# Development of Nanoprecipitation-Strengthened Cold-Rolled Batch-Annealed HSLA Uncoated Sheet Steels (>550 MPa Yield Strength) With Superior Formability





### Authors

Siddhartha Biswas (top left), Senior Plant Metallurgist, Big River Steel, A U. S. Steel Co., Osceola, Ark., USA sbiswas@bigriversteel.com

Shobhit Bhartiya (top right), Manager of Product Development & Product Quality – Cold Rolled and Coated Products, Big River Steel, A U. S. Steel Co., Osceola, Ark., USA

William Williams, Plant Metallurgist, Big River Steel, A U. S. Steel Co., Osceola, Ark., USA

V.S.Y. Injeti (bottom left), Metallurgist, Advanced Material Characterization, Big River Steel, A U. S. Steel Co., Osceola, Ark., USA

Amar K. De (bottom right), Director of Product Development & Product Quality, Big River Steel, A U. S. Steel Co., Osceola, Ark., USA

A low-carbon Ti-microalloyed chemistry was selected for the development of a family of cold-rolled and batch-annealed high-strength high-formable uncoated sheet steels. The batch annealing practice was optimized to retain precipitate strengthening in the final fully processed steel and microstructural attributes that resulted in superior forming characteristics. Production strategy, material characterization and formability of highstrength low-alloy steels with minimum yield strength of 550 MPa coldrolled batch-annealed uncoated sheet steel will be discussed, and results will be presented.

High-strength low-alloy steel (HSLA) sheets have become the material of choice where higher formability is required along with high strength. HSLA steels are utilized in oil and gas pipelines, heavy-duty highway and off-road vehicles, construction and farm machinery, industrial equipment, storage tanks, mine and railroad cars, barges and dredges, snowmobiles, lawn mowers, and passenger car components. Among the plethora of end-use part manufacturers, the automotive industry is looking for an HSLA steel option to reduce

weight without compromising safety as they must meet the fuel economy standards (vehicles with miles per gallon more efficient) set up by U.S. Department of Transportation's National Highway Traffic Safety Administration (NHTSA). Also, to meet net-zero carbon target, steel produced from continuous strip production (CSP) mills are being inquired about as the CSP technology has significant advantages in reducing  $CO<sub>2</sub>$  emissions.

Production of HSLA sheet steels with a yield strength of 490 MPa and above was presented and published

#### Table 1

Tensile Property Requirements of HSLA 550 Annealed Sheet Steels as Required by Various Specifications





Table 2

Chemistry of Ti-HSLA Steel Selected in the Current Study (wt.% max)

C Mn P S Si Ti N Nb CE<sub>IIW</sub> Pcm 0.060 1.0 0.015 0.003 0.30 0.12 0.009 Res 0.25 0.13 CE<sub>IIW</sub>: C+ Mn/6+ (Cr+Mo+V)/5+(Cu+Ni)/15; P<sub>cm</sub>: C + Si/30+(Mn+Cr+Cu)/20+Ni/60+Mo/15+V/10+5B

earlier by the authors. Continuous inquiry of the next higher-strength-level HSLA inspired the product development of HSLA550 with a minimum yield strength of 550 MPa. Mechanical property requirements for HSLA 550 sheet steels are listed in Table 1 as required by various common sheet steel original equipment manufacturer (OEM) specifications. This was used as target properties for developing sheet steel in this study.

In the current study, a very-low-carbon C-Mn-based ferritic steel with Ti-only microalloying similar to utilized for  $490X$  development<sup>4</sup> was selected to develop a family of HSLA sheet steels with a minimum yield strength of 550 MPa meeting SAE J2340 CR 550X specification.1 Thermomechanical treatment plays an important role in controlling the mechanical properties in microalloyed low-C steel.<sup>5</sup> Strength development was based primarily on precipitation strengthening and control of ferrite grain size. Details of alloy design, hot rolling processing approach and batch annealing parameter optimization through studies of microstructure evolution have been discussed. Finally, the mechanical properties and formability test results are presented to demonstrate the successful production of SAE J2340 CR 550 X.

