A NEW Ni-Cr-Mo ALLOY 59, UNS N06059, FOR PROVIDING COST-EFFECTIVE/RELIABLE SOLUTIONS TO VARIOUS MAINTENANCE AND CORROSION PROBLEMS IN NAVAL APPLICATIONS

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ABSTRACT

US Navy uses many metallic materials such as carbon steel, stainless steels, nickel alloys, copper alloys, titanium alloys and aluminum alloys in their ships. Some of the major concerns are pitting/crevice corrosion, stress corrosion cracking, hydrogen embrittlement, ability to weld repair small worn and corroded areas, prevention of galvanic corrosion and reliable usage of high strength fasteners. Even though several stainless steels and nickel-based alloys have shown promise and are used in marine environments, under very severe crevice corrosion conditions, most of these have suffered from localized crevice attack, including high alloys like alloy 625 and alloy C-276. Fasteners of Monel alloy K-500 have shown to suffer from stress corrosion cracking. Fasteners of alloy 718 have suffered from localized attack. The search for alloys that are essentially immune to localized crevice corrosion attack in marine environments and provide reliable high strength fasteners for replacing K-500 fasteners , led the US Navy to consider Ni-Cr-Mo alloys with the highest combination of chromium and molybdenum in a nickel matrix. One such pure ternary alloy, alloy 59 (UNS N06059) having a typical chemical composition of 59% nickel, 23% chromium, 16% molybdenum and iron levels of less than 1%, appears to have fulfilled this need. Extensive laboratory and field tests by various companies and corrosion laboratories in USA, U.K, Norway and France have shown this alloy to be essentially immune to crevice corrosion attack. Due to the SCC problems associated with high strength fasteners of alloy K-500, extensive testing of cold reduced bars of alloy 59 to yield strength levels greater than 150 KSI have been conducted. Cold reduced bars of alloy 59 have shown excellent resistance to localized corrosion, hydrogen embrittlement and stress corrosion cracking while possessing very high strength, ductility, fracture toughness, high-cycle fatigue resistance and very low crack growth rate both in air and sea-water.. This paper presents a brief description of this alloy's development, its physical metallurgical characteristics, and localized corrosion resistance data from various test programs, data on repair welding techniques with alloy 59 filler metal via electro-spark deposition technology and its potential use as high strength fasteners.

Key Words: Marine corrosion, seawater corrosion, alloy 59, UNS N06059, crevice corrosion, localized corrosion, stress corrosion cracking, chlorinated sea-water, stagnant sea-water, weld repair, overlay welding, electro-spark deposition, laser clad, high strength fasteners, cold reduced bars, fasteners, ASTM F467, ASTM F468

INTRODUCTION

Materials used in the marine environment, such as the naval, coastal and offshore applications, encounter numerous corrosion problems. The corrosion problems of primary concern are uniform corrosion, localized corrosion (pitting and crevice), stress corrosion cracking, galvanic corrosion, corrosion fatigue, and erosion corrosion. A large amount of corrosion data has been generated over the last few decades and is well publicized in the technical literature.[1-9] Even though the precise determination of all corrosion variables as related to site specific marine corrosion is not fully categorized, there is ample laboratory, field, and case history experience available to make cost effective and functionally reliable maintenance-free material selection. Table 1 lists the various classes of materials, usually specified and used in seawater service, whereas Table 2 lists the nominal chemistry of some of these alloys. Coated carbon steel, along with most of the materials listed in Table 1 and Table 2 have been successfully used in marine applications although in certain very specific severe crevice corrosion conditions, the performance has not been totally satisfactory. The following sections describe the general metallurgical characteristics, corrosion resistance, mechanical properties and results of marine testing programs conducted at or by various institutions on Nicrofer® 5923hMo, alloy 59 (UNS N06059) including the US Navy, along with a few applications in media with very high chloride contents.

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Table 1
Various Classes of Materials for Marine Application

Non-Metallic	Typical Material
• Fire glass reinforced plastic	FRP
• Carbon steel coated with	Epoxy resin/rubber
• Carbon steel coated with	Concrete/cement
Metallic	
• Carbon steel	-----
• Carbon steel coated with	Zinc
• Copper base alloy	90/10 CuNi, 70/30 CuNi
• Aluminum Alloys	Various grades
• Ferritic SS	29-4, 29-4-2
• Ferritic austenitic SS (Duplex)	Alloy 2205, 2506
• Standard austenitic SS	316, 317LMN
• 6Mo super-austenitic SS	Alloy 1925hMo, Alloy 31
• Nickel alloys	Alloy 400, K-500, 825
• Ni-Cr-Mo high performance alloys	Alloy 625, 22, C-276, 686, 59

Table 2
Nominal Composition of Some Marine Alloys
Nominal Composition

Alloy/Structure	Cr	Ni	Mo	Fe	Others
Ferritic					
29-4-2	29	2	4	Bal	---
29-4	29	-	4	Bal	---
Austenitic					
904L	20	25	4.5	Bal	Cu
825	22	40	3.2	31	Cu
Ferritic-austenitic					
(Duplex) 25Cr	25	5.5	3.2	Bal	Cu
Standard 6Mo Alloys	21	25	6,5	Bal	Cu , N
High Cr 6 Mo alloy 31	27	31	6.5	Bal	Cu , N
Cu-Ni Alloys					
90/10	-	10	-	1.5	Cu Bal
70/30	-	30	-	0.1	Cu Bal
Nickel Alloys					
400	-	66	-	1	Cu Bal
K-500	-	66	-	1	Al 2.7, Cu Bal
Ni-Cr-Mo Alloys					
625	21	61	9	3	Cb 3.5
C-276	16	58	16	5	W 4
22	21	57	13	3	W 3
686	21	57	16	2	W 4
59	23	59	16	<1	Al 0.3

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METALLURGICAL AND CORROSION CHARACTERISTICS OF ALLOY 59

Alloys of the Ni-Cr-Mo family, starting with alloy C, date back to the 1930's. Since then improvements in melting technology and a better fundamental understanding of the role of various alloying elements have led to newer Ni-Cr-Mo alloys. Their typical chemical composition is given in Table 3. The physical metallurgy and corrosion resistance (uniform corrosion, localized corrosion, thermal stability) of Ni-Cr-Mo alloys are very well documented in the open literature, including many applications of alloy 59 in chloride containing environments [10-13] & others.

