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# Phase Stability and Segregation in Alloy 22 Base Metal and Weldments

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**DOCUMENT CHANGE NOTICE  
(DCN)**

DCN No. 1 to Document No. SIP-UNR-037, Revision 0, Effective Date: 10/1/04.

Document Title: Phase Stability and Segregation in Alloy 22 Base Metal and Weldments

Identify applicable affected page, section, paragraph, attachment, exhibit, table, figure, or other:

Section 2.1 (Approach)

Scientific approach & technical methods (Subsection)

1. Reduce the number of specimens to a minimum of three different compositions, and make an effort to maximize the compositional range.
2. Reduce the number of welded samples produced to 3 temperatures for 3 anneal durations.
3. In the study of the point-to-point variability in the as-provided materials, reduce the number of plate locations from which samples are extracted, and perform the detailed study on only one mill-run (i.e., one manufacturer/composition).
4. Place on indefinite hold the study of sensitizing followed by replenishing (section 4c under Approach section below).
5. The examination of the residual stresses will be placed on indefinite hold.
6. Reduce scope of Electrochemical Testing to accommodate the above-described changes in the sample preparation plan.

Sequence of Work (Subsection)

7. Adjust schedule to accommodate above changes.
8. Delay procurement of backscatter detector until year 3 at the earliest.
9. Delay start of electrochemical testing until year 3.

Section 3 (Schedule of Work)

10. Adjust proposed schedule of work to accommodate the above changes.
11. Task Milestone schedule: Perform first interim data submittal in August of 2005 instead of May of 2005. Revert to original schedule after this.

Section 7 (Samples/Specimens)

12. Adjust plans for sample preparation to match the above described changes

This DCN, or elements thereof, may be cancelled to restore original scope to the SIP.

Approved by:

[Redacted Signature]

1110

PI: \_\_\_\_\_ Date: 3/17/2005

(Signature)

Print name: Jeffrey C. LaCombe

QA Manager: \_\_\_\_\_ Date: 3-28-05

(Signature)

Print name: Amy J. Smiecinski

QA Manager evaluated acceptability, that it does not violate quality requirements, and for impacts to other procedures; signature above documents this evaluation as successfully completed.

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University and Community College System of Nevada  
(UCCSN)

Scientific Investigation Plan  
(SIP)

**Task Title:** *Phase Stability and Segregation in Alloy 22 Base Metal and Weldments*

**Task Number:** **ORD-FY04-015**

**Subtask 1:** *Microstructural Characterization of Phase Stability and Variability in Alloy 22.*


**Subtask 2:** *Electrochemical Methods to Detect Susceptibility of Alloy 22 to Localized Corrosion*

**Document Number:** **SIP-UNR-037**

**Revision:** **0**


**Effective Date:** 10/25/, 2004

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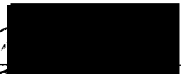
  
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9/14/04  
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10-22-04  
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## Revision History

<b>Revision Number</b>	<b>Effective Date</b>	<b>Description and Reason for Change</b>
0	10/25/04	Initial issue under cooperative agreement.

## 1. Scope and Objectives:

### Introductory Comments & Background

This SIP directly follows from the proposal submitted through the UCCSN to the Department of Energy, Yucca Mountain Project under the same title. The components of this study (to be discussed below) follow from critical issues that were largely identified in February of 2002, when the Waste Package Materials Performance Peer Review Panel reported [1] to the USDOE, Bechtel SAIC Co., and LLC on the waste package design intended for use by the Yucca Mountain Project. This document (SIP-UNR-037) is intended to serve as a planning document, resulting from the scope of work originally proposed.

The current design of the waste disposal containers relies heavily on encasement in a multi-layered container, featuring a corrosion barrier of Alloy 22, a Ni-Cr-Mo-W based alloy with excellent corrosion resistance over a wide range of conditions. The fundamental concern from the perspective of the Yucca Mountain Project, however, is the inherent uncertainty in the (very) long-term stability of the base metal and welds. Should the properties of the selected materials change over the long service life of the waste packages, it is conceivable that the desired performance characteristics (such as corrosion resistance) will become compromised, leading to premature failure of the system. To address this, we will study the phase stability and solute segregation characteristics of Alloy 22 base metal and welds. A better understanding of the underlying microstructural evolution tendencies, and their connections with corrosion behavior will (in turn) produce a higher confidence in the extrapolated behavior of the container materials over time periods that are not feasibly tested in a laboratory. Additionally, the knowledge gained here may potentially lead to cost savings through development of safe and realistic design constraints and model assumptions throughout the entire disposal system.

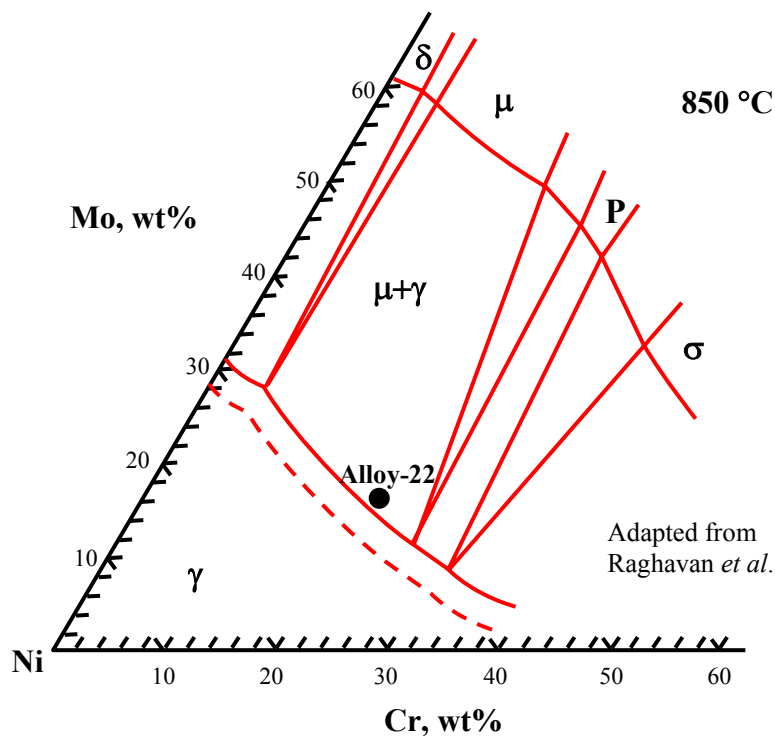
Ni base alloys with 16-22% chromium and 9-16% molybdenum are commonly used for higher corrosion resistance applications [2-7]. Alloy-22 (UNS N06022), a Ni-Cr-Mo-W alloy, is the current reference material for construction of the outer wall of nuclear waste containers [2-4] to be used by the Yucca Mountain Project. The nominal composition of Alloy-22 and the compositions of some of the reported phases seen in this alloy system are listed in Table 1.

