Revision 1

APPENDIX T

MICROSTRUCTURAL AND COMPOSITIONAL VARIATIONS OF ALLOY 22 (RESPONSE TO PRE 7.03)

Note Regarding the Status of Supporting Technical Information

This document was prepared using the most current information available at the time of its development. This Technical Basis Document and its appendices providing Key Technical Issue Agreement responses that were prepared using preliminary or draft information reflect the status of the Yucca Mountain Project's scientific and design bases at the time of submittal. In some cases this involved the use of draft Analysis and Model Reports (AMRs) and other draft references whose contents may change with time. Information that evolves through subsequent revisions of the AMRs and other references will be reflected in the License Application (LA) as the approved analyses of record at the time of LA submittal. Consequently, the Project will not routinely update either this Technical Basis Document or its Key Technical Issue Agreement appendices to reflect changes in the supporting references prior to submittal of the LA.

APPENDIX T

MICROSTRUCTURAL AND COMPOSITIONAL VARIATIONS OF ALLOY 22 (RESPONSE TO PRE 7.03)

This appendix provides a response for Key Technical Issue (KTI) agreement Preclosure Safety (PRE) 7.03. This agreement relates to providing information to demonstrate that the allowed microstructural and compositional variations of Alloy 22 (UNS N06022) and the allowed compositional variations in the weld filler metals used in the fabrication of the waste packages do not result in unacceptable waste package mechanical properties.

T.1 KEY TECHNICAL ISSUE AGREEMENT

T.1.1 PRE 7.03

Agreement PRE 7.03 was reached during the U.S. Nuclear Regulatory Commission (NRC)/U.S. Department of Energy (DOE) Technical Exchange and Management Meeting on Preclosure Safety held July 24 through 26, 2001, in Las Vegas, Nevada (Reamer and Gil 2001). No submittal related to this KTI agreement has been made to the NRC.

The wording of the agreement is as follows:

PRE 7.03

Demonstrate that the allowed microstructural and compositional variations of alloy 22 base metal and the allowed compositional variations in the weld filler metals used in the fabrication of the waste packages do not result in unacceptable waste package mechanical properties. DOE will provide justification that the ASME code case for alloy 22 results in acceptable waste package mechanical properties considering allowed microstructural and compositional variations of alloy 22 base metal and the allowed compositional variations in the weld filler metals used in the fabrication of the waste packages. DOE agrees to provide the information in FY 2003 and document the information in the Waste Package Design Methodology Report.

T.1.2 Related Key Technical Issue Agreements

There are no related KTI agreements.

T.2 RELEVANCE TO REPOSITORY PERFORMANCE

The waste package is important to safety. The waste package is credited to prevent release of radionuclides, in terms of dose to workers and the public, during the preclosure and postclosure periods. Therefore, the waste package outer shell is designed to be sufficiently ductile so that, working in conjunction with the stainless steel inner shell, the waste package does not breach for design basis drops or impacts due to rock or equipment falls.

The waste package is evaluated using finite element analysis based on numerical simulations of waste package dynamic events during preclosure and postclosure, including, but not limited to, vertical and horizontal drops, slapdowns, drops onto objects, collisions, and drops of equipment or rocks onto the waste package. The same mechanical properties are used for the analysis of the preclosure and postclosure events. These mechanical properties are important because they are used in the numerical simulations of waste package dynamic events.

T.3 RESPONSE

A testing program was conducted to study the microstructural and compositional variations of Alloy 22 base metal and compositional variations of weld filler metal ERNiCrMo-14 (UNS N06686) to determine if the compositional variations affected the mechanical properties. Several compositional variations within the specifications of the American Society of Mechanical Engineers (ASME) code (ASME 2001) were produced for the study. ASME SB-575 (ASME 2001, Section II, Part B) specification was used for Alloy 22 base metal, and ASME SFA-5.14 (ASME 2001, Part C) specification was used for the weld filler metal ERNiCrMo-14, as shown in *Nickel*-*Based Alloy Weld Filler Material and Base Metal Composition Test Program* (Allegheny Technologies 2004). The results of this study satisfy the requirements of PRE 7.03.

T.3.1 Effects of Compositional Variations on Mechanical Properties

The mechanical properties of Alloy 22 are well known, and the impact in varying the chemistry and how the mechanical properties are affected by the variations are shown in this study. Normal melt practice when producing this material "aims" at the mid-composition range of elements. The artificial chemical proportions tested here (dividing the chemistry range over seven incremental segments) are unlikely to be produced in any commercial operation. Test results in this study show that iron content of the material enhances the propensity toward the production of topologically close-packed (TCP) structure that results in poor corrosion properties.

The results of this study suggest that restrictions on the element iron are warranted. The final value for iron content to be specified for Alloy 22 procurement remains under evaluation. DOE expects that the final Alloy 22 specification will limit iron to less than the 6% limit allowed by ASME SB-575 (ASME 2001). This will minimize TCP phase precipitation when the materials are welded. Melt processes currently used in industry deliberately are designed to reduce many of the trace elements, particularly phosphorus and sulfur, that have been identified as deleterious to hot work and to the mechanical properties of both the base metal and the weld filler material. The same approach may also be taken with the weld filler metal ERNiCrMo-14. High trace elements in the melt results in materials that cannot be drawn, the process used to make weld wire. Nonetheless, the fabrication specification and weld control process will specify the acceptable ranges of chemistry, as appropriate.

For Alloy 22, none of the experimental compositions appear to closely match commercial plate product. While the compositions in the study increase chromium and molybdenum together, commercial producers tend to balance chromium against molybdenum, with tungsten being almost constant. For example, if a materials supplier chooses a formulation of Alloy 22 that is high in chromium (21.60% to 21.82%), similar to the compositions for Chemistry E or Chemistry F (see Table T-1), that formulation will contain lower molybdenum (13.11% to 13.31%), similar to the compositions for Chemistry B or Chemistry C (see Table T-1). If materials suppliers choose a formulation that is lower in chromium (21.03% to 21.22%), similar to the composition for Chemistry C (see Table T-1), then that formulation will contain higher molybdenum (13.71% to 13.81%), similar to the compositions for Chemistry E or Chemistry F. For the major alloying elements (chromium, molybdenum, and tungsten), the composition (21.22% to 21.31% chromium, 13.51% to 13.60% molybdenum, and 2.99% to 3.00% tungsten) for Chemistry D (see Table T-1) comes closest to typical commercial product. However, the composition for Chemistry D has high residuals (particularly cobalt, manganese, and vanadium) and, thus, is substantially different from typical commercial product (Allegheny Technologies 2004, Section 5.7.2).

