Heating Rate Effects During Non-Isothermal Annealing of AlK Steel

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The effects of heating rate on microstructural size and shape parameters during annealing of cold rolled aluminum killed steel strips have been examined under non-isothermal condition. It is shown that decrease in the heating rate results in accelerated grain growth behavior compared with the prediction by quasi-isothermal based kinetics. The {111} and {112} crystallographic orientations, which enhance the normal anisotropy and deep drawability of cold rolled annealed sheets, are found to exhibit a strong correlation with the grain shape anisotropy. This grain shape anisotropy itself is strongly dependent on heating rates. Lower heating rates result in higher aspect ratios and thus better drawability of the cold rolled sheets. A Hall-Petch type relationship is observed between grain size and hardness of the annealed samples.

Keywords annealing, grain growth, kinetics, microstructure, theory and modeling

1. Introduction

Control of microstructure during annealing of cold rolled steel sheets is important for critical applications such as automotive components and panels for white goods. Microstructural evolution during annealing is an outcome of a complex interplay of a number of phase transformation processes, namely precipitation, recrystallization, and grain growth.[1-5] The kinetics of these thermally activated phenomena are strongly dependent on the thermal profile as characterized by heating rate, annealing temperature, soaking time, and cooling rate. These heating parameters control the microstructural features such as grain size distribution, shape, and texture, which in turn determine the mechanical properties (such as strength, hardness, and drawability) that are crucial for the end applications. The influence of thermal profile on the microstructural size and shape parameters, texture, and mechanical properties is schematically shown in Fig. 1. Of the various components shown in the figure, the effect of heating rate on microstructural size and shape parameters as well as the relationship between grain shape and crystallographic orientation has not been extensively explored.

The kinetics of precipitation, recrystallization, and grain growth under isothermal conditions has been extensively investigated in the past.[6-9] For example, grain growth kinetics is conventionally represented by the Beck-type correlations:[5,9]

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

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

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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 for describing grain growth over non-isothermal temperature profiles by segmenting the annealing cycles into small isotherms[10-13] 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). One of the objectives of the present work is to examine the validity of the quasi-isothermal approach under non-isothermal annealing conditions.

Influence of heating rate on the normal anisotropy, which is a measure of deep drawability of steel sheets, has been experimentally studied[1] for different types of steels. Also, an empirical model—based on precipitation and recrystallization kinetics considerations—for determining the optimum heating rate during annealing of steel is available. However, the role of grain shape anisotropy and its quantitative relationship with crystallographic orientations and normal anisotropy are not well established.

The scope of the present work broadly covers all the components as well as relationships schematically shown in Fig. 1. Specifically, this work endeavors to examine the effect of heating rate on grain growth and grain shape of AlK grade cold rolled steels through detailed quantitative microscopy. Furthermore, the relationships between grain shape and crystallographic orientation as well as grain size and superficial hardness have been examined.

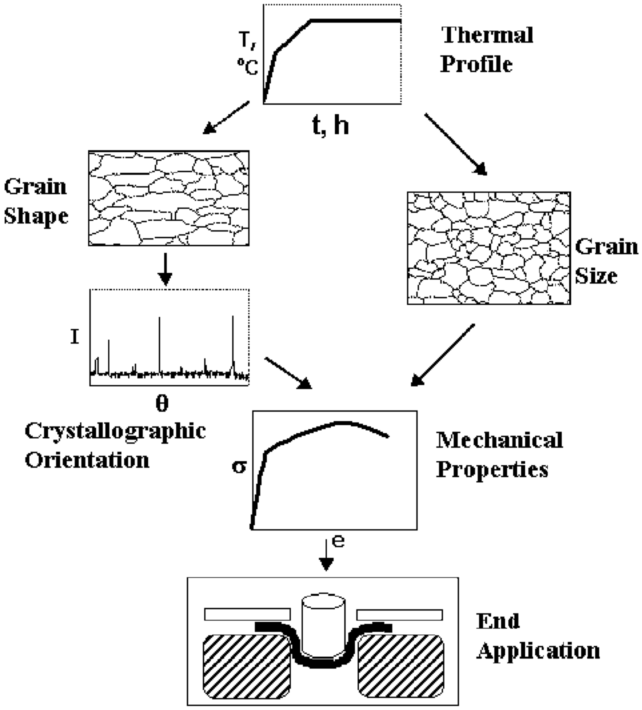

Fig. 1 Flow diagram illustrating the influence of thermal profile on mechanical properties and usability of structural sheet materials

