ROLE OF AUSTENITE IN WELD TOUGHNESS OF SUPER DUPLEX STAINLESS STEEL

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ABSTRACT

Microstructure control for welding super duplex stainless steel SAF2507 was carried out on a welded joint by GTA welding. The toughness of the bond region in the heat-affected zone (HAZ) of the advanced SAF2507 stainless steel was much lower than that of the base metal. The microstructure of the bond region for the as-welded sample was examined and the extreme grain growth of ferrite and the lowering of the amount of austenite phase were observed. In order to improve the toughness of the bond region, microstructure control was carried out using a cooling rate control process during welding. Various cooling times from 1 673 K to 1 073 K in the bond region were selected, which corresponded to the heat input from 1 kJ/mm to 6 kJ/mm. For the ferrite grain growth, the cooling time from 1 673 K to 1 473 K, that is, Δt_{16-14} was controlled using a Gleeble simulator. The ferrite grain size increased with increasing cooling time Δt_{16-14}. For austenite phase reformation, the cooling time from 1 473 K to 1 073 K, Δt_{14-10} was selected, since austenite phase reformation occurs within that temperature range. The amount of austenite increased with increasing Δt_{14-10}. Increasing the cooling rate caused both ferrite grain growth and an increase of the austenite phase. Improvement of the toughness was accomplished up to 60 s in the cooling time from 1 473 K to 1 073 K, however hardly any change in toughness was accomplished at the cooling time of 120 s, because the slow cooling rate caused both ferrite grain growth and an increase of the austenite phase.

IIW-Thesaurus keywords: *Austenite; Duplex stainless steels; Stainless steels; Steels; Toughness; Mechanical properties; Grain boundaries; Grain size; Heat affected zone; Weld zone; Microstructure; Thermal cycling.*

1 INTRODUCTION

Super duplex stainless steels of Fe-Ni-Cr alloys consist of austenitic-ferritic microstructures at room temperature and successfully combine good toughness, high strength, and high corrosion resistance due to the approximately equal volume fraction of the two phases [1].

Super duplex stainless steels also exhibit greater toughness and better weldability than ferritic stainless steels [1]. They have higher strength and better corrosion resistance than austenitic stainless steels [2]. Their good engineering performance has led to an increasing number of applications. However, it is well known that the impact toughness of duplex stainless steel weldments deteriorates with the increase in volume fraction of δ-ferrite within the Heat-Affected Zone (HAZ) [3, 4]. The bond region especially showed the lowest toughness in the HAZ for SAF2507 [5].

In this study, the volume fraction of austenite in the HAZ of the super duplex stainless steel SAF2507 was controlled by varying the cooling rate from the ferrite single-phase region using a weld simulator. The effectiveness of controlling austenite content with heat input was examined in terms of toughness improvement of the HAZ, and the effect of austenite on the toughness of the bond region in the HAZ has been investigated in terms of morphology of austenite and fracture surfaces.

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2 EXPERIMENTAL

The material used was super duplex stainless steel SAF2507 (25.4 % Cr, 6.7 % Ni, 3.8 % Mo, 0.27 % N, all mass %). Simulated weld thermal cycles were applied using a resistance heat weld simulator (Gleeble 1500). The specimens with the dimension of 11 × 11 × 60 mm were heated to 1 673 K in 10 s, held at the temperature for 5 s, and then cooled with different cooling rates. To determine the cooling rates, the following relation has been applied [6]:

$$\frac{Q}{d} = k \cdot \Delta t_{10-7}^{\;1/2} \qquad (1)$$

where

Q is the net heat input (J/mm),

d is the plate thickness (mm),

k is the thermal coefficient ($J/mm^2\ s^{1/2}$),

Δt_{10-7} is the cooling time from 1 073 to 773 K (s).

In this study, 25.52 $J/mm^2\ s^{1/2}$ for the k value and 16 mm for the plate thickness were used. The welding parameter Δt_{10-7} has some metallurgical foundation for the carbon and low-alloyed steels. For duplex stainless steels, the use of Δt_{14-10} has been suggested (cooling time from 1 473 to 1 073 K) since austenite reformation occurs within this temperature range. A relationship exists between Δt_{14-10} and Δt_{10-7}, giving the following expression [6]:

$$\frac{\Delta t_{10-7}}{\Delta t_{14-10}} = \frac{\dfrac{1}{(773 - T_0)^2} - \dfrac{1}{(1\,073 - T_0)^2}}{\dfrac{1}{(1\,073 - T_0)^2} - \dfrac{1}{(1\,473 - T_0)^2}} \qquad (2)$$

where

T_0 is the starting temperature (K).

