J Mater Sci (2009) 44:3736–3740
DOI 10.1007/s10853-009-3499-3

Microstructural evolution during batch annealing of boron containing aluminum-killed steel

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Received: 24 February 2009 / Accepted: 15 April 2009 / Published online: 7 May 2009

Abstract Role of boron in low carbon aluminum-killed cold rolled batch annealed steels has been critically examined. It was found that it is not the absolute boron but B/N ratio that controls the forming properties. Pancake shaped grains (high grain shape anisotropy) are highly desirable for improving the desirable {111} texture, normal anisotropy, and draw ability of the steel sheets. Microstructural analysis showed that the extent of pancaking decreases with increase in B/N atomic ratio and reaches ultimately to formation of equiaxed grains. Low B/N ratio (upto 0.3) resulted in improved mean plastic anisotropy ratio (r_m) value and high grain shape anisotropy, which has been characterized through grain aspect ratio. The desirable orientation in steel with low B/N ratio is attributed to sufficient availability of Al and N to precipitate during batch annealing. Optimum amount of boron, aluminum, and nitrogen in steel has resulted in coarse pancake structure, which is ideally suited for improved formability.

Introduction

Beneficial effect of small addition of boron in continuous annealed low carbon steel has been studied by many researchers in recent past [1–3]. The optimum combination of low yield strength and high r_m value has been reported at stoichiometric boron addition. The improvement on r_m has been attributed to ferrite grain growth after recrystallization in the annealed steel. Though boron has been extensively used in batch annealed IF steel for reducing cold work embrittlement, limited work has been reported on the influence of boron in batch annealed low carbon (C: 0.03–0.06%) aluminum-killed steel [4–6]. In all these investigations it has been concluded that boron addition results a pronounced deterioration in the [111] texture development and r_m value. Further, absolute boron rather than B/N ratio has been found to be the main contributory factor in deterioration of r_m value [4, 5]. However, in the literature there is no systematic study on the microstructural evolution during batch annealing of boron containing aluminum-killed steel particularly quantifying B/N ratio for improving the r_m value, which is reported in the present paper for the first time. This study will be helpful in development of cold rolled batch annealed formable steel even with higher nitrogen content (upto 80 ppm).

Experimental

The study has been carried out on the industrially produced low carbon (0.03–0.06 wt%) steel with varying B/N atomic ratio. The chemical composition of steels used for this study is shown in Table 1. Steel A is the typical chemistry used for producing EDD (Extra Deep Drawing) steel.

All the steels were continuously cast to 210 mm thick slabs and were hot rolled to 3.8 mm thickness. The hot rolled (HR) bands were finish rolled at 880 ± 10 °C and coiled with varying temperature from 560 to 640°C.

HR coils were cold reduced to 1.2 mm thickness. All the cold rolled coils, irrespective of B/N ratio, were annealed with the standard annealing cycles used for low carbon

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Table 1 Chemical composition of steels (wt%)

Steel	C	Mn	S	P	Si	Al	B ppm	N ppm	B/N atomic ratio
A	0.030	0.16	0.007	0.013	0.027	0.038	0	57	0
B	0.032	0.160	0.009	0.012	0.038	0.015	10	84	0.15
C	0.034	0.173	0.005	0.015	0.029	0.030	12	84	0.185
D	0.035	0.161	0.010	0.013	0.037	0.016	10	68	0.19
E	0.038	0.18	0.006	0.015	0.036	0.023	20	85	0.30
F	0.037	0.177	0.008	0.017	0.019	0.031	20	54	0.48
G	0.036	0.178	0.007	0.015	0.032	0.034	20	33	0.80
H	0.032	0.17	0.009	0.014	0.025	0.015	52	80	0.84
I	0.033	0.172	0.009	0.014	0.032	0.041	45	67	0.87

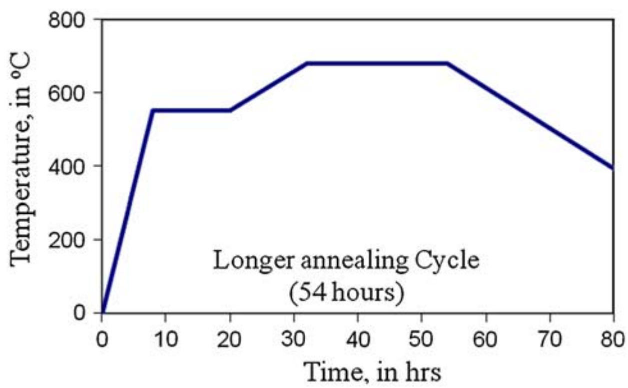

Fig. 1 Schematic representations of batch annealing cycle

EDD grade steel in Bokaro steel plant of Steel Authority of India Limited (SAIL) as schematically shown in Fig. 1. Intermediate holding at 550 °C has been intentionally kept to facilitate maximization of AlN precipitation before recrystallization.

