Intercritical Annealing of 22MnB5 for Hot Forming Die Quenching

Haotian Yan*, Massimo Di Ciano, Mohit Verma and Kyle J. Daun

*University of Waterloo, 200 University Avenue West, Waterloo, ON N2L 3G1, Canada

* corresponding author’s E-mail: h28yan@edu.uwaterloo.ca

Abstract. Austenitization models for ultrahigh strength steels like 22MnB5 are needed to: optimize existing furnace-based processes for hot forming die quenching (HFDQ); explore the potential of intercritical annealing to obtain softer, more ductile parts; and develop new HFDQ technologies such as direct-contact heating. This paper evaluates three such models using austenite phase fractions derived from Vickers hardness measurements made on Al-Si coated 22MnB5 coupons heated within lab-scale muffle furnace and then water-quenched. Overall, predicted trends are in good agreement with the experimental results at different soak temperatures, but experimental trends with increasing soak times are less obvious. The experimental results also highlight a deficiency in a recently-proposed physics based austenitization model at higher soak temperatures.

1. Introduction

In hot forming/die quenching (HFDQ), also called hot-stamping or press-hardening, blanks are first heated within a furnace, before being rapidly and simultaneously formed and quenched in a water-cooled die into martensitic parts. One of the key ultrahigh strength steels (UHSS) used for hot forming is Usibor® 1500 P, consisting of 22MnB5 with an Al-Si coating that reacts with iron in the substrate steel during heating to form a permanent Al-Si-Fe intermetallic layer that prevents oxidation and decarburization, and provides some long-term corrosion protection.

The heating/austenitization phase is a time- and energy-consuming activity in most HFDQ lines. Accordingly, there is considerable opportunity to improve process efficiency provided the crucial austenitization stage is fully-understood. There is also growing interest in intercritical annealing of UHSS, in which the blank is heated to a temperature between the austenite start and completion temperatures, $T_{A1}$ and $T_{A3}$, and then held for a set soak time to obtain an intermediate degree of austenitization. Instead of a fully-martensitic part, the final microstructure is a combination of martensite and ferrite, so hardness is partially sacrificed for improved ductility/elongation-at-failure [1]. Incomplete austenitization through non-uniform heating also enables parts having tailored microstructures and, thus, distributed mechanical properties that can enhance crash performance in certain situations [2].

These applications establish the need for a reliable constitutive model of steel austenitization at soak temperatures starting at $T_{A1}$. Several computational models have been developed to address this need. Recently, Di Ciano et al. [3] presented an empirical first-order reaction model to describe austenite formation kinetics of 22MnB5 during continuous heating, although this approach assumes the steel is heated beyond $T_{A3}$, and thus cannot be directly applied to intercritical annealing. Speich et al. [4] proposed a numerical austenitization model for manganese steel, that included rapid and slow
Table 1. Elemental composition of steels considered in this study. (Balance Fe.)

| Ref. | C    | Mn   | Si   | Cr   | Al   | Ti   | Ni   | S    | P    | Cu   | B    | Mo   | Nb   | V    |
|------|------|------|------|------|------|------|------|------|------|------|------|------|------|------|
| Pres. | 0.23 | 1.17 | 0.25 | 0.20 | 0.04 | 0.034 | 0.02 | 0.015 | 0.02 | 0.002 | <0.01 |
| [1]   | 0.234 | 1.224 | 0.003 | 0.188 | 0.035 | 0.033 | 0.003 | 0.015 | 0.01 | 0.0025 | 0.005 |
| [4]   | 0.20 | 1.53 | 0.25 | 0.023 | 0.010 | 0.009 |
| [5]   | 0.25 | 1.40 | 0.40 | 0.50 | 0.05 | 0.010 | 0.025 | 0.005 |
| [10]  | 0.266 | 1.167 | 0.306 | 0.216 | 0.032 | 0.036 | 0.0026 | 0.008 | <0.001 |

1Did not distinguish Cr and Mo

transformation of pearlite into austenite, slow growth of ferrite into austenite, and final equilibration of ferrite and austenite, based on the diffusion of carbon and manganese into austenite and ferrite phases. Li et al. [5] developed a semi-empirical austenitization model for 22MnB5 that includes detailed nucleation, growth, and impingement submodels, with parameters tuned using dilatometry measurements. Wolf et al. [1] developed an empirical polynomial model using metallography carried out on 22MnB5 samples heated in a muffle furnace and quenched.

