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Microstructure Evolution During Batch Annealing

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Design of industrial annealing cycles requires recrystallization and grain growth studies, which are typically carried out under isothermal laboratory condition. The kinetics coefficients of these phase transformations are obtained from such studies, which are subsequently used in designing the industrial non-isothermal cycles using the additivity principles. However, the strong heating rate effects on the grain growth kinetics necessitate such kinetics studies using industrial thermal profiles. In the present work, the hot and cold spot cycles of an industrial batch annealing cycle for AlK grade steel have been simulated in a programmable laboratory furnace. Subsequently, the effect of annealing temperature, soaking time, and heating rate on the microstructural features, such as grain size distribution, grain shape anisotropy, and grain orientation, have been investigated through extensive quantitative microscopy. The implications of these results on the design of industrial batch annealing cycles have been discussed.

Keywords AlK steel, annealing, grain growth, kinetics theory and modeling

1. Introduction

Batch annealing is one of the critical unit operations involved in the production of cold rolled and annealed flat steel coils. It influences all the key plant performance parameters, namely, overall efficiency, energy, productivity, and product quality.[1,2] In the batch annealing process, 4-5 cylindrical steel coils (weighing typically 15-30 tons each) separated by a convector plate are stacked on a furnace base. A protective cover encloses the coil where an inert or reducing H_2 gas is circulated. This enclosure is externally heated by a gas or oil fired furnace. The outer and inner surfaces of the coils are heated by convection from the circulating H_2 gas and by radiation between cover and coil. The inner portion of the coil is heated through conduction, which is retarded due to the air gaps between the sheets. As a result, during the annealing operation, different locations in the coil undergo different thermal cycles. In an industrial operation, the two locations of special interest are hot spot (near the coil surface with the highest temperature during the heating cycle) and cold spot (in the coil core with the lowest temperature), where by monitoring these two extreme locations, the product quality is controlled. Since recrystallization and grain growth are thermally activated processes, such thermal lag leads to spatial variation in microstructure, with an associated variation in the mechanical properties within a coil. Although an increase in soaking time usually results in reduction in microstructural and mechanical property variations, it also reduces the furnace productivity. Therefore, selection of soaking time in an industrial batch annealing operation requires an optimization between productivity and quality. In addition, appropriate selection of heating rate, which has a metallurgical implication on precipitation and recrystallization kinetics, and annealing temperature[3-5] form the crux of batch annealing thermal cycle design.

Recrystallization and grain growth studies are often required for designing industrial cycles. However, typically these studies are carried out under isothermal conditions in the laboratory furnaces for different duration of time. The results from these experiments are used to determine the kinetic coefficients using Johnson-Mehl-Avrami-Kolmogorov (JMAK) model for recrystallization kinetics or Beck-type correlation for grain growth kinetics. For example, grain growth kinetics is conventionally represented by the Beck-type correlation:[5,6]

$$d^n - d_o^n = k(T)\,t \quad \text{(Eq 1)}$$

$$k = k_o \exp\left(-\frac{Q^*}{RT}\right) \quad \text{(Eq 2)}$$

where, d is the mean grain size achieved at the end of an isothermal annealing cycle carried out at temperature T for a duration of t; d_o is the initial grain size; n is the grain growth exponent; k_o is the pre-exponential coefficient; R is the gas constant; and Q^* is the overall activation energy for grain growth, incorporating activation enthalpies of all the atomic processes that constitute the overall grain growth process. Even though Eq 1 and 2 are formulated for grain growth under isothermal conditions and the kinetics coefficients are generally obtained by carrying out isothermal studies, these equations are frequently used to describe grain growth over non-isothermal temperature profiles by segmenting the annealing cycles into small isotherms[7-10] and integrating the transformation kinetics over time. The basic assumption in this quasi-isothermal approach is that there is no change in kinetics of grain growth between isothermal and non-isothermal conditions. Evidently, this methodology of evaluating non-isothermal profiles will result in erroneous predictions if the kinetics of grain growth changes during non-isothermal annealing (e.g., heating rate or change in heating rate effects). In a recent work,[11] the strong influence of heating rate on grain growth kinetics as well as grain shape anisotropy and crystallographic texture was illustrated by carrying out grain growth studies at different heating rates. In another work,[12] the strong effect of non-isothermal

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cyclic annealing on the grain growth kinetics was demonstrated. Furthermore, Ref. 11 and 12 show that due to the strong heating rate effects, there can be significant error in describing the non-isothermal industrial cycles with the quasi-isothermal grain growth kinetics. Such strong heating rate effects on the grain growth kinetics suggest that the kinetic studies should be carried out using industrial thermal profiles.

