Influence of heating rate and temperature on austenite grain size during reheating steel

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Abstract. Controlling the final microstructure is one effective way to get HSLA steel with good mechanical properties. The structure of the desired item on the final microstructure depends on the initial grain size formed during the initial heating process, where to get super fine ferrite grains, it should form the initial austenite grain smooth during the heating process. Austenite grain size at the beginning of the heating process is important in order to obtain the size of the final microstructure that provides maximum mechanical properties. In this study, HSLA steel reheated to a temperature of 960°C, 1060°C and 1120°C with holding time variation of 10, 30 and 60 minutes at a heating rate of 5°C/minute, 7.5°C/minute and 10°C/minute, then water quenching. The austenite saw by using optic microscope and count by ASTM E112 method. From the results it is concluded that there is a relationship between temperatures interrelated heating, heating rate and holding time on the growth of austenite grain. The higher the temperature, the heating occur austenite grain size. While the most optimal results obtained for reheated temperature 1060°C with a heating rate 7.5°C/minute and the heating temperature 1120°C with a heating rate 5°C/minute.

1. Introduction
Controlling of microstructure is one of effective ways to get the alloyed steels with a good mechanical properties[1]. Grain structure desired in the end depend on grain microstructure formed during the heating process and the beginning of its deformation, where to get a ultrafine ferrite grain, it must be formed small austenite under reheating process [1, 2].

The research that originated from prior austenite where austenite grain size was reheating before deformed, has an important role where the prior austenite grain size will determine the grain size after recrystallization and grain growth after the steel is deformed [3,4]. If we examine first the heating of alloyed steel, the austenite grain coarsening has to be considered an important process for grain size development. The austenite grain size represents the initial grain size for the subsequent rolling process. According to A Kern [5] the influence factors for grain size evolution during heating steel are chemical composition, solution behavior of carbonitride, heating time and temperature. According to T.A. Kop, et. all, the ferrite-start temperature depends both on the manganese content and on the amount of niobium in solution [6]. The effect of deoxidants on size distribution of particles and austenite grains has been discussed by Karasev based on the relationship between surface area of particles and that of grains per unit volume of metal [7]. Most of the research based on the investigation of grain growth kinetic under isothermal annealed condition based on Beck formula according to the empirical equation \( D = k t^m \), where D is the average grain size at time t, \( t \) is holding time, and \( m \) (the time exponent) and k are temperature-dependent parameters [8].
During reheating the steel, the effects of heating rate on the precipitate distribution at the onset of grain growth will depend on the initial precipitate size, amount of solute in the matrix, and solute diffusivities, along with the time–temperature history controlled by the heating rate [9]. Further, grain growth in non-isothermal systems can be a very complex process, as recently summarized by Mishra and DebRoy [10]. For example, even in the classical grain growth experiments austenite grain growth kinetics at the holding temperature may depend on the heating rate employed to reach this temperature.

Currently in industrial applications, the grain size of the prior austenite as the basis for calculation is only obtained through a trial-and-error by heating the steel to a reheating temperature and hold in a several time under isothermal condition according to the kinetic grain growth based on Beck formula. To obtain the real condition, it is important to simulate the non-isothermal condition by changing the heating rate during annealing the steel. Therefore, the present work was undertaken to experimentally investigate the influence of temperature, holding time and heating rate on austenite grain size, designed for use in reheating a HSLA steel.

2. Experimental Procedure

The composition of the HSLA steel used was, as expressed in wt pct on table 1. A section of industrial steel plate with 6 mm thick has been cut for reheating specimens that were used to this study. The specimens were reheating at varied temperature (960°C, 1060°C and 1120°C) by varied reheating rate (5°C/minute, 7.5°C/minute and 10°C/minute) and hold for varied holding time (10, 30 and 60 minutes), while the use of heating rate with three different interval rates; during reheating has been performed in non isothermal condition.

| Table 1. HSLA Steel Composition (% wt) |
|----------------------------------------|
| C | 0.12 | Si | 0.266 | Mn | 0.645 | P | 0.009 | S | 0.005 | Al | 0.034 | Nb | 0.028 | V | 0.011 | N | 0.0037 | Fe | Balance |

On examining the final microstructure, the specimens were quenched until room temperature, which allowed the identification of the austenite grains. It is well known that revealing the austenite grain boundaries always be a challenge for some researchers. In this work, using a modification of Villella’s reagent [11] which is diluted with aquadest, the austenite grain boundary was successively revealed. The resulting grain size was measured with the linear intercept method [12].

