

***In situ* tracking of kinetics for phase transformation during Q&P process below Ms in 0.3C-2Mn-2Si steel**

Wu Gong^{1,2*}, Stefanus Harjo², Ruixiao Zheng³, Yo Tomota⁴, Tomoya Shinozaki⁵, Nobuhiro Tsuji^{1,3}

¹ Elements Strategy Initiative for Structural Materials, Kyoto University, Yoshida-honmachi, Sakyo-ku, Kyoto, 606-8501, Japan

² J-PARC Center, Japan Atomic Energy Agency, 2-4 Shirane Shirakata, Tokai-mura, Naka-gun, Ibaraki, 319-1195, Japan

³ Department of Materials Science and Engineering, Kyoto University, Yoshida-honmachi, Sakyo-ku, Kyoto, 606-8501, Japan

⁴ Research Center for Structure Materials, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki, 305-0047, Japan.

⁵ Steel Casting and Forging Division, Kobe Steel, Ltd., 2-3-1 Shinhama, Arai, Takasago, Hyogo, 676-8670, Japan

Abstract: Phase transformation behavior is essential in quenching and partitioning (Q&P) process for controlling the final microstructure with multi-phase. *In situ* neutron diffraction experiments were performed to examine phase transformation behavior in Q&P process in a 0.3C-2Mn-2Si steel. Carbon partition to austenite was observed. Quantitative phase fractions determined by neutron diffraction show that isothermal transformation occurs below Ms temperature. Microstructure observation reveals that the isothermally transformed product has the nature of bainite.

1. INTRODUCTION

Retained austenite (RA) plays a crucial role in mechanical properties of advanced high strength (AHSS) steels, especially for the ductility through transformation-induced plasticity (TRIP). Numerous researches have been carried out to study the contribution of RA to mechanical properties, in which the amount (i.e. volume fraction) and stability of RA were reported as the main factors [1, 2]. Quenching and partitioning (Q&P) process is an effective approach to control the amount and stability of RA [3,4]. Carbon partitioning from martensite formed in quenching stage to austenite is the fundamental theory in Q&P process. However, isothermal transformation may simultaneously occur, even below the Ms temperature [5,6]. Besides the RA, the matrix microstructure also plays an important role in ductility [7]. Therefore, evaluating the kinetics of isothermal transformation below Ms temperature and clarifying the isothermally transformed product are required for fully understanding the phenomena in Q&P process, which are essential for improving mechanical properties.

X-ray diffraction and the conventional microstructure observation methods such as SEM/EBSD and TEM have been developed to evaluate the phase transformation behavior at high temperature. However, because of the limited penetration, electron and X-ray cannot avoid the affect of the surface effect at high temperature such as oxidation and decarburization. Neutron diffraction with higher penetrability can probe a large volume of materials, which is more suitable to track the bulk-averaged information of the kinetic of phase transformation and element partition in real time. Recently, a thermomechanical processing simulator with broad capabilities has been developed on the engineering neutron diffractometer 'TAKUMI' in J-PARC. The intensity of the incident beam at MLF also increases gradually. It has become possible to follow the phase transformation associated with lattice parameter changes within several seconds at elevated temperature.

In the present study, *in situ* neutron diffraction was applied during Q&P process to quantitatively evaluate phase transformation behavior in a 0.3C-2Mn-2Si steel. Microstructure observation was performed for identification of transformed product.

* Corresponding author. E-mail: gong.wu.3x@kyoto-u.ac.jp, telephone: +81 29 284 3268.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

Chemical composition of the steel used in this study was Fe-0.30C-2Mn-2Si (wt%), of which the Ms temperature was 315°C. *In situ* neutron diffraction experiments were conducted on the beam line 19 ‘TAKUMI’ at J-PARC [8], with a thermomechanical processing simulator installed on it. The experimental setup is shown in Fig.1, a cylindrical specimen with length of 11 mm and diameter of 6.6 mm was set between anvils and on the center of induction coil. Incident neutron passed through the center of specimen along its radial direction. Two detector banks with 5 mm-width radial collimators positioned at +90° and -90° relative to the incident beam were used to collect the neutron diffraction profiles. The scattering vectors of both banks are parallel to the radial direction of the specimen. The specimen was fully austenitized at 900°C for 180 s, followed by rapid cooling to 280°C and held for 5400 s at the temperatures till cooling to ambient temperature finally. Neutron diffraction profiles were analyzed by Z-Rietveld [9] to obtain the phase fraction for evaluation of phase transformations kinetics. Microstructure characterization was carried out by SEM observation.