The present study evaluates these candidate austenitization models using hardness measurements carried out on Usibor® 1500 P coupons heated within a muffle furnace and then water-quenched. The experiments are done over a range of soak times and temperatures to represent the full intercritical annealing process envelope. Austenite phase fractions are then inferred from hardness measurements using a mixing rule, some of which are validated using quantitative metallography. Results obtained at different soak temperatures are generally consistent with trends predicted by the models, but the influence of soak time is less clear. The results also highlight a discrepancy between experimental results and a recently proposed model at higher phase fractions, likely due to an error in the treatment of grain impingement and model over-tuning.

2. Austenitization Models

Excluding Di Ciano et al.’s model [3], which does not apply directly to intercritical annealing, we consider three remaining candidate austenitization models, ranging from the purely empirical to ones that capture the detailed nucleation and growth mechanisms that underlie austenitization. The elemental composition of the steels considered in these studies, along with the present study, are summarized in Table 1. The simplest model is Wolf et al.’s [1] empirical polynomial fit to austenite volume fractions inferred from metallography carried out on furnace-heated and quenched 22MnB5 coupons. This correlation results in

\[ f_\gamma(t, T) = -253.963 + 0.9582T - 0.0012046T^2 + 5.0605 \times 10^{-7}T^3 + \frac{1.022}{t} - \frac{9.475}{t^2} \]  

where \( f_\gamma \) is the instantaneous austenite volume fraction.

Li et al. [5] take a more sophisticated approach in which austenite nucleation, growth, and impingement, are modeled explicitly according to coupled ordinary differential equations:

\[ \dot{N}_\gamma = \begin{cases} \dot{f}_\gamma \exp(-Q_\gamma/RT), & f_\gamma \leq f_p \\ 0, & f_\gamma \geq f_p \end{cases} \]  

\[ \dot{\gamma} = \dot{f}_\gamma N_v \]  

\[ \dot{f}_\gamma = \left( f_\gamma - f_\gamma^m \right) \frac{\dot{\gamma}}{(1 + \dot{f}_\gamma^m)^n} \]  

\[ m = \begin{cases} 1, & f_\gamma \leq f_p \\ 1 - m_0, & f_\gamma \geq f_p \end{cases} \]  

\[ n = n_0 f_\gamma^\nu \]
where \( N \) is the number of austenite nuclei, \( A_0 \), \( A_1 \) and \( \varphi_A \) represent the internal and external influencing factors, \( f_0 \) is the initial volume fraction of pearlite, \( B_0 \), \( B_1 \) and \( \varphi_B \) are material constants, \( \dot{v} \) is the volume growth rate of an austenite nucleus, \( m \) and \( n \) are related to the initial volume fraction of pearlite and transformation related parameters, and \( m_0 \), \( n_0 \), and \( \varphi_N \) are constants. The model parameters were fit to dilatometry measurements carried out on coupons heated within a Gleeble 3800 thermomechanical simulator.

Likewise, Speich et al. [4] present a multi-stage kinetics model consisting of rapid transformation of pearlite into austenite, slower transformation of ferrite into austenite, and final equilibrium between austenite and ferrite phases. Pearlite dissolution is modeled by instantaneous formation of an austenite film at the ferrite/pearlite interface, while the slower transformation of ferrite to austenite is found by numerically solving a diffusion-controlled moving boundary problem. Their simulation results were compared with austenite volume fractions inferred from metallography and dilatometry measurements carried out on coupons annealed in lead pots and subsequently quenched.

