen is
nearly same as that of AHR specimen, and its Trf temperature is nearly same as
that of Q specimen.
     This fact can be explained by the distribution of carbon in iron-matrix.
In equilibrium state, austenite phase of 0.8 %C is formed from ferrite phase of
very low carbon (0.02 %) and carbide phase (alloy carbide) of very high carbon
at the eutectoid temperature, Ae1. In QT and AHR specimens, the particles or
plates of carbide have to be dissolved in order to produce the region of 0.8
%C. Although carbide particles are precipitated in Q specimen during heating to
the test temperature, most of them are iron carbide, cementite, because the
time spent for the precipitation is too short to form alloy carbide. This
cementite is less stable than the alloy carbide present in AHR and QT speci-
mens. Therefore, the rise of Trs is little for Q specimen. In the case of
continuous heating, the dissolving the carbide will continues during transfor-
mation. The distribution of carbide has to be uniform at the end of transforma-
tion. The distribution of QT specimen will be more uniform than that of AHR
specimen, because the carbide precipitated by tempering is very   fine   and
uniformly distributed than the ferrite/pearlite mixed structure.

5.Size and number of austenite

Fig.10(a), (b) and (c) show the size and number of massive austenite grain in AHR, Q and QT specimens, respectively. The growth of grain begins immediately after the transformation starts, and continues during transformation. At the final stage of transformation, the growth rate becomes slow. The grain size is generally decreased with increasing heating rate, even though the transformation takes place in a higher temperature range. However, taking a shorter heating time in account for the rapid heating process, the growth rate in rapid heating process is larger than that in slow heating. The final grain size is not influenced by the conditions before transformation.

Number of grains continues to increase during transformation. That is, the nucleation of grain continues until the end of transformation. The rate of nucleation is largest at the time when transformation starts. This tendency is remarkable in Q and QT specimens, especially, for rapid heating of 200 K/s. In Q specimen, the increasing rate of number of massive grains becomes larger at the final stage of transformation, because lath austenite grains change into the massive form by coalescing each other.

Fig.11(a) and (b) show the size and number of lath austenite grain in Q and QT specimen, respectively. However, the influence of heating rate is smaller than that of massive austenite grain. The number of grains in Q specimen is larger than that in QT specimen, however the size itself of Q specimen is same as QT specimen. That is, the nucleation in Q specimen is more frequent than that
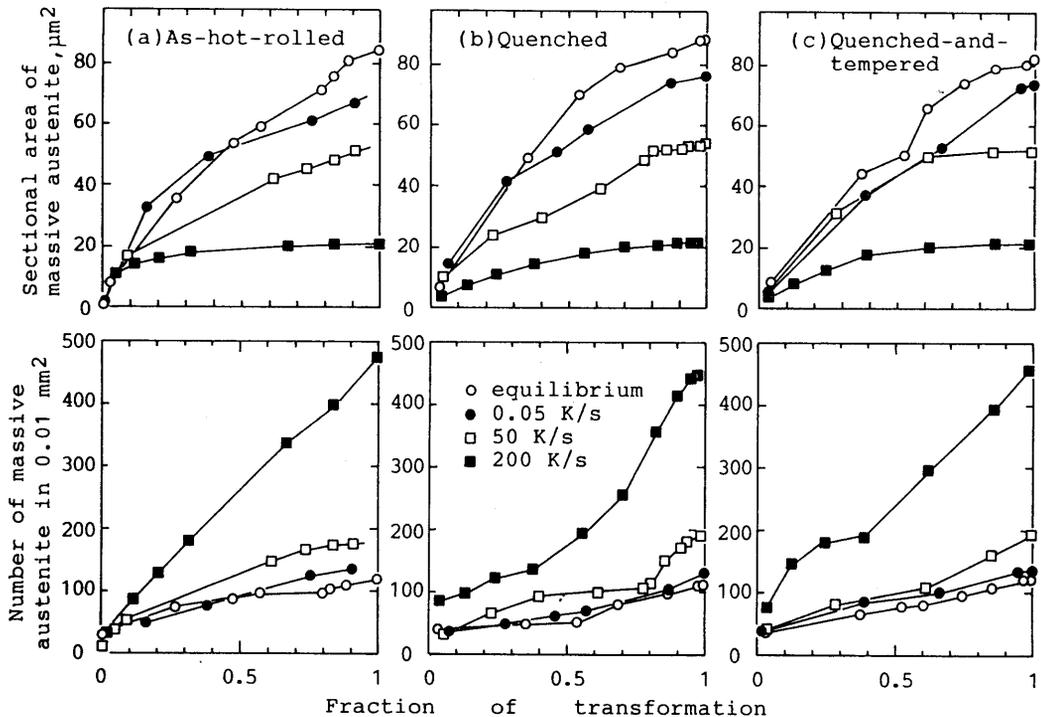