To determine the condition of the microstructure occur, it must be observed, especially the phase austenitic microstructure. In observing the austenite grain boundary phase, there was an obstacle in the observation using an existing etching substance. By etching solution of 4 g of picric acid in 100 ml of distilled water added with 8 drops Teepol and 8 drops of HCL, with the technique of etching solution in hot conditions and samples are also in hot conditions, which for the overall sample indicates the grain boundaries are fairly obvious.

3. Result and Discussion

Austenite Evolution

From the results of the evaluation of the etching substance sightings austenite grain boundaries, and the obtained mixture etching method that is appropriate for this study. For examples of the appearance of grain boundaries for each treatment process experienced by the sample can be seen in the picture below.
Figure 1. Austenite Grain formed during reheating 960°C with heating rate 5°C/minute and holding time 10, 30, 60 minute.

Figure 2. Austenite Grain formed during reheating 1060°C with heating rate 5°C/minute and holding time 10, 30, 60 minute.

Figure 3. Austenite Grain formed during reheating 1120°C with heating rate 5°C/minute and holding time 10, 30, 60 minute.

Figure 4. Austenite Grain formed during reheating 1060°C with heating rate 7.5°C/minute and holding time 10, 30, 60 minute.

Figure 5. Austenite Grain formed during reheating 1120°C with heating rate 7.5°C/minute and holding time 10, 30, 60 minute.
Austenite Grain Size

After the solution and etching method to show austenite grain boundaries, the grain size was calculated by using the intercept method, in accordance with ASTM E112 standard metallographic calculation. From the calculations, the average diameter of the austenite grain for each temperature, the rate of heating and holding time, such as shown in figure 7, 8 and 9.

Figure 6. Austenite Grain formed during reheating 1060°C with heating rate 10°C/minute and holding time 10, 30, 60 minute.

Figure 7. The relation graphs between grain size, reheating temperature and holding time at the heating rate 5°C/minute.

Figure 8. The relation graphs between grain size, reheating temperature and holding time at the heating rate 7.5°C/minute.
Figure 9. The relation graphs between grain size, reheating temperature and holding time at the heating rate 10°C/minute.

Figure 7, 8 and 9 shown that a heating rate of 5°C/min, 7.5°C/min and 10°C/min for each of the heating temperature by increased the holding time will increase the austenite grains size. When the heating temperature increased, the grains size formed increasing. This is due to the increase of holding time and heating temperature that will increase the activation energy of growth which will negate previous dislocations that exists on the grain boundaries so the growth of grain boundaries is not hindered by dislocation for growing.

In addition when we compared the austenite grains formed, the heating rate of 7.5°C/min will formed smaller. From this comparison it can be seen that the heating rate also plays a role in grain growth. From the pictures it can be presumed that on the low heating rate, the disintegration homogeneity of the dislocations more bigger so it will lowering obstacles that inhibit grain growth especially on the grain boundaries. In addition, the minimum energy needed for atoms to diffuse to obtain a more stable state (Q) will be smaller which will increase the grain boundaries migration to continue to grow even greater [13]. With a lower heating rate, homogeneity disintegration of sediment contained in the matrix is higher so it does not hinder the grain boundaries to continue to grow. However this needs to be investigated further by using Electron Microscope.

Figures 7, 8 and 9 shown that increased temperature will increase austenite grain that occurs. In addition to great affect grain growth activation energy, temperature rise will also increase the segregation of the solute contained in the matrix. Please also allege that at high temperatures will cause pressure maturation "Zener" which is a factor that can increase the growth of the grain size [14].

4. Conclusions

From the observation and discussion of the data obtained the following conclusions:
1. Large grain austenite formed with increasing heating temperature.
2. Austenite grain size increased with increasing holding time at the same heating temperature.
3. Austenite grain size will be smaller by increasing heating rate at the same heating temperature.
4. There is a relationship that is intertwined between the heating temperature, heating rate and holding time on the austenite grains growth.
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