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PROPERTIES OF MODIFIED NITINOL ALLOYS

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Naval Ship Research and Development Center
Annapolis, Maryland

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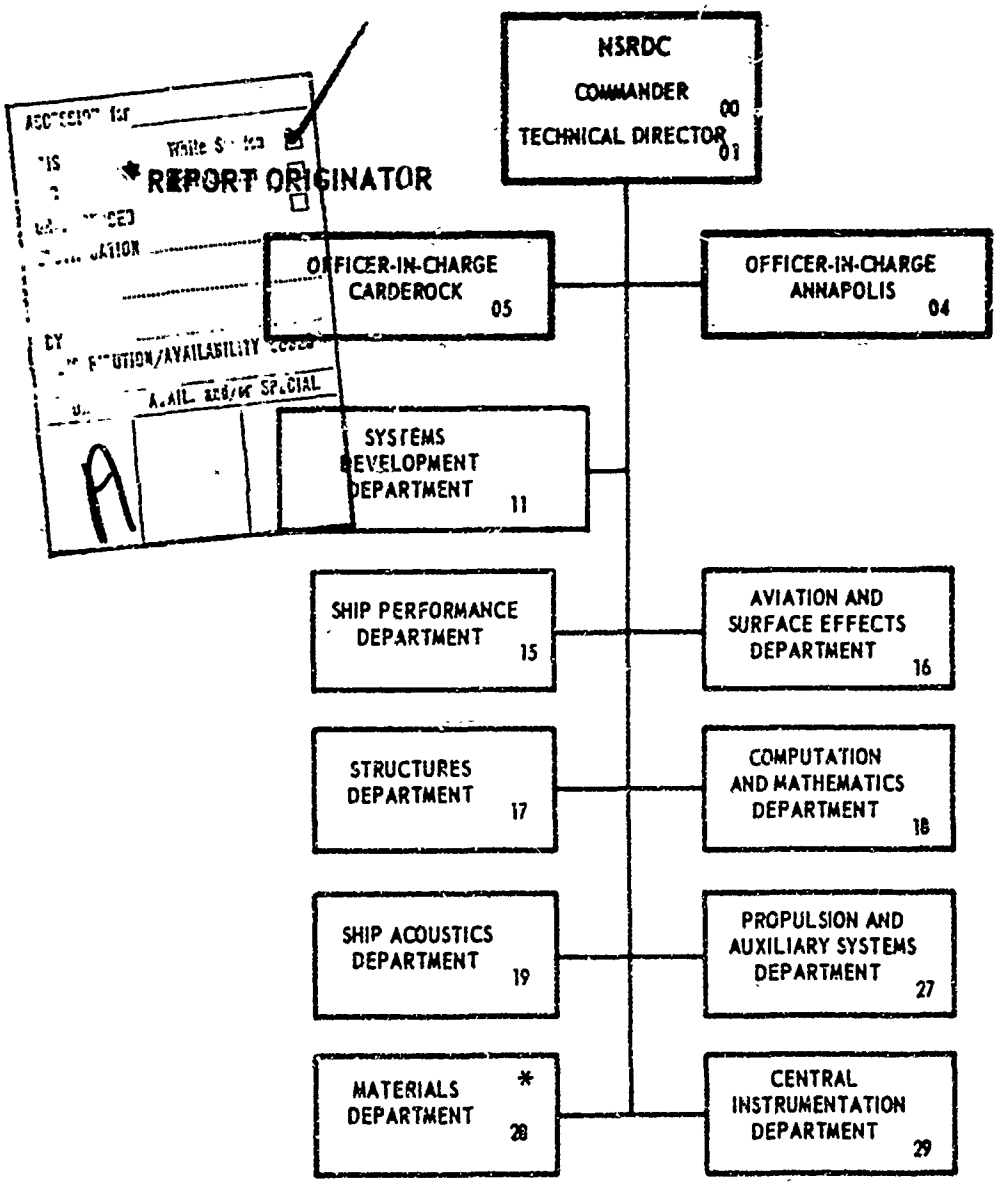

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13. ABSTRACT

Nitinol 55 is an equiatomic nickel-titanium intermetallic compound which has the capability of being restored to original shape after deformation. This alloy is potentially useful in many sea-water system applications. This investigation deals with the production and testing of 11 modified Nitinol alloys. Corrosion properties, mechanical properties, processing parameters, and microstructural characteristics have been determined as the function of alloy type. Results indicate that substitutional additions of Mo, Fe, and Cr are beneficial in preventing localized crevice corrosion. Further studies are being undertaken to determine long-term corrosion behavior of alloys produced as well as to broaden the data base describing the effects of the level of alloy content.

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INTRODUCTION

Nitinol 55 is an equiatomic nickel-titanium compound developed by Buehler and Wiley of the U. S. Naval Ordnance Laboratory.[1] It has generated significant interest due to its mechanical or shape memory effect. This is an ability of the alloy to recover shape after plastic deformation upon being heated through a unique temperature range. The bulk of research with this system has dealt with characterizing the memory effect and developing consistent explanations for that behavior.[2-4] The corrosion behavior of this family of alloys, however, has not been systematically described to date. Preliminary sea-water-corrosion testing of Nitinol alloy has indicated susceptibility to pitting and crevice attack. But this testing has also pointed up the possibility of developing corrosion-free alloys through ternary alloy addition. This program is directed toward characterizing the corrosion behavior and mechanical properties of a broad family of modified Nitinol alloys. The direction is toward the definition of alloys which will be acceptable for sea-water applications.

In order to carry out the description of the behavior of modified Nitinol alloys, a program of alloy production and testing has been undertaken. The scheme includes the determination of mechanical properties, processing characteristics, and corrosion behavior of experimental alloys as a function of alloy type and content. The initial phase, reported herein, consisted of the production of 11 alloys of the form $Ti_{0.5}$-$Ni_{0.47}$-$M_{0.03}$, where M was Co, Fe, Mo, V, Nb, Cr, Ta, Zr, Al, Cu, and Si, respectively. After production, each sample was hot rolled to sheet thickness, and subsequently, mechanical tests and corrosion studies were performed.