### 3. Experimental Methodology

Austenite phase fraction predictions from the three models described above are compared to experimental measurements carried out on 1 cm \( \times \) 1cm \( \times \) 2 mm coupons cut from a Usibor® 1500 P sheet, furnace-heated, and then water-quenched. The elemental composition of the coupons, shown in Table 1, was measured with an inductively coupled plasma per ASTM-E1097-12 [6] and the carbon content was determined per ASTM-E1019-11 [7].

The test matrix consists of eight soak temperatures between 730°C and 905°C and a range of soak times, as summarized in Table 2. The minimum and maximum temperatures were chosen to ensure the commencement and full completion of austenitization. The range of soak times vary with soak temperature, reflecting the fact that significantly longer soak times are required to reach phase equilibrium at lower soak temperatures (e.g. 24 hours for 740°C and 2 seconds for 906°C [4]). A sample temperature profile can be seen in Figure 1. In order to assess the repeatability of the results, three coupons were simultaneously heated, quenched, and analyzed for each test condition shown in Table 2.

**Table 2. Test matrix (number of samples)**

| Soak Temp. [°C] | 10 s | 18 s | 32 s | 56 s | 100 s | 6 min | 9.4 min | 16.7 min | 29.6 min | 1.56 hrs | 4.94 hrs | 27.78 hrs |
|-----------------|------|------|------|------|-------|-------|---------|----------|----------|----------|----------|-----------|
| 730             | 1    |      |      |      |       |       |         |          |          |          |          |            |
| 755             |      | 1    |      |      |       |       |         |          |          |          |          |            |
| 780             |      |      | 1    |      |       |       |         |          |          |          |          |            |
| 805             |      |      |      | 1    |       |       |         |          |          |          |          |            |
| 830             |      |      |      |      | 1     |       |         |          |          |          |          |            |
| 855             |      |      |      |      |       | 1     |         |          |          |          |          |            |
| 880             |      |      |      |      |       |       | 1       |          |          |          |          |            |
| 905             |      |      |      |      |       |       |         | 1        |          |          |          |            |

**Figure 1.** Heating curve for Usibor® 1500 P coupons soaked at 905°C for a 10 s hold time.
For each test, once the furnace equilibrated at its set-point temperature, the door was quickly opened and the three test samples placed inside. At the end of the soaking time, the samples were extracted from the furnace and quenched in water at room temperature to ensure the austenite was completely transformed into martensite. The coupons were then mounted, polished and etched using a 2% Nital solution. Vickers hardness measurements were used to infer the volume fraction of austenite formed during intercritical annealing according to a mixing rule [8]

\[ f_\alpha = \frac{H_0 - H}{H_0 - H_{eq}} \]  

(8)

where \( H_0 = 180 \text{ HV} \) and \( H_{eq} = 550 \text{ HV} \) are representative hardness values measured from as-received and fully-martensitic 22MnB5. Equation (8) assumes that the microstructure of the quenched sample consists entirely of ferrite and martensite, the latter formed from austenite, so the austenite volume fraction is equivalent to the calculated volume fraction of martensite.

4. Results and Discussion

Metallographic analysis of coupons heated for five hours or longer showed a gradation in grain size in the direction parallel to the coated surfaces, as shown in Figure 2 (a) and (b), with larger grains appearing near the unprotected coupon edges (i.e. where the coupon was cut from the larger sheet). In contrast, the microstructure is nearly homogeneous near the middle of the coupon, and there is negligible variation close to surfaces protected by the coating. This phenomenon is likely due to decarburization through the unprotected surface, which is known to occur above 700°C over long soak times [9]. Hence, as shown in Figure 2 (c) and (d), coupons heated for short soak times exhibit homogeneous microstructures. Accordingly, all subsequent metallography and hardness measurements are carried out at the coupon interior, away from the edge-affected zone.