Using the equations (1) and (2), six kinds of thermal cycles were created. The thermal cycles used in this study are illustrated in Figure 1, and the parameters for each thermal cycle are listed in Table 1.

After applying the thermal cycles the specimens were machined to Charpy V-notch samples with the dimension of 10 × 10 × 55 mm and tested in the temperature range from 77 K to 273 K.

The fracture surfaces after impact tests were observed by SEM using the technique of stereoscopy. From the obtained stereo images, three-dimensional topography of the fracture surfaces was reconstructed in a monitor using the software developed by the authors [7].

The microstructures of the test specimens were also studied by optical microscope. The samples were ground, polished mechanically, and etched electrolytically in 10 mol/l KOH solution at 3 V for 3 s.

3 RESULTS AND DISCUSSION

3.1 Austenite formation during cooling

Microstructures after applying the thermal cycles are shown in Figure 2. Ferrite is coloured darker and austenite lighter on the micrographs on the electrolytic etching. As shown in Figure 2, the morphology of the austenite is classified into grain boundary type and intragranular type. At the peak temperature of the thermal cycles, the microstructure of the specimen showed a single ferrite phase. Austenite forms during the cooling within the temperature range from 1 473 to 1 073 K. It is considered that grain boundary austenite forms in the early stage of cooling and forms the net structure, and then the formation of intragranular austenite follows. It can be seen that the grain boundary austenite was thickened and the formation of intragranular austenite was promoted by increasing the cooling time.

The austenite content after applying the thermal cycles was measured using optical micrographs. The point

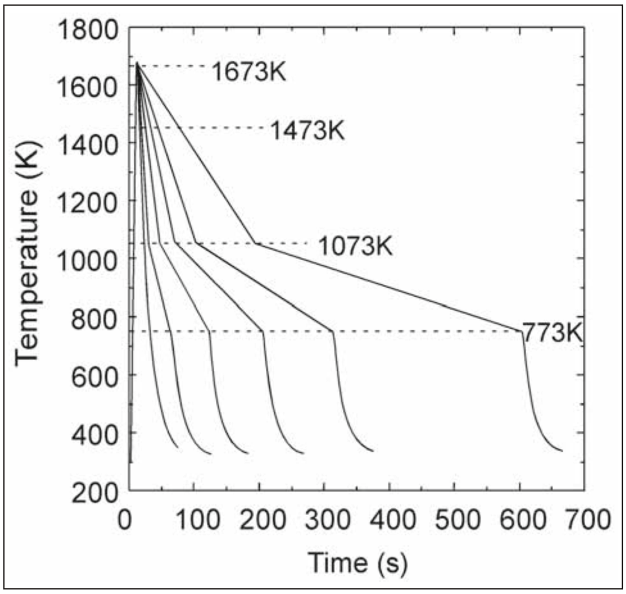


Figure 1 – Simulated thermal cycles applied

counting method (ASTM E562) was used for the measurement. The variation of austenite content as a function of Δt_{14-10} is plotted in Figure 3.

The austenite content increased from 35 % to 67 %, when Δt_{14-10} increased from 6 s to 114 s. It is known that phase transformation of the system containing two or more phases can be described by the following empirical function:

$$\frac{dy}{dt} = k^n \, t^{n-1} (1 - y)^m \qquad (3)$$

where

y is the transformation ratio,

t is time,

k, m and n are constants.

Nakao and Nishimoto *et al.* [8] reported that in duplex stainless steels the transformation of γ phase from α phase under continuous cooling condition is described by the following function, when the equilibrium austenite content is regarded as constant and the cooling cycle can be taken as approximately linear in the temperature range of the transformation:

$$\frac{f}{f_{eq} - f} = \Delta t_{14-10} \cdot k_0 \qquad (4)$$

where

f is the volume fraction of austenite after cooling,

f_{eq} is the equilibrium austenite content,

k_0 is the average value of transformation rate constant.