2. Experimentation

The starting material used in this study was 70% cold rolled 1.0 mm thick aluminum killed (AlK) grade steel sheet, with a composition of 0.05% C, 0.05% Al, and 45 ppm N. Samples cut from the cold rolled sheet were heat-treated in a programmable laboratory furnace having a multisegment PID controller. Metallographic preparation was done on the longitudinal and transverse sections of the heat-treated samples before examining them under an optical microscope. For each sample, eight digital micrographs were taken along the longitudinal direction, which gave a statistically significant number of over 2500 grains per sample. The large number of grains resulted in a reasonably precise measurement of mean grain size with an accuracy (95% confidence level) of ±0.3 μm. Subsequent to the grain boundary enhancement, image analysis (Scion Image Software, Scion Corp., Frederick, MD) was performed on the micrographs to quantify the area, perimeter, and major and minor lengths of 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 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. To measure the hardness of the annealed samples, the scales on the surface of the samples were cleaned by pickling with 10% HCl solution. The superficial hardness was measured on the "T" scale using a 15 Kgf major load. X-ray diffraction (XRD) was carried out on polished 10 mm × 10 mm samples in the 2θ range of 30-150 degrees at a scan speed of 4 degrees/min. The raw intensity data were smoothed using a 5-point moving average algorithm and the areas under all the major peaks were obtained by numerical integration.

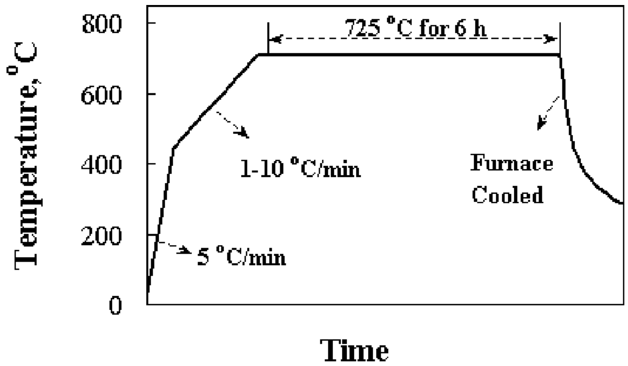

Fig. 2 Schematic of the annealing cycles used in this study

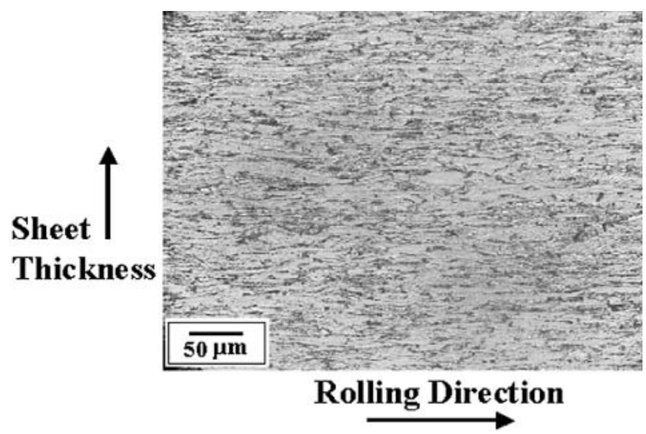

Fig. 3 Microstructure of the cold rolled steel sample

Figure 2 shows the four segments of the heat treatment cycle used in this work. In the first segment, the samples were preheated to 450 °C at a heating rate of 5 °C/min. No relevant solid-state transformation is expected to take place in this segment.[14] The second segment involved heating from 450-725 °C. In this segment, the effect of heating rate on the grain growth and grain shape was determined by varying the heating rates in the range of 1-10 °C/min. In the third segment, isothermal annealing was carried out at 725 °C for 6 h, followed by the fourth segment, where the samples were furnace cooled from 725 °C to room temperature. Note that except for the second segment, the remaining three segments were kept identical for all the samples.