The present work endeavors to investigate the microstructure evolution during batch annealing of aluminum-killed (AlK) cold rolled steel sheets for optimal design of batch annealing cycle. In this study, the hot and cold spot cycles of an industrial scale batch annealing cycle for AlK grade steel were simulated in a programmable laboratory furnace and samples with different soaking time were obtained. Extensive metallography, microscopy, and quantitative image analysis were carried out on the annealed samples. The effects of annealing temperature, soaking time, and heating rate on the microstructural features were examined. Quantification of several microstructural size, shape, and orientation parameters—namely mean grain size, grain size distribution, grain aspect ratio, specific grain boundary length, and angle of rotation—were carried out. The preliminary results from this work have been presented in a conference.[13] In this article, the results from this study have been presented and the implications of changes in the microstructural features on the final mechanical properties are discussed.

2. Experimental Procedure

The starting material for this study was ~70% cold rolled 1.0 mm thick, AlK grade steel sheet containing ~0.05% C, ~0.05% Al, and ~45 ppm N. The sheet was cut into rectangular samples (30 mm × 20 mm), and was heat-treated in a laboratory furnace. The furnace has a facility to program nine segments, where heating rate, temperature, and soaking time can be controlled. Two different types of heat-treatment cycles, simulating the industrial scale hot and cold spot cycles during batch annealing, were used in the laboratory furnace. The hot and cold spot refer to the two extreme thermal cycles experienced at the coil surface and coil core. These temperature-time profiles were obtained from a thermal model, which is described in section 3.1. After the heat-treatment, the samples were sectioned along the rolling direction, then mounted and polished on SiC and emery papers. The final lapping was done with alumina suspensions. The polished samples were etched with 2-4% nital solution for about 30 s and examined on an optical microscope. For a better statistical representation four micrographs containing over 1000 grains were analyzed for each of the samples. Subsequently the grain boundary delineated micrographs were digitized for image analysis. Image analysis was carried out on these digitized images and area, perimeter, major/minor lengths, and angle of orientation of the major axis with respect to rolling direction were measured for individual grains. Using the grain area measurements on individual grains, cumulative size distribution plots were generated for each sample. The mean grain size was determined from the cumulative size distribution plots and refers to the grain size corresponding to 0.5 cumulative fraction (strictly speaking, it is median grain size). Grain shape anisotropy was quantified by

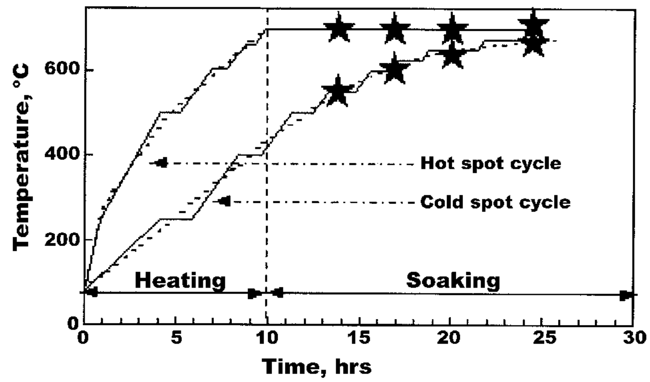

Fig. 1 Hot and cold spot cycles given in this study (solid lines) compared with the attempted industrial batch annealing cycles (broken lines)

the average grain aspect ratio (henceforth, grain shape anisotropy and grain aspect ratio will be used interchangeably), obtained from the ratio of major and minor lengths of individual grains.

3. Results and Discussions

3.1 Evolution of Thermal Profiles During Batch Annealing

The thermal profiles for the hot and cold spots were determined from a thermal model, where the transient temperature variation in the coil was obtained by solving the following energy equation in cylindrical coordinates:[1,2]

$$\rho_m C_m \frac{\partial T_m}{\partial \tau} = \frac{\partial}{\partial z}\left(k_z \frac{\partial T_m}{\partial z}\right) + \frac{1}{r}\frac{\partial}{\partial r}\left(r k_r \frac{\partial T_m}{\partial r}\right) \qquad \text{(Eq 3)}$$