Ele-		Chemistry A	Chemistry B			Chemistry C	Chemistry D		Chemistry E		Chemistry F		Chemistry G	
lment	Heat HC76	Heat HC77	Heat HC78	Heat HC79	Heat HC80	Heat HC81	Heat HD16	Heat HD17	Heat HC82	Heat HC83	Heat HC84	Heat HC85	Heat HC86	Heat HC87
lCr	20.31	20.22	20.78	20.81	21.22	21.03	21.22	21.31	21.60	21.60	21.73	21.82	22.36	22.39
Mo	12.71	12.63	13.28	13.31	13.11	13.13	13.51	13.60	13.73	13.71	13.81	13.77	14.23	14.23
Fe	2.51	2.5	3.00	3.02	3.99	3.98	3.02	2.98	4.97	5.07	2.98	3.04	5.74	5.78
W	2.64	2.66	3.00	3.01	3.01	2.98	3.00	2.99	3.00	2.99	2.99	2.97	3.39	3.36
Co	< 0.01	0.15	< 0.01	< 0.01	< 0.01	< 0.01	2.25	2.22	< 0.01	< 0.01	< 0.01	0.03	< 0.01	< 0.01
C	0.003	0.004	0.005	0.004	0.006	0.005	0.006	0.005	0.008	0.010	0.002	0.014	0.006	0.007
Si	0.02	0.03	0.03	0.03	0.03	0.02	0.08	0.06	0.04	0.04	0.04	0.05	0.05	0.05
Mn	< 0.01	0.02	0.02	0.02	0.01	0.01	0.40	0.40	0.03	0.04	0.02	0.02	0.02	0.04
V	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	0.25	0.25	0.01	0.01	0.01	0.01	0.01	0.01
P	0.003	0.003	0.004	0.004	0.004	< 0.003	< 0.003	< 0.003	0.006	0.006	0.005	0.005	0.006	0.006
S	< 0.0003	< 0.0003	< 0.0003	< 0.0003	< 0.0003	< 0.0003	< 0.0003	< 0.0003	< 0.0006	< 0.0003	< 0.0003	< 0.0003	< 0.0003	< 0.003
l Ni	61.6	61.57	59.67	59.59	58.39	58.64	56.03	56.00	56.37	56.33	58.24	58.05	53.95	53.90

Table T-1. Summary of Average Analyses of Alloy 22 Base Metal

Source: Allegheny Technologies 2004, Table 2-4.

NOTE: Values are based on Certified Material Test Reports.

T.3.2 Effects of Microstructural Variations on Mechanical Properties

ASME SB-575 (ASME 2001) specification indicates that the grain size requirement for sheet and strip shall be ASTM micrograin size number 1.5 or finer for plate thickness over 0.125 in. (ASME 2001, ASME SB-575, Table 5). The Alloy 22 base metal plate thickness is 1 in. (Allegheny Technologies 2004, Section 3.3).

Metallographic observation of the bead-on-plate welds showed the expected structures. The base metal showed an austenitic structure with annealing twins. The grain shape and size range for Chemistries A, B, C, D, E, and F are similar and are in the range of ASTM micrograin size numbers 4 to 6. The grain shape of Chemistry G is fairly equiaxed, but much smaller in size than the other chemistries. Grain size for Chemistry G is in the range of ASTM micrograin size numbers 10 to 12. Some minor grain growth was observable in the heat-affected zone near the fusion boundary. The autogenous weld showed a columnar dendritic structure. At higher magnification, a small amount of apparent interdendritic intermetallic phase was seen (Allegheny Technologies 2004, Section 4.2).

T.3.3 Summary

This appendix summarizes work completed to further evaluate the nickel-based alloy materials specified for the waste package outer corrosion barrier. None of the materials were normal material output because the compositions of chromium and molybdenum were segregated into segments over the range of each element. In this case, for each element set where a range was stipulated, the range was segregated into seven incremental values. Where a single value is listed in the requirements, the value is a maximum for that element and any lower value is acceptable. The stipulated chemical values for the samples were successfully achieved. Since the chemistries of Alloy 22 produce a variety of mechanical properties and microstructures, it is reasonable to assume that materials procured in the future with ranges based on this study would provide acceptable properties. The mechanical properties were reasonably within expectations for this material with these grain sizes, and the microstructures were typically equiaxial, with a size variation normal for the material.

The study showed that it is possible to produce base metals and weldments that exhibit the desired mechanical behavior using Alloy 22 base metal and ERNiCrMo-14 weld filler metal comparable to that produced commercially (Allegheny Technologies 2004, Section 5.7.1). For example, the heats of material containing the highest possible amount of impurities of the residual elements are unlikely in a commercial product. The two heats that did not meet the ASME SB-575 (ASME 2001) specification for mechanical property requirements (Chemistry G for the base metal) do not represent typical commercial products.

The information in this report is responsive to agreement PRE 7.03 made between the DOE and NRC. The report contains the information that DOE considers necessary for NRC review for closure of this agreement.

T.4 BASIS FOR THE RESPONSE

A testing program was conducted to study the microstructural and compositional variations of Alloy 22 base metal and compositional variations of weld filler metal ERNiCrMo-14. Several compositional variations within the specifications of the ASME Code were fabricated for the study. ASME SB-575 (ASME 2001, Section II, Part B) specification was used for Alloy 22 base metal and ASME SFA-5.14 (ASME 2001, Part C), was used for the weld filler metal ERNiCrMo-14 (Allegheny Technologies 2004). The results of this study satisfy the requirements of PRE 7.03.

T.4.1 Scope of Testing Program

The study produced seven sets of chemistries, segregated into roughly equal increments, for the compositional ranges provided in the applicable ASME specifications for both the base metal and the weld filler metal. The object was to generate seven incrementally higher sets of alloy compositions (chemistries) to study the effect of compositional variations on observable mechanical properties of the materials concerned.

