

1-4-2005

Sub-surface corrosion research on rock bolt system, perforated SS sheets and steel sets for the Yucca Mountain Repository – Quarterly technical report No. 2

Dhanesh Chandra
University of Nevada, Reno, dchandra@scs.unr.edu

Follow this and additional works at: https://digitalscholarship.unlv.edu/yucca_mtn_pubs



Part of the [Geotechnical Engineering Commons](#), [Metallurgy Commons](#), and the [Structural Materials Commons](#)

Repository Citation

Chandra, D. (2005). Sub-surface corrosion research on rock bolt system, perforated SS sheets and steel sets for the Yucca Mountain Repository – Quarterly technical report No. 2.

Available at: https://digitalscholarship.unlv.edu/yucca_mtn_pubs/24

This Technical Report is protected by copyright and/or related rights. It has been brought to you by Digital Scholarship@UNLV with permission from the rights-holder(s). You are free to use this Technical Report in any way that is permitted by the copyright and related rights legislation that applies to your use. For other uses you need to obtain permission from the rights-holder(s) directly, unless additional rights are indicated by a Creative Commons license in the record and/or on the work itself.

This Technical Report has been accepted for inclusion in Publications (YM) by an authorized administrator of Digital Scholarship@UNLV. For more information, please contact digitalscholarship@unlv.edu.



“Sub-surface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets for the Yucca Mountain Repository”

Quarterly Technical Report No. 2 (4th. Quarter for FY 2004)

Start Date of this Quarter, October 1, 2004 to December 31, 2004

Task No: ORD-FY04-019 and Document No: SIP-UNR-040

Prepared for U.S. DOE/UCCSN Cooperative Agreement Number DE-FC28-04RW12232

-submitted to-

*United States Department of Energy
Office of Civilian Defense Radioactive Waste Management
Yucca Mountain Project
1551 Hillshire Drive, Suite A
Las Vegas NV 89134-6321*

and

*Harry Reid Center
University of Nevada, Las Vegas
4505 Maryland Parkway
Las Vegas Nevada 89154-4009*

-submitted by-

*Metallurgical and Materials Engineering
Mail Stop 388
1664 N. Virginia Street
College of Engineering
University of Nevada, Reno
Reno NV 89557*

Dhanesh Chandra, PI (Corrosion Team Leader)

January 4, 2005

Task ORD-FY04-019 “Sub-surface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets for the Yucca Mountain Repository”

(A Quality-Affecting Task)

**Principal Investigator, Dhanesh Chandra
DOE Technical Representative, Jaime Gonzalez**

Quarterly Technical Progress Report

10/01/04 - 12/31/04

Statement of Work

The objective of this task is to conduct corrosion related research and predict the durability of rock-bolts and other underground metallic roof supports. Corrosion resistance of rock bolts (such as Swellex, CT, and hollow core anchor) made of high-strength low alloy steel, medium carbon steel (from the YM site), stainless steel, and other higher strength materials will be evaluated. In this quarter our SIP-UNR-040 Rev. 0 has been approved effective 12/17/04.

General Statements

- We have started work on “Baseline Materials” as suggested by the DOE meeting.
- Quality Assurance Training is in progress for all the staff working on the Yucca Mountain project
- Obtained some of the available specimens for testing
- A Ph.D student (Mr. Josh Lamb) and new Ph.D graduate students have been trained and started the QA work on the project.

Progress for the Period 10/01/04-12/31/04:

The SIP has been recently approved 12/17/04. We have started preliminary research on the following tasks:

Subtask 1: Selection of New High Strength Steels, Stainless Steels for Rock Bolts, Steel Sets and Perforated Roof Supports

Subtask 2: Electrochemical Corrosion Tests

Subtask 4: Environmentally Assisted Corrosion Tests

Subtask 7: Dry Oxidation Tests ASTM G-54

Subtask 8: Microstructure and Phase Characterization Studies

In addition we are working on a manuscript on stress corrosion cracking of steels for underground support. A manuscript “Paper No. MS30802E” has been sent to *Corrosion Journal* (NACE) and it is accepted is in print. We have obtained some of the calibrated instrumentation from Bechtel, Nevada, and started the electro-mechanical tests

1. Introduction

This report encompasses the work done for second quarter in accordance to cooperative agreement of UCCSN for the Task 019 “Subsurface Corrosion Research on Rock Bolt System, Perforated SS Sheets and Steel Sets for the Yucca Mountain Repository”, the objective of which was proposed earlier, to conduct corrosion research and predict the durability of rock-bolts and other underground metallic roof supports. We have recently started working on oxidation tests using Thermogravimetric

Analyzer, stress corrosion cracking/hydrogen embrittlement studies on rock bolts, other support materials including bench mark materials.