where T_m is the temperature, ρ_m is the density, C_m and k_z are temperature dependent specific heat and conductivity of the coil, respectively. The radial conductivity (k_r) of the coil depends on the sheet thickness and air gap between sheets. Using appropriate boundary conditions,[1,2] the solution to Eq 3 provides the complete transient temperature history during heating and cooling cycles, at different locations of the coil. Note that this thermal model is a component of the integrated batch annealing simulator, which can predict spatial and temporal thermal and microstructure evolution as well as the final mechanical properties of different cold rolled steel coils undergoing annealing in an industrial scale batch annealing furnace. The predictions from the thermal model have been extensively validated[1,2] with the data from industrial batch annealing furnaces through in-plant experiments, where temperatures were monitored across the coils during the annealing process by embedding thermocouples in the coils. The model predictions for the hot and cold spot cycles are presented in Fig. 1 as broken lines, whereas the actual set-points used in the laboratory furnace is given as solid lines. The soaking time was varied from 4-15 h (Fig. 1), with a reference to the hot spot cycle. As the minimum heating rate possible in the laboratory furnace was 1 °C/min, the hot spot and cold spot cycles were simulated by segmenting the entire cycle with intermittent

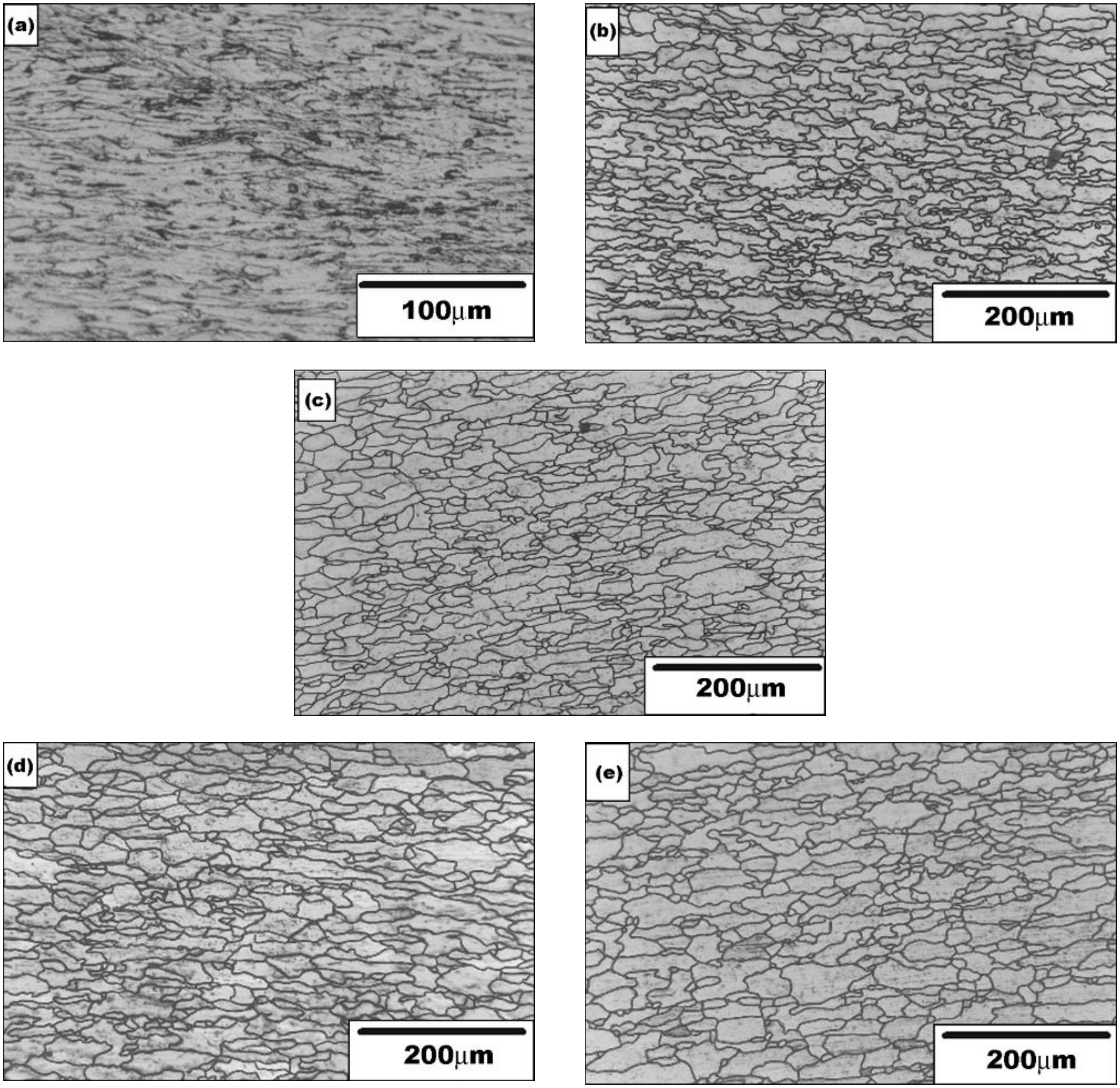

Fig. 2 Microstructure of **(a)** as received cold rolled sample, and annealed samples following the cold spot cycle with soaking time of **(b)** 4 h, **(c)** 7 h, **(d)** 10 h, and **(e)** 15 h

soaking. As shown in Fig. 1, the hot spot and cold spot cycles programmed in the laboratory furnace (solid lines) are very close to the thermal profiles predicted by the model (broken lines). The location of the various samples (with 4, 7, 10, and 15 h soaking time) is marked as stars in Fig. 1.

3.2 Microstructural Observations

The microstructure of the starting material (~70% cold-rolled AlK grade of steel) is shown in Fig. 2(a). Elongated and highly deformed grains are visible in this microstructure along the longitudinal section of the rolled sheet. Such a microstructure is common for the cold rolled steel strips. The micrographs in Fig. 2(b-e) show the evolution of the microstructure for the cold spot cycle samples. It can be observed that the grains are elongated and oriented along rolling directions, suggesting that the effect of rolling is visible even in the annealed microstructure. Such pancake-shaped grains are known to promote the desirable {111} texture in the steel.[14] The increase in grain size with the soaking time is also evident from this series of micrographs. Note that even for the 4 h cold spot sample (Fig. 2b), the microstructure revealed completion of the recrystalli-

