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Optimization of Annealing Parameters for Improvement in Formability of Extra Deep Drawing Quality Steel

Biswajit Sarkar, B.K. Jha, and Anjana Deva

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A number of annealing cycles were investigated in an attempt to find the optimum cycle that results in an attractive combination of mechanical and formability properties of an extra deep drawing (EDD) quality steel. It was found that the cycle that involved an intermediate anneal at 600 °C followed by further soaking at 700 °C resulted in the best combination of mechanical and formability properties. It was also found that the rate of heating up to 600 °C can be kept at 50 °C/h while the heating has to be done at a rate of 30 °C/h from 600 °C to the final annealing temperature of 700 °C. The desirable combination of mechanical and formability properties has been correlated with the microstructure that shows pancaking of the annealed grains accompanied by precipitation of carbides. Precipitates of carbides are more in number and smaller in size in the case of samples annealed by the cycle mentioned above compared to the ones annealed by other cycles. They are spherical in shape, which is desirable for forming applications.

Keywords batch annealing, double stage annealing, extra-deep drawing quality steel

1. Introduction

Extra-deep drawing (EDD) quality steel sheets are produced by either batch annealing or continuous annealing of cold-rolled steel sheets containing carbon (C) up to about 0.05% and manganese up to about 0.2%. Batch annealing cycles normally involve slow heating up to about 700 °C. Heating rates vary between 20 and 200 °C/h. The exact heating rate is chosen depending upon the productivity requirements, namely, the weight and number of coils to be annealed, the heating method used, the stacking arrangement of the coils, etc. A typical annealing cycle may last for as long as two to three days, which involve the heating of the coils then soaking at the appropriate annealing temperature, followed by cooling in the bell type furnace. In case of continuous annealing, on the other hand, the total process time is reduced to only a few minutes.

Notwithstanding the fact that continuous annealing leads to better productivity, traditionally the best qualities of formable steels have always been obtained through batch annealing. Evolution of this technology over many years has led to the understanding of the role of grain size, crystallographic texture and the distribution of carbides that are required for sheet formability.

In recent times, a two-stage annealing cycle has been adopted for the batch annealing of cold rolled EDD grade steels in some plants, namely, the Bokaro Steel Plant of the Steel Authority of India Limited (SAIL) and the Tata Iron and Steel Company (TISCO, Jharkhand), India. A typical double annealing cycle consists of heating a coil up to an intermediate temperature and soaking at this intermediate temperature followed by further heating to about 700 °C. At this point, the steel is soaked at this temperature for some prescribed time followed by furnace cooling. The heating rates in both segments are on the order of 30-50 °C/h. Slow heating after cold rolling is normally necessary to allow adequate time for the aluminum to diffuse, forming clusters or precipitates before recrystallization commences. While the first segment provides for precipitation, the second brings about recrystallization.

The optimum heating rate up to the precipitation stage has been calculated by Takahashi and Okamoto[1]: $\log(PHR) = 18.3 + 2.7 \log [(\mathrm{Al})(\mathrm{N})(\mathrm{Mn})/R_{CR}]$, where PHR is the peak heating rate and R_{CR} is the percentage reduction via cold working.

The objectives of the present study were to examine the effects of using different heating rates as well as intermediate temperatures to find an annealing cycle that results in an attractive combination of mechanical and formability properties.

2. Experimental Material

For the present work, a 3.55 mm thick hot band was obtained from Bokaro Steel Plant (Bokaro Steel City, Jharkhand, India) of the Steel Authority of India Limited (SAIL). The chemical composition of the hot band was determined by optical emission spectroscopy (OES). The hot-rolled sample was pickled in hydrochloric acid and then cold rolled by 70% in an experimental rolling mill supplied by M/s Hille, England. The chemical composition of the alloy is found in Table 1.

