Characteristics of Hot Ductility in Steels

Subjected to the Melting and Solidification*

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Synopsis
Hot ductility in steels was studied. Special emphases were placed on
the effects of thermal history, strain rate and fracture mode in order to clarify
the sensitivity of surface cracking during both continuous casting operation
and direct hot rolling.
There exist three temperature regions where typical embrittlement is noticed, i.e., Tm.'1200°C (I), 1200 N 900°C (II), and 900' 600°C (III). The cause of the embrittlement in the region I is the existence of residual liquid film along the dendritic interfaces. The ductility is found to be independent of the strain rate. In the region II, the precipitation of finely distributed oxy-sulfides at the austenite grain boundary weakens the boundary strength, and thus overaging treatments such as slow cooling, holding for certain time, or slow rate of straining result in good ductility. On the other hand, the embrittlement in the III region is manifested by the slower strain rate of test. Controlling factors of this embrittlement are precipitation of oxides, sulfides and nitrides, precipitation of proeutectoid ferrite film along austenite grain boundary as well as grain boundary slid-ing. Detailed mechanism is discussed.

I. Introduction

Increasing importance is being attached to the study of cracking in continuously cast slabs and of their hot workability during hot rolling, in order to increase the continuous casting ratio and accomplish hot direct rolling as means for reducing the consump-tion of energy and the number of processes involved in the manufacture of steel products. Laboratory ap-

proaches to the cracking in continuously cast slabs are represented by analysis of deformation characteristics by hot-stage tensile tests and theoretical analysis of thermal stresses. However, complete solutions to the cracks encountered in actual operations have not been obtained. Two of the reasons are that experiments which simulate the thermal history to which contin-uously cast slabs are subjected are difficult to perform and that the deformation mechanism and embrittle-

ment factors of steels in a high-temperature region are not fully understood. In a metallurgical view of the continuous casting process, the process starts with the solidification of liquid steel. As soon as the solidified shell is formed, the segregation and precipitation of solute atoms proceed. Thermal stresses set up during the intervening time combine with mechanical stresses as from ferrostatic pressure of liquid steel and mis-alignment of guide rolls, causing surface and internal cracks in the slab being cast. A better clarification of these problems calls for study on basic matters, such as the temperature region where embrittlement oc-curs, the factors that govern embrittlement and the strain rate dependence of embrittlement.
The works which have investigated the deforma-tion behavior of steel in the high-temperature region

from this standpoint are represented by those of Adams' and Lankford.2~ In Japan, there are re-ported the studies which have dealt with embrittlement in the vicinity of the melting point3-5) and those which have discussed embrittlement in the tempera-ture region of 900 to 6000C.68) However, there is no research that has systematically examined changes in strength and ductility of steel over a wide tempera-ture range from melting point to 600°C by simulating the thermal history of continuously cast slabs.
This report mainly describes the investigated results of thermal history, strain rate dependence and frac-ture mode concerning characteristic embrittlement which occurs on cooling after melting and solidifica-tion.

II. Experimental Method

One feature of the present experiment is that the embrittlement behavior of steel was investigated by melting a steel sample to simulate the actual contin-uous casting process and conducting tensile tests at re-spective temperatures on cooling following solidifica-tion. Gleeble testing machine (Fig. 1) was used for this purpose, which is a kind of horizontal tension tester and is designed to subject specimens to a wide

preprogrammed range of thermal histories by resist-ance heating through direct application of electric current to the specimen. Part of the machine was

Fig. 1. Schematic view of tensile test and thermal cycles.

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modified to get the strain rate of tensile deformation over a wide range. The experimental conditions are described below.

(1) Chemical Compositions of Test Steels and Shape of Test Specimens
The chemical compositions of the tested steels are given in Table 1. Heats A to C were vacuum melted in an induction furnace, while heats D to F were continuously cast slabs. Test specimens were round bars 10 mm in diameter and 120 mm long. Specimens were cut from hot-rolled plates in the rolling direction for vacuum-melted heats and from the columnar crys-tal zone near the surface in the casting direction for continuously cast slabs.

