Microstructure and Texture Formation in High Strength Cold Rolled and Annealed Sheet and Their Correlation With Formability Property

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A comprehensive understanding was developed on the evolution of microstructure and texture during annealing of Nb bearing microalloyed drawing quality high strength cold rolled (HSCR) sheet with specific reference to cold rolling and annealing practices. Further, this study aimed at establishing the best combination of these processing parameters to achieve an $\bar{r}$ value of 1.4 min. For a hot rolled microstructure of moderately coarse ferrite grain of size 16 μm, it was observed that 70% cold reduction guaranteed high intensity of (222) component accompanied with minimum intensity of (200) component. Further, investigation was carried out to understand the influence of annealing time on recrystallization behavior and texture development during intermediate annealing of 60% and 70% cold reduced specimens at 550 °C. It was found that recrystallization ceased after 12 h of intermediate annealing at 550 °C. The textures and microstructures produced during final annealing of 70% cold rolled specimens at various temperatures like 670, 690, 710, and 730 °C with varied duration of soaking (12-18 h) were critically examined. An $\bar{r}$ value of ~1.5 was achieved in HSCR microalloyed steel when 70% cold rolled specimens were intermediately annealed at 550 °C for 12 h followed by final annealing at 710 °C for 12 h.

Keywords high strength cold rolled (HSCR) steel, intensity of (222)/(200) components, intermediate annealing, pancake grains, $\bar{r}$ value, recrystallization

1. Introduction

High strength cold rolled (HSCR) steels are low-carbon microalloyed aluminum killed steels with increased yield strength (YS: 260 MPa min) and moderate forming capacity ($\bar{r}$: 1.2 min). This category of steels, owing to their higher strength in comparison to conventional Extra Deep Drawing (EDD) quality steel, save substantial amounts of material through application of thinner gauge sheets. These steels are now widely used for fabrication of specific automobile components[1] and of lighter structures[2] where dent resistance and rigidity are important design criteria to be fulfilled.

Enhancement of strength level in HSCR steels is brought about by deploying various strengthening mechanisms such as solid solutioning, grain refinement, and precipitation hardening, depending on the desired level of yield strength to be achieved. However, increase in strength level will pose problem in maintaining drawability properties at a moderate level. Therefore, an optimum balance in both strength and formability properties can be achieved through judicious control of steel chemistry and various processing parameters like hot and cold rolling and annealing practices. Since the early 1980s, development of such steels has been amply reported in the literature.[1-9] The scope of this investigation has, therefore, been designed to answer following unexplored areas:

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- Influence of cold reduction and annealing regimen (time and temperature) on the microstructure and texture development.
- Correlation between drawability property and texture.

The outcome of this study will ultimately help in determining the optimum percentage cold reduction and annealing cycle for the selected microalloyed chemistry and hot strip processing to produce superior formable quality HSCR-annealed sheet with $\bar{r}$ above 1.4 with YS in the range of ~260 MPa.

2. Experimental Work

The experimental material was collected from commercially produced hot rolled coil of 3.8 mm thickness. The composition of the steel (wt.%) was as follows (Table 1).

The typical finishing and coiling temperatures maintained during hot rolling were 860 °C and 610 °C, respectively.

The hot rolled samples were first cut into rectangular blanks of suitable size and pickled in 50% conc. HCl, prior to cold rolling in a 4 high (HI) experimental rolling mill. These blanks were subjected to varied amounts of cold reductions (e.g., 50%, 60%, 70%, and 80%) through a number of passes, while minimum reduction in each pass was maintained at 20% for ensuring homogeneous deformation. Subsequently, test specimens were prepared from these cold rolled sheets for detailed metallurgical investigation. The texture study was carried out in a

