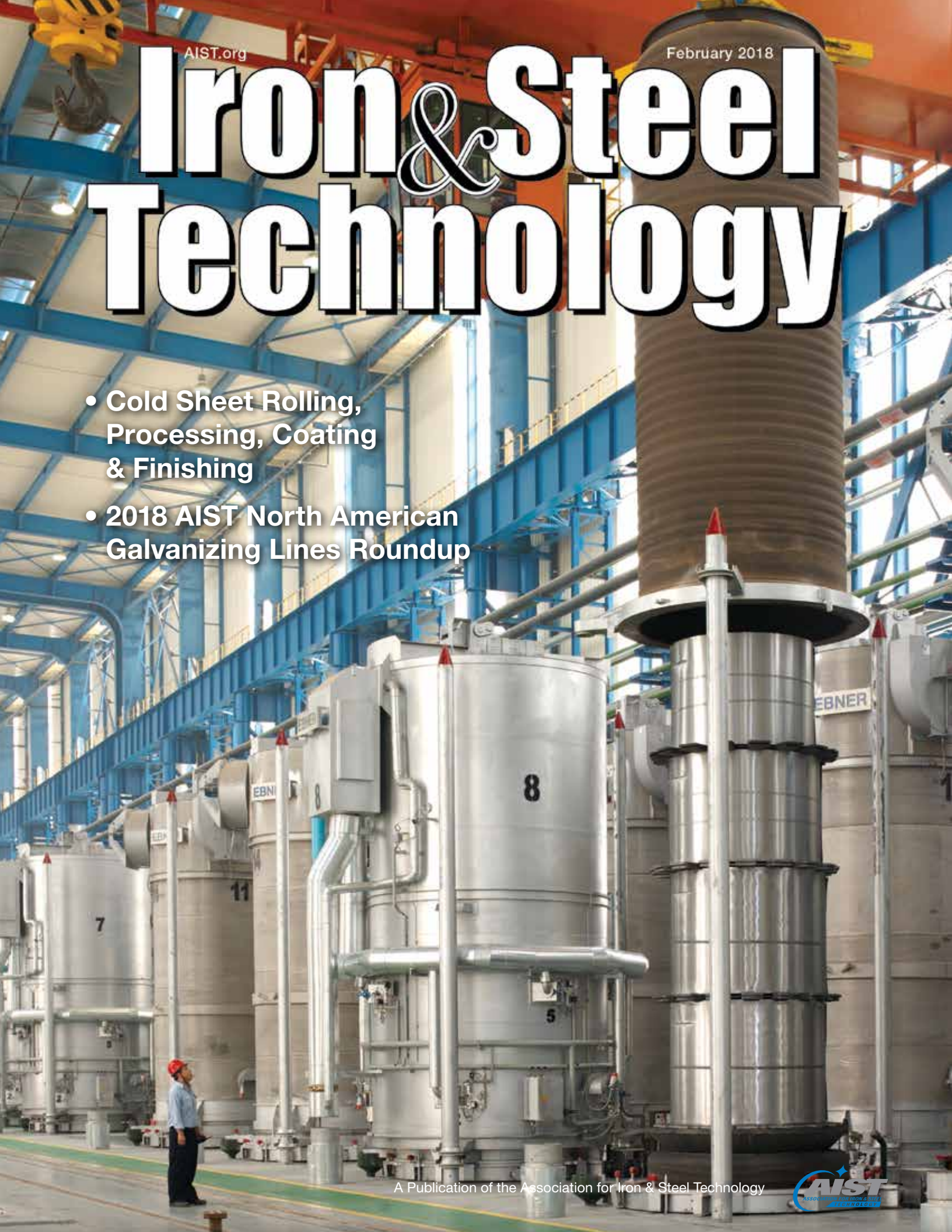


Iron & Steel Technology

- Cold Sheet Rolling, Processing, Coating & Finishing
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Improved Descaling Method for Advanced High-Strength Steels (AHSS) Using Fused Conditioning Salt

