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PROCESS CONTROL - EFFECT OF ACID CLEANING ON THE MECHANICAL PROPERTIES OF 17-4 PH STEEL IN VARIOUS HEAT TREATED CONDITIONS

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GENERAL DYNAMICS | FORT WORTH

PROCESS CONTROL TEST REPORT

NUMBER 4577

DATE Feb. 1961

MODEL

FORT WORTH

TITLE

PROCESS CONTROL - EFFECT OF ACID CLEANING ON THE MECHANICAL PROPERTIES OF 17-4 PH STEEL IN VARIOUS HEAT TREATED CONDITIONS

The Tests described in this report were conducted between 7-25-60 and 11-29-60

CONVAIR

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PROCESS CONTROL TEST REPORT

TITLE: PROCESS CONTROL-EFFECT OF ACID CLEANING ON THE MECHANICAL PROPERTIES OF 17-4 PH STEEL IN VARIOUS HEAT TREATED CONDITIONS

PURPOSE:

To determine if the cleaning procedure specified in Process Standard 61.32B* embrittles aged 17-4 PH steel parts.

SUMMARY:

Cleaning 17-4 steel parts as specified in Process Standard 61.32B will not produce embrittlement in 17-4 PH wrought forms at nominal strength levels.

Cleaning 17-4 PH castings as specified in Process Standard 61.32B could not be evaluated because of the inherent structural characteristic of low ductibility at 180 K.S.I. minimum strength.

Because of the difficulty in controlling aggregate immersion time, 17-4 PH steel parts, castings and wrought, should not be acid pickled. Acid pickling will not embrittle 17-4 PH steel parts, but susceptibility to failure due to intergranular attack will increase proportionally to increase in immersion time.

*General Dynamics/Fort Worth Process Standard
See Page 3 for procedure

TITLE: PROCESS CONTROL-EFFECT OF ACID CLEANING ON THE MECHANICAL PROPERTIES OF 17-4 PH STEEL IN VARIOUS HEAT TREATED CONDITIONS

OBJECT:

To determine if acid cleaning as specified in Process Standard 61.32, and other solutions, can be used to clean 17-4 PH steel parts.

SPECIMENS:

Compression Ring-Stress Bar specimens
Notched and Un-notched tensile specimens

Compression ring-stress bar specimens were produced from 17-4 PH castings and 17-4 PH bar material. The notched and un-notched tensile specimens were produced from 17-4 PH bar material.

TESTING APPARATUS:

Baldwin Southwark 60,000 # tensile machine, Industrial Filter and Pump Mfg. Co. - Salt Spray Cabinet

PROCEDURE:

Notched, un-notched tensile and compression ring-stress bar specimens were aged to both 900° and 1100°F and processed as follows:

A.-17-4 PH Casting - Compression ring-stress bar specimens

1. Control - as received - 900°F and 1100°F* age.
** 2. Annealed + Pickle solution + 900°F age.
** 3. Annealed + Pickle solution + 1100°F age.
4. As received (900°F age) + Pickle solution.
5. As received (1100°F age) + Pickle solution.
6. Annealed + P.S. 61.32B cleaning + 900°F age.
7. Annealed + P.S. 61.32B cleaning + 1100°F age.
8. As received (900°F age) + P.S. 61.32B cleaning.
9. As received (1100°F age) + P.S. 61.32B cleaning.
10. Annealed + Armco Pickle + 900°F age.
11. Annealed + Armco Pickle + 1100°F age.
12. As received (900°F age) + Armco Pickle.
13. As received (1100°F age) + Armco Pickle.

* .010" removed from O.D.
** .010" removed from O.D. prior to anneal

B.-17-4 PH Bar material - Compression ring - stress bar and notched and un-notched tensile specimens.

1. Control - 900°F and 1100°F age.
2. Pickle solution and 900°F age.
3. Pickle solution + 1100°F age.
4. 900°F age + Pickle solution.
5. 1100°F age + Pickle solution.

6. P.S. 61.32B cleaning + 900°F age.
7. P.S. 61.32B cleaning + 1100°F age.
8. 900°F age + P.S. 61.32B cleaning
9. 1100°F age + P.S. 61.32B cleaning
10. Armco pickle + 900°F age.
11. Armco pickle + 1100°F age.
12. 900°F age + Armco pickle.
13. 1100°F age + Armco pickle.

