Development of new additive for grain refinement of austenitic stainless steel

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Recently, a new additive for grain refinement of Ni based superalloys[1,2] has been developed. In those studies, it was considered that niobium carbide in Ni–Nb–C alloy additives would act as nuclei on the solidification of Ni based superalloys. It is known that the crystallographic characteristics of iron are quite similar to those of nickel. Thus, it was expected that niobium carbide would act as a grain refiner for iron base alloys, especially for austenitic steels. In this study, the effect of the additives on the microstructure of SUS316 steel was examined in various experimental conditions. The grain size of SUS316 specimens without inoculation was ~ 2700 μm. On the other hand, when NbC containing alloy additives were added into the SUS316 melt, fine equiaxed grains were observed and the grain size of the specimen was significantly reduced to ~ 200 μm.

Keywords: Grain refinement, Inoculation, Austenitic stainless steel, SUS316, Niobium carbide?>

Introduction

In recent years, it is strongly required to reduce the weight of transport machines, mainly automobiles, to save the natural resources such as metallic elements and fossil fuel. To reduce the weight of those machines, it is essential to improve the strength of the metallic components built in. It is well known that mechanical properties of metallic materials, such as hardness, tensile strength and low cycle fatigue property, will be improved by decreasing the grain size of metallic materials.[1–5] Thus it is expected that the weight of metallic components would be reduced efficiently by developing the grain refinement technique. Nowadays, several grain refinement techniques have been developed, such as rapid cooling process and low temperature casting process. However, those processes have some problems that the special equipments are required and that the size and the shape of the castings are limited due to their severe fabrication conditions. On the other hand, the inoculation method is the most promising method to solve those problems due to its very simple process. The authors have developed a new additive for grain refinement of Ni based superalloys[1,2] and it was suggested that niobium carbide in the additives would act as nuclei on the solidification of Ni based superalloys. Since the crystallographic characteristics of nickel and iron are quite similar, it was expected that niobium carbide would be effective as well for the grain refinement of iron based alloys, especially for austenitic steels. In

this study, the effect of a new additive containing NbC on the microstructures of SUS316 austenitic stainless steel was examined.

Experimental

The chemical composition of SUS316 steel used in this study is shown in Table 1. Four types of additives were prepared as follows: first, Fe–C alloys were prepared by melting electrolytic iron and electrode graphite in a high frequency induction furnace under an argon gas atmosphere. Then the Fe–C alloys and pure niobium (99·5% purity) were alloyed by arc melting to obtain Fe–Nb–C alloys as additives. Microstructures of the Fe–Nb–C alloys were observed by an optical microscope and a scanning electron microscope (SEM). Inoculation treatment was carried out as follows: SUS316 steel of about 90 g was melted at 1773 K in an alumina crucible with inner diameter of 20 mm. After holding at 1773 K for 30 min, the alloy melt was cooled to 1726 K and held for 20 min. Then, a certain amount of additive was added into the alloy melt. The alloy melt was stirred for 5 s with a silica tube and held for a predetermined time, followed by air cooling. The microstructure of the specimen was observed on the transverse sections with an optical microscope and the grain size was measured. The evaluation method of grain size is shown in Fig. 1. When grain size was macroscopic scale, it was measured on three concentric circles with 1/4r, 1/2r and 3/4r in radius (r is the radius of the specimen) as shown in Fig. 1a. When grain size was small, of the order of micrometre, it was evaluated by counting the number of intersections of grain boundaries with the several lines randomly drawn on the microstructures of transverse section (Fig. 1b).

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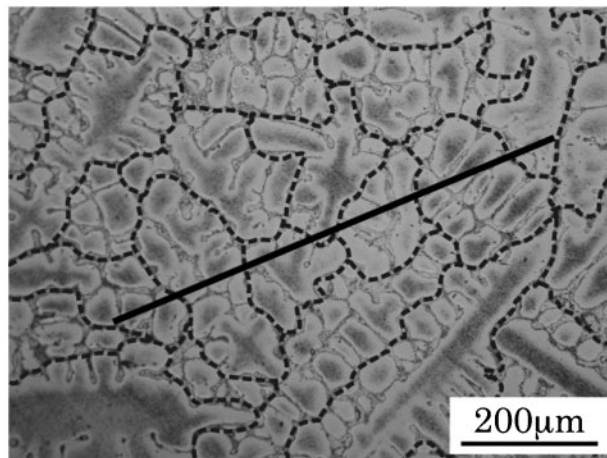
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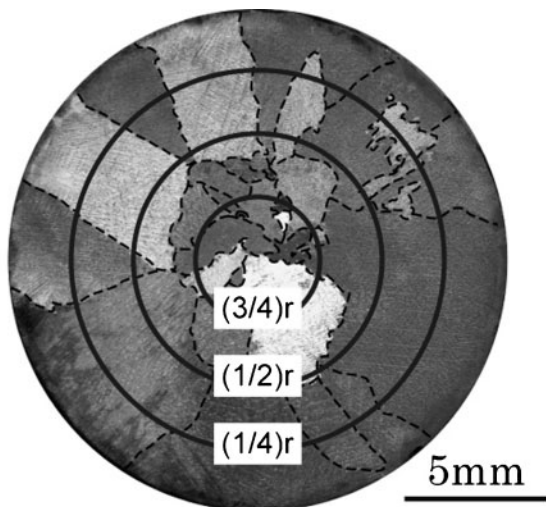
The average grain size : G=L/n (mm/number)