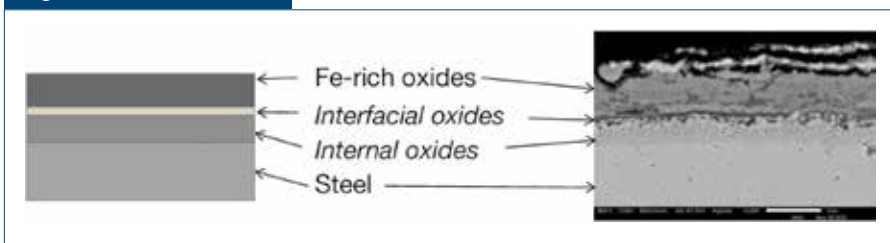
Third-generation advanced high-strength steels (AHSS) contain significantly high quantities of alloying elements. Many of these alloying elements create oxides in the surface scale of hot band that exhibit poor solubility in traditional pickling baths. The work presented here examines hot band samples of various quench and partition grades of steels and the effect of adding a fused salt conditioning step to the descaling process to both reduce the overall pickling time required and to improve the final surface quality of the metal. Also presented here are conceptual design elements for a production line possessing fused salt conditioning.

Historically, carbon steel has possessed less than 2% of alloying elements, and in many cases, the total alloy content has been less than 1%. This means the bulk of the mill scale formed is iron oxide, which is readily soluble in hot hydrochloric acid (HCl_{aq}). Recently, the demand for lighter-weight and more fuel-efficient vehicles has required steel manufacturers to offer new grades with ever-increasing strength. In order to retain a high degree of formability and have the required strength, the newest grades of advanced high-strength steels (AHSS) require levels of alloying elements in excess of 2%. Some of the elements used include manganese (Mn), silicon (Si), chromium (Cr), aluminum (Al) and titanium (Ti), among others.¹ Many of the oxides of the aforementioned

elements create problematic surfaces that are not easily cleaned in hydrochloric acid pickling baths.^{2,3} Moreover, oxides of most alloying elements tend to concentrate at the steel/scale interface, producing a film that is difficult for hydrochloric acid to penetrate. They may also appear as submerged oxides inside the steel matrix, just below the surface; again standard hydrochloric acid can remove these internal oxides but only with difficulty, if at all. The complex scale structure is depicted in Fig. 1.

Molten or fused salts historically have shown a significant appetite for the oxides of non-ferrous elements.⁴ This characteristic of molten salt has been used to a great extent in the conditioning and pickling of scale formed on stainless steel, Hastelloy, Inconel and other high-alloy steels.

Figure 1



Sketch of typical advanced high-strength steel (AHSS) scale layers, showing the interfacial and internal oxides of alloy elements absent in mild steel scales.



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In particular, various grades of stainless steel that have been annealed in open-atmosphere furnaces often go through molten salt immersion prior to pickling.⁵ It was the interest of this research to examine if using molten salt would improve the descaling of AHSS. Integrating a molten salt bath into a pre-existing carbon steel line, where the majority of the product mix might not be AHSS, is thought to be unfavorable. To ease integration and provide flexibility in line configuration, a process was envisioned where the salt would be applied as an aqueous solution. The strip would be heated to drive off the excess water, fuse the salt and drive the oxide dissolution. This proposed process presents several variabilities for process refinement including salt temperature, heat-up rate, pickling duration and order of pickling deemed to be the most prominent. Presented here are the initial findings and process refinement for the use of molten salt in the descaling of AHSS.

Experimental Procedure

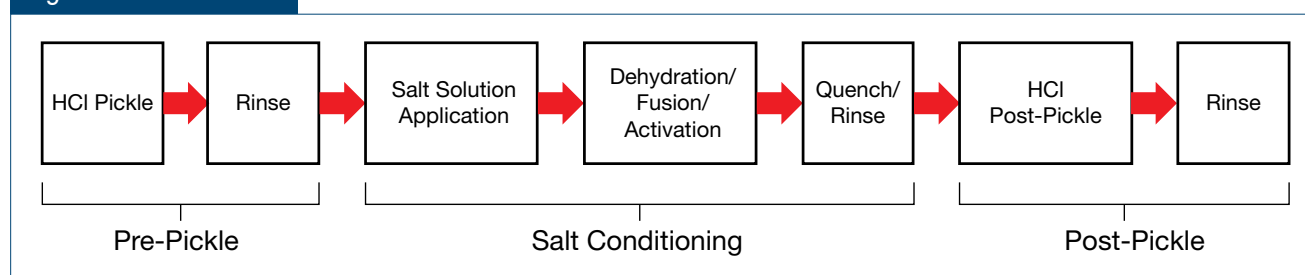
Steel samples were supplied by ArcelorMittal and were taken from industrially produced hot band coils. The AHSS had a tensile strength of 1,470 MPa and was alloyed with significant levels of silicon and manganese. The samples measured 100 mm x 200 mm. The pickling liquor was made by diluting 20% technical grade hydrochloric acid obtained from PVS-Nolwood (Detroit, Mich., USA), without inhibitor. Ferrous chloride was added to the diluted hydrochloric acid to give a “working bath” iron content. Typical pickling bath composition contained 190–200 g/l total chloride with 80–100 g/l iron. The pickling solution was heated on a hot plate and thermostatically controlled