Assuming that the equilibrium austenite content is 67 %, the austenite formation is well described by using the k_0 value of 0.14. The calculated austenite content is shown in Figure 3 as a solid line.

Table 1 – Parameters for simulated thermal cycles

Heat input (kJ/mm)	1	2	3	4	5	6
Δt_{14-10} (s)	6	9.3	21.3	37.3	58.7	114
Δt_{10-7} (s)	8	33	76	135	211	413

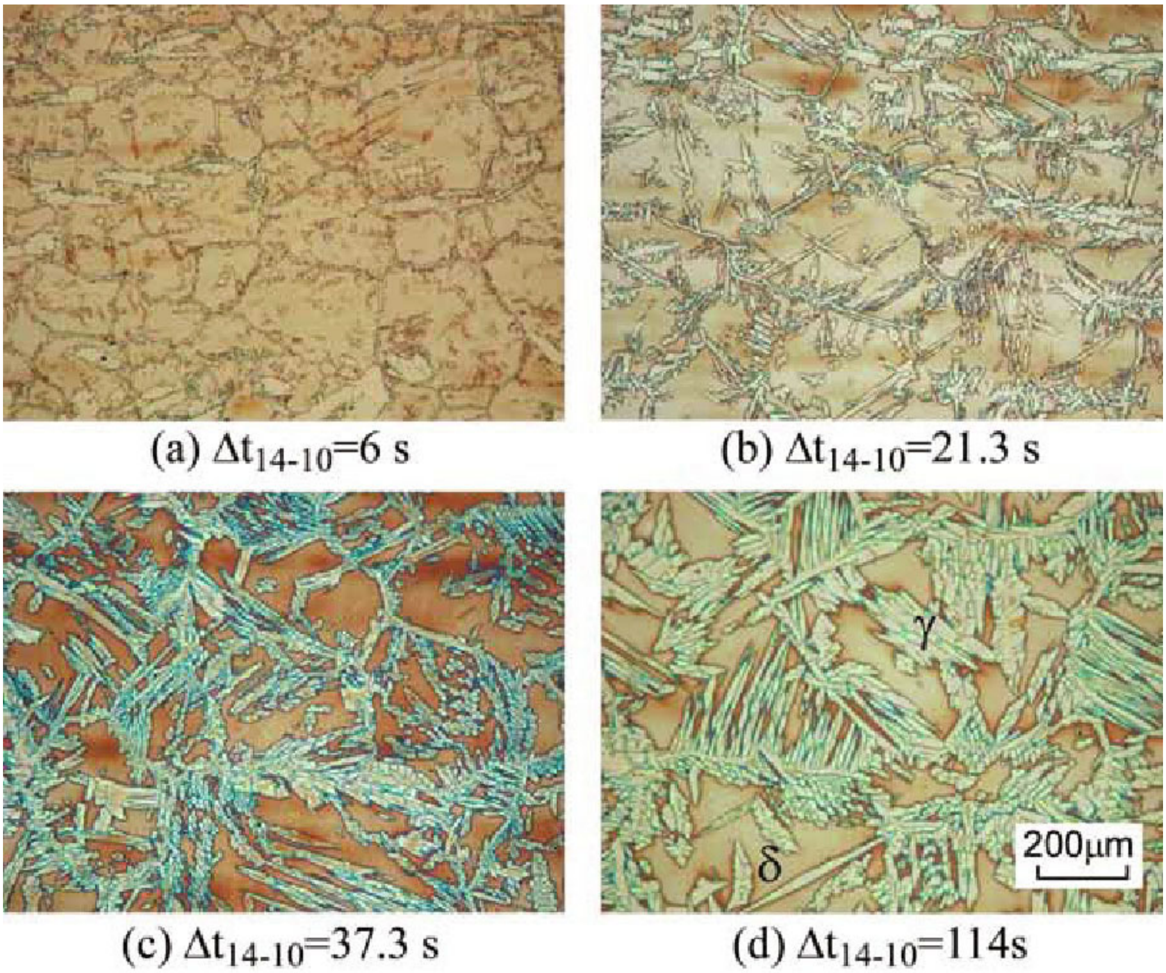


Figure 2 – Microstructures after applying the thermal cycles

3.2 Impact toughness of weld bond region

The variation of the impact energy at 193 K as a function of □$t_{14\text{-}10}$ is shown in Figure 4, and the ductile-brittle transition temperatures (DBTT) for each cooling condition is listed in Table 2. The toughness increased with the □$t_{14\text{-}10}$ when the □$t_{14\text{-}10}$ was below 60 s. However, when □$t_{14\text{-}10}$ exceeded 60 s, the toughness began to decrease.

