Deoxidation Process Change to Prevent Under-Riser Cracking Caused by Aluminum Nitrides on Heavy Sections

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Abstract. **A change of deoxidation procedure together with strictly following the thermal process avoided under-riser cracking caused by AlN in heavy section castings which were validated by fluorescent wet-mag particle method.**

Keywords—aluminum nitride, deoxidation, embrittlement, steel casting, heavy section, rock-candy, fracture, crack, grain boundary, segregation, heat, nitrogen, zirconium.

I. Introduction

Aluminum Nitride (AlN) embrittlement is the result of a solid-state precipitant in heavy section steel castings.

"For given temperature the inclusion forms, if the solubility product [Al] [N], which follows from the equilibrium constant of reaction" (D. Kalisz and S. 64) [1]:

$$[Al] + [N] = (AlN) \tag{1}$$

Embrittlement can lead to failure of sections with little loading, even during assembly. The AlN phase is a brittle phase which precipitates to primary grain boundaries. "The resulting fracture surface is dull, intergranular, and described as "rock-candy" fracture" (B. Allyn. 7, M. Svoboda. 1)[2,4]. It is also known that the AlN phase will not be dissolved by ordinary commercial heat treatment. "The two factors that control AlN embrittlement is the cooling rate and the composition" (C. Monroe And R. 27-28)[3]. Both the composition and cooling rate through proper rigging design should be used as tools for avoiding AlN embrittlement.

The cooling rate is strongly determined from the casting section size and the shape of the casting geometry. In a severe case the casting can have massive internal cracks in the riser area (both aluminum and nitrogen segregate) before it is even shaken out of the mold. "These AlN phases will always be found in much greater concentrations in the riser area" (W. Murphy. 6)[6].

"There is no guarantee of preventing rock candy fracture due to AIN formation if one controls the aluminum and nitrogen alone." (E. Daniel. 2)[5]. The following recommendations are made for prevention:

- Keep the nitrogen content as low as possible.
- Restrict aluminum additions to the minimum for complete deoxidation.

Deoxidation practice can contribute to grain boundary embrittlement (AlN). "In sections over 4 inches thick aluminum level must be kept low. Sections over 8 inches may require alternates to aluminum deoxidation" (C. Monroe And R. 27-28)[3].

"The AlN crack typically outlines the large prior-austenite grain boundaries" (R. Duncan. 47)[7] as shown in Figure 1.

Figure 1. Typical appearance of aluminum nitrides.

For castings under four inches thick, the cooling rate will allow aluminum levels in the range of 0.04% to 0.06% or higher. For castings between four inches and twelve inches thick, aluminum range of 0.01% to 0.04% can be safely used if an equal amount of a potent denitrifier such as Zirconium is also added. "Zirconium combines with nitrogen to form zirconium nitrides. These zirconium nitrides are relatively large blocky golden colored inclusions. They have a minimal effect on mechanical properties. (Denitrifier which form complex carbonitrides should be used with caution)" (W. Murphy. 6)[6]. The use of aluminum should be discouraged in castings which are greater than twelve inches thick. Castings over sixteen inches thick should have no aluminum added at any point in the melting process (B. Allyn. 7)[2].

II. Background

Once the rigging has been determined, control of the aluminum and nitrogen content in the steel is the best prevention of AlN embrittlement that is the main propose of this project. Due to the severe under riser cracks found by magnetic particle inspection MT (wet method) as shown in Figure 2 in hook castings identified as "Rock Candy / AlN" Acerlan had to change the deoxidation practice with lower levels in aluminum and supplemented with Zr. Acerlan also decided to reduce N from 100 PPM to 80 PPM maximum, to prevent this phenomenon to occur in our heavy section castings.

Figure 2. Example AlN cracking found in hook castings.

The selected alloy with AlN issues to validate the change of the deoxidation process was 4318 steel. The standard melting practice was in a 7.5 tons capacity electric arc furnace where N level was 100 PPM maximum. The deoxidation ranges were, aluminum between 0.020-0.060% and without additions of N stabilizers such as Zr.

