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Effects of Isotopically Controlled Boron Addition on Microstructure of
Nickel Irradiated at the Below Core Canister of FFTF

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Synopsis

Nickel specimens doped with several levels of ^{10}B were irradiated in the Fast Flux Test Facility (FFTF) for the purpose of examining the applicability of boron addition to the study of helium production effects. It was found that the boron has its chemical and transmutative effects, the former suppressing and the latter enhancing void nucleation. It seems that reliable estimation of helium effects is possible by well designed experiments separating some side effects of boron from its transmutation effects.

I. Introduction

For the purpose of examining the effects of high helium production rate during irradiation, which is a major characteristic of fusion neutron irradiation effect, irradiations of boron-doped specimens were performed or are going on in fast reactors^{1,2}). Although high helium production rate is achieved by this technique, it was pointed out that the boron addition causes unique influences on microstructures which are not typical of fusion neutron environment. For example, boron produces, in addition to helium, the same amount of lithium which may also affect microstructures. Moreover, boron has its chemical, not transmutative, effects on microstructures. Furthermore, segregation or precipitation of boron can lead to heterogeneous helium production in materials.

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The last issue has been examined by careful TEM observations and theoretical analyses^{1,3-5}). The microstructures in the vicinity of boron-precipitates clearly showed the effect of heterogeneous helium production. Moreover, they demonstrated considerable influence of lithium produced at the same time.

Chemical effects of boron on microstructures are likely to be estimated separately from its transmutative effects by the use of isotopically-controlled boron because only ^{10}B , whose natural abundance is about 20%, contributes to the transmutation. This is the major objective of the present study.

II. Experimental

Several kinds of boron-doped nickel specimens were loaded at the Below-Core-Canister of the Fast Flux Test Facility/Materials Open Test Assembly (FFTF/MOTA), for the irradiation at its cycle 10. Neutron irradiations were performed at 365°C and $3.33 \times 10^{26} \text{ n/m}^2$, ($E > 0.1 \text{ MeV}$), which corresponds to 12.2 dpa according to a preliminary calculation. The specimen matrix is listed in Table 1. A-1 is a pure nickel. The natural boron (20% ^{10}B) was doped in A-3 and A-5 and the enriched boron (91% ^{10}B) in A-2, A-4 and A-6, respectively. Table 1 also indicates helium production and He/dpa ratio. The helium production for A-1 and A-6 were measured in Rockwell International⁶) and those for other specimens were calculated from these data, chemical compositions and isotopic ratio of boron in the respective specimens. Using the measured amount of helium production, the time dependence of the He/dpa ratio is calculated. The results, shown in Fig. 1,

Table 1. Boron doping and helium production in the present specimens.

DESIGNATION	B(total) (appm)	^{10}B (appm)	He (appm)
A-1	<10	<10	12
A-2	59	54	29
A-3	761	152	61
A-4	411	378	134
A-5	5210	1040	347
A-6	3340	3070	1002

indicate that the He/dpa ratio decreases with time and finally becomes 2/3 of the initial value at the end of the cycle.

After irradiation, the specimens were unloaded, sorted and shipped to the Oarai Branch, Institute for Materials Research of Tohoku University. The specimens were electropolished and examined with JEM-200CX electron microscope.

III. Results

Voids and dislocations were observed in all specimens examined. Fig. 2 shows the voids in matrices. The swelling of all cases were within 1.5 and 2.5%. However, the void density is quite different in different boron-doping conditions. The void density measured are summarized as a function of the helium production in Fig.3. The remarkable difference in the void density between ^{10}B and ^{11}B doped specimens clearly suggests that the chemical effects of boron should be operating as well as its transmutative effects.

Boron-precipitates were rarely observed in the specimens. Fig. 4 is an example showing void distribution near a precipitate. Double halos of fine-void regions indicates lithium(inside) and helium(outside) projection effects respectively. Fig. 5 shows voids near grain boundaries. Void coarsening and denuded zones are observed