In this quarterly report we will discuss “Dry” oxidation of steels and other supporting materials that are susceptible to oxidation under the repository conditions. We initiated these studies to observe the rate of change in *sample mass* as a function of temperature and time using a Thermogravimetric analysis (TGA). In addition we started sample preparation of the Swellex rock bolts, and performed oxidation studies and electromechanical tests on baseline materials, namely, alloy C-22, and YM rock bolts.

2. Experimental

2.A. Thermal Analyses

A TA Instruments, Model Q500, Thermogravimetric analyzer has been used for oxidation research. This instrument can be operated in two modes for investigating thermal stability behavior in controlled atmosphere: (1) dynamic, in which the temperature is increased at a linear rate, and (2) isothermal, in which the temperature is kept constant. This instrument has a continuous weighing capacity of 1.0 g and a sensitivity of 0.1 μg , and a heating rate of 0.1 to 50 C/min. The Implementation Procedure (IP) for using the instrument has been written and is enclosed as an Appendix to the Technical Report. We have also procured and used Class 1 standard weight set from TGA accessory kit for calibration purposes.

TGA instruments operate on a null balance principle. Physically attached to a taut-band meter movement, the balance arm is maintained in a horizontal reference position by an optically actuated servo loop. When the balance is in a null position, a flag located on top of the balance arm blocks an equal amount of light to each of the photodiodes (the light is supplied by a constant current infrared LED). As sample weight is lost or gained, the beam becomes unbalanced, causing an unequal amount of light to strike the photodiodes. The unbalance signal, called the error signal, is acted upon the control circuitry and reduced to zero, or nulled. This is accomplished by an increase or decrease in the current to the meter movement, causing it to rotate back to its original position (null position). The change in current necessary to accomplish this task is directly proportional to the change in mass of the sample. This current is converted to the weight signal.

All experimental procedures are followed in accordance to the IP. Each set of operating conditions are complemented by weight and temperature calibration procedures. Sample temperature calibration is performed by using different materials show weight change at their Curie temperatures. The standards are placed in alumina pan with a strong magnet placed outside the TGA environmental furnace. Adjustments are made in case there is a difference. A multiple-point calibration is more accurate than a one-point calibration and is generally used. A two- or more-point calibration shifts the temperature by a constant amount below the first point, uses a smooth curve through the calibration points and maintains a constant temperature shift after the last point. The rate of heating is 3°C per minute in each case. The ‘observed’ curie transition temperatures in each case have been shown on the plots.

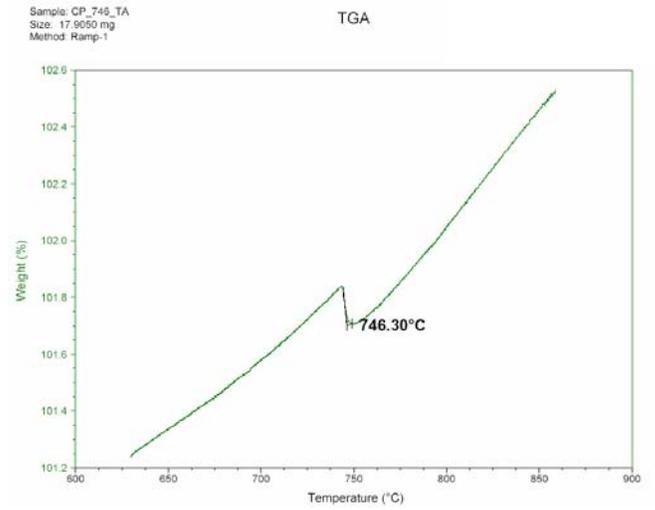
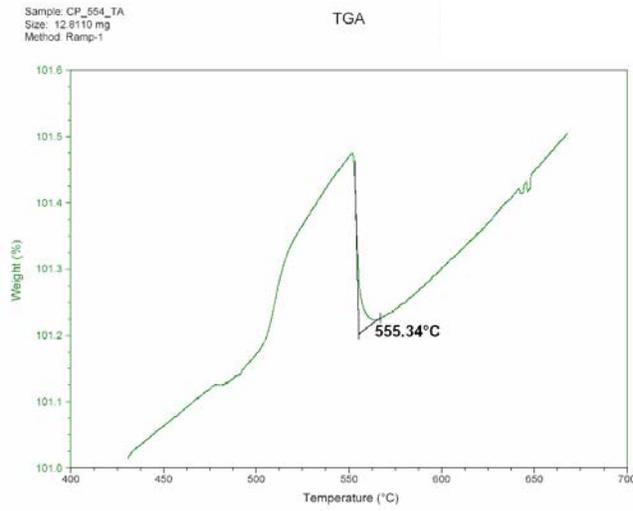
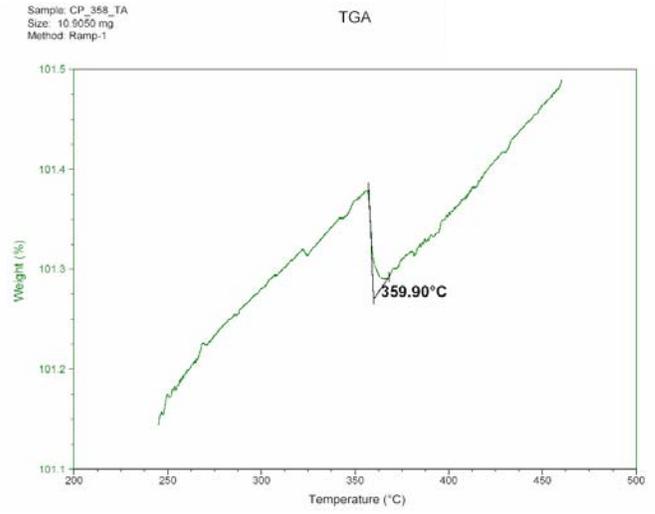
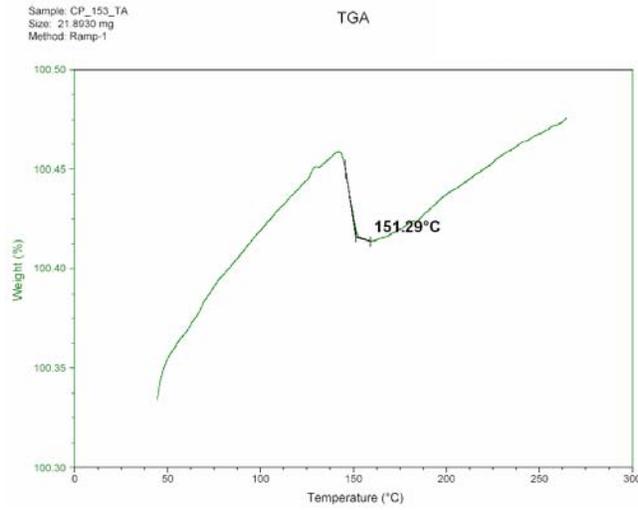