(2) Thermal History
As shown in Fig. 1 (a), a specimen was in situ melt-ed, solidified and tensile tested at the specified tem-perature on subsequent cooling. This type of speci-men was used as the basic one and is referred to as

`melted specimen' here. The heating rate was 25°C/s,
the cooling rate was 20°C/s, and the time of holding at temperature before the tensile test was varied as required. A quartz tube 10.2 mm in diameter and 30 mm in length was installed on the specimen to sup-port the melting zone (12 mm in length). It was con-firmed by chemical analysis that specimen contamina-tion by the quartz tube was negligible. For the pur-pose of comparison, some specimens were in situ melt-ed, cooled to a certain temperature, then reheated and tensile tested [Fig. 1 (b)], and as received speci-mens were reheated and tensile tested [Fig. 1 (c)]. These two types of specimens are called ` reheated specimens'.

(3) Specimen Temperature Control
Two pairs of PR-13 thermocouples were spot weld-ed to the surface center of each specimen. One pair was used for temperature control and the other for temperature measurement.

(4) Tensile Deformation
A pneumatic drive was used for high-speed defor-mation (~>2fs), and a pulling jig powered by a low-speed motor especially developed by the authors was used for low-speed deformation (<0.5/s). The aver-age strain rate is obtained by assuming that the uni-form deformation zone of the specimen was 10 mm long. Tensile strength and reduction of area were obtained as indices for strength and ductility and were used as characteristic values in the high-temperature regions under consideration.

(5) Temperature Difference between Surface and Axis of a Specimen
A 3-mm diameter hole was drilled perpendicular or parallel to the axis of a specimen (0.1 % C-steel) as shown in Fig. 2, and a PR-13 thermocouple was spot welded to the bottom of the hole and measurement of

the temperatures of the specimen at the axis and sur-face was done simultaneously. As the heating tem-perature increases, the temperature difference be-tween the axis and surface increases. If correction is

made for the energy dissipation at the surface, the temperature difference between them becomes around 50°C. In this report, test temperature denotes sur-face temperature for convenience.
(6) Solidification Pattern
Photograph 1 shows the solidification structure of 0.1 %C steel melted and then cooled at 20°C/s to am-

bient temperature. Columnar crystals are shown ex-tended toward the axis of the specimen. In this ex-periment, 1 to 2 % compressive strain was applied to the specimen during solidification to prevent the for-mation of shrinkage porosity.

III. Experimental Results and Discussion

1. Embrittlement Near Melting Point [Tm (melting point) to 1 zoo°C]
1, The Thermal History Dependence of Strength and Ductility
Specimens were cut from the columnar crystal zone of continuously cast slabs of austenitic stainless steel SUS 316 (heat F in Table 1). Some of the specimens were melted, solidified and tensile tested, while the others were reheated and tensile tested. The mea-

sured values of reduction of area and tensile strength

Fig. 2. Temperature difference between interior and outer surfaces of 10 mm diameter specimen (0.1 %C-
steel).

Table 1. Chemical compositions of alloys. (wt%)

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with temperatures are shown in Fig. 3 (=5/s). The deformation behavior (ductility change in particular) of this steel is extremely sensitive to the thermal his-tory. Especially, many of the melted specimens se-verely embrittle near the melting point or in the low-temperature region (embrittlement of carbon steels will be described later). When the problem is crack-ing during a process, such as continuous casting, direct rolling or welding, where the steel is subjected to the thermal history of melting and solidification, it is ne-cessary to investigate cracking susceptibility on melted specimen as shown in Fig. 1 (a).
2. Fracture Mode
The observed fracture appearance of the above-mentioned SUS 316 steel near the melting point is

shown in Photo. 2. The melted specimen was melted at 1420°C and tensioned to fracture at 1 350°C on cooling, while the reheated specimen was heated to 1 350°C and fractured at that temperature. The for-mer is fractured along the dendrite interfaces where en-riched residual liquid steel often remains. The latter exhibits austenite grain boundary fracture. Accord-ing to the detailed observation of the process leading to the fracture of the reheated specimen, an incipient grain-boundary melting occurs first. Under tensile stress, voids initiate at the grain boundaries and grow along the grain boundaries as the melting zone ex-

pands, eventually leading to the final failure. When attention is focused on ductility near the melting point in Fig. 3, the zero ductility temperature (ZDT) of the