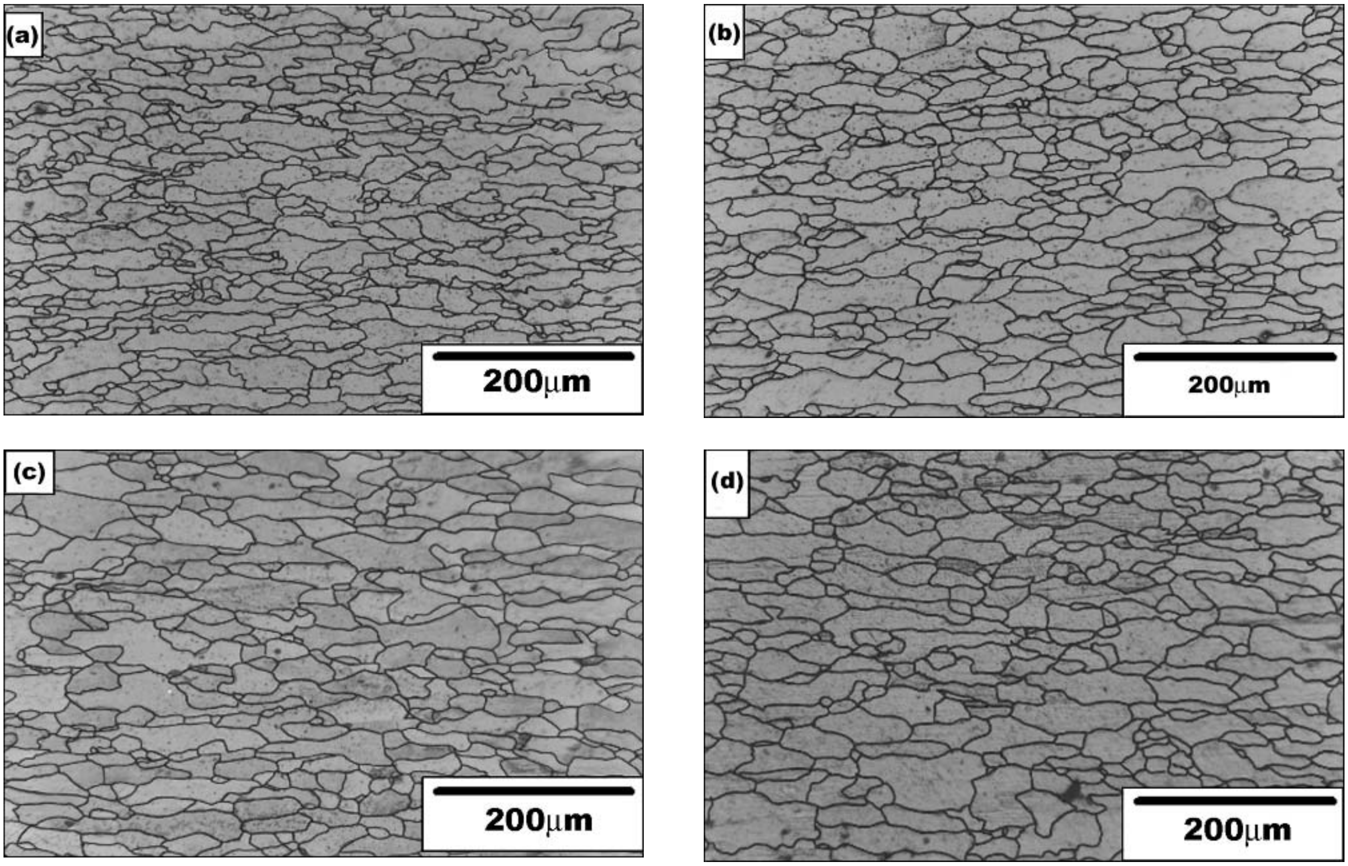

Fig. 3 Evolution of microstructure during annealing of samples corresponding to the hot spot cycle with a soaking time of **(a)** 4 h, **(b)** 7 h, **(c)** 10 h, **(d)** 15 h

zation process. The corresponding outlined micrographs for the hot spot cycles are shown in Fig. 3. The general trends of growth of elongated and oriented recrystallized grains with time, as observed for the cold spot cycles, are also seen here. In addition, it can be readily observed that the micrographs for the cold spot cycles are smaller than the hot spot cycles, which is a direct consequence of more time spent at higher temperature by the hot spot samples. Note also that it is practically impossible to obtain these microstructures from an industrial scale operation without destroying a few batches of coils.

3.3 Grain Growth During Batch Annealing

Quantification of grain size involved analysis of grain size distribution for individual samples and subsequent determination of mean grain size corresponding to the 0.5 cumulative fraction. Figure 4(a) shows the cumulative grain size distributions for different cold spot samples. This figure shows that the grain size distributions are overlapping for the different cold spot samples, indicating low grain growth rate along this profile. This is primarily due to the low temperature along the cold spot profile, which results in lower growth rates (Eq 1 and 2). A similar result is also reflected in the evolution of the mean grain size for the cold spot samples (Fig. 4c). Even though the soaking time is increased from 4-15 h