Microstructural observations were performed on polished and etched (2–4% natal) hot rolled and cold rolled annealed samples. All the strips were observed in a plane perpendicular to the rolling plane and along the rolling direction. The major and minor axis for individual grains for all the cold rolled batch annealed samples was measured to determine aspect ratio using image analyzer Leica 600. Mean plastic anisotropy ratio (r_m) was measured for aspect ratio of 1.82 and 3.75 and having B/N ratio 0.87 and 0.185, respectively.

Results and discussion

Quinto et al. [4] carried out extensive study on the role of boron in low carbon (C: 0.06–0.07%) unalloyed steel with B:10–40 ppm, N: 60–120 ppm processed through batch

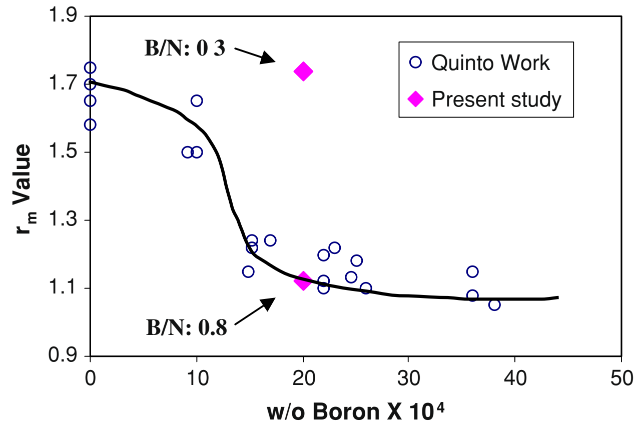

Fig. 2 Effect of boron addition on r_m value of batch annealed aluminum-killed steel [4]

annealing route. They have observed (Fig. 2) that addition of more than 15 ppm of boron to Al-killed drawing quality reduces its drawing properties to that essentially of rimmed steel. The change of r_m value from 1.7 to 1.2 is associated with characteristic change from elongated to equiaxed, an inhibition of aluminum nitride precipitation in annealed sheet and lowering of intensity of {111} poles. Lyudkovsky and Rastogi [5] have also shown that addition of boron has resulted in substantial retardation of AlN precipitation and suppression of {111} orientation in low carbon batch annealed steel.

It has further been reported [4] that AlN precipitation, which is critical to the development of texture, is inhibited by boron additions to such an extent as to nullify its effect in aluminum-killed steel. The authors have concluded that absolute value of boron is the deciding factor in decreasing the r_m value even if sufficient amount of Al and N are present in solution during batch annealing, conventionally a prerequisite for formation of pancake structure and high r_m value [7]. The suppression of AlN precipitation by reducing the AlN nucleation sites in the presence of boron has been reported to be the main reason of low r_m value. It has further been explained that the presence of segregated boron, coarse borocarbides, or fine borocarbides act to reduce the free energy of grain boundaries [4, 8, 9] and so the number of effective sites that ultimately effect suppression of AlN precipitation.

In the present study, we have found significant difference in r_m value in the industrial steels with similar absolute boron content (20 ppm) and different B/N atomic ratio, which has been indicated in Fig. 2. As a result of change in B/N ratio from 0.3 to 0.8, the r_m has decreased from 1.74 to 1.12. Further XRD results conforms to a higher (111)/(100) ratio of 8.6 in the steel with B/N ratio of 0.3 compared to that of 4.5 in the steel with B/N of 0.8. The favorable orientation in steel with low B/N ratio can be attributed to sufficient availability of Al and N to precipitate during batch annealing. These findings are quite revealing and not in line with the work of Quinto [4]. A systematic microstructural evolution has been carried out to explain the importance of B/N ratio rather than absolute boron in influencing r_m value.

In the cold rolled batch annealed steel, in addition to the grain size, grain shape is known to strongly influence the properties [10]. For example in the aluminum-killed grade steel, pancake shaped grains (high grain shape anisotropy) are highly desirable for improving the desirable {111} texture, normal anisotropy, and draw ability of the steel sheets [7]. In the current study, the grain shape anisotropy has been characterized through grain aspect ratio, which is the ratio of major and minor grain length. Figure 3 shows the variation of aspect ratio with varying B/N atomic ratio.

It is interesting to note that aspect ratio of steel B (B/N: 0.185) is found to be higher (shown by dotted line) as compared to that of steel A (B/N: 0). As the B/N ratio increases, aspect ratio decreases. A definite logarithmic trend emerged while co-relating B/N ratio vis-à-vis aspect ratio, showing a good correlation coefficient ($R^2 = 0.94$).