※L : The circumference of
a circle measured.

n : The number of grains which

(a) were intersected by the circle.

Microstructure

The average grain size : G=L/n (μm/number)

※L : The circumference of the line.

n : The number of grains which

were cut across by the line. (b)

1 Evaluation method of grain size

Results and discussion

Microstructure of Fe–Nb–C alloy additives

For comparison, the Ni–Nb–C alloy additive, which showed a significant effect on the grain refinement of nickel based superalloys,[1,2] was added into molten SUS316 steel. The fine equiaxed grains were observed at the bottom part of the specimen and the rest was coarse columnar structure. This result indicates that the additive was not dispersed throughout the melt and sedimented at the bottom part of the crucible. In addition, the fine equiaxed grains observed at the bottom of the specimen suggest that a component in the Ni–Nb–C additive, possibly NbC, has the effect on grain refinement of SUS316 steel. The calculated specific weight of Ni–Nb–C alloy additive and molten SUS316 steel are 7980 and 7250 kg m^{-3} at 1450 K respectively. The specific weight of the Ni-Nb-C alloy additive is much larger than that of SUS316 steel. This difference of the specific weight is considered to be attributable to the sedimentaion of the additive at the bottom part of the specimen. To avoid the sedimentaion of the additive, Fe–Nb–C alloys which involve NbC crystals and have almost the same specific weight
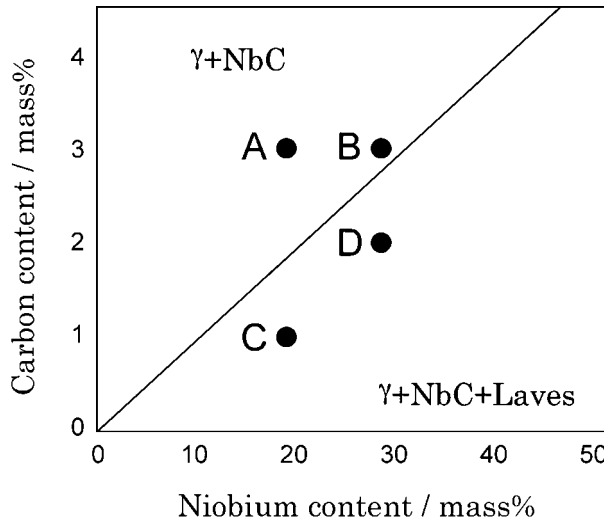

2 Phase constituents of Fe–Nb–C ternary system

as that of molten SUS316 steel were prepared as additives. Figure 2 shows the phase constituent diagram of ternary Fe–Nb–C system[6] and the chemical composition of four Fe–Nb–C alloys used in this study. Figure 3 shows the microstructures of the four types of Fe–Nb–C alloy additives. Coarse primary NbC crystals were observed in the microstructure of all specimens. The area fractions of primary NbC