3. Experimental Procedures

3.1 Annealing

Eight annealing cycles were investigated. In these cycles, the rate of heating up to the intermediate temperature, the intermediate annealing temperature, and the rate of heating from

Biswajit Sarkar, B.K. Jha, and **Anjana Deva,** Steel Authority of India Limited, R&D Centre for Iron & Steel, Doranda, Ranchi 834002 India. Contact e-mail: dm@rdcis.bih.nic.in. The authors are with the R&D Centre for Iron and Steel, Steel Authority of India Ltd., Ranchi. Contact e-mail: aec@rdcis.bih.nic.in.

Table 1 Nominal Composition in (Weight Percent) of Extra Deep Drawing Steel as Determined by Optical Emission Spectroscopy

C, wt%	Mn, wt.%	Si, wt.%	S, wt.%	P, wt.%	Al, wt.%	O, ppm	N, ppm
0.05	0.19	0.022	0.008	0.024	0.051	77	96

the intermediate annealing temperature to the final annealing temperature were varied. Two heating rates, namely, 50 and 75 °C/h, were chosen for the intermediate annealing temperature-heating stage, whereas for the heating stage up to the final annealing temperature, heating rates of 15 and 30 °C/h were chosen. Two intermediate annealing temperatures, 550 and 600°C, were selected. The samples were soaked for half an hour at 550 or 600 °C, as well as at the final annealing temperature of 700 °C. All the samples were cooled in the furnace.

Based on the aforementioned variables, the following eight annealing cycles were investigated (Table 2). After annealing, samples from each of the eight cycles were pickled in hydrochloric acid and samples were prepared for tensile and formability tests.

3.2 Tensile Testing

Tensile samples, having gauge lengths of 50 mm, were prepared in the longitudinal and transverse directions, as well as 45° to the rolling direction. Tensile tests were performed at a crosshead speed of 5 mm/min in the Instron 1195 Static Tensile Testing Machine (UK). Yield strength, ultimate tensile strength, percentage total elongation, uniform elongation, and strain hardening exponent (n) were determined for all the samples in these three directions. The results are shown in Table 3. The strain hardening exponents are shown in Table 4.

3.3 Determination of Average Plastic Anisotropy (r_{avg})

For the determination of r, 50 mm gauge length tensile samples in three directions, 0°, 90°, and 45° to the rolling direction, were prepared. These were pulled in tension by 15%. The plastic anisotropy ratios (i.e., the r values) were determined as follows.

$$r = \frac{\varepsilon_w}{\varepsilon_t} \quad \text{(Eq 1)}$$

$$\varepsilon_w = \ln\left(\frac{w_f}{w_i}\right) \quad \text{(Eq 2)}$$

$$\varepsilon_t = \ln\left(\frac{t_f}{t_i}\right) \quad \text{(Eq 3)}$$

Since volume is conserved during plastic deformation, $l_i w_i t_i = l_f w_f t_f$ and so it follows that, $t_f/t_i = l_i w_i/l_f w_f$. Usually an elongation of 20% is given to the samples for the measurement of strains, but in view of the limited values of total elongation in the case of these samples from some of the annealing cycles, the samples were elongated by only 15%. Therefore, it follows that,

Table 2 Investigated Annealing Schedules

Cycle No.	Description
1	Heating to 550 °C at 50 °C/h and 550-700 °C at 15 °C/h
2	Heating to 550 °C at 50 °C/h and 550-700 °C at 30 °C/h
3	Heating to 550 °C at 75 °C/h and 550-700 °C at 15 °C/h
4	Heating to 550 °C at 75 °C/h and 550-700 °C at 30 °C/h
5	Heating to 600 °C at 50 °C/h and 600-700 °C at 15 °C/h
6	Heating to 600 °C at 50 °C/h and 600-700 °C at 30 °C/h
7	Heating to 600 °C at 75 °C/h and 600-700 °C at 15 °C/h
8	Heating to 600 °C at 75 °C/h and 600-700 °C at 30 °C/h

