Microstructure Control of a Medium Manganese Steel by Combined Interrupted Quenching and Intercritical Annealing

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Fe-5Mn-0.1C alloy was subjected to interrupted quenching to a temperature between Ms and Mf followed by intercritical annealing in the ferrite and austenite dual-phase region. As a result, a core-shell type second phase, which consisted of a fresh martensite core surrounded by a film-like retained austenite shell, was formed. Characteristics of the microstructure and mechanical properties of the medium Mn steel with core-shell type second phase will be introduced.

Keywords: *high strength steel, medium manganese steel, interrupted quenching, intercritical annealing, core-shell type second phase, retained austenite, TRIP effect, work hardening, element partitioning, mechanical property*

1. Introduction

Medium-Mn steels, which are expected as the next-generation high-strength steel sheets for automobiles, contain retained austenite stabilized by Mn and C in the microstructure [1-10]. The retained austenite leads to an excellent strength-elongation balance of this type of steels due to the TRIP effect. However, since sufficient strength cannot be obtained simply by quenching and tempering (intercritical annealing), cold or warm working is usually performed before the intercritical annealing to increase yield strength by grain refinement. However, it would be difficult to perform cold or warm working of hard martensite in the actual process. In contrast, the interrupted quenching and intercritical annealing (IQ-IA) process proposed in this study [1,2] aims to simultaneously increase the strength and elongation of medium-Mn steels by heat treatment alone, without using working. Specifically, Fe-5Mn-0.1C alloy was subjected to interrupted quenching to a temperature between Ms and Mf followed by intercritical annealing in the ferrite and austenite dual-phase region at around 923 K. As a result, a core-shell type second phase, which consisted of a fresh martensite core surrounded by a film-like retained austenite shell, was formed. The resulting steel is strengthened by the fresh martensite core and its ductility is improved by the TRIP effect of the film-like retained austenite shell. Characteristics of the microstructure and mechanical properties of the medium Mn steel with core-shell type second phase will be introduced.

2. Experimental procedures

The chemical composition of the steel used in this study is shown in **Table 1**. The specimens were solution-treated at 1173 K in the austenite single-phase region for 1.8 ks, followed by interrupted quenching (IQ) in a salt bath held at different temperatures between M_s and M_f at 461 K, 518 K, and 546 K. Then, the specimens were kept there for 60 s so that each volume fraction of untransformed austenite could become approximately 10, 20, and 30 vol%, respectively. After that, the specimens were moved to another salt bath held at 923 K for intercritical annealing (IA) and kept there for various times up to 100 ks, followed by water quenching to ambient temperature. The above heat treatments are schematically shown in **Fig. 1**. In addition to the above IQ-IA process, full quenching (1173 K) and intercritical annealing (FQ-IA) process was also performed to the same steel for comparison.

Changes in the phase transformation ratio during IA at 923 K were estimated by dilatation tests using a dilatometer with rod-like specimens (φ3×10 mm). The heat pattern for the dilatation tests was simulated similarly to the heat treatments of the specimens shown above. Following the final quenching, the volume fraction of the retained austenite was estimated by saturation magnetization measurements at ambient temperature. The microstructure was analyzed with an optical microscope, as well as a transmission electron microscope (TEM). The partitioning of Mn was evaluated via energy-dispersive X-ray spectroscopy (EDS) analysis using scanning transmission electron microscopy (STEM). Tensile tests were conducted on test pieces, with gauge dimensions of 6 mm × 3 mm × 1 mm, using an Instron testing machine at an initial strain rate of 5.56×10^{-4}/s.

Table 1 Chemical composition of the steel used in this study. (mass%)

C	Si	Mn	P	S	Al	N	O	Fe
0.10	1.19	5.0	0.003	0.002	0.017	0.0013	0.0010	Bal.

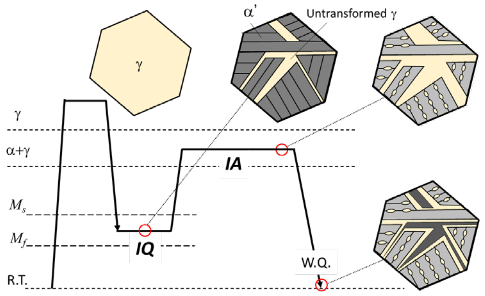

Fig. 1 Schematic diagram showing the IQ-IA process and the resulting microstructure.