The change in microstructure from pancaking to equiaxed with increasing B/N ratio has been shown in Fig. 4a–e. The coiling temperature was kept less than 600 °C for all the steels. To observe the effect of coiling temperature on the morphological changes, microstructure (Fig. 4f) of steel sample with higher B/N ratio of 0.87 and processed with higher coiling temperature of 640 °C has also been included. As is evident, the extent of pancaking has decreased with increasing B/N ratio. Even with higher B/N ratio (0.87), microstructure of steel coiled with lower temperature (Fig. 4e) shows minor extent of pancaking, whereas samples coiled at higher coiling show completely equiaxed grains.

Deep drawing aluminum-killed steel are processed keeping low coiling temperature (<550 °C) leaving Al and N in super saturated solid solution. AlN particle or AlN

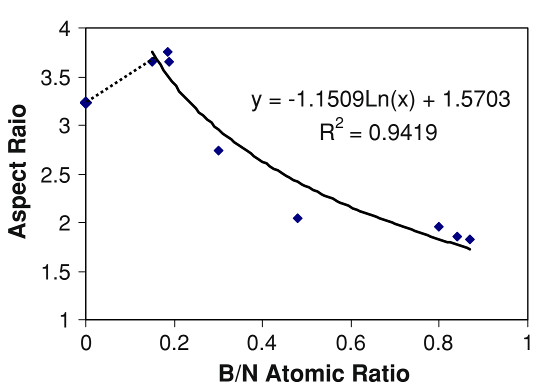

Fig. 3 Variation in mean aspect ratio with varying B/N atomic ratio

clusters precipitate at the early stage of batch annealing at the boundaries of sub grain in the deformed samples [7, 11]. The dominant effect of these clusters or precipitate is to retard nucleation of recrystallization.

More precisely, nucleation by sub grain growth is selectively inhibited by these clusters or precipitate at sub grain boundary [10]. At the end of batch annealing treatment, the pancake grains are a consequence of the initial pancake deformed microstructure and the precipitation of aluminum nitrides in sheets on prior grain boundaries and sub boundaries which lies in the rolling plane, therefore providing anisotropy barrier to growth [7, 11]. The pancaking behavior of microstructure has also been found in boron containing steel (Fig. 4b), which is an indirect evidence of presence of sufficient Al and N in super saturated solid solution during batch annealing.

Further, the mechanism can be discussed in light of microstructural changes occurring during hot rolling and batch annealing. Increase in r_m value and thereby aspect ratio has been discussed by Sudo [1], wherein boron addition has resulted in increase in r_m for continuous annealed steel. The author has attributed it to the ferrite grain growth in the annealed sheet subsequent to ferrite grain growth in hot band. The latter is caused by decrease in nucleation site during transformation from austenite to ferrite [12]. Further, Takahashi [2] has explained this by the size of BN vis-à-vis AlN. AlN, which precipitate during annealing, is generally fine and tends to inhibit growth of grains. Thus, the decrease in precipitates of AlN and consequent increase in precipitates of BN induce the grain size to become larger as the boron content increases. With increase in B/N ratio, the extent of pancaking decreases and reaches ultimately to formation of equiaxed grains.

Finding of the present study can further be explained based on precipitation behavior of BN and AlN in austenite region of Al–B–N system. It has been shown [13, 14] that boron nitride precipitate preferentially in the austenite region in presence of AlN and BN precipitation rate has not been found to be affected by coiling temperature [2] for higher B/N ratio. The finding is particularly significant in our present study as it confirms that in hot rolled steel, significant amount of free nitrogen will be present particularly when both coiling temperature and B/N are low. For B/N of 0.87, the aspect ratio is almost independent of coiling temperature and fully equiaxed grains are obtained after batch annealing (Fig. 4e and f).

Conventionally, steel without boron and steel with lower B/N ratio coiled at lower temperature (<600 °C) should have similar aspect ratio and extent of pancaking due to availability of sufficient N and Al in solid solution. However, in the present study coarse pancaked structure with higher aspect ratio has been found. This improvement in microstructure with boron addition may be due to ferrite

