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Characterization of the Microstructure and Precipitates Formed During the Thermomechanical Processing of a CrNiMoWMnV Ultrahigh-Strength Steel

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Abstract

The effect of total applied strain (TAS) and finish forging temperature (FFT) on the microstructure and precipitation kinetics of a newly developed low-cost, low-alloy CrNiMoWMnV ultrahigh-strength steel has been investigated. A Gleeble 3800 thermomechanical simulator was used to simulate the hot forging process and its influence on the precipitation kinetics. Field emission scanning electron microscopy combined with electron backscattered diffraction was employed to characterize the final overall microstructures while transmission electron microscopy on carbon extraction replicas was used to characterize the precipitates in terms of morphology, size distribution, mean equivalent circular diameter (ECD), the 90th percentile in the cumulative diameter distribution ($D_{90\%ppt}$), chemical composition and crystallography. Thermo-Calc software was used to predict the precipitates expected in austenite at equilibrium. The final microstructure consists of lath martensite and a small fraction of the precipitates AlN, TiN and composite TiN-AlN. Differences in the degree of strain-induced precipitation caused by variations in TAS and FFT have been shown to greatly influence precipitate size distributions. Variations in the degree of precipitate dissolution and coarsening cause variations in the prior austenite grain size, which subsequently cause variations in the effective grain size of the final microstructure, i.e. that defined by high-angle grain boundaries.

1. Introduction

Different microalloying elements such as Al, V, Nb or Ti can be used in order to prevent grain growth and obtain fine-grained microstructures through the precipitation of finely distributed nitrides, carbides or carbonitrides on austenite grain and sub-grain boundaries. The precipitates affect recovery, recrystallization and grain growth during thermomechanical processing i.e. hot forging or rolling^[1], and thereby have a great influence on the flow stress during the process^[2] and the resultant strength and toughness of the product^[3].

Precipitation kinetics is enhanced by thermomechanical processing through an increase in the diffusion of the solute atoms and an increase in the density of nucleation sites, i.e. dislocations and new grain boundaries or sub-grain boundaries^[4]. For example, Mucsi^[5] studied the effect of grain size on the precipitation kinetics of hot-rolled, Al-killed, low-carbon steel and concluded that the refinement of the grain size has a significant effect on the precipitation of nitrides, which probably formed on the grain boundaries and dislocations. Also, Radis et al.^[6] showed that AlN precipitates on grain boundaries in ultralow-carbon steel.

Of the microalloying elements Al, V, Nb and Ti, the nitride of the latter has the highest stability and lowest solubility in austenite. In the case of the commonly encountered concentrations of Ti and N, complete dissolution of TiN is only achieved at very high temperature near to the melting point or even higher^[7].

Al is commonly used as a deoxidizer in steel leaving some Al in solution. Under suitable conditions, this Al is available to form AlN which can hinder grain growth through the pinning effect^[7]. There are two crystallographic structure of AlN precipitates, face-centered cubic (fcc) and hexagonal close-packed (hcp)^[8]. AlN precipitates appear in a wide range of morphologies, i.e. dendritic, prismatic cuboidal, plate, rod or needle depending on the Al and N contents, temperature and the environment in which it is formed^[9]. It can be formed at the grain boundaries during the

solidification or at dislocation loops resulting from deformation. In austenite, AlN often precipitates at grain boundaries as a result of its large misfit with the steel matrix and the relatively high diffusivity of Al at grain boundaries ^[10].

No information is available about the precipitation occurring during the thermomechanical processing of the newly developed CrNiMoWMnV ultrahigh-strength steel that is the topic of this paper. This work has focused on studying the effect of hot forging parameters, i.e. finish forging temperature (FFT) and total applied strain (TAS). Field emission scanning electron microscopy combined with electron backscattered diffraction was employed to characterize the final overall microstructures in terms of phase formed, prior austenite grain size (PAGS), mean effective grain size (EGS) as defined by high-angle grain boundaries, and the 90th percentile in the cumulative grain area distribution (D90%_{EGS}). Transmission electron microscopy on carbon extraction replicas was used to characterize precipitates in terms of morphology, size distribution, mean equivalent circular diameter (ECD), the 90th percentile in the cumulative diameter distribution (D90%_{ppt}), chemical composition and crystallography.