Table 3
Typical Chemical Composition of the "C" Family Alloys

Alloy (UNS #) - Decade Introduced	Ni	Cr	Mo	W	Fe	Others	PRE*
C (N10002) - 1930's	Bal	16	16	4	6	--	69
625 (N06625) - 1950's	Bal	22	9	--	3	Cb	51
C-276 (N10276) 1960's	Bal	16	16	4	5	--	69
C-4 (N06455) - 1970's	Bal	16	16	-	2	--	69
22 (N06022) mid-1980's	Bal	21	13	3	3	--	65
686 (N06686) mid-1990's	Bal	21	16	4	2	--	74
59 (N06059) early1990's	Bal	23	16	-	<1	--	76

* PRE = Pitting Resistance Equivalent = % Cr + 3.3 (% Mo) + 30 N

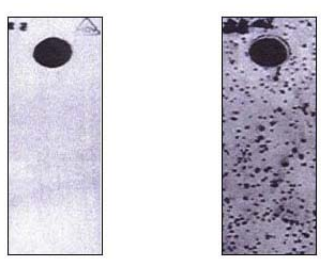
Alloy 59- UNS N06059 is one of the highest nickel containing alloy of the Ni-Cr-Mo family without addition of other alloying elements such as tungsten, copper, or titanium and hence can be classified as the purest ternary form of a "Ni-Cr-Mo" alloy. It also has the highest PRE number (Table 3), which is responsible for its superior crevice corrosion resistance behavior as measured by the ASTM G-48, 10% $FeCl_3$ test solution (Table 4). It has been shown by Garner [14] that the ASTM G-48 ferric-chloride test does provide a conservative prediction of crevice corrosion behavior in ambient sea-water for a wide range of alloys.

Table 4
Localized Corrosion Resistance in 10% $FeCl_3$ Solution (ASTM G-48)

Alloy	Cr	Mo	PRE	CPT(°C)	CCT (°C)
625	22	9	51	77.5	57.5
22	21	13	65	>85 *	58
C276	16	16	69	>85	>85
59	23	16	76	>85	>85

* Above 85°C, the 10% $FeCl_3$ solution chemically breaks down, i.e. decomposes by hydrolysis

Absence of tungsten in alloy 59 is responsible for its excellent thermal stability, a point which becomes very important in overlay welding and multi-pass welding of thick sections. Alloy 59 was the only alloy without any localized or inter-granular attack as shown by the results in Figure 1 and Table 5. Clearly shown in Figure 1 is the extent of the severe pitting attack on the tungsten containing alloy 22. Other tungsten containing alloys like C-276 and 686 as well as the copper containing alloy C-2000 suffered attack similar to alloy 22 to varying degrees. Alloy 59 was the only alloy free of any localized attack expect where steel stamping was done for identification purposes. (Figure 1)



59 22

Figure 1: Thermal Stability of Alloy 22 vs Alloy 59

Table 5
Thermal Stability per ASTM G-28A and G-28B after Sensitization for 1 hr at 1600°F (871°C)

	Corrosion Rate (mpy)				
Media	**C-276***	**22***	**686***	**N06200***	**59****
ASTM G-28A	>500*	>500*	872*	116*	40**
ASTM G-28B	>500*	339*	17*	>500*	4**
Pitting Attack	Severe	Severe	Severe	Severe	None
Intergranular Attack	Severe	Severe	Severe	Severe	None

* Alloy C-276, 22, C2000 & 686 – Heavy pitting with grains falling due to deep inter-granular attack.
** Alloy 59 No Attack

LOCALIZED CORROSION TESTS ON ALLOY 59 IN MARINE ENVIRONMENTS

Shifler and Aylor's paper on considerations for the testing of materials and components in seawater [15] adequately describes the various factors affecting complexity of the seawater environment and the design of seawater corrosion testing to minimize experimental variations and best simulate service conditions. Pitting and crevice corrosion tests were conducted by various laboratories both in USA and Europe in seawater, a very complex and aggressive environment. These are briefly described below:

I. LaQue Center for Corrosion Technology, Inc. , Wrightsville Beach, N.C.

(This laboratory does not exist any more. It has been dis-banded and the ground leveled)

a) In 1990 a series of multiple crevice assembly (MCA) corrosion tests on sheet/plate material in filtered natural seawater at 30°C were conducted on alloy 59 for a period of 90 days. This alloy was totally resistant and no localized attack occurred.[16]

b) In 1992 a "joint alloy producer test program" was developed by LaQue to study the crevice corrosion in natural seawater of various sheet/plate materials. The test was run in filtered natural seawater for 60 days utilizing acrylic plastic washers. Alloy 59 was fully resistant in all tests with various metal and different crevice former surface conditions. Alloy C-276, another high alloy of the Ni-Cr-Mo family with 16% Mo, surface ground with 120 grit SiC paper showed some crevice attack [17] in one test.

c) Another series of severe crevice corrosion testing on alloy 59 tubulars was done in 1993 for a 60 day period in fresh strained natural seawater. The ambient temperature of once through seawater ranged from 26.0°C to 31.3°C. The daily average temperature during the course of 60 day test was 28.6°C. Despite a constant flow rate of 2.4 m/s, accumulation of a bio-film developed on tube ID walls. However, no hard shell fouling was observed during the 60 day test period. Tight crevice formers were prepared by cutting a 3 inch (76mm) long segment of vinyl tubing and these covered approximately 25 mm of the alloy 59 tube. Serrated nylon hose clamps were used to tightly secure the vinyl sleeve to alloy tube assembly. Alloy 59 tubes were totally resistant in this severe crevice test in seawater.[18]

d) In 1998, another series of crevice corrosion tests were included in a test program sponsored by the U.S. Navy for evaluation of materials for use in a new generation of seawater valves. The 180 days test results in filtered seawater at 85+/-5°F (29.5+/- 2.5°C) on both wrought and cast series of alloys (bronze, copper-nickel, nickel-copper, stainless steels, titanium alloys, nickel base Ni-Cr-Mo alloys and cobalt base alloys) were presented by Aylor in a Corrosion/99 paper [19]. Crevices were formed by PTFE and/or cloth impregnated rubber gaskets.

The basic conclusion of the results on Ni-Cr-Mo alloys from this study [19] were:

- In wrought condition alloy 59, alloy 22, alloy 686 and alloy UNS N06200 exhibited full resistance in quiescent seawater.
- In flowing seawater – wrought alloy 59 had a minor **superficial** indication of less than 0.01 mm (0.0004") on two of the 4 exposed sites, whereas wrought alloy 22, UNS N06200 and alloy 686 were totally resistant.

- On cast alloy condition in quiescent seawater only cast 59 (ASTM A494 grade CX2M) was fully resistant to localized corrosion. Cast 625 (ASTM A 494 grade CW-6MC) , cast C-276 and cast 22 (ASTM A 494 grade CX2MW), all showed significant crevice attack.
- On the cast alloy condition in flowing seawater, again cast alloy 59 and cast C-276 were the only alloys fully resistant, whereas cast alloy 625 and cast 22 showed significant crevice attack.

e) Due to the best overall performance in both the wrought and cast condition of alloy 59 taken together, it was decided to test components in a butterfly valve application, where severe stagnant corrosion conditions exist. The butterfly valve was disassembled after one year exposure at LaQue and the materials of construction evaluated. Alloy 59 was free from any localized attack [20].