Fig.10   Sectional area and number of massive austenite grain as the function of fraction of transformation
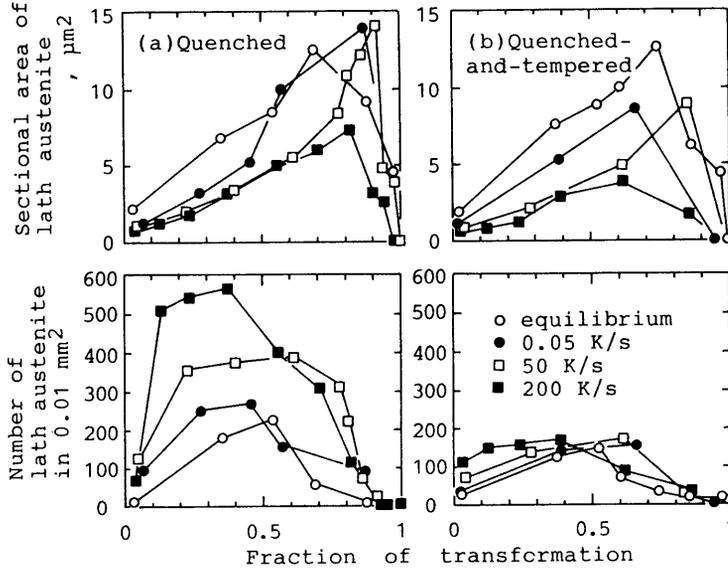
Fig.11   Sectional area and number of lath austenite grain as the function
         of fraction of transformation

in QT specimen. The number of lath austenite decreases by joining together. The
joining of lath austenite begins at earlier stage of transformation with rapid
heating. The number reaches zero until the end of transformation.

6.Segregation of phosphorus caused by transforming process

During transformation, the position of ferrite/austenite interface moves
toward ferrite phase. The moving speed of interface depends on heating rate and
grain size of austenite. The time from starting to finishing becomes short with
increasing heating rate. However, in rapid heating, a large number of austenite
grains grow simultaneously. Therefore, growth rate of each grain is much small-
er than expected. In this section, the moving speed of interface is estimated
for the massive austenite grain using the experimental results of grain size.
For the calculations, the following assumptions were made.
    1) Massive austenite is a sphere.
    2) The size of all grains are same.
Therefore, the moving speed mentioned below is the mean value. The assumption 2)
is against the experimental results, as shown in Fig.10, which indicates that
the nucleation of new grains occurs at each time period during transforming
process.
    If the volume of a grain is V, an expectation of sectional area, A is