Photo. 1. As cast structure
ified at 20°C/s.

of 0.1 %C-steel melted and solid-

Photo. 2. Fracture surface of the
1 325°C on heating (B).

austenitic stainless steel (heat F), fractured at 1 350°C on cool ing (A) and

Fig. 3. Hot ductility and strength curves for the austenitic stainless steel (heat F).

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melted specimen indicates the temperature at which solid phases connect with each other and start defor-mation after a considerable progress of solidification. The temperature that ductility commences to decrease in the reheated specimen denotes the incipient grain boundary melting temperature.
3. Strain Rate Dependence of Ductility
Using high-purity electrolytic iron (heat A) and 0.4%C steel (heat B), the strain rate dependence of ductility near the melting point was investigated. The results of reheated specimens are plotted in Fig.
4. Figure 4 clearly indicates that the ductility near the melting point does not depend on the strain rate. With melted specimens, similar results were obtained. If the embrittlement near the melting point is caused by the presence of a liquid film along the grain boundary or dendrite interface as described in Section 111.1.2, it naturally follows that the ductility does not depend on the strain rate.

2. Embrittlement in Austenite Region (1200 to 900°C)
1. Thermal History Dependence
The reheated and melted specimens of a low-car-bon steel (heat D in Table 1) were tested for tensile strength and reduction of area, with the former speci-mens heated at different maximum temperatures. The test results are given in Fig. 5 (=5/s). As the maximum heating temperature is raised, marked em-brittlement occurs in the temperature region of 1200 to 800°C. At the maximum heating temperature of 1400°C, the reheated specimens (denoted by open circles) show almost the same strength and ductility as the melted specimens (denoted by solid circles). When this low-carbon steel is heated to 1400°C, the axial portion of the specimen partly melts. Some of the impurity elements which contribute to this em-brittlement are believed to dissolve into the liquid phase and the rest are considered to go into solid solu-tion.

Embrittlement in this temperature region is sensi-tive not only to the maximum heating temperature, but also to the subsequent thermal history. The em-brittlement of a melted specimen, for example, may

be diminished if the specimen is cooled at a lower rate after melting and solidification, held at a certain temperature during cooling, or cooled just below the Arl point and reheated. Figure 6 shows the isoduc-tility curves (~=5f s) obtained when the same steel as shown in Fig. 5 was isothermally held at between 1200°C and 700°C on cooling after melting and solidification. The isoductility curves are reversed C in shape, and the nose is located at around 900°C. Holding at 1 100°C for 80 s or 900°C for 500 s or 700°C for 30 s yields 60% or more reduction of area (increment of ductility by holding).
The second characteristic of the embrittlement in

this temperature region is that it is extremely sensitive to the amounts of impurities, such as sulfur and oxy-gen, in the steel. When the steel has sulfur content of over 0.01 % and low Mn/S ratio as the one shown

in Fig. 6, for example, it embrittles pronouncedly.

Fig. 4. Strain rate dependence of near-melting temperature ductility of the electrolytic iron (heat A) and 0.4%
C steel (heat B).

Fig. 5. Hot ductility and strength curves for the plain car-bon steel (heat D) subjected to various thermal
cycles (=5/s). ~

Fig. 6. Effect of holding in the temperature range between 1200°C and 800°C on the hot ductility for the plain
carbon steel (heat D) subjected to the melting

treatment.

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When the steel is vacuum melted to sulfur and oxygen contents of 0.01 % or less (such as heats A and C in Table 1), on the other hand, it does not suffer any embrittlement. Similar results have been already re-ported by Lankford.2~ Decreasing the sulfur content or increasing the Mn/S ratio by manganese additions reduces the likelihood of the embrittlement.

