Solidification Behavior of Iron-Niobium and Iron-Carbon-Niobium alloys

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Abstract. The undercooling and solidified structure of Fe-Nb binary alloys and Fe-0.1mass%C-Nb ternary alloys were investigated by changing Nb concentration from 0.5 to 6mass%. A glass-encased method was used for achieving high undercooling, in addition to a conventional solidification method. The specimen was furnace-cooled followed by quenching from 1723K or air-cooling from 1473K. As a result, the degree of undercooling in the Fe-Nb alloys solidified by the glass-encased method was higher than that in the specimen solidified by the conventional method, while that in the Fe-0.1mass%C-Nb alloy solidified by the glass-encased method was not so high. The solidified structure in all alloys that were quenched at γ formation temperature has dendritic morphology in spite of δ / γ transformation. The secondary arm spacing of the dendrite became fine with increase in niobium concentration.

Introduction

It is important to control the α grain size of commercial carbon steel because the mechanical properties of carbon steel is controlled by the size of α crystal grain. Following concepts have been suggested in order to fine α crystals in iron-based alloys［1］. The first is refining γ crystal grain, the second is increase of a cooling rate at the γ / α transformation, the third is utilization of deformation such as cold working, and the forth is the utilization of non-metallic inclusion for nucleating α crystal grain. The third and fourth methods have already been applied to commercial steels. However, there are some kinds of steels to which these methods cannot be applied. Then, the first is an attractive method to refine the α grain size. The γ crystal grain grew rapidly when δ phase was completely disappeared in the hypo-peritectic carbon steel and when liquid phase decreased to a certain value in hyper-peritectic carbon steel［2,3］. This indicates that if a certain phase such as the δ phase and liquid phase coexists with γ phase to lower temperature, the γ grain size comparable to secondary dendrite arm is formed. Particularly when niobium as a ferrite former is added to the hypo-peritectic carbon steel, the δ / γ coexisting region is extended［2］. However, there is less information on solidification behavior of steel containing niobium as a main solute element, even if solidification behavior of steel containing niobium as a minor element was investigated［4,5］. Furthermore, there is no study whether niobium-carbide formed before solidification in Fe-C alloy acts as a heterogeneous nucleus or not.

In this study, changes in undercooling and secondary dendrite arm spacing in the Fe-Nb and Fe-C-Nb alloys which were solidified by a equiaxed solidification method and a glass-encased

method were investigated by changing niobium concentration in order to clarify the structure change from δ dendrite to γ grain and the possibility of heterogeneous nucleation due to niobium carbide.

Experiment

Phase diagram of a Fe-Nb system. The Fe-Nb binary phase diagram is illustrated in Fig.1［6］. The starting temperature of the γ single phase decreases from 1667K at 0%Nb to 1451K at 1.7 mass% Nb. Two ideas are considered in order to coexist two phases. The one is increase the Nb concentration over 1.7mass% and the other is the addition of third element such as carbon to change the phase diagram. Furthermore, the γ grain growth is expected to be suppressed by the pinning effect of NbC if it forms during solidification of the Fe-C-Nb alloys. On the other hand, the nucleating potency of NbC was investigated since fine dendrite formation is also expected if NbC acts as heterogeneous nuclei.

Experimental procedure. The specimens of Fe-Nb binary alloys and Fe-0.1mass%C-Nb ternary alloys were prepared by changing Nb concentration from 05 to 6mass%. Hereafter the mass% is abbreviated as %. An alumina crucible was set in a uniform temperature zone in an electric furnace for achieving equiaxed solidification. The alumina crucible with 35mm in inner diameter and 45mm in depth with a round bottom was used and the 140g specimen was melted in an argon atmosphere after evacuation. The specimen was kept at 1823K for 3.6ks and was cooled with following two methods. One was rapid quenching from 1673K after cooling at 0.028K/s and the other was air-cooling from 1473K after cooling at 0.028K/s. The experiment was done by a conventional solidification method and a glass-encased method. In the latter method, the sample was melted and solidified together with commercial glass. The temperature was measured by B-type thermocouples located in the sample at 10mm from the crucible bottom. A cross section of the specimen was etched by a mixture of ethanol of 80ml, nitric acid of 20ml, picric acid of 1g. The secondary dendrite arm spacing was measured by the linear intercept method. The niobium concentration crossed primary dendrites was measured by an X-ray micro-analyzer.