II. **Corrosion Test Results at the US Navy's, Key West Test Facilities** :

Vinyl sleeves tube test configuration producing a severe crevice test to determine localized corrosion resistance was conducted in a flow loop under two conditions:

a) Continuous chlorination of 0.15 ppm chlorine in natural seawater – test duration up to 1324 days, temperature of natural seawater varied between 21 to 30°C in the summer and winter,

b) Seawater trough test – un-chlorinated continuous refreshed natural seawater – circulating at three gallons/minute – test duration from 92 days to 1324 days.

The following alloys were tested: Alloy 686 , alloy 625, alloy 59, and alloy C-276.

The test data was presented at the Sea-Horse conference meeting, Wrightsville Beach, NC in 1996. The major conclusions of this study of 1324 days exposure (approximately 4 years) are presented below:

- Alloy C-276 showed crevice / pitting attack up to 4 mil (0.1 mm) depth in the trough test (un-chlorinated seawater). In the flow loop test deep etch pitting was observed.
- Alloy 59 in the loop test showed some discoloration in two of the loops, etch is one loop and no attack or change in the other three loops after the 1324 days (approx. 4 years) exposure.
- In the trough test there was no indication on alloy 59 after 876 and 1000 days when the test was stopped. Alloy 625 showed some etch only in one loop, no damage in the other 5 loops and no damage in the trough test after 1324 and 876 days respectively.
- Another sample of alloy 625 showed etching in three of the loop tests and no damage in other 4 loops, whereas in trough test alloy 625 showed crevice attack after 180 days.
  - Alloy 686 showed deep etch in the loop test after 229 days.

III Corrosion Tests in the U.K.

John W. Oldfield [21] showed that over a two year crevice corrosion exposure tests conducted at UK's Defense Research Agency on contract to Cortest in Holten Heath, Poole, U.K. , alloy 59 gave the best performance. The theoretical modeling in the 20-25°C range predicted alloys ranking order from best to worst as: Alloy 59 > Alloy 22 > Alloy C-276 > Alloy 654SMo > Alloy 625 > Alloy 24. At 50°C the ranking from best to worst changed to: Alloy 59 > Alloy C-276 > Alloy 22 > Alloy 24 > Alloy 625 > Alloy 654SMo. The obtained data from exposure tests, did confirm the ranking in resistance to initiation of crevice attack i.e. alloy 59, alloy 22, and 654SMo performed the best (however, a small pit was observed at the crevice edge of both alloy 22 and alloy 654SMo) followed by alloy 24, then alloy 625, with alloy C-276 showing the greatest number of attack initiation sites in the PVC/metal crevice geometry in both natural seawater and chlorinated seawater. Overall alloy 59 gave the best performance, both as predicted per the model and verified by exposure tests.
Agreement with the model predictions of resistance to initiation of attack at ambient temperature showed general agreement in the relative performance of alloys 59, 22 and 654SMo, but agreement was **not shown** with the results of alloy C-276, which from all published data predicted to be better than alloy 625 due to its significantly higher molybdenum content and higher PRE number. Generally alloy C-276 has outperformed alloy 625 in numerous exposure tests in marine/seawater environments. This discrepancy in this exposure could be explained by the surface roughness of the sample. It is generally accepted that finer the roughness of the sample surface, greater is the severity for crevice corrosion attack. In these tests, the finer surface roughness values on alloy C-276 samples ($R_A \approx 0.47$ microns) in comparison to alloy 625 ($R_A \approx 1.90$ microns), created a much more severe crevice condition on alloy C-276, thus the more severe attack in comparison to alloy 625. At least this is a possible explanation for this discrepancy.

IV Corrosion Tests at SINTEFF Material Technology, Trondheim, Norway

A series of corrosion tests were done on weld overlays of nickel based alloys to determine the localized corrosion behavior. Three types of tests were done. These are defined and explained in Corrosion/98 paper (7).

1) Pitting corrosion test per ASTM G48A
2) Crevice corrosion test per Material Technology Institute. MTI manual No. 3 procedure MTI-2.
3) Crevice test per SINTEFF's test method.

Various nickel based alloys (alloy 625, 59, C-276, C-4, 22) were over-layed using three welding methods, i.e. GTAW, SMAW, and PTA (Plasma Transferred Arc). The substrate used in all cases was a 6 Mo alloy. The conclusion from this study was that alloy 59 weld overlay was the best amongst all alloys tested and for all welding processes employed. Details on the test procedures & results obtained were previously presented at Corrosion/98 (7).

V Crevice Corrosion Tests at Cherbourg Naval, France

Various tests were conducted, which involved potentiostatic tests at 300 mV vs. SCE, crevice corrosion test assemblies and potentiodynamic tests in natural seawater. One of the conclusions of this study was that alloy 59 was superior to alloy 625 in the various tests conducted.

The other major conclusion of this study was that even though initiation of crevice corrosion under certain conditions can occur on even 16% Mo alloys like alloy 59 and C-276, the propagation rate is significantly lower than alloy 625. The details on the various tests were published in Euro Corr'99 Proceedings (22).

VI Corrosion Testing in Natural Seawater with CO_2 + H_2S Addition

Some oil companies were interested in getting corrosion data on various materials on commingled seawater (aerobic and anaerobic) with produced water. Mixing produced water with chlorinated seawater increases the injection temperature and due to chemical reaction with organic material in the produced water, generally all chlorine is removed. Additionally, H_2S in produced water is oxidized to sulfur in contact with aerobic seawater, potentially introducing an additional corrodent, which increases the corrosiveness of the media.

Tests in elevated temperature natural seawater were conducted for a period of 134 days with addition of CO_2 in one test and CO_2 plus H_2S in another test.
The particulars of testing parameters were as follows:

- Seawater with CO_2 added

Duration 134 days, Temperature 70-80°C, CO_2 addition pH 4.92 to 5.2, Flow rate - 3m/s, Oxygen 2.6 ppmw

- Seawater with CO_2 + H_2S added

Duration 120 days with H_2S, 134 days with CO_2, Temperature 69-75°C, Addition of CO_2 resulted in a pH of 5.0 and H.$_2$S was added to give a residual value of 50 ppmw, Flow rate – approx. 3 m/s, Oxygen – None
Conclusion from this study was:

- No corrosion was observed on alloy 59 tube samples in either flow loop. It was concluded that alloy 59 is totally suitable for marine environment in the presence of H_2S and CO_2. The details of this test are provided in a private communication with CAPCIS, U.K.

VII Sulfide Stress Corrosion Cracking

Alloy 59 bars in the cold reduced condition from 3 different heats to 0.2% YS strength levels of 135 ksi, were tested per NACE TMO177 (24) sulfide stress cracking tests for 720 hours at 100% of the yield stress. No failures were observed. These tests were repeated with specimens coupled to steel. Again no failures occurred in 720 hours. Tests were also done in uniaxial constant load mode at 100% YS applied stress in high temperature/high pressure autoclave at 400 deg. F. The environment consisted of a high chloride brine solution of 25% NaCl with 150 psi partial pressure of H_2S and 200 psi of CO_2 partial pressure. Additionally one gram per liter of sulfur was also added. No failures occurred in 720 hours, after which the test was stopped. This indicated that the stress corrosion cracking of alloy 59 was relatively resistant in the cold reduced condition in sulfide and chloride environments. These results led to incorporation of alloy 59 up to a hardness level of RC 35 max in the NACE MRO-175 specification [24] and the ISO 15156 specification.