III. Method

For this project, the alloy designed to validate the change of the deoxidation process was 4318-Zr melted ~~at~~ in a 7.5 tons capacity electric arc furnace that reduces the N in the arc by boiling in C of 0. 30 C as minimum for N less than 80 PPM[7]. The aluminum range was maintained between 0.015-0.035% and with additions of N stabilizers such as Zr. The zirconium range was from 0.04 to 0.12%.

The sulfur content was at maximum of 0.015% and the basic fusion practice was used. Castings followed the process described below:

A. Melting & Pouring

B. Shake out after 24 hrs temperature must be below 600°C [1110 F].

C. Heat treat: Normalize at 980°C [1796 F]

D. Burn & Arc at 250°C min [480 F] leaving ½" high riser contact

E. Quench & Temper. BHN=269-321

F. Inspection: MSS-SP-55/ MT

a) Craks or any other defect - Defect Maps needed

G. Weld Repair (if needed)

a) Preheat to 250°C [480 F], then all defects must be completely removed. The preheating temperature throughout the welding process was maintained.

b) PWHT at 640°C [1185 F] for 6 hrs.

H. Final NDT (Wet Mag) to validate no delayed cracks were found during the entire process.

I. Final Validation. No cracks were found validated by Define, Meassure, Analize, Improve, Control process (DMAIC).

A. Melting & Pouring

A completely killed in a 7.5 EAF melting practice tons with a boiling C of at least 30 points by oxygen injection was used to obtain less than 80 PPM of nitrogen content for a heat of 4318-Zr material. It is a typical alloy of the AISI 43XX series designation [Ni-Cr-Mo] with 0.18% C and "Zr" which requires zirconium deoxidation. The actual customer specification is ASTM A1001 Sec. Range 2 gr. B, see Table 1 with limits in N, 100 PPM Max and O, 90 PPM Max. The ladle additions were 1 kg/ton of Al, 2.6 kg/ton of FeSiZr. The pouring temperature was maintained at ~ 1590 ° C [2900 F]. A total of 8 castings were poured with a 14 inches riser diameter. See Figure 3. See mechanical properties in Table 2.

Alloy	C	Mn	Si	Ni	Cr	Mo	P	S	Al	OTHERS
4318-Zr	.16-.20	.95-1.15	.35-.60	1.8-2.0	.65-.85	.40-.45	<.020	<.015	.01-.035	N< .010 O< .009 Zr=.04-0.12

Table 1. Chemistry range for 4318-Zr material according to ASTM A1001 Sec. Range 2 Gr. B

		Chemistry grade	TS ksi		YS ksi		Elongation % Min		ROA % Min	
Sec. Range	**Class**		**T/8**	**3T/8**	**T/8**	**3T/8**	**T/8**	**3T/8**	**T/8**	**3T/8**
2	B	II		95	85	76	16	12	32	24

Table 2. Mechanical Properties 105according to ASTM A1001 Sec. Range 2

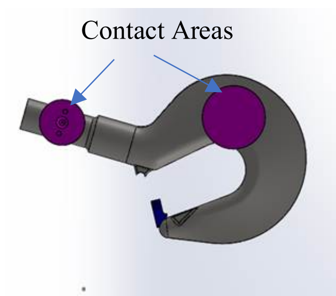

Figure 3. Contact areas. Part Description: 130 Ton Hook, Gross Weight 580 kg.

B. Shake Out

The castings were shaken out after 24 hrs. when they reached a temperature <600 ° C [1110 F].

C. Heat Treat

Normalize was performed at 980°C [1800 F] for 4 hours of soaking time with air cooling.

D. Burn & Arc (Riser Removal)

Minimum preheat to 250°C [400 F] leaving 1/2 inch of riser from the surface of the casting and then the castings were ground.

E. Quench & Temper

Quench: Castings were heated up to 955°C [1750 F] for 3.5 hours. then quench in water; the water temperature range was 35-45°C [95-110 F]. The transfer time to the water tank was <1 minute and held in the water for approximately 20 minutes.

Temper: at 660°C for 6 hrs. The cooling was in water.