2. Experimental Methods

2.1. Materials and thermomechanical simulation procedure

The steel with the chemical composition given in **Table 1** was melted in an air induction furnace (IF) and refined using electroslag remelting (ESR) in the Steel Technology Department, Central Metallurgical Research and Development Institute (CMRDI), Egypt. The steel ingots were forged in the temperature range 1100-950 °C followed by air cooling at about 0.3 °C/s and further details are given in ^[11].

A Gleeble 3800 thermomechanical simulator was used to simulate the hot forging process. Fifteen cylindrical specimens with a diameter of 5 mm and length of 7.5 mm were cut from the forged bar with their axis parallel to that of the forged bar. **Table 2** shows the five thermomechanical treatments used to examine the influence of FFT combined with the TAS on

precipitation during the hot forging process. In the first thermomechanical treatment, the specimens (1100-0) were austenitized at 1100 °C for 180 s then quenched in water to retain the high temperature microstructure, see **Figure 1a**. In the second thermomechanical treatment, the specimens (1100-0.2) were austenitized at 1100 °C for 180 s before getting the first hit with total strain of 0.2 with strain rate of 1 s^{-1} and held for 20 s before water quenching (WQ). In the third thermomechanical treatment, the specimens (1050-0.4) were heated to 1100 °C at 5 °C/s , held for 180 s before getting the first hit with strain 0.2 and strain rate 1 s^{-1} and held for 20 s followed by cooling at 5 °C/s to 1050 °C and held there for 20s before the second hit with the same parameters to give a TAS 0.4. Then the specimens were held for 20 s before WQ. The same sequence and process parameters have been used in order to get the specimens from the fourth and fifth thermomechanical treatment with TAS 0.6 and 0.8 and FFTs of 1000 °C and 950 °C respectively as shown in Table 2 and Figure 1a.

2.2. Microstructure

Figure 1b illustrates the cutting plane and the section selected for metallographic investigation for all the thermomechanically treated specimens. One specimen from each thermomechanical treatment was mounted in conductive materials for microstructure examination and the other two specimens were mounted in non-conductive materials for replica preparation. All specimens were mechanically ground and polished using $3 \text{ }\mu\text{m}$ and $1 \text{ }\mu\text{m}$ diamond suspension.

The final microstructures of all specimens have been investigated using field emission scanning electron microscopy (FE-SEM, Zeiss Sigma) on samples lightly etched in fresh 2 vol.% nital. Grain boundaries with misorientations higher than 15° were considered to define the effective grain size from a toughness point of view. The effective grain size was defined by their equivalent circle diameter (ECD), which was measured using an EDAX electron backscatter diffraction (EBSD) system on the FESEM with an accelerating voltage 15 kV, a magnification 1500x and a step size of $0.15 \text{ }\mu\text{m}$. The filtering value is 3 pixels which means that the smallest grain size included in the

calculation of the mean effective grain size is 0.22 μm . Using EBSD data, the original PAGs were reconstructed using Matlab software with the aid of the MTEX texture and crystallographic analysis toolbox as described by Javaheri et al ^[12] and Nyssönen et al ^[13,14].

Transmission electron microscopy (TEM) (JEOL JEM-2200FS EFTEM/STEM) at 200 kV was employed to investigate the extracted precipitates on carbon extraction replicas. The carbon extraction replicas were prepared in five steps. Firstly, the samples were lightly etched in fresh 2 vol.% nital, then coated with a 10-20 nm thick carbon film. Secondly, the carbon film was scored using sharp knife into small rectangles about 2mm x 3mm. Thirdly, the replica samples were exposed to 10 % HNO_3 at a potential of 10 V till the replicas lifted off with the extracted precipitates. Fourthly, the extracted replica was washed properly in ethanol, HCl and distilled water. Finally, copper grids were used to pick up the replica from the washing solution and to investigate the precipitates using TEM.

TEM images were analysed using ImageJ software in order to determine the number densities and mean ECD of the precipitates from thirty-two 3.16 x 3.16 μm fields with 319 μm^2 total investigated area. Energy dispersive X-ray spectroscopy (EDS) was employed to determine the chemical compositions and types of the precipitates by analysis of at least 80 precipitates / thermomechanical treatment with various sizes and morphologies from at least three different replicas to get a reliable analysis. Also, the crystallographic structures have been determined using the TEM diffraction pattern.