It is considered to be attributed to the fact that austenite reformation starts to slow down when □$t_{14\text{-}10}$ exceeds 60 s and grain growth of the ferrite matrix increases.

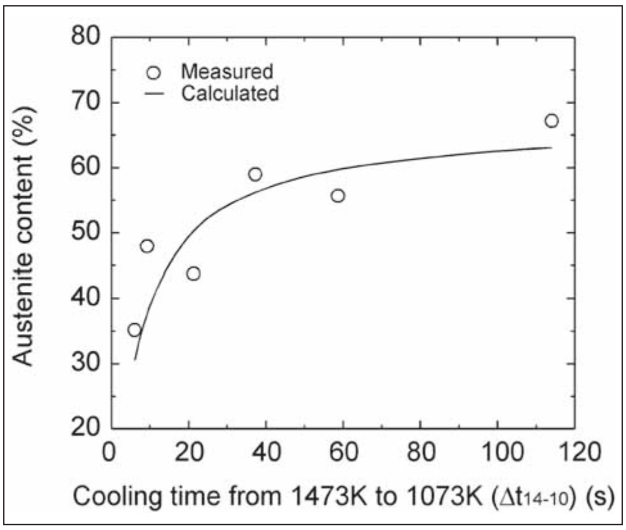


Figure 3 – Variation of the austenite content as a function of the cooling time from 1 473 to 1 073 K (□$t_{14\text{-}10}$)

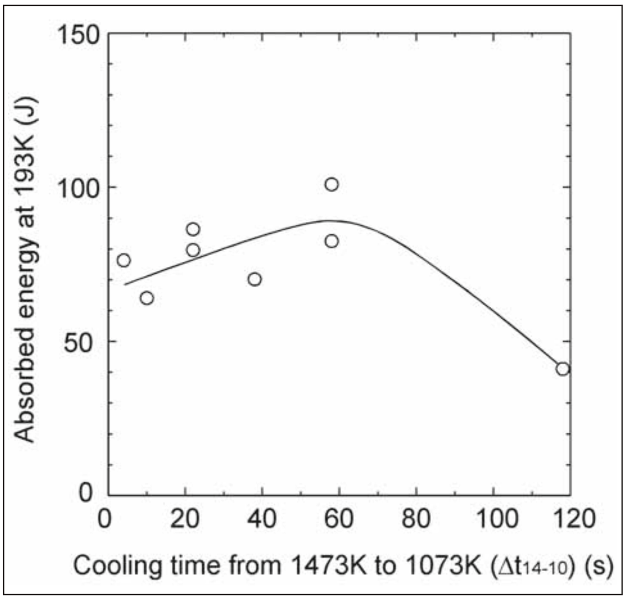


Figure 4 – Variation of the impact energy at 193 K as a function of the cooling time from 1 473 K to 1 073 K (□$t_{14\text{-}10}$)

Table 2 – □t_{14-12} and DBTT of the simulated weld specimens

□t_{14-10} (s)	6	9.3	21.3	37.3	58.7	114
DBTT (K)	219	220	211	212	210	222

3.3 Grain growth of ferrite during cooling

Austenite reformation occurs in the temperature range below 1 473 K; therefore it is considered that grain growth of the ferrite matrix occurs above 1 473 K, since there is no austenite which prevents the migration of ferrite grain boundaries.

Thermal cycles with rapid cooling in the temperature range below 1 473 K were applied to suppress the austenite reformation. The applied thermal cycles are illustrated in Figure 5.

After heating to 1 673 K, the specimens were cooled to 1 473 K in 3, 19, and 57 s which corresponds to the heat input of 1, 4, and 6 J/mm, followed by rapid cooling below 1 473 K.

Microstructures, after applying the thermal cycles, are shown in Figure 6. It can be seen that the formation of austenite is limited to the grain boundary of ferrite and it is apparently observed that the grain size of ferrite increased as the cooling rate decreased.