Even though Alloy-22 in wrought form is considered to have good phase stability at the operating temperatures (<200 °C) of the repository, exposure to elevated temperatures during the fabrication processes (such as welding and stress relieving) could cause alteration of microstructure and associated deterioration of mechanical and corrosion properties. Welding causes microstructural changes in Alloy-22, such as formation of dendrites in the weld metal, segregation of Mo and W in the interdendritic

regions, formation of topologically close packed phases both in the weld metal and the heat affected zone (HAZ), and possibly precipitation of long range ordered phases [3, 5-7]. Formation of such secondary phases could make the material susceptible to intergranular corrosion and/or reduce strength and ductility [8-10]. Whereas the equilibrium solidification

Phase	Ni	Mo	Cr	W	Fe	Co
Nominal	56.96	13.43	21.22	3.29	3.17	0.84
$\gamma$ (Matrix)	58.5	12.7	21.6	2.9	3.4	0.9
$\mu$	33.1	38.7	19.3	6.3	2.1	0.6
$P$	32.6	37.4	21.7	5.3	2.2	0.9
$\sigma$	34.5	34.9	23.4	4.2	2.2	0.9

phase of Alloy-22 is the austenitic  $\gamma$  phase (which can be obtained by quenching), at lower temperatures, secondary phases such as  $\mu$  are thermodynamically stable at the Alloy-22 composition. **Figure 1** (adapted from [11]) illustrates that at 850 °C, the equilibrium phases to be expected include  $\mu+\gamma$ . At still lower temperatures, the  $\gamma \rightleftharpoons \mu+\gamma$  boundary is expected to move even closer to the Ni corner of the diagram (see dashed line in the figure). This may make the alloy potentially susceptible to phase separation at lower temperatures.



**Figure 1:** Isothermal section of Cr-Ni-Mo alloy phase diagram at 850 °C.

In low carbon Ni-Cr-Mo alloys, sensitization has been reported to occur mainly because of the precipitation of topologically close packed (TCP) phases like  $\mu$  and  $p$  [2-7]. Increased corrosion rates of aged Ni-Cr-Mo-W alloys were observed because of this sensitized microstructure [10]. The sensitization of Mo-rich nickel base alloys is found to be different from the sensitization of common austenitic stainless steels and other Ni-Cr alloys such as Inconel 600. Sensitization of austenitic stainless steel resulted in depletion of chromium adjacent to the chromium rich  $M_{23}C_6$  carbides [12, 13]. In the Ni-Cr-Mo alloys, sensitization resulted in depletion of molybdenum near the TCP or  $M_6C$  precipitates [10, 14]. When the sensitized Ni-Cr-Mo alloys were exposed to a reductive environment the Mo depleted regions were preferentially attacked and in an oxidizing environment the TCP phases themselves were dissolved, giving rise to the corrosion rate [14]. Raghavan *et al.* [11] and Cieslak *et al.* [5-7] observed the TCP phases containing only the nominal chromium as that of the bulk chemistry. Whereas, Hodge [14, 15] reported that the  $\mu$  phase was enriched with Cr and the suggested phase was  $(Ni, Fe, Co)_3(W, MO, Cr)_2$ . This issue is not fully resolved as of yet. There are no reports available on the depletion profiles of aged Alloy-22. Depletion of alloying elements in the vicinity of secondary phase precipitates will impair the corrosion resistance of the alloy. Therefore, it is imperative to develop an understanding of how the microstructure changes during fabrication or exposure to service conditions so that the integrity of the waste package container can be ensured.

The scope and objectives of this task, “Phase Stability and Segregation in Alloy 22 Base Metal and Weldments” will support the Yucca Mountain Project (YMP) by characterizing and developing our understanding of the waste package material performance and formulating these findings in a manner that contributes to the direction of design and fabrication decisions. In doing so, we aim to build confidence in the long-term performance of the waste package system. This task (ORD-FY04-015) is divided into two connected subtasks that will improve our

understanding of the stability and variability of the microstructure in Alloy 22 and relate this to corrosion behavior of the material. The two subtasks are:

**Subtask 1: *Microstructural Characterization of Phase Stability and Variability in Alloy 22.*** Develop an improved understanding of Alloy 22 and the extent to which compositional and microstructural variations are present in otherwise “nominal” as-procured material.

**Subtask 2: *Electrochemical Methods to Detect Susceptibility of Alloy 22 to Localized Corrosion.*** Study the influence that compositional and microstructural variations have on the corrosion performance of Alloy 22.

### **Key Findings of Prior Scoping Study**

Whereas this task is a new line of formal investigation, a preliminary scoping study of the evolution and influence of various TCP phases in Alloy-22 (UNS N006022) on corrosion was previously conducted during the prior Cooperative Agreement under Task 14. In summary, the initial findings confirmed that Alloy 22 has the potential for forming grain boundary precipitates that are rich in Molybdenum and Tungsten. This has the potential effect of making the grain boundaries susceptible to corrosion in a reducing environment. Below is a selection of key findings of this scoping study.

- **Wrought Microstructure:** In un-aged wrought samples (not welded), there do appear to be grain boundary precipitates that can influence corrosion. The extent and nature of these inhomogeneities in mill-provided material must be better understood. Furthermore, we must evaluate these data in the context of subsequent thermal processing steps in the fabrication of nuclear storage casks.
- **Weld Microstructure:** The microstructure of the weld metal (even that aged at 760 °C for 140 h) revealed grain boundary precipitates along with interdendritic precipitates. The nature and extent of these precipitates, as well as their response to thermal treatments needs further investigation.
- **Aging:** Consistent with the well-established understanding of alloy-22, annealing of the material can lead to enhanced precipitation and grain growth of the “undesirable” TCP phases, especially along grain boundaries. These precipitates are accompanied by solute depletion in the surrounding regions that can be detrimental to corrosion resistance of the material.
- **EPR test methods:** Electrochemical Potentiokinetic Reactivation (EPR) test methods can be adapted for use with alloy-22 to determine the susceptibility to intergranular corrosion and correlate this with the accompanying microstructure. As part of the scoping study at UNR, suitable electrolytes for use in EPR tests were developed for detecting Cr and Mo depletion. Promising results were obtained with the following electrolytes:

For Cr-depletion	1M H <sub>2</sub> SO <sub>4</sub> + 0.5M NaCl + 0.01M KSCN	@ 30 °C
For Mo-depletion	2M HCl + 0.01M KSCN	@ 60 °C

- **Cr Depletion:** Both single and double loop EPR tests show an increase in reactivation current with an increase in aging time at 650 °C, but a decreasing reactivation current with increase in aging time at 700-750°C. Micrographs and EDX line scans of the



samples aged at higher temperatures and for longer time periods show no measurable Cr depletion. This suggests that at either lower temperatures or at earlier times, there are regions of Cr depletion that disappear later. This suggests an underlying diffusion-related process in which Cr is depleted and then eventually replenished.

- **Mo & W Depletion:** EPR tests indicate that Mo and/or W depletion is present in both wrought and welded samples after aging occurs. This is confirmed in EDX line scans that reveal Mo and W-rich grain boundary precipitates.
- **Weld Material Phase Separation and Corrosion:** Weld metal of Alloy-22 showed Mo segregation at the inter-dendrites and some Mo rich intermetallic phases. During aging of the weld metal, no chromium depletion was observed. Some aged weld specimens showed very high ratios of reactivation to activation current as compared to other aged specimens. This is consistent with the presence of Mo and W depleted regions and a greater susceptibility to corrosion.

### Scope of Work

The research program will focus on the following interrelated components. They are divided into two subtasks.

#### Subtask 1    **Microstructural Characterization of Phase Stability and Variability in Alloy 22**

*Develop an improved understanding of Alloy 22 and the extent to which compositional and microstructural variations are present in otherwise “nominal” as-procured material.*

**Issue 1.1 Characterize the as-fabricated Alloy-22 base metal.** To formulate an understanding of the long term stability of the Alloy-22 base metal and welds, it will be necessary to first develop an understanding of the initial state of the material and its properties, as well as supplier-to-supplier and heat-to-heat variation in characteristics.

*We will conduct a metallurgical analysis of the base metal using optical microscopy, SEM, and TEM imaging and diffraction methods. These analyses will also include compositional analysis and phase identification. Electrochemical methods that are selectively sensitive to material composition may also be implemented to complement this analysis. We will work to determine whether a more restrictive chemical composition specification for the alloy would reduce uncertainty and increase confidence with respect to the stability of the alloy and the corrosion behavior of waste packages.*

**Issue 1.2 Characterize Alloy-22 welds:** The closure weld and its response to the attending high temperatures during the welding are critical to long-term performance of waste packages. The primary issues affecting the long-term integrity of the welds in Alloy 22 are the as-fabricated microstructure of the welds and the weld heat affected zones (HAZs), the level and nature of residual stresses in the welds and HAZs and the long-term stability of the weld zone microstructure. These microstructures and stresses are the most significant material variables affecting the resistance of the waste package to localized failure by corrosion or stress corrosion cracking.

*We will determine the extent to which variations in the welding and post weld processes, as well as base material and filler compositions affect the microstructure of the welds and HAZs. Variables that would be included in the scope of work would include base material and filler metal compositions at the extreme limits of the ASTM or YMP specifications. Activities will include procuring and characterizing weld samples (including characterization of base metal and filler materials) for microsegregation, precipitate formation, long-range ordering, and a residual stress scoping study.*

**Issue 1.3 Long-term metallurgical stability: Cr-Mo depletion and Long Range Ordering.** Long term exposure of Alloy 22 to moderately elevated temperatures can lead to diminished corrosion resistance by species depletion and degradation of mechanical properties by long range ordering.

*We will expand the scoping study presented above (Section 1.1) to determine how element depletion (such as chromium or molybdenum) occurs adjacent to the grain boundaries. We will identify the time and temperature relationship for this depletion and additional effort will be directed towards evaluating the amount of heat-to-heat variability of the source material (within the alloy specifications).*

**Issue 1.4 Segregation of sulfur and phosphorous.** Sulfur and phosphorus are minor constituents in Alloy 22, and when segregated (which can occur upon welding), they can have detrimental effects on corrosion and stress corrosion cracking resistance. Additionally, sulfur has been observed in stainless steel to influence the weld pool behavior and the weldment properties.

*The extent of segregation of minor constituents will be measured to determine the potential for grain boundary impurity segregation as a function of time and temperature in the bulk and weld metal and in the heat affected zone of welds.*

### **Subtask 2 Electrochemical Methods to Detect Susceptibility of Alloy 22 to Localized Corrosion.**

*Study the influence that compositional and microstructural variations have on the localized corrosion performance of Alloy 22.*

**Issue 2.1 Develop an EPR test solution and Cr depletion test procedure.** This will facilitate detection and quantification of the level of chromium depletion in Alloy-22 base metal and weldments.

**Issue 2.2 Develop an electrochemical test solution and Mo segregation test procedure.** This will facilitate detection and quantification of the level of Mo segregation in weld metal and Cr and Mo depletion in the matrix due to the formation of TCP phases.

**Issue 2.3 Study the effect of precipitation of secondary phases on the corrosion resistance of Alloy-22,** especially in chloride and fluoride containing environments.

This work is subject to the University and Community College System of Nevada (UCCSN) Quality Assurance (QA) Program requirements.

This Scientific Investigation Plan (SIP) presents an independent confirmatory study for comparison with previously gathered information.

## 2. Approach:

**Scientific approach & technical methods:** The work plan essentially consists of the following major parts discussed in detail below.