Figure 1. Curie point transitions for four standard metals used for temperature calibration of Thermogravimetric analyzer(TGA). The results match the standards.

2B. Environmentally Assisted Corrosion Tests - Stress-Corrosion Cracking and Hydrogen Embrittlement Tests

A screw-driven SSRT universal machine (United STM-10ES) along its software and a Gamry Potentiostat (DC-105) were used for environmental cracking susceptibility determinations for I-beams. A schematic of the experimental set up is given in Figures 2. All SSRT were carried out with 100X simulated YM water at 85 °C. The simulated waters were conditioned (de-aerated) by purging nitrogen gas for at least one hour before specimen immersion. The specimens were prepared as per Yucca mountain specifications. We first studied the most stable materials such as Alloy 22.

Then, freshly prepared wet-ground specimens with a 600-grit emery paper finish were immediately installed into the cell through the top and bottom central necks using o-ring compression fittings. Then the conditioned de-aerated 100X YM water transferred to the test cell within 10 to 15 seconds without exposing it to air for too long. The cell openings are then closed quickly by threaded o-ring fittings and neck joints, which hold the entire elements of the cell sealed. The installation of the cell on to the universal joints of the SSRT machine should not exceed a total of 4-5 minutes after finishing the specimen preparation. By this rapid installation, possible de-aeration loss of the conditioned test solution, subject to effect the open circuit potential of the LCS by letting excessive oxygen in to the cell, should be prevented. After setting the temperature controller to the desired test temperature, open circuit potential monitoring with the Gamry Potentiostat is started. From previous experience, the open circuit potential (E_{corr}) will stabilize around $-700 \text{ mV}_{Ag/AgCl}$ while the temperature will have already reached to the desired values to an accuracy of $\pm 0.1 \text{ }^\circ\text{C}$. De-aeration by purging nitrogen gas into the test solutions was continuous throughout the tests at a flow rate of 150 ml/min. The conditioned specimens in the test solutions were charged for approximately 45 minutes by imposing desired potentials through Potentiostatic method, until the current values were stabilized prior to monitoring stress-strain curves and currents. The SSRT is started with appropriate strain rates from $10^{-3}/\text{s}$ to $10^{-7}/\text{s}$. During the tests, the changes over the test solutions and specimens were examined, and recorded. After the failure of the specimens, the cell was uninstalled and the pH changes of the test solutions were recorded.

3. Results and Discussion

Dry Oxidation Tests: Thermogravimetric tests were performed on Rock bolt and Alloy 22 samples using a heating rate of 1K/min. The time and temperature of beginning and end of the weight gain were continuously recorded. The percentage weight gain and absolute weight gain are also shown on the plot. The composition of the rock is already reported in previous reports, and the composition of the Alloy 22 is listed in Table 1.