to be 185°F. The salt is a proprietary mixture of halide, alkaline and oxidizing salts. The salt mixture was provided as an aqueous solution from Kolene as the commercial product Oxigen-X™. The salt was applied by dipping the whole sample into a container of the solution. The solution was dehydrated to deposit the salt, then fused, and finally activated by using a Lab-Line laboratory oven (Model 4232) pre-set to different temperatures. The final temperature of the conditioning salt was controlled by the oven dwell time. After the coupons were conditioned with the fused salt, the panels were quenched in cold water, any remaining salt was rinsed off, and the coupon was dried prior to the next step. These steps are summarized in the process flow diagram seen in Fig. 2.

The weight of each sample was measured before treatment and then after treatment. The resulting weight loss was normalized by dividing the change in weight by the total initial surface area; the weight loss was then reported as g/m². Most of the tests were run in duplicate to assess variability in the system. The first sample for a given test was labeled with an A and the second sample was labeled with a B. The results for both A and B samples are reported in Table 1 and are indicated by the added A/B label. The surface profilometry was only measured on one of the coupons.

Scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDS) were performed by ArcelorMittal in East Chicago, Ind., USA. Planar view SEM analysis of pickled samples was performed on a JEOL 6060 LV Scanning Electron Microscope using 20-kV acceleration voltage. EDS analysis was carried out via NSS Version 3 software provided by Thermo Scientific Inc. with the same 20-kV acceleration voltage. Surface profilometry was carried out

Figure 2



Displayed is the process flow diagram for the studied system. The system can be broken down into three different stages: pre-pickle, salt conditioning and post-pickle. Within each of these stages, different variables were tested to optimize the system.

using a Nanovea PS50. Scans were performed with lateral dimensions of 4 mm x 4 mm, with X and Y indexing step size of 20 μm and 10 μm , respectively, and an XY resolution of 1.7 μm . A chromatic confocal optical pen was used with a Z measuring range of 300 μm . Scans were made over 10–11 minutes with a scan velocity of 10 mm/second and an acquisition rate of 1,000 Hz. The resulting data was analyzed with Expert Mountain Surface 3D Analysis software.

Results and Discussion

To study the effect of molten salt conditioning on the scale described above, an experimental matrix of 14 samples was established to test the most pertinent variables of the system. The experimental parameters of the matrix are given in Table 1. The matrix was further broken down into five different phases. Phase 1 studied the effects of pickling only. The second phase studied pickling, then salt conditioning at different temperatures. Phase 3 studied pre-pickling, salt conditioning and then post-pickling with varying salt temperatures. Phase 4 adjusted the pre- and post-pickling times. Finally, Phase 5 studied the effect of the heat-up rate for salt conditioning.

An initial means of determining the effectiveness of the pickling process was done by visual inspection and by measuring weight loss. Surfaces that appeared to be light gray and uniform in appearance were believed to be clean. Also, it was assumed that the more scale removed, the greater the weight loss would be, and therefore, the samples with the greatest weight loss had the cleanest surfaces. The final appearance and weight loss for the samples are reported in Table 1. More precise measurements were made with SEM and EDS to determine the ultimate surface cleanliness, and these results are reported later in this paper.

Initial testing in Phase 1 was done to determine the effect pickling alone had on scale removal. This was accomplished by stepping samples 1A/B to 4A/B through progressively longer pickling dwell times. The visual results for 1B, 3B and 4A are pictured in Fig. 3. Ten seconds of pickling time is clearly insufficient for removing scale as the surface of sample 1B can be seen with a large amount of discolored scale remaining on the surface. When the steel has been pickled for 30 seconds, as seen for sample 3A, the surface starts to become uniformly gray in color, however there are still significant amounts of dark gray streaks that can be seen with the naked eye. By the time the steel had been pickled for a full 60 seconds, the

Table 1

Parameters and Results From the Testing Matrix Used to Evaluate the Variables Involved With Alkali Salt Conditioning (Oxygen-X) of the Mill Scale Found on AHSS