The compositions of the materials met the following applicable requirements (Allegheny Technologies 2004, Section 1.2):

- 1. Base metal composition meeting the general requirements of AMSE SB-575 (ASME 2001) Alloy 22 (see Table T-2).
- 2. Weld filler material ERNiCrMo-14 meeting the requirements called out in ASME SFA-5.14 (ASME 2001) specification (see Table T-3).

Table T-2. Chemical Composition Limits for Alloy 22

Source: ASME 2001, ASME SB-575, Table 1.

	ERNiCrMo-14 Composition Limit
Element	(%)
Carbon	0.01 , max
Manganese	1.0 , max
Iron	5.0, max
Phosphorus	0.02, max
Sulfur	0.02, max
Silicon	0.08 , max
Copper	0.5 , max
Nickel	Remainder
Cobalt	
Aluminum	0.5 , max
Titanium	0.25, max
Chromium	19.0 to 23.0
Niobium plus Tantalum	
Molybdenum	15.0 to 17.0
Vanadium	
Tungsten	3.0 to 4.4
Other Elements, Total	0.50 , max

Table T-3. Chemical Requirements for ERNiCrMo-14

Source: ASME 2001, ASME SFA-5.14, Table 1.

The results of the Certified Material Test Reports for the base metal and the weld filler metal are shown in Tables T-1 and T-4, respectively. The specimen sets that were produced are shown in Table T-5.

A total of 98 weldments were manufactured using the base metal and weld filler metal combinations detailed in Table T-5. Table T-5 contains 49 possible material combinations, which were doubled to allow the production of one-half of the test specimens in a solution-annealed condition. Subsamples were also prepared and test specimens were machined to facilitate the following testing study (Allegheny Technologies 2004, Section 1.2):

- 1. Tensile testing of 980 specimens (10 specimens per weldment) was completed on 0.505-in. (12.8-mm) specimens, as detailed in ASTM/ASME SA-370 (ASME 2001, Figure 4).
- 2. Charpy impact testing on 1,960 subsized specimens (20 specimens per weldment) was completed in accordance with ASTM/ASME SA-370 (ASME 2001, Figure 11).
- 3. A total of 490 metallographic mounts were prepared and evaluated.

	Chemistry	Chemistry 2	Chemistry З	Chemistry 4	Chemistry 5	Chemistry 6	Chemistry
Element	Heat HC70	Heat HC71	Heat HC72	Heat HD-15	Heat HC73	Heat HC74	Heat HC75
Chromium	19.34	19.79	20.50	20.59	21.58	22.29	22.86
Molybdenum	15.10	15.75	16.25	16.27	16.25	16.28	16.82
Tungsten	3.16	3.47	3.74	3.82	3.79	4.04	4.33
Iron	< 0.02	0.42	0.39	4.03	0.28	0.35	0.14
Carbon	0.004	0.005	0.002	0.005	0.001	0.001	0.002
Silicon	0.02	0.02	0.03	0.075	0.02	0.03	0.03
Manganese	< 0.01	< 0.01	< 0.01	0.89	< 0.01	< 0.01	< 0.01
Cobalt	< 0.01	< 0.01	< 0.01	0.02	< 0.01	< 0.01	< 0.01
Vanadium	< 0.01	< 0.01	< 0.01	0.105	< 0.01	< 0.01	< 0.01
Phosphorus	< 0.003	0.006	0.007	0.003	0.008	0.008	0.010
Sulfur	< 0.0003	< 0.0003	< 0.0003	0.0003	< 0.0003	< 0.0003	< 0.0003
Copper	< 0.01	0.01	0.01	0.42	0.01	0.01	0.01
Nickel	61.94	60.35	58.84	53.51	57.84	56.79	55.59

Table T-4. Summary of Average Analyses of ERNiCrMo-14 Weld Filler Material

Source: Allegheny Technologies 2004, Table 2-6.

NOTE: Values are based on Certified Material Test Reports.

Table T-5. Test Matrix

Test Set 1	Test Set 2	Test Set 3	Test Set 4	Test Set 5	Test Set 6	Test Set 7
1 & A	1 & B	1 & C	1 & D	1 & E	1 & F	1 & G
2 & A	2 & B	2 & C	2 & D	2 & E	2 & F	2 & G
3 & A	3 & B	3 & C	3 & D	3 & E	3 & F	3 & G
4 & A	4 & B	4 & C	4 & D	4 & E	4 & F	4 & G
5 & A	5 & B	5 & C	5 & D	5 & E	5 & F	5 & G
6 & A	6 & B	6 & C	6 & D	6 & E	6 & F	6 & G
7 & A	7 & B	7 & C	7 & D	7 & E	7 & F	7 & G

NOTE: The set of specimens was produced using the base materials of Table T-1 and the weld filler metals of Table T-4.

T.4.2 Material Manufacturing

T.4.2.1 Alloy 22 Base Metal Production

Melting for bar material production was completed through vacuum induction melting-electroslag remelting (VIM-ESR) using commercial facilities, which is a normal melting practice for Alloy 22 and similar alloys. The VIM-ESR ingots were forged to reroll billets using a rotary forge press. Reroll billets were rolled to flat bars. The bars were cut to length, inspected, and certified to ASME SB-575, N06022 (ASME 2001). Product identity and ingot location was maintained from the melt and during each step of the manufacturing process to maintain the identification and traceability of bar materials manufactured for the study (Allegheny Technologies 2004, Section 2.1.1).

T.4.2.2 ERNiCrMo-14 Weld Filler Material Production

The objective was to produce 0.0625 in. diameter ERNiCrMo-14 weld filler metal in accordance with ASME SFA-5.14 (ASME 2001) specification. A production method was chosen to allow use of commercial processing equipment (Allegheny Technologies 2004, Section 2.1.2).

As with the base metal products, melting was completed through VIM-ESR using commercial facilities. The VIM-ESR ingots were forged to reroll billets using a rotary forge. Reroll billets were rolled to 0.219-in. diameter redraw coil. The redraw coil was inspected, shaved, and drawn to weld wire that was certified to ASME SFA-5.14, ERNiCrMo-14 (UNS N06686) (ASME 2001). A system was employed during each step of the manufacturing process to maintain the identification and traceability of weld wire materials manufactured for this project. Each spool of wire was uniquely identified to ensure that it is traceable back to the product heat from which it was manufactured (Allegheny Technologies 2004, Section 2.1.2).