3. Results and discussion

3.1. Precipitates

3.1.1. Thermodynamic calculations

Thermodynamic calculation was performed using Thermo-Calc software version 2018a together with the database TCFE7. **Figure 2** shows that the equilibrium precipitates in austenite in the temperature range 950 - 1400 $^{\circ}\text{C}$ are AlN and TiN. The volume fraction of the precipitates

increased by decreased temperature while there is no change in the chemical composition of the precipitates over the temperature range used in the calculations (see **Table 3**). The calculated solubility temperatures of AlN and TiN of the investigated steel are 1224 °C and 1396 °C respectively. This indicates that some undissolved precipitates existed in the steel even after holding at 1100 °C, which indeed is what was observed experimentally in the non-strained specimens (1100-0) as described below.

3.1.2. Chemical composition and structure of the precipitates

According to the morphology, EDS analysis and diffraction analysis, the precipitates formed before and during the forging process can be divided into two main types: 1) aluminium nitride AlN, 2) titanium nitride TiN with traces of V, Nb, Mo and W which substitute Ti without a change in the lattice structure (see **Table 4** and **Figure 3**). In some cases, AlN appears to have nucleated and grown on TiN to form composite precipitates denoted in the following as TiN-AlN (see **Figures 3** and **4**). In the composite precipitates, the AlN nucleates on pre-existing TiN precipitates to form a shell or cap.

It should be noted that in the EDS spectra the peaks of Cu and C are partly or completely due to the copper grid and the carbon replica. So, those are neglected in addition to the EDS peaks resulting from the preparation process such as O, S and Fe ^[15].

In our previous publication ^[16], we predicted that different carbides formed in this type of steel from the solid phase during the cooling of the ingots from the IF and ESR and these carbides dissolved completely during the homogenization at 1100 °C prior to the forging process as austenite is the only stable phase at 1100 °C. So, the observed carbides in the investigated steel are probably autotempered martensite carbides that form immediately after the martensite forms during air cooling. But in ^[16], we did not talk about the nitride precipitates that can form at high temperatures and during the thermomechanical process. The current experimental results proved our expectation that no carbides are formed during the forging process. From the present results, it is now clear that

only nitrides form during the forging process and that the carbides observed in ^[16] must have formed during air cooling after the forging process through a martensite autotempering process.

3.1.3. Size and frequency of precipitates

The morphology of the extracted precipitates formed at different TAS / FFT are shown in **Figure 5 a-e**. The size distribution of the extracted precipitates are illustrated in **Figure 5f**.

There is no effect of 0.2 strain on the size distribution of the precipitates, as can be seen by comparing the results for the 1100-0 and the 1100-0.2 specimens. This may be for the following reasons, a) the precipitates formed in the 1100-0 specimens may be fairly close to what is expected in equilibrium, which means there is a very small driving force for further precipitation at this temperature, b) the small magnitude of the strain (0.2) and associated changes in the substructure and c) the short additional time at the same temperature. As it is well known that the amount of introduced dislocations and sub-grains which represent the main sites for nucleating of the strain induced precipitates depend mainly on different parameters such as FFT, TAS and strain rate.

However, the decrease of the FFT and increase in the TAS in case of 1050-0.4 specimens compared to 1100-0 and 1100-0.2 specimens, have a great effect of the formation of a new and finely distributed precipitates as can be observed from Figure 5c compared to Figure 5a and b. These led to a decrease in the frequency of the largest precipitates and an increase in the frequency of the smallest precipitates with ECD lower than 40 nm as shown in Figure 5f. These can indicate the slow growth kinetics at this stage of the forging process in spite of the large number of the small precipitates. This is in line with Jeanmaire et al. ^[10] who investigated the precipitation of AlN in low-nitrogen high-aluminium maraging steel and found that the slow growth of AlN precipitates at low temperatures may be due to the lower diffusivity of Al at 1000 °C compared to 1200 °C: using the mobility database in Thermo-Calc Dictra MobFe2, they showed that the diffusion coefficient of Al (D_{Al}) at 1000 °C is 35 times lower than at 1200 °C. Also, these have a great effect on the mean