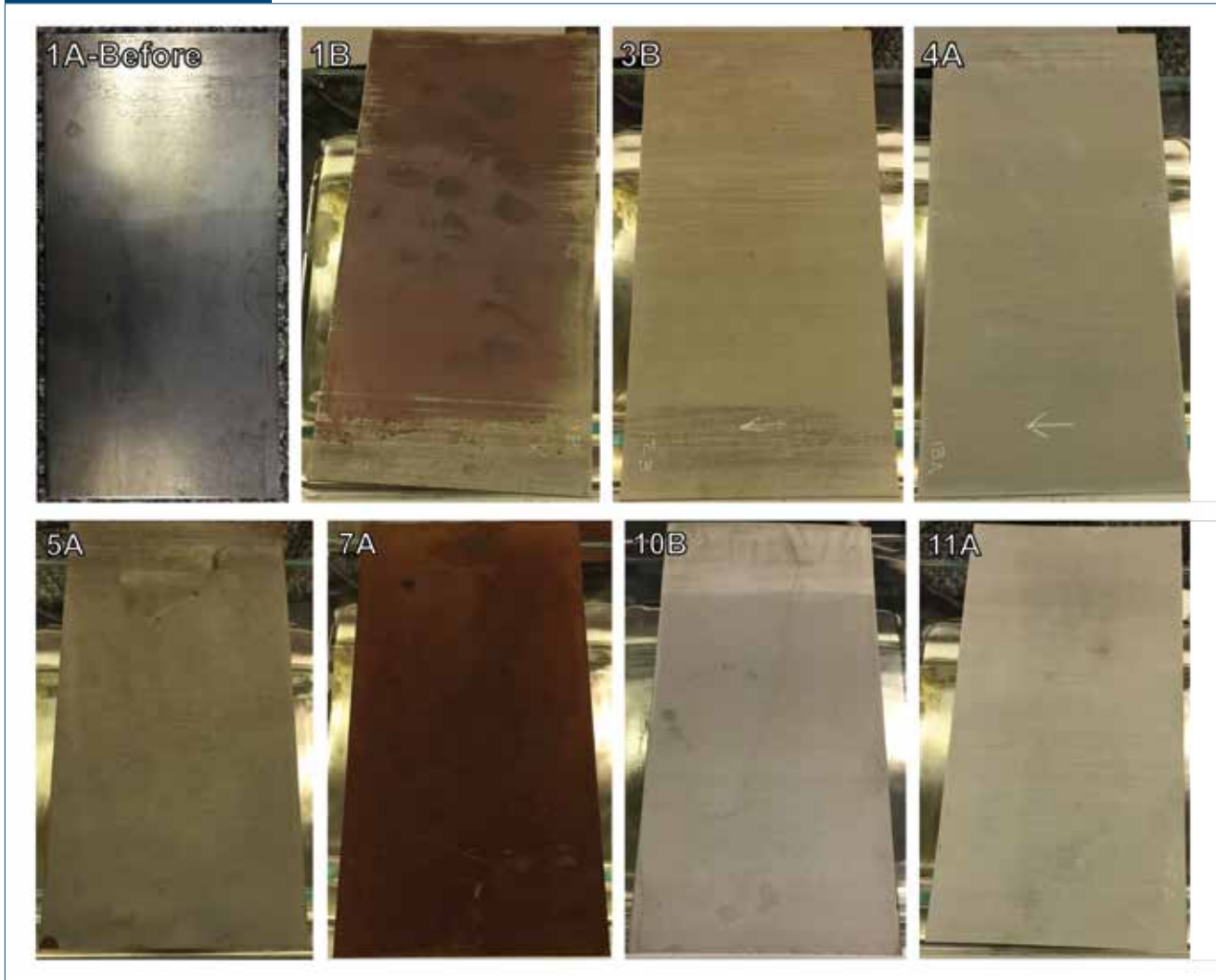
Test phase	Sample	Pre-pickle time (s)	Oven temp. ¹ (°C)	Final coupon temp. ² (°C)	Post-pickle time (s)	Appearance	Weight loss (g/m ²)	Ra (μm)
1	1A/B	10	—	—	—	Rusty	24.6 \pm 1.3	0.794 \pm 0.402
	2A/B	20	—	—	—	Light streaks	30.7 \pm 0.9	0.792 \pm 0.430
	3A/B	30	—	—	—	Light streaks	33.1 \pm 0.2	0.822 \pm 0.480
	4A/B	60	—	—	—	Light streaks	37.3 \pm 1.7	0.834 \pm 0.616
2	5A/B	20	540	230	—	Dark	38.2 \pm 1.0	0.801 \pm 0.603
	6A/B	20	540	345	—	Rusty	29.0 \pm 2.7	0.839 \pm 0.584
	7A/B	20	540	455	—	Rusty	23.8 \pm 0.7	2.500 \pm 0.673
3	8A/B	20	540	230	20	Clean	43.7 \pm 2.5	—
	9A/B	20	540	345	20	Clean	58.8 \pm 4.1	1.290 \pm 0.491
	10A/B	20	540	455	20	Clean	64.5 \pm 2.6	1.290 \pm 0.508
4	11A/B	20	540	230	10	Clean	41.1 \pm 1.2	1.332 \pm 0.910
	12A/B	—	540	345	20	Light streaks	32.2 \pm 0.4	1.239 \pm 0.605
5	13A/B	20	400	345	20	Clean	62.4 \pm 2.3	1.261 \pm 0.505
	14A/B	20	815	345	20	Clean	57.4 \pm 3.0	—