$$r = \frac{\ln\left(\frac{w_f}{w_i}\right)}{\ln\left(\frac{w_i}{1.15\, w_f}\right)} \quad \text{(Eq 4)}$$

where w_i is the initial width of the tensile sample, w_f is the final width of the tensile sample, t_i is the initial thickness, t_f is the final thickness, ε_w is the strain in the width direction, and ε_t is the strain in the thickness direction. Thus, the average plastic anisotropy ratio (r_{avg}) was determined in the following manner:

$$r_{avg} = \frac{r_o + 2r_{45°} + r_{90°}}{4} \quad \text{(Eq 5)}$$

where r_o is equal to r in the rolling direction, $r_{45°}$ is equal to r at 45° to the rolling direction and $r_{90°}$ is equal to r at 90° to the rolling direction.

3.4 Formability Test

Plane strain forming limits (FLDo) were determined for the samples from each of the eight annealing cycles. For the determination of FLDo, strips measuring 250 × 125 mm were prepared from the annealed samples. Earlier trial experiments were performed to determine the width that gave rise to plane strain conditions. It was found that a width of 125 mm for the strip gave rise to the condition wherein the minor strain was zero when the sample was stretched in the other direction. The eight annealed strips were electrolytically grid marked with 5 mm diameter circles. These were then punch stretched with a 100 mm diameter hemispherical punch at the rate of 10 mm/min in a universal formability-testing machine supplied by M/s Erichsen (Germany). The blank holder force was kept at 180 kN for all the samples. The stretching of the samples was conducted until fracture.

After the punch stretching was over, the samples were removed from the formability tester. The original circles had been deformed into ellipses. The minor and major axes of the

Table 3 Mechanical Properties

Cycle	YS, kg/mm^2				Ultimate Tensile Strength, kg/mm^2				Uniform Elongation (ε_u), %				Total Elongation, %			
No.	0°	45°	90°	Av.	0°	45°	90°	Average	0°	45°	90°	Average	0°	45°	90°	Average
1	22.2	24.5	22.6	23.5	32.3	34.2	31.4	33.0	22.1	18.5	20.9	20.5	30.4	26.1	29.6	28.0
2	23.8	22.2	23.3	22.9	33.2	34.3	33.3	33.8	23.4	22.1	23.4	23.0	33.8	28.9	32.2	31.0
3	22.3	25.9	24.6	24.7	32.2	34.9	32.5	33.6	17.4	13.9	16.2	15.8	26.7	21.1	19.2	22.0
4	21.0	20.9	21.5	21.1	28.3	24.0	28.7	26.3	18.5	15.0	17.4	17.0	28.3	24.0	28.7	26.3
5	19.8	20.7	21.2	20.6	27.9	31.4	28.4	29.8	20.9	19.7	24.6	21.8	32.4	25.2	38.9	28.9
6	20.7	21.3	19.5	20.7	30.8	32.1	29.5	31.1	24.6	24.6	23.4	24.2	32.1	32.9	30.9	32.0
7	20.8	22.3	21.7	21.8	30.9	32.8	29.9	31.6	20.9	11.6	19.7	17.4	35.9	22.5	30.0	27.7
8	22.5	23.8	23.8	23.5	33.9	37.8	36.3	31.4	19.7	20.7	23.4	21.3	30.0	31.2	33.0	31.3

Table 4 Formability Properties

Cycle No.	n	r_{avg}	FLDo, %
1	0.19	1.25	28.5
2	0.21	1.33	27.0
3	0.15	1.10	25.0
4	0.16	1.35	27.5
5	0.20	1.70	30.0
6	0.22	1.92	35.0
7	0.17	1.20	30.0
8	0.20	1.40	30.0

ellipses were measured and were then used to calculate the minor and major strains, respectively. The ellipses just adjacent to fracture were classified as the necked ones. The necked ellipses were classified as failed and the ellipses next to them were considered safe. The strain was measured both manually as well as by the grid pattern analyzer (GPA) supplied by Camsys, USA. The highest reading of major strain for zero minor strain was taken as the FLDo.