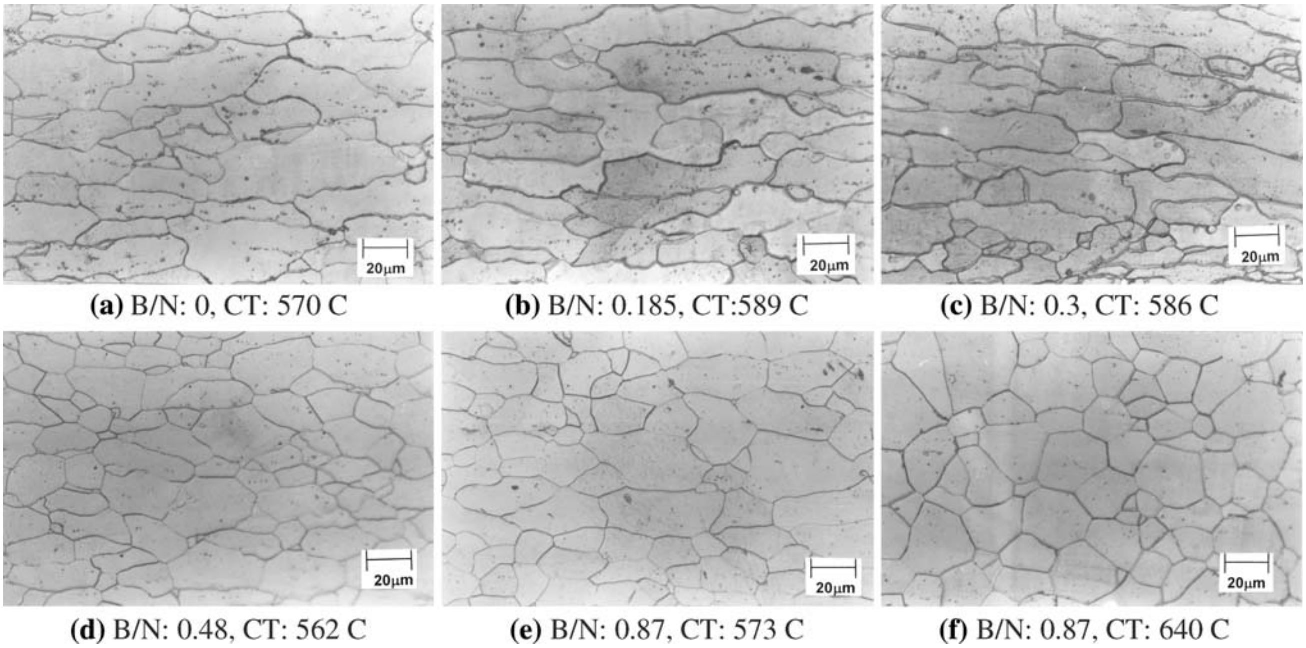

Fig. 4 Microstructure of cold rolled batch annealed steel with increasing B/N ratio

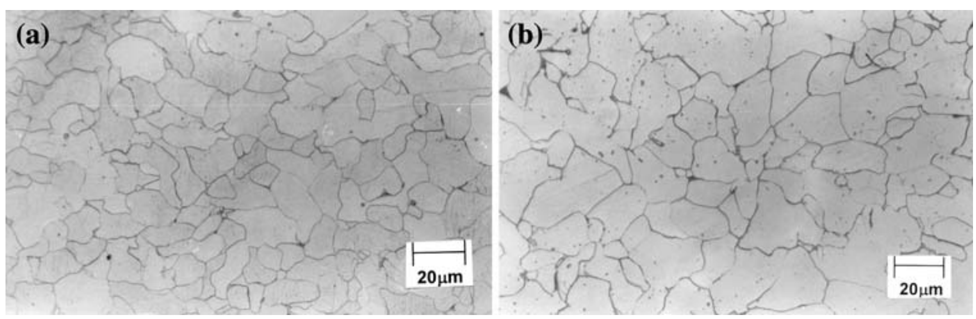

Fig. 5 Microstructure of hot rolled steel **a** B/N: 0 and **b** B/N: 0.185

grain growth after recrystallization in the annealed sheets as a consequence of ferrite grain growth in the hot band [15]. This has been confirmed in the present study wherein the hot band microstructure (Fig. 5a and b) shows coarse ferrite grain size of 22 μm for steel with B/N: 0.185 compared to that of steel without boron, i.e., 15 μm.

Conclusion

The above study concludes that it is not the absolute boron but B/N ratio that controls the properties of batch annealed aluminum-killed steel. The mechanism of non availability of nucleating sites for AlN precipitation and thereby deterioration in forming properties is not valid, rather availability of Al and N in solid solution is the main governing factor for its precipitation during batch annealing, even if boron is present in steel. Optimum amount of boron, nitrogen, and aluminum can lead to coarse pancake structure ideally suited for improved formability.

Acknowledgements The authors thankfully acknowledge the help and support extended by the management of Research and Development Centre for Iron and Steel, Ranchi. Special thank to Mr S. K. De for the help in conducting plant trials.

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