Table 1 Chemical composition of SUS 316 steel, mass%

C	Si	Mn	P	S	Ni	Cr	Mo	Fe
0·08	1·00	2·00	0·045	0·030	18	12	2·5	Bal.
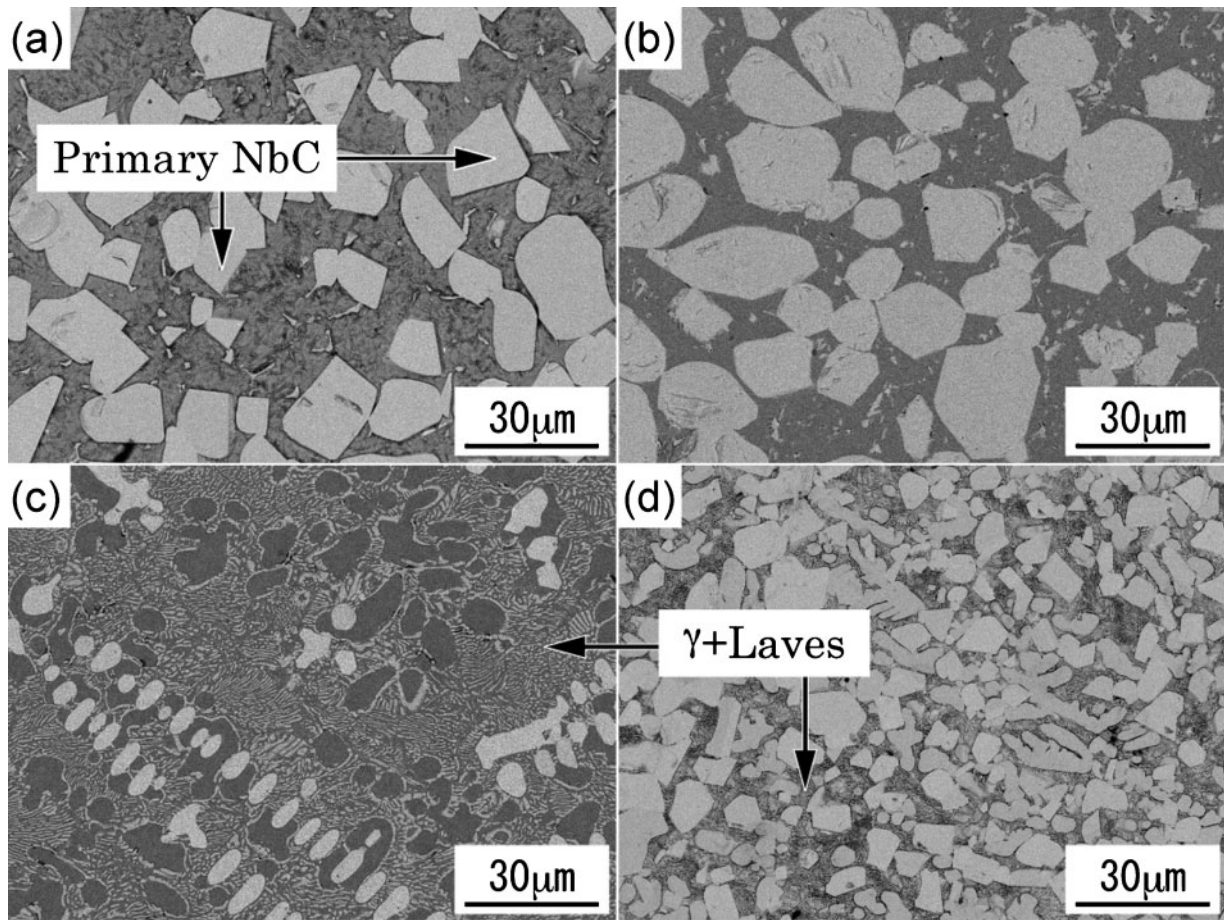
a Fe–20Nb–3·0C alloy (additive A); *b* Fe–30Nb–3·0C alloy (additive B); *c* Fe–20Nb–1·0C alloy (additive C); *d* Fe–30Nb–2·0C alloy (additive D)