3.5 Metallography

Samples from the as-received hot band and cold-rolled and annealed strips from all the eight annealing cycles were prepared for optical metallography. These samples were etched in 2% nital and were microscopically examined using a Neophot-30 optical microscope. The fracture surfaces of the samples that fractured during formability testing were observed in JSM 840 Scanning Electron Microscope (SEM) supplied by JEOL (Tokyo, Japan).

Thin foils for transmission electron microscopy (TEM) were prepared by a twin jet polishing method using an electrolyte consisting of perchloric acid and acetic acid. These thin foils were examined in a JEOL Transmission Electron Microscope operating at 150 kV.

4. Results and Discussion

Figure 1 shows the optical micrograph of the as-received hot band. The nominal chemical composition of the hot band is shown in Table 1. The results of the tensile tests are shown in Table 3. Table 4 shows the results of the formability tests as well as the average plastic anisotropy ratios and the strain hardening exponents. It was observed that for the eight annealing cycles, cycle 6 resulted in the best combination of mechanical and formability properties, namely $n = 0.22$, $r_{avg} = 1.92$ and FLDo = 35%. This cycle involved heating the cold rolled sample at a rate of 50 °C/h to 600 °C and then soaking it there for half an hour before heating it from 600 to 700°C at the rate of 30 °C/h. It was soaked at this temperature for half an hour before being cooled to room temperature in the furnace. Cycle 3, which consisted of heating the sample at the rate of 75 °C/h to 550°C and then to 700 °C at a rate of 15 °C/h, yielded the poorest combination of properties with $n = 0.15$, $r_{avg} = 1.10$ and FLDo = 25%. It is well known that the strain hardening exponent (n) is strongly correlated with the FLDo.[2] The results obtained in this research are consistent with this observation.

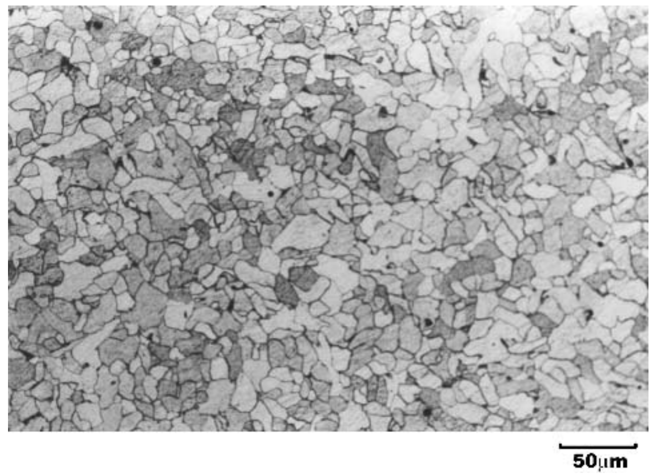

Fig. 1 Optical micrograph of as-received hot band showing equiaxed ferrite grains of average 10 μm in size, 200×

Normally it was believed that the desirable properties for EDD quality steel resulted from the "pancaking" of annealed grains, which imparts anisotropy in the mechanical and formability properties of the steel. In the present case, however, it has been observed that although there is pronounced pancaking of the grains for the sample annealed using cycle 6 (Fig. 2) the sample from annealing cycle 3 also has also shown some pancaking (Fig. 3). However, the mechanical and formability properties of the cycle 3 sample were not commensurate with the extent of pancaking. It is to be noted that the formability parameters, in particular *n, r,* and FLDo, are inferior in case of the cycle 3 sample. Hence, the attractive combination of mechani-

Fig. 2 Optical micrograph of the sample annealed as per cycle 6 showing prominent pancaking, 200×