ECD and $D90\%_{ppt}$ of the extracted precipitates as can be seen from **Figure 6**. This is because of the high stored energy introduced by the deformation and the reduction of FFT led to decrease the activation energy for nucleation and increased the driving force for precipitation in addition to the increased diffusion rate of the microalloying elements in the strained austenite ^[17]. Also, in addition to the prior austenite grain boundaries, the new introduced sub-grain boundaries and dislocations as a result of deformation led to increasing the nucleation sites and the kinetics of precipitation ^[17] through the pipe diffusion along with dislocation core ^[18]. Okaguchi and Hashimoto ^[19] concluded that the precipitation kinetics in the Nb-Ti bearing HSLA steel is accelerated in deformed austenite compared to undeformed austenite and becomes more significant at 900 °C than at 1000 °C. The precipitates formed in the deformed austenite were also finer than those formed in the undeformed austenite.

Further decrease in the temperature and increase in the total strain in the case of the 1000-0.6 specimens, together with the help of bulk and dislocation pipe diffusion, caused growth and coarsening of the large precipitates or the newly formed strain-induced precipitates at the expense of smaller precipitates. This was clearly observed from the reduction of the frequency of precipitates smaller than 40 nm and the increase in the frequency of large precipitates (see Figure 5f). This behavior may result from the lower supersaturation of the solution as well as the nucleation rate ^[20]. At this stage, the particles with radius lower than the critical radius will dissolve while the particles with a radius larger than the critical radius will grow up continuously. These changes led to a sharp increase in the mean ECD and $D90\%_{ppt}$ of the extracted precipitates as can be seen from Figure 6. Further decrease in the temperature and total strain in the case of 950-0.8 compared to 1000-0.6, led to a slight increase in the frequency of the newly formed small precipitates (less than 40 nm) without any change in the frequency of the largest precipitates. These are accompanied by a small reduction in the mean ECD and $D90\%_{ppt}$ of the precipitates (see Figure 6).

Generally, there is a good agreement between the experimental results for the precipitate types with the thermodynamic calculation results even without taking the applied strain in account for the thermodynamic calculations.

3.2. Grain structure

For the whole range of TAS, i.e. from 0 to 0.8, and FFT, i.e. 1100 to 950 °C, the final microstructure comprises lath martensite with a tiny fraction of fine precipitates as shown in **Figure 7**. It can be seen that the precipitates are located on grain boundaries, sub-grain boundaries and within the martensitic laths.

The variation in the precipitate characteristics discussed above i.e. precipitate distribution, mean ECD and $D_{90\%ppt}$ resulting from the formation, dissolution, growth and coarsening of the precipitates has a clear impact on the grain structure as discussed below. The reconstructed PAG structure, the PAGS expressed as a mean ECD and their size distributions after removal of all edge grains are given in **Figure 8**. **Figure 9** shows inverse pole figures (IPF) with superimposed image quality (IQ) maps, ECD mapping of each grain and ECD grain size distributions after removal of all edge grains. **Figure 10a** shows grain boundary misorientation distributions for the as-quenched microstructures.

At the beginning of the forging process, in the case of the 1100-0.2 specimens, the high solubility of nitrogen at 1100 °C leads to the dissolution of some of the precipitates still present in the 1100-0 specimens. This leads to a decrease in the grain boundary pinning effect of the precipitates. The observed grain growth seen as an increase in the PAGS may result from a decrease in the grain boundary pinning effect of the precipitates or, maybe, it is just a coarse recrystallized grain size resulting from the high temperature and small applied strain. The increase in the PAGS is seen in an increase in the mean effective grain size (EGS) and the effective grain size at 90% in the cumulative grain area distribution ($D_{90\%EGS}$) of the final microstructure. However, with a further decrease in the forging temperature and the additional strain in the case of specimen 1050-0.4, there

is an increase in the formation of small strain induced precipitates, as shown above. This increases the grain boundary pinning effect, thereby retaining a finer recrystallized austenite grain size at this stage of the forging process. In the case of the specimen 1000-0.6, there is no difference in the PAGS compared to the 1050-0.4 specimens. At the end of the forging process, i.e. specimen 950-0.8, precipitate coarsening, which started at 1000 °C, reduces the grain boundary pinning effect leading to grain growth and an increase the PAGS. Also, this coarsening in the microstructural features may result from incomplete recrystallization, recrystallization to a coarser grain size or recrystallization followed by grain growth. Further investigations are needed to identify the mechanisms leading to the observed changes in the PAGS.