Results and Discussion

Undercooling. The change in undercooling against Nb concentration in Fe-Nb and Fe-0.1%C-Nb alloys solidified by the conventional method is shown in Fig.2. Most alloys show the small undercooling, while the undercooling in the Fe-1%Nb and Fe-0.1%C-Nb alloys is somewhat higher. As shown in Fig.3, the undercooling of the Fe-3%Nb and Fe-0.1%C-3%Nb alloys solidified by the glass-encased method is higher than that in the specimen solidified by the conventional method. Summarizing above results, the degree of undercooling in the specimen including carbon became smaller in spite that the glass-encased melted was used. The solubility of Nb in the molten iron with 0.1%C is shown in Fig.4［7］. Since NbC can form before the crystallization of δ phase in the specimen including Nb over about 1.0%, the reduction of undercooling in the Fe-C-Nb alloy may be caused by heterogeneous nucleation due to the NbC.

Solidification structure. Figure 5 shows the solidification structure of Fe-3%Nb alloy quenched

from 1673K. The structure is dendritic and much liquid was retained among dendrites. The remaining liquid in the inter-dendrite region became more with increasing niobium concentration

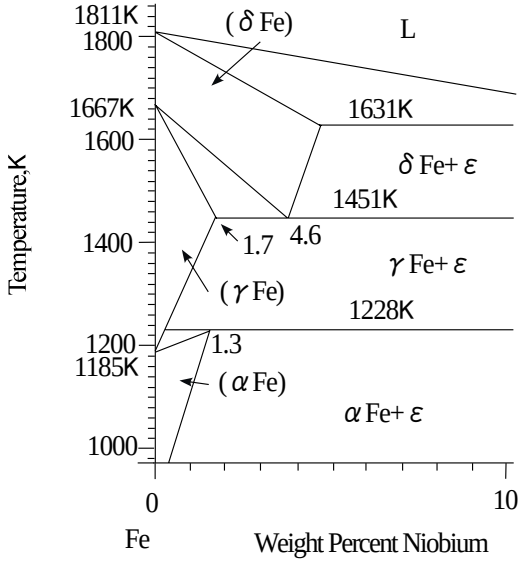

Fig.1 Phase diagram of Fe-Nb.

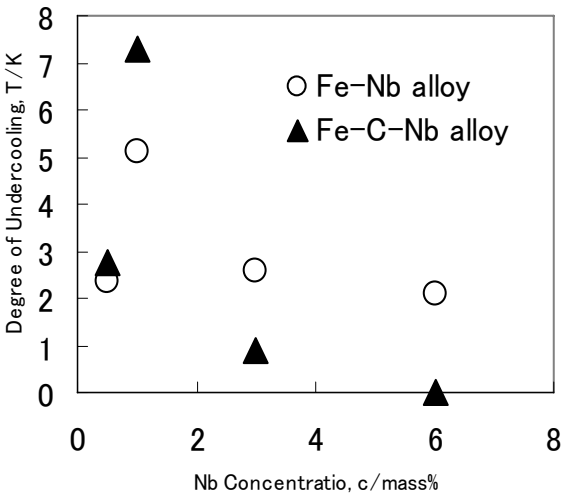

Fig.2 Change in undercooling in Fe-Nb and Fe-0.1%C-Nb alloys by conventional method.

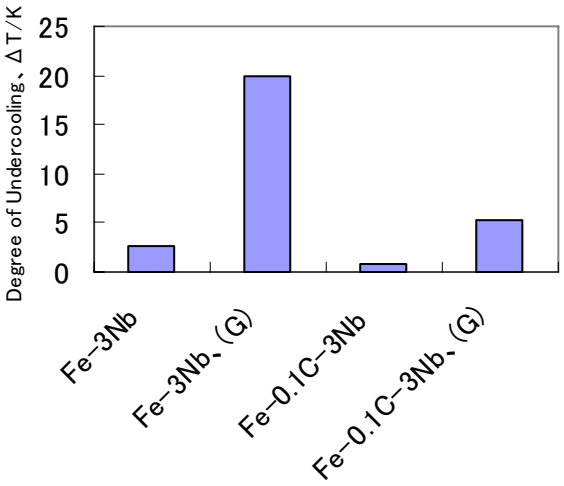

Fig.3 Difference in undercooling due to two solidification methods.
G:Glass-encased method.