Table 1 Composition of Experimental Material

C	Mn	P	S	Si	Al	Nb	N
0.05	0.40	0.013	0.014	0.017	0.044	0.030	0.0051

Kristalloflex (Siemens Model no. XRD500, Germany) 800 x-ray diffractometer using Mo-Kα radiation for assessment of integrated x-ray diffraction (XRD) intensity of (200) and (222) components in the rolling plane. To study the recrystallization behavior during annealing, the 60% and 70% cold rolled specimens were first annealed at an intermediate temperature of 550 °C for durations of 3, 6, 9, 12, 15, and 18 h followed by air cooling. Test specimens from these sheets were prepared and x-ray diffraction study was carried out. Based on the analysis of these test results, a final annealing scheme was worked out for 70% cold rolled specimens, which were intermediately annealed at 550 °C for 12 h The final annealing was carried out at 670, 690, 710, and 730 °C for durations of 12, 15, and 18 h in a muffle furnace (Fig. 1). To prevent oxidation of specimens during annealing aluminium based anti-oxidation seizure fluid was used as coating material. The texture study was again carried out for assessment of integrated XRD intensity of (200) and (222) components in the rolling plane. Further, microstructural evaluation using optical techniques was carried out and $\bar{r}$ values were measured by using module r. For transmission electron microscopy (TEM), foils (3 mm disc) were cut from 1 mm thick specimens prepared from 3.8 mm thick hot rolled sheets. Each disk was then jet thinned using a twin jet polisher with a solution of 5% perchloric acid in methanol at 0 °C. A JEOL (Model no. 4000EX, Japan) transmission electron microscope was used for identification of NbC precipitates.

3. Results and Discussions

3.1 Hot Rolling

The optical microscopy of hot rolled specimen showed that the microstructure comprised equiaxed ferrite with average grain size ~16 μm. Evolution of such grains had taken place because the finish rolling temperature was maintained above Ac_3 and coiling temperature was close to 600 °C so as to facilitate suppression of AlN precipitates.[10] Higher finish rolling temperature favored grain coarsening, which, however, was restricted by the presence of fine, 18 nm NbC precipitates (identified in TEM) (Fig. 2). Note that finer ferrite grain size in hot rolled structure is desirable for developing (222) texture in cold rolled and annealed coils and therefore, formation of such moderately coarse ferrite grains in hot rolled HSCR coil was expected to favor the evolution of desired texture.

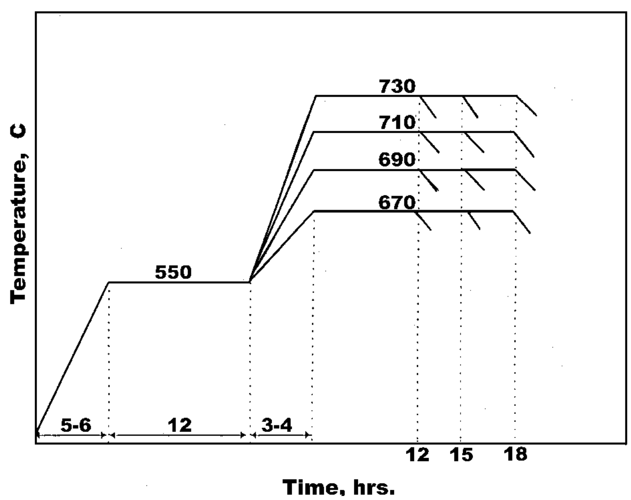

Fig. 1 Schematic presentation of annealing scheme adopted for present investigation

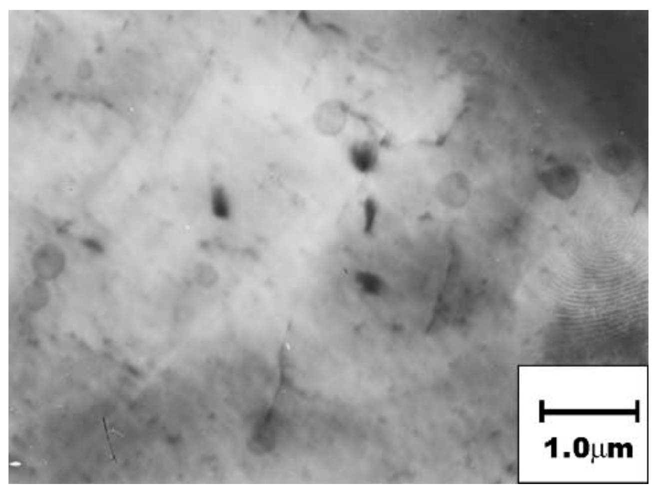

Fig. 2 TEM micrograph of hot rolled HSCR steel showing NbC precipitates (18 nm)