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[1] Superscripts refer to similarly numbered entries in the Technical References at the end of the text.

EXPERIMENTAL APPROACH AND RESULTS

ALLOY PRODUCTION

Processing of experimental alloys began with the production of equiatomic Ti-M alloys for substitutional additions. These "stock" buttons were arc-melted and radiographed as a check for homogeneity. Laboratory grade purity metals and commercially pure titanium were used in preparing the buttons. Experimental buttons, each weighing approximately 150 grams, were then arc-melted from an induction-melted master Ti-Ni alloy (D4006) and addition of the "stock" alloy to carry out a 3 a/o substitution for Ni. Table 1 includes the results of chemical analysis carried out after completion of processing. Fe and Nb additions were substantially below the level sought with both alloys exhibiting excess levels of Ti.

TABLE 1 - CHEMICAL ANALYSIS OF NITINOL ALLOYS AFTER PROCESSING (ATOMIC PERCENT)

Alloy	Ni	Ti	C	Other Elements
Ni-Ti(D4006)	49.56	50.43	0.01	
Ni-Ti+Cu	46.66	50.27	-	Cu 3.07
Ni-Ti+Co	47.50	49.68	-	Co 2.91
Ni-Ti+Nb	46.22	52.17	-	Nb 1.60
Ni-Ti+Si	45.7	51.7	-	Si 2.5
Ni-Ti+V	46.45	50.51	-	V 3.03
Ni-Ti+Cr	46.93	50.21	-	Cr 2.85
Ni-Ti+Ta	45.99	51.79	-	Ta 2.21
Ni-Ti+Mo	46.5	50.4	-	Mo 3.0
Ni-Ti+Fe	45.48	53.54	-	Fe 0.97
Ni-Ti+Zr	46.5	50.5	-	Zr 2.9
Ni-Ti+Al	46.5	50.4	-	Al 3.0

Prior to hot rolling, radiographic analysis was performed to detect undissolved additions. Initial melts of Co, Fe, V, Cr, Zr, Al, Cu, and Si showed complete solubility of the substitutional alloy addition. The Nb-modified alloy was homogenized after one remelt. Mo- and Ta-modified alloys contained small, dispersed particles after several remelts but were nevertheless forwarded for processing. Hot rolling was carried out for all alloys including the master Ti-Ni under the following format:

- Grind button surface where required.
- Hot roll button to 0.125-inch thickness at 850° C.*
- Cut small section of tang for metallography.
- Hot roll to 0.080-inch thickness at 800° C.
- Hot roll to 0.050-inch thickness at 750° C.
- Hot roll to 0.030-inch thickness at 675° C.
- Roll last 20% thickness reduction at 600° C.
- Clamp sheets into flattening fixture and anneal for 10 minutes at 500° C; air cool.

Severe cracking occurred during the initial pass with the Mo-, Al-, and Zr-modified alloys. As a result, these specimens were eliminated from further processing, while the remaining alloys were processed according to the scheme described above. Co-, Cr-, and Cu-modified alloys showed very slight surface checking on one side as the result of the hot-rolling process. Si- and Ta-modified alloys displayed general surface cracking on both sides as well as edge cracking. The Fe-modified specimen underwent general surface cracking on a single side. The Nb modification resulted in surface cracking on both sides, and lastly V-modified material displayed large surface cracks on both sides with some through-cracking. All surface cracks were ground locally during processing. All melting and processing was performed at the Naval Ordnance Laboratory (NOL) White Oak, Maryland.

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*Abbreviations used in this text are from the GPO Style Manual, 1967, unless otherwise noted.

METALLOGRAPHY

Longitudinal and transverse orientation specimens were obtained during hot rolling. Standard metallographic polishing techniques were employed followed by etching in a solution of 82% H_2O, 14% HNO_3, and 4% HF for periods of 2 to 10 seconds. The Ti-Ni alloy (D4006) shows a homogeneous Ti-Ni matrix with dispersed stringers of Ti_4Ni_2O,[5] figure 1. This microstructure is typical of that seen with induction-melted alloys processed into sheet. Alloys modified with Co and Fe and Mo displayed lamellar dispersions of Ti_4Ni_2O characteristic of the arc-melted alloy, figure 2. Alloys with Cu, Cr, and Si additions displayed highly homogeneous dispersions of a reduced amount of oxide, figure 3. Each of these alloys also showed evidence of formations of limited secondary phases. Pits observable with Cr-modified and other alloys are etch pits resulting from oxide and solute segregation. Ta-, V-, Nb-, Al-, and Zr-modified alloys showed high degrees of secondary phase formation, indicating limits of each elemental solubility in the Ti-Ni system, figure 4.

MECHANICAL PROPERTIES

Room temperature microhardness tests were carried out with a Tukon hardness tester. Average Diamond Pyramid Hardness (DPHN) values for both longitudinal and transverse orientations are reported in table 2.

TABLE 2 - ROOM TEMPERATURE DIAMOND PYRAMID HARDNESS VALUES OF MODIFIED NITINOL ALLOYS

Alloy	DPHN (Transverse)	DPHN (Longitudinal)
Ni-Ti(D4006)	168	180
Ni-Ti+Cu	143	154
Ni-Ti+Co	168	167
Ni-Ti+Nb	191	213
Ni-Ti+Si	216	223
Ni-Ti+V	228	218
Ni-Ti+Cr	235	229
Ni-Ti+Ta	240	240
Ni-Ti+Mo	283	293
Ni-Ti+Fe	285	295
Ni-Ti+Zr	-	281
Ni-Ti+Al	-	253

As has been stated, Nitinol alloys possess the property of shape recoverability after plastic deformation by heating through a temperature span known as the transition temperature range (TTR). This range has been shown to be a function of alloy content as well as type and degree of mechanical work introduced. Seven of the nine processed alloys were tested in order to determine the TTR of each alloy. The test method for this determination involved bending narrow sheet specimens 180° over a mandrel of such size as to induce approximately 6% plastic deformation in the outer fibers. Deformation took place in an isothermal bath whose temperature was well below estimated values for onset of recovery. The bath temperature was then raised and the angle of recovery determined as a function of bath temperature. Allowing Ø to be the angle of recovery, percent recovery (Ø/180°) versus temperature is plotted for various alloys in figures 5 and 6. TTR figures are reported in table 3. The values reported for Fe- and Cr-modified alloys were estimated from noncontrol bend experiments in liquid N_2 baths.