3 Microstructures of Fe–Nb–C alloys

crystals in the additives A, B, C and D measured by image analysis were 36·9, 38·2, 8·3 and 36·3% respectively. The matrix structure in the additives C and D are a eutectic consisting of γ and Fe_2Nb (Laves phase).
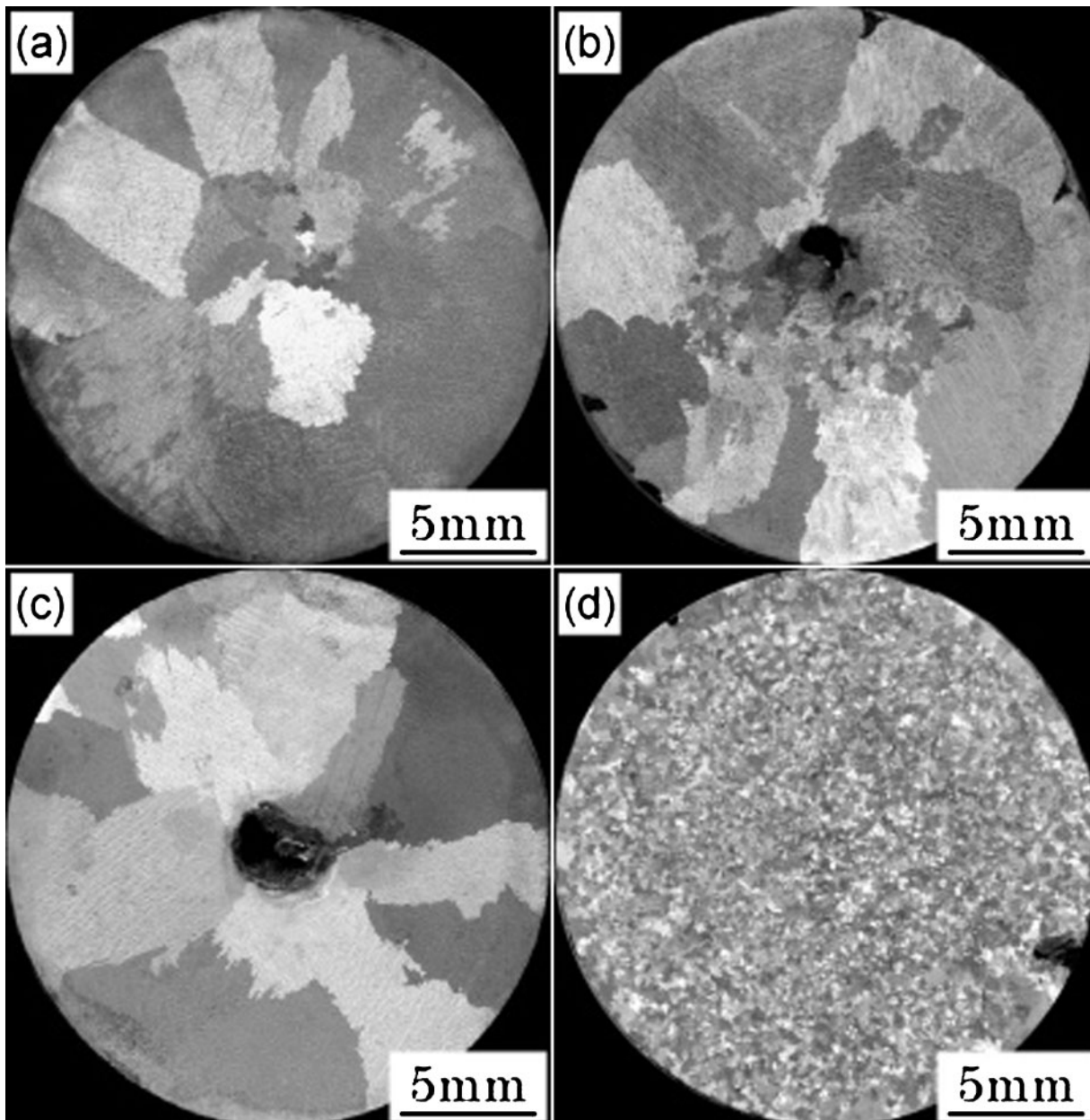
Effect of amount of additives on grain size

To investigate the effect of amount of additive on the grain size of SUS316 steel, the additive D (1·0–3·0 mass%) were added into molten SUS316 steel. The granule size of the additive was sieved to 0·5–1 mm. The holding time was 0 s. Figure 4 shows the macrostructures of SUS316 steel after addition of various amounts of the additive D. The relationship between the grain size and the amount of additive is shown in Fig. 5. When the amount of additive was 0–2 mass%, coarse columnar structure was observed as shown in Fig. 4a–c. On the other hand, when the amount of additive was 3 mass%, fine equiaxed grains were observed over the whole area of the section (Fig. 4d) and grain size was significantly reduced to 210 μm. These results indicate that the amount of additive more than 3 mass% is required to achieve the grain refinement of SUS316 steel.

Effect of holding time on grain size

To investigate the effect of holding time on the grain size, molten SUS316 steel was held for various periods after the addition of additive D of 3 mass% at 1726 K. Macrostructures and average grain sizes of the specimens for different holding time are shown in Figs. 6 and 7 respectively. When the holding time was 0 s without stirring, fine equiaxed grains were observed at the centre of the specimen as shown in Fig. 6a. When the holding time was 0 s after stirring for 5 s, fine equiaxed grains were observed (Fig. 6b) and the grain size was ~210 μm. However, when the holding time was 5–10 s, coarse columnar structures were observed (Fig. 6c and d) and the effect of additives seems to disappear. It is suggested that the Fe–Nb–C alloy additives with granule size of 0·5–1 mm would have very high dissolution rate in the melt of SUS316 steel.