VIII Chloride Stress Corrosion Cracking test in 42% boiling $MgCl_2$

The material as a U Bend was tested in boiling 42% $MgCl_2$. After 720 hours no cracks were observed, the test was then stopped. This boiling $MgCl_2$ is one of the most severe test for determining stress corrosion cracking susceptibility of any alloy.

IX Resistance in Chlorinated sea-water

Recent tests conducted in chlorinated sea-water showed alloy 59 to be totally resistant to any pitting or crevice attack up to 75 deg C. Detail of the work are published in open literature [25] .

WELD REPAIR USING NEW TECHNOLOGIES

Repair of shipboard components due to wear and corrosion as a result of exposure to marine environment, leads to periodic but frequent maintenance. Methods to repair these components in a cost effective manner led the US Navy to evaluate a micro-welding process known as " Electrospark Deposition" (ESD) [26]. The research conducted indicated that ESD was a viable in-situ method for repairing small worn and corroded areas on nickel and copper base alloys. Crevice corrosion testing of electrodeposited alloy 59, 625 and C276 showed that alloy 59 was free from any localized attack after 365 days immersion in seawater, similar to the wrought alloy 59 control test coupon, whereas both ESD alloy 625 and C276 did initiate crevice corrosion attack.

Another technology bearing promise is " Laser Clad".Details are published in a paper presented at the 2005 International Congress on Applications of lasers and Electro-Optics [27]. The title of the paper is " Pitting resistance of Laser Clad Alloys 625, C276, 59 and 686" with one of the co-authors being Seanda Williams of the Naval Undersea Warfare Center Division Keyport, Keyport, WA 98345. This paper showed that alloy 59 and 686 had better pitting resistance than alloy 625 and C76 and in Tale 4 of this paper alloy 59 was free from any pitting attack whereas alloy 686 was pitted in ASTM G28 test (see below).

ASTM G28 Test

Alloy	Condition	Corrosion Rate (mpy)	Pitting
686	Laser clad with wire	4.6	Yes (0.013 mm deep)
59	Laser clad with wire	1.0	No
59 Powder	Laser clad with powder	2.7	No

EROSION RESISTANCE TESTING

Due to significant costs associated with maintenance of seawater valves and piping in fire main and auxiliary seawater cooling systems in Navy, a project to evaluate candidate valve materials in simulated severe seawater piping system service was initiated. Details of the erosion corrosion test loop design, material tested and results obtained are published in Corrosion/2001 paper by Ruedisueli and Aylor [28]. Alloy 59 among all Ni-Cr-Mo alloys (59, 22, 686 and C2000) had the lowest mass loss with alloy 22 showing the highest mass loss. The most resistant alloys in this evaluation were cobalt base alloys, 316L, alloy 654 and CP Ti Grade 2.

HIGH STRENGTH FASTENERS FOR SEAWATER SERVICE

Fasteners used in naval applications not only require high strength but must also have resistance to crevice corrosion, resistance to SCC and hydrogen embrittlement and must posses sufficient toughness. Alloy K500 has been extensively used but has suffered from SCC and hydrogen embrittlement and accelerated corrosion due to galvanic interaction with more noble metals. The US Navy has an on going program to find suitable material/s to replace alloy K500. Alloy 59 has been selected by the US Navy as one of the candidate materials for high strength fastener applications, which will be machined from cold reduced bars of alloy 59. Alloy 59 was selected due to its excellent resistance to crevice corrosion in marine environments, resistance to stress corrosion cracking and hydrogen embrittlement , high strength, excellent ductility and very high charpy impact values and toughness properties and high fracture toughness values meeting the design requirements of high strength fasteners.

Mechanical properties of cold reduced bars of alloy 59

Table 6 gives the various mechanical property data at room temperature, 400° F and 700° F of cold reduced bars of alloy 59. As is evident, the alloy exhibits excellent strength and ductility even in the heavily cold worked condition. Figure 2 shows the mechanical property (UTS & YS) as a function of cold work at room temperature. Table 7 shows the Charpy impact values at room temperature and – 40°C. Even in the highest cold reduced condition, the impact values are close to 200J at – 40°C.

Table 6
Mechanical properties of cold reduced bars of alloy 59 for fastener applications

% CW	Temp. °F	UTS (KSI)*	0.2% YS (KSI)*	El.%	R.A %
Annealed	Room Temp.	106	50	68	76
Annealed	400	98	44	62	71
Annealed	700	90	36	65	61
9.6% CW	Room Temp.	123	77	55	75
9.6% CW	400	111	67	58	70
9.6% CW	700	103	64	55	60
14.2% CW	Room Temp.	133	98	45	73
14.2% CW	400	118	85	42	71
14.2% CW	700	110	76	39	70
22.15% CW	Room Temp.	149	128	36	71
22.15% CW	400	130	112	33	65
22.15% CW	700	123	102	32	60
29.42% CW	Room Temp.	164	152	25	65
29.42% CW	400	146	130	25	63
29.42% CW	700	141	125	24	60
42% CW	Room Temp.	225	192	16	52
42% CW	400	177	175	16	50
42% CW	700	170	168	15	46

To covert ksi to MPa, multiply by 6.9

Table 7
Charpy Impact Values (J)* vs cold reduction of alloy 59 bars

% CW	Temp. °F	1 (J)	2 (J)	3 (J)	Average (J)
Annealed	Room Temp.	> 300	> 300	> 300	> 300
Annealed	- 40 °F	> 300	> 300	> 300	> 300
9.6% CW	Room Temp.	> 300	> 300	> 300	> 300
9.6% CW	- 40 °F	> 300	> 300	> 300	> 300
14.2% CW	Room Temp.	282	280	285	282
14.2% CW	- 40 °F	284	280	284	282
22.15% CW	Room Temp.	246	248	2511	248
22.15% CW	- 40 °F	246	245	243	245
29.42% CW	Room Temp.	235	241	233	236
29.42% CW	- 40 °F	229	233	233	231
42% CW	Room Temp.	206	227	193	205
42% CW	- 40 °F	181	187	186	185