Table 1. The composition of the alloy C-22*.

Elements	Ni	Co	Cr	Mo	W	Fe	Si	Mn	C	P	S	V
%Composition	56	2.5	22	13	3	3	0.08	0.5	0.01	0.02	0.02	0.35

*[Electronic Space Products International]

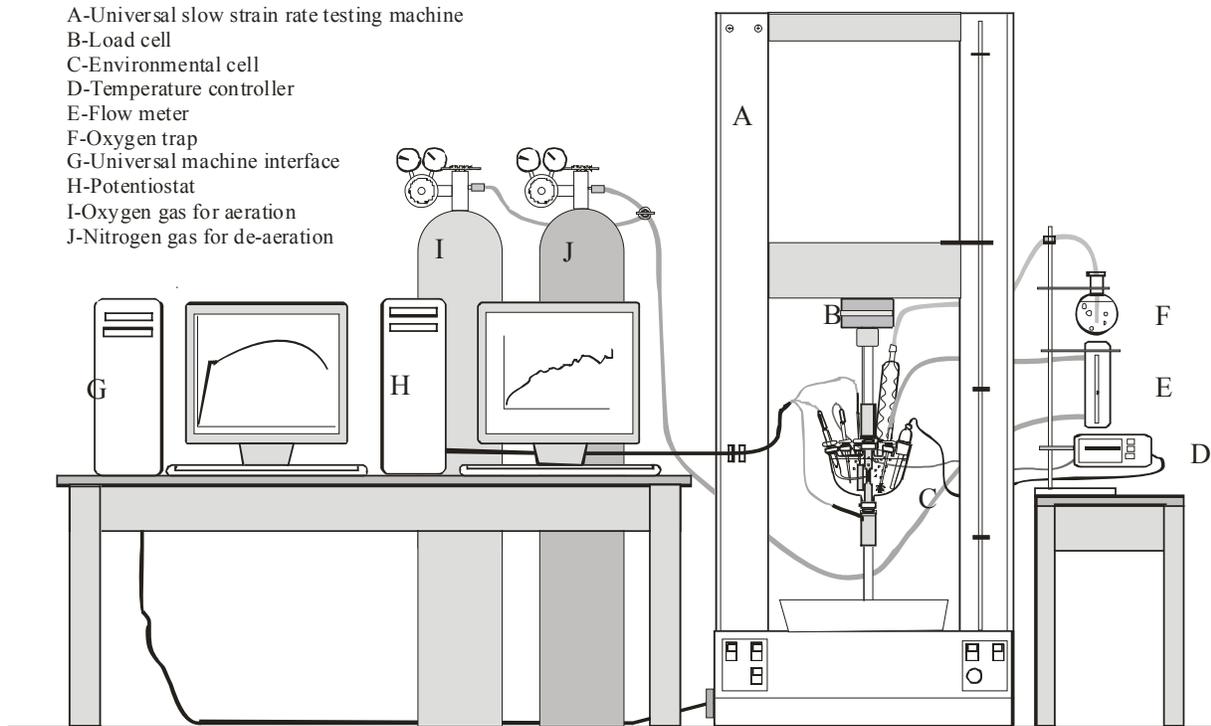


Figure 2. Experimental set-up for environmental cracking, such as SCC and HIC, and strain aging determination.

Thermal analyses of the rock bolt performed on the TGA showed that there was weight gain of the sample suggesting oxidation of the rock bolt, as seen Figure 3. The actual weight gain was ~ 0.084 mg, which is 0.074% increase. This is expected in these types of materials, and analyses are in progress. We also evaluated alloy C-22 as a baseline material with high corrosion resistance, for comparison purposes. These tests were performed under two different conditions; in flowing nitrogen and oxygen. The samples were heated to 950°C . Results for this alloy C-22 are shown in Figure 4. There are two curves in this Figure; top one (heating) is obtained by nitrogen flow and bottom one (heating) is under oxygen flow. Time, temperature and wt.% at the start and end of weight loss have been pointed out in Figure 4. It is surprising that there was weight loss during heating, although only 0.07% change.

We have repeated the experiment; so far all results indicate weight loss. In one case, we held the sample at 950°C for 4 hours after initial temperature increase. Results showed that there was initial weight loss and then a weight gain (at 950°C); these results are shown in Figure 5A. Figure 5B shows the same plot expect the x- axis is plotted in terms of "time". Rock bolt and Alloy 22 are being used as bench mark metals for evaluating the corrosion resistance of other materials that will be tested.