there is a nominal change (14-17 μm) in the mean grain size (Fig. 4c). Note that even for a very low soaking time of 4 h, recrystallization was complete. The higher temperature along the hot spot cycle resulted in significant grain growth, which is reflected in the cumulative size distribution (Fig. 4b) as well as the mean grain size (Fig. 4c). In the case of hot spot samples, the mean grain size increased from 15.5-21.5, when the soaking time increased from 4-15 h. The mean grain size at the end of hot spot and cold spot annealing lie close to the commonly observed grain size range of ASTM 8-9 numbers (~15-25 μm) during the industrial scale batch annealing of AlK grade steel. The good match between these two values confirms that the laboratory experiments simulates closely to the industrial scale batch annealing operation. As the grain growth takes place the grain boundary length per unit area should decrease. This behavior can be readily observed in Fig. 5, where the variation in specific grain boundary length (per unit area) is shown with the soaking time for the hot and cold spot cycles. This is an indirect measure and confirmation of the grain growth during batch annealing process. In addition to the grain growth, the change in specific grain boundary length also includes other microstructural features namely grain roundness, and serration of the grain boundaries, which can change during the grain growth. Therefore, it is not expected that the variation in the mean specific grain boundary length (Fig. 5) will be directly correlated to the variation in mean grain size (Fig. 4c).