1. **Material Selection & Procurement:** Suppliers will be consulted with to identify the heats that are available and their corresponding compositional makeup. Plate stock will then be procured from multiple suppliers with specific heats selected to cover the desired range of compositions to be studied.
2. **Sample Preparation:** Test samples will be labeled for identification and machined from the provided plates into the desired form(s) for subsequent processing and testing in both Subtasks 1 & 2. A sample preparation IP will be developed early on to standardize the methodology as much as possible.
3. **Welding:** Weld specifications will be established and samples will be procured from a QA-approved supplier for subsequent study. Gas Tungsten Arc Welding (GTAW) will be used, both autogenously and with filler. Single and multi-pass welds will be evaluated to determine the influence of thermal cycling on previous weld passes and the cumulative effect after multiple passes.
4. **Heat Treatment:** Heat treatment schedules will be established and samples will be heat treated. Early on, an IP will be developed to help govern this process. An example heat treatment test matrix would include
  - a. Solution Annealing of the wrought mill-annealed will be conducted at various temperatures *ca* 1120°C. Annealing will be conducted for various durations (ex: 5, 10, 20, 60 minutes). At the conclusion of the anneal, samples will be quenched at various cooling rates.
  - b. Sensitizing heat treatments will be conducted at 650, 700 and 750°C for 1, 10, and 100 hours.
  - c. Sensitizing heat treatments at 700°C for 24 h, followed by a replenishing treatment (800 and 850°C for 100h)
    - a. For the welded material, the heat treatment would include aging at 700, 750 and 850°C for 1, 10, 100, 1000 and 3000 h.
    - b. As the investigation proceeds, other welding and heat treatment conditions will be included as required (example: diffusion tests).
5. **Microstructural Characterization:** The as-provided and processed samples will be characterized using a variety of techniques to develop an understanding of the attending microstructures for each set of conditions. Key methods will include optical microscopy, X-Ray and electron diffraction, and Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM), both accompanied by Energy Dispersive X-Ray techniques (EDX). IPs will be developed for each of these instruments/techniques.

6. **Electrochemical testing (Subtask 2):** All electrochemical tests will be carried out following the general procedure described in ASTM standard G-5. No aeration or deaeration will be carried out. Double loop EPR tests will be carried out on the specimens, which will be polished to 600 grit. The specimens will be polarized from the corrosion potential to an appropriate potential in the passive range (300-500 mV (SCE)) at a predetermined scan rate (50-100 mV/min) and the potential will then be reversed back to free corrosion potential at a test temperature of 30°C. The ratio of peak currents recorded during forward scanning ( $I_a$ ) and reverse scanning ( $I_r$ ) will be used to determine the degree of sensitization. A higher value of  $I_r/I_a$  implies a higher susceptibility to IGC.
- a. **Detection of Cr Depletion:** Fine tuning of EPR test procedures for Cr depletion described earlier (1M H<sub>2</sub>SO<sub>4</sub> + 0.5M NaCl + 0.01M KSCN) will be conducted.
  - b. **Detection of Mo segregation and TCP phases:** Fine tuning of EPR test procedures for Mo depletion described earlier (2M HCl + 0.01M KSCN) will be conducted.
  - c. **Comparison with conventional chemical weight loss measurements:** Weight loss measurements will be carried out on all the heat treated specimens after the Ferric sulfate-sulfuric acid and mixed acid oxidizing salt tests according to the ASTM G-28 standard. EPR results will be correlated with the weight loss data of ASTM G-28 tests.
  - d. **Correlation of electrochemical data to microstructural changes:** Results from (5) and (6) above will be studied to identify correlations.
  - e. **Evaluation of Localized Corrosion Resistance Degradation, and Data Analysis:** Potentiodynamic polarization curves will be constructed in chloride containing solutions on select wrought and welded specimens with and without secondary phases. The heat treatment for the formation of secondary phases will be selected based on the EPR results. The temperature of the corrosive environment will range from 20 – 90°C. Other polarization procedures and temperatures will be considered and implemented as required. The key parameters are passive current density and passivation potentials.
  - f. **Data Analysis:** Double loop EPR tests will be carried out on a set of specimens with all the proposed heat treatment conditions in test solutions of different compositions. If there is no distinction between the peak current ratios in a given solution composition for two extreme heat treatment conditions (for example 650° C for 1 h and 700° C for 100 h), no further tests will be conducted for that solution. That solution composition will be considered as unsuitable for EPR evaluation. If the peak current ratios are resolvable between heat treatments in a given solution composition, at least three test runs will be carried out in that solution and the data will be statistically analyzed for comparison with other test solutions/procedures. The proposed test matrix for the EPR studies is shown in [Table 2](#).

**Table 2:** Proposed test matrix for EPR studies

Experiment	Heat Treatment Condition				
	Mill Annealed	650° C 1, 10, 100 h	700° C 1, 10, 100 h	750° C 1, 10, 100 h	850° C 1, 100, 1K h
<b>EPR for Cr depletion</b>	BM, WM (as welded)	BM, WM (100h)	BM, WM (100h)	BM, WM (100h)	BM (1000h) WM
<b>ASTM-G28 Weight loss</b>	BM, WM (as welded)	BM, WM (100h)	BM, WM (100h)	BM, WM (100h)	BM (1000h) WM
<b>Polarization chloride</b> in	BM, WM (as welded)	BM, WM (100h)	BM, WM (100h)	BM, WM (100h)	BM (1000h) WM
<b>EPR for Mo segregation and TCP phase</b>	As welded			WM	WM
<b>Microstructural evaluation</b>	BM, WM	BM (100)	BM	BM, WM	WM

BM: Base metal in wrought condition, WM: Weld metal; WM(100h): Weld metal aged for 100 h. Aging will not be done for other durations.

7. **Residual Stress Measurements:** Time permitting, a scoping study, characterizing the residual stresses in the weld and surrounding metal will be conducted and correlated with microstructural evolution and corrosion behavior. These tests will be conducted by Lambda Research (on the UCCSN Qualified Supplier List).

### Sequence of Work:

All processes will be described fully in scientific notebooks and Implementation Procedures. Special skills (such as instrument operation) will be identified as appropriate and incorporated into IPs and instrumentation operation checklists.

An outline of the work sequence is as follows.

- 1) To take advantage of synergies between this task and ORD-FY04-014 (investigating variability in electrochemical behavior), an effort is being made to identify common compositions for study in both tasks, making for more efficient procurement and characterization, and permitting additional cross-referencing of data between tasks.

To start, Alloy 22 plate stock will be obtained from 3-5 suppliers. Three different mill heats will be purchased from the selected suppliers (selected to also accommodate ORD-FY04-014's needs). Certification sheets for all available heats will be requested from each supplier for choosing the heats to be obtained. Heats will be chosen to represent mid, upper and lower limits of chromium, molybdenum, and iron concentrations as specified by ASTM - B575. An attempt will be made to procure the following combinations (as available):

- i) High Cr, Low Mo, with Cu
- ii) High Cr, low Mo, with Co and V
- iii) Low Cr, high Mo, with Cu
- iv) Low Cr, high Mo, with Co and V
- v) Mid Cr, Mid Mo, low Fe
- vi) Mid Cr, Mid Mo, low Fe

It should be noted, however, that there are a limited number of suppliers of Alloy-22, each producing alloys with characteristic compositions. The constituents that we will attempt to monitor most closely will be those that affect the precipitate nucleation process and those that directly affect the corrosion behavior.