TABLE 3 - EXPERIMENTALLY DETERMINED TRANSITION TEMPERATURE RANGE VALUES OF MODIFIED NITINOL ALLOYS

Alloy	TTR °C	
Ni-Ti(D4006)	+60	+85
Ni-Ti+Cu	+50	+80
Ni-Ti+Co	0	+65
Ni-Ti+Nb	+65	+95
Ni-Ti+Si	+70	+95
Ni-Ti+V	-10	+30
Ni-Ti+Ta	+75	+100
Ni-Ti+Cr*	- 196 < TTR < -80	
Ni-Ti+Fe*	< - 196	

*Approximate determinations.

ACCELERATED CORROSION

Potentiostatic accelerated crevice corrosion tests were carried out according to a method recently developed at this laboratory. Figure 7 is a schematic of the cell describing the O-ring setup employed to limit area of corrosion attack as well as create an artificial crevice. The tests were run according to the following technique:

- After each specimen was ground to 600-grit finish, it was placed in the cell and open-circuit potential was recorded.

- The potential was varied in a step scan mode, beginning at a potential 5 millivolts (mv) more negative than open-circuit potential. The scan was carried out in +50-mv increments until the +200-mv level was reached or current growth began. Hold time for each step was 2.5 minutes. For each experimental run, terminal potential, or the potential at which current growth occurred, was maintained for 2 hours. Applied potential and specimen current were continuously recorded.

- If no appreciable current increase was noted at the end of 2 hours at +200-mv applied potential, the test was repeated as described to reach potential levels of +400, +600, +800, and +1000 mv or until current growth occurred.

- Two series of tests were run and are reported. The first tests were carried out under conditions wherein the specimen was electrically grounded, and the auxiliary and reference electrodes kept at same potential. This led to specimen current measurement which is only valid from a comparative standpoint. The second series was run under conditions wherein only the specimen was grounded, thereby yielding intrisically valid current level results.

Interpretation of these accelerated corrosion test data is complex. The variables include potential at which current growth (corrosion) occurred, current growth rate, maximum current, specimen appearance, and rate and time considerations during potential scan. For purposes of this analysis, potential at which current growth occurred, time at potential prior to current increase, current after 20 minutes of current growth,

and specimen appearance have been reported for both the grounded and ungrounded tests. These data are presented in tables 4 and 5. Nine of the original modified alloys have been tested as well as Ti-Ni (D4006), Ti-Ni (D4031) which is another heat of induction-melted Nitinol, and a Co-modified alloy ($Ni_{0.5}$-$Ti_{0.43}$-$Co_{0.07}$) obtained from the Naval Ordnance Laboratory.

NATURAL SEA-WATER TESTS

Sea-water trough corrosion tests were performed upon fully processed sheet specimens employing a multiple crevice configuration test. A 1/2-inch hole was machined in the center of each 2- x 5-inch specimen, and a multiple crevice device shown in figure 8 was attached to the specimen. The free surface to crevice area ratio was 20:1 for these 35-day tests which were made in the filtered sea-water trough at the Francis L. LaQue Corrosion Laboratory.

TABLE 4 -- POTENTIOSTATIC CELL CREVICE CORROSION TEST RESULTS; AUXILIARY AND REFERENCES ELECTRODE GROUNDED

Alloy	Open-Circuit Potential mv, Versus Ag-AgCl	Potential at which Current Growth Occurred mv	Time at Potential Prior to Current Growth min	Specimen Current 20 Minutes after Start at Current Growth ma	Specimen Appearance
Ni-Ti(D4006)	-285	+400	0	0.79	Moderate to heavy pitting; extensive general attack
Ni-Ti+Cu	-350	+400	1	0.23	Moderate to heavy pitting; extensive general attack
Ni-Ti+Co	-335	+400	11	0.18	Light pitting; mild general attack
Ni-Ti+Nb	-475	+400	3	0.44	Light pitting; moderate general attack
Ni-Ti+Si	-365	+400	11	0.23	Light pitting; moderate general attack
Ni-Ti+V	-345	+400	1	0.58	Moderate to heavy pitting; extensive general attack
Ni-Ti+Cr	-397	+600	52	0.37	Limited light pitting; moderate general attack
Ni-Ti+Ta	-316	+600	13	0.61	Limited light pitting; moderate general attack
Ni-Ti+Mo	-350	+1000	∞	0.0	No pitting or general attack
Ni-Ti+Fe	-537	+400	28	0.07	No pitting; mild general attack
Ni-Ti(D4031)	-347	+400	10	0.96	Moderate to heavy pitting; extensive general attack
$Ti_{0.5}$-$Ni_{0.42}$-$Co_{0.08}$	-372	+500	3	0.27	Light pitting; light general attack

TABLE 5 - POTENTIOSTATIC CELL CREVICE CORROSION TEST RESULTS; SPECIMEN ONLY GROUNDED

Alloy	Open-Circuit Potential mv, Versus Ag-AgCl	Potential at which Current Growth Occurred mv	Time at Potential Prior to Current Growth min	Specimen Current 20 Minutes after Start of Current Growth ma	Specimen Appearance
Ni-Ti(D4006)	-295	+350	2.5	14.7	Heavy pitting; moderate to extensive general attack
Ni-Ti+Cu	-200	+300	0	1.65	Light pitting; moderate general attack
Ni-Ti+Co	-262	+350	0.5	2.45	Moderate pitting; moderate to heavy general attack
Ni-Ti+Nb	-310	+300	1.5	17.1	Moderate to heavy pitting; no general attack
Ni-Ti+Si	-225	+250	2.5	1.60	Light pitting; light general attack
Ni-Ti+V	-215	+650	1.5	41.5	Heavy pitting; heavy general attack
Ni-Ti+Cr*	-298	+400	2.0	192	Heavy pitting; light general attack
	-365	+550	0	1.93	Light pitting; light general attack
Ni-Ti+Ta*	-300	+300	0.5	22.5	Moderate to heavy pitting; no general attack
	-358	+400	1.5	28.5	Heavy pitting; no general attack
Ni-Ti+Mo	-275	+1000	∞	0	No pitting; no general attack
Ni-Ti+Fe	-337	+400	0	1.2	Light pitting; moderate general attack
Ni-Ti(D4031)	-377	+350	3.5	6.75	Moderate pitting; moderate general attack
$Ti_{0.5}$-$Ni_{0.42}$-$Co_{0.08}$	-383	+400	0	1.7	Light pitting; moderate general attack

*Repeated tests.