Figure 8 shows the experimental result of the addition of additive D with a larger granule size 1~1·5 mm for different holding times. In this case, refining effect of the additive was maintained up to 45 s (Fig. 7) and this suggests that the grain refinement of SUS316 steel in this study resulted from the addition of Fe–Nb–C alloy additives and not from the rapid cooling of the melt caused by the inoculation. Therefore, the optimum conditions for the grain refinement of SUS316 are as follows: the inoculation temperature 1726 K, the stirring time 5 s, the holding time 0 s for the granule size of the

a no addition; *b* 1 mass%; *c* 2 mass%; *d* 3 mass%
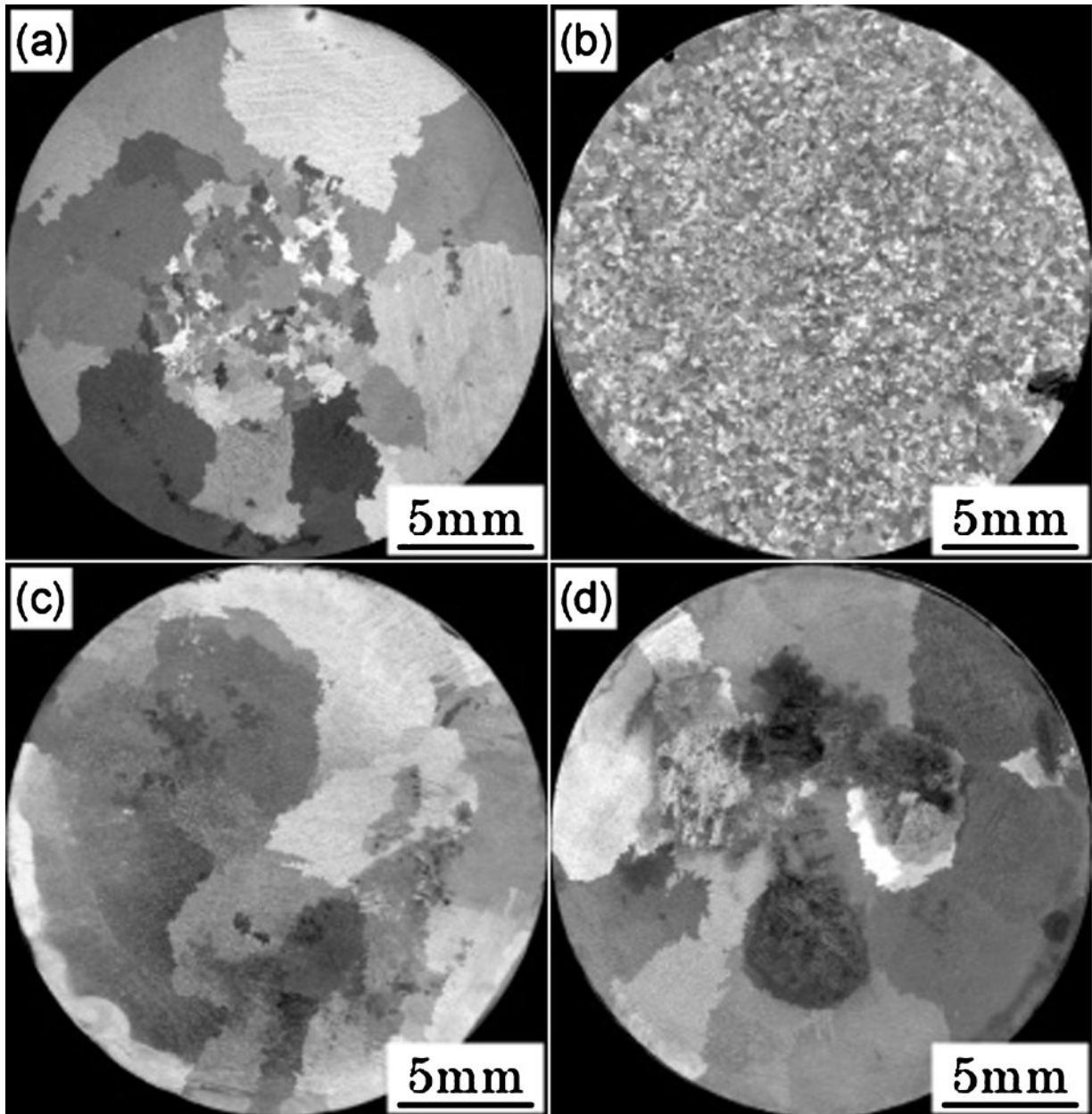**4 Macrostructure of SUS316 steel after addition of various amounts of additives D**
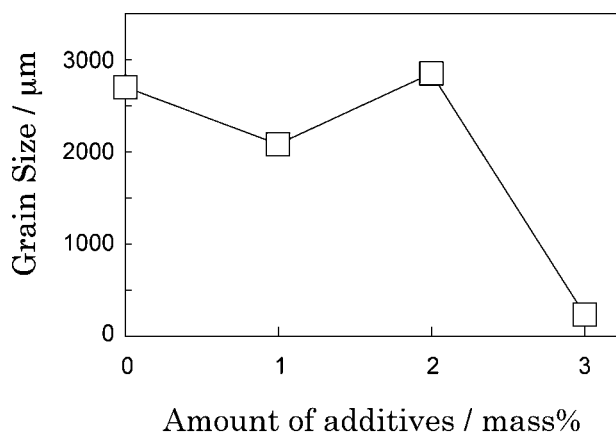
5 Effect of amount of additive D on grain size