T.4.3 Material Properties of Base Metal

Table T-6 compares the mechanical properties of the test Alloy 22 base metal to those of the ASME SB-575 (ASME 2001) specification.

Heat Number	Chemistry Set	Yield Strength (ksi)	Ultimate Tensile Elongation in Strength (ksi) 2 in. (%)		Reduction of Area
HC76	A	45.6	111.2	73.5	79.4
HC77	Α	44.9	112.0	73.3	79.3
HC77	A	46.6	114.0	72.5	78.5
HC77	A	45.0	111.0	75.2	81.1
HC78	B	46.3	110.6	74.6	80.1
HC79	$\sf B$	47.7	113.1	72.8	79.9
HC80	C	45.4	110.3	71.9	79.5
HC81	C	45.4	110.7	72.8	80.1
HD16	D	47.3	112.3	74.5	81.0
HD17	D	46.6	111.2	76.7	82.2
HC82	E	50.5	121.9	58.5	63.5
HC83	E	51.6	122.2	57.6	61.2
HC84	F	48.6	114.2	70.7	75.6
HC85	F	52.9	122.4	64.2	65.8
HC86	G	64.5	136.2	41	39.3
HC86	G	63.7	135.6	40	35.9
HC86	G	64.4	136.8	40.8	39.5
HC87	G	63.8	135.3	39.4	32.0
HC87	G	62.6	133.3	36.9	32.6
HC87	G	63.1	134.5	33.7	30.1
ASME SB-575 Alloy 22 (ASME 2001, Table 4) (minimum values)		45	100	45	Not Specified

Table T-6. Comparison of Mechanical Properties of Experimental Heats of Alloy 22 Base Metal

Source: Allegheny Technologies 2004, Table 2-5.

NOTE: Base materials are indicated by a letter.

Although Alloy 22 base metal Chemistry G meets the requirements specified in ASME SB-575 (ASME 2001) as listed in Table T-2, it does not meet the mechanical properties required by ASME SB-575 (ASME 2001), as listed in Table T-6.

Bar heats for Chemistry G, heat HC86 and HC87 materials showed high strength and low elongation. Examination of the as-received bars shows that the grain size (microstructure) of these heats is much finer than that of the other heats. Also, the grains seem to be outlined by fine second-phase particles. For this very highly alloyed composition, it appears that the standard commercial anneal that was applied (2,050°F for 30 minutes) was not sufficient to obtain the desired effects. A higher anneal temperature might have produced a softer, more ductile product with a reduced second phase fraction and larger grain size (Allegheny Technologies 2004, Section 2.2).

Regarding the commercial application of the base metal and weld filler metal chemical compositions studied, material with high residual levels of phosphorus and sulfur have poor workability and would be expected to be uneconomic or impossible to manufacture because of significantly reduced product yields due to cracking (Allegheny Technologies 2004, Section 2.2).

On the other end of the spectrum, those materials with ultra-low levels of residual elements would also be uneconomic to manufacture because, in order to attain these low levels, they would be restricted to vacuum induction melting, and the melt charge would probably include 100% carefully selected virgin raw materials. It would probably not include any revert (Allegheny Technologies 2004, Section 2.2).

The electric-arc furnace/argon-oxygen decarbonization process appears to be ideally suited as the primary melt step for the economical manufacture of large quantities of plate because it is designed to minimize the trace elements that cause degradation in mechanical and metal working processes (Allegheny Technologies 2004, Section 2.2).

T.4.4 Fabrication of Weldments and Test Specimens

T.4.4.1 Material Selection

Incoming bar material was visually and dimensionally inspected for conformance to the order requirements. The statement of work required that these bars be inspected by ultrasonic examination before welding. This inspection was done as a C-scan with a reference defect standard of 0.039-in. (1 mm). Bars that passed the ultrasonic test examination were then released for weld joint preparation machining (Allegheny Technologies 2004, Section 3.1).

T.4.4.2 Bead-on-Plate Welding

One bead-on-plate weld per base metal composition was made using autogenous gas tungsten arc welding (Allegheny Technologies 2004, Section 3.2).

T.4.4.3 Fabrication of Weldments

The welding process used in this study was the cold-wire gas tungsten arc process, which is the same process that will be used to close waste packages after they are filled with waste. The 1-in. nominal thickness bar was machined to produce a double-U groove. This welding procedure can be summarized as follows (Allegheny Technologies 2004, Section 3.3):

- The root of the weld was joined using two partial-penetration autogenous passes, one from each side. Each pass produced about 75% penetration of the land, for a total of about 150% penetration. Autogenous root fusion was chosen to simulate the process to be used at the repository and the fabrication shop for corrosion-resistant alloys (Allegheny Technologies 2004, Section 3.3).
- Subsequently, three passes per side were made using cold-wire feed gas tungsten arc welding. The plate was cooled (using forced air) to below 200°F, and manually wire brushed to remove any surface oxidation before each subsequent weld pass was started. Many welds were ground between passes to further remove surface oxides (Allegheny Technologies 2004, Section 3.3).

Two weldments were produced for each of the 49 combinations of seven base metal chemistries and seven filler metal combinations. Each weldment was given a unique identification number. Table T-7 relates the weldment numbers to the base metal and weld filler metal compositions (Allegheny Technologies 2004, Section 3.3).

Weld Filler Base Metal	1	$\mathbf{2}$	3	4	5	6	
A	4, 5	14, 15	64, 65	84, 85	42, 43	50, 51	30, 31
В	6, 7	16, 17	66, 67	82, 83	44, 45	56, 57	32, 33
C	10, 11	120, 21	168, 69	92, 93	148, 49	60, 61	36, 37
D	94, 95	96, 97	80, 81	86, 87	78, 79	74, 75	177, 176
Е	8, 9	18, 19	70, 71	190, 91	46, 47	58, 59	34, 135
F	2, 3	12, 13	72, 73	88, 89	38, 39	54, 55	26, 127
G	24, 25	122, 23	63, 162	98, 99	40, 41	52, 53	28, 29

Table T-7. Weldment Numbers—Base Metal Reference Number

Source: Allegheny Technologies 2004, Table 3-1.

NOTE: Base metals are indicated by a letter and weld wires by a number. For example, the weld numbers for combination D3 are 80 and 81.