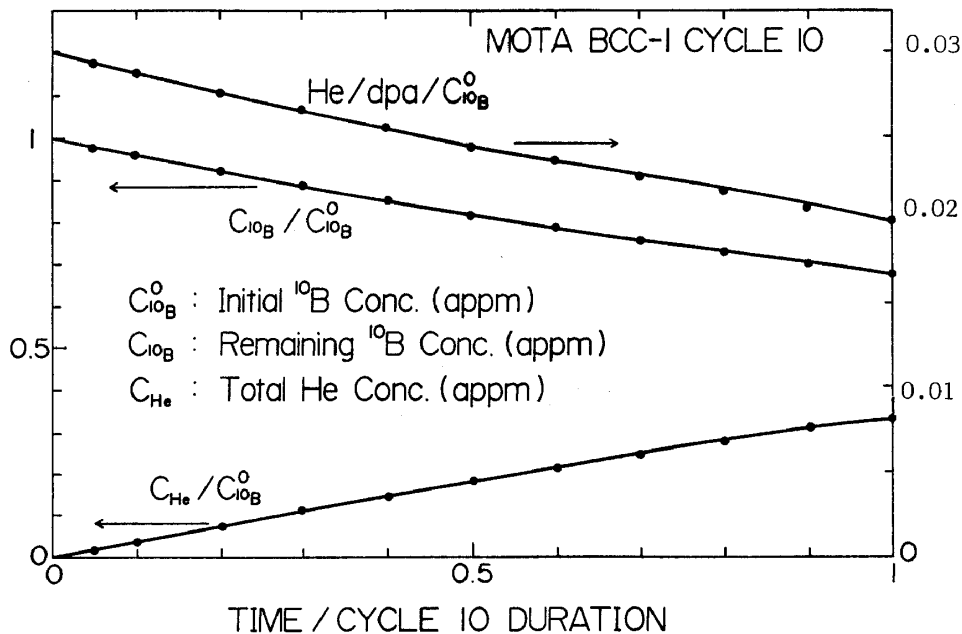


Fig. 1. Time dependence of helium production from ^{10}B at the Below-Core-Canister of FFTF during Cycle 10 operation.

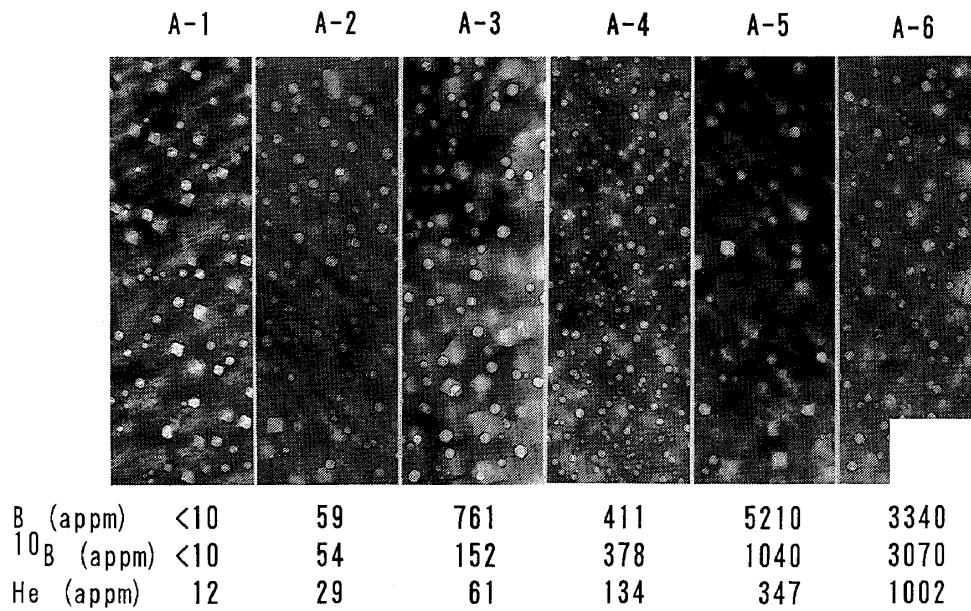


Fig. 2. Voids observed in the matrix. (365°C, 12.2 dpa)