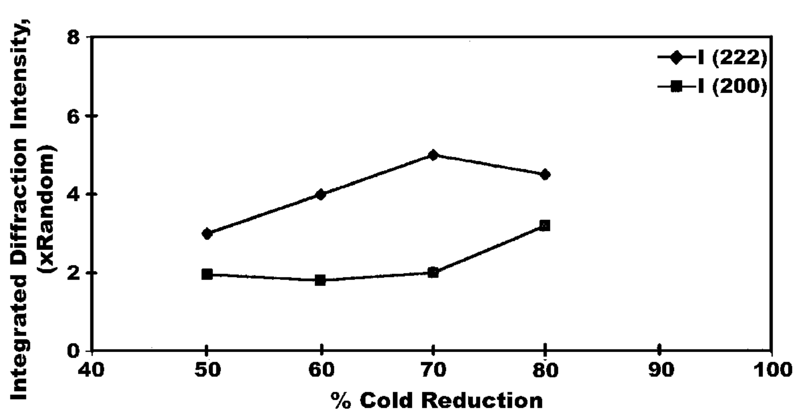

Fig. 3 Variation in integrated diffraction intensity of (222) and (200) components with cold reduction

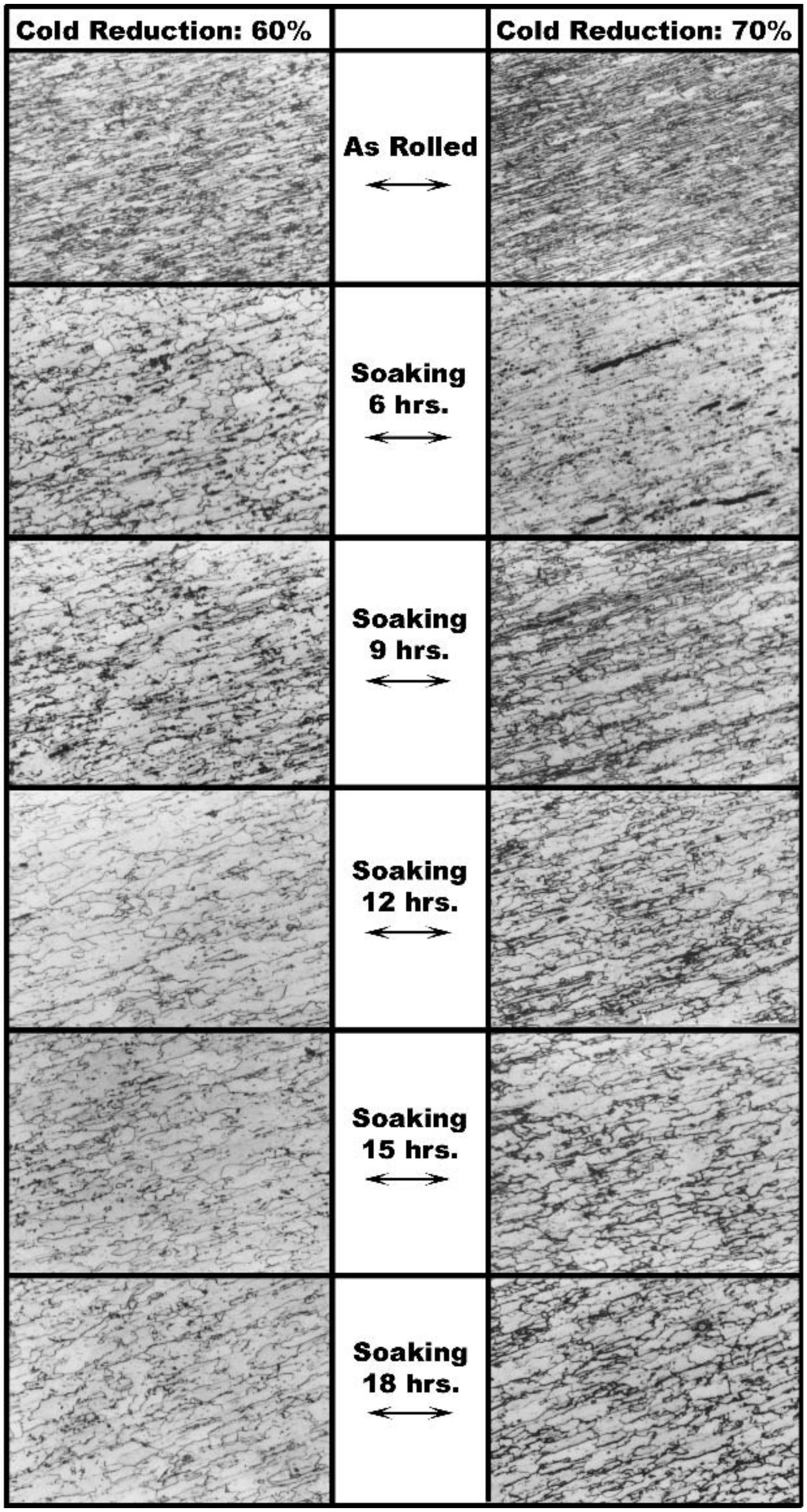

Fig. 4 Sequence of microstructural change with increasing annealing time at 550 °C for specimens with 60% and 70% cold reductions (X200)

3.2 Cold Rolling

Figure 3 shows that the intensity of (222) component increased linearly up to 70% cold reduction. Further increase in cold reduction lowered the intensity of (222) component. This observation contrasts with the evolution of texture in cold rolled Al-killed EDD steel where the (111) component keeps increasing up to 90% cold reduction.[11] Further, the (200) intensity was found to remain constant up to 70% cold reduction and it increased sharply with a further increase in cold reduction. Based on these findings, it is suggested that 60-70% cold reduction should be considered as desirable range for achieving suitable texture after cold rolling.

3.3 Intermediate Annealing

Examination of microstructures produced after intermediate annealing revealed that recrystallization ceased after 12 h of soaking in both 60% and 70% cold rolled specimens (Fig. 4). Explanation in support of such observation has been drawn from the time-temperature-transformation (TTT) diagram of AlN formation and concurrent recrystallization of Al-killed low carbon steel.[11] In the current study, it seems that intermediate annealing had caused the formation of small fraction of recrystallized ferrite grains followed by precipitation of AlN, which must have started after 12 h of annealing and, therefore, recrystallization discontinued as expected from the TTT diagram of AlN formation. The limited