At the completion of the test period, specimens were cleaned, and type and severity of attack were characterized. There were distinct differences in the corrosion behavior of the alloys tested. The Fe- and Cr-modified alloys shown in figure 9 are seen to suffer very limited general and localized crevice attack. Ti-Ni (D4006), V-, and Cu-modified alloys are shown to suffer moderate to severe pitting (figure 10). Lastly, the alloys modified with Co, Nb, Si, and Ta, respectively, suffered various degrees of crevice attack as shown in figures 11 and 12.

DISCUSSION

It has been previously stated that this program is a parametric evaluation of Nitinol alloys as a function of alloy content and type. Several factors have emerged from this initial phase. Principally, the degree to which the TTR of the alloys is affected by substitutional additions of various elements has been detailed. Previous work by Goldstein, et al,[6] has examined the effects of additive alloy content of many of the alloys examined in this study upon the hardness of the wrought alloy. These data indicate that substitutional additions produce correspondingly lower or at least equal hardness values compared to the nonsubstitutional type. This points to the degree to which lattice strain in dependent upon type of addition as well as, in the case of Cu, Co, Nb, Si, and V additions, the ability of the room temperature Ti-Ni structure to accommodate appreciably increased levels of alloy content.

The data accumulated in determining the TTR for each alloy have shown a wide variation of alloy contribution with respect to that parameter. Fe and Cr additions appeared to reduce the TTR drastically compared to that observed with unalloyed Nitinol. Co and V additions were shown to depress the TTR moderately. The remaining alloys, which were processed, displayed little or no effect upon the TTR. It should be noted that with the exception of the Co-modified alloy, recovery from 6% plastic strain was nearly complete.

The results of the potentiostatic accelerated corrosion testing are the most difficult to interpret. The tendency toward drawing narrow conclusions concerning the predicted long-term crevice corrosion behavior must be resisted. Furthermore, it must be stated that the reproducibility of these results has not been adequately examined. However, analysis of both sets of data indicates that Mo, V, Ta, Cr, Fe, and possibly Co additions are beneficial in preventing this type of localized attack. This statement is made with respect to heavily weighting the potential at which current growth occurred and in analyzing all of the data. Past experience with this test indicates that its greatest accuracy is seen in determining "immune" alloys as described by a lack of current growth at the +1000-mv applied potential. The Mo-modified alloy was the only one of those tested to meet this criterion. Ta-, Cr-, Fe-, and V-modified alloys underwent light to moderate attack at

substantially lower potentials. These alloys are considered as possibly resistant to attack because of the fact that they suffered corrosion to a lesser degree and at somewhat higher potential than the remaining alloys. The only case where correlation exists with long-term data is that for the Ti-Ni alloy (D4031) and Co-modified alloy produced by NOL. Specimens of both of these alloys were placed in sea-water crevice corrosion test for a period of 1 year at the Francis L. LaQue Corrosion Laboratory, Wrightsville Beach, North Carolina. Figure 13 shows the specimens after test. It is seen that unmodified Nitinol suffered pitting and crevice attack. The Co-modified alloy was nearly unattacked. This long-term test indicates that alloys which resisted attack with applied potentials up to +400 to +500 mv in the potentiostatic cell tests are possibly resistant to long-term crevice attack.

The corrosion behavior observed in the 35-day sea-water trough exposures showed more resolution than that seen in the potentiostatic cell test. The fact that Fe- and Cr-modified alloys displayed resistance to pitting, crevice attack, and general attack in this test points to the likelihood of long-term corrosion resistance. However, the existence of inhomogeneities in the rolled product, short test duration, and lack of test repetition place the results of this test more in the range of speculation than strict characterization.

Straightforward correlation of the properties studied with microstructure resulting from alloy additions does not exist according to the data reported herein. From a corrosion standpoint the pitting and crevice attack occurred with alloys showing both secondary phase formation and homogeneous structure with various distributions of Ti_4Ni_2O. The TTR determinations indicate sensitivity to type and level of substitution, regardless of classification of microstructure. This points up the hypothesis that TTR variation occurs according to the number of available electron bonds rather than bulk mechanical consideration. This is most obvious with the V-modified alloy which shows significant secondary phase formation, high hardness, but also possesses an appreciably reduced TTR as compared to Ti-Ni.

Future research in this program will entail broadening the data base reported herein. The level of addition of alloying element will be varied to determine the effect upon the corrosion behavior and mechanical properties. Possible quaternary combinations will be considered. However, particular attention will be paid to verifying the trends indicated by these data as well as clarification of corrosion behavior.