- 2) Each plate obtained will be sampled and tested according to ASTM standards for mechanical, properties, chemical analysis, and grain size by a laboratory on the Qualified Supplier List (QSL) to qualify it for work under the HRC QA program.
- 3) Welding will be performed on selected samples by either a service provider on the QSL, or in cooperation with ORD-FY04-014, which is planning to develop some basic welding procedures.
- 4) Characterization will be performed with the metal in each of three metallurgical states:
  - i) Mill-annealed (as received)
  - ii) Solution annealed (at various temperatures and quenches)
  - iii) Aged (at various temperatures, durations, and quenches)
- 5) Characterization will be performed on specimens derived from a single common source plate (minimum of 4 locations) to assess microstructural point-to-point variability in the provided material. Additionally,
- 6) Electrochemical (EPR) tests will be performed on specimens from (4) and (5).
- 7) Microstructure data will be assembled and analyzed to identify trends and correlations with processing history.
- 8) Corrosion data will be assembled and analyzed to identify any correlations between the observed microstructures and corrosion behavior. The need for additional specimens and testing methods will be determined.
- 9) As a scoping study, a selection of welded and un-welded specimens will be analyzed (via an external facility) to quantify the residual stresses and identify any correlations between the stress state, the microstructure, and the corrosion behavior. It is possible that this secondary component of the task may incorporate non-QA data if a testing facility on the Qualified Supplier List cannot be identified or a new supplier cannot be qualified.
- 10) As secondary scoping study, the multicomponent diffusion behavior of the principal components of Alloy-22 will be conducted. This will likely necessitate the use of an electron microprobe for precise composition profile measurements in diffusion couples, triples, and multiples. If qualified microprobe data cannot be obtained, this secondary scoping study may not produce QA results.

#### **Justification for non-QA-affecting subtasks, purchases:**

The overall efforts in Subtasks 1 and 2 will be Quality Affecting (QA). Within Subtask 1, two scoping studies are planned to study residual stresses and the diffusional behavior of Alloy 22. Reasonable attempts will be made to perform this work in compliance with QA requirements. However, these activities are intended to be small (non-critical) scoping studies and additional resources will not be allocated for QA compliance if these costs are deemed prohibitive. If difficulties are encountered in identifying suppliers or testing facilities on the QSL, then these

contributing activities may be designated non-QA-affecting components of Subtask 1 that will be used for scoping or corroborative purposes only.

**Subtask change from Q to non-Q under Q-task:** Not applicable.

### 3. Schedule of Work

#### Year 1 (7/1/04 - 9/30/04)

- Development and submission of Scientific Investigation Plan (SIP, this document)
- Quality Assurance (QA) training
- Procurement of non-quality affecting equipment and instruments (furnace(s), polishing equipment & supplies, data logger, etc.)
- Literature studies
- Secure funding for FE-SEM Backscatter detector and supporting equipment
- Scoping work for development of Implementation Procedures (IPs)

#### Year 2 (QA Work Starts. Emphasis on Weld Materials)

- Procure FE-SEM Backscatter detector and install onto existing equipment and integrate the new equipment into the experiment plan.
- Procurement of sample material and fabrication of test samples (wrought and welded).
- Develop sample preparation methods (SEM, TEM, Optical Microscope).
- Characterization and cataloging as-provided material.
- Procurement and setup of heat treatment furnace and data logging system.
- Conduct literature review supporting study of multicomponent diffusion in Alloy 22. Develop detailed diffusion study test plan.
- Fine tune EPR methods and conduct experiments on aged wrought material.
- Conduct conventional ASTM G-28 tests on aged wrought Alloy-22 coupons and correlate results with the EPR data.
- Investigate process for specification and procurement of welded material samples.
- Collaborate with the activities being undertaken in ORD-FY04-014.
- Develop and maintain collaborative dialog with other researchers (including “external interfaces”).

#### Year 3 (Emphasis on Weld Materials)

- Establish detailed weld study test parameters. Procure welds.
- Commence microscopy studies (optical and electron) of sample weld microstructures and correlate results with corrosion behavior. Microstructural changes due to thermal cycling of adjacent weld passes (in multi-pass welds) will be evaluated.
- Conduct experiments to elucidate the diffusion behavior of the various components of Alloy-22 for various microstructural conditions and correlate with microstructural evolution and corrosion behavior.

- Conduct hardness tests (including microhardness maps) of weld and base metals.
- Apply EPR test for detecting Mo segregation and TCP phase formation in welds.
- Conduct conventional ASTM G-28 tests on aged welded Alloy-22 coupons and correlate results with the EPR data.
- Carry out optical and SEM microstructural characterization on some selected specimens of welded and aged Alloy-22 for correlation with the observed intergranular susceptibility by EPR method.
- Conduct electrochemical localized corrosion tests at 30 °C on both aged wrought and weld specimens of Alloy-22.
- Preparation for residual stress measurement study.

**Year 4:**

- Continue preparation of and analysis of samples along the lines of earlier years in an iterative manner, to develop a more comprehensive understanding of the microstructure and corrosion performance.
- Study of the solutal segregation behavior including microstructural characterization and elemental profiling across the grain boundaries and dendrite/interdendritic regions of all material conditions will be completed.
- Conduct residual stress measurement study.
- Electrochemical localized corrosion tests will be carried out at elevated temperatures in the concentrated chloride environments simulating YM repository water.

**Year 5:**

- Continue preparation of and analysis of samples along the lines of earlier years in an iterative manner, to develop a more comprehensive understanding of the microstructure through microscopy and corrosion studies.
- Complete various activities begun in earlier years.
- Conduct task closeout and prepare final report.