C.-Compression ring - stress bar specimens were also given the following evaluation:

1. Control - 900°F + 1100°F age + salt spray for 6 days.
2. 900°F age + Pickle * solution + salt spray for 6 days.
3. 1100°F age + Pickle * solution + salt spray for 6 days.
4. 900°F age + P.S. 61.32B * cleaning + salt spray for 6 days.
5. 1100°F age + P.S. 61.32B * cleaning + salt spray for 6 days.
6. 900°F age + Armco * pickle + Salt spray for 6 days.
7. 1100°F age + Armco * pickle + salt spray for 6 days.

* The compression ring - stress bar specimens were given 3 immersion time (maximum) cycles prior to salt spray evaulation.

The acid solutions used are as follows:

1. P.S. 61.32B cleaning

a. Vapor Degrease
b. Clean - Nitric acid - 17/20%
Water - 80/83%
Soduim Dichromate - 3/5 oz/gal.
c. Immersion for 30 mins. at 143°F.
d. Rinse

2. Pickle Solution

a. Vapor Degrease
b. Pickle - Nitric acid - 7/17%
Ammonium Bifluoride - 6/9 oz/gal.
Water - remainder
c. Immersion for 15 mins. at 135°F.
d. Rinse

3. Armco Pickle Solution

a. Vapor Degrease
b. Pickle - Sulfuric acid - 15%
Water - remainder
c. Immersion for 30 mins. at 190°F
d. Hot water rinse
e. Pickle - Nitric acid - 10%
Hydrofluoric acid - 2%
Water - remainder

f. Immersion for 3 mins. at 140°F
g. Hot water, high pressure water or brush scrub.

Compression ring-stress bar specimens of Tests A and B stressed after processing.

Compression ring-stress bar specimens of Test C stressed before processing and salt spray evaluation.

Results: Results are listed in Tables I, II, III, IV and V.

TABLE I

17-4 PH Casting - H900 Condition-Compression Ring-Stress Bar-Control

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	71.5 - 72.0	540	.210
2.	71.5 - 72.0	605	.310
3.	71.5 - 72.0	750	.500*
4.	71.5	470	.130
5.	71.5 - 72.0	660	.390
6.	71.5 - 72.0	575	.240
7.	71.5 - 72.0	525	.170
8.	71.5 - 72.0	550	.210
9.	71.5 - 72.0	430	.110
10.	71.5 - 72.0	510	.160

* No failure. Where stress bar was not used, .500" deflection chosen, arbitrarily, as end point. At .500" deflection, compression ring O.D. parallel.

17-4 PH Casting-H1100 Condition-Compression Ring-Stress Bar-Control

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	62.0 - 62.5	470	.500
2.	63.5 - 64.0	390	.500
3.	63.5 - 64.0	430	.500
4.	66.0 - 67.0	540	.500
5.	64.5 - 65.5	450	.500
6.	67.0 - 67.5	535	.500
7.	67.0 - 67.5	420	.200 *
8.	62.5 - 63.0	385	.500 -
9.	63.5 - 64.5	380	.500 -
10.	66.0 - 67.5	425	.500 -

* Failure
\- .010" removed from O.D.

TABLE II

17-4PH Bar Material-H900 Condition-Compression Ring-Stress Bar-Control

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	72.0	773	.500
2.	72.0	786	.500

17-4 PH Bar Material-H 1100 Condition-Compression Ring-Stress Bar-Control

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	66.0 - 66.5	570	.500
2.	66.0	590	.500

17-4 PH Bar Material-H900 Condition-Compression Ring-Stress Bar-Control*

Specimen #	Lbs. Deflection	Inches Deflection
1.	565	.200
2.	555	.200

17-4 PH Bar Material-H1100 Condition-Compression Ring-Stress Bar-Control*

Specimen #	Lbs. Deflection	Inches Deflection
1.	445	.200
2.	465	.200

* These compression rings were stressed to the inches deflection as noted, stress bar inserted and subjected to salt spray for 6 days.

TABLE III

A. - 17-4 PH Casting-Compression Ring-Stress Bar

* Annealed + Pickle solution + 900°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	380	.085
2.	505	.500
3.	325	.014
4.	435	.037

* Annealed + Pickle solution + 1100°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	330	.023
2.	530	.500
3.	380	.500
4.	395	.500

* .010" removed from O.D. prior to anneal

As received (900°F age) + Pickle solution

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	71.5 - 72.0	590	.500
2.	71.5 - 72.0	370	.190
3.	71.5 - 72.0	450	.390

As received (1100°F age) + Pickle solution

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	62.0 - 62.5	280	.500
2.	66.5 - 67.0	525	.500
3.	66.5 - 67.0	395	.500