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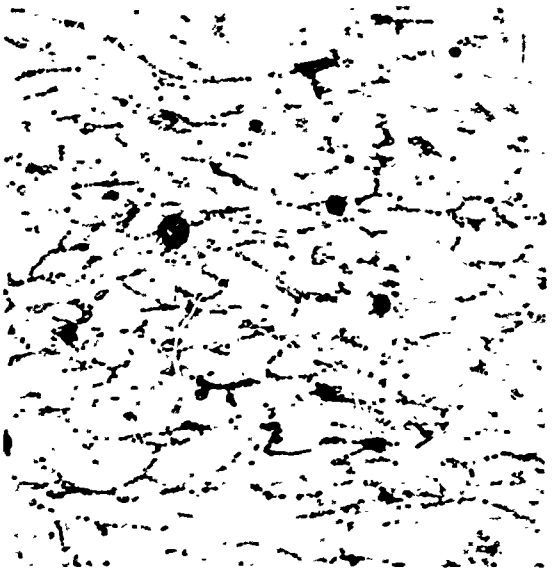
Item (a)
Longitudinal

Item (b)
Transverse

Figure 1 - Microstructures of Ti-Ni
Master Alloy (D4006)
(250X)

Item (a)
Co-Modified



Item (b)
Fe-Modified

Item (c)
Mo-Modified

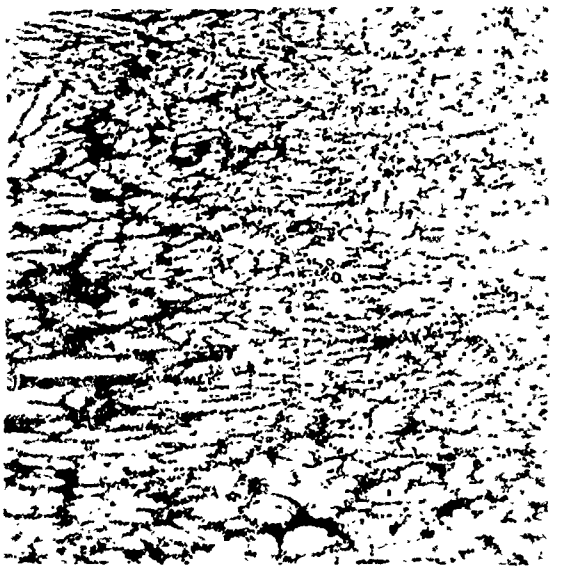
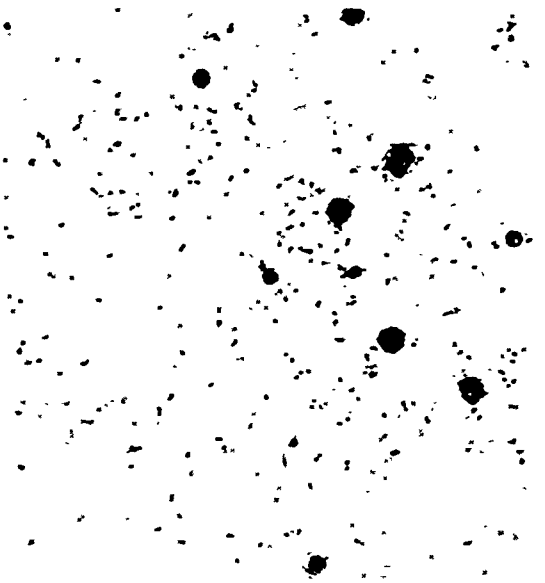


Figure 2 - Microstructures of Co-, Fe-, and Mo-Modified Nitinol Alloys (Transverse) (250X)

Item (a)
Cu-Modified

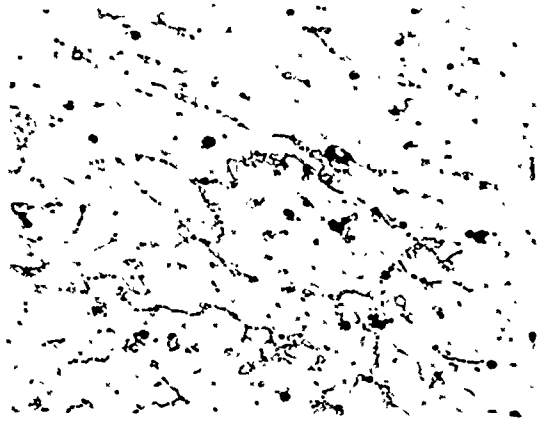
Item (b)
Cr-Modified



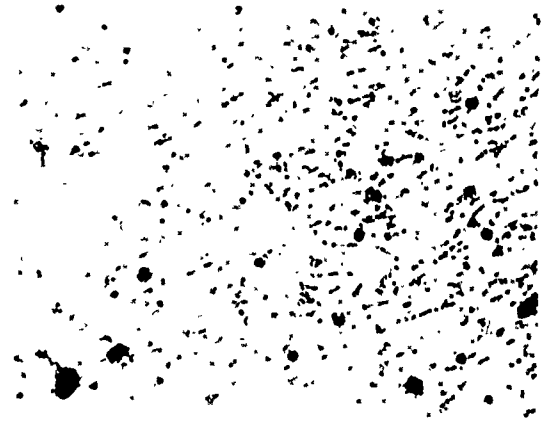
Item (c)
Si-Modified

Figure 3 - Microstructures of Cu-, Cr-, and Si-Modified Nitinol Alloys (Transverse) (250X)

Item (a) - Ta-Modified



Item (b) - V-Modified



Item (c) - Nb-Modified

Item (d) - Al-Modified

Item (e) - Zr-Modified

Figure 4 - Microstructures of Ta-, V-, Nb-, Al-, and Zr-Modified Nitinol Alloys (Transverse) (250X)