**Task Milestone Schedule**

<b>Date</b>	<b>Milestone</b>
7/9/2004	QA Indoctrination for key personnel
7/31/2004	QT Files completed for key personnel
8/13/2004	Submit Scientific Investigation Plan for technical review Submit Scientific Investigation Plan for QA review
9/1/2004	SIP approval and commencement of QA work.
5/10/2005	Interim Data submittal (REPEAT EVERY 6 MONTHS, May/November) Notebook/data technical and QA review completed
5/15/2005	Interim Data submittal (REPEAT EVERY 6 MONTHS, May/November)



	Notebook/data submitted to TDA
4/1/2008	End data collecting/testing/measurements
5/15/2008	Perform final calibrations or calibration checks
6/1/2008	Complete data reduction/analysis
6/15/2008	Submit scientific notebook for final technical review Submit scientific notebook for final QA review
7/15/2008	Submit final data to TDA
8/10/2008	Submit final technical report for technical review Submit final technical report for QA review
9/23/2008	Submit final report to DOE
9/30/2008	Final records submittal

#### 4. Interface Controls

Dr. Jeffrey LaCombe of the University of Nevada, Reno, will serve as the Task's Principal Investigator. Dr. LaCombe will oversee Subtask 1 and assure the integrity and scientific soundness as the work progresses. Dr. Shantanu Namjoshi will oversee Subtask 2 and assure the integrity and scientific soundness as the work progresses.

Administrative interfaces at UNR include Dr. Ted Batchman, Dean, College of Engineering, and Dr. Manoranjan Misra, Division Chair, Metallurgical and Materials Science Engineering.

Planning, data, results, analysis, and conclusions will be shared and discussed with Dr. Raul Rebak, Dr. Tammy Summers, Dr. Frank Wong, and Dr. Tian Lian at Lawrence Livermore National Laboratories (LLNL), Gerald Gordon and V. Pasupathi of the Bechtel-SAIC team, and Robert Fish of Booz-Allen & Hamilton, the technical support contractor for DOE in Las Vegas. The DOE Technical Task Representative will be Paige Russell.

#### 5. Standards

The following published industry standards and criteria will be followed and incorporated (or adapted, where appropriate) into Implementation Procedures (IPs):

##### A. Materials

- Standard Specification for Low-Carbon Nickel-Molybdenum-Chromium, Low-Carbon Nickel-Chromium-Molybdenum, and Low-Carbon Nickel-Chromium-Molybdenum-Tungsten Alloy Plate, Sheet, and Strip, B 575-94

##### B. Grain Properties

- Standard Test Methods for Average Grain Size, E 112-96
- Characterizing Duplex Grain Sizes, Test Methods for, E 1181
- Assessing the degree of banding or orientation of microstructures, E 1268

- Determining the orientation of a metal crystal, E 82-91
- Estimating the largest grain observed in a metallographic section (ALA Grain Size), E 930
- Determining the average grain size using semiautomatic and automatic image Analysis, E 1382

### **C. Metallography**

- Metallographic Laboratory safety, E 2014
- Metallographic laboratory evaluation, E 807-96
- Preparation of Metallographic specimens, E 3
- Macroetching Metals and Alloys, Test Method for, E 340
- Microetching Metals and Alloys, Practice for, E 407
- Electrolytic polishing of metallographic specimens, E 1558-99
- Standard Practice for determining the inclusion or second-phase constituent content of metals by automatic image analysis, E 1245-95 the inclusion content of steel, E 45-97

### **D. Microscopy**

- Calibrating the magnification of a Scanning Electron Microscope, E 766-98
- Scanning Electron Microscope Beam size characterization performance Characterization, E 986-97
- Measuring and reporting tip shape in scanning probe microscopy, E 1813
- Standard Guide for Quantitative Analysis by Energy-Dispersive Spectroscopy, E 1508-98

### **E. Hardness and Micro-hardness Testing**

- Micro-hardness of Materials, Test Method for, E 384
- Vickers hardness of metallic materials, E 92
- Rockwell hardness and Rockwell superficial hardness of metallic materials, E 18-98
- Macro-Rockwell hardness testing of metallic materials, E 1842-96

### **F. XRD of Metals (Alloy 22)**

- Standard test method for verifying the alignment of X-Ray Diffraction instrumentation for residual stress measurement, E 915-96
- Standard test method for determining the effective elastic parameter for X-Ray diffraction measurements of residual stress, E 1426-98
- Determining the Orientation of a Metal Crystal, Test Method for, E 82-91
- Preparing quantitative pole figures, E 81-97

## **6. Implementing Procedures**

Implementing procedures (IPs), will be developed for routine project tasks. All non-routine work without an associated IP will be documented in the scientific notebook as described in “Scientific Investigation Control”, QAP-3.0. Established, published standards will be incorporated wherever possible.

**Existing IPs:** The following existent IP’s will be reviewed and revised (as needed) prior to use:

- IPLV-003, 08/05/04, Analytical & Top-Loading Balance Use

- IPR-018 Electrochemical Corrosion Testing
- IPR-020 User Calibration of Gamry Potentiostats PC4/300mA/DC105 and PC4/750mA/DC105
- IPR-025 User Calibration and Use of the Cole-Parmer/Oakton pH/mV/°C Meter

**New IPs:** The following new Implementation Procedures (IPs) are planned to be written and used:

- Alloy 22 specimen identification and machining (complementing QAP-8.0)
- Heat treatment of Alloy 22. This “Special Process” will be developed in compliance with QAP-9.0.
- Magnification calibration of optical microscope.
- Magnification calibration of scanning electron microscope.
- Quantitative analysis using EDS system on scanning electron microscope.
- Quantitative analysis using EDS system on transmission electron microscope.

## 7. Samples/Specimens

A survey of available Alloy-22 suppliers (ex: Haynes, etc.) will be conducted to determine the selection of compositions (within the UNS specifications) is available in the overall marketplace. Once specific mill runs (heats) are identified, plate stock will be procured. Rod stock may be procured if necessary. The heat numbers and batch numbers will be recorded for traceability. The plates will be sampled and tested by a laboratory on the Qualified Suppliers List in accordance with ASTM standards in order to be qualified for use in the UCCSN/DOE Cooperative Agreement. UNR has a plate of Alloy 22 that has been qualified for use under the UCCSN QA Program and this material will be used for the initial proposed work; new material will be acquired and qualified as well.

Specimens will be cut from the parent plates and uniquely identified to maintain traceability to orientation with respect to the rolling direction and location in the parent plate. Specimens will be prepared as follows. Additional specimen preparation steps or procedures may be necessary and when implemented, will be fully described in Scientific Notebooks and/or IPs.