additive 0·5–1 mm and 0–20 s for the granule size of 1–1·5 mm.

Effect of area fraction of primary NbC crystals on grain size

In the previous works on the grain refinement of Ni based superalloys, it was found that more than 10% of the area fraction of primary NbC crystal in the Ni–Nb–C alloy additives was required to achieve the grain refinement. Thus, it was expected in this study, that the area fraction of primary NbC in the Fe–Nb–C alloy additives would influence the grain refinement. In the experiments, four additives which have various area fractions of primary NbC shown in
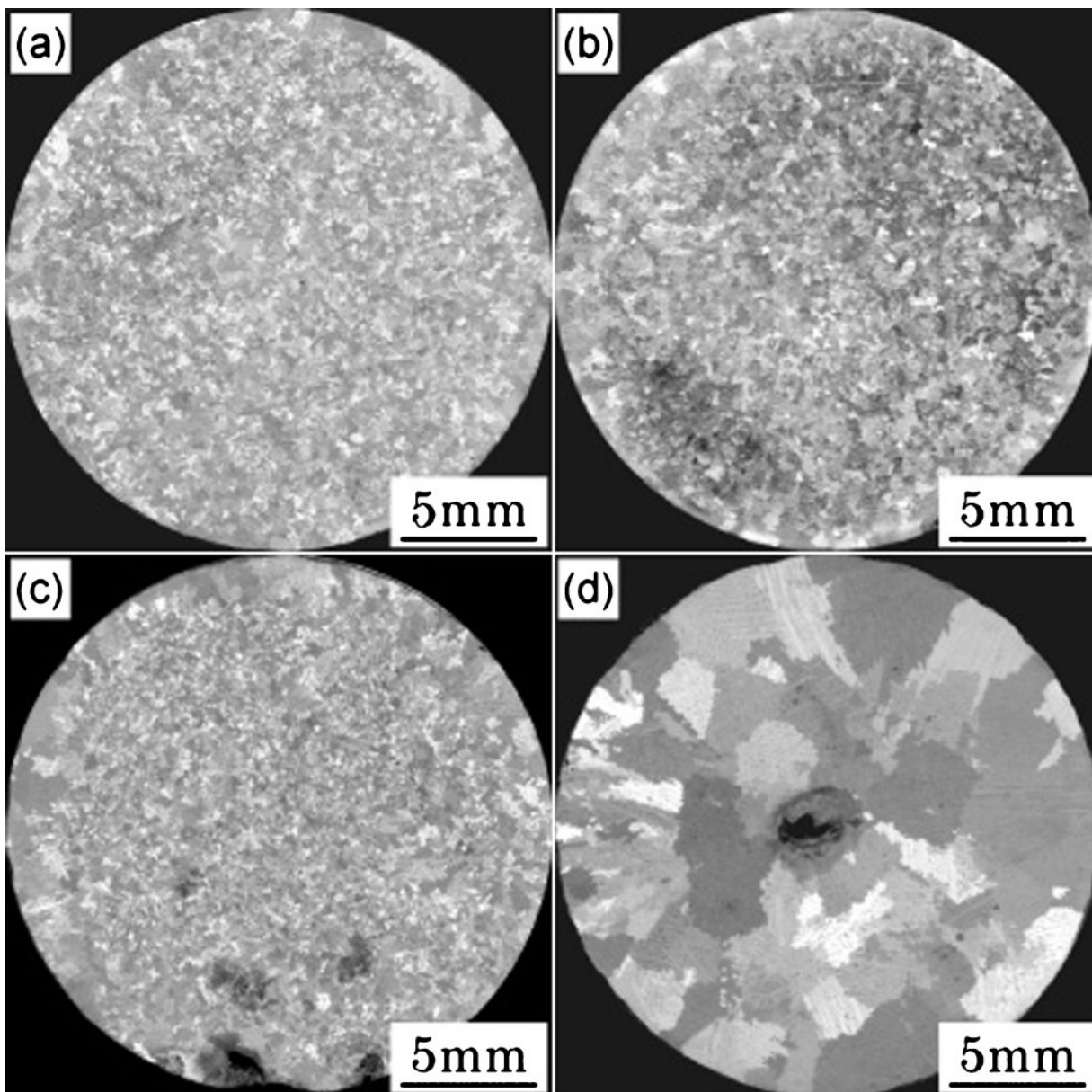
a no stirring and holding for 0 s; *b* stirring for 5 s and held for 0 s; *c* stirring for 5 s and held for 5 s; *d* stirring for 5 s and held for 10 s