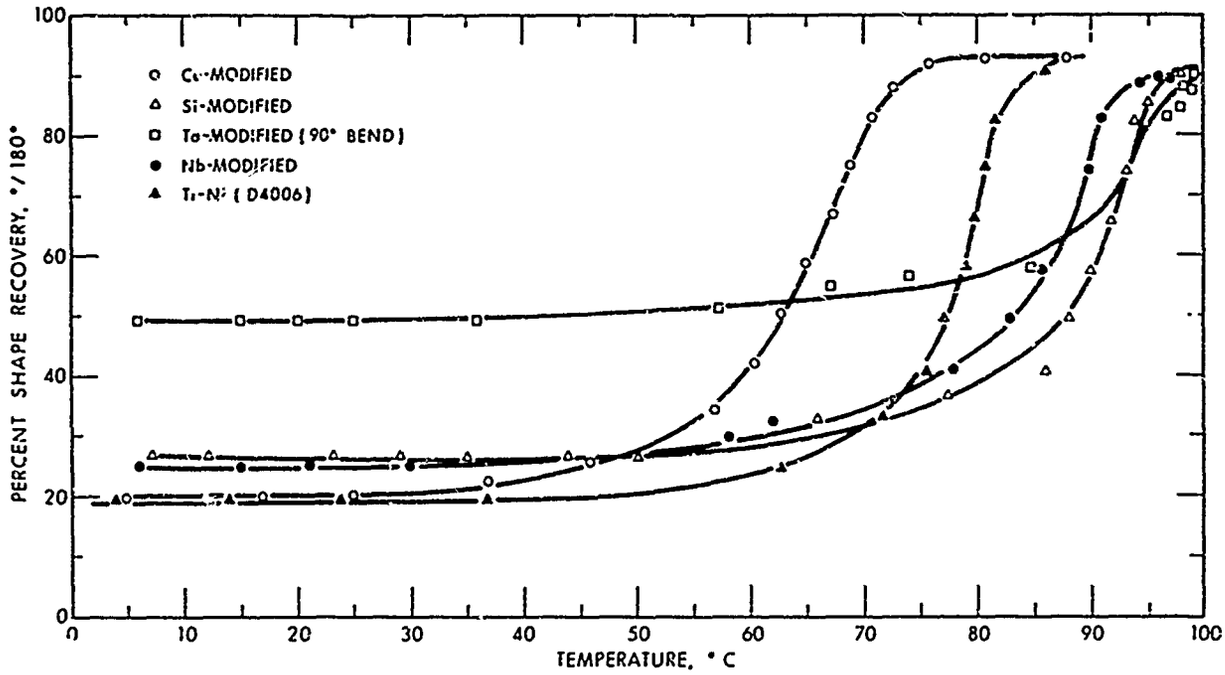


Figure 5 - Percent Shape Recovery Versus Temperature for Cu-, Si-, Ta-, Nb-Modified Alloys and Ti-Ni(D4006)

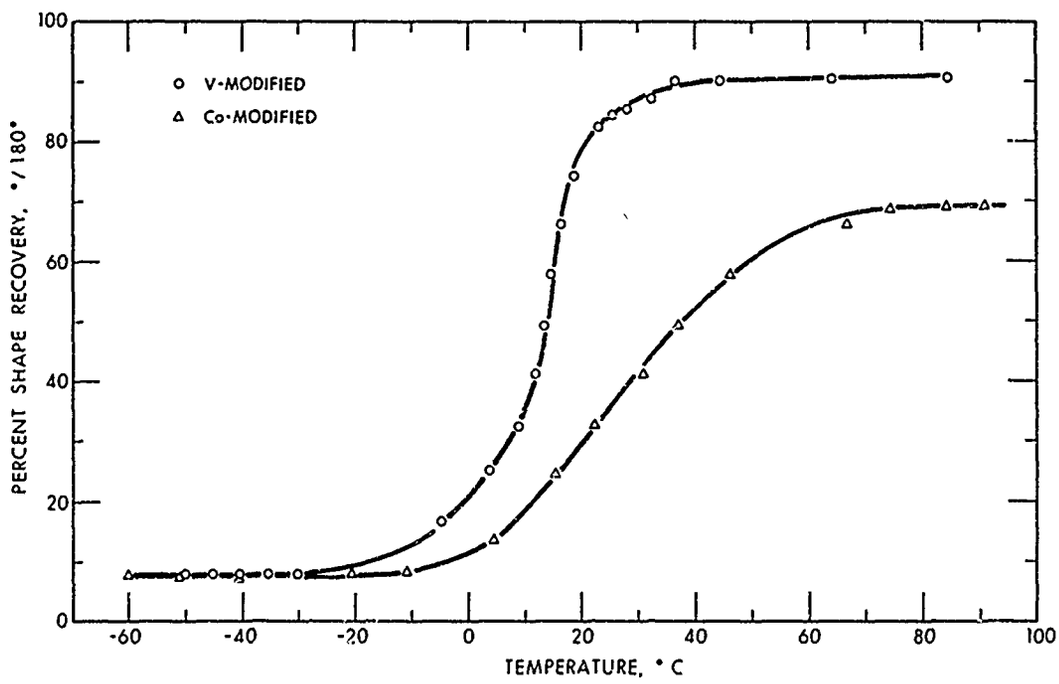


Figure 6 - Percent Shape Recovery Versus Temperature for V- and Co-Modified Alloys

