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LITERATURE SURVEY OF STRESS CORROSION CRACKING
AND HYDROGEN EMBRITTLEMENT IN HIGH
STRENGTH STEELS

John L. Briggs

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THE DOW CHEMICAL COMPANY
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401

Prepared under Contract AT(29-1)-1106
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LITERATURE SURVEY OF STRESS-CORROSION CRACKING AND HYDROGEN EMBRITTLEMENT IN HIGH STRENGTH STEELS

John L. Briggs

Abstract. A literature survey has been conducted to determine the stress-corrosion cracking properties of three high strength steels. The steels of concern are Vascomax 250[®] (an 18Ni maraging steel), Almar 362[®] (a stainless maraging steel), and HP9Ni-4Co-.20C (a quenched and tempered martensitic steel). Other topics included in the survey are hydrogen-stress cracking, general corrosion resistance, welding, and a suitable method for cleaning the high strength steels.

INTRODUCTION

Three high strength steels, Vascomax[®] 250 (proprietary name of Vanadium-Alloys Steel Company), Almar[®] 362 (a registered trademark of Allegheny Ludlum Steel Corp.) and HP9Ni-4Co-.20C[®] (proprietary name of Republic Steel Corp.), have been designated as possible fabricating materials in production processes at Rocky Flats. All high strength steels are susceptible to stress-corrosion cracking (SCC) in specific environments,¹ therefore the projected use of these alloys has created a need for information on their SCC properties. A literature survey has been conducted with special emphasis on the SCC susceptibilities of these alloys. Other areas of interest which were included in the survey were general corrosion resistance, welding characteristics, and hydrogen embrittlement as related to the cleaning of high strength steels.

Where limited data were available on one of the three high strength steels, information is presented on a similar alloy. The results of various laboratory and atmospheric SCC tests in specific environments are included in the report. Factors other than corrosion environment, such as heat treating, handling, and fabricating may contribute to SCC so that qualified interpretation of reported test results is necessary. Care in interpretation applies especially when comparing the SCC resistances of different alloys.¹⁻³

Special consideration is also necessary when SCC test results indicate that cracking has occurred under experimental conditions. Such results do not mean the alloy will necessarily crack under all service conditions but do indicate that consideration must be given to environmental and stress conditions.

STRESS-CORROSION CRACKING

Stress corrosion is defined by Uhlig as any combined effect of stress and corrosion on the behavior of metals.⁴ He defines SCC as the combined action of stress and corrosion which leads to cracking and embrittlement of the metal. LaQue defines SCC broadly as failure by cracking as the result of combined action of static tensile stress and corrosion.⁵ The "Metals Handbook" defines SCC as "... failure by cracking under (the) combined action of corrosion and stress, either external (applied) or internal (residual). Cracking may be either intergranular or transgranular, depending on the metal and corrosive medium."⁶ Boyd describes SCC as a combination of electrochemical and hydrogen induced causes.⁷ Although there is controversy over the mechanism of SCC, there is general agreement that SCC and hydrogen stress cracking (hydrogen embrittlement) may be defined separately. SCC results from the electrochemical corrosion of a metal under stress. Hydrogen stress cracking (HSC) results when hydrogen is present in a metal lattice under stress (no corrosion or metal dissolution is required for this type fracture).¹ This report will treat HSC and SCC as separate phenomena, although some fractures may include both cracking mechanisms.

One method of distinguishing between the two types of cracking has been described by Fletcher who states that cracking failures which occur under conditions of complete cathodic protection with hydrogen evolution may be classified as HSC. Stress-corrosion fractures occur when the current is reversed and the specimen is made anodic.¹

HYDROGEN EMBRITTLEMENT

Hydrogen embrittlement (or HSC) is a cracking process caused by the combined action of stress and nascent (atomic) hydrogen in the metal lattice.^{1,2} The penetration of hydrogen into a susceptible alloy usually results in a loss of ductility as measured by a tensile test. In some cases, hydrogen embrittlement cannot be detected in a tensile test, but is made manifest as a loss in ability to support a sustained (static) load. No embrittlement of hydrogen-charged iron-base and nickel-base alloys is detected with

impact loading. Thus, hydrogen embrittlement is a low strain rate phenomenon. The reason for the strain rate dependence is that the mechanism for hydrogen embrittlement requires the diffusion of hydrogen to sites where failure initiates and there is not sufficient time for hydrogen diffusion under fast strain-rate conditions. Consequently, the most sensitive method for detecting hydrogen embrittlement is the static fatigue test with notched tensile specimens.⁴

The presence of trace amounts of hydrogen are difficult to detect, therefore fracture failures caused by hydrogen embrittlement are especially insidious. Average hydrogen contents of less than 1 ppm can cause cracking and the maximum amount of hydrogen that can be introduced into martensitic steel is 12-15 ppm.

It is generally agreed that processes that introduce hydrogen should be avoided.^{1, 2}

Maraging steels such as Almar 362 and Vascomax 250 are susceptible to hydrogen embrittlement but are less susceptible than conventional martensitic steels.⁸ Summarized in Table 1 are tests conducted to compare the embrittlement susceptibilities of Almar 362 and Type 410 (martensitic) stainless steel.

Table 1. Comparative Resistance to Hydrogen Embrittlement (50% HCl with 1% SeO₂).⁹

Almar 362		Type 410 Stainless Steel	
Ultimate Tensile Strength (psi)	Hours to Failure (hr)	Ultimate Tensile Strength (psi)	Hours to Failure (hr)
185,000	0.25	150,000	0.2
170,000	4.75	140,000	0.5
150,000	6.0	120,000	1.0
140,000	6.0 ^a	110,000	2.2

^aDiscontinued after 6 hours, no cracks.