Annealed + P.S. 61.32B cleaning + 900°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	480	.120
2.	720	.500
3.	715	.500
4.	525	.175

Annealed + P.S. 61.32B cleaning + 1100°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	470	.400
2.	480	.430
3.	470	.500
4.	470	.500

As received (900°F age) + P.S. 61.32B cleaning

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	71.5 - 72.0	465	.100
2.	71.5	680	.450
3.	71.5 - 72.0	565	.270

As received (1100°F age) + P.S. 61.32B cleaning

Specimen #	Rockwell A Hardness	Lbs. Deflection	Inches Deflection
1.	63.5	380	.500
2.	66.5	420	.360
*3.	63.5 - 64.0	385	.500

* .010" removed from O.D.

Annealed + Armco Pickle + 900°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	670	.500
2.	680	.500
3.	645	.500
4.	685	.500

Annealed + Armco Pickle + 1100°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	480	.500
2.	515	.500
3.	500	.500
4.	500	.500

As received (900°F age) + Armco Pickle

Specimen #	Lbs. Deflection	Inches Deflection
1.	470	.450
2.	505	.470
3.	330	.100

As received (1100°F age) , Armco Pickle

Specimen #	Lbs. Deflection	Inches Deflection
1.	180	.500
2.	375	.500
3.	215	.500

TABLE IV

B. - 17-4 PH Bar stock-Compression Ring-Stress Bar

Pickle solution + 900°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	640	.500
2.	780	.500
3.	690	.500

Pickle solution + 1100°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	625	.500
2.	620	.500
3.	580	.500

900°F age + Pickle solution

Specimen #	Lbs. Deflection	Inches Deflection
1.	725	.500
2.	680	.500
3.	675	.500

1100°F age + Pickle solution

Specimen #	Lbs. Deflection	Inches Deflection
1.	450	.500
2.	515	.500
3.	480	.500

P.S. 61.32B cleaning + 900°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	830	.500
2.	795	.500
3.	820	.500

P.S. 61.32B cleaning + 1100°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	610	.500
2.	555	.500
3.	600	.500

900°F age + P.S. 61.32B cleaning

Specimen #	Lbs. Deflection	Inches Deflection
1.	795	.500
2.	790	.500
3.	810	.500

1100°F age + P.S. 61.32B cleaning

Specimen #	Lbs. Deflection	Inches Deflection
1.	550	.500
2.	620	.500
3.	620	.500

Armco pickle + 900° F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	785	.500
2.	700	.500
3.	720	.500

Armco pickle + 1100°F age

Specimen #	Lbs. Deflection	Inches Deflection
1.	530	.500
2.	555	.500
3.	555	.500

900°F Age + Armco Pickle

Specimen #	Lbs. Deflection	Inches Deflection
1.	510	.500
2.	505	.500
3.	500	.500

1100°F age + Armco Pickle

Specimen #	Lbs. Deflection	Inches Deflection
1.	365	.500
2.	410	.500
3.	360	.500

B. - 17-4 PH Bar stock Mechanical Properties

Notch - Un-notch

900°F age - Controls

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	181.9	197.0	15.4	52.3
notched	---	289.0	---	---

Notched-Un-notched ratio - 1.47-1

1100°F age - Controls

Specimen #	.2% Y.S.(K.S.K.)	T.S.(K.S.K.)	Elongation (% in 2")	R. of A. (%)
Un-notched	150.8	155.8	17.9	63.8
notched	---	237.3	---	---

Notched-Un-notched ratio - 1.52 - 1

Pickle solution + 900°F age

Specimen #	.2% Y.S.(K.S.K.)	T.S.(K.S.K.)	Elongation (% in 2")	R. of A. (%)
Un-notched	186.9	198.0	15.4	53.1
notched	---	289.6	---	---

Notched-Un-notched ratio - 1.46 - 1

Pickle solution + 1100°F age

Specimen #	.2% Y.S.(K.S.K.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	145.8	151.0	17.9	63.9
notched	---	235.1	---	---

Notched-Un-notched ratio - 1.56 - 1

900°F age + Pickle solution

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	184.0	195.3	15.4	51.8
notched	---	280.1	---	---

Notched-Un-notched ratio - 1.43 - 1

1100°F age + Pickle solution

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% of 2")	R of A. (%)
Un-notched	147.6	148.6	16.1	62.3
notched	---	229.1	---	---

Notched-Un-notched ratio - 1.54- 1

P.S. 61.32B cleaning + 900°F age

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	185.8	197.8	15.4	52.5
notched	---	274.8	---	---