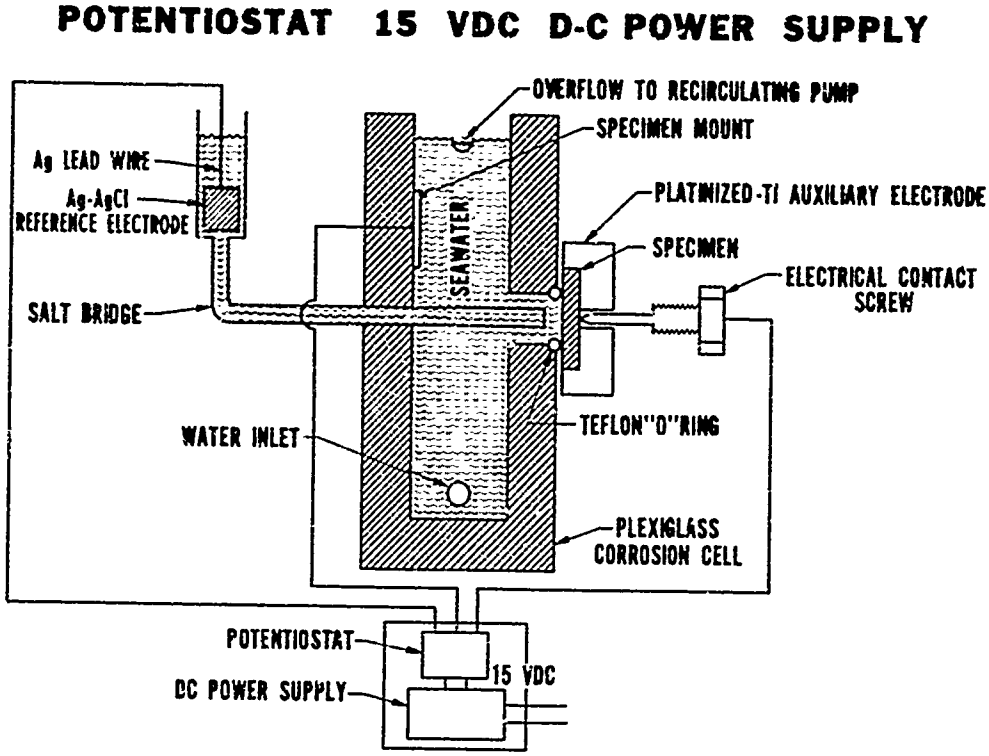
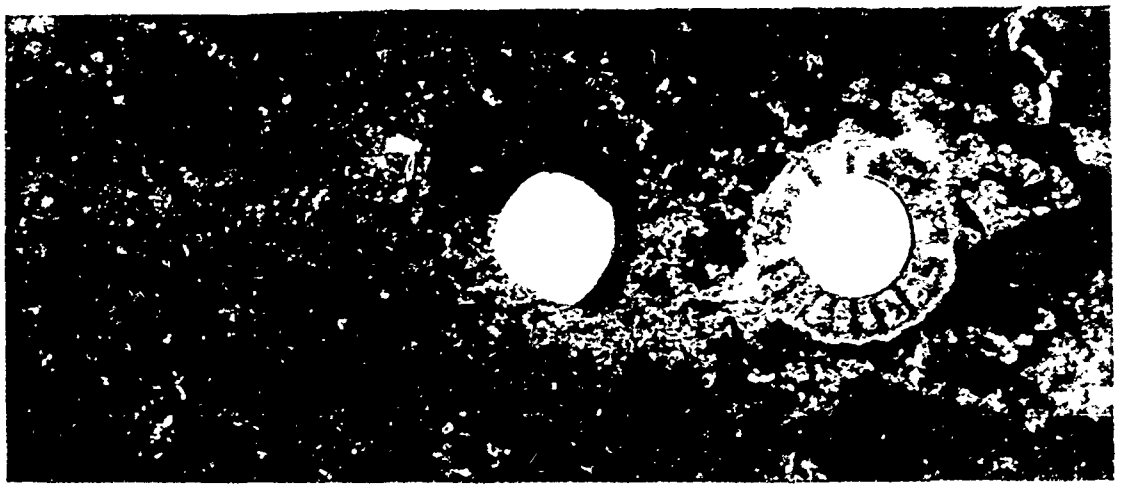


Figure 7
Accelerated Crevice Corrosion Test Setup

Figure 8 - Multiple Crevice Device Employed in Sea-water Trough Corrosion Tests

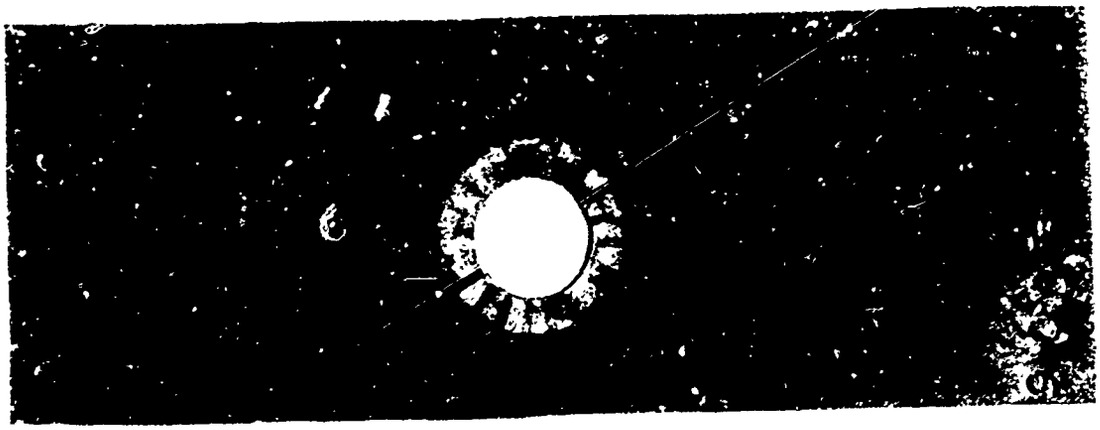
Item (a)
Fe-Modified



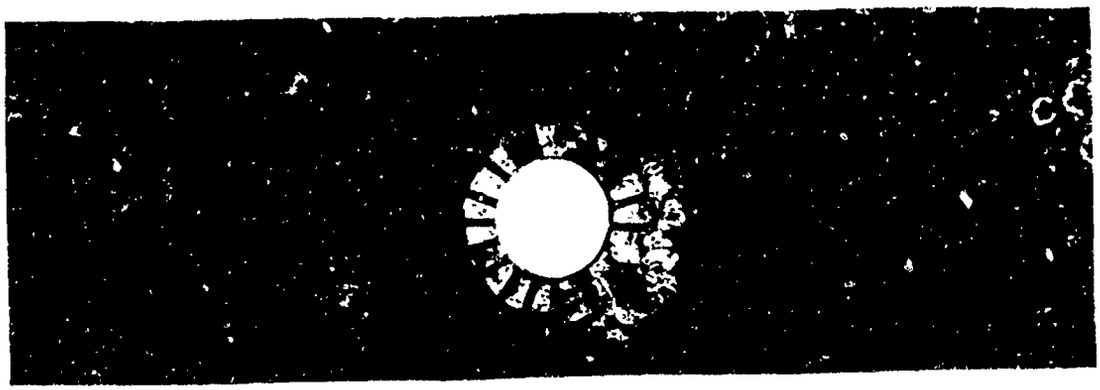
Item (b)
Cr-Modified

Figure 9
Fe- and Cr-Modified Specimens after
Sea-water Corrosion Tests

Ite · (a) - Ti-Ni(D4006)