3. Results and Discussion

3.1 Microstructural Observations

The microstructure of starting material (~70% cold rolled AlK grade steel) taken along the longitudinal section of the rolled sheet is shown in Fig. 3. In this microstructure, elongated

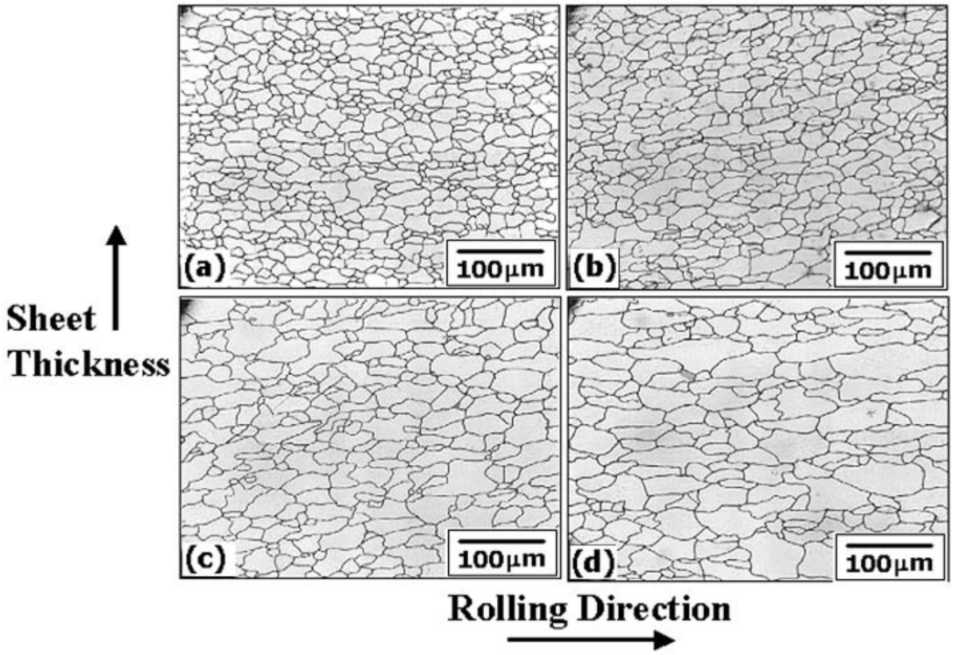

Fig. 4 Microstructures of samples annealed at 725 °C for 6 h with heating rates of **(a)** 10 °C/min, **(b)** 5 °C/min, **(c)** 3 °C/min, and **(d)** 1 °C/min

and highly deformed grains, characteristic of the large amount of deformation given during cold rolling, are discernible. The effect of heating rate on the microstructure along the longitudinal section is shown in Fig. 4(a-d). In these samples, heating rates were varied between 1-10 °C/min and the samples were annealed at 725 °C for 6 h followed by furnace cooling. It can be seen that the grains are elongated and oriented along the rolling direction in all the microstructures. Such microstructures are known to promote the desirable {111} and {112} textures in the steel.[15] It can be readily observed from this series of micrographs that the grain size increases with decrease in the heating rate. For example, grain size of the sample heated at 10 °C/min shown in Fig. 4(a) is significantly smaller than the sample heated at 1 °C/min shown in Fig. 4(d). In addition, increase in grain aspect ratio with decrease in heating rate can also be perceived. Quantitative results on the grain size and aspect ratio are presented in the subsequent sections.

3.2 Accelerated Grain Growth Behavior Due to Reduction in Heating Rate

The cumulative grain size distributions for samples with different heating rates are presented in Fig. 5(a). The increase in grain size with decrease in heating rate observed in the micrographs (Fig. 4) is further confirmed from the shift in cumulative grain size distribution plots (Fig. 5a) towards larger grain size. Quantitative analysis of the cumulative size distribution plots shown in Fig. 5(b) indicates that the mean grain size increases by 67%, when the heating rate is decreased from 10 °C/min to 1 °C/min. The following paragraph evaluates the ability of the quasi-isothermal approach to model such large variations in grain size with heating rates.

When the heating rate is decreased from 10 °C/min to 1 °C/min, the time to reach annealing temperature (725 °C) in-

