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Transformation Kinetics in Controlled-Power and Controlled-Temperature Cycle Testing

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Abstract

On-heating transformation kinetics were investigated for several steels by using a Gleeble capable of programmable power input as well as programmable temperature cycling. Transformation kinetics determined in both modes are reported. The temperature cycles are significantly different between the two modes due to the latent heat associated with the phase transformations. Both diffusion rates and transformation driving force increase with temperature above the eutectoid temperature, therefore the latent heat can potentially have a significant impact on the transformation kinetics. Experiments with plain carbon steels illustrate that the latent heat of austenite formation causes an appreciable temperature arrest during transformation, and the dilatation response is similarly altered. A kinetic transformation model, based on the decomposition of pearlite and the diffusional growth of austenite, reproduced the transient dilatation data obtained from both control modes reasonably well using the same kinetic parameter values.

Introduction

On-heating transformations are an important part of microstructural development in many metallurgical processing and fabricating schemes. In the case of steels, welding, hot working, and heat treating cycles all result in heating into the austenite plus ferrite and/or austenite phase fields. At the present time, there is widespread interest in modeling these processes as an aid in optimization and control of post process microstructure and properties. For these models to be applicable, they must describe thermal cycles and the phase transformation kinetics associated with the both the on-heating and on-cooling transformations, and these descriptions must be experimentally validated.

Most experimental studies of transformation kinetics utilize controlled-temperature cycling. This approach, however, is not strictly representative of typical industrial processes, which are inherently controlled energy input processes. The difference between these two control modes can be pronounced during on-heating endothermic transformations, where the latent heat of the reaction can result in a significant thermal arrest. For temperature-controlled Gleeble testing, this arrest results in a significant increase in applied power in response to the increased temperature deviations from the programmed temperature cycle. As a consequence, under some conditions, this can result in an artificially high temperature relative to that which would have been experienced (at a given fraction transformed) during a controlled-power input cycle. Since the nucleation and growth rates for on-heating transformations increase rapidly and at different rates above the equilibrium transformation temperature [Ref 1], it is conceivable that the progress of the transformation will differ between the two cycles. The purpose of the present study was to modify a Gleeble weld thermomechanical weld simulator to operate in either controlled-power or controlled-temperature modes, and to compare the temperature cycles and austenitization kinetics of medium carbon pearlitic steels in both control modes. The transformation kinetics from both experiments are then compared to the predictions of a numerical model.

Experimental Procedures

Materials and Metallography - Several plain carbon steels were used in this investigation. Initial comparisons of the thermal cycles and dilatation response in controlled-power and controlled energy modes were conducted on 0.45 and 0.29 wt. % C alloys. Additionally, a kinetic model of the austenization process was used to compare the response of the 0.29 wt. %

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C alloy in both control modes. Both alloys were in the normalized condition prior to testing, and therefore consisted of fine pearlite and proeutectoid ferrite. Samples for metallography were prepared using standard polishing techniques and were etched using 2% nital. Determinations of initial grain size and relative fractions of individual microstructural constituents were performed using quantitative image analysis software.

Controlled-Temperature Cycle Testing - Conventional controlled-temperature cycle dilatometry was conducted on a DSI Gleeble 1500 thermomechanical simulator. Diametral dilatation was measured on 6.35 mm diameter samples using a high resolution dilatometer at the location of the Cr-Al control thermocouple percussion welded to the sample, and a free span of 30 mm was used. The dilatometer was calibrated for each of the heating rates by using pure nickel samples of the same geometry. All tests were conducted in a vacuum purged enclosed chamber under flowing high purity argon with computer data acquisition. The tests were conducted using programmed linear heating rates ranging from 25 to 200°C/sec.

Gleeble Modifications - As it is currently configured, the modification provides for open loop controlled power inputs. The modification is compared with the conventional Gleeble feedback temperature control methodology in Fig. 1. The changes essentially consist of the addition of circuitry that supplies a voltage proportional to either the heat potentiometer setting on the Gleeble 1531 control module or to a millivoltage equivalent which is entered as a variable in the Gleeble Programming Language program codes, and bypassing of the comparator circuitry. The modification therefore allows for any power versus time cycle, although only constant power inputs were used in the current study.