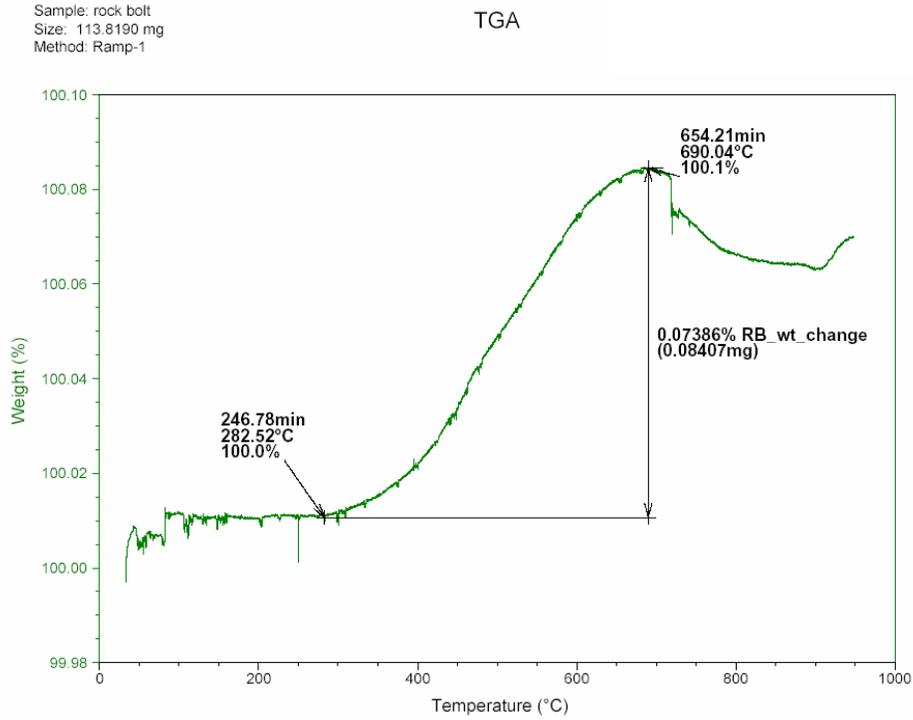


Figure 3. Rock bolt sample heated to 950°C in Nitrogen atmosphere, shows a weight gain.

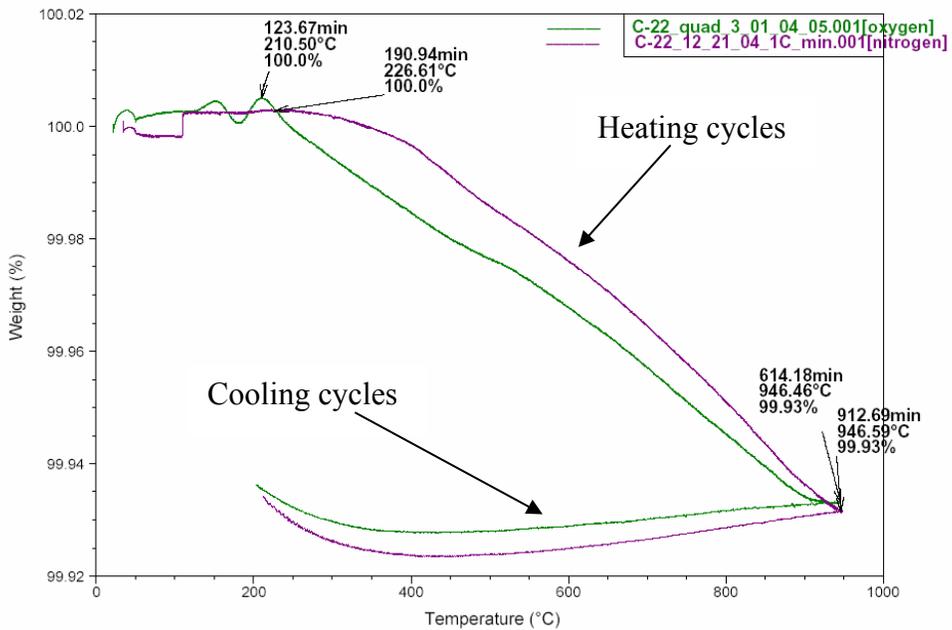


Figure 4. Combined plots of C-22 heated to 950°C in nitrogen and oxygen atmospheres showing a small weight loss during the TGA scan.