2. Strain Rate Dependence of Ductility
Figure 7 shows changes in strength and ductility when the same steel (heat D) as shown in Fig. 5 was tensile tested at different rates of deformation at

1 000°C on cooling after melting and solidification. As the strain rate decreases, the ductility improves (embrittlement is relaxed). This result has been ob-tained with the low-carbon steel which has high sulfur and oxygen contents or a low Mn/S ratio. With melted specimens of steels with low sulfur and oxygen contents (for example, heats A, C and E), ductility in the temperature region of 1 200 to 900°C does not depend on the strain rate, and the reduction of area is 70% or over (~=5 x 10_3 to 20/s). Lankford2~ has reported that the embrittlement of a steel whose com-position is similar to that of heat D does not depend on the strain rate in the temperature region of 1 200 to 900°C. His results differ from those of the present experiment in this point.
3. Causes of the Embrittlement

To investigate the causes of the embrittlement in this temperature region, the fractured surfaces of ten-sile specimens were observed and analyzed. Scan-ning electron microscope fractographs of representa-tive specimens are presented in Photo. 3 for correla-tion with the embrittlement behavior shown in Fig. 6. Almost all of the fracture surfaces of specimens with 20% reduction of area show austenite grain boundary failure, while the fracture surfaces of specimens with over 60% reduction of area change to transgranular dimples. A detailed examination shows that inter-granularly fractured surfaces in the low-ductility spec-imens consist of dimples a few microns in diameter and that precipitates 0.2 to 0.51a in diameter are pres-ent in each dimple (A and B in Photo. 3). Charac-teristic X-ray analysis of these individual precipitates detected Fe-Mn-O, Fe-Mn-S and Al-0 signals (E, F and G in Photo. 3). Although some of the Fe sig-

nals are believed to have come from the iron matrix, these precipitates are presumably (Fe, Mn)O, (Fe, Mn)S and A1203. The fracture surface of the speci-men tensioned to rupture at 1 200°C is composed of transgranular dimples, and optical microscopy of a cross section near the fracture surface reveals few or

no precipitates on the grain boundaries. The frac-ture surfaces of specimens with ductility improved by holding for a long period of time in the temperature region of 1 100 to 900°C change to transgranular dimples. The sulfides and oxides present in the dim-ples are shown to grow to a few microns in diameter, and the spacings between the particles are increased (C and D in Photo. 3). The fracture surfaces of spec-imens tensioned to rupture (s=5/s) at temperatures of 800°C or less are mostly composed of transgranular dimples, and the observed precipitates are large in size (2 to 5 f in diameter) and small in quantity. These results suggest the following causes of the embrittlement in the temperature region of 1 200 to 900°C. The sulfur and oxygen which exist oversat-urated precipitate on the austenite grain boundaries in the form of (Fe, Mn)S and (Fe, Mn)O in the tem-perature range of 1 150 to 900°C during cooling after melting and solidification. The specimen exhibits the lowest ductility when it is subjected to tensile stress under this condition. When the specimen is cooled at a lower rate after solidification or held isothermally in the temperature region of 1 200 to 900°C, these precipitates coarsen and do not contribute to the em-brittlement. When the specimen is first cooled to just below the Arl point and then reheated, the pre-cipitates are retained in the grains and made harmless. When the maximum heating temperature is set at 1200°C or lower, these precipitates which contribute to the embrittlement are fixed within the grains and rendered harmless. Decreasing the strain rate also improves hot ductility (Fig. 7), and the fracture sur-face changes from intergranular failure to trans-granular dimples. Also in this case, improvement is probably caused by coarsening of the precipitates and reduction of intergranular cracking susceptibility dur-ing deformation.