$$A = \frac{V}{2r} = \frac{2\pi r^2}{3} \tag{1}$$

where, r is a radius of grain. Therefore,

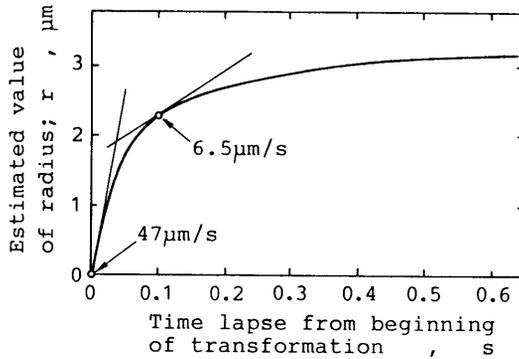$$r = \sqrt{\frac{3A}{2\pi}} \tag{2}$$

Fig.12 Estimated radius of massive austenite grain in as-hot-rolled
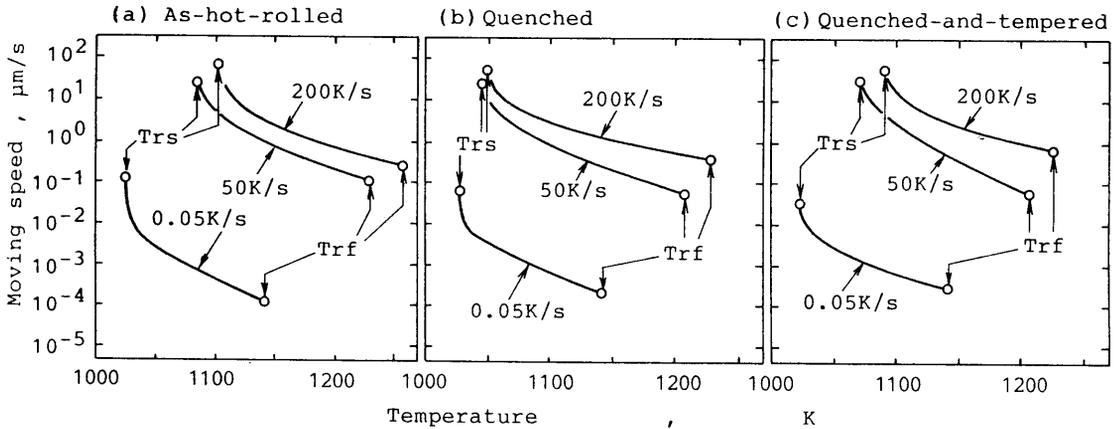   specimen with the heating rate of 200 K/s

Fig.13 Moving speed of the interface between ferrite and austenite, estimated
        from the data about sectional area of massive austenite grain, as shown
        in Fig.10

The equivalent radius of massive austenite in Fig.10 is calculated by eq.(2),
as shown in Fig.12. As the radius is plotted against the time lapse from begin-
ning of transformation, the moving speed of grain surface (interface of ferr-
ite/austenite) can be obtained as the inclination of tangent to the curve. The
moving speed is largest at the beginning of transformation and decreases with
time.

     The moving speed of grain surface of massive austenite are summarized for
AHR, Q and QT specimens with heating rate of 0.05, 50 and 200 K/s in Fig.13.
The microstructure before transformation does not affect on the moving speed of
interface at every heating rate.

     Austenite nucleates in ferrite matrix, and the interface of ferrite/aus-
tenite moves toward ferrite region. The solubility of phosphorus in austenite is
smaller than that in ferrite matrix. Equilibrium distribution coefficient is
about 0.4 [4]. Therefore, phosphorus is swept out from austenite to ferrite
region, and phosphorus segregate at final stage of transformation.

     If the amount of diffusion into ferrite is equal to the amount of sweep
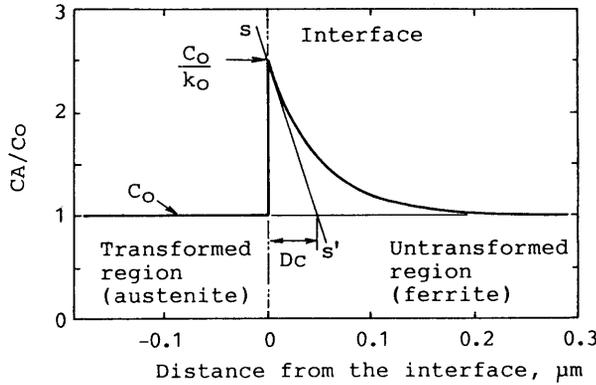out of the transforming interface, the phosphorus concentration is in the