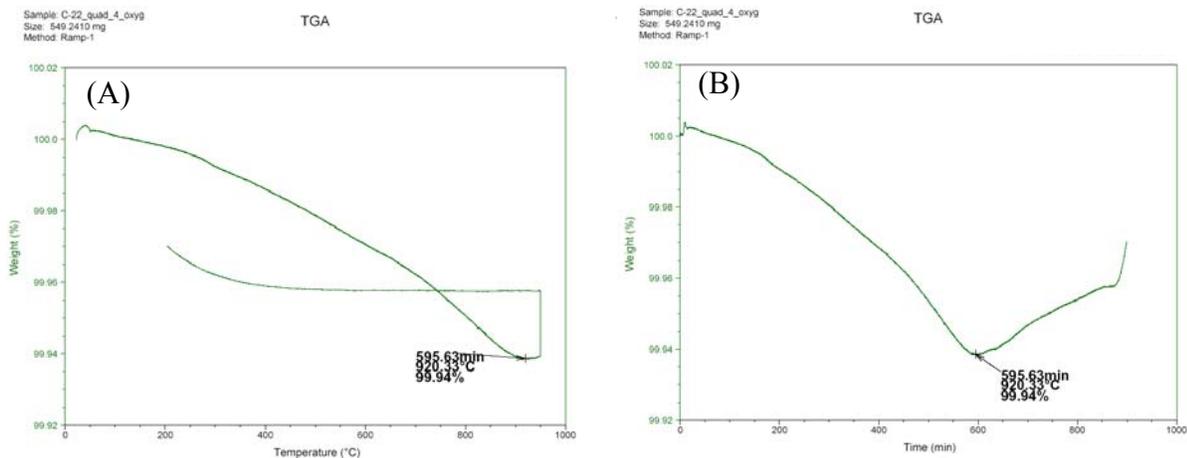


Figure 5. Plots of alloy C-22 heated to 950°C in oxygen atmosphere and held at that temperature for 4 hours.

Characterization of the Samples Before and after Heating:

X-ray diffraction analyses were performed on the alloy C-22 samples before and after the TGA heating run. The XRD pattern of the alloy C-22 sample in as received wrought condition is showed the alloy shown in Figure 6; and matches the FCC typical super alloy with similar compositions (*JCPDS card No. 35-1489 Ref 1*). The XRD patterns of the TGA heated specimens under N₂ and O₂ treatment showed sharpening the Bragg peaks indicating there the micro-strains are released due to annealing (Figure 6). The results were similar for both thee the samples that were heated with nitrogen and oxygen. The comparisons of the Bragg peak (111) for theses three samples are also shown in Figure 6. In Figure 6, the Si standard peak is also included to compare the broadened peaks of the alloy C-22 sample (before heat treatment).

Scanning microscopic samples of alloy C-22 samples were also examined that show the morphology of the surface film on Alloy-22 after heating in nitrogen atmosphere (Figure 7). *The inset* in Figure 7 shows a micrograph at lower magnification, covering a larger area, showing uniform covering on the surface. The EDAX scans on the sample before and after heating in nitrogen atmosphere indicate possible nitride film formations are also shown (Figure 8). It can be noted the XRD patterns have some non-indexed Bragg peak of very low intensities indicative of new phases formed during heating. Alloy-22 heated in a nitrogen atmosphere showed a thin film of uniform coating all over the outer surface of the sample. Since it is very thin it did not yield peaks of sufficient intensity during XRD and EDAX and its exact composition could not be evaluated; research is in progress to understand the mechanisms.

[1] R. Pfoersch, ICCD Grant-in-Aid (1984), Penn State University, University Park, Pennsylvania, USA