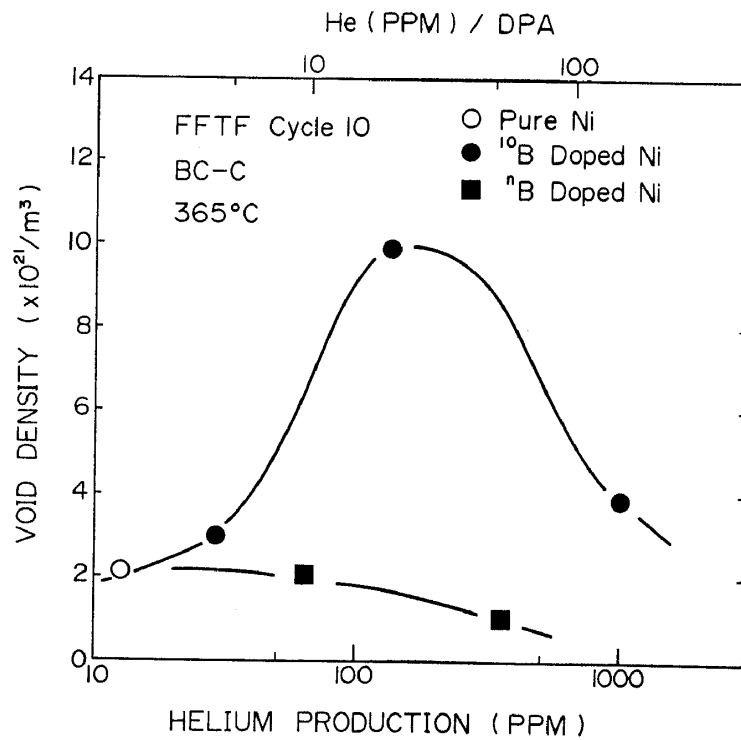


Fig. 3. Void densities as a function of the amount of helium production. (365°C, 12.2 dpa)

in some cases. However, fine voids as observed near the precipitates were not observed.

Fig. 6 shows an example of dark-field weak-beam image of the matrix. Very low density of Stacking Fault Tetrahedra(SFT) are observed as well as voids and low density of dislocations.

IV. Discussion

We believe that the boron doped in the specimens is distributed in the matrix fairly homogeneously. The reasons why we believe are as follows:

1. Chemical analyses of the unirradiated specimens and helium analyses of the irradiated specimens showed reasonable boron and helium concentrations, respectively.
2. Precipitates were only rarely observed in matrix. The density and size of voids were significantly homogeneous in matrix except the vicinity of the low density of precipitates.
3. Fine voids were not observed near grain boundaries. Thus segregation or precipitation of the boron at grain boundaries are believed to be small.

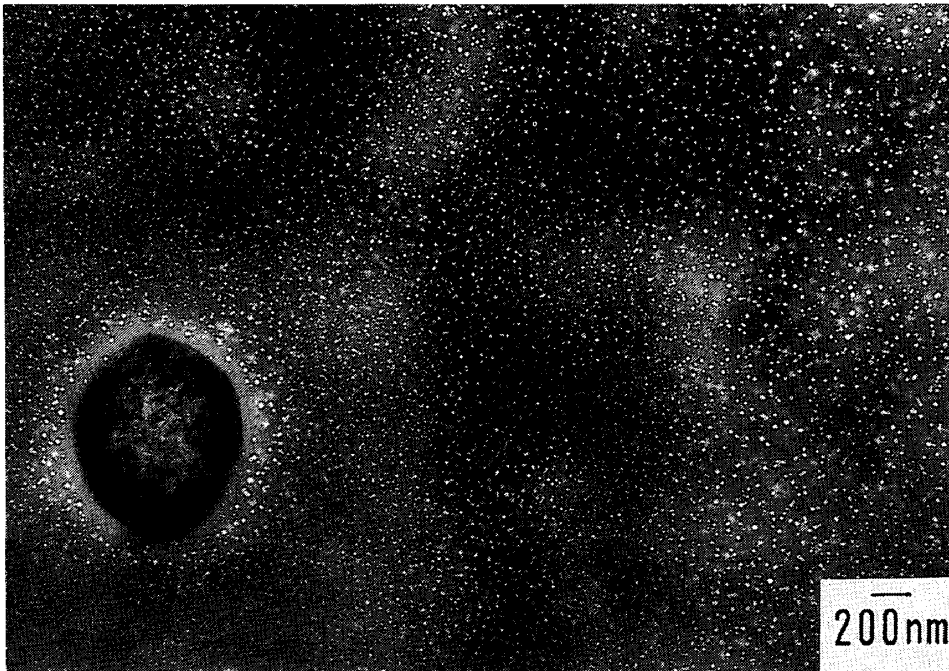


Fig. 4. Voids near a boron precipitate. (365°C, 12.2 dpa)

This means, however, that lithium is also generated homogeneously in matrix. This effect is remaining to be examined in the future.

The fact that the void density increases with increasing the boron content for ^{10}B -doped specimens, shown in Fig. 3, suggests that the transmutation products should enhance the void nucleation. On the other hand, the decrease in the void density after the peak with increasing the ^{10}B addition and the lower void densities in ^{11}B -doped specimens relative to those in ^{10}B -doped specimens suggest that the boron should suppress void nucleation as its chemical effect. It is, therefore, of urgent importance to estimate this effect by charged particle irradiations which do not cause transmutation of boron.