In general, one expects smaller-sized lath microstructures in steels austenitized for short soak times, compared to those soaked for longer times, which results in a harder steel after quenching, and thus, steel coupons held at 830-900°C for two seconds were harder than those held at 950°C for 300 seconds [10]. The influence of soak time and temperature on austenitization is further summarized in terms of the incipient volume fraction, \( f_{\text{inc}} \), and equilibrium volume fraction, \( f_{\text{eqp}} \), at each soak temperature, shown in Figure 3 (a) and (b), respectively. The former represents the volume fraction between 2-18 seconds after reaching the soak temperature, while the latter corresponds to phase equilibrium after an extended soak time. Both these parameters indicate a slight softening as the soak temperature increases beyond 855°C. In order to confirm that this phenomenon corresponds to grain

![Figure 2](image.png)

**Figure 2.** (a, b) Inhomogeneous grain sizes observed close to the unprotected surface for coupons heated to (a) 730°C and (b) 755°C, both for a 24 hour soak time; and (c, d) homogeneous grain sizes observed close to the Al-Si coated surface for coupons heated to (c) 730°C and (d) 755 °C for soak times of 18 s.
growth, scanning electron microscopy (SEM) was performed on samples held at 855°C for 18 seconds and 905°C for 30 minutes, shown in Figures 4 (a) and (b) where the lighter regions indicate martensite and the darker regions indicate ferrite.

Smaller grains are observed in Figure 4 (a) compared to Figure 4 (b), thus supporting this theory. In a study that focused on rapid austenitization of 22MnB5, Andreiev et al. [10] reported that short-soaked coupons are slightly harder at similarly high soak temperatures compared to long soak durations typical of furnaces. In general, soak time contributes to austenite formation as it promotes the diffusion of carbon at grain boundaries during phase transformation. However, an excessively long soak time also increases the risk of decarburization and coalescence of small grains, resulting in softening and the large variations in $f_{\gamma}$ with soak time. As a result, the decreasing hardness values at long soak times would lead to a false interpretation of decreasing austenite volume fraction. In reality, the austenite carbon content, originating mainly from pearlite dissolution, becomes progressively diluted as the ferrite converts to austenite.

This effect was also neglected in Equation (8), which assumes the presence of only ferrite and homogeneous austenite phases. Therefore, this approach was validated by performing quantitative metallography (systematic manual point count, ASTM standard E562 using 100 points and 10 fields) for coupons heated at temperatures close to $T_{Ac1}$, where the evolution of carbon content would have the strongest effect on hardness. A comparison of these results in Table 3 shows that the hardness-inferred austenite phase fractions are consistent with those found by quantitative metallography. Thus, although the secondary effect of evolving carbon content in austenite was neglected in Equation (8), it can still be used as a quick and simple technique to estimate austenite phase fraction.

Figure 3. (a) Incipient and (b) equilibrium austenite volume fractions inferred from hardness values, and compared with modelled results. Error bars correspond to the three samples measured for each annealing condition.

Figure 4. Scanning electron micrograph of specimens: (a) soaked at 855 °C for 18 seconds; and (b) 905 °C for 30 minutes.
Table 3. Comparison between inferred Vickers hardness results and quantitative metallography

| Temperature (°C) | Soak Times   | Volume fraction of austenite (%) | Hardness | Quantitative Metallography |
|------------------|--------------|----------------------------------|----------|---------------------------|
| 780              | 18 seconds   | 55.23 ± 3.81                     | 56.6 ± 5.54 |
|                  | 9 minutes    | 76.85 ± 3.88                     | 67.39 ± 3.43 |
|                  | 5 hours      | 65.95 ± 4.24                     | 60.16 ± 10.28 |

Figure 5 (a) shows the inferred austenite volume fraction for different soak times and temperatures. For coupons held at 730°C for 18 seconds, $H = 190$ HV, corresponding to $f_\gamma = 0.03$ according to Equation (8). Given that the as-received samples have a hardness of 180 HV, this finding supports that the eutectoid temperature is near 730°C. In general, $f_\gamma$ increases with soak temperature. One would also expect the austenite phase fraction to increase with soak time, but this is less apparent in the experimental results. Figure 5 (b) shows that, at a soak temperature of 780°C, the Li et al. [5], Speich et al. [4], and Wolf et al. [1] models predict that $f_\gamma$ increases monotonically up to around 0.72 with increasing soak time, while the experimental data covers a similar range but does not exhibit a clear trend with soak time.