Item (b) - V-Modified



Item (c) - Cu-Modified

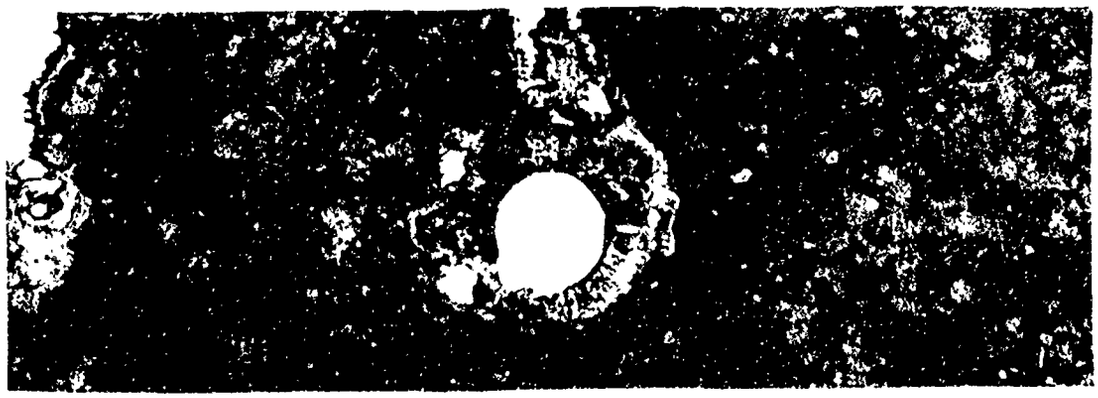
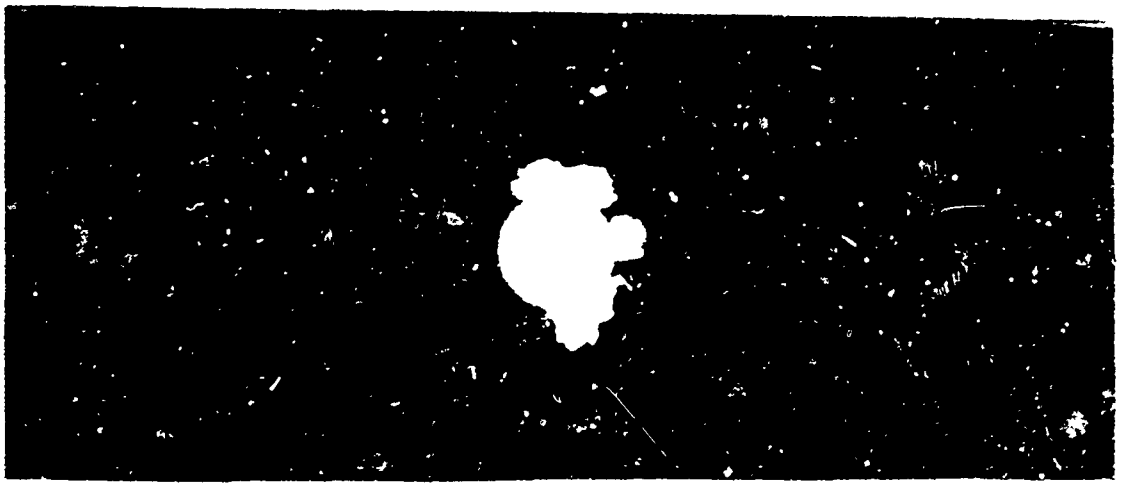


Figure 10 - Ti-Ni(D4006)-, V-, and Cu-Modified Alloys after Sea-water Corrosion Tests

Item (a)
Nb-Modified



Item (b)
Si-Modified

Figure 11 - Nb- and Si-Modified Alloys after Sea-water Corrosion Tests

Item (a)
Ta-Modified

Item (b)
Co-Modified

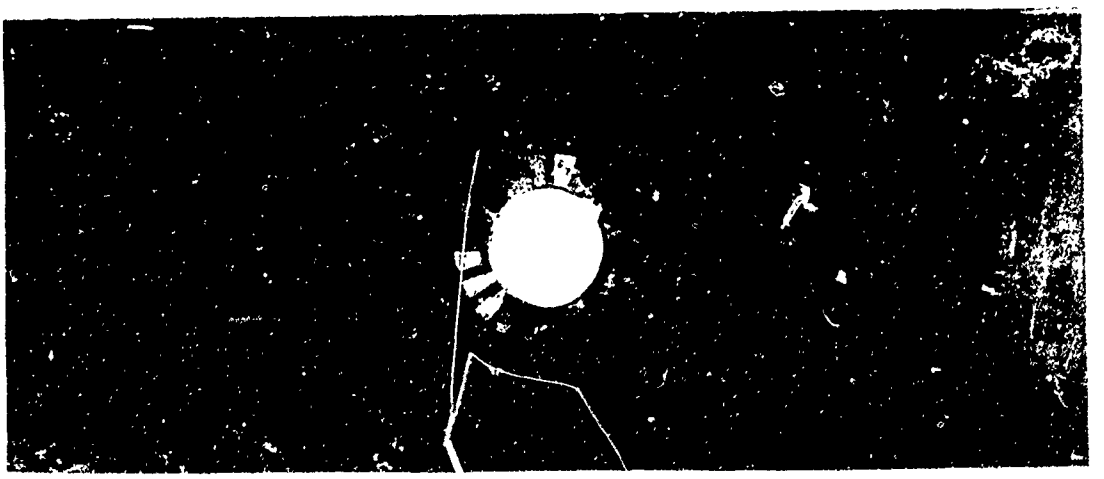


Figure 12 - Ta- and Co-Modified Alloys after Sea-water Corrosion Tests

(1) Ti-Ni(D4031)
(2) $Ti_{0.5}$-$Ni_{0.42}$-$Co_{0.08}$

(1) (2) (1) (2)

Figure 13 - Ti-Ni(D4031) and $Ti_{0.5}$-$Ni_{0.42}$-$Co_{0.08}$
Crevice Corrosion Specimens after
1-Year Sea-water Exposure