¹Where: 400°C = 750°F; 540°C = 1,000°F; 815°C = 1,500°F.

²Where: 230°C = 450°F; 345°C = 650°F; 455°C = 850°F.

— Indicates step was not conducted or measurement was not made.

Figure 3



Images of the various coupons tested. The first coupon, 1A, shows the AHSS samples in their “as-received” condition with the original mill scale still present.

surface started to become completely uniform, with the dark streaks seen on sample 4A becoming difficult to distinguish.

For the first four samples, where only HCl_{aq} was used, a non-linear correlation was observed between amounts of material loss versus the time in the pickling bath. Sample 1A/B had a weight loss rate of 2.46 (g/m^2)/second; however, this rate dropped to 0.62 (g/m^2)/second for sample 4A/B. This is attributed to the reduction in the amount of iron oxide ($\text{FeO}/\text{Fe}_3\text{O}_4$), which is rapidly soluble in HCl_{aq} . At the same time, as the coupon was being pickled, the less-soluble refractory oxides, formed from the alloying elements, were exposed on the surface.

Samples 5A/B, 6A/B and 7A/B composed the second phase of testing. These samples were pickled for 20 seconds and then treated with the fused salt (Oxygen-X). With the oven temperature being kept at a constant 540°C , the required dwell time increased to obtain a fused salt temperature of 455°C versus 230°C . There was a noticeable difference between these samples after salt conditioning and quenching. The sample conditioned at 230°C appeared dark gray with some light streaks (see image 5A in Fig. 3). However, the samples heated above 230°C had reddish-brown, rusty-looking surfaces. Sample 7A/B, which was processed to a final temperature of 455°C , appeared the darkest and heaviest. A picture of 7A can be seen in Fig. 3. Though sample 7A/B was

processed to a higher temperature of 455°C, it only displayed 62.3% of the weight loss that sample 5A/B (230°C) had shown. Though not fully characterized, the reddish-brown appearance of 7A/B suggests that the surface is heavily populated by Fe_2O_3 , FeOOH , and other oxyhydroxides.⁶ It is then believed that the weight loss due to alloy removal was significantly counteracted by the addition of mass added in the form of oxygen, which was present in the $\text{Fe}_2\text{O}_3/\text{FeOOH}$ rich surface.