* To convert J to ft-lbs, multiply J values by 0.7375

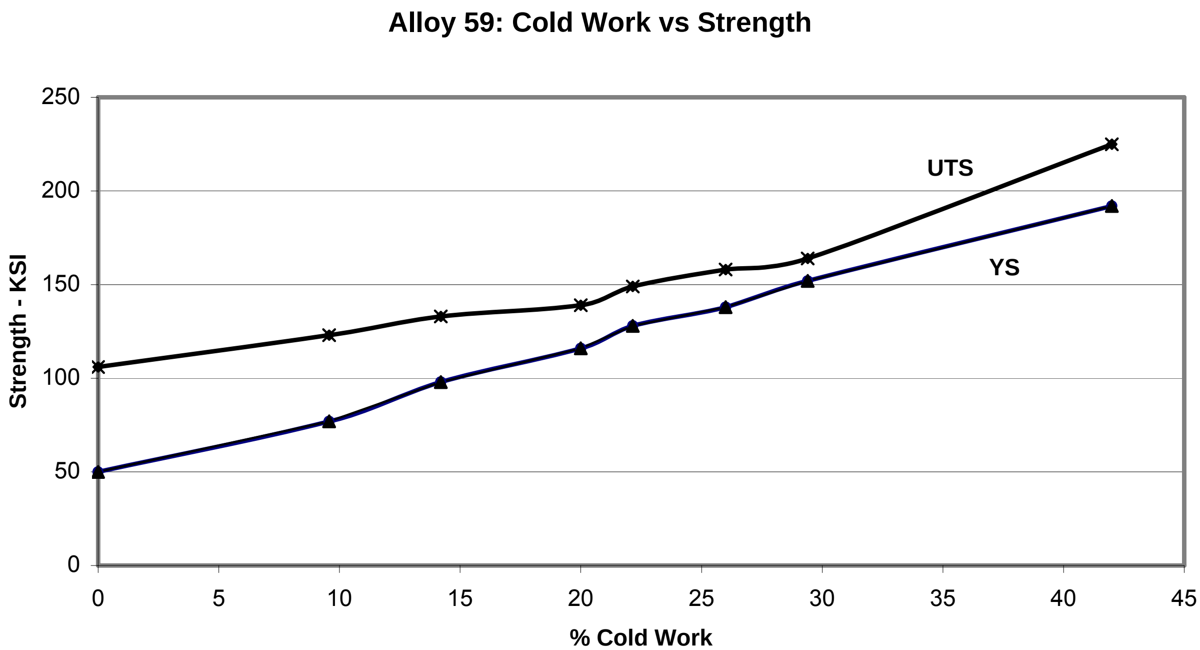


Figure 2: Cold work vs room temperature strength of alloy 59 bar

Corrosion Resistance

Concern had been raised that the cold work may degrade the corrosion resistance of alloy 59 bars meant for sea-water fasteners applications. To prove or disprove this, many standard lab tests were conducted in various solutions. These are presented below in Table 8.

Table 8

Tests Conducted on Annealed and Cold Worked Bars of Alloy 59 at various cold work levels

Corrosion Media	**Solution Annealed**	**9.57% CW**	**14.2 % CW**	**22.15 % CW**	**29.4 % CW**	**42% CW**
ASTM G28A	1.11 mm/y	1.13 mm/y	1.38 mm/y	1.28 mm/yy	1.23 mm/y	1.28 mm/y
ASTM G 28B	0.25 mm/y	0.25 mm/y	0.35 mm/y	0.36 mm/y	0.28 mm/y	0.25 mm/y
ASTM G48 C	CPT > 85 °C	CPT > 85 °C	CPT > 85 °C	CPT > 85 °C	CPT > 85 °C	CPT > 85 °C
ASTM G48 D	CCT > 85 °C	CCT > 85 °C	CCT > 85 °C	CCT > 85 °C	CCT > 85 °C	CCT > 85 °C
ASTM G61	**See Fig. 3**	**------**	**------**	**------**	**See Fig. 4**	**See Fig 4**
Green Death*	CPT > 95 °C	CPT > 95 °C	CPT > 95 °C	CPT > 95 °C	CPT > 95 °C	CPT > 95 °C

* Green Death Solution: 11.5% H_2SO_4 + 1.2 % HCL + 1% $FeCl_3$ + 1% $CuCl_2$

In the ASTM G28 A and B test, even in the 42% cold worked condition, the values are within the statistical scatter for fully solution annealed bars. In the localized corrosion test in 10% $FeCl_3$, there is no difference between the critical pitting and critical crevice temperatures of annealed and cold reduced bars. No degradation of properties occurred. Similar results were also obtained in the “Green Death” pitting test. This is a very corrosive solution with very low pH and the presence of oxidizing species. Cyclic potentiodynamic polarization measurements for localized corrosion susceptibility were conducted per ASTM G61 in 3.56 weight percent NaCl solution. An indication of the susceptibility to initiation of localized corrosion in this test is given by the potential at which the anodic current increases rapidly. The more noble this potential, obtained at a fixed scan rate, the less susceptible is the alloy to initiation of localized corrosion. Figure 3 shows the cyclic curve for the annealed alloy 59 and Figure 4 shows the comparison curve data for annealed, 29% and 42% cold worked material. As is evident, there is no difference between the curves. **Hence it can be safely stated that cold work does not degrade the corrosion resistance properties of alloy 59 even to very high strength levels.**

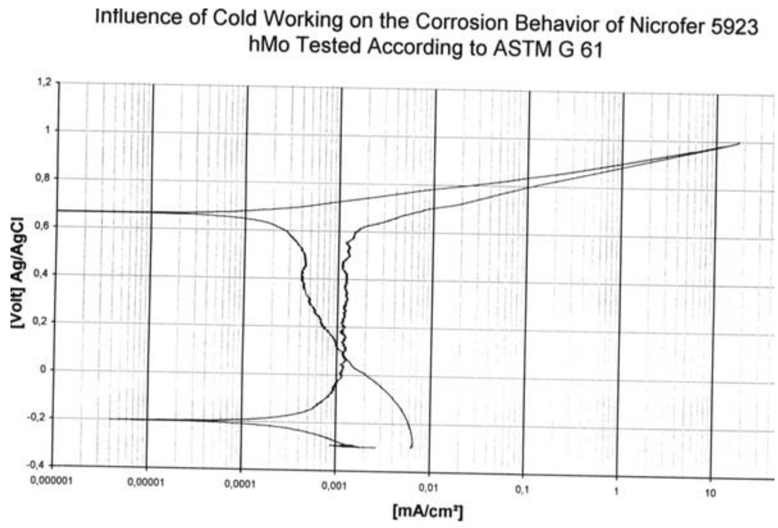


Figure 3 : Potentiodynamic curve for annealed alloy 59

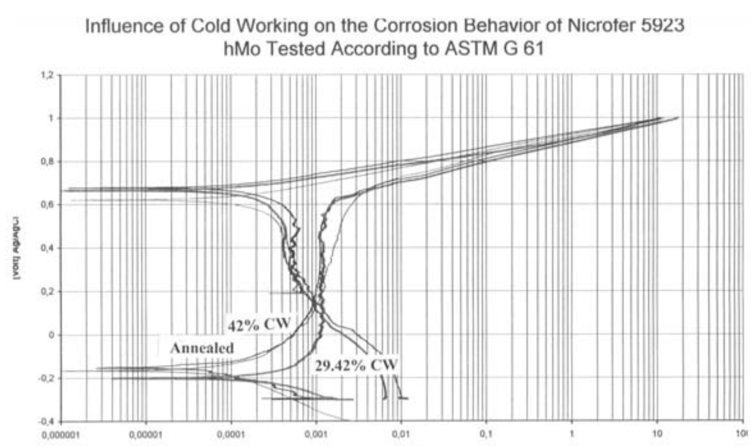


Figure 4 : Combined Potentiodynamic curves for annealed and cold worked alloy 59

At the request of US Navy, some other tests were conducted on cold reduced bars of alloy 59 as shown in Table 9 below. These tests were done at outside laboratories (Westmoreland Testing Labs and Laque labs). The results of these tests are presented in Corrosion/2005 paper [29]. A brief summary of the tests and conclusions from this paper are presented here as well.