T.4.4.3.1 Inspection of Weldments

As-welded bars were ultrasonically examined immediately after welding and cooling. This examination was performed using 45° angle beams, supplemented with additional angles as required, with a reference defect standard of 0.039 in. (1 mm). The ultrasonic testing calibration was performed using a 0.039-in. (1-mm) reference standard. This is the same calibration standard used in *Weld Flaw Evaluation and Nondestructive Examination Process Comparison Results for High-Level Radioactive Waste Package Manufacturing Program* (Smith 2003), where the ultrasonic testing process was shown to be capable of locating a flaw smaller than 1.0 mm. Flaws in that study were oriented parallel to the hoop stresses. For Alloy 22, a flaw of 1 mm would not be reportable using ASME acceptance criteria. Bars that passed the ultrasonic test examination were then released for heat treatment (if required) or test specimen machining (Allegheny Technologies 2004, Section 3.3.1).

T.4.4.3.2 Heat-Treatment of Weldments

One of each pair of weldments was solution-annealed in air at 2.075° F nominal (\pm 25°F) for 1 hour and rapidly quenched. For the vast majority of the weldments, heat-treating was completed in a batch mode in a large commercial plate-annealing furnace with spray quench capability. Table T-8 shows the identities of the heat-treated weldments (Allegheny Technologies 2004, Section 3.3.2).

Weld Filler Base Metal		$\mathbf{2}$	3	4	5	6	
A	5	15	65	85	43	51	31
B		16	67	83	45	57	33
C	11	21	69	93	49	61	37
D	95	97	81	87	79	75	177
Е	9	19	71	91	47	59	135
F	3	13	73	89	39	55	127
G	25	23	63	99	41	53	29

Table T-8. Identities of Heat-Treated Weldments

Source: Allegheny Technologies 2004, Table 3-2. Base metals are indicated by a letter and weld wires by a number. Here, the identity of heat treated weldments for combination D3 would be 81.

A representative number of plates per load were instrumented with embedded thermocouples and the heating and quenching cycles were recorded. All specimens showed conformance to the heating requirement (Allegheny Technologies 2004, Section 3.3.2).

T.4.4.3.3 Filler Metal Weldments

The welding procedure selected for this investigation (two autogenous passes and six filler metal passes to join 1-in.-thick nickel alloy material) is somewhat aggressive, especially compared to laboratory-type practices. This procedure was chosen deliberately as outlined below (Allegheny Technologies 2004, Section 3.3.3):

- Simulation of probable fabrication parameters. The economics of large-scale manufacture will drive fabrication processes toward the highest productivity possible.
- Identification of weldability problems that might exist. Using a greater number of smaller weld passes might mask such problems, especially hot cracking.

T.4.4.3.4 Fabrication of Test Specimens and Mechanical Testing

Half-inch diameter tensile test specimens were produced from the welded plates and tested per ASME SA-370 (ASME 2001). These specimens were transverse to the weld, so base metal, heat-effected zone, and weld filler were all equally stressed. Ten specimens per weldment were

tested. The test specimen was centered on the plate thickness and the weld was centered within the gauge length, as shown in Figure T-1 (Allegheny Technologies 2004, Section 3.3.4).

Source: Allegheny Technologies 2004, Figure 3-1.

Half-size Charpy impact specimens were produced and tested per ASME SA-370 (ASME 2001). Half-size specimens were used because Alloy 22 base metal is so tough that it was observed to stop the hammer of normal 300 ft-lb capacity machines. Whenever this happens, or whenever a specimen absorbs energy greater than 80% of the machine capacity, ASTM E 23-02a, *Standard Test Methods for Notched Bar Impact Testing of Metallic Materials,* requires that the machine be removed from service, inspected, repaired if necessary, recalibrated, and recertified before being returned to use (Allegheny Technologies 2004, Section 3.3.4). NIST does not certify Charpy machines larger than 300 ft-lb in capacity.

Tests were performed at room temperature. Ten specimens per weldment were tested with the notch located near the centerline of the weld metal fusion zone, as shown in Figure T-2 (Allegheny Technologies 2004, Section 3.3.4).

Source: Allegheny Technologies 2004, Figure 3-2.

Figure T-2. Charpy Test Specimen: Notch near Weld Metal Fusion Zone Centerline

Ten additional specimens per weldment were tested with the notch root located at the fusion boundary, as shown in Figure T-3 (Allegheny Technologies 2004, Section 3.3.4).

Source: Allegheny Technologies 2004, Figure 3-3.

Figure T-3. Charpy Test Specimen: Notch Root at Fusion Boundary

These specimens were polished and macroetched to facilitate locating the notch root at the fusion boundary, as shown in Figure T-4 (Allegheny Technologies 2004, Section 3.3.4).

Source: Allegheny Technologies 2004, Figure 3-4.

T.4.4.3.5 Metallography

Metallographic preparation and examination of the bead-on-plate and weldments were performed. ASME SB-575 (ASME 2001) specification indicates that the grain size requirement for sheet and strip shall be ASTM micrograin size number 1.5 or finer for plate thickness over 0.125 in. (ASME 2001, ASME SB-575, Table 5). The Alloy 22 base metal plate thickness is 1 in. (Allegheny Technologies 2004, Section 3.3). The ASTM E 112-96, *Standard Test Methods for Determining Average Grain Size*, comparison method was used to evaluate the grain size of the as-received bars (Allegheny Technologies 2004, Section 3.3.5).

T.4.4.4 Test Results

T.4.4.4.1 Base Materials

The high-purity, low alloy content composition for Chemistry A material showed low strength (see Table T-6). When the standard commercial anneal (2,050°F for 30 minutes) was applied, it satisfied the specification mechanical property requirements. The higher annealing temperature provided by the postweld heat treatment softened the material further so that it did not satisfy the specification yield strength requirement (Allegheny Technologies 2004, Section 4.1.1).

Chemistry G base metal was stronger and less ductile, showed a much finer grain size than the other heats, and contained a second phase presumed to be TCP. The 2,050°F anneal applied to the bars was insufficient to dissolve the second phase. Metallographic examination of the postweld heat-treated bars showed that 2,075°F annealing was also insufficient to dissolve the second phase. Autogenous welds on this material showed a greater amount of apparent interdendritic intermetallic phase than seen in the lower alloy heats (Allegheny Technologies 2004, Section 4.1.1).