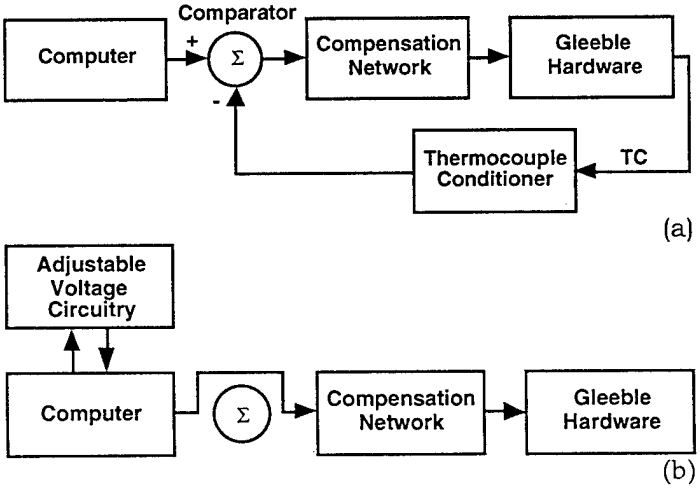

Figure 1. Block diagrams showing (a) normal closed loop Gleeble temperature-control mode, and (b) open loop power-control mode.

In the controlled-power tests, all other conditions such as free span, atmosphere, *etc.*, were held constant during the experiments. A simple power profile, consisting of a 1 sec ramp to the desired power level, followed by a constant power hold sufficient to allow the sample to reach a temperature of at least 1000°C was used. The power levels were selected to provide heating rates in the 600 to 700°C range which were similar to those used in the controlled-temperature cycles. Thus, the heating rates near the lower critical temperature in both modes are similar, and the results can be directly compared.

Results and Discussion

Comparison of Thermal Cycles - Fig. 2 compares the thermal cycles resulting from the constant power and controlled-temperature cycle tests for the 0.45 wt % C alloy at two heating rates. As shown, the thermal

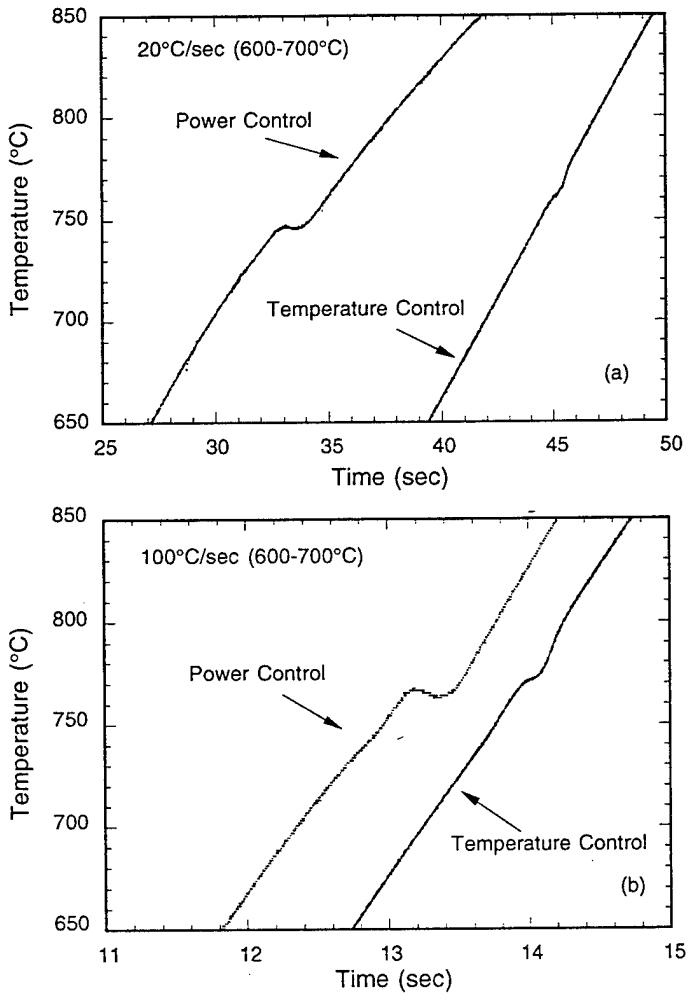

Figure 2. Comparison of on-heating thermal cycles of 0.45 wt% C steel for controlled-power and controlled-energy modes for (a) 20°C/sec and (b) 100°C/sec.

arrest is more evident in the constant power experiments, even at the lower heating rates. For the conventional controlled-temperature cycle tests, the Gleeble control algorithm compensates for the arrest by the application of additional power, so that the cycle more nearly approximates the programmed ramp. This application of power also results in a small but detectable overshoot of the programmed cycle after the initial arrest. The response in these tests is, of course, not surprising, but illustrates the differences in the two control modes. For the controlled-power tests at the higher heating rates, it is also interesting to note that the sample temperature decreases slightly following initiation of austenitization. Differences in the observed lower critical temperature (at the same heating rate) can also be seen in Fig. 2. These differences are on the order of 10°C for the 20°C/sec heating rate of Fig. 2(a), but are not thought to result from differences in control mode since duplication of the experiments resulted in similar variations irrespective of the control mode.