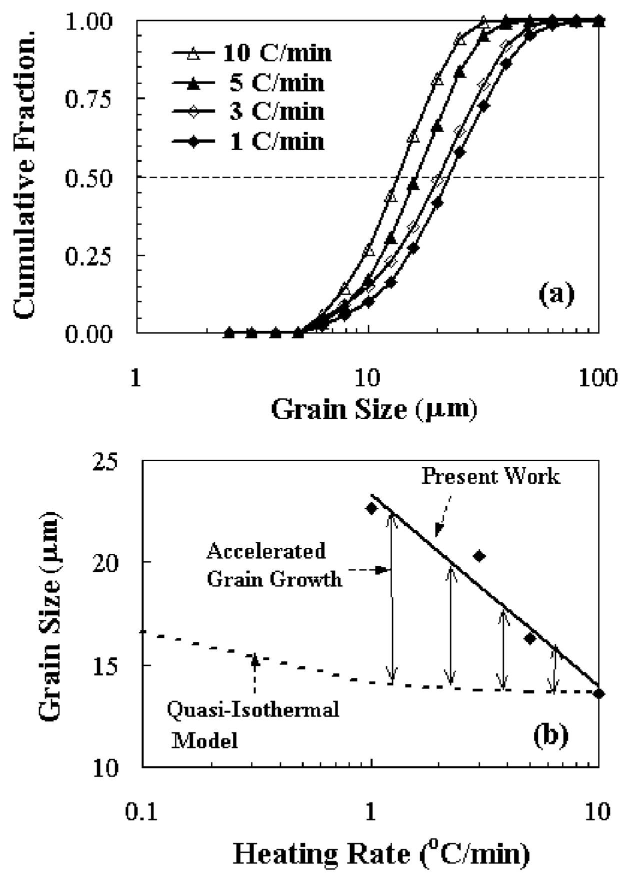

Fig. 5 **(a)** Variation of cumulative grain size distribution with heating rate, and **(b)** accelerated grain growth behavior observed during annealing of AlK steel sheets

creases. However, in the AlK grade steel used in the current study, recrystallization is complete and the grain growth starts around a temperature of 575 °C, which continues in the remaining portion of the second segment as well as in the third segment of isothermal annealing. Consequently, the effective duration of the grain growth in the second segment increases from 25-250 min (time to heat up from 575-725 °C in the grain growth regimen) when the heating rate is decreased from 10-1 °C/min. Considering the quasi-isothermal grain growth kinetics—by segmenting the ramp portion of the curve in Fig. 2 as small isothermal steps and integrating Eq 1 and 2 over time—the variation in grain size with heating rate has been computed[16,17] for the entire cycle. For the quasi-isothermal model, the activation energy for grain growth was taken as 230 KJ/mole, which is close to the value reported in Ref. 5 as well as that obtained in previous studies[17] conducted on the same cold rolled material. The result from this quasi-isothermal model is plotted as a broken line in Fig. 5(b). The model predictions show an insignificant increase in grain size when the heating rate is decreased from 10-1 °C/min. In the quasi-isothermal model, the effect of heating rate can arise due to increase in temperature or time. When the heating rate is decreased from 10-1 °C/min, although the time in grain growth regimen during the second segment increases from 25-250 min, it still remains lower than the subsequent isothermal annealing time of 6 h. Moreover, irrespective of the variation in heating rate, for all the samples the equivalent isothermal temperature[1] of the heating segment remains at 670 °C, which is significantly lower than the subsequent isothermal annealing temperature of 725 °C. This should result in dominance of the isothermal segment over the heating segment, which is reflected by nearly constant grain size prediction by quasi-isothermal model. However, as can be observed in Fig. 5(b), the experimental results indicate far higher grain sizes at lower heating rates compared with the predictions of the quasi-isothermal model. It is known that in the low carbon steel,[9] increasing the heating rate retards the recrystallization kinetics by affecting the amount of carbon in the solid solution as well as due to the interaction with AlN precipitation. However, the observed acceleration in grain growth (Fig. 5b) with decrease in heating rate is high and cannot be explained by the recrystallization retardation. Therefore, it can be concluded that the increase in grain size with decrease in heating rate observed in the current study cannot be described or captured by quasi-isothermal kinetics and there is acceleration in grain growth kinetics.

Furthermore, it may be observed that as the heating rate is decreased, the discrepancy between quasi-isothermal predictions and experimental results increases. In the non-isothermal heating regimen, the grain growth kinetics is accelerated to a greater extent compared with those predicted by the quasi-isothermal kinetics. Similar behavior was observed in 1100 grade aluminum sheets annealed at 360 and 480 °C,[18] where it was noted that the rate of heating to the annealing temperature considerably affects the grain size of aluminum alloys. It was also shown that the slower heating rate results in much larger grain size in aluminum alloys compared with rapid heating, which is in agreement with the present work on AlK steel. These observations suggest that there is a strong non-isothermal effect on the grain growth behavior. Similar non-isothermal effects and reduction in apparent activation energy under non-isothermal conditions have also been recently reported for several other phase transformations. For example, the activation energy for crystallization of the amorphous $Pd_{40}Cu_{30}P_{20}Ni_{10}$ was found[19] to decrease from 336 KJ · mol^{-1} under isothermal condition to 258 KJ · mol^{-1} under isochronal condition. In another case, the activation energy for reduction of Fe_2MoO_4 by hydrogen gas was reported[20] to be reduced from 173.5 KJ · mol^{-1} under isothermal condition to 158.3 KJ · mol^{-1} under isochronal condition. Similar reduction in activation energies and the resultant acceleration in phase transformation kinetics were observed during non-isothermal cyclic grain growth in aluminum-killed steel,[21] cyclic sintering of zinc powders,[22] and cyclic hardening of Ti-6Al-4V alloys.[23]