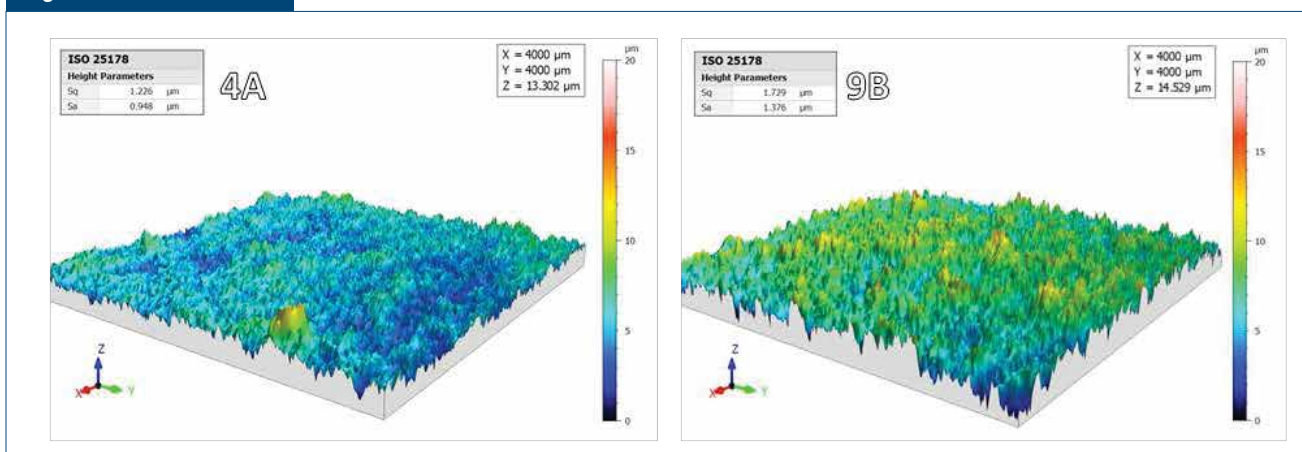
Phase 3 was composed of samples 8A/B, 9A/B and 10A/B, and was used to study the addition of a second post-pickling step. Again, the oven temperature was kept at a constant 540°C, and the samples were left in at progressively longer times to obtain a final salt temperature of 230°C, 345°C and 455°C. Both the pre- and post-pickling steps lasted 20 seconds. These samples showed a significant increase in the amount of material removed and reversal in the weight loss trend seen in Phase 2. Sample 10A/B, which had experienced the highest temperature, displayed the greatest weight loss of 64.5 g/m². Sample 10A/B had a 147.6% increase in weight loss when compared to sample 8A/B, which had been heated to 450°F. When compared to sample 7A/B from Phase 2, which had been heated to 450°F also, sample 10A/B from Phase 3 had 271% greater weight loss. Some of the increase in the weight loss of Phase 3 samples over Phase 2 samples can be attributed to having experienced twice the amount of pickling, yet the weight loss

experienced by all of Phase 3 was greater than sample 4A/B from Phase 1, which had spent a full 60 seconds in a pickling bath with no salt conditioning.

To further test the effect of a multiple pickling regime, Phase 4 varied the pre- and post-pickling times for samples 11A/B and 12A/B. For sample 11A/B, the amount of time spent in the second pickle bath was cut in half to 10 seconds, which resulted in a total pickling time of 30 seconds. Both sample 11A/B from Phase 4 and sample 8A/B from Phase 3 were heated to 230°C during the salt conditioning step; yet sample 11A/B only experienced a 5.9% reduction in weight loss despite being pickled for three-fourths of the time of sample 8A/B. This would suggest that pre-pickling has a stronger effect than does the post-pickling on final surface cleanliness and pickling rate. Sample 11A, pictured in Fig. 3, showed an extremely uniform light gray surface. To further assess the effect of pre- versus post-pickling, sample 12A/B was processed with no pre-pickling but with a post-salt-treatment pickling time of 20 seconds. Sample 12A/B had an increase in weight loss when compared to 6A/B from Phase 2, which was also heated to 345°C but only pre-pickled. Despite this increase in weight loss, the effect of only doing a post-pickle is less than the effect of splitting the pickling time before and after conditioning.

Additional tests were done to determine if the heating rate had a significant effect on the material removed from salt conditioning. This was

Figure 4



Shows 3D renderings of the profilometry measurements made on samples 4A and 9B.