The high residual heat of Chemistry D base metal exhibited a somewhat increased tendency toward TCP phase precipitation. Since the differences between the residual levels of Chemistry D and materials of Chemistries B, C, E, and F, which are meant to represent typical commercial residual levels, are not great, any tightening of the specification restrictions on these elements would have to be done very cautiously (Allegheny Technologies 2004, Section 4.1.1).

T.4.4.4.2 Bead-on-Plate Welds

Metallographic observation of the bead-on-plate welds mostly showed the expected structures. The base metal showed an austenitic structure with annealing twins. The grain shape and size range of chemistries for Chemistries A, B, C, D, E, and F are similar and are in the range of ASTM micrograin size numbers 4 to 6. The grain shape of Chemistry G is fairly equiaxed but much smaller in size than the other chemistries. Grain size for Chemistry G was in the range of ASTM micrograin size numbers 10 to 12. Some minor grain growth was observable in the heat-affected zone near the fusion boundary. The autogenous weld showed a columnar dendritic structure. At higher magnification, a small amount of apparent interdendritic intermetallic phase was seen (Allegheny Technologies 2004, Section 4.2).

The bead-on-plate welds of the higher residual Chemistry D material, which is otherwise similar to Chemistry C, showed a greater amount of apparent interdendritic intermetallic phase (Allegheny Technologies 2004, Section 4.2).

The somewhat higher alloyed materials for Chemistries E and F base metals began to exhibit a noticeable quantity of second phase, distributed as irregular rafts. These particles did not appear to change the grain size as compared with the other, lower-alloyed heats. The high alloy Chemistry G base metal showed a much finer grain size than the other heats. Bead-on-plate welds of the higher alloyed Chemistries E and G material showed an amount of apparent interdendritic intermetallic phase that was greater than seen in the lower alloy heats and about equivalent to what was seen in the high residual Chemistry D (Allegheny Technologies 2004, Section 4.2).

T.4.4.5 Filler Metal Weldment Examination

Optical metallography of the filler metal weldments showed features consistent with those seen in the bead-on-plate welds. Typically, a small amount of isolated intermetallic phase was seen in the weld deposit (Allegheny Technologies 2004, Section 4.3.2).

Heat treatment of such welds appeared to provide some homogenization, as revealed by the disappearance of the dendritic structure. It also caused recrystallization, as evidenced by the appearance of grain boundaries within the weld, but did not dissolve the second phase particles (Allegheny Technologies 2004, Section 4.3.2).

The high residual content weld filler metal Set 4 produced somewhat more second phase within the weld, but the levels varied from location to location within a given weld and even within a given pass within that weld (Allegheny Technologies 2004, Section 4.3.2).

Combining the high residual content weld filler metal Chemistry Set 4 or the highly alloyed weld filler metal Chemistry Set 7 with the high residual content base metal Chemistry D or the highly alloyed base metal Chemistry G increased the amount of second phase. However, the increase did not appear to be large enough to be especially notable, given the general variability observed (Allegheny Technologies 2004, Section 4.3.2).

Heat treatment did not decrease the amount of second phase but rather increased it in these high alloy or high residual combinations or both (Allegheny Technologies 2004, Section 4.3.2).

Optical metallography was supplemented by scanning electron microscopy to clarify some of the features noted. Weldment number 99 (heat-treated Chemistry G with Set 4 weld filler metal) was examined using both secondary electron and qualitative energy dispersive spectroscopy modes. Particles seen in optical metallography were bright in secondary electron mode, indicating probable enrichment in heavy elements. Energy dispersive spectroscopy showed these to be highly enriched in molybdenum and tungsten and slightly enriched in chromium relative to the surrounding matrix. This supports the assumption that the particles are TCP-type intermetallics. Evidently, the 2,075°F annealing heat treatment was insufficient to dissolve the intermetallic phases in these highly alloyed compositions (Allegheny Technologies 2004, Section 4.3.2).

T.4.4.6 Tension Testing

Summaries of the tensile test results are shown in Tables T-9 to T-12.

Review of the scatter in the data revealed that the difference in mechanical properties of base metal Chemistry G was a major contributor to the standard deviations for the various filler wires (Allegheny Technologies 2004, Section 4.4).

Test specimens exhibiting low tensile strength failed in the weld. These fracture surfaces frequently exhibited features that were assumed to be small weld defects. Test specimens exhibiting low yield strength always fractured in the base metal (Allegheny Technologies 2004, Section 5.2).

Elongation and reduction of area were measured. While elongation is a specified property in the ASME SB-575 (ASME 2001) specification for Alloy 22 base metal, there is no elongation requirement in the ASME SFA-5.14 (ASME 2001) specification for ERNiCrMo-14 weld filler metal. Also, the composite nature of the welded specimen, with zones of different strength characteristics, makes any attempt to impose the base metal requirement upon the welded material unreasonable. Reduction of area is not a specified property for ASME SB-575 (ASME 2001) specification for Alloy 22 base metal or for the ASME SFA-5.14 (ASME 2001) specification for ERNiCrMo-14 weld filler metal; however, ASME specifications are based on ASTM specifications, which do include reduction of area (Allegheny Technologies 2004, Section 5.2). Reduction of area is an indication of the ductility of the material.

Table T-9. Summary of Tensile Strength Results

Source: Allegheny Technologies 2004, Table 4-2.

NOTE: HT = heat-treated; AW = as-welded; UTS = ultimate tensile strength.

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Table T-10. Summary of Yield Strength Results

Source: Allegheny Technologies 2004, Table 4-3.

NOTE: HT = heat-treated; AW = as-welded.

Table T-11. Summary of Tensile Elongation Results

Source: Allegheny Technologies 2004, Table 4-4.

NOTE: HT = heat-treated; AW = as-welded.

Table T-12. Summary of Tensile Reduction of Area Results

Source: Allegheny Technologies 2004, Table 4-5.

NOTE: HT = heat-treated; AW = as-welded.