Tests performed comparing 18Ni maraging steels to 4340 steel also indicated maraging steels are less susceptible to hydrogen embrittlement than conventional tool steels of equivalent yield strengths.⁸

Other time-to-failure data support the lower susceptibilities of maraging steels to hydrogen embrittlement.¹ However, the maraging steels will eventually fail by hydrogen embrittlement, albeit, after longer exposure periods to embrittling environments. Therefore, the time-to-failure parameter is not significant for demonstrating the relative susceptibilities of stored materials and the above relative susceptibilities to hydrogen embrittlement are inconsequential.

The 9Ni-4Co alloy displays excellent resistance to stress-corrosion cracking, but no data are available on susceptibility of this particular alloy to hydrogen embrittlement. However, the martensitic structure would make the alloy susceptible to hydrogen embrittlement.

MARAGING AND PRECIPITATION HARDENING STEELS

Almar 362 and Vascomax 250 are both members of the maraging (martensitic, age-hardened) steel family. Maraging steels are high nickel, iron-base alloys with high yield strengths up to 300 ksi (kips per square inch). The high strength levels of these steels are achieved by age hardening and/or precipitation hardening a stabilized martensitic structure.

Age hardening for maraging steels refers to the alloys being strengthened by aging in the annealed or martensitic condition. Precipitation hardening occurs when the precipitation of an intermetallic compound is involved in the strengthening mechanism. A variety of alloying elements (Ti, Al, Cu, Mo) included in maraging steels serve in the age hardening and precipitation hardening processes.

PROPERTIES

The following sections give information concerning the metallurgy, SCC, general corrosion resistance, welding, and cleaning methods of the three high strength steels.

Vascomax 250 [18Ni(250) Maraging Steel] — Metallurgy

Vascomax 250 is a proprietary name of the Vanadium-Alloys Steel Corporation and is an 18% nickel maraging steel with a nominal yield strength of 250 ksi. Most of the data available in the literature pertain to 18Ni(250) maraging steels which are included in this report rather than the proprietary-named alloy. Yield strength is included in the designation of maraging steels, e.g., 18Ni (250).

The 18Ni (250) maraging steel is solution annealed to austenite at 1500° F for one hour. The steel is transformed from austenite to martensite by air cooling from the annealing temperature to room temperature. Solution annealing at 1500° F for one hour allows for the highest strength and ductility when the steel is subsequently hardened.¹⁰ The M_s temperature is about 310° F and the M_f temperature is about 210° F.¹¹

The solution-annealed and air-cooled steel is maraged (aged in the martensitic condition) by heating to 800° to 900° F for one to four hours and then air cooled. The aging mechanism is not fully understood. It is related to ordering and precipitation reactions.^{10,11}

The nominal composition of 18Ni (250) maraging steel is summarized in Table 2.

Table 2. Nominal Composition of 18Ni (250) Maraging Steel.¹¹

Element	Weight Percent
C	0.03 max.
Mn	0.10 max.
Si	0.10 max.
S	0.01 max.
P	0.01 max.
Ni	17.0-19.0
Co	7.0-8.5
Mo	4.6-5.2
Ti	0.3-0.5
Al	0.05-0.15

Combining a low carbon content with high (18 wt%) nickel content provides a dislocation structure and a base martensite hardness to which hardening may be applied. The toughness of the alloy is increased by maintaining low levels of silicon, manganese, and sulfur. Cobalt, molybdenum, titanium, and aluminum are included for age hardening purposes.¹²

18Ni (250) Maraging Steel – SCC

The 18Ni (250) maraging steel exhibits excellent resistance to SCC. This is probably the result of low silicon, sulfur, manganese and carbon content, high nickel composition and the aging process involving molybdenum and cobalt. However, some susceptibility to SCC does exist.¹²

Investigators at The International Nickel Co., Inc. have reported that 18Ni (250) maraging steel has good resistance to SCC in the Bayonne, New Jersey industrial atmosphere and in aerated artificial ocean water. The specimens, which were loaded to a yield strength of 246 ksi in the ocean water tests, had not broken within 180 days.¹³

In a two-year SCC study,^{14,15} bent beam and U-bend specimens of 18Ni maraging steel with variable yield strengths (181 to 232 ksi) were comparatively tested with several low alloy steels. After one year, the results of this test program indicated that 18Ni maraging steels were susceptible to

SCC in various environments, which are listed in Table 3. Shot peening the surface of the maraging steel improved resistance to SCC.^{2,14}

Table 3. Comparative Corrosivity of 18Ni Maraging Steel in Various Environments.¹⁴ The environments are arranged in order of decreasing corrosivity from top to bottom.

1. Natural ocean water immersion
2. 140° F water-saturated air
3. Aerated 3% NaCl solution
4. Aerated distilled water
5. Kure Beach 50 ft. lot marine atmosphere
6. Bayonne, N. J., industrial atmosphere
7. Newport Beach marine atmosphere
8. Aerated tap water
9. 0.25% sodium dichromate solution
10. 4% soluble oil (18% nickel-grade only)
11. Trichloroethylene
12. Cosmoline (no failures)
13. Laboratory air (no failures)

The summary and conclusions after the second year of SCC testing are included in Table 4.