Table 9

At US Navy's request :Other Mechanical and Corrosion Tests for Cold Reduced Bars of Alloy 59

1) Fastener Tests on 3/4-in UNC fasteners machined from cold reduced bar stock:
- Fastener Tensile per ASTM F606
- Cone Proof Load Test per ASTM F606
- Wedge Tensile Tests per ASTM F606 with 10° angle
- Torque-Tension Tests per SAE J174

2) Tests on samples machined from bar stock:
- High cycle fatigue using notched specimens in air and artificial seawater (ASTM E466)

Other mechanical and corrosion tests for cold reduced bars of alloy 59

- Fatigue Crack Growth Rate in air and artificial seawater (ASTM E647)
- Fracture Toughness (JIC) in air only (ASTM 1820)

3) Corrosion Testing:
- Crevice corrosion per ASTM G78 for 60 days in seawater
- Galvanic Compatibility per ASTM G71 with Alloy 625, Monel 400 and Ti-grade 2 for 60 days in seawater.

Brief Summary of the mechanical tests conducted at Westmoreland Labs [30]

The alloy 59cold reduced bar supplied for this test program had the following room temperature mechanical properties:

Size (inches)	UTS (ksi)	0.2% YS (ksi)	% R.A.	% El.
1.65”	165	146	50	25

Fastener Tests

Ten (10) ¾-10 x 5 UNC nut and bolt sets were machined from the bar for tensile test, proof load, cone proof load, 10° wedge test (all per ASTM F606) and fastener torque / tension per SAE J174. All testing was done at room temperature in laboratory air environment. The results are presented below in tables 1 through table 5.

Bolt tensile tests per ASTM F606

Sample #	UTS, ksi	0.2% YS, ksi	Failure Location
1	171	164	Threads*
2	170	163	Threads*

* Failure location in threads indicated that the test was valid.

Cone Proof Load test per ASTM F606

Sample #	Proof Load (lbs.)
1	44,020
2	44,020

Note: The test load used for the cone proof load tests was determined per ASTM F606-95B using the lowest calculated tensile stress as the specified proof stress of the nut. After testing, a wrench was used to break the nut loose from the mandrel. After loosening, the nut was removed by hand. Both parts passed the cone proof test without stripping or rupture. This also indicated that there was no measurable permanent deformation of the nut at these high proof loads and that the fastener is suitable for high strength fastener applications.

Nut Proof Load Test

Sample #	Proof Load (lbs.)	Pass / Fail
1	56,800	Pass
2	56,800	Pass

The test load for the nut proof test was the lowest recorded value. No permanent measurable deformation was noted at these high proof loads.

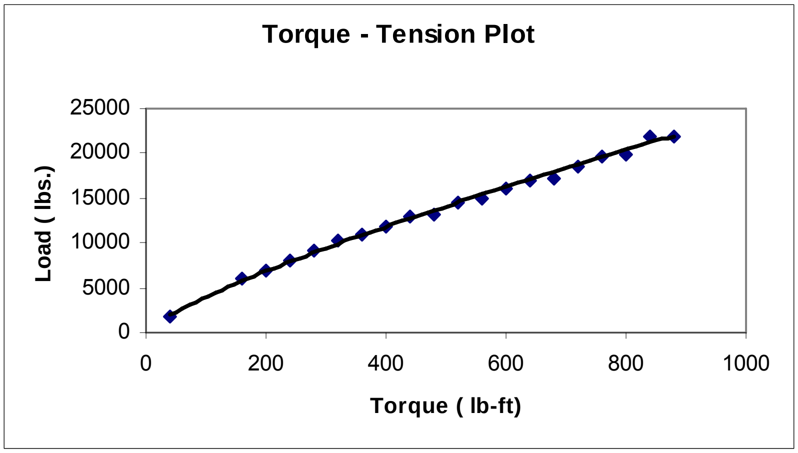
10° Wedge Tensile Test

Sample #	Ultimate tensile load (lbs.)	UTS (ksi)	Failure Location
1	57,030 lbs.	170	Threads*
2	56740 lbs.	170	Threads*

* Failure location in threads, indicated that the “head quality” of the bolt is excellent with good ductility. Failure at the junction of the head and shank is prohibited at any load in this test.

Torque – Tension Test

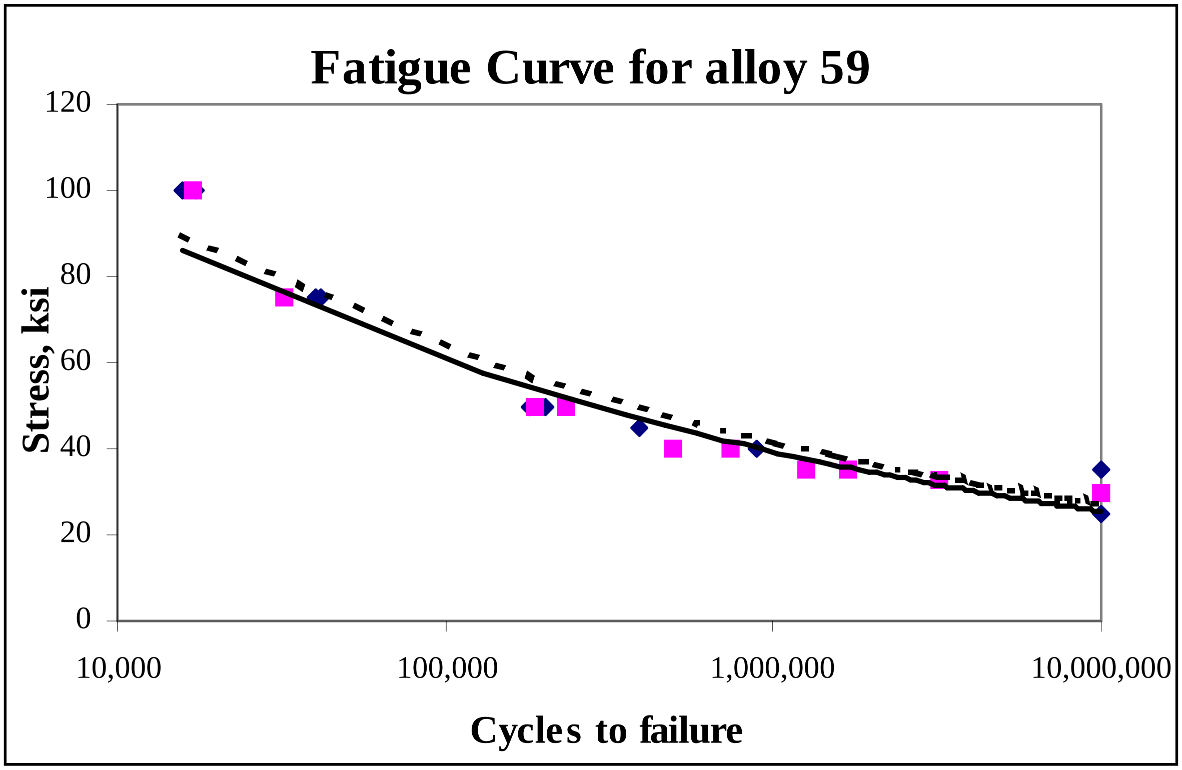
Torque Lb-ft	40	160	200	240	280	320	360	400	440	480
Tension load, bs	1826	6080	6870	8100	9250	10,180	10,910	11860	12910	13,240
Torque Lb-ft	520	560	600	640	680	720	760	800	840	880
Tension load, bs	14,580	15,060	16,110	16950	17290	18,450	19,610	19,860	21,900	21900