6   Effect of holding time on macrostructure of SUS316 steel (additive D with 0·5–1 mm in granule size)
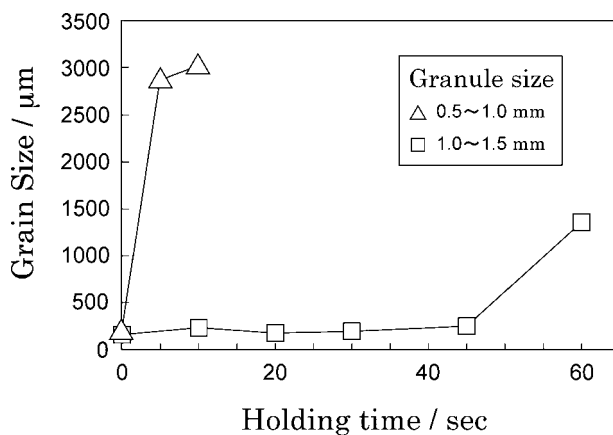
7   Relationship between grain size and holding time for different granule size of additive D

Fig. 2 were added into the molten SUS316 steel. The experimental condition was the optimum conditions for the grain refinement as mentioned before. The macrostructures of four specimens with the addition of additive A–D are shown in Fig. 9. In the cases of the additive A, B and D, fine equiaxed grains with the grain size of ~ 200 μm, were observed, while slight coarse equiaxed grains, ~ 1000 μm, were observed in the case of additive C, as shown in Fig. 8*c*. Figure 10 shows the relationship between the area fraction of primary NbC and the grain size. When the area fraction of primary NbC was lower than 10%, the effect of the additive on the grain refinement was not so significant.

a stirring for 5 s and held for 0 s; *b* stirring for 5 s and held for 10 s; *c* stirring for 5 s and held for 20 s; *d* stirring for 5 s and held for 60 s

8 Effect of holding time on macrostructure of SUS316 steel (additive D with 1–1·5 mm in granule size)

Conclusions

1. The area fraction of primary NbC crystals in Fe–Nb–C alloy additives tends to increase with increasing Nb and C concentrations of the additives.

2. When the amount of additives was less than 3 mass%, coarse columnar structure was observed. On the other hand, when the amount of additive was 3 mass%, fine equiaxed grains were observed and grain size was significantly reduced to 210 μm. This indicates that the primary NbC crystals would act as nuclei for primary γ on solidification of SUS316 steel.

3. When the holding time after the addition of additives and the subsequent stirring of the melt was over 5 s, no effect was observed for a finer additive with granule size of 0·5–1 mm, indicating that the additive dissolved rapidly into the melt. On the other hand, when a coarser additive with granule size of 1–1·5 mm was employed, the refiner effect was maintained up to 45 s.

4. Optimum conditions for the grain refinement of SUS316 are as follows: the inoculation temperature 1726 K, the stirring time 5 s, the holding time 0 s for the additive the granule size of 0·5–1 mm and 0–20 s for the additive with granule size of 1–1·5 mm.