recrystallization of cold rolled structure combined with the precipitation of AlN had influenced the texture that evolved in intermediately annealed specimens. It was found that the integrated intensity of (222) component initially increased with annealing time and reached its maximum value after 9 h and 12 h of soaking for 60% and 70% cold reduced specimens, respectively (Fig. 5-6). Again, the intensity of (222) component was substantially higher after 6 and 9 h of soaking for 60% cold reduced specimens than for 70% reduced specimens. The higher intensity of (222) component evolved in specimens with 60% reduction was due to more rapid coarsening of recrystallized grains in the specimens of samples with 60% reduction up to 9 h of soaking (Fig. 4). The grain size became almost similar after 12 h of soaking in both categories of specimens, which resulted in almost identical intensity values of intensity of (222) component. The subsequent drop in intensity of (222) component with duration after reaching its maximum value was accompanied by an increase in intensity of (200) component indicating the dissolution of carbides in ferrite matrix.

Texture studies in terms of integrated intensity of (222) and (200) components and their ratios for cold rolled and intermediate annealed specimen are summarized in Table 2. This table shows that I_{222}/I_{200} ratio was highest when 70% cold reduced specimen was subjected to intermediate annealing at 550 °C for 6 h or 12 h However, 6 h of soaking may not be sufficient for

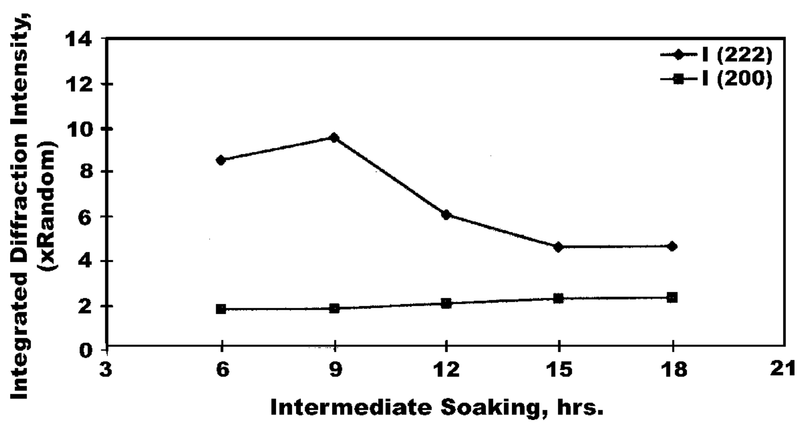

Fig. 5 Variation in integrated diffraction intensity of (222) and (200) components with time during intermediate soaking at 550 °C for 60% cold rolled specimens