Table 4. Conclusions of SCC Tests.¹⁵

1. Susceptibility to stress-corrosion failure is found in all 18Ni alloy heats having yield strengths from 181 to 232 ksi. Susceptibility becomes greater with increasing titanium content and yield strength.
2. At a given strength level, 18Ni maraging steel is less susceptible to failure than is H-11 steel or a hot-worked die steel. Cold working of 18Ni maraging steel before aging further reduces its susceptibility to stress-corrosion failure.
3. Distilled water is a more severe SCC medium than 3% salt water, and the U-bend test is more severe than the bent-beam test for SCC testing 18Ni maraging steel.
4. The 18Ni maraging steel increases in susceptibility to stress-corrosion cracking with an increase in environmental temperature. Conventional high strength steels at the same stress level have a higher susceptibility to failure at room temperature, but show a lesser increase in susceptibility with temperature rise.
5. An inhibited epoxy coating designated as 454-1-1 was effective in delaying SCC of both maraging steels and H-11 steel. An inorganic zinc coating designed to impart cathodic protection was not effective.
6. The prevention of SCC failure upon application of cathodic current demonstrates that the maraging steels crack by an electrochemical mechanism in 3% NaCl solution.
7. SCC susceptibility of maraging steel decreases with the increasing pH of the environment solution.

Greene and Haney studied the correlation of SCC with pH using potentiostatic techniques. The tests were conducted with 18Ni maraging steel specimens immersed in aqueous

chloride solutions with adjusted pH values. High susceptibility to SCC was observed in the 10-12 pH range and was associated with the onset of passivation.¹⁶

Commercial and laboratory heats of 18Ni maraging steels showed excellent resistance to SCC when exposed for long periods in ocean water and in marine and industrial atmospheres as summarized in Tables 5 and 6. The investigators emphasized the importance of structure, thermal history, and composition on the SCC properties of the alloy. All cathodically protected maraging steels cracked in ocean water except the 18Ni (200).

Cathodic protection similar to the previous studies was demonstrated in SCC tests conducted at Kure Beach. Less resistance to SCC is observed in these tests (shown in Table 7) compared to the previously described tests. This investigation showed that the cracking behavior was influenced by processing history.¹⁸

Other investigations have indicated that susceptibility to SCC is dependent on test method. U-bend specimens of 18Ni (250) steel in natural ocean water did not fail after

Table 5. Results of Stress-Corrosion Cracking Tests in Ocean Water on Commercially Produced Alloys.¹⁷

Alloy Type	Days in Test	Time to Visible Cracking, days ^a		
		Unprotected U-bends	Cathodically Protected U-bends -0.85 volts ^a	-1.2 volts ^b
18Ni (200)	490	OK,OK ^c	OK,OK	OK,OK ^b
18Ni (250)	490	OK,OK ^c	OK,OK	16,240
18Ni (250)	490	OK,OK ^c	6,OK	14,290
18Ni (300)	490	355,OK ^c	9,29	6,6
20Ni (250)	461	4,21	211,396	1,34
25Ni (250)	413	180,OK	OK,OK	163,OK
4340	6	6,6	6,6	6,6

^aOK signifies no failure to the number of days in the test.

^bPotential against a saturated calomel electrode.

^cRemoved at 218 days for microexamination.

750 days. Pre-notched specimens of the same alloy in natural ocean water failed with applied stresses of 15 to 25% of the yield strength. The difference between notched and

Table 6. Visible Stress-Corrosion Cracking in Atmospheric Tests on Commercially Produced Alloys.¹⁷

Time To Visible Cracking, Days ^a (2-Point Loaded Specimens.)						
Bayonne Atmosphere						
Alloy Type	Latest Inspection Days	U-bend	150 ksi ^b	200 ksi ^b	250 ksi ^b	300 ksi ^b
18Ni (200)	514	OK, OK, OK, OK ¹	OK, OK, OK, OK ²	405, 448, 448, OK ²	—	—
18Ni (250)	514	OK, OK, OK, OK ¹	OK, OK, OK, OK ²	OK, OK, OK, OK ²	OK, OK, OK, OK ²	—
18Ni (250)	514	OK, OK, OK, OK ¹	OK, OK, OK, OK ²	OK, OK, OK, OK ²	OK, OK, OK, OK ²	—
18Ni (300)	514	61, 67, OK, OK ¹	OK, OK, OK, OK ²	OK, OK, OK, OK ²	OK, OK, OK, OK ²	481, OK, OK, OK ²
20Ni (250)	266	22, 53, 266, OK ⁵	22, 22, 22, 22	14, 15, 22, 22	7, 14, 14, 14	—
25Ni (250)	420	OK, OK, OK, OK ⁶	OK, OK, OK, OK	OK, OK, OK, OK	—	—
4340	49	14, 14, 14, 28	22, 28, 46, 49	14, 14, 14, 18	14, 14, 14, 14	—
Kure Beach Atmosphere						
18Ni (200)	491	OK, OK, OK, OK ³	350, 350, 350, OK ⁴	288, 288, 288, OK ⁴	—	—
18Ni (250)	491	OK, OK, OK, OK ³	OK, OK, OK, OK ⁴	OK, OK, OK, OK ⁴	OK, OK, OK, OK ⁴	—
18Ni (250)	491	OK, OK, OK, OK ³	OK, OK, OK, OK ⁴	OK, OK, OK, OK ⁴	OK, OK, OK, OK ⁴	—
18Ni (200)	491	155, 345, OK, OK ³	OK, OK, OK, OK ⁴	350, OK, OK, OK ⁴	327, OK, OK, OK ⁴	56, 245, 288, OK ⁴
20Ni (250)	13	13, 13, 13, 13	13, 13, 13, 13	13, 13, 13, 13	13, 13, 13, 13	—
25Ni (250)	414	OK, OK, OK, OK	OK, OK, OK, OK	OK, OK, OK, OK	—	—
4340	7	2, 7, 7, 7	7, 7, 7, 7	7, 7, 7, 7	1, 2, 7, 7	—

NOTE: Superscript numbers indicate the following lengths of exposure in days before removal for microexamination:

¹ 261, ² 272, ³ 219, ⁴ 247, ⁵ 185, ⁶ 127.