## **Experimental**

## Alloy Design and Processing Considerations

Based on the success of producing sheet steel with minimum 490 MPa yield strength, a titanium (Ti) microalloying approach was adopted. Laboratory study was done with Ti-HSLA sheet steel to obtain the necessary mechanical properties to meet the minimum 550 MPa yield strength. The experimental chemistry of the HSLA 550 steel is shown in Table 2. The alloy design considerations adopted were described in an earlier paper.4

## Hot Rolling Parameters

The steel was produced by melting a suitable scrap mix in an electric arc furnace. Alloying and optimized steel cleanliness practices to modify and eliminate inclusions were done at the ladle metallurgy furnace. The steel was cast into slabs at the continuous caster and subsequently sent to the 6-stand hot strip processing mill.

The slabs were hot rolled to 2.0–3.0 mm thickness using a 6-pass reduction schedule and finish hot

deformation temperature of 900–925°C. The hot-rolled strip was immediately accelerated cooled to a coiling temperature of 600–675°C. A cooling rate of more than 30°C/second was employed using super-reinforced laminar cooling to result in non-polygonal ferrite grains with substructures if possible. Hot-rolled samples were collected from cropping two outer laps of coils after cooling to evaluate microstructure and mechanical properties.

## Cold Rolling and Choice of Annealing Parameters

The hot-rolled coils were cold rolled to 1.0–1.5 mm thickness (50–60% cold reduction) and then annealed in a batch annealing furnace. The annealing cycle to be used for these cold-worked coils was initially determined using laboratory simulations of batch annealing. Strip samples prepared from cold-rolled sheet were subjected to a programmed batch annealing cycle at various temperatures in a box furnace and tested after furnace cooling for mechanical properties and microstructure. Fig. 1 shows variation in yield strengths as a function of simulated batch annealing temperature. From the simulation results, it was evident that a batch annealing temperature window of 580–630°C was suitable in achieving yield strength above 550 MPa. A batch annealing cycle was

## Figure 1

Yield strength of cold worked samples of selected steels after simulated batch annealing.



2024 I Iron & Steel Technology I [AIST.org](http://www.aist.org)

Oct

## 76 Technical Article

#### Table 3

### Tensile Properties of Batch-Annealed Ti-HSLA Coils



### Figure 2

Head and tail end yield strength comparison for coldrolled batch-annealed sheet steel.



## Figure 3

700 Yield Strength Max 670  $\Diamond$ CRFP  $\Box$  $\Box$  $\Box$ HR 650 Tensile Strength, MPa Tensile Strength, MPa  $\Box$ 680  $\Diamond$ Yield Strength, MPa Yield Strength, MPa ♦  $\Diamond$ 630 ♦ 660 610  $\frac{\Diamond}{\Box}$  $\Box$  $\overline{5}$ 590 640  $\Diamond$ CRFP 570 **DHR** Yield Strength Min Tensile Strength Min 550 620 Quarter Center Quarter Quarter Center Quarter Location Location  $(a)$  (b)

Variation in tensile properties: yield (a) and tensile strength (b) for hot-rolled and cold-rolled batch-annealed sheet steels across the coil width.



Based on this simulation result, cold-rolled coils with Ti-HSLA chemistry were then batch annealed in a hydrogen annealing furnace at temperatures between 580 and 630°C. After cooling, samples were collected from head and tail ends for mechanical and microstructural property evaluation.

## Mechanical and Microstructural Properties Evaluation

Samples from hot-rolled and batch-annealed coils were collected for various property evaluations such as tensile properties, hole expansion ratio and microstructures. Tensile samples across the width were also tested to check

cross-width property variation.

Microstructural Characterization: The full thickness section along the rolling direction was examined in an optical microscope (Leica DMI5000-M) as well as a scanning electron microscope (SEM) (Hitachi SU3500) for microstructural features and cleanliness study. Metallographic samples were mechanically polished to 1 mm diamond paste suspension followed by surface treatment in a Hitachi IM4000 ion milling system for orientation imaging mapping. An area of 128 mm x 100 mm with a step size of 0.3 mm was chosen for electron backscatter diffraction (EBSD) analysis for orientation imaging and texture evaluation in a Hitachi SU3500 SEM.