1. Alloy 22 plate (or rod stock) will be cut into cylindrical cross section form to minimize edge/corner effects in electrochemical tests. Samples not used in electrochemical tests (i.e., heat treatment/microscopy only) may be machined into square/rectangle shapes if this significantly lowers machining costs.
2. Specimens that will be used in electrochemical measurements will be mounted in epoxy. Samples used exclusively for metallographic analysis may be mounted if necessary for polishing. A threaded hole on the reverse face of the samples will be machined to facilitate proper mounting in the Scanning Electron Microscope.
3. Specimens will be polished to a mirror finish with successively finer polishing media. Experience has found that the sequence: 240 grit Silica carbide paper, 400 grit SiC paper, 600 grit SiC paper, 1.0  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  suspension, 0.3  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  suspension and 0.05  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  suspension yields a suitable surface for microstructure analysis.
4. Specimens examined under the optical microscope may require etching. They will be etched using the best available etching method (to be determined in a scoping study).

- Measurements specifically designed to examine the grain boundary region will implement a backscatter detector with no etching (polished sample surface only) to preserve the grain boundary structure.
5. Examination of the microstructure using optical microscopy and Scanning Electron Microscopy will be performed. Micrographs will be made to record the structure. The Metallurgical and Materials Science Engineering Department at UNR has an Olympus metallographic optical microscope and a Hitachi SEM that will be used for this task.
  6. Specimens other than those for study of the mill-annealed and/or subsequent processing history will be solution heat treated in a tube furnace at 1200°C for two and one half hours. This heat treatment was determined experimentally at UNR using recommendations from Haynes International; Alloy 22 specimens with obvious precipitates were examined before and after this heat treatment, and this time and temperature treatment was found to be effective.
  7. After initial characterization and heat treatment, some specimens will be remounted, re-polished and examined to characterize the post-processing microstructure and/or conduct electrochemical experiments.

## **8. Equipment and Instrumentation**

The following equipment have been identified for likely use in this task.

- Gamry potentiostats: PC4/750mA/DC105/EIS300, PCi4/750mA/DC105/EIS300
- Thermocouple thermometers: Type J, K, and R
- Digital multimeter
- Calibrated electrical resistors
- pH meters: Cole-Parmer or Oakton
- Immersion heating baths with digital temperature controllers
- Weighing Scales / Balances
- Class A volumetric glassware: Volumetric flasks and pipettes
- Tube furnace with digital temperature controller
- Box furnace with digital temperature controller
- Data Logger
- Metallurgical sample preparation equipment
- Digital caliper
- Optical Microscope
- Scanning Electron Microscope / EDS
- Transmission Electron Microscope / EDX
- X-Ray Diffractometer
- Electron beam microprobe
- Hardness testing (macro)
- Microhardness testing

All M&TE will be controlled and calibrated according to QAP-12.0, "Control of Measuring and Test Equipment." All these are in-house equipment and are dedicated for UCCSN/DOE Cooperative Agreement tasks; they are not available for public use. Password protection will be placed on all computers and PC-based potentiostats. All computers to be used have password protection capability. Physical access to calibrated instruments and test materials will be restricted whenever possible by storage in locked cabinets, in locked offices, or locked laboratories where they are being used. Some instruments will be user calibrated by following applicable IP's. Some will be calibrated by qualified suppliers.

## 9. Software and Models

Experimental data will be plotted and analyzed using one or more of the following: Microsoft Excel, SigmaPlot for Windows, Origin, or Gamry Instruments Echem Analyst. SigmaPlot will be used for graphical/display purposes only and is exempt according to QAP 3.2 paragraph 2.1.2. Any user-written Excel macros will be verified by hand calculation and documented in the Scientific Notebook as prescribed in QAP 3.2 paragraph 2.1.5. Gamry Echem Analyst will also be used according to QAP 3.2 paragraph 2.1.5. Microsoft Office suite will be used for general computing needs. Operating systems to be used are Microsoft Windows 98, ME, XT, and XP. Qualified software will be obtained from Software Quality Management in accordance with QAP-3.2, "Software Management." Unqualified (and non-exempt) software including macros and other routines in Excel or other commercially available qualified software suites will be qualified in accordance with QAP-3.2.

Application Software for quantitative image analysis will be selected and used. It is considered exempt under QAP 3.2 paragraph 2.1.4, "*Software that is embedded in the equipment or integral to the operations, maintenance, or calibration of measuring and test equipment and has not been developed or modified by the User.*" The quantitative image analysis software is an enhancement to the microscopy instruments, whose accuracy is fundamentally tied to the magnification calibration. Therefore it will be controlled in accordance with QAP-12.0 and the magnification calibration IPs to be developed in this task.

Application Software for analysis of multicomponent diffusion kinetics (microstructural evolution) is freely available (software written by John Morral and distributed directly or with diffusion textbook written by M.E. Glicksman). It will be qualified in accordance with QAP-3.2.

## 10. Procurements and Subcontracts

Quality-affecting calibration items and services will be purchased from qualified suppliers in accordance with QAP-7.0, "Control of Procurement and Receipt." Metal specimens may also be procured from a qualified supplier. Metal specimens not purchased from a qualified supplier will be qualified by a testing laboratory on the Qualified Suppliers List or by use of a qualified standard or calibrated measuring and test equipment.

## 11. Hold Points

Hold Date	Purpose of Hold
10/1/2004	Prior to procurement of Alloy-22 material, finalize the specimen test plan (ex; identify available mill heats, etc.)
12/1/2004	Prior to specimen machining, finalize specimen geometry and machining process.
1/15/2005	Prior to microscopy, establish techniques for specimen preparation for microstructural analysis (grinding, polishing, etching, mounting, etc.)
8/1/2005	Internal assessment of the EPR test method's ability to reveal information characterizing localized corrosion behavior and grain boundary composition
12/1/2005	Review progress/results of the characterization of the as-provided material. Identify necessary actions for further progress or to complete study.
4/1/2007	Review progress/results of the characterization of the weld and base metal specimens. Identify necessary actions for further progress or to complete study.
12/1/2008	Review overall project status and progress to identify and prioritize remaining issues.
4/1/2008	Terminate data acquisition for final analysis
5/1/2008	Terminate data analysis for final report.

## 12. Quality Control—Accuracy, Precision, Error, and Uncertainty

In this task, the following objectives for accuracy and precision (and methods of measurement) will be applied.