Dilatation Response - The dilatation for the 0.45 wt % C alloy in both control modes is shown in Fig. 3 for the 20 and 100°C/sec heating rates. For these plots, the dilatation is given in arbitrary length units (transducer output) and have been displaced vertically for clarity. As shown, there are significant differences between the two control modes. The primary difference is in the region of the curve associated with the decomposition of the pearlite which occurs nearly isothermally (or with a slight temperature drop) for the controlled-power cycle, and over a range of temperature for the controlled-temperature cycle.

The dilatation response can also be compared in terms of time and this is shown in Fig. 4 for a 20 °C/sec heating rate. As expected from the temperature cycles of Fig. 2(a), the temperature-controlled curve is compressed in time relative to the power-controlled cycle. Qualitatively, this compression results from the higher temperatures experienced by the sample during the early stages of the transformation (*i.e.* during the pearlite decomposition). Relative to typical welding processes, therefore, the controlled-temperature cycle testing results in artificially high temperatures (and apparent reaction rates) during the early portion of the transformation.

Model Fits of Controlled-Temperature Cycle Tests - As noted in the Introduction, it is conceivable that the artificially high temperatures can result in a different balance between nucleation and growth rates in the controlled-temperature cycle versus controlled-power experiments. In order to evaluate this possibility, a

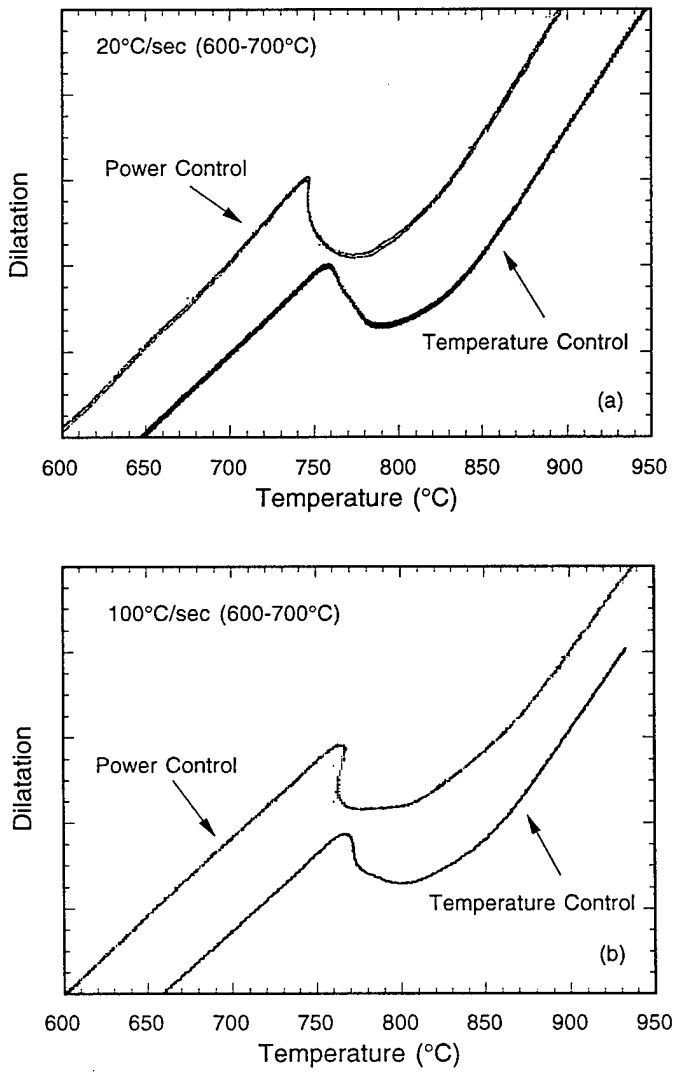

Figure 3. Dilatation versus temperature for controlled-power and controlled-temperature heating modes for 0.45 wt. % C steel. (a) 20°C/sec and (b) 100°C/sec.