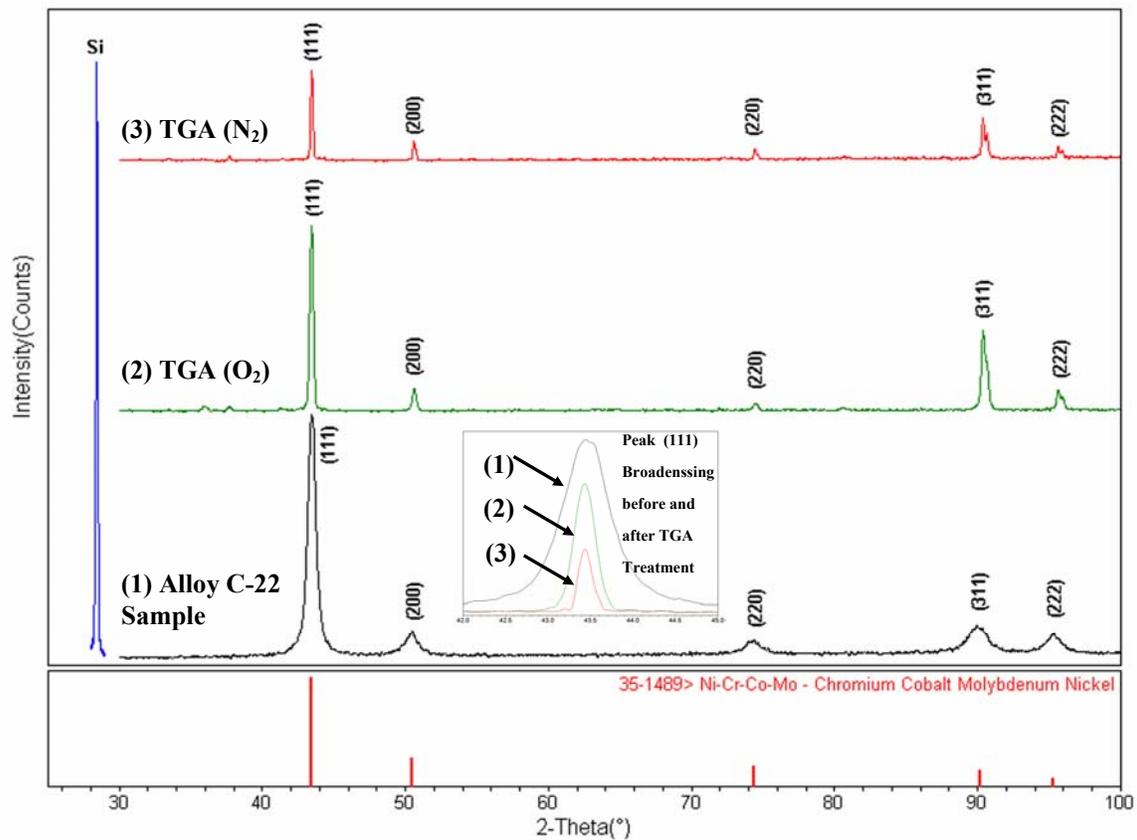


Figure 6. X-Ray diffraction scans of (1) Alloy-22 (2) Alloy-22 heated in oxygen atmosphere (3) Alloy-22 heated in nitrogen atmosphere.

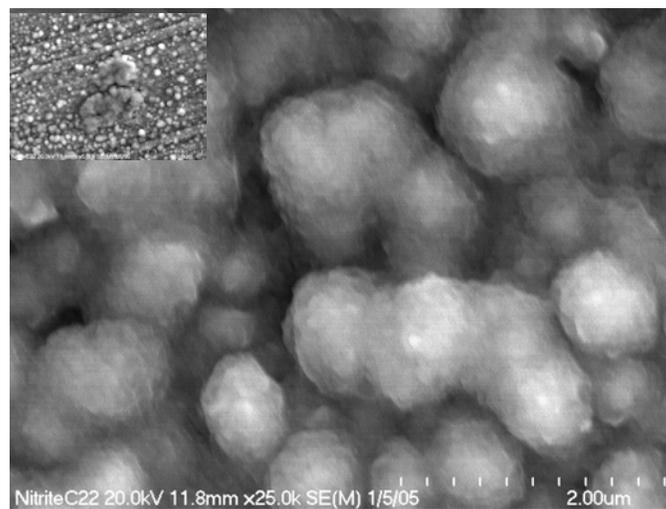


Figure 7. SEM micrographs showing the morphology of the film on Alloy-22 after heating in nitrogen atmosphere. The *inset* shows a micrograph at higher magnification, covering a larger area, showing uniform covering on the surface.