^aOK signifies no failure

^bApplied stress

Table 7. Stress-Corrosion Cracking Behavior of U-Bends in Kure Beach Marine Atmosphere 80 Feet from Ocean.^{1,8}

Alloy	0.2% Offset Y.S. (ksi)	Total Testing Period, days ^a	Failure Ratio ^b	Days to Failure
18Ni(250) Maraging Steel	240-255 272	550 —	5:12 ^c 4:4	402-546 82-188
12-5-3 Maraging Steel	186 205	550 550	0:4 ^c 0:8	— —
4340	240	—	4:4	2-7

^aAll unfailed specimens remain in test.

^bRatio of failed specimens to those originally placed on test.

^cMicrocracks observed in one specimen of each heat after 183 days.

un-notched specimens indicated resistance to crack initiation but not to crack propagation. This author pointed out that selection of a material for resistance to crack initiation is not a recommended procedure.¹⁹

In comparative SCC tests, notched specimens of 18Ni (300) maraging steel exhibited the best resistance to SCC of materials tested (9Ni-4Co, 4340, H-11). When smooth, unnotched specimens were used, the 9Ni-4Co was superior to the other materials tested.²⁰

Steigerwald has described the use of precracked specimens in SCC tests of various high strength steels including the 18Ni and HP9Ni-4Co-.25C alloys.²¹ Emphasis was directed on the advantages of using precracked specimens and employing linear elastic fracture mechanics to interpret test results. The advantages were summarized as follows:

1. Precracking specimens reduces the time normally necessary to initiate environmentally induced cracking by a factor of 10^5 . Pit formation which initiates environmentally induced cracking is statistically controlled resulting in scatter. Precracked specimens reduce this scatter and simplifies mechanism analysis.
2. With no pitting in a given environment, SCC immunity may be indicated in smooth specimens. Environmentally induced failure may occur if a stress raiser is present.
3. The sharpest possible stress raiser is provided by a precrack which should result in conservative predictions.
4. Precracking produces a stress-raiser to which many fracture mechanisms may be applied and allows crack growth kinetics to be accurately evaluated.

Novak and Rolfe used a fracture mechanics approach to results of precracked bend specimens of 18Ni (250) maraging steel in synthetic ocean water. They showed that K_{Ic} (plane-strain critical stress intensity factor) and K_{Isc} (lower limit of stress intensity factor required to cause brittle fracture by SCC) are material properties (for a specific environment) independent of geometry. The K_{Ic} and K_{Isc} values obtained in this study for 18Ni (250) maraging steel were 67 and 36 ksi $\sqrt{\text{inch}}$ respectively.

The use of precracked samples eliminated the long incubation period necessary for crack initiation in smooth specimens. Correlation of these studies with smooth specimen SCC tests showed that K_{Isc} values may be used to calculate critical pit depths that form prior to SCC.²²

Judy and Goode have also described procedures based on fracture mechanics for detecting the susceptibility to SCC of high strength steels. They also indicated that flaw size stress conditions for subcritical crack growth may be calculated from K_{Isc} values.²³

18Ni (250) Maraging Steel — General Corrosion

In comparative tests, the corrosion rates of 18Ni maraging steels were about one-half the rates of low alloy steels, and pitting was shallower in atmospheric tests. Rust and pitting were observed on the 18Ni maraging steels in the atmosphere and rust and fouling were observed in ocean water. The results of these tests are summarized in Table 8.

Nearly identical corrosion rates were measured by other investigators with 18Ni (250) maraging steels exposed to similar environments. The maraging steel showed better corrosion resistance in quiet ocean water and marine environments than quenched and tempered steels and showed much better corrosion resistance than mild steels. All the specimens displayed uniform rusting corrosion. High corrosion rates were measured for all the specimens in moving ocean water as shown in Table 9.

18Ni (250) Maraging Steel — Welding

All the principal joining processes have been used to weld 18Ni maraging steels. Optimum weld-metal toughness and strength equivalent to base metal is provided by the TIG (gas tungsten arc process) in plate thicknesses up to 1 inch. Poor weld-metal toughness has resulted from all other welding processes.^{24,25}

Table 8. Corrosion of 18Ni (250) Maraging Steel in Industrial and Marine Environments.¹⁷