#### Figure 4

Optical microstructures of Ti-HSLA steel after hot rolling.



Formability Evaluations: Hole expansion ratio (HER) tests were done on hot-rolled and annealed sheet samples using an Erichsen 142-20-Basic test equipment. For the hole expansion tests, five square coupons of 100 mm x 100 mm size were cut from each of three locations across the width, quarter width, center and three-quarter width locations for examining property homogeneity. Holes of 10-mm diameter (*do* ) were punched at center of each coupon for HER tests.

The test coupons were clamped between a holder and die with a clamping force of 85 kN. Die selection for punching was chosen based on ISO 16630:2009(E) specification. For the gauges of this study, 1.44 mm and 3.03 mm, the clearance during punching was 10.4% and 12.1%, respectively. A conical punch with 60° angle was pierced through the hole at a speed of 0.25 mm/second and the crack appearance during piercing was monitored using a digital imaging system. The piercing was done at least 30 minutes after punching the hole. Diameter of the holes after crack appearance was measured and hole expansion

ratio, λ was calculated as  $\lambda = \frac{(d_f - d_o)}{d_f}$ *d f o*  $\frac{d}{d\rho}$ , where  $d_{\rho}$  and  $d_{f}$ 

are initial and final diameter of the hole, respectively.

## **Results**

Head and tail tensile properties of batch-annealed sheet steels are shown in Table 3. It is seen from Table 3 that the sheet steel successfully met the minimum yield strength of 550 MPa in fully annealed condition. Excellent total elongation values were obtained. Tensile properties were also outstandingly uniform from head to tail of the coils as well as across the width of the coils as indicated in Fig. 2. The yield and tensile strength of hot band were compared to the batch annealed in Fig. 3a and 3b, respectively. The chosen batch annealing temperature was optimized to retain the precipitates size and distribution from the hot mill.

### Figure 5



Optical (a) and scanning electron (b) micrographs of batch-annealed Ti-HSLA steel showing nearly full recrystallized microstructures.



Oct

SEM Ti-HSLA steel annealed

# 78 Technical Article

### Table 4

## Hole Expansion Ratio Testing Results Performed on Annealed HSLA Sheet Steels



### Figure 6

3-mm-thick hot-rolled and 1.5-mm Ti-HSLA coldrolled batch-annealed steel sheet blanks after hole expansion tests showing fracture initiation sites.



### **Microstructure**

Fig. 4 shows through-thickness microstructures of hotrolled Ti-HSLA steel along the rolling direction. Ferrite grains indicated some non-polygonality due to accelerated cooling from finishing temperature. Fig. 5 reveals microstructures of fully processed Ti-HSLA steel after batch annealing. Ferrite grains were much refined and showing near completion of recrystallization. Scanning electron images indicated further clarity on the near completion of recrystallization and onset of ferrite grain growth.

## Formability Evaluation: Hole Expansion Ratio **Results**

Table 4 lists the HER values obtained for both the Ti-HSLA steel in hot-rolled and batch-annealed conditions. HER data from three locations across the width were obtained and an average of at least three samples from each location are summarized. As indicated from

### Figure 7

Optical micrographs of box annealed steel at 580°C (a) and 640°C (b).



(a) (b)

Figure 8

Grain boundary mapping constructed from orientation imaging from electron backscattered diffraction (EBSD). Blue lines represent high-angle grain boundaries with critical misorientation of 15° or red lines representing low-angle boundaries with misorientation of 2 to 15°.





exceeding 1° or differing in phase is considered a boundary (total number =  $462862$ , total length =  $2.67$  cm)

the results, outstanding HER values were obtained at the hot-rolled stage and were preserved after batch annealing. The values are very uniform across the width of the coil. The HER values are indicative of excellent edge ductility or stretchability of the steel and is expected to perform well during stamping operations. The fracture initiation sites during the hole expansion tests are shown in Fig. 6. The punched holes retained their circular configuration after piercing. The cracks developed in both the rolling direction as well as the transverse direction, indicating complete recrystallization of the ferrite grains at the hot-rolled stage. The cracks for batch-annealed sample developed in one direction as the recrystallization was near completion, as seen in Fig. 6.