BHN = 222-264.

F. MSS-SP-55/MT

Visual and MT inspection to corroborate that the cracks did not appear during Q & T.

G. Weld Repair

Just visual spec MSS-SP-55 repair was performed.

H. PWHT

At 640°C for 6 hours and air cool.

I. Final NDT

Kept for 36 hours after PWHT to validate there were no delayed cracks before shipment.

J. Final Validation

Final customer validation for problems related to cracks was performed after machining.

K. Results

Heat number 17T7B has been poured using Zirconium as deoxidant (4318-Zr). See Table 3 for Final Chemical analysis.

Pouring report:

- Pour Temperature: 1582°C [2880 F]
- Ladle Additions:
  1. Aluminum 5 kg
  2. Zirconium 15 kg
- Argon stir: 3 minutes
- 8 Hooks - 130 ton
- 4 Test Blocks

%	Al	N*	O*	Zr
Final	.033	.006	.010	.12

Table 3. Chemistry Results with Zirconium deoxidation.

N and O results with LECO analyzer.
See pour list in Table 4.

DESCRIPTION	GR	QTY	DATE	M. HEAT	HEAT CODE
TEST BLOCK 6X6X12	4318-Zr	4	04-11-2017	17T7B	A457
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A083
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A084
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A085
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A086
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A087
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A088
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A089
130 TON HOOK	4318-Zr	1	04-11-2017	17T7B	A090
		12		17T7B	

Table 4. Pour report heat: 17T7B

L. All castings were shaked out at <200°C.

M. The castings were loaded to the heat treatment furnace powered on with the door opened for 45 minutes to avoid direct thermal impact of the casting before the ramp up began. Then the furnace temperature was raised at 700°C for 2 hours and the castings held at 955°C for a soaking time of 4.0 hours and followed by air cool.

N. Castings should be pre-heated to avoid thermal cracks formed by local martensite in the riser contact areas. All castings were pre-heated at >250°C. Table 5 shows the pre-heat temperatures for burn and arc.

HEAT CODE	PRE-HEAT TEMP.BURN / ARC
A083	300/320°C
A084	390/330°C
A085	300/340°C
A086	340/360°C
A087	320/340°C
A088	330/350°C
A089	380/380°C
A090	250/300°C

Table 5. Preheat temperatures and MT results before Q&T

O. The castings were loaded to the heat treatment furnace powered on with the door opened for 45 minutes to avoid direct thermal impact of the casting before the ramp began. then it was directed at 700°C for 2 hours and then to 955°C, soak time of 4.0 hours and cooling with water tank. The water temperature was at the beginning of quech 35°C and at 45°C at the end of cooling, the castings were kept in the water for 20 minutes. The transfer time to the water tank was 45 seconds.

Temper: castings were charged to the heat treatment furnaces at a temperature below 300°C, then heated to 660°C for 6 hours and then cooled in the cooling tank to obtain the best mechanical properties. See Table 6 for mechanical test block results.

BHN	TS ksi		YS ksi		Elongation % Min		ROA % Min		Charpy -40°C, Av.	
	T/8	3T/8	T/8	3T/8	T/8	3T/8	T/8	3T/8	T/8	3T/8
228	105,832	100,677	89,883	84,779	23	19	43	36	39	42

Table 6. Mechanical properties from 6X6X12 inches test block from 17T7B heat

P. After riser removal and Q&T heat treatment castings were inspected by Magnetic Testing flouresent method, customer acceptance criteria: LEVEL II. covering 100% using heads and coil. See Figure 4 for inspection areas. After MT and visual inspections no AlN cracks under riser areas were detected after Q&T. See Figure 5.

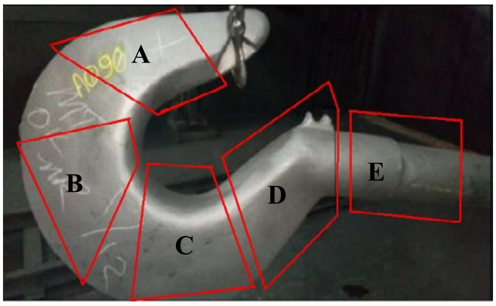

Figure 4. Areas to be inspected by MT

Figure 5. Photos of castings after visual and MT inspections, after Q&T. heat codes: A083, A084, A085, A086, A087, A088, A089, A090.