Alloy Type	Bayonne Atmosphere						Kure Beach Atmosphere						Ocean Water		
	Corrosion Rate (mpy)			Pit Depth (mils)			Corrosion Rate (mpy) ^a			Corrosion Rate (mpy) ^b			Corrosion Rate (mpy)		
	3 Mo.	6 Mo.	12 Mo.	3 Mo.	6 Mo.	12 Mo.	3 Mo.	6 Mo.	12 Mo.	3 Mo.	6 Mo.	12 Mo.	3 Mo.	6 Mo.	12 Mo.
18Ni(200)	1.2	0.86	0.50	1.1	2.5	3.1	0.35	0.45	0.42	0.70	0.65	0.64	3.2	2.3	2.6
18Ni(250)	1.2	0.91	0.52	1.4	2.5	3.0	0.40	0.45	0.38	0.65	0.70	0.56	3.0	2.5	2.8
18Ni(250)	1.4	0.84	0.50	2.0	2.1	2.3	0.55	0.50	0.42	0.75	0.65	0.60	2.8	2.4	2.4
18Ni(300)	1.6	0.89	0.51	2.0	2.2	2.5	0.45	0.50	0.40	0.70	0.60	0.53	3.0	2.6	2.0
20Ni(250)	—	1.36	0.81	—	2.8	3.6	—	0.50	0.45	—	1.00	0.42	—	2.8	3.6
25Ni(250)	—	0.72	0.60	—	2.2	3.1	—	0.30	0.30	—	0.50	0.58	—	2.8	3.0
4340	2.8	1.82	0.80	4.7	5.2	5.0	1.65	1.25	0.92	2.30	1.50	1.34	3.1	2.8	4.3
HY-80	3.0	1.77	0.88	5.1	5.7	4.5	1.50	1.15	1.02	2.40	1.50	1.41	2.8	2.6	3.3

^a 800 ft. from ocean^b 80 ft. from oceanTable 9. Corrosion in Ocean Water Flowing at 2 FPS (temperature range 40-85° F).¹⁸

Alloy	Corrosion rate (mpy)	
	6 mo.	1 yr.
18Ni maraging steel	11	7
12-5-3 maraging steel	*	*
HY 80	—	12
Mild steel	12	9

*All corrosion was in the form of pitting to a maximum depth of 20 mils at 6 months, 35 mils at 1 year.

Three sequences for welding and heat treating which have been used are:²⁵

1. Solution anneal, age, weld, and re-age the entire assembly or weld zone only.
2. Solution anneal, weld, and age the entire assembly.
3. Weld (solution annealed or aged), solution anneal, and age.

A significant property of the maraging steels related to welding is the reversion of the martensitic structure to austenite when heated.^{11, 26} A dark etching band containing austenite results in the weld heat-affected zone because of austenite reversion. A coarse-grained martensite band and a light etching band also result. Studies of the weld heat-affected zone using synthetic specimen techniques indicated decreased tensile strength of about 6% in the dark etching band and about 5% in the coarse-grained martensite band. Increased toughness was also observed in these regions. The author concluded that these changes were not significant.^{25, 27} Full post-weld heat treatments (annealing at 1600 to 1800° F) produces joint efficiencies of 100%; however, a post-weld maraging heat treatment provides joint efficiencies of 90%.²⁸ The investigations by Travis also indicated higher weld joint efficiencies for annealing and aging after welding than for direct aging after welding.²⁹

Filler metals for welding 18Ni maraging steels have been examined by several investigators.²⁵ Titanium content appears to be critical and should be the same as the base metal. Minor elements should be kept at a minimum because their presence decreases fracture toughness. It is generally agreed that hydrogen content be kept as low as possible.³⁰ Table 10 summarizes weld properties of 18Ni maraging steels using filler wire with the following impurity limits: Al-0.2%, C-.03 max., and H-5 ppm maximum.

Some SCC studies using precracked specimens have been conducted comparing TIG welded 18Ni maraging steel and unwelded base material in synthetic ocean water. Preliminary results indicated the SCC resistance of the weld was lower than the unwelded base material.²⁵ Sandoz's investigation with precracked specimens of TIG welds on 18Ni maraging steel in 3.5 wt% NaCl solution unexpectedly showed the weld metal more resistant to SCC than the base plate.³¹

Table 10. Properties of Welds in 18Ni Maraging Steel.²⁸

	Gas, Metal Arc ^a		Covered Electrode ^b		Submerged Arc ^c	
	As Welded	Maraged	As Welded	Maraged	As Welded	Maraged
Yield strength (0.2% offset), psi	134,000	220,000	130,000	225,000	130,000	220,000
Tensile strength, psi	154,000	235,000	160,000	242,000	155,000	235,000
Reduction in area, %	40	30	35	26	24	25
Elongation in 1 in., %	5	7	5	6	6	6
Notched: unnotched tensile strength	—	1:2	—	1:1	—	1:1
Rockwell C hardness	32 to 42	49 to 51	30 to 40	49 to 51	30 to 35	48 to 50
Charpy V-notch impact (70°F) ^d	39 ft-lb	15 ft-lb ^e	25 ft-lb	10 ft-lb	33 ft-lb	10 ft-lb

One-half-inch-thick plate (250,000 psi yield strength) welded in maraged condition. Transverse tensile tests; all fractures occurred in weld. Maraging was carried out at 900°F for 3 hours.

^aSemi-automatic, using 0.062-in. diameter filler wire.

^bLow hydrogen flux coating, manual weld.

^cFour passes, welded from one side.

^dNotch in weld running normal to plate surface.

^e12 ft-lb at -320F.

Almar 362 – Metallurgy

Almar 362 is a low carbon, martensitic, precipitation-hardened, stainless steel member of the maraging steel family. The alloy is austenitic at annealing temperatures (1500–1650°F) and is martensitic at room temperature. The M_s and M_f temperatures are 320°F and 51°F, respectively. Aging the alloy in the 900 to 1100°F temperature range causes the precipitation of intermetallic (nickel-titanium) compounds which strengthens the martensitic structure. The nominal composition of Almar 362 is summarized in Table 11.