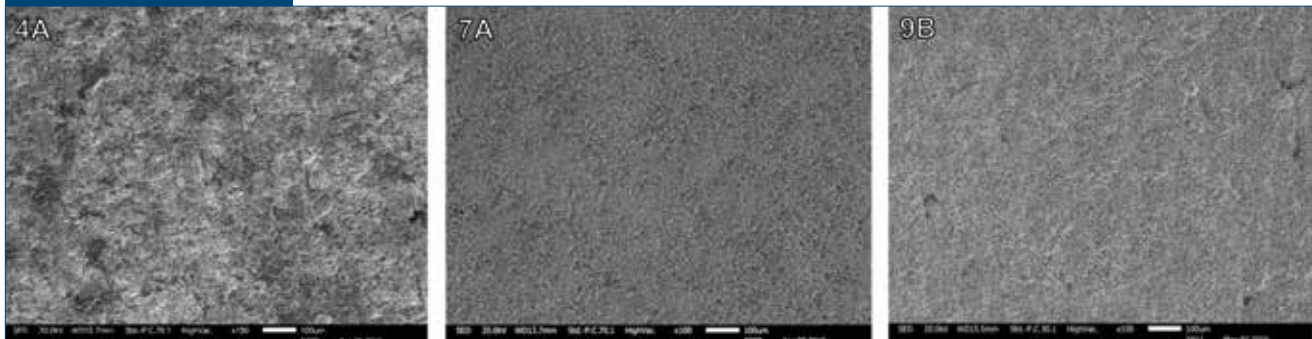
accomplished with Phase 5, which kept the final salt conditioning temperature, pickling times and pickling order consistent, but changed the oven temperature. The results can be seen by observing the measurements made for samples 13A/B, 9A/B and 14A/B, which in this given order, the oven temperature was increased from 400°C to 540°C to 815°C. There does appear to be a decreasing trend in material loss going from low oven temperature to higher oven temperature or, in other terms, low heat-up rates to high heat-up rates. When one looks at the differences in the measurements between 62.4 g/m² for 13A/B (400°C oven temperature/345°C salt temperature) and 57.4 g/m² for 14A/B (815°C oven temperature/345°C salt temperature), the result does not appear to be as significant as the result for samples that had a constant oven temperature but increasing surface temperature. For example, when one compares the previous results to the difference between 43.7 g/m² for 8A/B (540°C oven temperature/230°C salt temperature) and 64.5 g/m² for 10A/B (540°C oven temperature/455°C salt temperature). Therefore, the final conditioning temperature of the molten salt has a stronger influence on the amount of material removed than the heating rate.

Profilometry measurements were made in order to quantify any differences in surface texture between material that had only been pickled (Phase 1) and material that had been treated with salt and pickling (Phases 2 and 3). The results of these measurements are reported in Table 1 as Ra values. In the first four samples, which had only been pickled, the Ra values ranged from 0.792 μm to 0.834 μm. There was a general trend toward larger Ra values as the pickling

time increased. A similar result was observed with samples 5A/B, 6A/B and 7A/B, which were pickled and then treated with fused salt; though sample 7A/B showed a significant jump in Ra value, this was most likely due to the uneven growth of Fe₂O₃ on the surface of the sample. Consistently higher Ra values, ranging from 1.239 μm to 1.332 μm, were measured for those samples that were treated with a pre- and post-pickling step (samples 9A/B through 13A/B in Table 1) as compared to samples that were pickled only. Representative images of the profilometry measurements can be seen in Fig. 4, plotted in a three-dimensional projection. Sample 9B appears to have a number of “islands,” which may represent material that was resistant to both acid and salt. Even though there were measurable differences between Phase 1 samples and Phase 3 samples, their differences fall within the statistically measured variance and don’t represent a statistically significant difference in roughness. When observed macroscopically, pickled only and pickled/salt/pickled treated samples showed little difference.

SEM with EDS analysis was used to further explore the surface of the samples. Representative SEM images are displayed in Fig. 5, and the EDS measurements are summarized in Table 2. EDS measurements were considered to be semi-quantitative and used to compare the different surfaces, but are not thought to accurately represent the true surface compositions. Samples 1A, 2A, 3A and 4A, which were only pickled with HCl_{aq}, displayed patchy surfaces with dark localized regions. These dark regions can be clearly seen in the image of 4A, shown in Fig. 5. EDS analysis of the overall surfaces showed oxygen present in the range

Figure 5



Resulting scanning electron microscopy (SEM) images of AHSS samples that were treated through various pickling regimes. Sample 4A was pickled for 60 seconds. Sample 7A was pickled for 20 seconds and conditioned with fused salt at 455°C. Sample 9B was pre- and post-pickled for 20 seconds and conditioned with salt at 345°C.

Table 2

EDS Results From the Testing Matrix, Grouped by Treatment (pickle only Oxygen-X with pre-pickling, Oxygen-X with post-pickling, or three-stage process). EDS measurements are semi-quantitative and not thought to represent actual surface composition.