The PAGS has a clear impact on the sub-structures of the quenched microstructures. This can be seen in Figure 9 and **Figure 10b**, which show the dependence of $D_{90\%_{\text{EGS}}}$ on the PAGS.

4. Conclusions

The precipitation behavior during the thermomechanical processing of low-cost low-alloy CrNiMoWMnV ultrahigh-strength steel has been studied. The investigated steel has been melted in an induction furnace and then refined using ESR technology with a CaF_2 -based slag. The effects of the total applied strain and finish forging temperature have been evaluated in terms of precipitate type, morphology and size, and austenite grain size development. In addition, the effects of water quenching from different stages of the hot forging on the final microstructure have been characterized in terms of mean effective (high-angle) grain size (EGS) and the 90th percentile in the cumulative grain area distribution ($D_{90\%_{\text{EGS}}}$). The following conclusions can be drawn.

1. Whatever the applied total strain and the finish forging temperature, the microstructure of the investigated steel after water quenching consists mainly of martensite with a small fraction of precipitates. The microstructure features such as PAGS, EGS and $D_{90\%_{\text{EGS}}}$ show variation during the forging process as a result of the formation, dissolution and coarsening behavior of the TiN and AlN precipitates.

2. At the beginning of the forging process with TAS 0.2 and FFT 1100 °C, compared to the non-strained specimen austenitized at 1100 °C, there is not much difference in the precipitate statistics i.e. size distribution, ECD, D90%. This is due to the fact that the precipitates present after heating to 1100 °C are probably close to the equilibrium state and the small additional strain of 0.2 does not change the situation.
3. The decrease in the FFT to 1050 °C with TAS 0.4 led to the formation of small strain induced precipitates, which increases the frequency of precipitates finer than 40 nm in ECD and reduces the frequency of the largest precipitates, leading to significant decreases in the values of ECD and D90%.
4. At the later stages of the forging process, the lower supersaturation with respect to the precipitation of AlN reduced the nucleation rate of new precipitates with the result that precipitate coarsening led to an increase in the mean precipitate size.
5. At the beginning of the forging process, the high temperatures and small strain led to a coarse recrystallized grain which is seen as a larger PAGS and D90%_{EGS} of the quenched martensite. This coarsening may result from a decrease in the grain boundary pinning effect of the precipitates as a result of dissolution of some precipitates.
6. The recrystallization process of the investigated steel started at 1050 °C after 0.4 total strain.
7. The reason for the observed increase in the PAGS after a total strain of 0.8 with a finish forging temperature of 950 °C requires further investigation.

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Figures

Graphical abstract

Figure 1. Gleeble thermomechanical procedures (a), cylindrical sample dimensions, cutting plane and selected section for investigation (b).

Figure 2. Thermodynamic calculations for precipitates types formed at high temperatures.

Figure 3. Typical TEM micrographs, corresponding SAD patterns and corresponding EDS analysis of large hcp plate-like AlN precipitate with an enlarged HRTEM micrograph (a, b, c), TiN precipitate with enlarged HRTEM micrograph (d, e, f) and AlN-TiN composite (g, h, i)

Figure 4. Elemental EDS mapping of composite TiN-AlN precipitates with a cap of spherical morphology of AlN on the pre-existing TiN precipitate (a), TiN and composite TiN-AlN precipitates with a cap of plate-like morphology of AlN on the pre-existed TiN (b), precipitate and

TiN and composite TiN-AlN precipitates with a shell of spherical morphology of AlN and core of TiN precipitates (c).

Figure 5. Typical STEM micrographs of carbon extraction replica for the morphology and distribution of the extracted precipitates for the investigated steel at different TAS and FFT, 1100-0 (a); 1100-0.2 (b); 1050-0.4 (c); 1000-0.6 (d) and 950-0.8 (e). Size distribution of the precipitates at

different FFT and applied TAS (f). red dotted circle in (c) refer to some of the new and finely distributed precipitates.