The grain size distributions of the various hot and cold spot

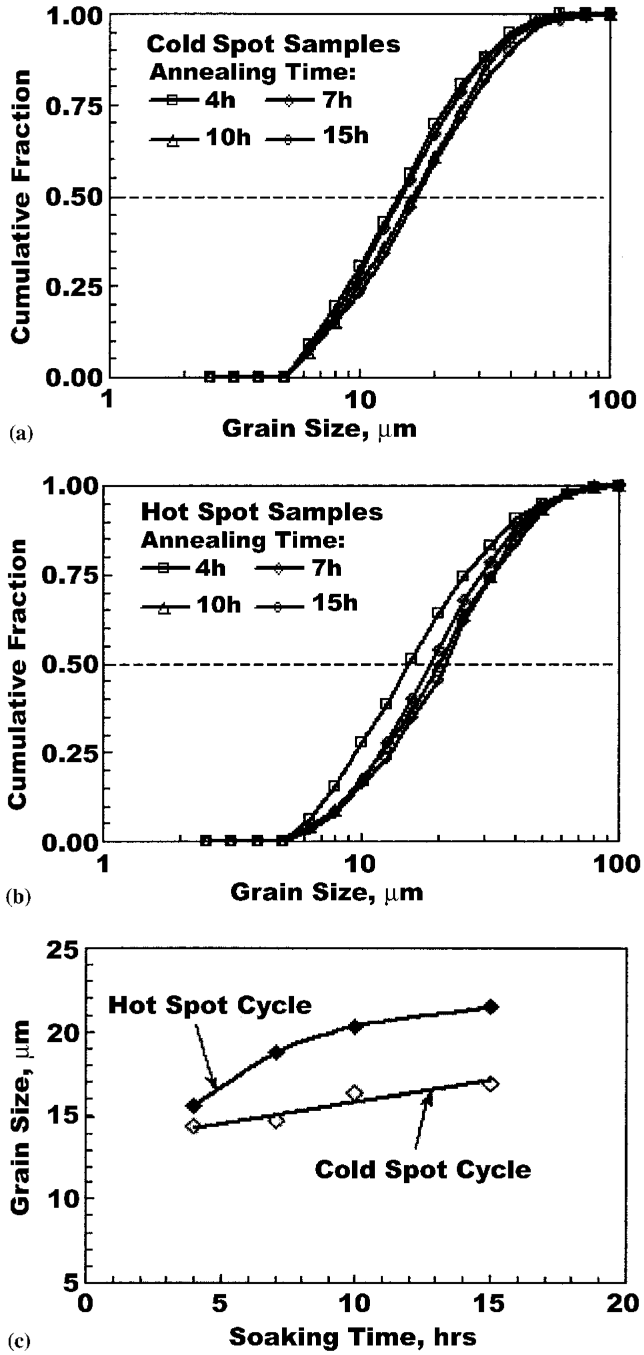

Fig. 4 Evolution of the cumulative grain size distribution along the **(a)** cold spot and **(b)** hot spot profiles; **(c)** variation of mean grain size with soaking time along the cold and hot spot locations.

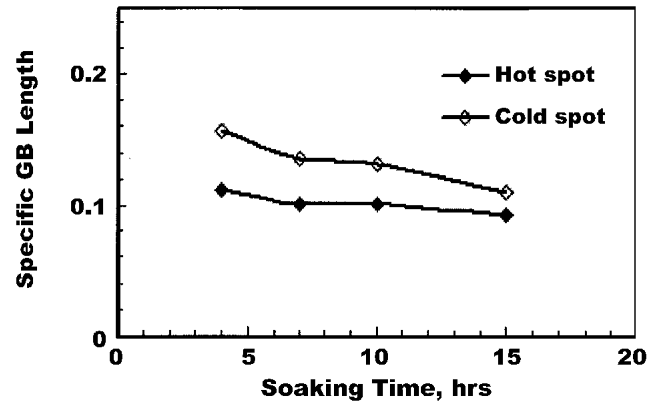

Fig. 5 Variation in specific grain boundary length with soaking time for hot spot and cold spot samples

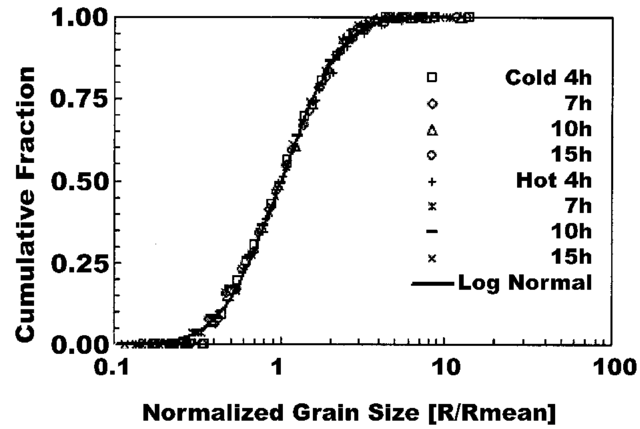

Fig. 6 Invariant nature of the normalized grain size distributions

samples were normalized with respect to their mean grain sizes. The normalized grain size distributions are plotted in Fig. 6. From this figure it is evident that the normalized grain size distributions remain invariant or self-preserving. Furthermore, as shown by the solid line in Fig. 6, the normalized grain size distributions follow a log-normal distribution. Similar invariant nature of the grain size distribution following log-normal distribution was exhibited in a recent work,[11] where samples were heat-treated with different heating rates in the range of 1-10 °C/min. The invariant nature of the normalized grain size distribution and its conformity with a log-normal distribution are in agreement with Feltham's model,[15,16] which is based on topological considerations as well as recent results using stochastic grain growth.[17]