3. Results and discussion

As an example of the microstructure obtained by the IQ-IA process, a TEM bright-field image and its illustration of a specimen subjected to IA at 923 K-5.4 ks are shown in **Fig. 2** 1). The fresh martensite grains with high dislocation density are distributed in a tempered martensite matrix with low dislocation density, surrounded by film-like retained austenite observed in dark contrast. In other words, "core-shell type second phase" is formed with martensite as the core and retained austenite as the shell. The analysis of Mn distribution by STEM-EDS shown in **Fig. 3** 1) indicates that Mn is concentrated only in the film-like retained austenite of the shell section, where the Mn concentration is increased to around 10%. Such Mn-enriched austenite would be stable to room temperature. On the other hand, the Mn concentration in the fresh martensite of the core section is maintained at almost the initial concentration of 5%. In the tempered martensite, the Mn concentration tends to decrease slightly. Although the distribution of C concentration is not shown here, all the added C should be concentrated in the core-shell type second phase, so its internal C concentration is estimated to reach 0.22%, hence, the fresh martensite should have a high hardness value of more than 500 HV.

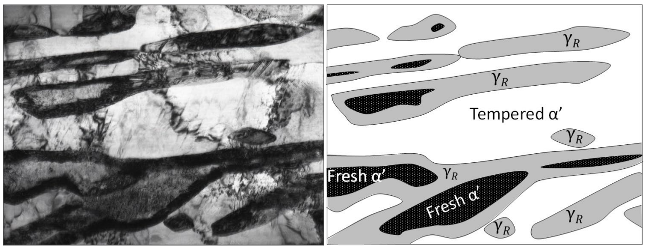

Fig. 2 Transmission electron microscopy (TEM) bright field images and its trace of image of interrupt-quenched (IQ) and intercritically annealed Fe-5%Mn-0.1%C alloy. 1)

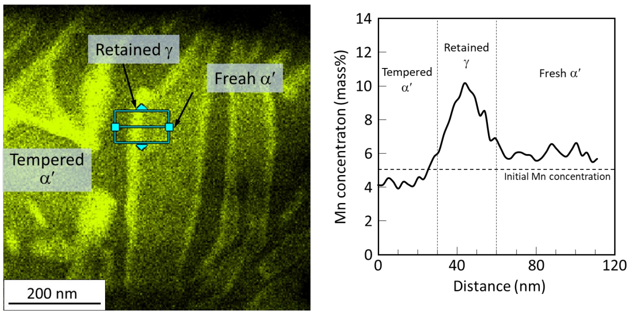

Fig. 3 EDS element map showing Mn distribution for the IQ specimen. The line analysis result on the right shows Mn profile along the line in the left figure. 1)

The formation mechanism of the core-shell type second phase is schematically shown in **Fig. 4**. When the untransformed austenite partially remaining after IQ is heated to the (α+γ) two-phase region, α'→γ reverse transformation proceeds due to migration of the interface. During this process, Mn, an austenite stabilizing element, is supplied from the matrix martensite and becomes enriched. However, the diffusion rate of Mn in austenite is so slow at this temperature that it cannot diffuse into the untransformed austenite and remains there. When the specimen is quenched at room temperature from such a state, the outer periphery with enriched Mn becomes retained austenite and constitutes a shell section, while the interior with low Mn concentration undergoes martensitic transformation to form a fresh martensitic core.

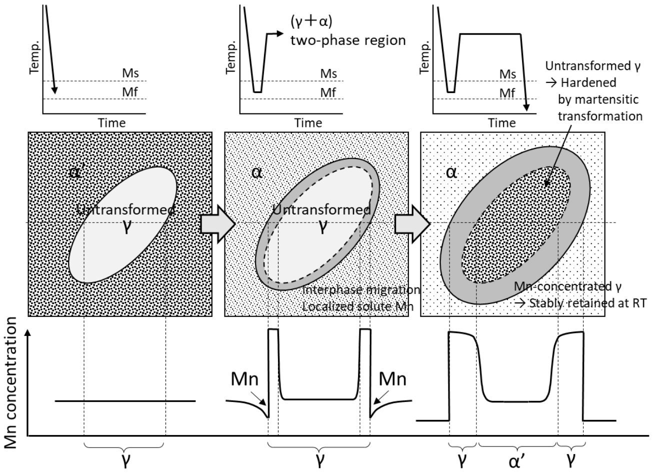

Fig. 4 Schematic illustration showing the mechanism of core-shell type second phase formation through IQ and IA process in Fe-5%Mn-0.1%C alloy.