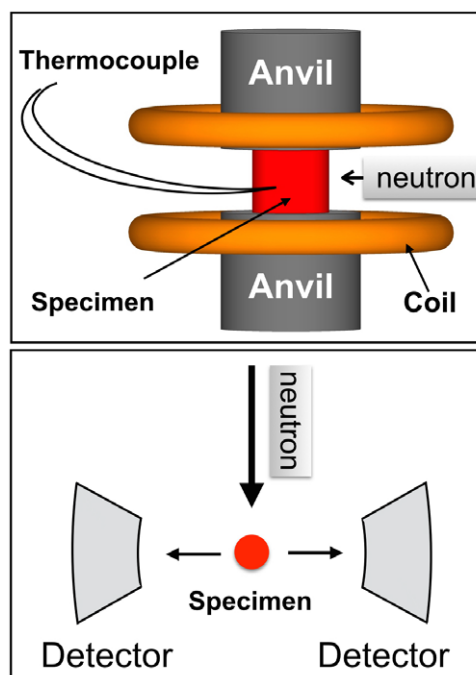


Fig. 1 Setup of *in situ* neutron diffraction experiment in Beamline 19 ‘TAKUMI’

3. RESULTS AND DISCUSSION

3.1. *In situ* tracking of phase transformation behavior below Ms

Fig. 2 shows the evolution of diffraction profiles during austempering process. The reverse phase transformation and isothermal transformation can be observed roughly in Fig. 2a. After austenitization at 900°C, the microstructure is full austenite (see profile ① in Fig.2b). Austenite transformed to martensite immediately when the temperature below Ms temperature. The diffraction profile ② was captured at the time when the temperature reached 280°C. It shows that more than half of the austenite was transformed to martensite. Martensite shows typical broad peaks due to the high dislocation density. Meanwhile, an apparent peak broadening was observed in austenite during the martensitic transformation. It attributed to dislocations introduced in austenite through stress relaxation of transformation strains [10]. In the subsequent isothermal holding, the intensity of the 111 FCC peak decreased significantly as the 110 BCC peak intensity increased, which indicated the decomposition of austenite. An apparent shift of austenite peaks to higher *d*-spacing can be observed during the holding process. It attributed to the carbon partition to austenite, which increased lattice parameter of austenite. After holding for 5400 s, a carbon-enriched austenite with high stability was retained after cooled down to ambient temperature.

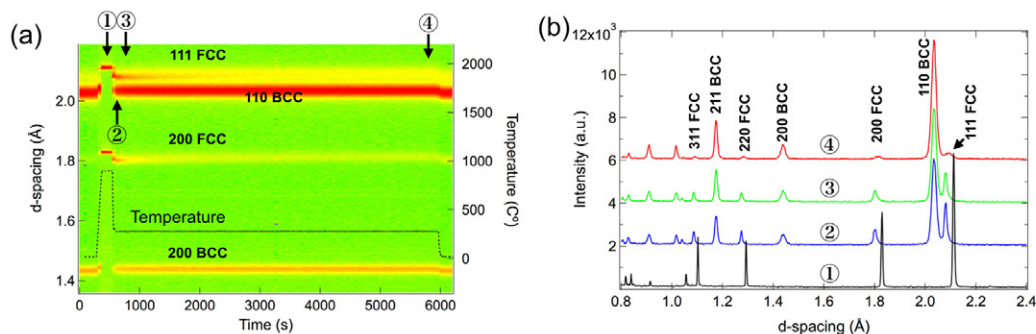


Fig. 2 Neutron diffraction results: (a) evolution of diffraction profiles during austempering at 280°C, and (b) diffraction profiles at ① 900°C; cooling to 280°C for ② 0 s; ③ 60 s and ④ 5400 s as marked in (a).