Notched-Un-notched ratio - 1.39 - 1

P.S. 61.32B cleaning + 1100°F age

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	142.4	150.7	18.0	63.0
notched	---	230.1	---	---

Notched-Un-notched ratio - 1.53 - 1

900°F age + P.S. 61.32B cleaning

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	182.8	197.0	15.4	50.0
notched	---	283.0	---	---

Notched-Un-notched ratio - 1.47 - 1

1100°F age + P.S. 61.32B cleaning

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	150.8	151.8	17.9	62.5
notched	---	239.0	---	---

Notched-Un-notched ratio - 1.57 - 1

Armco pickle + 900°F age

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	188.9	195.0	14.3	49.0
notched	---	289.0	---	---

Notched-Un-notched ratio - 1.48 - 1

Armco pickle + 1100°F age

Specimen #	.2% Y.S. (K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	141.4	145.7	14.3	49.0
notched	---	221.4	---	---

Notched-Un-notched ratio - 1.52 - 1

900°F age + Armco Pickle

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	187.4	199.1	14.3	49.1
notched	---	296.0	--	---

Notched-Un-notched ratio - 1.49 - 1

1100°F age + Armco pickle

Specimen #	.2% Y.S.(K.S.I.)	T.S.(K.S.I.)	Elongation (% in 2")	R. of A. (%)
Un-notched	148.5	150.7	16.o	62.4
notched	---	212.8	---	---

Notched-Un-notched ratio - 1.41 - 1

TABLE V

C. - Compression ring-stress bar specimens

900°F age + Pickle solution + Salt spray for 6 days

Specimen #	Lbs. Deflection	Inches Deflection
1.	485	.190
2.	460	.200
3.	490	.200

1100°F age + Pickle solution + Salt spray for 6 days

Specimen #	Lbs. Deflection	Inches Deflection
1.	250	.190
2.	265	.200
3.	270	.190

900°F age + P.S. 61.32B cleaning + Salt spray for 6 days

Specimen #	Lbs. Deflection	Inches Deflection
1.	580	.200
2.	550	.200
3.	570	.200

1100°F age + P.S. 61.32B cleaning + Salt spray for 6 days

Specimen #	Lbs. Deflection	Inches Deflection
1.	445	.200
2.	435	.200
3.	440	.200

900°F age + Armco Pickle + Salt spray for 6 days

Specimen #	Lbs. Deflection	Inches Deflection
1.	65	.130
2.	65	.130
3.	60	.130

1100°F age + Armco Pickle + Salt spray for 6 days

Specimen #	Lbs Deflection	Inches Deflection
1.	70	.140
2.	70	.130
3.	65	.130

Discussion of Results:

17-4 PH was acid cleaned as specified in P.S. 61.32B and in two other acid solutions-Nitric-Ammonium Bifluoride-Water and Armco Pickling Method (Sulfuric-Water and Nitric-Hydrofluoric-Water solutions).

The test specimens- Compression Ring - Stress Bar, Notched and Un-notched tensiles - aged at 900°F and 1100°F before and after cleaning and pickling produced the following:

1. Compression test rings from 17-4 PH castings aged at 1100°F before and after immersion in the acid cleaning and pickling solutions revealed a greater ability to withstand deflection than the test rings aged at 900°F.

2. Compression test rings from 17-4 PH bar material aged at 900°F and 1100°F before and after acid immersion produced the same amount of deflection at different strength levels.

3. Tensile specimens from 17-4 PH bar material subjected to the same conditions as the compression test rings produced no significant evidence of deleterious effects of acid immersion.

4. The cleaning and pickling solutions did not embrittle 17-4 PH, but metallographic examination did show evidence of intergranular attack. Only the pickling solutions produced this evidence. Using 1100°F before acid pickling in the Nitric-Ammonium Bifluoride-Water and combination solutions of Sulfuric-Water and Nitric-Hydrofluoric - Water produced the greatest amount of intergranular attack.

5. Whether the compression ring-stress bar specimens were immersed once or three times for maximum accumulation of time in the acid solutions made no significant difference maximum accumulation of time in the acid solutions produced only reduction of wall thickness of the ring specimens. Maximum accumulation of time in the acid solutions did not product failures of the compression ring - stress bar specimens.

Recommendations:

Because of the difficulty in controlling aggregate immersion time, 17-4 PH steel parts should not be acid cleaned or pickled. Acid cleaning or pickling will not embrittle 17-4 PH, but susceptibility to failure due to integranular attack will increase with increase of immersion time in the pickling solutions.