4. Embrittlement in Temperature Region of 900 to 600°C
1. Strain Rate Dependence of Ductility
This temperature region is the r-a transformation
region for low-carbon steels, and is near the recrystal-lization starting temperature and also grain boundary sliding commencing temperature for a r single-phase steel. The largest feature of the embrittlement in this temperature region is that the lower the rate of deformation, the greater the embrittlement. Thus, this type of embrittlement is essentially different from the aforementioned embrittlement in the higher tem-perature region. Since it becomes more accentuated as the rate of deformation decreases, this embrittle-ment is closely related to transverse facial cracks in continuously cast slabs. This is the reason why the behavior of the embrittlement in the temperature re-gion of 900 to 600°C has come to draw wide atten-tion in recent years. As one example of this embrit-

Fig. 7. Strain rate dependence of ductility and strength at 1000°C for the plain carbon steel (heat D) sub-
jected to the melting treatment.

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tlement, Fig. 8 shows the dependence of reduction of area on temperature and strain rate, as investigated by repeating and tension testing specimens of a low-carbon steel (heat C in Table 1). The ductility is lowest at 750°C and decreases with decreasing strain

rate. When the Ar3 transformation temperature dur-ing cooling at a rate of 20°C/ s was measured by a dilatometer, it was 757°C. Thus, the temperature at which ductility shows a minimum value agrees closely with the temperature at which r-a transformation
commences.

2. Effect of Chemical Composition
The second feature of the embrittlement in the tem-

perature region of 900 to 600°C is that it is sensi-tive to the chemical composition involved in steels. Figure 9 shows the strain rate dependence of Nb-bearing steel (heat E). The minimum value of duc-tility is located at the same 750°C as the low-carbon steel shown in Fig. 8, but the temperature range where the embrittlement occurs as the rate of defor-

mation decreases is expanded. The Ar3 point of this steel is 620°C. This means that the embrittlement

occurs at a temperature higher than the apparent transformation temperature. The embrittlement of the Nb-bearing steel is probably due to the difficulty of recrystallization upon NbCN precipitation in the austenite region as well as by the r-a transformation, as discussed partly by Bernard et a1.9>

Photo. 3. Austenite grain
fracture (C, D)

boundary fracture (A,
taken from the plain

B) and
carbon

transgranular
steel (heat D).

dimple

Fig. 8. Dependence of ductility on the strain rate and test temperature ranged from 1 200 to 650°C for the
low carbon steel (heat C).

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The high-purity electrolytic iron (heat A) does not embrittle in this temperature region, but the r single-phase steel (heat F) exhibits embrittlement as the rate of deformation decreases in the same way as shown in Figs. 8 and 9. It is also found that the temperature range where carbon steels with high sulfur and oxy-gen contents (for example, heat D) embrittle is wid-ened.
3. Modes of Fracture and Causes of the Embrittlement

First, the investigated results of the fracture prop-agation process are described. The 750°C deforma-tion (E=5 X 10-3/s) of the Nb-bearing steel (heat E) was interrupted to investigate its structural change. In the initial stage of deformation, voids are initiated along austenite grain boundaries, and grain boundary sliding is observed at other sites. Near these austenite grain boundaries, proeutectoid ferrite films are form-ed. When deformation is carried out just before the final failure, the voids coalesce, and some of them show wedge-shaped intergranular cracks (Photo. 4). Scanning electron microscope fractographs of a bro-ken specimen are given in Photo. 5. The fracture