Q. During all weld repairs preheating to 250°C [480 F], throughout the welding process was maintained.

R. PWHT at 640°C [1185 F] for 6 hours was performed.

S. After 36 hours of PWHT, the castings were inspected as final validation before being shipped to the customer. See Figures from 6 to 13 for the fluorescent method photos of hooks castings. No cracks were found in these heat code castings: 6) A083, 7) A084, 8) A085, 9) A086, 10) A087, 11) A088, 12) A089, 13) A090.

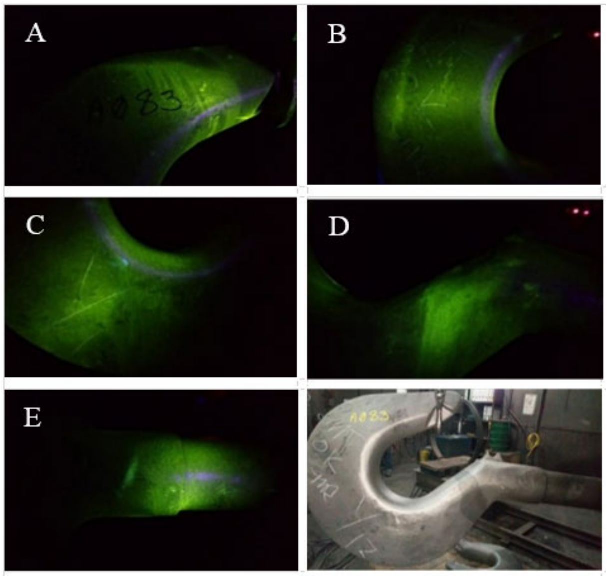

Figure 6. heat code: A083, Photos of castings after final MT. No cracks were found.

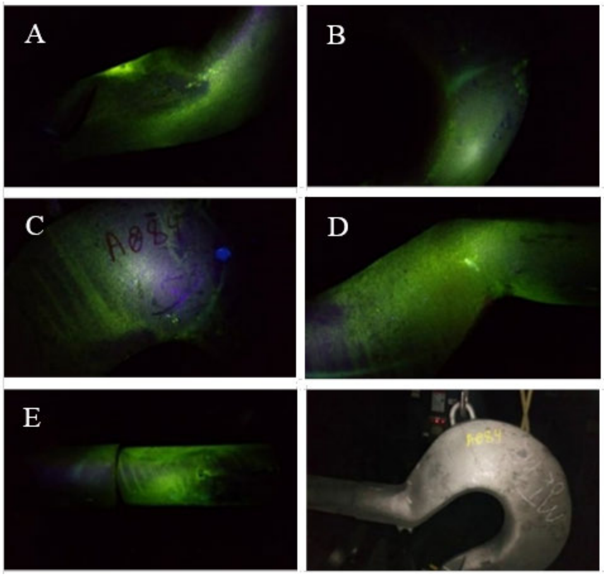

Figure 7. heat code: A084, Photos of castings after final MT. No cracks found.

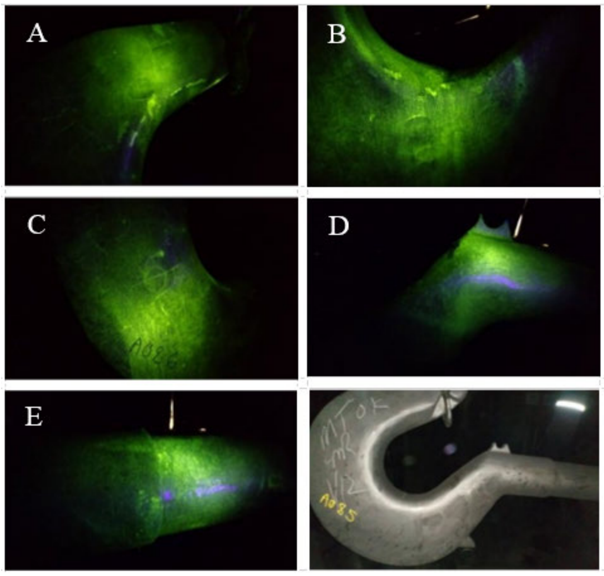

Figure 8. heat code: A085, Photos of castings after final MT. No cracks found.