## **Discussion**

Ti-HSLA steel was selected with lean alloy compositions which yielded a yield strength minimum of 550 MPa in cold-rolled batch-annealed condition in laboratory simulation and was successfully replicated in production. Ti-HSLA steel represented a fully recrystallized microstructure after finish deformation rolling and was manifested in final ferrite grain structure after hot rolling. Hot rolling processing parameters were optimized for achieving maximum precipitation strengthening in the hot-rolled coils.<sup>6</sup> Batch annealing parameters after cold rolling were optimized to attain near full recrystallization but stopped before ferrite grain growth commences.

Softening behavior of cold-worked samples as studied through batch annealing simulation (Fig. 1) indicated an operable temperature window of 580–630°C for the selected alloy composition. Beyond this temperature, ferrite grain coarsening was observed and a substantial loss in strength was detected. Box-annealed microstructures at 610°C and 640°C are shown in Figs. 7a and 7b, respectively. It can be observed on the microstructure at 640°C that the recrystallization is complete and grain growth has started.

Studies of texture evaluation and grain boundary orientation of ferrite grains were carried out using EBSD. Fig. 8 shows the grain boundary mapping of batchannealed Ti-HSLA steel constructed from orientation imaging indicating significant fraction of high-angle grain boundary (HAGB) (~70%) revealing near complete recrystallization. Grain size distribution as estimated from grain boundary mapping is plotted in Fig. 9 and an average ferrite grain size of 3.5 mm was found for the batch-annealed sample which was 4.8 mm at the hotrolled stage.

The Ti-HSLA steel revealed increasing γ-fibre with the presence of some α-fibre as well (Fig. 10). The γ-fibre showed significant intensity concentration or maximum at {111}<112> positions, albeit non-uniform along the γ-fibre location. The ferrite grain boundary orientation indicated in Fig. 8 revealed a mixed grain structure which is probably the reason for not obtaining a sharp and uniform γ-fibre texture.<sup>7</sup> Sharper γ-fibre texture is typical of extra deep drawing quality steels with a coarser and equiaxed ferrite grain structure.

Precipitation studies revealed significant nanosized TiC precipitates (1.5 to 8 nm) in Ti-HSLA steels strongly contributing toward the yield strength increment.4 The precipitates that formed after hot rolling remained mostly

## 80 Technical Article

### Figure 9

Grain size distributions of hot-rolled and cold-rolled batch-annealed Ti-HSLA sheet steel sample as calculated using grain boundary mapping.



intact in final batch-annealed steels as the size and distribution of fine precipitate did not change during recrystallization annealing. The batch annealing temperature was chosen to retain these TiC size and distribution and through partial recrystallization.

Hole expansion ratio data obtained in the current batch-annealed steel indicated superior stretchability or edge ductility suitable for most stamping operations. In general, the sheet steel indicated high HER values because of internal cleanliness, finer ferrite grain size and nanoprecipitates. It is interesting to note that the HER values and tensile properties of this steel makes them immensely attractive for autobody stamping applications requiring high stretch ductility. Successful stamping performance of this Ti-HSLA steel has been assessed and can be attributed to the mechanical and formability results. An example of stamped part (seat cushion front link) is shown in Fig. 11.