Note that most industrial annealing cycles are non-isothermal and their design involves selection of heating rate, annealing temperature, and soaking time. During the cycle design, heating rates are generally perceived to reduce the furnace productivity and therefore the highest possible heating rates—generally constrained by the thermal inertia of the components and sometimes by product quality (e.g., deep drawability in AlK steel sheets)—are selected. The present work indicates that it may be possible to reduce the heating rate and yet enhance the furnace productivity due to accelerated grain growth kinetics and the reduced isothermal annealing time. In effect, although reduction in heating rate will increase the heating segment time, this may be compensated by a reduction in isothermal annealing time, along with enhancement of product quality (e.g., anisotropy and deep drawability). The present work also highlights the limitation of using the kinetics coefficients in quasi-isothermal kinetic models that are determined under isothermal conditions.

3.3 Invariant Nature of the Grain Size Distribution

The grain size distributions of the samples annealed with different heating rates, described in the previous section, 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 lognormal distribution. The invariant nature of the normalized grain size distribution and its conformity with a lognormal distribution are in agreement with Feltham's model,[24,25] which is based on topological considerations as well as recent results using stochastic grain growth.[26]

3.4 Grain Size-Hardness Relationship

Superficial hardness value is one of the important and readily measurable quality parameters for cold rolled annealed steel sheets. Superficial hardness measurements were carried out on all the samples annealed with different heating rates. The microstructures shown in Fig. 4 indicate that all these samples are in the grain growth regimen subsequent to the

[1]The equivalent isothermal temperature is given by: [575 °C + 0.63*(725-575)] = 670 °C. The factor of 0.63 arises due to the exponential dependence on temperature 1− (e^{-1}) = 0.630.

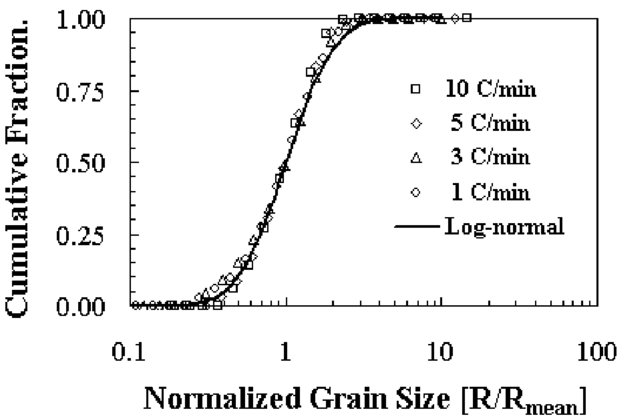

Fig. 6 Invariant nature of the normalized grain size distributions

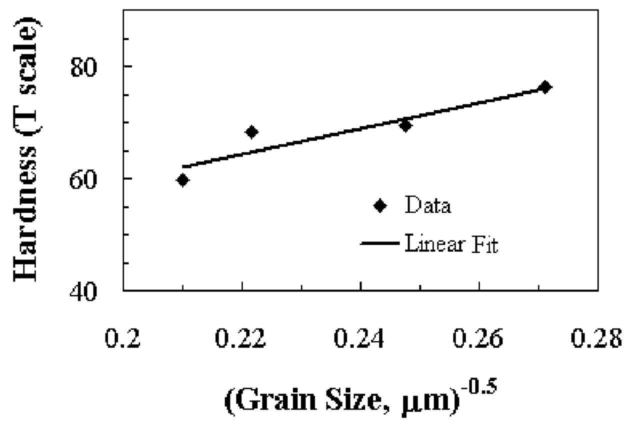

Fig. 7 Hall-Petch type relationship between grain size and superficial hardness

completion of recrystallization. Therefore, variation in hardness values among various samples will be primarily due to the variation in grain sizes. At low temperatures, the grain boundaries act as obstacles to dislocation motion and provide strengthening through stress concentrations due to pileup of dislocations at the grain boundaries.[27] The effect of grain size on yield strength is given by the widely invoked Hall-Petch relationship[28]:

$$\sigma_{YS} = \sigma_o + \frac{k_{hp}}{\sqrt{d}} \quad \text{(Eq 3)}$$

where σ_{YS} is the yield stress, σ_o is the frictional stress required to move dislocations, k_{hp} is the Hall-Petch slope, and d is the grain size. Analogous to yield strength, the hardness values are expected to be correlated to the grain size. The inverse of the square root of mean grain size is plotted against hardness in Fig. 7. As expected, for the samples with different heating rates, a good linear correlation is exhibited, suggesting that the Hall-Petch type mechanism can explain the decrease in hardness with decrease in heating rate.

3.5 Influence of Heating Rate on Grain Shape Anisotropy

Although it is known that decrease in heating rate results in pancaking of the microstructure, a direct quantitative relationship between the effects of heating rate on the grain aspect ratio in AlK steel has not been demonstrated. In the present work, grain aspect ratio was measured in the samples annealed with different thermal profiles, described by temperature, time, and heating rate variations.

The effect of temperature and time on the grain shape anisotropy was recently studied[17] on the same AlK grade steel as the current study, under conditions of industrial batch annealing of steel coils. The temperature profiles in the earlier study corresponded to the hot spot (coil surface) and cold spot (coil core) locations of the steel coils. In the hot spot cycle, the samples were heated to 710 °C at the rate of 43 °C/h and given isothermal annealing at 710 °C for 4-15 h. In the cold spot cycle, the samples were heated to 610 °C at the rate of 35 °C/h followed by a slower heating rate of 10 °C/h. The annealing time for cold spot samples was the same as those of the hot spot

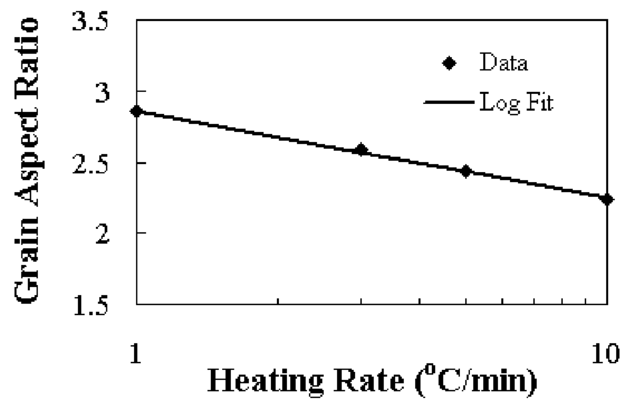

Fig. 8 Effect of heating rate on grain shape anisotropy

samples. The grain aspect ratio of the hot spot samples remained nearly constant at 3.0, whereas in the cold spot samples the grain aspect ratio decreased from 3.72-3.1, when the annealing time was increased from 4-15 h. These observations were explained on the basis of preferential precipitation along the transverse direction and precipitate coarsening limiting the grain growth. The rate of precipitate coarsening has a stronger temperature dependence (growth rate 0.2 nm/°C) than the time dependence (0.45 nm/hr).[8] The isothermal condition of the hot spot samples resulted in small increase (10%) in precipitate size and hence insignificant variation in grain aspect ratio (<5% change), when the annealing time was increased from 4-15 h. In contrast, in the case of the cold spot cycle where in addition to annealing time there was significant increase in temperature (150 °C), considerable precipitate coarsening (60-70%) took place that explains the observed large decrease (22%) in grain aspect ratio. In summary, the changes in grains shape anisotropy were attributed to the precipitate coarsening arising from the change in temperature, whereas the soaking time was found to have insignificant effect.