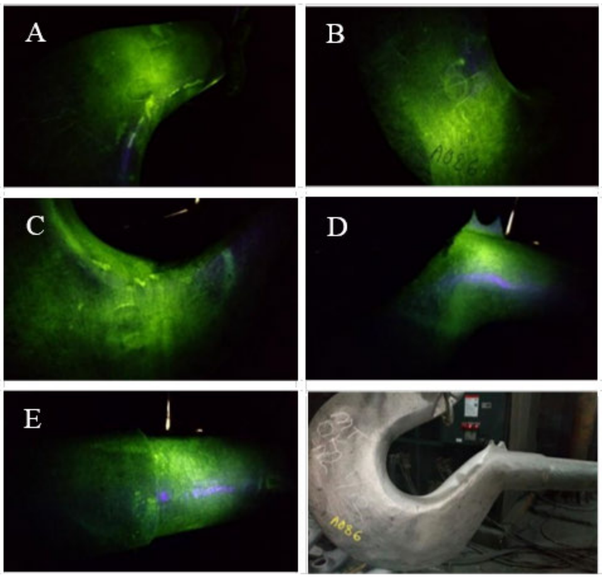

Figure 9. heat code: A086, Photos of castings after final MT. No cracks found.

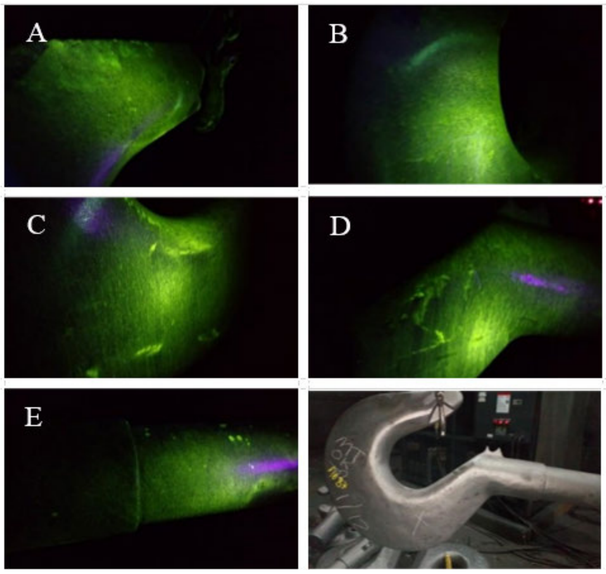

Figure 10. heat code: A087, Photos of castings after final MT. No cracks found

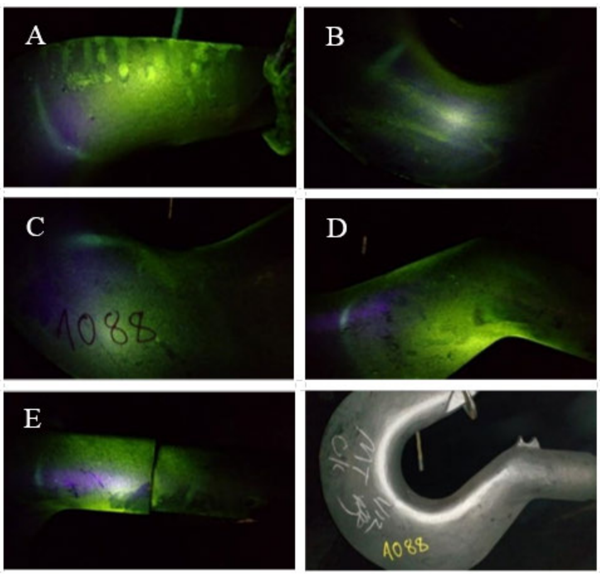

Figure 11. heat code: A088, Photos of castings after final MT. No cracks found.