This test shows that alloy 59 can be torqued to high levels without yielding as shown by the linear relationship between the torque and the tension load.

High Cycle Fatigue Test in Air and Artificial sea-water

Twenty notched high cycle fatigue specimens were machined from the cold reduced bar of alloy 59 (notch K_T = 3.0) . Testing was done at room temperature per ASTM E466-99 with an R.ratio of 0.1; the loading was sinusoidal with a frequency of 30 Hz. Ten specimens were tested in air and the other ten in artificial sea-water (ASTM D1141) at various stress levels. A comparison of the stress-life curves indicates that alloy 59 exhibits very similar fatigue properties in both air and sea-water environments in the stress range tested. At 50 ksi, the data in both environments overlap. At lower stress levels, there is a very slight reduction of fatigue life in the sea-water environment. The graphical plot below shows the comparison of the fatigue curves in air vs artificial sea-water.



Fracture Toughness

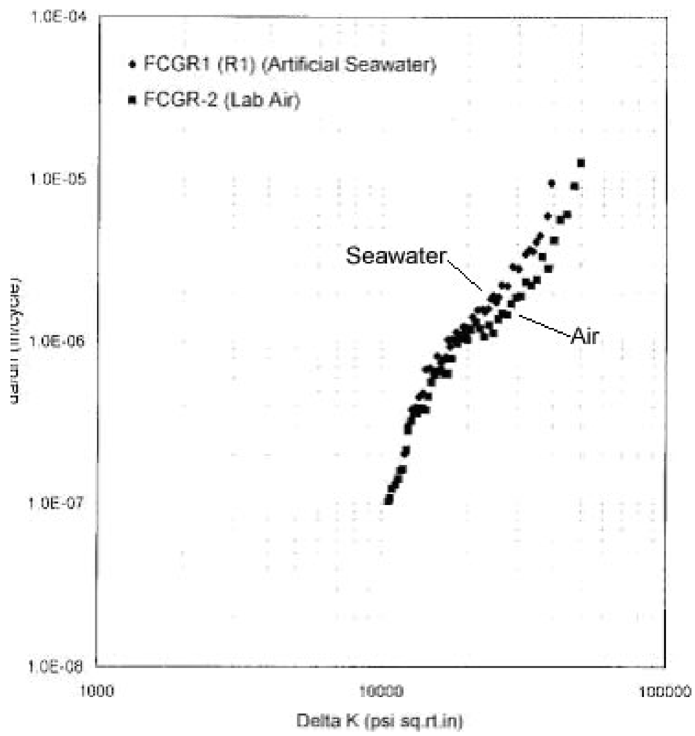
One 0.6-T compact tension (CT) sample was machined and tested in air per ASTM E1820-99a for J_{IC} fracture toughness. The specimen was machined in the L-R orientation. The sample was fatigue pre-cracked to a final a/w, of approx. 0.50 and then side grooved to a depth equal to 20% of the nominal thickness (10% per side). Based on the test result as shown below it is evident that the alloy 59 bar in the cold worked condition to a YS, of 146 ksi exhibits a very high fracture toughness value of Kjic of 287.12.

Sample #	Jic (E1820) (in- lb/in^2	Kjic (E1820) ksi (sqrt.in)	P_Q (E1820) lbs	K_Q (E1820) ksi (sqrt.in)	Unstable
JIC-1 L-R	2500.7	287.12	4612.8	76.2	No

Verification checks for J_{IC} and K_{JIC} were valid. Test for K_Q is invalid.

Fatigue Crack Growth Rate Testing

Two compact tension specimens were machined, pre-cracked and fatigue crack growth rate tested per ASTM E647-00. Both specimens were machined in the L-R orientation. Testing was done using an “r” ratio of 0.05 and a frequency of 10 hertz. One test was in air and the other in artificial sea-water at room temperature. The specimen crack length was continuously measured automatically using the electric potential drop technique. The log-log plot of “da/dn “(inches per cycle) vs ΔK (psi square root inch) characterizes, a material’s resistance to stable crack extension under cyclic loading. Figure 1 below shows that up to ΔK values of 20,000 psi√inch, the crack growth rate values of da/dn (inches per cycle) in air and sea-water are very similar. The curve for the sea-water begins to diverge very slightly at higher values of Delta K.



Plot of fatigue crack growth rate da/dn (inches per cycle) in air and sea-water vs Delta K
Where, Delta K is Stress Intensity Factor Range defined as K_{max} – Kmin.

Galvanic/ Crevice Corrosion Testing of Cold reduced alloy 59 bars in Sea-Water (Laque Labs) [31]

Testing comprised of exposing alloy 59 fasteners machined from the cold reduced bars in direct crevice-forming contact with plate material of alloy 625, alloy 400 and Ti-grade 2. Two area ratios of 5:1 (Metal: Alloy 59) and 10:1(Metal: Alloy 59) were tested. Like metal assemblies of the three plate materials with similar crevice geometries were also tested as controls. In addition, non-metal to metal crevice tests were performed with alloy 59 bar and alloy 625 pipe of the same diameter using vinyl sleeves.