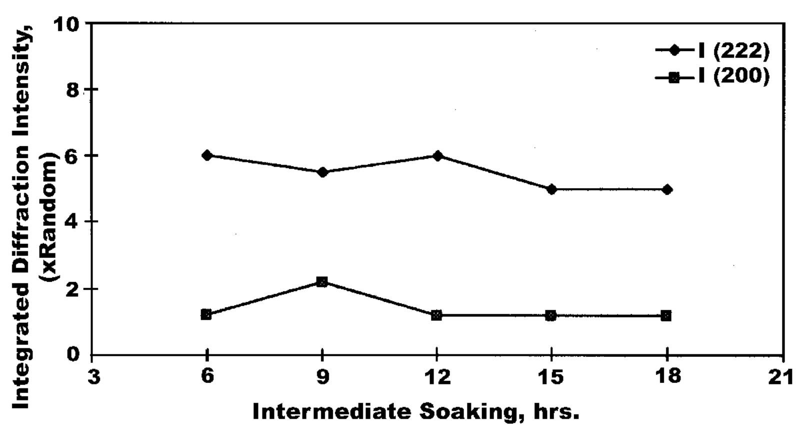

Fig. 6 Variation in integrated diffraction intensity of (222) and (200) components with time during intermediate soaking at 550 °C for 70% cold rolled specimens

initiating the precipitation of AlN particles during intermediate annealing. Therefore, 12 h was considered the optimum duration of soaking at 550 °C to achieve metallurgically favorable conditions in 70% cold reduced specimen for ensuring higher $\bar{r}$ value after final annealing.

Given these results, 70% cold reduced specimens that were subjected to intermediate annealing at 550 °C for 12 h were selected for final annealing.

3.4 Final Annealing

Metallographical examination of a 70% cold reduced specimen showed that the microstructure was predominantly pancaked ferrite grains (Fig. 7). Presence of pancaked structure is linked with the nucleation of recrystallized ferrite grains at the AlN precipitates. During soaking, these grains grew along a preferred direction and ultimately assumed a coarse pancaked structure—a classic phenomenon observed in Al-killed low carbon EDD steel. However, annealing at 690 °C for 12 h had resulted in formation of much finer pancaked ferrite grains compared with grains formed after annealing at lower temperature of 670 °C for 12 h. This indicates that no fresh recrystallization took place during annealing at 670 °C and formation of coarser grains in this case was due to growth of recrystallized grains evolved during intermediate annealing. On the other hand, presence of finer pancaked grains in specimen annealed at 690 °C indicates that recrystallization had resumed at a temperature somewhere between 670 °C and 690 °C. Annealing at higher temperature (710-730 °C) resulted in further coarsening of recrystallized pancaked ferrite grains freshly formed between 670 °C and 690 °C. Interestingly, no appreciable grain coarsening was observed when annealing duration increased from 12-18 h at different annealing temperatures. The average ferrite grain size varied from 13.6 ± 1.6 μm to 16.4 ± 1.9 μm corresponding to annealing condition of soaking at 690 °C for 12 h and 710 °C for 18 h. A detailed analysis of ferrite grain size for different combinations of annealing temperature and time is shown in Table 3.

Table 2 Integrated Intensity of (222) and (200) Components and Their Ratios for Cold Rolled (CR) and Intermediate Annealed (IA) Specimens

	I_{222}		I_{200}		I_{222}/I_{200}	
CR 60% (A)	4.0		1.8		2.2	
CR 70% (B)	5.5		2.0		2.75	
	A	B	A	B	A	B
IA 6 h	8.5	6.0	1.8	1.0	4.7	6.0
IA 9 h	9.5	5.5	1.8	2.5	5.3	2.2
IA 12 h	6.0	6.0	2.3	1.0	2.6	6.0
IA 15 h	4.0	5.0	2.3	1.0	1.7	5.0
IA 18 h	4.0	5.0	2.3	1.0	2.2	5.0