V. Conclusions

The effects of boron on microstructural evolution under neutron irradiations seem to be twofold, namely production of helium and lithium by transmutation promoting void nucleation and chemical effects of boron suppressing void nucleation. The competition of these two effects is likely to result in the void density as a function of ^{10}B or ^{11}B content obtained in the present experiment. Reliable estimation of the helium effects seems to be possible using the boron addition technique by well designed experiments separating some of its side effects. Especially the chemical effects of boron and possible lithium effects must be investigated in the future.

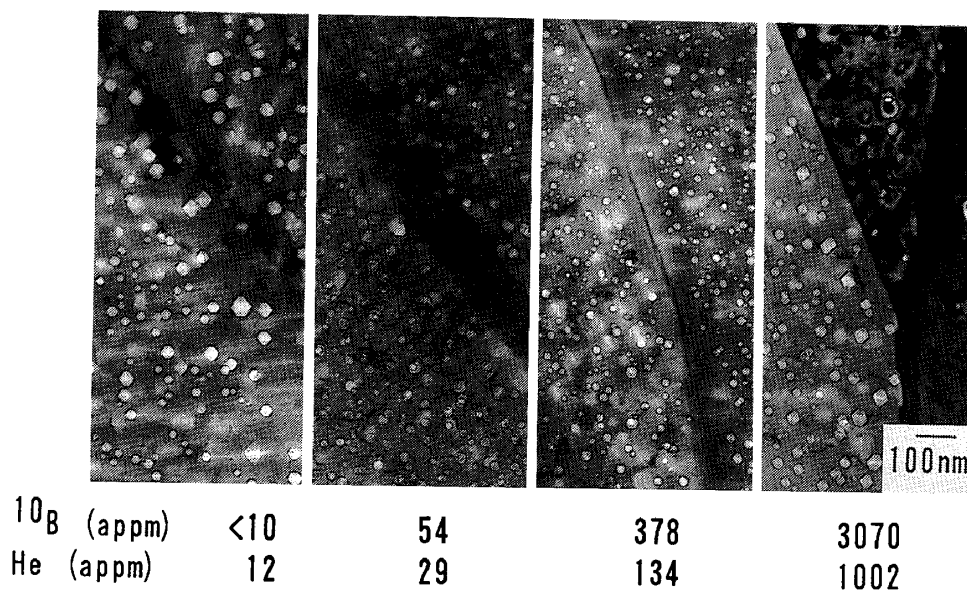


Fig. 5. Voids near grain boundaries. (365°C, 12.2 dpa)

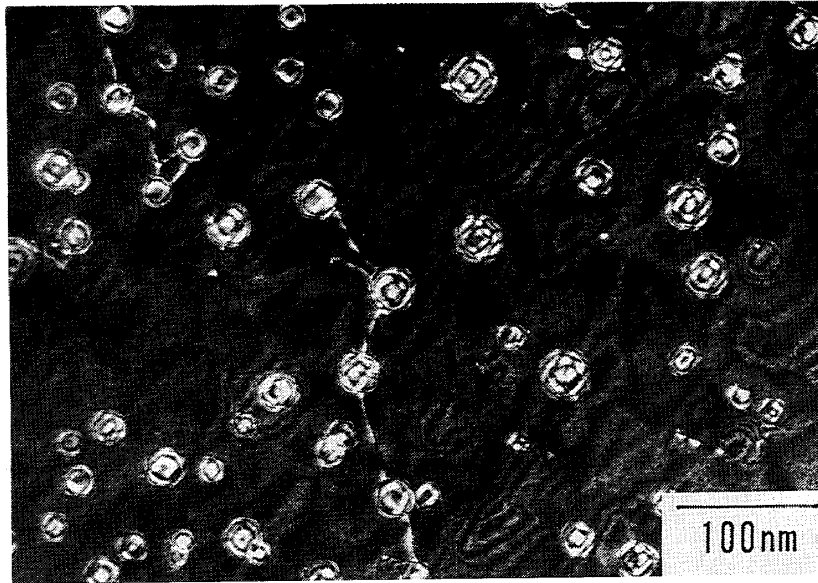


Fig. 6. Dark-field weak-beam image of the matrix.
(365°C, 12.2 dpa)

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