3.4 Grain Shape Anisotropy During Batch Annealing

In the cold rolled annealed steel sheets, in addition to the grain size, grain shape is known to strongly influence the mechanical properties.[11] For example, in the AlK grade steel, pancake shaped grains (high grain shape anisotropy) are highly desirable for improving the desirable {111} texture, normal anisotropy, and drawability of the steel sheets.[14] In the current study, the grain shape anisotropy has been characterized through grain aspect ratio, which is the ratio of the major and minor grain length. Figure 7 shows the variation in mean aspect ratio with soaking time for the hot and cold spot samples. Note that the mean aspect ratio of the hot spot cycle samples remains nearly constant with soaking time. Also, the mean aspect ratios of the cold spot cycle samples are higher than the hot spot cycle samples and it decreases with increase in soaking time. The higher aspect ratio of the cold spot samples (cold spot samples have lower heating rate as compared with hot spot samples) is

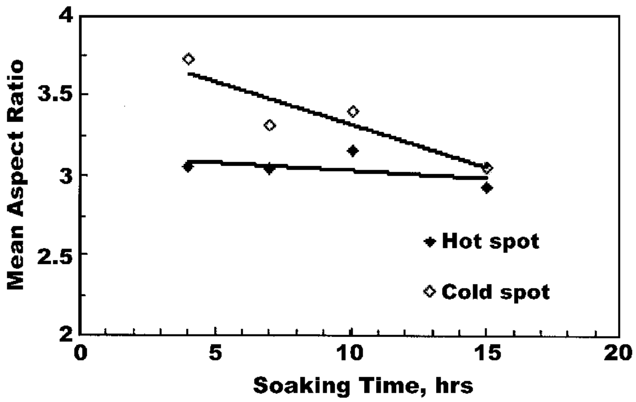

Fig. 7 Variation in grain shape anisotropy (mean aspect ratio) with soaking time for hot spot and cold spot samples

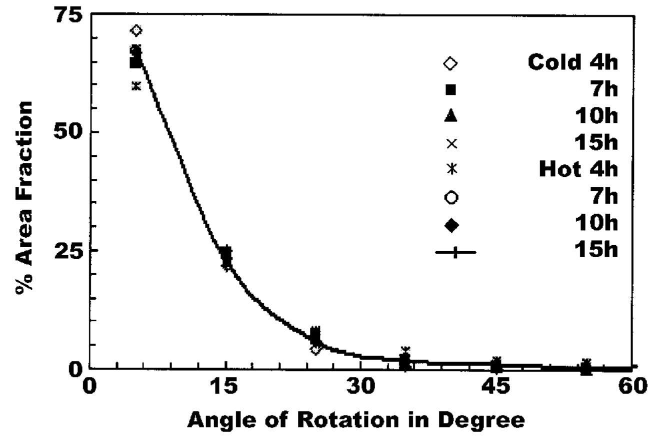

Fig. 8 Invariant nature of the orientation distribution of the major axis of grains

consistent with the recent observation of inverse relationship between heating rate and grain aspect ratio.[11]

These behaviors can also be qualitatively explained on the basis of interacting microstructural phenomenon—such as precipitation, recrystallization and grain growth—during the annealing process.[3-5] The grain shape anisotropy is a manifestation of difference in grain growth rate along the rolling and transverse directions. Due to higher number of grain boundaries along the transverse direction and preferential precipitation along the grain boundaries, the grain growth due to the precipitate coarsening will be more affected in the transverse direction. Therefore, due to the precipitate coarsening, the grain growth in transverse direction will take place at a higher rate than the rolling direction. This difference in grain growth rates along the two directions will result in decrease in the grain aspect ratio. Also, as has been recently[18] shown, the rate of precipitate growth has a stronger temperature dependence (growth rate ~0.2 nm/°C) than the time dependence (0.45 nm/h). In the case of hot spot cycles, the temperature remains constant and 11 h of soaking time will result in ~10% increase in the precipitate size. In contrast, in the case of cold spot cycle, in addition to 11 h of soaking time, the difference in temperature between the first and last sample is over 100 °C. The combined effect of time and temperature effect will result in significant precipitate growth (~60-70%). The large difference (~60-70%) in the precipitate growth between cold and hot spot cycles, explains the observed larger decay in the grain shape in the cold spot samples compared with the hot spot samples.