The quantitative change of the volume fraction of BCC phase is shown in Fig. 3. The detail of cooling stage shows that when the specimen was cooled down to the Ms temperature, the volume fraction of martensite increases accompanied by the slope of temperature curve changes significantly. Because the martensite transformation is exothermic reaction, thereby slowed down the cooling rate. 61.6% austenite transformed to martensite during cooling down to 280°C. In the subsequent holding process, the transformation rate slowed down, approximately 31.2% austenite was isothermally transformed in 5400 s, resulting in 7.2% RA after final cooling to ambient temperature.

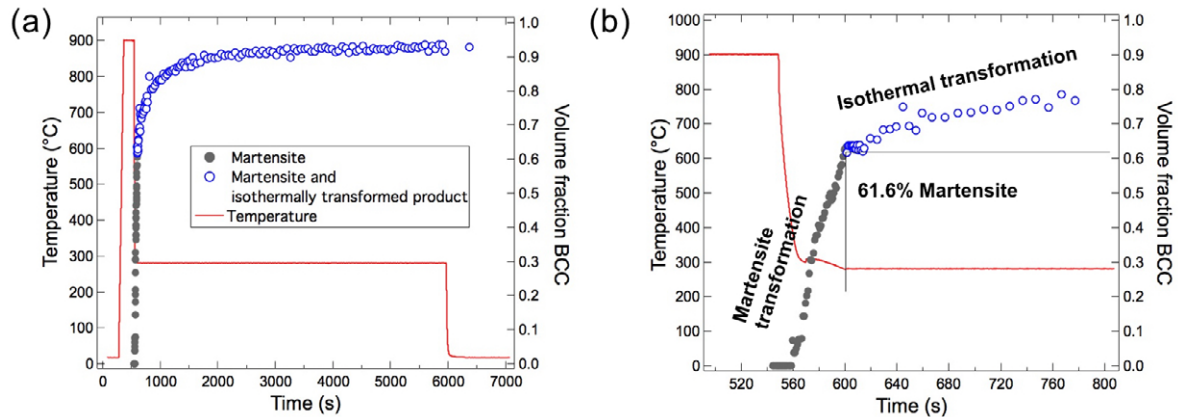


Fig.3 Changes of volume fraction of BCC phase: (a) during the whole process and (b) details of cooling stage. Thermal history was added as reference.

3.2. Identification of isothermally transformed product

There are two possibilities for isothermal transformation below Ms temperature; one is isothermal martensite transformation and the other is bainite transformation. Here, it was attempted to identify which transformation took place in the present case by microstructure observations of the transformed product.

To distinguish a tempered martensite structure from a bainite structure, two reference samples were prepared. Sample A was quenched from 900°C to the ambient temperature in order to obtain nearly full martensite structure, followed by tempering at 280°C for 5400 s. On the other hand, sample B was held at 330°C for 5400s to have a fully bainite structure. SEM microstructures of samples A and B exhibited the characteristic differences. That is, the lath width was smaller in sample B and carbide precipitation was found inside the lath only in sample A. The carbon enriched thin film austenite has been observed in bainite for Si bearing steels [10], carbide would precipitate along lath boundaries in bainite of sample B if tempered for a long time. Hence, “tempered martensite” and “(tempered) bainite” must be distinguished by the lath size and precipitation sites of carbide.

Fig. 4(a) shows SEM microstructure of sample A. i.e., tempered martensite. As seen tiny precipitated particles are dispersed within a relatively coarse lath martensite. On the other hand, as shown in Fig. 4(b), the microstructure of the sample austempered below Ms temperature is found to consist of characteristic two regions. The aspect of most area is similar to Fig. 4(a) suggesting tempered martensite. However, the area indicated as “bainite” looks differently and is similar to