Analysis of the integrated intensity ratio of (222) and (200) components with varied conditions of annealing treatments in 70% cold reduced specimens are shown in Fig. 8. Note that the intensity ratio decreased initially when annealing temperature increased from 670-690 °C then again increased with further increase in temperature. Initial drop in intensity ratio was probably due to formation of finer pancake grains at 690 °C compared with pancake grains formed at 670 °C. At temperatures higher than 690 °C, presence of coarser pancake grains resulted in higher intensity of (222) texture. Here the grain growth process is essentially cannibalistic in nature with larger grains consuming the smaller ones. Therefore, (222) oriented grains, owing to their favored nucleation, have the best opportunities for growth, and so prosper during this competitive process.[11] The highest value of intensity ratio was found in the specimen,

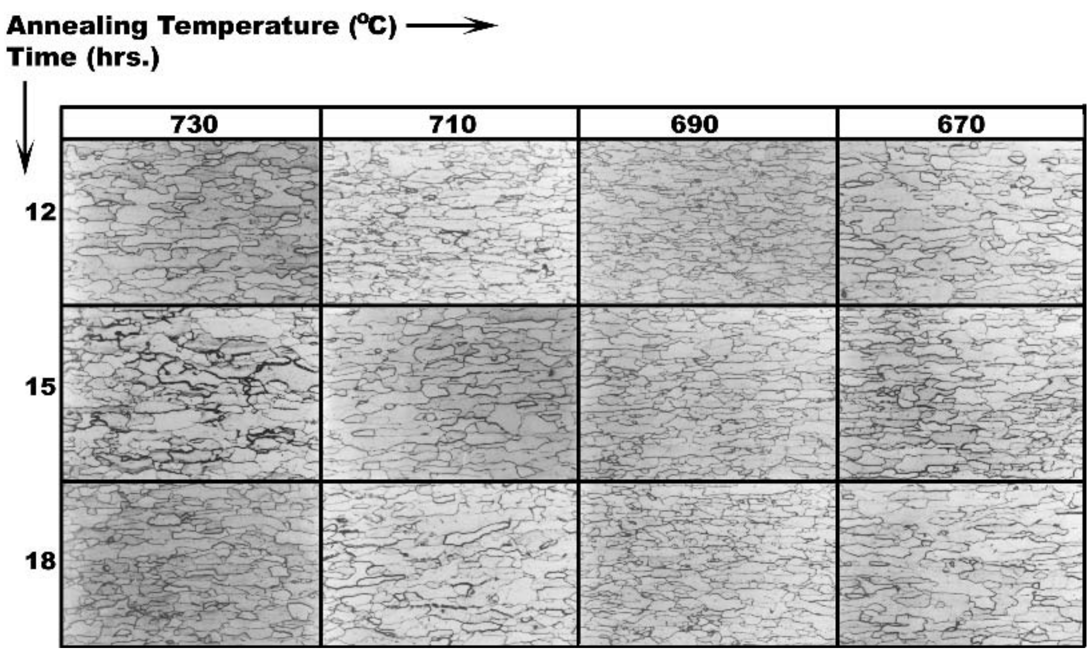

Fig. 7 Influence of varied annealing conditions on microstructural evolution in 70% cold rolled and intermediately annealed specimens (X200)

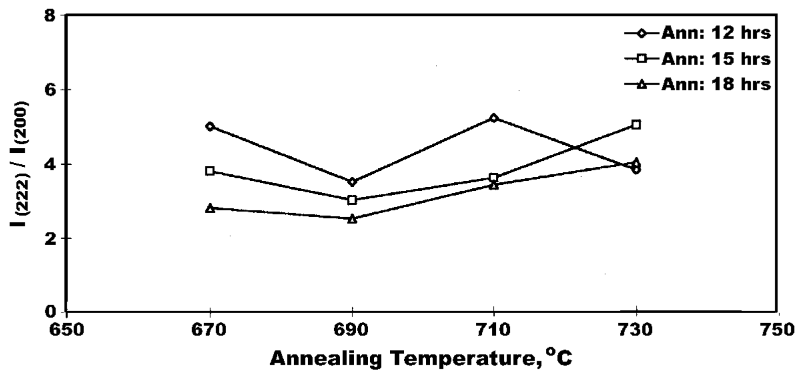

Fig. 8 Influence of varied annealing conditions on integrated diffraction intensity of (222) and (200) components for 70% cold rolled specimens with intermediate soaking at 550 °C for 12 h