These results suggest that with decrease in temperature and heating rates, grain shape anisotropy is increased. Furthermore, temperature has more profound effect on grain shape anisotropy than soaking time.

3.5 Invariant Nature of Grain Orientation Distribution

The preferred orientation of the major axis of the grains along the rolling direction can be readily observed from the microstructures shown in Fig. 2-3. This tendency was quantitatively estimated by determining the cumulative fraction of the area as a function of the angular orientation of the major axis (Fig. 8). The distribution of angular orientation of the grains also remains invariant for all the samples, where soaking time, heating rate, and annealing temperatures have been varied. This result suggests that the grain growth progresses while maintaining a constant angular distribution.

3.6 Industrial Implications

The key variables in designing a batch annealing cycle are heating rate, annealing temperature, and soaking time to obtain a coil within specified range of microstructure and mechanical properties. Traditionally, the temperature differential between hot and cold spot locations is taken as a completion criterion for the batch annealing process. Increase in soaking time is expected to decrease the temperature differential as well as variation in mechanical properties and improve the product quality, though it is also associated with an adverse effect on some of the other plant performance parameters such as furnace productivity and specific energy consumption. However, the present work revealed that the recrystallization for the cold spot cycle gets completed even after a short soaking time of 4 h. Moreover a closer examination of Fig. 4(c) suggests that the grain size difference between the hot and cold spot samples was minimum during initial stages (4 h) of soaking time, indicating a possibility of batch annealing termination with advantages of significant productivity improvements as well as reduction in product quality variability. This puts a strong case for complete recrystallization in the cold spot location as a better criterion for batch annealing. In an industrial batch annealing operation, the completion of recrystallization along the cold spot cycle can be monitored via integrated batch annealing furnace simulator.[2] The present work also suggests that increase in soaking temperature and heating rate will increase the productivity, however with an associated loss in grain shape anisotropy (grain aspect ratio) and product quality. Therefore, the annealing cycle should be designed such that the soaking time is within the close limits of specified range of microstructure and mechanical property variation. In addition, the soaking temperature and heating rate should be such that pancake shape grains (high shape anisotropy) are obtained without having significant impact on the furnace productivity. The work carried out in this study provides quantitative estimates of rate of grain growth and grain shape anisotropy decay in the hot and cold spot cycles. These kinetic parameters form the input to the integrated batch-annealing simulator[1,2] from which the optimum soaking temperature and time can be estimated. The optimum soaking temperature and time—where the productivity is maximum and specific energy consumption is minimum, while meeting the specified product quality—can be obtained through such studies.

4. Summary

In the present work, the thermal profiles for the hot and cold spots were obtained from a thermal model, which involved solving the transient energy equation. These thermal profiles were used to heat treat the AlK steel samples in a laboratory furnace using a programmable controller. This methodology enables the simulation of industrial batch annealing cycles in a laboratory furnace. Samples with different soaking times were obtained along the hot and cold spot cycles to study the microstructure evolution during batch annealing operation. A number of microstructural features, such as grain size distribution, grain shape anisotropy, and grain orientation, were examined and correlated to the annealing temperature, soaking time, and heating rates through extensive quantitative microscopy. As expected, due to the thermally activated grain growth process, the grain sizes for the hot spot samples were higher than the cold spot samples and both these grain sizes evolved with time. The grain size distribution for all the hot and cold spot samples, normalized to their median grain size, were found to be invariant and self-preserving following the log-normal distribution. The grain shape anisotropy increased with decrease in annealing temperature as well as heating rate. The decay in grain shape anisotropy with soaking time was more pronounced for the non-isothermal cold spot samples compared with the isothermal hot spot samples. This behavior has been explained on the basis of temperature rise and interaction between precipitate coarsening and grain growth. The angular distribution of the grain orientation was found to remain invariant for all the samples with a wide range of heating rate, annealing temperature, and soaking time. Finally, the industrial implications of this work include completion of recrystallization along the cold spot location as a better cycle termination criterion for the industrial batch annealing operation.

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