5. When the area fraction of primary NbC crystals in the additives was lower than 10%, the effect of the Fe–Nb–C alloy additives was not significant and thus additives having a higher area fraction of NbC are desirable to achieve the grain refinement of SUS316 steel.
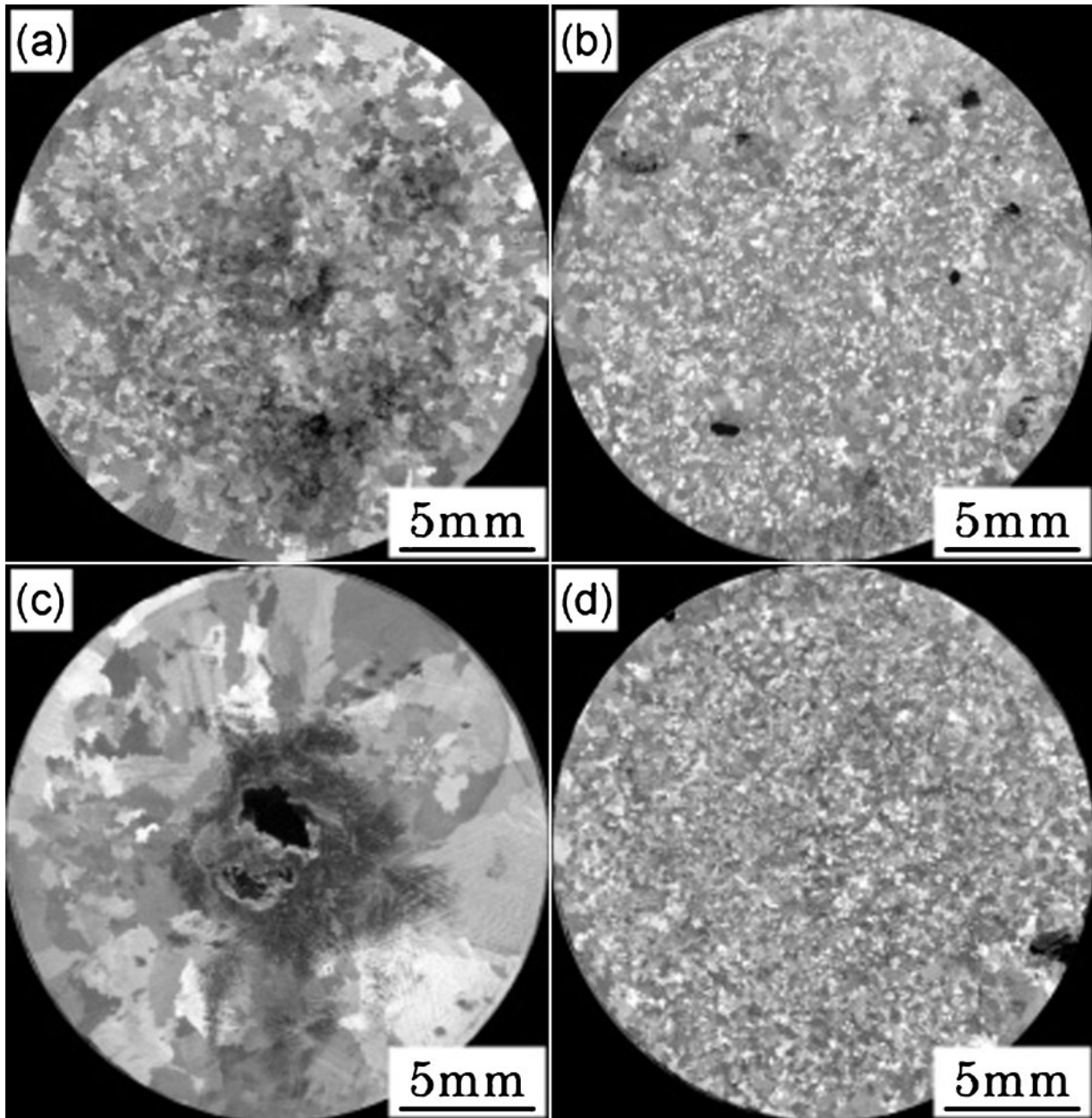
a additive A; *b* additive B; *c* additive C; *d* additive D

9 Macrostructures of SUS316 steels after inoculation with additives having different area fractions of NbC
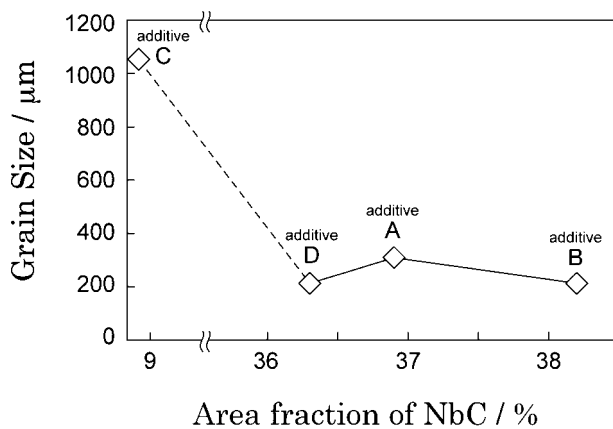
10 Effect of area fraction of NbC in additives on grain size of SUS316 steel

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