The grain size of ferrite was measured using the line intercept method (ASTM E112). The variation of average grain size of ferrite as a function of the cooling time from 1 673 to 1 473 K (□t_{16-14}) is plotted in Figure 7. It was found that the average grain size of ferrite increased from 172 to 459 μm as the cooling time increased.

It is known that the grain growth under isothermal conditions is described by the following empirical function [9]:

$$d_f^{1/n} - d_0^{1/n} = k \exp\left(-\frac{Q_{app}}{RT}\right) t \quad (5)$$

where

d_f is the average grain size after time t at temperature T,

d_0 is the initial grain size,

Q_{app} is the activation energy for grain boundary motion,

n is the time exponent,

k is a kinetic constant,

R is the gas constant.

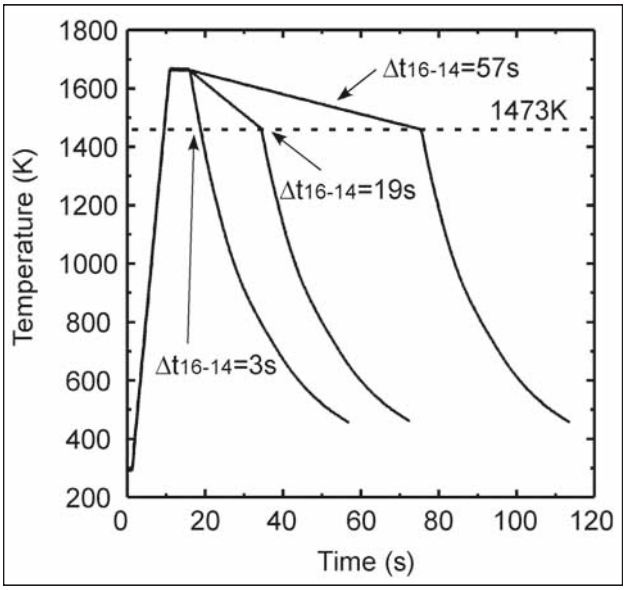


Figure 5 – Thermal cycles with rapid cooling below 1 473 K

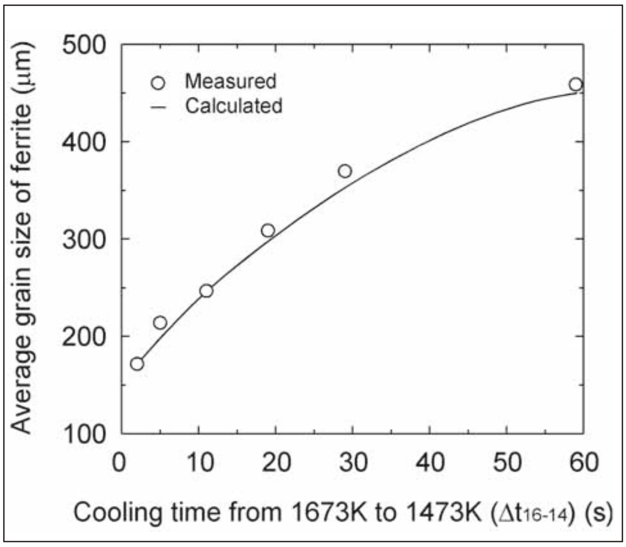
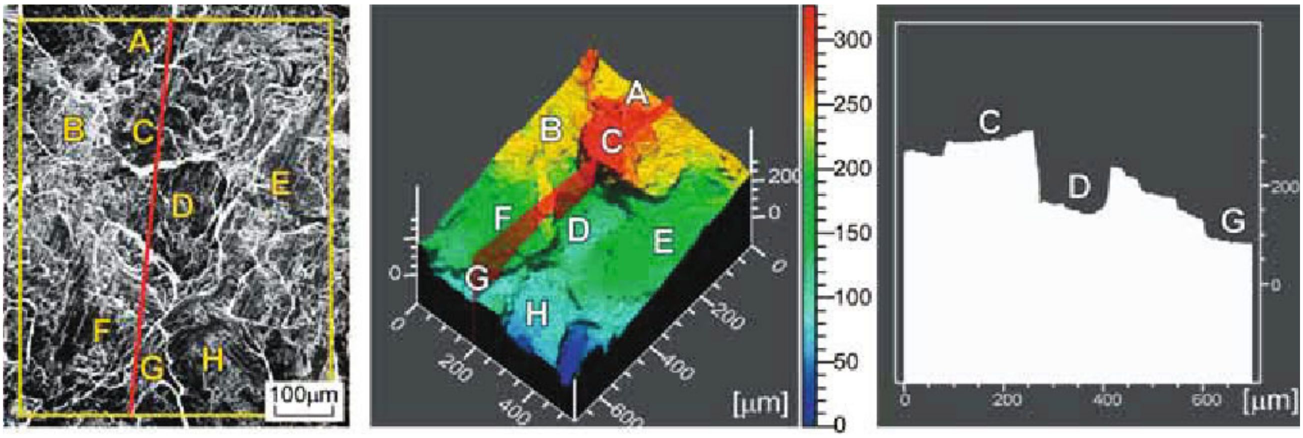