Summary of the results obtained from the above corrosion testing:
The 60 day test in natural sea-water conducted at LaQue Center at Wrightsville Beach, North Carolina, USA to assess any galvanic effect resulting from direct metal coupling of cold reduced high strength alloy 59 with other marine grade materials of alloy 625, alloy 400 and titanium grade 2. Based on the 60-day test at a mean sea-water temperature of 25.1°C, the following conclusions are drawn:

- All Ti-grade 2 components were resistant to attack as were their mated alloy 59 studs.
- Alloy 625 studs surfaces in contact with alloy 59 incurred light crevice attack up to 0.02 mm in depth. No attack was observed on alloy 59
- Mass losses for alloy 625 and alloy 59 were insignificant and negligible.
- Alloy 400 components with or without alloy 59 attachments incurred the most attack. Significant mass loss of alloy 400 is attributed to a combination of classical pitting and crevice related damage.
- Relatively little difference in final and average corrosion potentials was measured for the various alloys 625 and Ti-grade 2 assemblies with and without alloy 59 studs. The couple assembly potentials were ~ 200 to 225 mV, slightly more noble, than alloy 59 control stud and alloy 59 bar assemblies fitted with vinyl sleeve crevice formers.
- Alloy 59 can be considered an excellent material of construction for marine fasteners.

Full details of this sea-water test program, is reported elsewhere [31].

Slow Strain rate test conducted by US Navy

Some other slow strain rate tests were conducted on cold reduced bars of alloy 59 by US Navy's Naval Surface Warfare Center, Carderock Division [32]. These tests were conducted in accordance with ASTM G129. The freely corroding and cathodically polarized specimens were immersed in ASTM ocean water (Method D1141). In all the specimens, the fracture surfaces showed a completely ductile trans-granular appearance which indicates no environmental cracking susceptibility. The notched specimens continued to yield even after max load was achieved, sometimes for a couple of hours before breaking. Generally specimens fail soon after max load is achieved. This indicates excellent resistance to stress corrosion cracking of cold reduced alloy 59. The data is presented below in Table 10.

Table 10

SSRT Tests on Cold Reduced Bars of Alloy 59 per ASTM G129

Alloy Identification	SSRT Environment	Time to failure (hours)	Max. Load (lbs)	Notch Dia. (in)	Max. Stress (psi)	Env.: Air ratio Average
59-1 59-2	Air Air	18 15.1	3070 2958	0.1259 0.1259	246602 237606	----- -----
59-3 59-4	Freely corroding	15.6 10.9	2985 2472	0.1257 0.1258	240538 198883	0.99 0.82
59-3 59-4	-850mV -850mV	14.9 12.7	3049 2936	0.1256 0.1252	246087 238483	1.01 0.97
59-3 59-4	-1000mV -1000mV	13.5 13.2	2844 2905	0.1258 0.1258	228812 233720	0.94 0.96

US Navy tests conducted at Navy Metalworking Center

Alloy 59 cold reduced bars were also tested under the "Metallic Materials Advanced Development and Certification Project (MMADCP) Task 3 (Certification of High Strength Marine Grade Mechanical Fasteners and Attachment Systems)". Alloy 59 was one of the several alloys that the Navy metalworking Center (NMC) , in co-operation with both the Naval Sea Systems Command (NAVSEA) and the Naval Surface Warfare center – Carderock Division (NSWCCD), has tested in support of certifying those materials for use as marine grade fastener as on US Navy surface ships and submarines. The results of the work are reported in a document prepared by Kevin L. Klug, report # TR No. 05-057 dated 23 November, 2005 [33]. The conclusion of this report was:

1) Alloy 59 offers a substantially higher impact toughness than the other two materials tested (MP98T and alloy 686) at the nominal 100+ ksi yield strength.
2) At the 150+ ksi yield strength level, alloy 59 displayed impact toughness that was substantially higher than MP98T and comparable to alloy 686
3) The mechanical test results contained in this document support the use of alloy 59 as a high strength marine grade fastener material in the nominal YS of 100+ ksi and 150+ ksi

DISCUSSIONS

Crevice corrosion in marine environments is of significant interest to the U.S. Navy and companies operating off-shore platforms because it has long been recognized as a limiting factor for use of stainless steels. Many researchers such as Lennox and Peterson [34] have concluded the statistical nature of crevice corrosion in higher alloys, i.e. the materials' response to crevice corrosion is unpredictable; it may or may not occur. Extremely small differences in the crevice geometry and/or surface conditions of the materials in a given environment can either lead to crevice corrosion or have immunity to crevice corrosion. The statistical nature of crevice corrosion behavior has also been discussed and argued by the other researchers [(35-38)] as well some recent data from tests using deeper and tighter crevices has revealed some susceptibility to crevice corrosion initiation for alloys even containing 16% Mo with a high pitting resistance equivalent number of 69 such as alloy C-276. **But the research has also shown the very high resistance of the 16% Mo Ni-Cr-Mo alloys to crevice corrosion propagation**.

Hence, looking at all the test data on corrosion resistance of Ni-Cr-Mo alloys as presented in this paper and by various other researchers, it can be safely concluded that amongst the various alloys of the Ni-Cr-Mo family such as alloy 625, C-276, 22 and alloy 59, the only alloy, which most closely provides optimum resistance and immunity to crevices corrosion in seawater, is alloy 59 (UNS N06059). This alloy is covered in the U.S. in the various specifications such as ASTM, ASME, AWS and NACE (MRO-175) and other various international specifications.
For fastener applications, the data generated, so far indicates that alloy 59 can be cold worked to high strength levels, is essentially immune to crevice corrosion in sea-water /marine environment, and has excellent resistance to hydrogen embrittlement and stress corrosion cracking. Its corrosion resistance properties are not degraded by cold working. Additionally the alloy offers an exceptional combination of strength, ductility and toughness.

CONCLUSIONS

- Alloy 59 (UNS N06059) having a typical chemical composition of Ni 59, Cr 23, Mo 16 and Fe <1, is the purest ternary alloy of the Ni-Cr-Mo family.
- It has the highest Pitting Resistance Equivalent number of 76, which accounts for its superior localized corrosion resistance in seawater and other chloride bearing environments.
- The low iron content, high PRE number and absence of tungsten or copper, makes alloy 59 more thermally stable than alloys like C-276, 22, 686 and UNS N06200. This is critical for overlay welding and multipass welding of thick sections.
- Data generated on localized corrosion behavior at the various test facilities clearly show alloy 59 to be superior to other Ni-Cr-Mo alloys and is essentially immune to crevice corrosion.
- US Navy's evaluation for repair of small worn out or corroded areas by electro spark deposition coatings, or laser cladding shows alloy 59 to be superior to other alloys of the Ni-Cr-Mo family.
- Alloy 59 showed superior performance over other Ni-Cr-Mo alloys during erosion tests for candidate seawater valve materials.
- For fastener applications, alloy 59 exhibits excellent mechanical properties & toughness, corrosion resistance similar to annealed material without any degradation and, stress corrosion resistance in various environments including sea-water.
- Various tests conducted on fasteners machined from cold reduced bars clearly show that alloy 59 offers an exceptional combination of strength, ductility, toughness, fatigue resistance in sea-water and fatigue crack growth resistance for fastener applications
- Alloy 59 is galvanically compatible with alloy 625 and Ti grade 2 which are extensively used in marine environments.

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