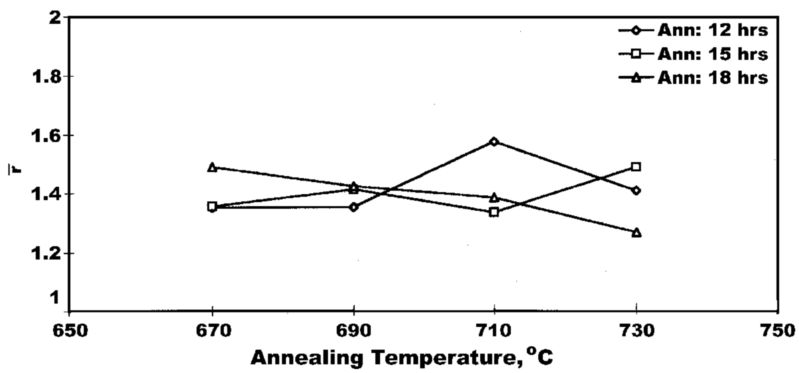

Fig. 9 Change in $\bar{r}$ value with annealing temperature and time for 70% cold rolled specimens with intermediate soaking at 550 °C for 12 h

Table 3 Ferrite Grain Size for Different Combination of Annealing Temperature and Time

Serial No.	Annealing Temperature and Time	Avg. Grain Size, μm	Aspect Ratio
1	670 °C, 12 h	15.0 ± 2.3	2.50
2	670 °C, 15 h	14.8 ± 1.8	2.57
3	670 °C, 18 h	15.6 ± 2.0	2.53
4	690 °C, 12 h	13.6 ± 1.6	3.05
5	690 °C, 15 h	14.4 ± 2.1	2.66
6	690 °C, 18 h	14.6 ± 1.8	2.86
7	710 °C, 12 h	15.4 ± 2.2	2.86
8	710 °C, 15 h	15.9 ± 2.4	2.34
9	710 °C, 18 h	16.4 ± 1.9	2.56
10	730 °C, 12 h	15.0 ± 2.0	2.84
11	730 °C, 15 h	15.6 ± 2.1	2.04
12	730 °C, 18 h	14.7 ± 1.8	2.98

which was annealed at 710 °C for 12 h. Also, Fig. 9 shows that the $\bar{r}$ value was maximum when annealing temperature was 710 °C and duration was 12 h. In addition to pancaked ferrite grain size, other metallurgical factors like extent of carbides dissolution and quantity of austenite formed (at annealing temperatures higher than Ac_1) play significant roles in deciding the evolution of texture in finally annealed Al-killed sheets. In the current study, it seems that annealing of 70% cold reduced specimen at 710 °C for 12 h had resulted in evolution of most favorable conditions for achieving a higher intensity ratio of (222)/(200) leading to superior formability with $\bar{r}$ above 1.5.

4. Conclusions

Based on the above understanding, the following conclusions have been drawn:

- In HSCR steel, the upper limit of cold reduction prior to annealing should not exceed 70% when the intensity of (222) component attained its highest value without appreciable increase in the intensity of (200) component. Therefore, 60-70% cold reduction should be accepted as an optimum reduction for evolving favorable texture.
- Intermediate annealing at 550 °C for both 60% and 70% cold rolled specimen confirmed that recrystallization practically ceased to proceed beyond 12 h of soaking, when integrated diffraction intensity of (222) component reduced significantly. Therefore, intermediate annealing at 550 °C for 12 h can be considered an optimum condition for facilitating development of favorable texture after final annealing.
- The microstructures developed after final annealing at 670 °C, 690 °C, and 710 °C in 70% cold rolled specimens remained unaffected with varied duration (12-18 h) of soaking. However, the grain size of annealed specimens was substantially influenced by the annealing temperature.

- Presence of finer grains at 690 °C in comparison to that at 670 °C showed that recrystallization commenced again between 670 °C and 690 °C in the 70% cold reduced specimens.
- The integrated intensity ratio of (222) and (200) components varied widely with the final annealing conditions. However, a minimum intensity ratio of 5.0 and $\bar{r}$ value of 1.5 was achieved in 70% cold reduced and intermediately annealed specimens were finally annealed at 710 °C for 12 h.

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