Another feature of IQ-IA process is that the volume fraction of the constituent microstructures can be adjusted by changing the IQ temperature. **Fig. 5** shows the volume fraction of fresh martensite and retained austenite obtained by changing the IQ temperature in three steps (546 K, 518 K, and 461 K) as a function of the time of IA (923 K). The volume fractions of the untransformed austenite of these IQed specimens are controlled to be 30 vol%, 20 vol%, and 10 vol%, respectively. As the IA time increases, the amount of austenite at the IA temperature increases, but its stability decreases due to dilution of carbon in the austenite. As a result, the longer the IA time, the more fresh martensite and the less retained austenite in the microstructure obtained when quenched to room temperature.

Fig. 6 2) shows stress-strain curves for samples treated with IQ at 546 K, 518 K, and 461 K, respectively, followed by IA at 923 K. For comparison, results are also shown for specimens that were fully quenched and then IAed (FQ-IA), as well as for a conventional DP steel (Fe-0.15C-1.0Mn alloy) 11). All three IQ-IA processed specimens have higher strength than the FQ-IA processed specimens, and they are not only stronger but also more ductile than the DP steel. This excellent strength-ductility balance is attributed to the simultaneous effects of increased work hardening due to fresh martensite and the TRIP effect due to retained austenite. The difference in mechanical properties among the three IQ-IA processed specimens appears to be strongly dependent on the volume fractions of fresh martensite and retained austenite: The higher the IQ temperature, the more increased volume fraction of fresh martensite and the lower amount of retained austenite, which has resulted in the higher strength and the reduced elongation. As described above, one of the characteristics of the IQ-IA process is that the mechanical properties of the same steel grade can be varied depending on the IQ temperature.

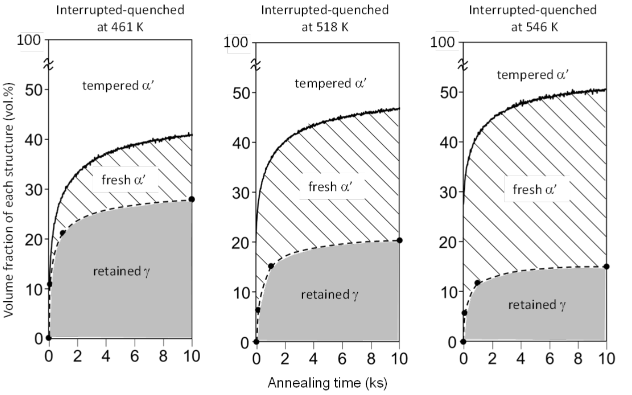

Fig. 5 Change in volume fraction of fresh martensite and retained austenite measured after IQ-IA process as a function of IA treatment time.[2)]

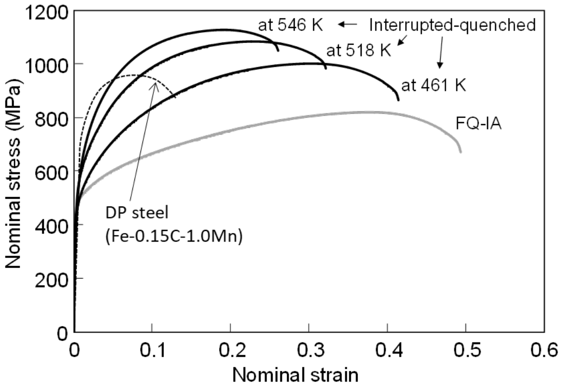

Fig. 6 Nominal stress-strain curves for samples treated with IQ at 546 K, 518 K, and 461 K, respectively, followed by IA at 923 K. For comparison, results are also shown for specimens that were fully quenched and then IAed (FQ-IA), as well as for a conventional DP steel (Fe-0.15C-1.0Mn alloy)[11)].

4. Conclusion

Fe-5%Mn-0.1%C alloy forms a microstructure characterized by the core-shell type second phase within tempered martensite matrix, through the heat treatment of interrupted quenching to a temperature between Ms and Mf followed by intercritical annealing at around 923 K (IQ-IA process). As a result, high strength steel with excellent strength-ductility balance is obtained owing to the increased work hardening rate due to fresh martensite core and the TRIP effect by retained austenite shell. Another feature of the IQ-IA process is that the mechanical properties can be adjusted by changing the IQ temperature. Although the difficulty of temperature control remains a problem in practical use, it is noteworthy as one heat treatment that achieves both strength and elongation in medium Mn steels without pre-working of martensite.

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