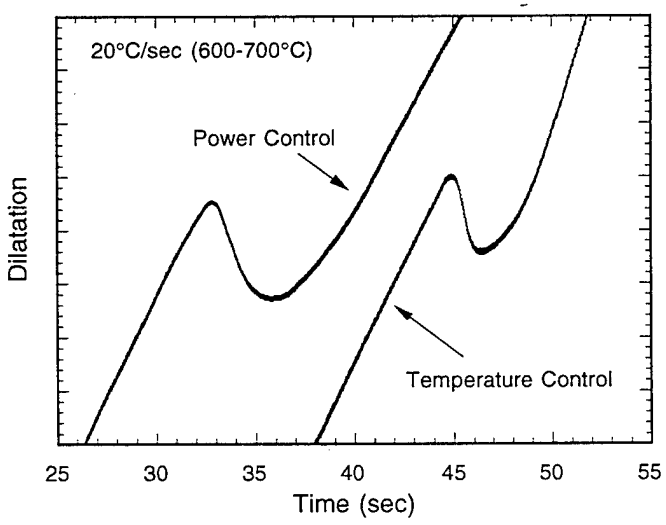

Figure 4. Dilatation versus time for 0.45 wt. % C steel using controlled-power and controlled-temperature modes for 20°C/sec heating rate.

series of controlled-power experiments were conducted on a 0.29 wt. % C steel for which the austenitization kinetics had previously been evaluated under temperature control. The details of the kinetic model which was developed for the pearlitic 0.29 wt. % steel have been presented elsewhere [Ref 2], and will only be summarized here.

In the previous study [Ref 2], the formation of austenite in a pearlitic hypoeutectoid carbon steel was studied using a combined experimental and numerical approach. The progress of the austenitization reaction over a range of heating rates was tracked experimentally through dilatometric measurements which differed from the conventional approach. In the conventional approach, the fraction transformed is obtained from extrapolated dilatation versus temperature curves for the parent and product constituents. However, recent work by Onink *et al.* [Ref 3] on the on-cooling decomposition of austenite has demonstrated that the use of the level rule in such an approach can introduce significant errors. These errors essentially result from the fact that three phases are involved in the transformation, and the dilatation (lattice parameter) of the austenite is strongly dependent on its carbon content as well as temperature. Thus, the fraction transformed is not a linear function of the relative length change, and cannot be directly inferred from a dilatation versus time/temperature record. In order to avoid this difficulty the dilatation is predicted based on the transient temperature and phase concentrations. The kinetic parameters are then adjusted so that the best fit of the dilatation data is achieved. The transformation model used is similar to that recently described by Oddy *et al.* [Ref 4]. From this direct fit, the fraction austenite versus the time/temperature path is obtained. The model treated the formation of austenite from the pearlite/ferrite mixture as a two-part process, one describing the transformation of pearlite and one describing the diffusional growth of austenite into the proeutectoid ferrite. For the pearlite decomposition, an Avrami expression [Ref 1] coupled with the additivity principal was used to describe the progress of the reaction [Ref 1]. Growth of the austenite into the ferrite was described by means of a one-dimensional diffusion model. Three kinetic fitting parameters were used, two of which are associated with the Avrami description of the pearlite decomposition, and one of which was a geometric factor associated with the ferrite transformation. Fitting was conducted simultaneously on dilatation measurements from three heating rate experiments, and used the actual thermal cycle including the deviations from the programmed cycle (such as those shown in the temperature-controlled cycles in Fig. 2).

The process is complicated by random and systematic experimental errors, and incomplete material property data. Therefore, a self-calibration process was also developed which uses the dilatation data to obtain the density variation of the various phases with temperature. This enables the model to be used in situations where accurate thermal expansion data is not available.

The procedure described above was used to determine the kinetic fit parameters of the 0.29 wt. % C alloy. For these determinations, data obtained at temperature ramp rates of 50, 100, and 200 °C/sec were fit by the model. Inputs to the model were the experimentally determined room temperature density, 7.84 g/cm^3, the metallographically determined pearlite volume fraction, 0.333, the metallographically determined grain size, 24 mm, an estimate [Ref 5] of the equilibrium eutectoid temperature, 720°C, and the diffusion coefficient of carbon in austenite [Ref 6]. Each dilatation data set was self-calibrated so that errors in the individual experiments could be separately accounted for, but a single set of kinetic parameters was used to fit all three experimental data sets simultaneously. Figure 5 shows comparisons between the experimental data and best model fits for dilatation versus time. In general, the model captures the detailed characteristics of the experimental data. For example, the 200°C/sec heating rate shows two local minima in the dilatation versus time curve and this behavior is reflected in the model. Further, comparison of the model estimates with replicated experimental determinations indicated that the model predictions typically fall within the experimental variance.