Table 11. Nominal Composition of Almar 362.⁸

Element	Weight %
C	0.03
Mn	0.30
P	0.015
S	0.015
Si	0.20
Cr	14.50
Ni	6.50
Ti	0.80

One author recommends aging at 900°F to produce maximum strength and hardness, 1125°F for maximum toughness, and 1000°F for optimum combined toughness and strength.⁹

Almar 362 – SCC

One writer describes the SCC resistance of Almar 362 as exceeding the SCC properties of precipitation hardening steels and better than the martensitic stainless steels.⁹

Some pitting was detected, but no cracks occurred in SCC tests of Almar 362 exposed to 5% salt spray. The results are summarized in Table 12.⁸

Almar 362 – General Corrosion

The inclusion of 12% chromium plus low carbon in the composition of Almar 362 has provided the alloy with stainless steel properties which are not available in the other nickel maraging steels. (Plating is often used to protect the nonstainless maraging steels.) General corrosion resistance of Almar 362 is similar to 430 stainless steel.⁹ Table 13 summarizes the corrosion rates of Almar 362 in several acid environments.⁸

Normal atmospheres or humid environments will not cause rust or staining of Almar 362 although some pitting will occur (similar to 304 stainless steel) in salt spray or marine environments.⁹

Table 12. Stress-Corrosion Cracking Tests of Almar 362 in 5% Salt Spray.⁸

Material	Heat Treatment		Applied Stress (ksi)	Percent of 0.2% Yield Strength	Total Time	Condition
	Temperature (°F)	Time (hr)				
0.019 in. strip transverse	900	8	190.0	90	Discontinued at 196 days, no cracks.	1 small pit, 13 days.
	1050	2	150.0	95	Discontinued at 196 days, no cracks.	1 small pit, 13 days. 1 small pit, 27 days.
0.060 in. strip, transverse	900	8	137.0	75	Discontinued at 250 days, no cracks.	No rust or pits.
	975	4	125.0	75	Discontinued at 250 days, no cracks.	No rust or pits.
	1050	2	112.0	75	Discontinued at 250 days, no cracks.	No rust or pits.
1 in. diam. bar, longitudinal	900	8	169.5	90	Discontinued at 140 days, no cracks.	No rust scattered small pits.
	975	4	155.3	90	Discontinued at 140 days, no cracks.	No rust scattered small pits.
	1050	2	136.7	90	Discontinued at 140 days, no cracks.	No rust, scattered small pits.

Table 13. Corrosion of Almar 362 in Acid Environments.⁸

Environment	Rate, mpy
Boiling nitric acid (25%)	13
Boiling phosphoric acid (20%)	1
Boiling acetic acid (60%)	1
Concentrated sulfurous acid	0.1
Sulfurous acid vapor	0.4

Almar 362 – Welding

Any of the conventional methods used for welding austenitic stainless steels may be used for welding Almar 362. Post-weld annealing or pre-heating is not required to improve weld metal ductility or to prevent cracking. It is necessary to age the alloy after welding to increase strength. As with the maraging nickel steels, a high titanium content makes Almar 362 susceptible to weld metal segregation in multipass welds. Optimum weld results are obtained using light-gage material and single pass TIG welds. The TIG process will produce welded and aged properties similar to the base metal in sections up to $\frac{1}{8}$ -inch thick. Because of titanium segregation, multipass welds on heavier gages are obtained by limiting the titanium content of the filler wire.^{9,32,33} The weld properties of Almar 362 welded strip are summarized in Table 14.

Table 14. Properties of Almar 362 Welded Strip.⁹

Post-Weld Heat Treatment	Ti (%)	0.2% YS	UTS	Elongation
		(ksi)	(ksi)	in $\frac{1}{2}$ in. (%)
8 hr at 900°F (0.031-in. thick)	0.70	192.6	193.0	2.5 ^a
8 hr at 900°F (0.050-in. thick)	0.88	196.8	199.0	3.0 ^b
1500°F + 8 hr at 900°F	0.70	188.6	189.2	8.0 ^b
1500°F + 8 hr at 900°F	0.88	180.4	182.7	5.0 ^b
3 hr at 1000°F	0.70	170.5	172.1	4.5 ^a
3 hr at 1000°F	0.88	159.4	169.4	8.0 ^b
1500°F + 3 hr at 1000°F	0.70	158.5	161.0	5.5 ^b
1500°F + 3 hr at 1000°F	0.88	149.8	157.6	7.0 ^b

^aFractured in base metal.

^bFractured in weld metal.

For multipass welding, filler wire with a titanium content of 0.45%–0.60% is recommended by one writer who described successful welds on pressure vessels with 0.5% Ti filler wire. A thorough description of welding methods is included in that study.⁹

9Ni-4Co-.20C – Metallurgy

The 9Ni-4Co-.20C alloy is a member of a family of martensitic steels which develops high strengths from

quenching and tempering processes. The composition limits of this alloy are summarized in Table 15. The composition of 9Ni-4Co-.25C is included for comparative purposes.