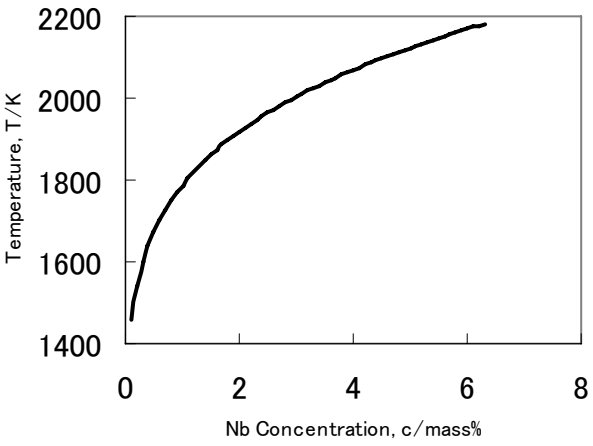

Fig.4 Formation temperature of NbC in Fe-0.1%C-Nb alloy.

since the specimen was quenched at a constant temperature. The solidification structure of Fe-0.1%C-3%Nb alloy, shown in Fig.6, is also composed of dendrites and inter-dendrite liquid. Therefore, the effect of C on the dendritic morphology might be small because the dendrite structures of Fe-3%Nb and Fe-0.1%C-3%Nb alloys are similar. The solidification structures of Fe-3%Nb and Fe-0.1%C-3%Nb alloys solidified by the glass-encased method show also dendrite structure even though the glass might influence dendrite growth. On the other hand, the grain

structure of the Fe-0.1%C-3%Nb alloy solidified with air-cooling corresponded to secondary dendrite arm spacing. This means that fine grain comparable to the secondary dendrite arm spacing can be obtained by extending the coexistent region of two phases.

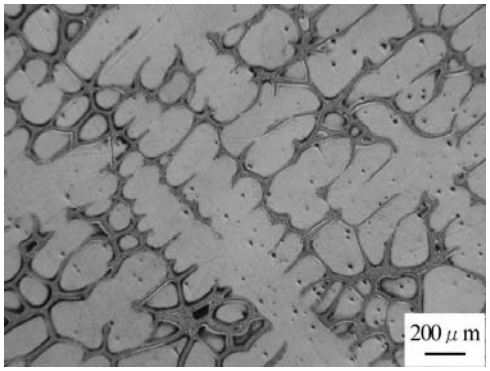

Fig.5 Dendrite morphology of Fe-3%Nb alloy.

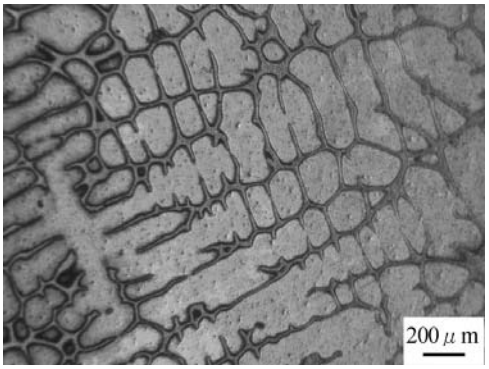

Fig.6 Dendrite morphology of Fe-0.1%C-3%Nb alloy.

Secondary dendrite arm spacing. Secondary dendrite arm spacing in the Fe-Nb and Fe-0.1%C-Nb alloys decreases with increasing niobium concentration and the decreasing tendency is seemed independent on the solidification methods. The secondary arm spacing was almost independent of the degree undercooling, since the maximum degree of undercooling was about 19K in this experiment.

Summary

The undercooling and solidification structure of Fe-Nb binary alloys and Fe-0.1%C-Nb ternary alloys were investigated by changing Nb concentration from 0.5 to 6% and carrying out the conventional solidification method and the glass-encased method. As a result, the degree of undercooling in Fe-Nb alloys solidified together with glass was higher than that in the specimen solidified by the conventional method, while that in Fe-0.1%C-Nb alloy solidified by the glass-encased method was low. The secondary dendrite arm spacing became finer with increasing niobium concentration.

References

［1］T.Maki: CAMP-ISIJ, 13(2000),730.
［2］T.Maruyama,K.Matsuura,M.Kudoh and Y.Itoh: Tetsu-to-Hagane, 85(8)(1999),585.
［3］T.Maruyama,M.Kudoh and Y.Itoh: Tetsu-to-Hagane, 86(2)(2000),86.
［4］M.Leonhardt, W.Löser andH.-G.Lindenkreuz: Acta mater, 47(10)(1999),2961.
［5］M.Leonhardt, H.-G.Lindenkreuz, W.Löser and J.Eckert: Materials Science Forum, 312-314 (1999),275.
［6］T.B.Massalaski, et al: Binary phase diagrams, vol.2 (1990),1732.
［7］Metals Data Book, JIM, Maruzen, (2004),170.