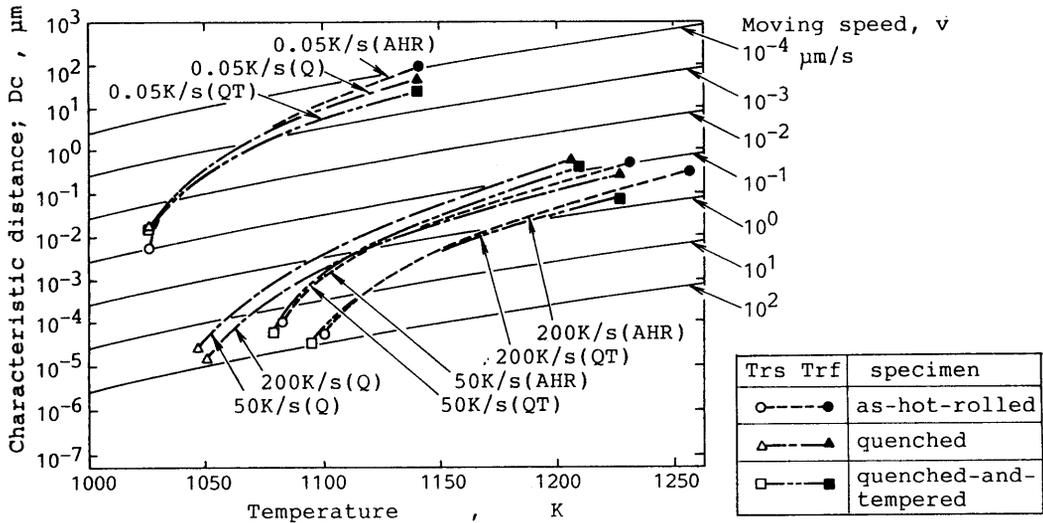Fig.14 The profile of phosphorus concentration near the interface

Fig.15 The characteristic distances and courses with various heating rates

steady state near the interface. The diffusion coefficients of phosphorus in ferrite and austenite are 2.75×10^{-6} and 4.91×10^{-8} m^2/s at 1200 K [5], respectively. Then the profile of phosphorus concentration, CA will be given by the following equation assuming that phosphorus does not diffuse in austenite [6].

$$CA = Co + Co \frac{1 - k_o}{k_o} \exp\left(-\frac{v}{Da} x\right) \qquad (3)$$

Where, Co is bulk concentration of phosphorus, ko is equilibrium distribution coefficient, v is moving speed of interface, Da is diffusion coefficient in austenite and x is the distance from the interface. Fig.14 shows the estimated profiles of phosphorus concentration, where the moving speed and diffusion coefficient are 1μm/s and 4.91×10^{-8} m^2/s. In this case, the thickness of segregation layer is about 0.2 um. The characteristic distance; Dc can be defined as shown in Fig.14. The line, ss' is tangent to the concentration curve at x=0. The characteristic distance is

$$Cd = \frac{Da}{v} \qquad (4)$$

In the case of continuous heating process, the transformation occurs over a range of temperature. Therefore, the diffusion rate will change during transformation. And the moving speed of interface also will change. Fig.15 shows the D_c at the temperature range of 1000 to 1250 K. The moving speed of interface was changed from 0.0001 to 100 μm/s. The characteristic distance increases with temperature, because the diffusion coefficient of phosphorus in ferrite increases with temperature. The broken lines indicate the courses of temperature and moving speed of interface for three heating rates. The D_c value is small at the beginning of transformation, and increases with the progress of transformation, because of the increase in temperature and the decrease in moving speed of interface. The D_c value is largest in the of 0.05 K/s.

At the final stage of transformation, eq.(3) will not express the phosphorus concentration, because phosphorus can not diffuse into ferrite region. Phosphorus will be enclosed in the narrow ferrite region and phosphorus concentration rapidly will be increased. Phosphorus is the ferrite stabilizing element. Therefore, if the phosphorus concentration in ferrite region exceeds the solubility limit of austenite, ferrite can not transform to austenite.

7.Conclusions

The transforming process was traced on 1%Cr-0.5%Mo steel with a rapid heating rate such as 200 K/s. The location of nucleation and shape of austenite grain is changed by the microstructure before transformation. There exists only massive austenite grain from ferrite/pearlite mixed structure. Both of massive and lath austenite grains grow from quenched and tempered structure including lath structure of martensite grain. The transforming temperatures raise with increasing heating rate. The rise in as-hot-rolled specimen is larger than that in quenched specimen. The size of austenite grain decreases as the heating rate increases, however, the condition of specimen before transformation does not affect on the final size at the end of transformation.

The estimated moving speed of the ferrite/austenite interface is small at the final stage of transformation. And the thickness of segregation layer exceeds 0.1 μm even though the heating rate is large as 200 K/s.

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