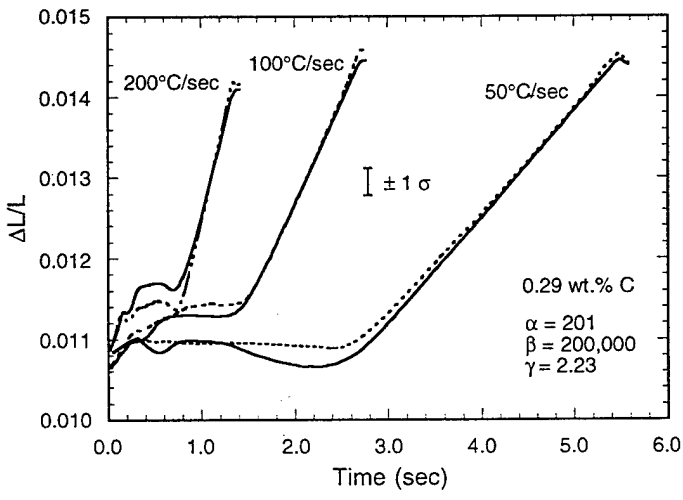

Figure 5. Comparison of experimental dilatation data (dashed) to prediction (solid) for three different heating rates for 0.29 wt. % C steel. Controlled temperature-cycle tests. Error bar is ± 1s and represents variance of dilatometer data at 700°C for three determinations at each heating rate.

Figure 6 shows the calculated individual contributions of the pearlite and ferrite to the total fraction of austenite as a function of temperature for the 50°C/sec temperature-controlled cycle. As might be expected, decomposition of the pearlite occurs at a significantly higher rate than the decomposition of the ferrite, and the model captures this behavior even though it is not readily apparent in the original dilatation curves (Fig. 4).

Model Fits of Controlled-Power Cycle Tests - Fits to the dilatation data for the controlled-power tests yielded essentially identical kinetic parameters. Hence, the controlled power data is equally well fit using the coefficients derived from the controlled-temperature cycle experiments, and vice-versa. Fig. 7 shows the experimental dilatation and model fits for the controlled-power experiments using the coefficients derived from the controlled-temperature cycle tests. The structure and accuracy of the fitted curves are similar to those observed for the controlled-temperature tests (Fig. 5). Fig. 8 shows the fraction austenite as a function of temperature for a controlled-power heating rate of 44°C/sec, and can be compared directly with Fig. 6. The curves are very similar, but are slightly different as a result of the differences in the time-temperature path (since the same kinetic parameter set was used to generate both figures).

Based on these results, the Avrami/diffusion model captures the differences in transformation rate caused by the differences in thermal cycle for the two control modes. Thus, it is not necessary to modify the kinetic parameters for the 0.29 wt. % C steel at these heating rates. Of course, it is also possible that the current model and experiments are not sensitive enough to detect subtle differences in the kinetic parameters. Based on the rationale that the primary differences between the two control modes would be in the details of nucleation, the effect would be expected to be largest during the transformation of the pearlite constituent. If the nucleation rate of austenite in pearlite is sufficiently high for both thermal cycles, then changes in this rate may not have an appreciable effect on the kinetic fit parameters irrespective of the model used. Also, since the pearlite contributes only approximately 1/3 volumetrically to the total dilatation, small changes in its decomposition parameters due to the temperature

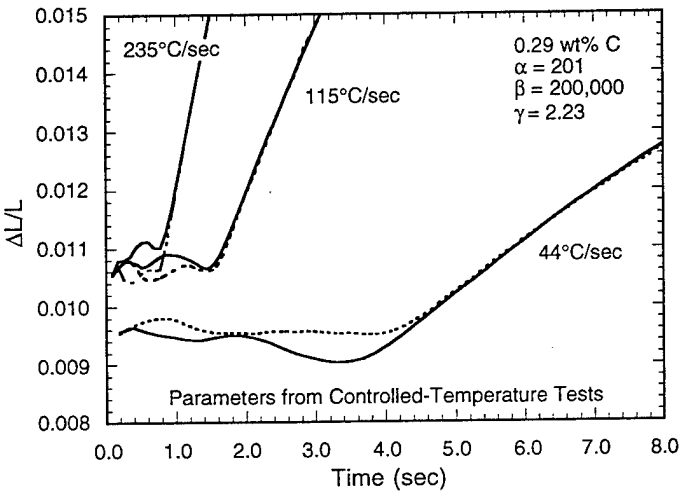

Figure 7. Comparison of experimental dilatation data (dashed) to prediction (solid) for three different heating rates for 0.29 wt. % C steel. Controlled-power cycle tests.