surface shows austenite grain boundary cracks when observed at low magnification. At high magnifica-tion, the fracture surface is revealed to consist of fine dimples, and precipitates are present at the bottom of each dimple. The precipitates shown in Photo. 5 are those identified as (Fe, Mn)S and (Fe, Mn)0. Em-brittled specimens of the low-carbon steel (heat C) shown in Fig. 8 are similar to the Nb-bearing steel in fracture mode, but the size of individual dimples is larger than in the case of the Nb-bearing steel. The optical microscope observation of areas near the frac-ture surfaces of specimens tensioned to rupture at dif-ferent temperatures and strain rates confirmed the following facts. In the structure which shows a minimum value of ductility (at 750°C in the case of heat C) as the rate of deformation decreases, pro-eutectoid ferrite films are formed along coarse aus-tenite grain boundaries, and deformation mostly oc-curs in this constrained ferrite region. When ferrite transformation is promoted (at 700°C or less) or when high-speed deformation is effected, however, the entire specimen is uniformly deformed with no stress con-centration near the austenite grain boundaries.
These results suggest the following mechanism of deformation. When tensile stress is imposed on car-bon steels or the Nb-bearing steel with sulfides, carbides or nitrides precipitated at austenite grain boundaries, these precipitates become sources of stress concentration, causing the formation, growth and coa-lescence of voids. When grain boundary sliding and deformation occur during this process, void growth is accelerated. Precipitation of proeutectoid ferrite along the grain boundaries acts to localize deforma-tion near the grain boundaries. The fact that the tensile strength of the proeutectoid ferrite is several times smaller than that of austenite (at the same tem-perature)10~ may be taken to support this finding. The reasons why embrittlement occurs in a tempera-

Fig. 9. Dependence of ductility on the strain rate and test temperature for the Nb bearing steel (heat E).

Photo. 4.
Optical micrographs showing void forma-
tion (A), and wedge type crack (B) at prior
austenite grain boundary, taken by interrup-tion of tensile test (750°C, i = 5 X 10_3 Js) for the Nb bearing steel (heat E).

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ture region higher than the apparent Ar3 temperature as in the case of the Nb-bearing steel are that NbCN precipitates during deformation to make recrystalliza-tion difficult9~ and that deformation accelerates the 1-a transformation.11 Figure 10 schematically illus-trates the intergranular embrittlement of carbon steel. This concept can fully explain why the embrittlement is difficult to occur in alloys which have difficulty in forming proeutectoid ferrite films along the austenite grain boundaries as the high-purity electrolytic iron (heat A) does. It can be easily inferred also that grain-refined steels become more difficult to embrittle. The principal cause of the embrittlement in the r single-phase steel (heat F) cannot be, however, ex-plained by what has been described above. There is a possibility that the embrittlement may occur by the same mechanism as similar embrittlement12~ occurring in nonferrous alloys and grain boundary sliding13,14~ recognized under high-temperaturecreep. This sub-ject calls for further study.

Iv. Conclusions

As one aid in clarifying the mechanism of cracking
in continuously cast slabs, representative steels were tensile tested on cooling after melting and solidifica-tion by simulating the thermal history to which slabs are subjected, and the characteristics of embrittlement which occurs in high-temperature regions were studied.

(1) Embrittlement which occurs in the tempera-ture range between the melting point and 600°C can be divided into three regions of occurrence according to the temperature range where embrittlement occurs

and the characteristics of embrittlement. Figure 11 schematically illustrates ductility variations from one region to another.
(2) In region I which is close to the melting point, the liquid phase causes the embrittlement, and duc-tility does not depend on the strain rate.

Photo. 5. Fracture surfaces and characteristic X-ray spectra showing (Fe, Mn)S and
(Fe, Mn)0 precipitates at the bottom
of fine dimple. Tensile tested at
750°C and i=5 X 10_3/s for the Nb
bearing steel (heat E).

Fig. 10. Schematic presentation of grain boundary embrit-tlement in the temperature range between 900°C
and 600°C.

Fig. 11. Schematic presentation of ductility
pearing in hot tensile test.

troughs ap-

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(3) In region II which corresponds to a stable austenite region, embrittlement occurs along grain boundaries as a result of intergranular precipitation of sulfides and oxides. This embrittlement becomes

more pronounced as the rate of deformation increases. Melted specimens are more susceptible to the cracking than reheated specimens. This cracking susceptibil-ity of melted specimens can be diminished by decreas-ing the cooling rate or by isothermal holding at be-tween 1 200°C and 900°C on cooling.

(4) In region III which covers temperatures of 900 to 600°C, the degree of the embrittlement in-creases as the rate of deformation decreases. This

embrittlement is caused by such factors as intergran-ular precipitation, formation of proeutectoid films along austenite grain boundaries, and grain boundary sliding.

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