Table 15. Nominal Compositions of 9Ni-4Co-.20C and 9Ni-4Co-.25C.³⁴

Element	wt%, .20C	wt%, .25C
C	0.18-0.24	0.24-0.30
Ni	7.00-8.50	7.00-9.00
Co	3.50-4.50	3.50-4.00
S	0.010 Max.	0.010 Max.
Si	0.10 Max.	0.10 Max.
Mn	0.10-0.35	0.10-0.35
P	0.010 Max.	0.010 Max.
Cr	0.80-1.10	0.35-0.60
Mo	0.80-1.10	0.35-0.60
V	0.06-0.12	0.06-0.12

Available data indicate that there is little difference in the carbon content in the 9Ni-4Co-.20C and 9Ni-4Co-.25C alloys.²⁵ The 9 wt% Ni gives the alloy hardenability and toughness, and promotes retention of austenite. Cobalt counteracts the austenite retention and also aids self-tempering by raising the M_s and M_f temperatures to 590°F and 480°F respectively in the low carbon alloy. The self-tempering of the martensite toughens the as-quenched or as-welded metal. Carbon increases the strength of the alloy, but it also promotes brittleness; therefore the carbon content is kept as low as possible, consistent with the desired strength level.³⁵ The 9Ni-4Co-.20C alloy was specifically developed for weldability. Chromium, silicon and molybdenum are included in the alloy for solid solution strengthening and hardenability. The higher molybdenum and chromium contents of the 9Ni-4Co-.20C alloy result in high toughness and increased strength owing to secondary hardening or intermetallic precipitation. Nominal mechanical properties of the 0.20 and 0.25 carbon content alloys are summarized in Table 16.

9Ni-4Co-.25C – SCC

Although a higher carbon alloy (9Ni-4Co-.45C) has displayed marginal resistance to SCC, the lower carbon 9Ni-4Co-.25C alloy has excellent resistance to SCC.³⁶ This SCC resistance appears to be related to the high fracture toughness of the low or carbon alloy.³⁷ Bieffer immersed three spring-loaded bent beam specimens of 9Ni-4Co-.25C (190 ksi YS) in 3.5% NaCl solutions and none of the specimens failed in 7 days. Specimens of the

Table 16. Nominal Mechanical Properties of 9Ni-4Co-0.25C and 9Ni-4Co-.20C Alloy Plate*³⁴

Mechanical Property	9Ni-4Co-.25C	9Ni-4Co-.20C (Cr, Mo)
Yield strength, ksi	190	180
Tensile strength, ksi	200	190
Reduction of area		
(L)	60	65
(T)	55	60
Elongation in 1 in., %		
(L)	16	18
(T)	14	16
Charpy impact, ft-lb	40-50	45-55
Precracked charpy W/A, (in.-lb/in. ²)		
(L)	3300	3900
(T)	2800	3500
Slow bend – K_{Ic} (fatigue cracked), (ksi $\sqrt{\text{in.}}$)	135-165	150 (nom)

*Oil quenched from 1500/1550°F and subsequently tempered at 1000°F – 2Hr + 2Hr tempering time.

same type were unbroken in normal sulfuric acid after 4 hours. Similar specimens immersed in "poisoned" (15 drops of 2 g phosphorous in 40 ml CS₂) normal sulfuric acid failed in 0.8 hour and failed in boiling calcium ammonium nitrate in 2.7-3.0 hours.³⁸

The effects of heat treatment were investigated in SCC tests with axially loaded specimens of a 9Ni-4Co alloy cycled through a 5% NaCl solution. The results are shown in Table 17. This research showed that the 9Ni-4Co had about one-fifth the susceptibility to stress-corrosion cracking as an 18Ni maraging steel. Better resistance to SCC was also observed for the 9Ni-4Co alloy in distilled water.³⁹

Davis observed no failures in 1000 hours when low carbon 9Ni-4Co steel specimens, loaded to 80% yield strength, were alternately cycled for 8 minutes in a 3.5% NaCl solution and for 52 minutes in air. Similar tests on the higher carbon (.45%) alloys resulted in failure between 263 to 389 hours. It was concluded that the high fracture toughness of the low carbon alloy was responsible for its good SCC resistance.^{36, 37}

Bent beam specimens of 9Ni-4Co-.25C were SCC tested in distilled water, tap water, salt water and high humidity air at 140°F. No failures were observed in any of the four environments with material tempered at 800°F. Failures were observed for specimens tempered at 400°F and 600°F in the high humidity atmosphere and also for the 400°F tempered material in distilled water.¹⁴

Table 17. Effect of Heat Treatment on Stress-Corrosion Cracking of High Nickel Alloy Steels.

Material ^a	Draw Temp. (°F)	Test Stress (ksi)	Time-To-Failure (hours)	Average Time-To-Failure (hours)
9% Ni-4Co				
Forging V	400	200	23.1, 48.2, 129.6	67
	450	200	26.5, 46.4, 128.1	67
Forging T ^b	400	200	24.5, 17, 2.7	19
	450	200	30.8, 5.8, 19.4	15
Forging N	450	200	11.7, 46.3, 91.7	67
			65.2, 53.8, 162.9	
	550	186	86.1, 139.4, 146.5	124
	700	169	NF ^c , NF	
	500 AQ	186	NF, NF	
18Ni Maraging Steel				
Forging W	900	200	5.5, 10.1, 7.6	8
	950	200	9.0, 10.0, 5.1	8
Forging S ^b	900	200	16.9, 5, 4.1	9
	950	200	7.6, 10.1, 5.5	14
Forging P	900	200	8.5, 7.3, 5.7, 24.8, 25.3	14

^aInitials T, V, N, W, S, and P are arbitrary designations of particular forgings.

^bMaterial overheated during forging.

^cNo failure after 200 hours.

9Ni-4Co — General Corrosion

No data were available in the literature on the general corrosion properties of 9Ni-4Co alloy. Somewhat better corrosion resistance would be expected for 9Ni-4Co-.20C alloy than for typical quenched and tempered steels, e.g., 4340 steel, due to the nickel in the 9Ni-4Co alloy.