### Summary

A low-carbon, lean Ti-bearing alloy was chosen to develop ferritic HSLA 550 sheet steel in cold-rolled batchannealed condition. Strengthening in the final sheet steel was intended primarily through partial recrystallization of ferrite grains and TiC precipitation strengthening with moderate solid solution strengthening. Precipitation of TiC nanoprecipitates in hot-rolled steel was induced through proper control of finishing deformation temperature, cooling rate and coiling temperature. A batch annealing cycle appropriate of the current steel was evolved through presimulations to decide full recrystallization annealing temperature. Actual mill production of sheet steels of various thicknesses revealed HSLA 550 steels could be successfully developed with excellent elongation and strain hardening index using a lean Ti-only HSLA approach. Studies of microstructure revealed the controlled batch annealing produced nearly complete recrystallized very fine ferritic microstructure. Outstanding hole expansion ratio values of more than 50% were obtained for 1.5-mm-thick sheet steels. Nanosized TiC precipitates, fine recrystallized grain size and excellent internal cleanliness contributed to the HER values. Critical automotive components were successfully stamped and more customer orders accepted based on performance results.

### Figure 10

Orientation distribution function of batch annealed T09 steel showing increasing  $\gamma$ -fibre orientations.



Texture Name: Harmonic Texture Calculation Method: Harmonic Series Expansion Series Rank (I): 16 Gaussian Smoothing: 5.0\* Sample Symmetry: Triclinic Representation: Euler Angles (Bunge)  $max = 8.444$  $-5.917$ 4.147 2.906 2.036 1,427  $-1.000$  $-0.701$ Constant Angle: 62  $\bullet$   $\varphi$ l (0.0°-90.0°)  $\Phi$  (0.0°-90.0°)

### Figure 11

HSLA 550 XF end use seat cushion front link.



## Acknowledgments

Authors are thankful to all employees of Big River Steel – A U. S. Steel Co. for their support and motivation for the successful development of the HSLA 550 batch-annealed

sheet steel. The authors are grateful to the management of Big River Steel for full support for the commercial development of this steel.

*This article is available online at AIST.org for 30 days following publication.*

## **References**

- 1. SAE J2340, "Categorization and Properties of Dent Resistant, High Strength, and Ultra High Strength Automotive Sheet Steel," 2017.
- 2. ASTM A1008, "Standard Specification for Steel, Sheet, Cold-Rolled, Carbon, Structural, High-Strength Low-Alloy, High-Strength Low-Alloy With Improved Formability, Solution Hardened, and Bake Hardenable," 2018.
- 3. GMW3032, 10th Edition, "High Strength Sheet Steel, 180 MPa Through 700 MPa Yield Strengths," General Motors Worldwide Standard, April 2019.
- 4. S. Biswas, B. Chen, S. Bhartiya, V.S.Y. Injeti, M. Sexton and A. De, "Development of Nanoprecipitation-Strengthened Cold-Rolled Batch Annealed HSLA Sheet Steels (>490MPa Yield Strength) With Superior Formability," *Iron & Technology,* Vol. 19, No. 10, 2022, pp. 60–72.
- 5. D. Roy, A.K. Gupta, M.S. Alam, S. Srikanth and B.K. Jha, "Enhancement of Properties of Micro-Alloyed Low-Carbon Ni-Added Steel by Thermomechanical Treatment," *Journal of Materials Engineering and Performance,* Vol. 29, No. 12, 2020, pp. 7952–7963.
- 6. C. Mahimkar, V. Natarajan, V.S.Y. Injeti, J. Martin, D. Hennessy and A.K. De, "Fundamentals of Alloy and Processing Design for the Successful Production of Ferritic, TiC-Strengthened Ultrahigh-Strength 100 ksi (Yield Strength) Hot-Rolled Steel with Low-Temperature Toughness Through a Flex Mill," *AISTech 2021 Conference Proceedings,* 2021, pp. 900–911.
- 7. F. Boratto, R. Barbosa, S. Yue and J.J. Jonas, "Effect of Chemical Composition on Critical Temperature of Microalloyed Steels," Proceedings of Thermec 88, Tokyo, Japan, 1988, pp. 383-390.



This paper was presented at AISTech 2024 — The Iron & Steel Technology Conference and Exposition, Columbus, Ohio, USA, and published in the AISTech 2024 Conference Proceedings.

