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In-situ EBSD analysis on reverse transformation behavior of Fe-Ni-C alloys

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Abstract: In the reverse transformation of steels, initial structures as well as heating rates and chemical compositions of steels strongly influence formation behaviors of austenite. For example, when the lath martensite is reheated at a heating rate ≦10K/sec, a formation of granular and acicular austenite has been reported. However, the formation mechanism of the acicular austenite has not been fully clarified by using quenched specimens. In this study, we investigated formation behaviors of granular and acicular austenite through detailed microstructural observations by means of the *in-situ* SEM/EBSD techniques in which a sample is reheated in a SEM chamber and observed its surface while heating. Through *in-situ* SEM/EBSD investigations on a 9Ni-0.003C (mass%) steel, it was confirmed that austenite nucleated on grain boundaries in the initial structure. On the other hand, nucleation within ferrite grains could not be confirmed. Compared with the previous studies in which microstructures were indirectly observed after cooling, it could be concluded that the present *in-situ* SEM/EBSD method was certainly effective to observe nucleation and growth processes of reversely transformed austenite during heating.

1. INTRODUCTION

In the reverse transformation of steels, initial structures as well as heating rates and chemical compositions of steels strongly influence formation behaviors of austenite [1-5]. Therefore, the metallurgy of reverse transformation is extremely complicated. It is important to investigate the effect of each factor on formation behaviors of reversely transformed austenite separately. When the lath martensite is reheated at a heating rate ≦10K/sec, a formation of granular and acicular austenite has been reported [6-8]. Table1 summarizes the characteristic of granular and acicular austenite obtained by previous studies. It is reported that each acicular austenite formed within a prior austenite grain has the same crystallographic orientation with the prior austenite grain which transformed to martensite (It is called memory effect.). Three kinds of formation mechanisms for the acicular austenite with the same orientation have been proposed: (1) displacive transformation from lath martensite to austenite [6]; (2) satisfying the orientation relationships among cementite, ferrite and austenite [7]; (3) coarsening of retained austenite between martensite laths [8]. It is also suggested that cementite and retained austenite influence formation behaviours of acicular austenite. However the nucleation and growth mechanism in the reverse transformation has not been identified.

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Table1. Characteristics of granular and acicular austenite

	Characteristics of austenite
Granular austenite	・Nucleated at large-angle grain boundaries. ・Formed by diffusion mechanism
Acicular austenite	・Nucleated at block and lath boundaries. ・Acicular austenite formed within an identical prior austenite grain have the same orientation ・Three types of formation mechanisms are proposed. ① Displacive transformation from lath martensite to austenite. [6] ② Satisfying the orientation relationships among cementite, ferrite and austenite. [7] ③ Coarsening of retained austenite between martensite laths. [8]

Many previous studies were mainly obtained by *ex-situ* observations by using samples water-quenched after reheating at temperatures higher than A_{c1} [1-10]. However, in these quenched samples, austenite which was formed during heat treatment had been transformed to ferrite already. In order to understand the formation process and characteristics of reversely transformed austenite, it is better to directly observe austenite microstructures at high temperatures. Recently, a new microstructural observation techniques called *in-situ* SEM/EBSD has been developed [11,12]. In this technique, a sample is reheated in a SEM chamber and observed by EBSD during heating. By the use of the *in-situ* SEM/EBSD technique, it would become possible to directly observe the formation process of austenite during heat treatments. In this study, we investigated formation behaviors of granular and acicular asutenite through detailed microstructural observations by means of the *in-situ* SEM/EBSD techniques.

2. EXPERIMENTAL PROCEDURE

9Ni-0.003C (mass%) steel was used in this study. Chemical compositions of this steel and transformation temperatures are shown in Table2. The samples cut out from forged ingots were solutionized at 1523K for 72h in an Ar atmosphere. The samples were austenitized at 1373K for 900s and slowly cooled in a furnace, followed by treatment at 723K for 7.2ks to obtain homogenized samples. The initial microstructure was fully ferrite structure and cementite and retained austenite had not been detected by TEM observation and XRD measurement respectively.

Table 2. Chemical compositions in the steel (mass%) and transformation temperatures (K).

C	Si	B*	Ni	Fe	A_{c1}	A_{c3}
0.003	<0.01	9	9.09	Bal.	898	998

In the *in-situ* SEM/EBSD, the samples were heated at a heating rate of 1K/sec (≧673K), and held at temperatures between 863K and 998K to perform *in-situ* SEM/EBSD observations. Then, the temperature was increased again to the next observation temperature. Fig.1 shows such an *in-situ* SEM/EBSD heat pattern. The *in-situ* SEM/EBSD observations were performed in the same position.

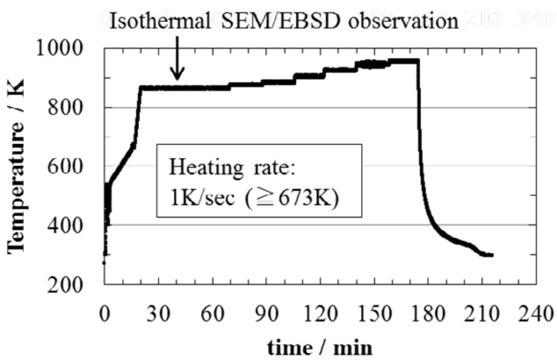

Fig.1. Heat pattern of *in-situ* SEM/EBSD.