In general, Figure 3 (b) shows an overall good agreement between the experimental and modeled data for $f_\text{eq}$. On the other hand, Figure 3 (a) shows that while the measured $f_\text{eq}$ is consistent with Wolf et al.’s model [1], Speich et al.’s model [4], and other experimental data reported in the literature [10], they are significantly higher than ones predicted using Li et al.’s [5] model for soak temperatures above 830 °C. This discrepancy could be explained by three factors. First, Li et al.’s model [5] contains a large number of free parameters that were fitted using dilatometry data, and it is possible that this process resulted in over-tuning. Second, this model may overpredict the influence of impingement on the transformation rate during the late stage of austenitization, per Equations (5)-(7). This seems plausible considering that the modeled austenite formation rate drops past $f_\gamma = 0.7$, at which point the overlapping particles may begin to impinge on each other. This effect is particularly visible in Figure 5 (b) at longer soaking times. In contrast, the model proposed by Speich et al. [4] excludes this effect, and is in much better agreement with the data. Finally, it should be noted that the elemental composition of the materials used in each model is different from each other, as reflected in Table 1, and thus, it could also result in the variation of hardness values/austenite phase fraction among different samples.

Heating rate may also influence the incipient austenite fraction, in part because it allows some austenitization to occur prior to reaching the soak temperature. A faster heating rate shortens the heating duration, thus lowering the austenite volume fraction upon reaching the soak temperature.

![Figure 5](image-url)  
**Figure 5.** Austenite volume fraction: (a) as a function of soak temperature and time; (b) plotted as a function of soak time at a soak temperature of 780 °C. Error bars on the experimental data show results obtained from the three samples for each annealing condition.
Since the austenite volume fractions obtained with Li et al.’s [5] model are found using experimentally-measured heating rates, and it took less time to reach 855°C (180 s) compared to 830°C (280 s) due to fluctuations in the furnace conditions, this may also explain the slightly lower austenite volume fraction observed at 855°C shown in Figure 3.

Overall the models are able to capture experimentally-observed trends, but Li et al’s model [5] significantly underpredicts \( f_a \) at higher soak temperatures. For this model to be deployed in a quantitative way, it should be reexamined, with particular focus on the impingement sub-model. Moreover, the polynomial fit provided by Wolf et al. [1] only applies to a limited range of soak temperatures and times. Its capabilities could be extended by increasing the set of experimental data used to obtain the fitting parameters.

5. Conclusions and Future Work

There is a growing need for models that can predict austenite phase fractions in order to optimize hot forming die quenching processes and to develop new techniques that exploit intercritical annealing and tailoring. The present study evaluates the validity of candidate models for austenitization of 22MnB5. The results demonstrate that the steel properties can be adjusted by varying soak temperature and time. It is also found that austenite formation is more sensitive to soak temperature than soak time due to the fact that an excessive soak time may promote grain coalescence or decarburization, leading to the softening of the steels or inhomogeneous hardness properties.

Austenite volume fractions ranging from zero to nearly unity were observed for coupons heated according to the soak temperatures and soak times used in this study. Upon reaching 830°C, austenite volume fraction becomes less sensitive to soak time, and, at soak temperatures above 855°C, the soak time has negligible influence on austenite formation. This suggests that intercritical annealing should be carried out at lower temperatures, in order to control incomplete austenitization, and nearly complete austenitization may be achieved without soaking the steels at or above \( T_{AC3} \), which could realize both energy and time savings during hot stamping.

Experimentally-inferred austenite phase fractions from the current study and Andreiev et al.’s [10] experimental data do not always show the expected monotonic trends with increasing soak times, which may be explained by grain growth. More generally, variations between experimental data and model predictions may be due to variations in the elemental compositions of the tested materials, processing conditions, and, especially, model over-tuning. These models should be developed into more robust tools for process design and optimization.

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