9Ni-4Co-.25C — Welding

The low carbon grades of the 9Ni-4Co steels have been reported to have excellent welding properties due to the self-tempering of the weld metal and heat affected zone. No preheat or post-weld heat treatment is necessary for the low carbon alloys.³⁶ Welding properties of the 9Ni-4Co steels are similar to the 12Ni maraging steels and have the advantage of not requiring post-weld heat treatments. Welding is done in the quenched and tempered conditions.²⁵ Table 18 summarizes the best weld mechanical properties for 9Ni-4Co steels reported to the Defense Metals Information Center (DMIC).

Filler wires of various compositions have been used to weld 9Ni-4Co-.25C steel but no single composition has been recommended.²⁵ The addition of deoxidizing elements such as Zr, Ti, and Al in the filler wire is beneficial.³⁴

Studies by the U.S.A.F. showed that TIG welds, using filler metals of base metal or 7.5Ni-4Co, were comparable to the base metal. MIG (gas metal arc process) welds

Table 18. Best Reported Mechanical Properties for Welds in 9-4-X Alloys.^{25 a}

Material Thickness inch	Yield Strength, ksi ^b	Plane-Strain Fracture Toughness (K _{IC}), ^c ksi √inch	Charpy V-Notch Energy Adsorption, ft-lb	Tensile Strength, ksi ^b	Elongation in 1 inch, %	Welding Process	Energy Input, kj/in.
½	174 (L)	133 (W)	—	194 (L)	16 (L)	TIG	21
½	191 (T)	101* (W) 97* (HAZ)	—	217 (T)	10 (T)	TIG	20.5
2	181 (T)	—	52 at 32°F (W)	199 (T)	—	TIG (manual)	42
—	192 (L)	—	54 at 32°F (W)	208 (L)	17 (L)	TIG	—
1	187 (T)	119 (W)	—	—	—	—	—
1	187 (T)	119 (W)	32 at -80°F (W)	202 (T)	15 (T)	TIG	—

^aThe welds in the 9-4-25 alloy are in the as-deposited condition.

^bL indicates test parallel to weld; T perpendicular to weld.

^cAsterisk indicates values reported were less than the actual toughness. W indicates weld fusion zone; HAZ indicates weld heat-affected zone.

under the same conditions were not satisfactory due to carbon loss which lowered the yield strength. Weld porosity was a problem in these filler wire evaluation tests.³⁰

DMIC also concluded that TIG welding methods are preferable to MIG methods due to porosity and to the inability to obtain sufficiently inert atmospheres. DMIC also reports that the MIG welds have less strength than TIG welds due to loss of carbon. The same authors indicate that for weldable steels below the 200 ksi level, 9Ni-4Co-.25C is superior to 18Ni maraging steels on the basis of weld impact toughness, material costs, and post-weld heat treatment.²⁵

TIG weld joint efficiencies of nearly 100% may be obtained if the steel is welded in the annealed condition and then fully heat treated. The tensile properties and charpy V-notch properties of TIG welds have been shown to be equal to or greater than the base plate values.³⁵

Various welding studies conducted during the development of the alloy indicate that weld porosity may be minimized by good cleaning techniques. The excellent results now achieved in the low carbon alloy weldments are attributable to alloy toughness and self-tempering of the weld metal and heat-affected zone.³⁶

Cleaning Process

Since cleaning operations can introduce hydrogen into high strength steels resulting in HSC, special procedures are required to clean these steels.

A number of cleaning processes such as acid pickling, acid cleaning, vapor blasting, and cathodic cleaning introduce hydrogen into the metal.⁴⁰ Although the steels included in this survey do exhibit good resistance to HSC,⁴¹ the possibility of failure by HSC is not precluded. Consequently, these methods would be unacceptable for cleaning high strength steels.

A cleaning method which included an inhibited acid bath for cleaning martensitic steel was investigated; hydrogen embrittlement resulted which was subsequently eliminated by bakeout techniques.⁴² Other studies have been conducted to determine the efficiency of proprietary acid inhibitors for retarding hydrogen embrittlement. It was concluded that little protection from hydrogen embrittlement was afforded by acid inhibition and acid pickling of high strength steels should be avoided.^{40,43-45}

The importance of cleaning the weld filler wire as well as the base metal has been described by one researcher.

Contaminant residue from wire drawing lubricant introduces hydrogen which can cause underbead cracking.³³ Cleaning techniques that do not introduce hydrogen into the metal include abrasive cleaning (grit blasting), anodic pickling, solvent degreasing and alkaline cleaning.^{40,42} Grit blasting and anodic pickling would not be acceptable methods for cleaning due to excessive metal removal. Thus, by elimination, solvent degreasing and alkaline cleaning remain as satisfactory cleaning methods which preclude hydrogen embrittlement and excessive metal removal.

Ultrasonic cleaning employing alkaline detergents would be adequate for soil removal if the parts to be cleaned exhibit a minimal amount of oil and dirt. Solvent degreasing or an alkaline soak followed by ultrasonic cleaning with a mild alkaline detergent would be necessary if heavy dirt residues are to be removed.

The following procedure is tentatively recommended for cleaning the high strength steels (Almar 362, HP-9Ni-4Co, and Vascomax 250):

1. Preclean the parts by vapor degreasing with Freon TF[®] (5 minutes).
2. Air dry.
3. Ultrasonically clean the parts with a mild alkaline detergent such as Immunol 517-6[®] or Oakite 162[®] (15 minutes).
4. Rinse in flowing tap water (5 minutes).
5. Rinse in distilled or deionized water (5 minutes).
6. Dry the parts with forced dry air (filtered and oil free).
7. After cleaning, handle the parts with *clean* white gloves.

This method would be suitable for cleaning the steels at any point during the fabrication process.

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