The variation in grain aspect ratio with heating rate is plotted in Fig. 8, which indicates a strong correlation between heating rate and mean aspect ratio; the grain aspect ratio increases when the heating rate is decreased. For example, the grain aspect ratio increases by 28% when the heating rate was

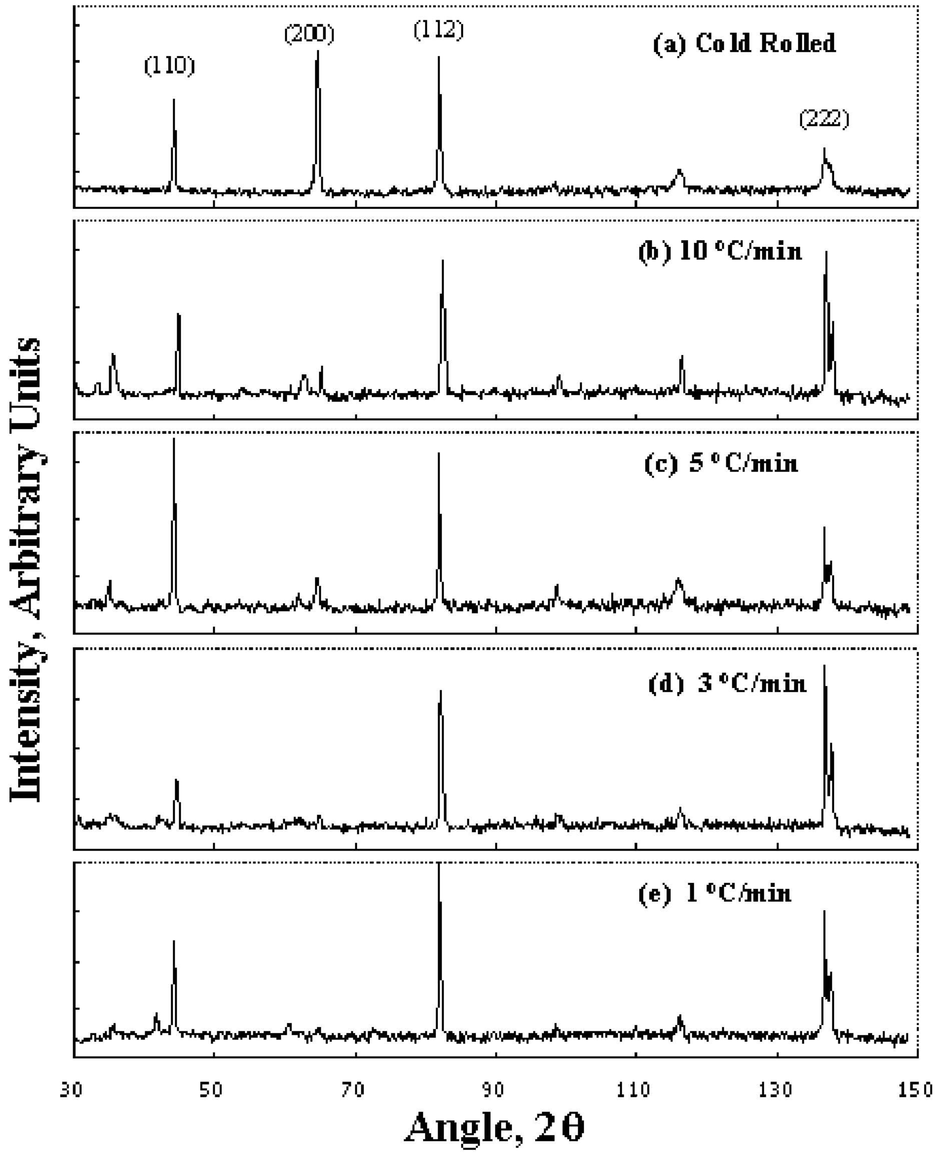

Fig. 9 X-ray diffractograms of **(a)** cold rolled sample and other samples annealed at 725 °C for 6 h with heating rates of **(b)** 10 °C/min, **(c)** 5 °C/min, **(d)** 3 °C/min, and **(e)** 1 °C/min

decreased from 10-1 °C/min. The observed variation in grain shape anisotropy for different samples can arise either at the recrystallization stage (heating rate affecting grain shape anisotropy of the nuclei) and/or during the grain growth stage (annealing temperature and/or soaking time affecting the grain shape anisotropy). In the current study, the equivalent isothermal temperatures in the second segment being equal (670 °C) for all the samples, the observed variation in grain shape anisotropy among various samples can arise due to the differences in heating rates and soaking times in the second segment. Furthermore, with insignificant effect of soaking time, only the heating rate is expected to influence the grain shape anisotropy. It can be thus concluded that the decrease in heating rate results in an increase in grain shape anisotropy of the recrystallized microstructure, and the difference in the grain shape anisotropy of samples is maintained during the subsequent heating (second) and isothermal annealing (third) segments.