3. RESULTS

Fig.2 shows the microstructural evolution during reverse transformation which has been obtained by *in-situ* SEM/EBSD, (a) indicates IPF map of initial structure and (b-d) indicate IPF maps of only austenite phase during heat treatment. Black lines in (a) signify high angle grain boundaries ($\angle\theta \geqq 15°$). Through *in-situ* SEM/EBSD investigations, it was confirmed that austenite nucleated on grain boundaries in the initial structure and specific austenite grains grew up by encroaching other grains. With increasing the temperature, austenite grew into ferrite grain gradually. Austenite orientations were clearly analysed at each temperature. It was identified that austenite grains formed on one grain boundary had various orientations. On the other hand, nucleation within ferrite grains could not be confirmed.

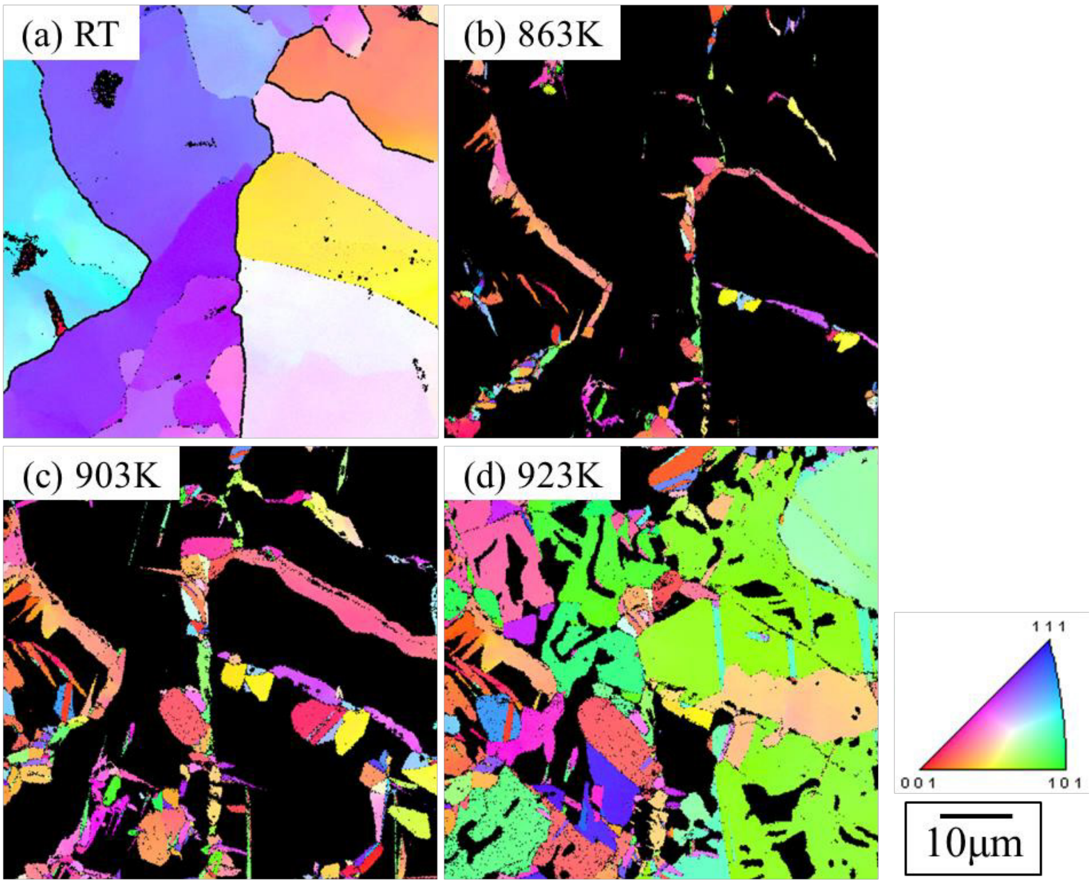

Fig. 2. *In-situ* SEM/EBSD images : (a) IPF map of initial structure (b-d) IPF maps of only austenite phase during heat treatment.

4. SUMMARY

In this study, formation behaviors of granular and acicular austenite were investigated through detailed microstructural observations by means of the *in-situ* SEM/EBSD techniques. Through *in-situ* SEM/EBSD on 9Ni-0.003C (mass%) steel, it was confirmed that austenite nucleated on grain boundaries in the initial structure and specific austenite grains grew up by encroaching other grains. On the other hand, nucleation within ferrite grains could not be confirmed. Compared with the previous studies in which microstructures were indirectly observed after cooling, it could be concluded that the present *in-situ* SEM/EBSD method was certainly effective to observe nucleation and growth processes of reversely transformed austenite during heating.

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