Figure 6. Effect of TAS and FFT on the mean ECD and D90%_{ppt} of the extracted precipitates.

Figure 7. Inlens SEM micrographs of the investigated steel for the samples 1100-0 (a, b); 1100-0.2 (c, d); 1050-0.4 (e, f); 1000-0.6 (g, h) and 950-0.8 (i, j) showing the positions of precipitates within the microstructure. Some of the precipitates are marked with yellow circles in b, d, f, h and j.

Figure 8. PAG structures, ECD mapping of each PAG, size distribution and mean PAGS obtained by analyzing EBSD data with MTEX and MATLAB software for the samples 1100-0 (a, b, c); 1100-0.2 (d, e, f); 1050-0.4 (g, h, i); 1000-0.6 (j, k, l) and 950-0.8 (m, n, o). The images (a, d, g, j, m) show are combinations of austenite orientation with image quality.

Figure 9. Inverse pole figures (IPF) with superimposed image quality (IQ) maps, ECD mapping of each grain, ECD grain size distribution and mean EGS for the samples 1100-0 (a, b, c); 1100-0.2 (d, e, f); 1050-0.4 (g, h, i); 1000-0.6 (j, k, l) and 950-0.8 (m, n, o).

10.

boundary misorientation distributions for all samples (a) and influence of the forging parameters on the PAGS, $D_{90\%EGS}$ and mean EGS (b).

Table 1. Chemical composition of the investigated steel [wt.%].

C	Cr	Ni	Mo	W	Mn	Si	V	Ti	Nb	Cu	Al	P	S	N	O	Fe
0.29	2.37	2.45	0.33	1.23	0.53	0.81	0.085	0.003	0.001	0.020	0.058	0.0186	0.014	0.018	0.007	balance

Table 2. Specimen codes and conditions used in the current study.

Thermomechanical treatment	Specimen Code	Total Applied Strain (TAS)	Finish forging temperature (FFT) [°C]
1	1100-0	0	1100
2	1100-0.2	0.2	1100
3	1050-0.4	0.4	1050
4	1000-0.6	0.6	1000
5	950-0.8	0.8	950

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Table 3. Predicted equilibrium precipitate volume fractions and compositions.

Temperature [°C]	Precipitate [Vol. %]	Total [Vol. %]	Chemical composition [wt.%]							
			C	N	Al	Ti	V	Cr	Nb	W
1100	AlN	0.075	0.081	--	34	66	--	--	--	--
	TiN	0.006		2	21	--	72	4	1	1
1050	AlN	0.093	0.099	--	34	66	--	--	--	--
	TiN	0.006		2	20	--	72	4	1	1
1000	AlN	0.105	0.111	--	34	66	--	--	--	--
	TiN	0.006		3	19	--	72	4	1	1
950	AlN	0.113	0.119	--	34	66	--	--	--	--
	TiN	0.006		4	18	--	72	4	1	1

Table 4. Compositions of different precipitate types measured with EDS.

Precipitate		N	O	Al	S	Ti	V	Fe	Nb	Mo	W
AlN	wt. %	35 ± 1	3 ± 1	60 ± 0	1 ± 0	1 ± 0	--	--	--	--	--
	at. %	51 ± 1	3 ± 1	44 ± 1	1 ± 0	1 ± 0	--	--	--	--	--
TiN	wt. %	33 ± 2	2 ± 1	--	--	49 ± 1	7 ± 0	1 ± 0	2 ± 1	2 ± 1	4 ± 1
	at. %	61 ± 2	3 ± 1	--	--	28 ± 2	4 ± 0	1 ± 0	1 ± 0	1 ± 0	1 ± 0
AlN-TiN	wt. %	34 ± 1	1 ± 0	35 ± 3	--	21 ± 2	3 ± 0	1 ± 0	--	--	--
	at. %	57 ± 2	1 ± 0	26 ± 3	--	13 ± 1	2 ± 0	1 ± 0	--	--	--

Table of Contents (TOC) text

The forging parameters total applied strain and finish forging temperature applied to a CrNiMoWMnV ultrahigh-strength steel affect precipitate size distributions through their effect on the degree of strain-induced precipitation as well as precipitate dissolution and coarsening. Precipitate size distributions, in turn, affect the grain size of the prior austenite and the final microstructure.