Review of the tensile test data (Allegheny Technologies 2004, Section 5.2) shows that:

- Yield strength is controlled by the base metal and heat treatment; it is not significantly influenced by weld filler metal composition.
- Tensile strength is influenced by base metal composition, weld filler metal composition, and heat treatment. These parameters interact in a complex fashion and interaction effects appear to be significant. As a particular example, the tensile strength of heat-treated weldments of base metal Chemistry D is reduced when weld filler metal Sets 4 or 7 are used (Allegheny Technologies 2004, Section 5.2).
- Elongation is influenced by base metal composition, weld filler metal composition, and heat treatment. Elongation is improved by heat treatment for most conditions, with the exception of weld filler metal Sets 4 and 7, for which heat treatment reduces tensile elongation. As seen for tensile strength, the elongation of heat-treated weldments of base metal Chemistry D is reduced when weld filler metal Sets 4 or 7 are used (Allegheny Technologies 2004, Section 5.2).
- Reduction of area is influenced by base metal composition, weld filler metal composition, and heat treatment. The location of the fracture (base metal or weld deposit) is particularly important. This interaction is especially complex. Reduction of area is improved by heat treatment for weld filler metal Sets 1, 2, 3, 5, and 6, but reduced for weld filler metal Sets 4 and 7 (Allegheny Technologies 2004, Section 5.2).

T.4.4.7 Charpy Testing

Charpy impact testing is used to measure resistance to low-energy brittle fracture. Resistance is measured as energy absorbed by the specimen deformation and fracture. For carbon and low alloy steels, energies above about 20 ft-lb are generally accepted as indicating toughness at the test temperature when full size specimens are used. The fully austenitic materials of this investigation would be expected to exhibit higher energies, but since half-size specimens were used, the energy will be correspondingly reduced by about half (Allegheny Technologies 2004, Section 5.3).

A 7-by-7 matrix, equally proportioned (the chemistry of the base metal and weld metal), was produced for this study. Ten Charpy impact specimens were produced for each set within the matrix. As-welded specimens and as-welded and solution heat treated specimens were produced. The two sets produced 980 total specimens; one of which was not tested due to preliminary tensile results that indicated it to be very brittle. Out of 979 tests, 817 provided valid lateral expansion values. In 162 tests, the test specimens failed to separate after testing, and, as a result, valid lateral expansions could not be obtained. In three of the 98 data sets (base metal, weld filler metal, heat treatment combinations), lateral expansion values were not obtained because the bars did not fracture completely. The failure to obtain valid lateral expansion values for these tests should be regarded as a demonstration of high toughness of the materials involved (Allegheny Technologies 2004, Section 5.3).

Summaries of the Charpy test results are shown in Tables T-13 to T-16.

Table T-13. Summary of Charpy Impact Absorbed Energy Results, Half-Size Weld Centerline Specimens

Source: Allegheny Technologies 2004, Table 4-6.

NOTE: 1 ft-lb = 1.36 J.

Table T-14. Summary of Charpy Impact Absorbed Energy Results, Half-Size Weld Fusion Boundary Specimens

Source: Allegheny Technologies 2004, Table 4-7.

NOTE: HT = heat-treated; AW = as-welded; 1 ft-lb = 1.36 J.

Table T-15. Summary of Charpy Impact Lateral Expansion Results, Half-Size Weld Centerline Specimens

Source: Allegheny Technologies 2004, Table 4-8.

NOTE: HT = heat-treated; AW = as-welded; 1 mil = 0.001 in. = 0.0254 mm. NA = Not available because specimens did not break.

Table T-16. Summary of Charpy Impact Lateral Expansion Results, Half-Size Weld Fusion Boundary Specimens

Source: Allegheny Technologies 2004, Table 4-9.

NOTE: HT = heat-treated; AW = as-welded; 1 mil = 0.001 -in. = 0.0254 mm. NA = Not available because specimens did not break.

The range of observed impact energies, 5 to 151 ft-lb, shows that the variables of this investigation (base metal chemistry, weld filler metal chemistry, heat treatment, and notch location) have significant effects. The range of observed lateral expansions, 5 to 69 mils (0.005 to 0.069 in.), support this conclusion (Allegheny Technologies 2004, Section 5.3).

Since the fusion boundary tests do not involve a significant volume of weld filler metal, weld filler metal chemistry would be expected to exert little influence on fusion boundary impact energy. This expected result is what was observed. In both the as-welded and the heat-treated conditions, all base metals, except Set G, show high-impact energies. Set G shows 27 and 21 ft-lb average energies for fusion boundary tests for the as-welded and heat-treated conditions, respectively. These low values, while higher than those for several metals of construction, are severely reduced compared to the expected values for this alloy. Lateral expansion data also support this observation (Allegheny Technologies 2004, Section 5.3).

The weld centerline tests would be expected to exhibit more clearly the influence of weld filler metal chemistry on toughness. Again, this expected result was observed. In both the as-welded and the heat-treated conditions, there was a general decrease in toughness going from weld filler metal Sets 1 to 7, with one significant exception. The high residual weld filler metal Set 4 exhibited lower toughness than any weld filler metal except for the highly alloyed weld filler metal in Set 7 (Allegheny Technologies 2004, Section 5.3).

- The variables of this investigation (base metal chemistry, weld filler chemistry, and heat treatment) have significant effects on toughness (Allegheny Technologies 2004, Section 5.3).
- Fusion boundary toughness is influenced mostly by the composition of the base metal. In both the as-welded and the heat-treated conditions, Set G shows low toughness, as demonstrated both by impact energy and lateral expansion (Allegheny Technologies 2004, Section 5.3).
- Weld centerline toughness is influenced by weld filler metal chemistry, as well as by heat treatment and, to a lesser extent, by base metal composition. In both the as-welded and the heat-treated conditions, there was a general decrease in toughness going from weld filler metal Set 1 to 7, with the exception that weld filler metal Set 4 exhibited lower toughness than any filler except weld filler metal Set 7 (Allegheny Technologies 2004, Section 5.3).

Through the use of the half-size specimens, all test results fell within the certified range of the impact test machines used. In 162 tests, the test specimens failed to separate after testing, and valid lateral expansions could not be obtained. Thus, the lateral expansion data represent fewer values than do the absorbed energy data. For three of the 98 data sets (base metal, weld filler metal, and heat treatment combinations) no valid lateral expansion values were obtained (Allegheny Technologies 2004, Section 4.5).

Given the measured high toughness of Alloy 22 plate material and weldments fabricated with commercial composition type weld filler metal, any flaws not detected by the rigorous nondestructive examination that will be implemented will not impact the calculated mechanical response of the waste package. This insensitivity to the presence of flaws under the range of potential preclosure loading conditions considered for waste package performance is discussed in Appendix S of this technical basis document.