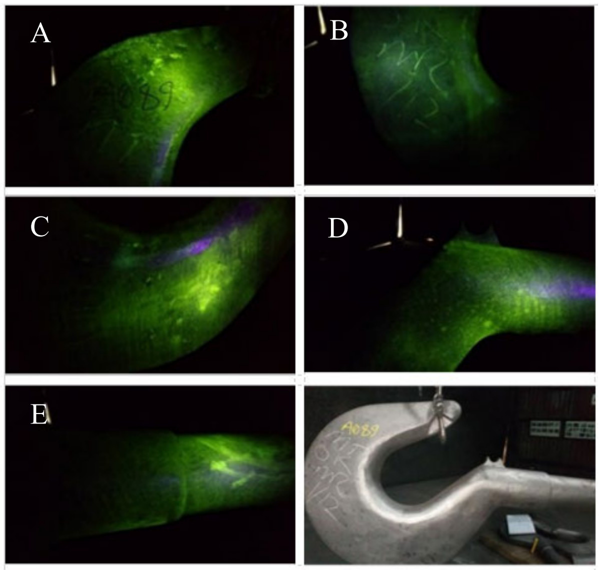

Figure 12. heat code: A089, Photos of castings after final MT. No cracks found.

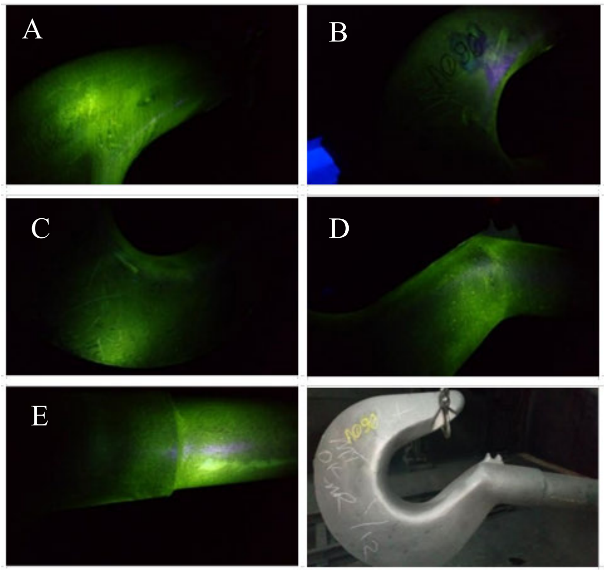

Figure 13. heat code: A090, Photos of castings after final MT. No cracks found.

T. Final Validation; all process described above were validated by using DMAIC. Also a Customer validation was received after castings were machined. No cracks were found. After validation, a change in the procedure was implemented as a control measure.

IV. Conclusions

After the deoxidations process change to the 4318-Zr alloy for castings > 6 inches riser contact areas by adding Zirconium from 0.04 to 0.12% and 0.035% max Aluminum in the ladle after a C boil of .30 points in E.A.F. and a follow up with the Metallurgical Flow (thermal process) we corroborated that the process change has been successful in avoiding AlN cracks.

The evidence of this project suggests the following conclusions:

1. Reduce N in arc furnace by C boil of .30 C minimum for N less than 80 PPM.

2. Control residual Al from 0.015 to 0.035% to prevent AlN formation.

3. In very heavy section, replace Al deoxidation with additions of N stabilizers like Zr.

4. Range of Zr from 0.04 to 0.12% to stabilize N and avoid another type of inclusions.

5. AlN cannot be eliminated by heat treat, but heat treat can eliminate other type of cracks with the proper cycle.

6. It was validated that by following the procedure no other cracking problems appear, such as thermal cracks (preheating cracks). After inspection all eight hook castings showed good quality results.

To achieve similar results the authors have implemented the new deoxidation practice to different alloys prone to AlN embrittlement in heavy sections. In the future other type of deoxidants such as Ti and Ca will be considered as well.

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