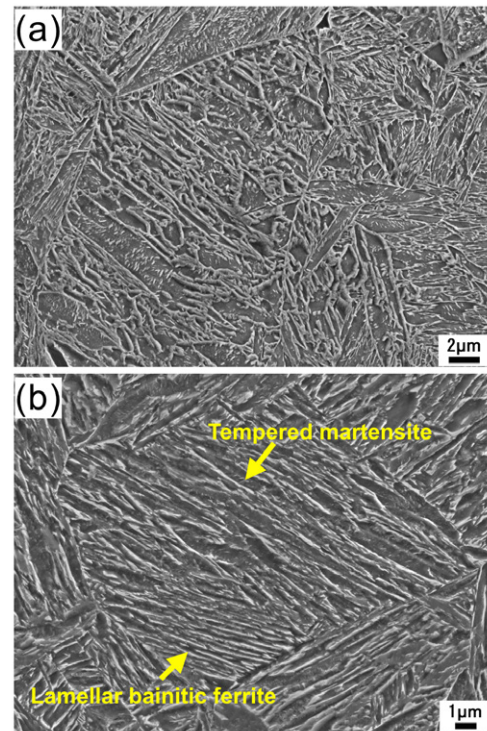


Fig.4 SEM microstructures obtained by (a) quenching to ambient temperature & tempering at 280°C for 5400 s, and (b) austempering at 280°C for 5400 s.

the microstructure of sample B. Therefore, the present isothermal transformation below Ms is very likely to bainite transformation, not isothermal martensite transformation. Some researchers have concluded similarly [5, 6].

4. SUMMARY

In the present study, the isothermal transformation behavior below Ms temperature was investigated by *in situ* neutron diffraction combined with microstructure observation in a 0.3C-2Mn-2Si steel. Quantitative phase fraction evolution was evaluated from neutron diffraction profiles. Austenite firstly transformed to martensite in the cooling stage when the temperature below Ms temperature. Isothermal transformation occurred in the subsequent holding process accompanied by carbon partition. The microstructure observation confirmed that the isothermally transformed product in the present steel has the nature of bainite.

Acknowledgements: This work was supported by the Elements Strategy Initiative for Structural Materials (ESISM) from the Ministry of Education, Science, Sport and Culture, Japan. The neutron diffraction experiments at the Materials and Life Science Experimental Facility of the J-PARC was performed under a user proposal of 2016B0252. The authors sincerely thank Dr. T. Kawasaki, T. Iwahashi and T. Harada from JAEA for their help in neutron diffraction experiments.

REFERENCES

- [1] Sugimoto, K.I., Usui, N., Kobayashi, M., Hashimoto, S.I.: ISIJ international, 32(1992), 1311-1318.
- [2] Tomota, Y., Tokuda, H., Adachi, Y., Wakita, M., Minakawa, N., Moriai, A., Morii, Y.: Acta Materialia, 52(2004), 5737-5745.
- [3] Speer, J.G., Assunção, F.C.R., Matlock, D.K., Edmonds, D.V.: Materials Research, 8(2005), 417-423.
- [4] Edmonds, D.V., He, K., Rizzo, F.C., De Cooman, B.C., Matlock, D.K., Speer, J.G.: Materials Science and Engineering: A, 438(2006), 25-34.
- [5] Van Bohemen, S.M.C., Santofimia, M.J., Sietsma, J.: Scripta Materialia, 58(2008), 488-491.
- [6] Samanta, S., Biswas, P., Giri, S., Singh, S.B., Kundu, S.: Acta Materialia, 105(2016), 390-403.
- [7] Caballero, F.G., García-Mateo, C., Chao, J., Santofimia, M.J., Capdevila, C., De Andres, C.G.: ISIJ international, 48(2008), 1256-1262.
- [8] Harjo, S., Ito, T., Aizawa, K., Arima, H., Abe, J., Moriai, A., Iwahashi, T., Kamiyama, T.: Materials Science Forum, 681(2011) 443-448.
- [9] Oishi-Tomiyasu, R., Yonemura, M., Morishima, T., Hoshikawa, A., Torii, S., Ishigaki, T., Kamiyama, T.: Journal of Applied Crystallography, 45(2012), 299-308.
- [10] Gong, W., Tomota, Y., Harjo, S., Su, Y.H., Aizawa, K.: Acta Materialia, 85(2015), 243-249.