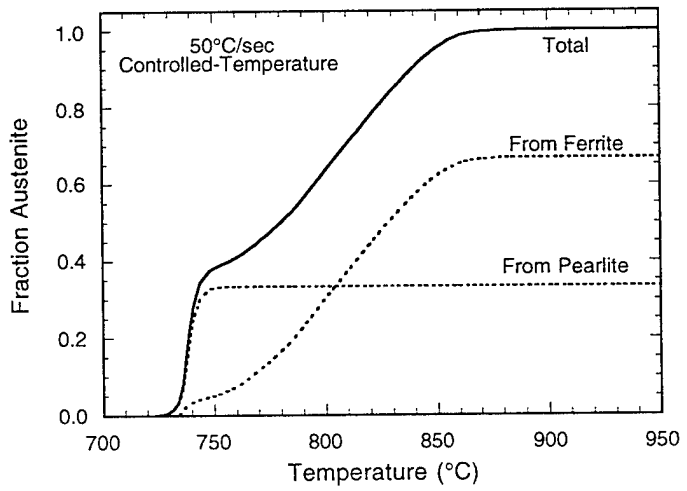

Figure 6. Fraction austenite as a function of temperature for the 50°C/sec controlled-temperature cycle. The contributions from the pearlite and ferrite constituents are shown. 0.29 wt. % C steel.

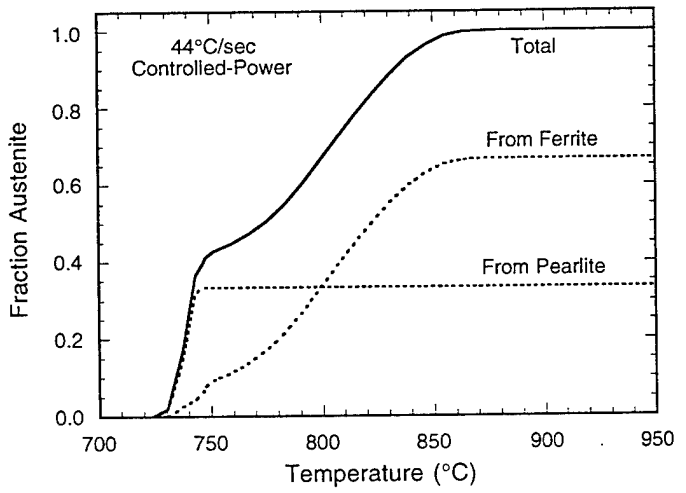

Figure 8. Fraction austenite as a function of temperature for the 44°C/sec controlled-power cycle. The contributions from the pearlite and ferrite constituents are shown. 0.29 wt. % C steel.

differences between the two modes may not be detectable within the experimental error. Finally, the current model assumes that the cementite and ferrite within the pearlite are consumed proportionally, so that neither phase remains when the pearlite is transformed to austenite. Speich and Szirmae [Ref 7] and others [Refs 8,9] have shown that cementite (and/or carbon concentration gradients) can remain in austenite formed from pearlite. This effect, which is one that is likely to influence the details of the growth of austenite in the pearlite, is not captured in the Avrami description (or other similar descriptions) of the pearlite decomposition. Further studies on higher carbon and alloy steels should help clarify these issues.

Conclusions

Several control methodologies for conducting controlled-power heating experiments using a Gleeble have been developed and the first of these, power cycle open loop control, has been implemented. Heating experiments with a 0.45 wt. % carbon steel demonstrate the differences between power- and conventional temperature-controlled cycling, and are associated with the different response of the system in supplying the latent heat of the transformation. A kinetic model of the austenitization process could predict the behavior in both modes with the same kinetic parameter set. Hence, for the particular steel and conditions examined, differences between the observed transformation rates can be explained by differences in the time-temperature path, without the need to invoke different kinetic parameters. Further experiments are planned to confirm this observation for a broader range of steels and conditions.

Acknowledgments

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