3.6 Grain Shape-Crystallographic Orientation Relationship

In the cold rolled and annealed sheet, preferred crystallographic orientations, i.e., {111}, {112} are desired to promote normal anisotropy,[1,15,29] which in turn enhances the deep drawability. To examine the effect of heating rate on crystallographic orientation, XRD studies were carried out on the samples heated at rates of 1-10 °C/min to the annealing temperature of 725 °C and soaked for 6 h. The diffractograms for these samples along with that for the cold rolled sample are presented in Fig. 9 where various intensity peaks have been identified and indexed.

As seen in the Figure 9, in the cold rolled steel sample, the (200) peak is strongest, followed by (112) and (110). The other peaks, such as (222), are very weak. The (200) peak is undesirable as it is associated with very low normal anisotropy. High value of normal anisotropy is desirable as it results in deep drawability, whereas low value of planar anisotropy is desired to prevent a tearing defect during deep drawing. For the annealed steels the preferred orientations are (222) and (112), whereas (200) and (110) are the undesirable crystallographic orientations.[1,15,29] Note that in the annealed samples, as the heating rate is decreased from 10-1 °C/min, intensities of (200) and (110) peaks decrease, whereas intensities of (112) and (222) peaks increase. Since these trends are favorable for deep drawing applications, it can be concluded that decreasing the heating rate results in a desirable crystallographic orientation for increasing the deep drawability of steels.

Figure 10 shows the variation in intensities of (222) and (112) peaks as a function of grain aspect ratio. The intensity of these peaks has been normalized with respect to the (200) peak. Such normalization is helpful in comparing the intensities of different samples. The strong dependence of intensities of the desirable (222) and (112) peaks on the grain shape anisotropy is evident from this figure. For example, when the heating rate is decreased from 10-1 °C/min, the grain shape anisotropy increases by 28% which results in an over three-fold increase in the normalized intensity of the (222) peak, from 7-22. Similarly, in the case of the (112) peak, there is an over four-fold increase from 5-23, when the heating rate is decreased from 10-1 °C/min. Although, increase in normal anisotropy by decrease in heating rate is experimentally well established,[26] the present work relates this effect to the grain shape anisotropy. It indicates the important role of grain shape anisotropy in developing better deep drawing characteristics in steel.

4. Conclusions

Most of the industrial annealing cycles are non-isothermal in nature, whereas the grain growth equations and the kinetics coefficients are obtained under isothermal conditions. Generally, the non-isothermal industrial cycles are described by the quasi-isothermal grain growth kinetics with the help of the additivity principle and with an assumption that the kinetics of grain growth does not exhibit any special non-isothermal effects. In the present work, the effect of reduction in heating rate was observed to show acceleration in grain growth kinetics

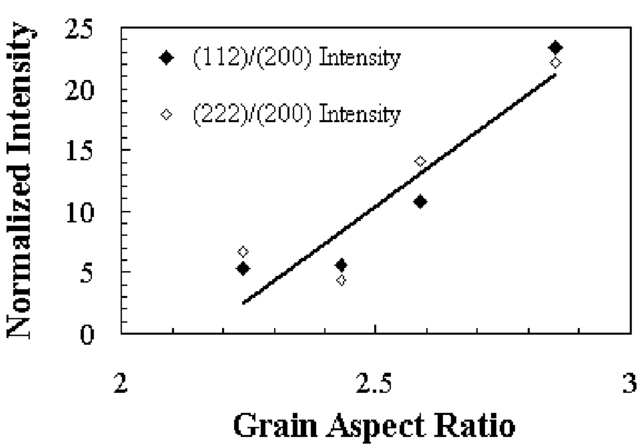

Fig. 10 Effect of grain shape anisotropy on normalized intensities of crystallographic orientations

attributable to non-isothermal effects and reduction in activation energy, thereby rendering the quasi-isothermal model seriously deficient in describing the grain growth kinetics. The mean grain size of the annealed sheets exhibited a Hall-Petch type relationship. The reduction in heating rate increases the grain shape anisotropy of the recrystallized microstructure. Furthermore, a strong correlation was observed between the grain aspect ratio and the {111} and {112} crystallographic orientations, which are known to enhance the normal anisotropy and deep drawability of cold rolled annealed sheets. The implications of these results for accelerated grain growth kinetics for productivity enhancement and quality improvement have been highlighted.

Acknowledgments

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