Figure 7 – Variation of the average grain size of ferrite as a function of the cooling time from 1 673 K to 1 473 K (□t_{16-14})

(a) Δt_{16-14}=3 s (b) Δt_{16-14}=19 s (c) Δt_{16-14}=57 s

Figure 6 – Microstructures after applying the thermal cycles with rapid cooling below 1 473 K



(a) SEM micrograph (b) Three-dimensional image (c) Cross sectional profile

Figure 8 – Fractographic observation using three-dimensionally reconstructed topography of the simulated specimen ($\Delta t_{12\text{-}8}$ = 114 s) after Charpy impact tested at 77 K

To calculate the grain growth in the continuous cooling process, the right-hand side of Eq. (5) was integrated over the thermal cycles. By using values of $k = 2.488 \times 10^{15}$, Q_{app} = 318 (kJ/mol) and n = 0.395, a good correlation between calculated and measured values was obtained. The result of the calculation is shown in Figure 7 as a solid line.

3.4 Fracture surface observation

In Figure 8, an SEM micrograph and three-dimensional reconstructed image of the specimen cooled with $\Delta t_{14\text{-}12}$ of 114 s and fractured at 77 K are shown. Several cleavage facets marked as A to H are observed in the SEM micrograph. According to the three-dimensional image, these cleavage facets are separated by steps, the height of which is below 200 μm. Using three-dimensional images, the facet size of cleavage fracture of this specimen was measured. Over 200 facets were measured on the cross sections extracted from three-dimensional images. As a result, the average facet size of this specimen was 108 μm. As mentioned above, the average grain size of ferrite of this specimen was 459 μm; i.e., the fracture of one ferrite grain was separated into four smaller facets on average. Close observation of the three-dimensional image shown in Figure 8 (b) showed groups of adjacent cleavage facets D, E and B, F, G, H have almost the same orientation.

The result suggests that the group of cleavage facets having the same orientation belongs to one ferrite grain. It is considered that intragranular austenite separated the cleavage fracture and formed steps at the facet boundaries. It was found that intragranular austenite played a critical role in lowering the DBTT of the ferrite matrix by partitioning the cleavage fracture in a ferrite grain.

4 CONCLUSIONS

The toughness of the bond region in the heat-affected zone (HAZ) of the advanced super duplex stainless steel SAF2507 was much lower than that of the base metal. In order to improve the toughness of the bond region, microstructure control was carried out using a cooling rate control process during welding.

1) Various cooling times from 1 673 K to 1 073 K were selected, which corresponded to the heat input from 1 kJ/mm to 6 kJ/mm. For the ferrite grain growth, the cooling time from 1 673 K to 1 473 K, that is, $\Delta t_{16\text{-}14}$, was controlled using a Gleeble simulator. The ferrite grain size increased with increasing cooling time $\Delta t_{16\text{-}14}$.

2) For austenite phase reformation, the cooling time from 1 473 K to 1 073 K, $\Delta t_{14\text{-}10}$, was selected, since austenite phase reformation occurs within that temperature range. The amount of austenite increased with increasing $\Delta t_{14\text{-}10}$.

3) The improvement of the toughness was accomplished up to 60 seconds in the cooling time from 1 473 K to 1 073 K, however hardly any improvement in toughness was accomplished at the cooling time of 120 s, because the slow cooling rate caused both ferrite grain growth and an increase of the austenite phase.

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