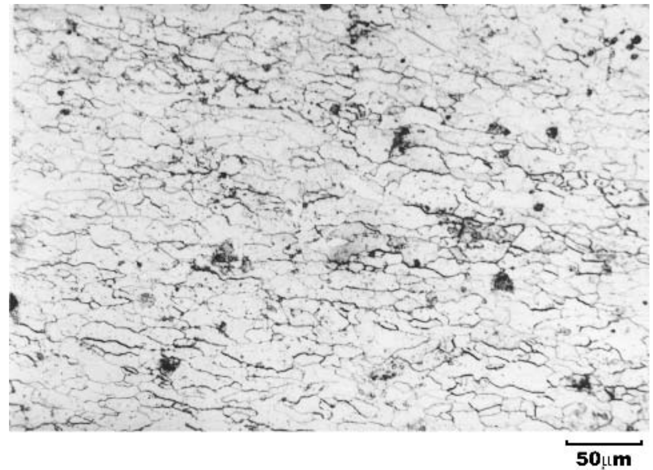

Fig. 3 Optical micrograph of the sample annealed as per cycle 3 showing mild pancaking, 200×

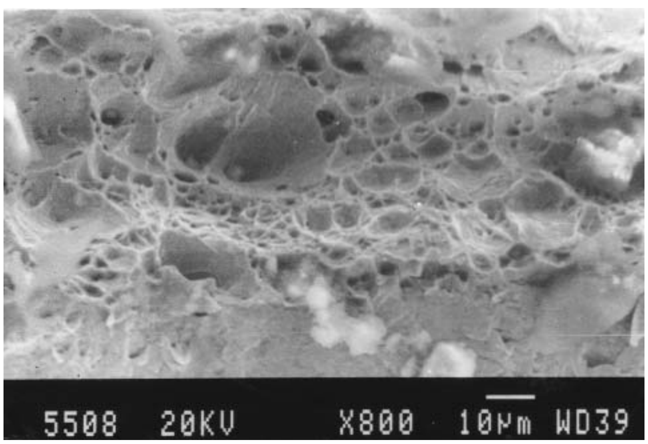

Fig. 4 SEM micrograph of the fracture surface of the sample annealed according to cycle 6 and punch stretched to fracture

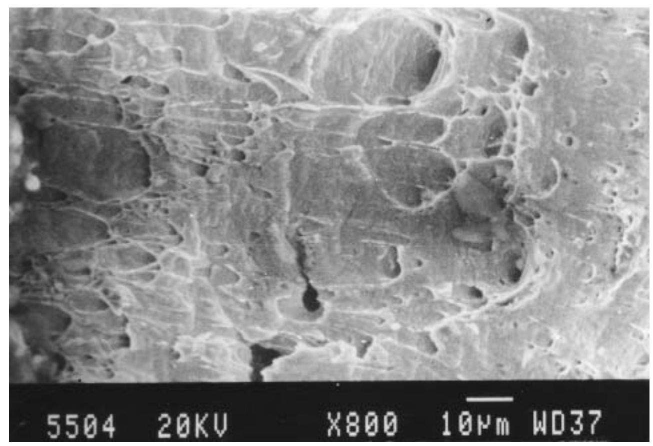

Fig. 5 SEM micrograph of the fracture surface of the sample annealed according to cycle 3 and punch stretched till fracture

cal and formability properties for the cycle 6 sample is not entirely attributable to pancaking.

That the formability property of the cycle 6 sample is superior to the one annealed according to cycle 3 can be corroborated from SEM micrographs of the fracture surfaces. These images are shown in Fig. 4 and 5. Figure 4 shows the fracture surface of the sample annealed according to cycle 6, which fractured during punch stretching. The fracture surface has many dimples, which is indicative of ductile fracture. On the other hand, the sample from annealing cycle 3 shows signs of cleavage fracture (Fig. 5). This behavior is more indicative of brittle failure and results in inferior formability properties. It should be noted that both these samples were stretched under identical conditions. Hence, it can be concluded that the difference in the mode of fracture can be attributed to the difference in their intrinsic forming properties.