Phase	Sample		EDS chemistry, overall surface		EDS chemistry, oxygen-rich areas*	
			O	Si	O	Si
			Wt. %	Wt. %	Wt. %	Wt. %
Pickle only	1A	Side 1 (S)	8.1	3.7	10.4	4.2
		Side 2 (R)	19.6	2.1	(No such areas)	
	2A	Side 1 (S)	5.9	3.5	(No such areas)	
		Side 2 (S)	8.1	4.0		
	3A	Side 1 (S)	6.5	3.5	11.1	3.5
		Side 2 (S)	9.3	4.7	13.9	4.9
	4A	Side 1 (S)	7.7	4.7	9.4	5.3
		Side 2 (S)	8.0	4.9	11.4	5.7
Pre-pickle + Oxygen-X	5A	Side 1 (D)	3.2	1.6	17.3	0.9
		Side 2 (D)	2.4	1.4	13.1	1.3
	6A	Side 1 (R)	27.9	1.2	(No such areas)	
		Side 2 (R)	27.0	1.0		
	7A	Side 1 (R)	29.2	1.1	(No such areas)	
		Side 2 (R)	30.1	1.3		
Oxygen-X + post-pickle	12A	Side 1 (S)	8.8	4.2	12.8	5.3
		Side 2 (S)	8.0	3.6	8.4	3.9
Pre-pickle + Oxygen-X + post-pickle	8A	Side 1 (C)	0.0	3.2	(No such areas)	
		Side 2 (C)	2.5	2.6		
	9B	Side 1 (C)	1.9	1.8	(No such areas)	
		Side 2 (C)	1.4	2.1		
	10A	Side 1 (C)	0.8	1.9	6.7	1.6
		Side 2 (C)	1.2	1.8	11.7	1.5
	11A	Side 1 (C)	0.0	1.4	(No such areas)	
		Side 2 (C)	0.9	1.9		
	13A	Side 1 (C)	2.0	2.0	(No such areas)	
		Side 2 (C)	1.4	2.1		

(C) = Clean, (D) = Dark, (R) = Rusted, (S) = Streaks

*Dark areas in SEM images, on samples where these were seen

of 6–9%, with one side of panel 1A showing an elevated oxygen level of 19.6%. Within the dark regions found on these samples, the oxygen content was in excess of 9.4% and as high as 13.9%. For samples that were only pickled, silicon was measured to be in the range of 2.1–4.9%, with concentrations increasing to 3.5–5.7% in the dark areas on the sample surface. Samples that were treated with a fused salt conditioning step showed very different surfaces depending on the order in which the conditioning took place. For example, sample 7A (pictured in Fig. 5), which was pickled and then treated with salt, showed a uniform surface but one that was dark brown in color. EDS analysis showed a 30% oxygen surface concentration, but despite this elevated concentration of oxygen, the sample only had concentrations of silicon in slight excess of 1%, which was lower than any other sample tested. Samples 8A through 11A, and 13A possessed very homogeneous light gray surfaces. The SEM image of sample 9B in Fig. 5 represents the typical surface observed for these samples. These samples had the lowest oxygen concentration, 0.0–2.5%, out of any of the samples examined with EDS. Also, the surface concentration of silicon on these samples was the lowest for any samples that did not have a rusty surface. The above observations illustrate that the three-stage process is the only one that gives clean, scale-free surfaces with no visible signs of rust and no alloy-rich areas (which would likely be unpickled oxides) on the surface.

Conclusions

An array of tests has been carried out on AHSS samples to evaluate the effect of using a fused salt conditioning step in the descaling process. It was determined that splitting the order of pickling into pre- and post-pickling steps provided the most benefit to increasing the overall pickling rate. The addition of a salt conditioning step also improved the final surface quality as EDS showed lower oxygen and alloying content at the surface when compared to samples that had been pickled alone. The final salt temperature was more important to maximizing scale removal

than the heat-up rate. Optimal salt conditioning temperature was found to be between 230°C and 345°C. Higher salt conditioning temperatures appeared to overcondition the samples and generated an iron oxide-rich surface that required increased secondary pickling to remove. When optimized, an added salt conditioning step can allow a clean, scale-free surface to be obtained with 30 seconds of pickling. This represents at least a 50% improvement in pickling time, as 60 seconds failed to yield a clean surface in an HCl_{aq} pickling bath.

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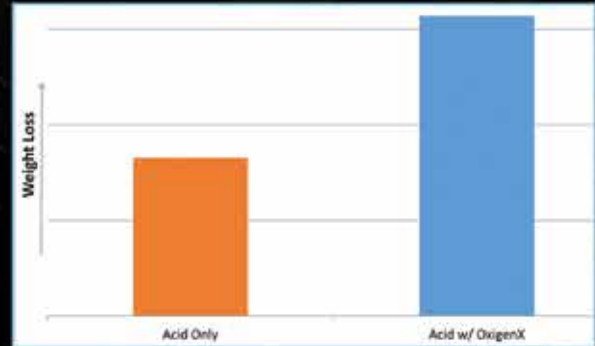


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