T.4.5 Conclusions

The high-residual chemistry for Alloy 22, where the residual elements are proportioned at the top of the ASME SB-575 (ASME 2001) specification range, results in such poor hot workability that it is not possible to produce rectangular bar product with any reasonable commercial practice. All other chemistries were successfully rotary-forged and rolled to rectangular bar. This includes the high residual heats for Chemistry D (see Table T-1), which had the same high-residual element contents except for sulfur and phosphorus (Allegheny Technologies 2004, Section 5.7.1). The following conclusions can be made from the results of this study:

- The high-residual chemistry for the weld filler metal ERNiCrMo-14, where the residual elements, including phosphorus and sulfur, are placed near the top of the ASME SFA-5.14 (ASME 2001) specification, demonstrated extremely poor hot workability to the extent that it was not possible to roll coil with any reasonable commercial practice. Chemistry Set 4 (see Table T-4) with phosphorus and sulfur present at typical commercial levels (0.003% phosphorus and 0.0003% sulfur) and other residuals at the same high levels, the material was hot-processed successfully (Allegheny Technologies 2004, Section 5.7.1).
- The high-purity, low-alloy content of weld filler metal Chemistry Set 1 showed low strength. When the standard commercial anneal (2,050°F for 30 minutes) was applied, the resulting samples barely satisfied the specified mechanical property requirements. The higher annealing temperatures used for the postweld heat treatment softened the material to the point that it did not reliably satisfy the specified 45 KSI yield strength requirement of ASME SB-575 (Allegheny Technologies 2004, Section 5.7.1).
- Yield strength of weldments is controlled by the base metal and heat treatment, and it is not significantly influenced by filler metal composition (Allegheny Technologies 2004, Section 5.7.1).
- Tensile strength of weldments is influenced by base metal composition, filler metal composition, and heat treatment. These parameters interact in a synergistic fashion, and these effects appear to be significant. As a particular example, the tensile strength of heat-treated weldments of base metal Chemistry D is reduced when weld filler metal Chemistry Sets 4 or 7 are used (Allegheny Technologies 2004, Section 5.7.1).
- Elongation of weldments also is influenced by base metal composition, filler metal composition, and heat treatment. Elongation is improved by heat treatment for most conditions, with the exception of weld filler metal Chemistry Sets 4 and 7, for which heat treatment reduces tensile elongation. The elongation of heat-treated weldments of base metal Chemistry D is reduced when weld filler metal Chemistry Set 4 or 7 are used (Allegheny Technologies 2004, Section 5.7.1).
- Reduction of area of weldments is influenced by base metal composition, filler metal composition, and heat treatment. Reduction of area is improved by heat treatment for weld filler metal Chemistry Sets 1, 2, 3, 5, and 6 but reduced for weld filler metal Chemistry Sets 4 and 7 (Allegheny Technologies 2004, Section 5.7.1).
- The variables of this investigation (base metal chemistry, weld filler chemistry, heat treatment, and notch location) have significant effects on toughness of weldments (Allegheny Technologies 2004, Section 5.7.1).
- Fusion boundary toughness is influenced mostly by the composition of the base metal in both the as-welded and the heat-treated conditions. Samples from Chemistry G demonstrate low toughness as demonstrated both by impact energy and lateral expansion (Allegheny Technologies 2004, Section 5.7.1).
- Weld centerline toughness is influenced by weld filler chemistry, as well as by heat treatment and, to a lesser extent, by base metal composition. In both the as-welded and the heat treated conditions, there was a general decrease in toughness going from weld filler metal Chemistry Sets 1 to 7, with the exception that weld filler metal Chemistry Set 4 exhibited lower toughness than any filler except weld filler metal Chemistry Set 7 (Allegheny Technologies 2004, Section 5.7.1).
- Postweld heat treatment at temperatures substantially above the 2.075°F nominal used in this study may be required to homogenize ERNiCrM0-14 welds and substantially eliminate the presence of TCP phases. Higher temperature postweld heat treatment may be beneficial for improving the ductility and toughness of welds. The effect of such higher temperature treatments upon the base metal would have to be examined (Allegheny Technologies 2004, Section 5.7.1).
- Lean compositions near the specification minimum levels (e.g., base metal Chemistry A) may have marginal strength. This is especially true if very high temperature (greater than the 2,075°F used in this study) postweld heat treatments are used (Allegheny Technologies 2004, Section 5.7.1).
- Rich compositions near the specification maximum levels (e.g., base metal Chemistry G) may have excessive tendency toward TCP phase precipitation. Higher temperature postweld heat treatments may mitigate this effect (Allegheny Technologies 2004, Section 5.7.1).
- Iron-rich compositions (e.g., base metal Chemistries G and E) may also exhibit increased tendency toward TCP phase precipitation, especially in the presence of high chromium, molybdenum, and tungsten (Allegheny Technologies 2004, Section 5.7.1).
- The high residual element content of base metal Chemistry D caused some problems in processing, ultrasonic examination, and weldment properties. However, the differences in composition between this heat and the others were small. The net effect of these differences is to increase the tendency toward TCP phase formation (Allegheny Technologies 2004, Section 5.7.1).
- The high residual element content of weld filler metal Chemistry Set 4 caused problems in processing and in weldment properties. While the difference between this composition and the others may be attributed to the influence of iron, other elements should not be discounted (Allegheny Technologies 2004, Section 5.7.1).
- This investigation showed that it is possible to produce weldments that exhibit the desired mechanical behavior using Alloy 22 base metal and the weld filler material ERNiCrMo-14 comparable to that produced commercially (Allegheny Technologies 2004, Section 5.7.1). It also showed that forced segregation of chemistries over the range of allowed compositions can produce poor mechanical properties. This is particularly true in the extreme lower and higher ranges. Normal commercial processes adhering to ASME SB-575 (ASME 2001) specifications produce good mechanical properties.
- Based on this study, the chemistry of Alloy 22 to be used in the waste package will be procured in accordance with ASME SB-575 Alloy N06022 (ASME 2001), having the required chemistry and limiting iron to less than (the upper limit of 6%) allowed by ASME SB-575 (ASME 2001).

T.5 REFERENCES

T.5.1 Documents Cited

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T.5.2 Codes, Standards, and Regulations

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