An interesting observation in these samples has been the precipitation of carbides. Figures 6 and 7 show bright field TEM images of the carbides in samples annealed according to cycles 3 and 6. The sample annealed as per cycle 6 shows profuse precipitation of carbides compared to the sample annealed according to cycle 3. The former sample was given an intermediate anneal at 600 °C, whereas the latter was annealed at 550 °C during this step. In addition to the amount of carbide precipitation, it is also observed that the carbides are smaller and greater in number for the cycle 6 sample. Also, it is to be noted that the carbides are spherical in shape.

As a result of the profuse precipitation of carbides, the ferrite contains less C (i.e., the ferrite is "purer")[3,4] for cycle 6 sample. It is well known that the "purification" of ferrite grains leads to better formability properties. In fact, this forms the basis of the continuous annealing of extra deep drawing quality steels. Besides the fact that the ferrite is purer in case of the samples annealed as per cycle 6, the presence of smaller and more spherical carbides has conferred superior formability properties compared to the sample that has been annealed as per cycle 3. In the latter case, carbides are not as numerous and they are not spherical in shape.

An examination of the strain hardening exponents (n values) and the FLDo values in Table 4 reveals that the heat

Fig. 6 TEM micrograph showing the bright field image of carbides in the sample annealed as per cycle 6. The carbides are spherical in shape and smaller in size, 6000×.

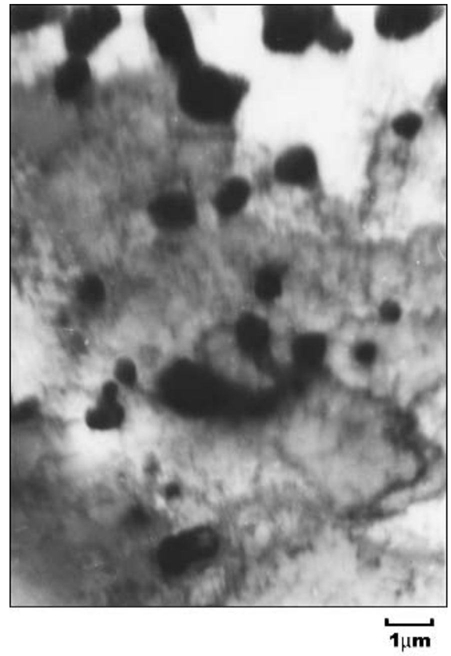

Fig. 7 TEM micrograph showing the bright field image of carbides in the sample annealed as per cycle 3. The carbides are non-spherical in shape and larger in size, 6000×.

treatments that involved intermediate anneals at 600 °C resulted in higher FLDo values compared to the ones given intermediate anneals at 550 °C. Moreover, from the magnitude of the *n* values, it is apparent that a slower rate of heating up to the intermediate annealing temperature yields a superior combination of the *n* and FLDo values. Hence, the best annealing heat treatment consists of slow heating up to 600 °C, and then to 700 °C, as contrasted to the one procedure that involved rapid heating up to 550 °C followed by slow heating to 700 °C. This resulted in the poorest formability properties.

5. Conclusions

The annealing cycle, which involved heating the 70% cold rolled strip up to 600 °C at 50 °C/h and then soaking at 600 °C, followed by heating from 600 to 700°C at 30 °C/h and soaking at this temperature followed by furnace cooling, resulted in the best combination of mechanical and formability properties. For this annealing heat treatment schedule, the following values were obtained: an average strain hardening exponent (n) of 0.22, average plastic anisotropy ratio (r_{avg}) of 1.92, and a plane strain forming limit (FLDo) of 35%. The fracture surface of this sample showed dimples, which is indicative of ductile fracture. The attractive combination of properties found in the samples annealed as per the above schedule can be attributed to the profuse precipitation of small spherical carbides, which resulted in a more pure ferrite (i.e., lower C). This leads to desirable forming properties.

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