### A. Quantitative Microscopy & Mechanical Properties

Applicable QAP's and IP's will be used to maintain data accuracy. Accuracy of measurements and data will be determined by comparison with known standards whenever possible. Whenever measurable or reported by image analysis software, statistical uncertainties for individual measurements will be recorded along with the reported measurement values. Acceptable levels of uncertainty/error will be determined with the assistance of published standards whenever possible. When not published, the level of acceptable uncertainty will be determined through a scoping test and implemented in the measurement procedures.

Potential sources of error include instrument calibration errors, incorrect specimen preparation (polishing, etching, etc.), specimen contamination, and human error. All of these will be minimized by IPs, personal qualification requirements, indoctrination, and training.

### B. Electrochemical Measurements

Applicable QAP's and IP's will be used to maintain data accuracy. Accuracy of measurements and data will be determined by comparison with known standards whenever possible. Accuracy of each complete electrochemical test apparatus (consisting of reaction flask, temperature control, gas and electrolyte flow control, counter electrode, and reference electrode) will be determined

using ASTM G5, “Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements” [2]. This method uses a standard ferric Type 430 stainless steel (UNS S43000) sample available from ASTM Headquarters. Measured current-potential curves should fall within the limits presented in the ASTM G5 method. Corrective action to the test apparatus will be taken if the system response is outside of the standard limits.

A minimum of three points in the conditions test matrices (Tables 1 – 4) will be tested in duplicate, and one point in triplicate, to determine precision. The conditions chosen for duplicate and triplicate testing will be determined during the course of experimentation based on equipment availability and time constraints. Comparisons within data sets and with similar data in the literature will demonstrate representativeness.

Uncertainty or scatter can be of the order of a factor of 2 in typical electrochemical data, such as passive corrosion current densities. This uncertainty is acceptable because these parameters will often change by several orders of magnitude as a function of time and metallurgical conditions. Uncertainties are presumably due to the stochastic nature of the metal surfaces and bulk solution properties. Potential sources of error include variation of flow condition of electrolyte, dissolved oxygen content, pH, temperature, surface condition of the working electrode from initial polishing and the effects of the test conditions, etc.; these factors will be controlled and maintained as closely as practical under laboratory conditions. Human error will be minimized by personal qualification requirements, indoctrination, and training. Instrumental error will be minimized by calibration of testing equipment and following the requirements of all applicable IP’s.

### **13. Data Recording, Reduction and Reporting**

#### **A. Subtask 1**

Data from this subtask will consist of

- 1) Specimen supplier and mill batch information
- 2) specimen sampling location and orientation
- 3) welding parameters (as appropriate)
- 4) heat treating parameters (as appropriate)
- 5) sample microscopy preparation parameters
- 6) specimen micrographs and attending instrument parameter data
- 7) microstructure quantification data (such as grain size, phase distributions and fractions, ordering, precipitate sizes, etc.)
- 8) composition data (average, point and profile, depending on needs)
- 9) reduced diffusion data
- 10) mechanical property data.

#### **B. Subtask 2**

Data from this subtask will consist of EPR current densities and potentials and the corresponding fixed test parameters, passive current densities at multiple potentials, passive current density transients resulting from step changes in potential, potentiostatic Electrochemical Impedance Spectroscopy (EIS) data, and current-voltage curves for hydrogen evolution/oxygen reduction.

Electronic data will be collected, reduced, and recorded according to QAP-3.1, "Control of Electronic Data." The data generated will be plotted and/or tabulated where appropriate. Data analysis and reduction will be performed using the above listed software packages (spreadsheet calculations, image processing, diffusion data reduction) as appropriate. Significant graphical results will be printed and pasted into or attached to the appropriate Scientific Notebooks (SNs). Numerical results will be recorded in the appropriated SN's. Data will be submitted to the UCCSN Technical Data Archive (TDA) in accordance with QAP-3.6, "Submittal of Data" through the UCCSN data coordinator.

Data that have not undergone technical review or are otherwise non-qualified, will be labeled "for scoping or corroborative use only," and traceability to their origin and nature will be maintained in documents in which the data are presented. Non-qualified data will be used for preliminary scoping purposes only and will not be used to reach any conclusions.

#### **14. Reviews and Verifications**

Technical reviews of any reports and publications containing quality-affecting data will be conducted in accordance with UCCSN QA Program requirements. Scientific notebooks and associated data are to be reviewed in accordance with QAP-3.0, "Scientific Investigation Control." Technical reports and other data are to be reviewed in accordance with QAP-3.4, "Technical Reports."

#### **15. Records and Submittals**

QA records generated as a result of the records sections of the implementing procedures and QA procedures and deliverables are controlled and transmitted to the Document Control Coordinator in accordance with QAP-17.0, "Quality Assurance Records." Technical reports will be written in accordance with QAP-3.4, "Technical Reports". Data will be submitted to the TDA in accordance with QAP-3.6, "Submittal of Data" through the UCCSN data coordinator. New IPs that are developed as part of this effort (see Section 6, above) may involve additional records (such as procedure checklists). It is anticipated that these records will be added to Scientific Notebooks as attachments. Technical reports will be the primary deliverable. Non-qualified reports will be submitted quarterly and the final report will be submitted at the end of the task.

#### **16. References**

Note: Square brackets, [], denote citations from within the text of these references.

1. Final Report: Waste Package Materials Performance Peer Review Panel, Submitted in response to request of USDOE, Bechtel SAIC Co.  
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2. SUMMERS, T.S.E., WALL, M.A., KUMAR, M., MATHEWS, S.J., and REBAK, R.B.  
Phase Stability and Mechanical Properties of C-22 Alloy Aged in Temperature Range 590 to 760 C for 16,000 Hours. in Scientific Basis for Nuclear Waste Management XXII, Materials Research Society Symposium Proceedings Vol. 556, Materials Research Society, 1999, 916-926.



3. SUMMERS, T.S.E., REBAK, R.B., PALMER, T.A., and CROOK, P. Influence of Thermal Aging on the Mechanical and Corrosion Properties of GTAW welds of Alloy N06022. in Scientific Basis for Nuclear Waste Management XXV, Materials Research Society Symposium Proceedings, Vol. 713, Materials Research Society, 2002, 45-52.
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13. FANG, Z., WU, Y.S., ZHANG, L., and LI, J.Q., Application of the Modified electrochemical Potentiodynamic Reactivation Method to Evaluate Intergranular Corrosion susceptibility of Stainless Steels. Corrosion, 1998. 54, p. 339.
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15. HODGE, F.G. and KIRCHNER, R.W., Corrosion, 1976. 32, p. 332.