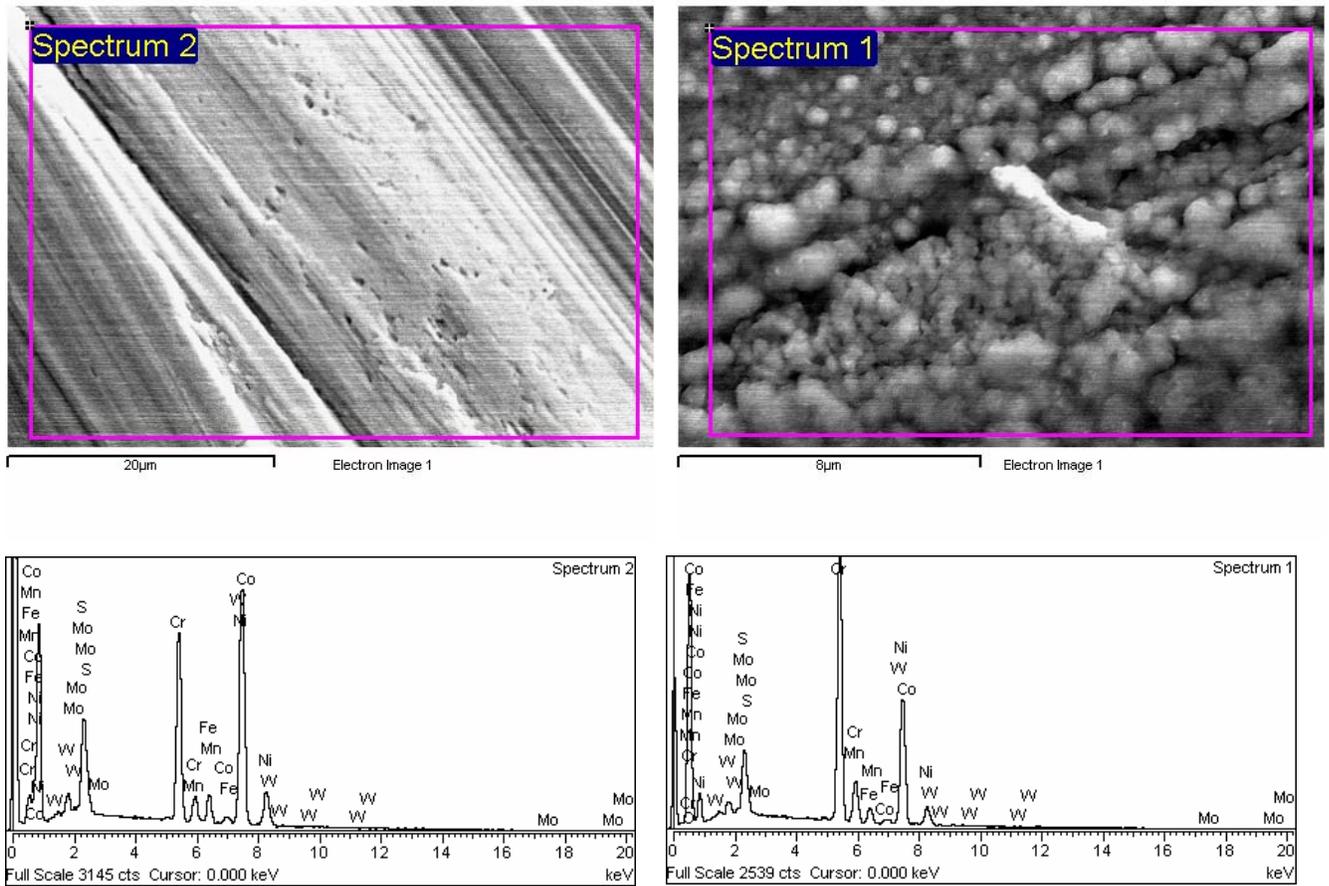


Figure 8. SEM micrographs and EDAX spectrum of Alloy-22 before and after heating in nitrogen atmosphere.

Environmental Stress Corrosion Tests:

Stress corrosion cracking experiments were performed on the baseline material of Alloy C-22 in 100x YM water for these experiments. Electromechanical tests were performed using the instrument in Figure 2, and the stress strain plot results are shown in Figure 9. The experiment had to be terminated due to a software problem, and thus the fracture was not observed. The photo in Figure 9 shows the length difference between the unbroken specimen and the specimen before pulling.

Stress vs. strain for alloy 22

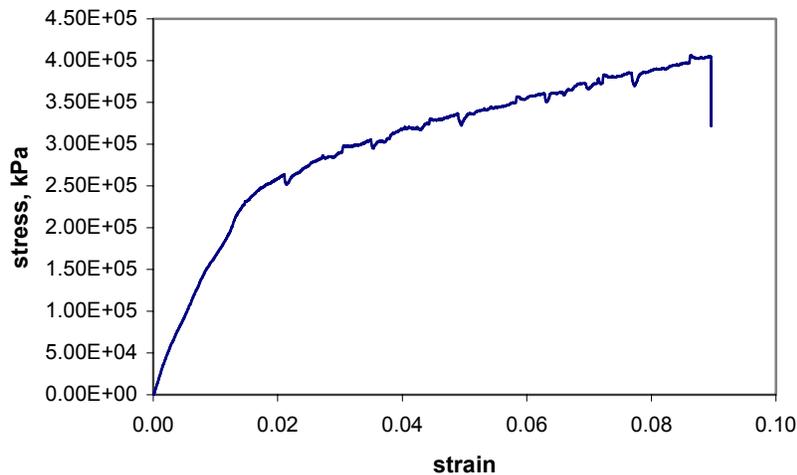


Figure 9. Stress strain plot of the alloy 22 in wrought condition. A photo of the specimens before and after the pulling in YM 100x water electrochemical environment are shown (right side). Please note the difference in the length the specimen before and after loading.

Time line

Our SIP for Task ORD-FY04-019 was approved on December 17, 2004. We have started Potentiodynamic tests and X-ray diffraction analyses as per timeline. We were not able to perform the EIS scans at this time as we have only one working Potentiostat that was being used for the stress corrosion cracking experiments. We could not start the immersion experiments as we do not have all the specimens machined due to delays. To compensate for this we have started the Stress corrosion task which is scheduled to start in Quarter No. 1 (12/1/2006) to effectively utilize the time. The machine shop machined these specimens first and the rest of them are being prepared. These delays due to lack of machined materials and others are not very significant, and we will be able to catch up with the tasks during the next quarter.