METALLURGICAL EVALUATION OF RECYCLED STAINLESS STEEL (U)

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Metallurgical Evaluation Of Recycled Stainless Steel (U)

Summary

Recycled Type 304 stainless steel from both Carolina Metals Inc. (CMI) and Manufacturing Science Corporation (MSC) met all the requirements of ASTM A-240 required by Procurement Specification G-SPP-K-00005 Rev. 4. Mechanical strength and corrosion resistance of the material are adequate for service as burial boxes, overpacks, and drums. Inclusion content of both manufacturer's material was high, resulting in a corresponding decrease in the corrosion resistance. Therefore, an evaluation of the service conditions should be performed before this material is approved for other applications. These heats of stainless steel are not suitable for fabricating DWPF glass canisters because the inclusion and carbon contents are high. However, MSC has recently installed a vacuum induction furnace capable of producing L grade material with a low inclusion content. Material produced from this furnace should be suitable for canister material if appropriate care is taken during the melting/casting process.

Background

The Materials Technology Section (MTS) of the Savannah River Technology Center (SRTC) was requested by Waste Minimization and Certification to evaluate mechanical properties and metallurgical condition of recycled stainless steel sheet from both CMI (heat number 006) and MSC (heat number 131). Sheet material was produced for the Beneficial Reuse Program and was used to fabricate 55 gallon drums, 85 gallon overpacks and burial boxes. The material characterization was required to verify that the material was in compliance with the applicable sections of ASTM A-240 [1] and ASTM A-480 [2] as specified by Procurement Specification G-SPP-K-00005 Rev. 4 [3] and to evaluate the material for other applications, i.e. DWPF canisters. Evaluations included tensile testing of wrought and welded samples, weld bend tests, chemical analysis (including carbon), microstructural evaluation, and corrosion testing. Feed stock for the sheet material consisted of SRS Reactor heat exchanger heads, process water pipe, and slug buckets.

Mechanical Test Results

The evaluation of mechanical properties included tensile testing of both wrought and welded specimens in accordance with ASTM A 370 [4]. ASTM A 240 requires a minimum of 75 ksi tensile strength, 30 ksi yield strength, and 40% elongation for a Type 304 stainless steel. All specimens exceeded the minimum requirements. Test results for the CMI and MSC material are presented in the following table.

Table 1. Mechanical Properties of CMI and MSC Recycled Stainless Steel.

Manufacturer	Specimen No.	Tensile Strength (psi)	Yield Strength (psi)	Elongation (%)
Minimum		75,000	30,000	40
CMI	1	104,270	56,090	50
CMI	2	104,550	52,640	52
CMI	3	105,150	54,450	52
CMI	4	103,930	55,010	52
CMI	5	106,010	56,950	50
MSC	6	116,860	57,780	52
MSC	7	111,310	57,470	52
MSC	8	108,140	55,990	54
MSC	9	111,920	56,610	52
MSC	10	109,590	55,180	55

Tensile test data and data from other mechanical and metallurgical tests can be found in the Materials Technology Section, Materials Consultation Group, METLAB Data Management System under Lab Numbers 199607025, 19961044, and 19961044.

Weld bend tests were performed in accordance with ASTM E 190 [5] and ASME Section IX [6]. Bend specimens were cut from sheet that had been welded with 308-L filler material. Four specimens were prepared (2 root and 2 face specimens) from each manufacturer and were bent 180 degrees. None of the specimens showed any evidence of cracking following the test and all the specimens met the appropriate ASTM and ASME performance requirements.

Metallurgical Test Results

Chemical Analyses

Elemental analyses were performed using a LECO glow discharge carbon analyzer and X-ray Fluorescence Analyses. Results are presented in the following table and fall within the chemical specification for Type 304 stainless steel (UNS No. S30403).

Table 2. Elemental analyses of CMI and MSC Recycled Stainless Steel.

Manufacturer	Elemental Analyses (wt%)												
	Fe	Cr	Ni	C	Mn	Mo	P	Cu	S	V	Si	Co	Nb
MSC	69.586	18.609	9.465	0.033	1.066	0.228	0.022	0.357	0.019	0.032	0.494	0.086	0.035
CMI	70.025	18.715	9.391	0.067	0.876	0.161	0.021	0.281	0.015	0.034	0.375	0.094	0.012
S30403	Rem	18 - 20	8 - 12	0.030	2.000	------	0.045	------	0.030	------	0.750	------	------

Corrosion Testing

Several corrosion tests were performed on samples from both MSC and CMI. The first was the ASTM A 262 [7,8] Practice A (oxalic acid screening test). Both samples failed the oxalic acid test therefore, ASTM A 262 Practice C (boiling 65 wt% nitric acid test) was performed. Again both samples exhibited excessive corrosion rates and suffered from severe end grain attack. A standard control sample was also tested at the same time and exhibited an acceptable corrosion rate thus demonstrating that the failure of the CMI and MSC samples was due to the metallurgical condition of the materials. Weight loss results are presented in the following table.

Table 3. Results of ASTM A 262 Practice C Test.

Sample ID	Dimensions (in)			Weight (g)				Weight Loss (g)	Weight Loss (%)
	Length	Width	Thickness	Initial	96 hr	48 hr	96 hr		
STD	0.9540	0.7660	0.2430	22.7614	22.7328	22.7208	22.7086	0.0528	0.23
CMI	1.0290	0.9840	0.1180	14.9347	14.3728	13.5547	11.7160	3.2187	21.55
MSC	1.0080	1.1180	0.0680	8.8843	8.5999	8.0353	6.1172	2.7671	31.15

Electrochemical corrosion tests, linear and cyclic polarization, were also performed to obtain corrosion data in process water (untreated well water) at ambient temperature. These tests were used to determine corrosion rate and pitting susceptibility, respectively. Process water was obtained from the 773-A process water supply and had a conductivity of 100 µS/cm. Samples of ASTM A 537 carbon steel and an actual DWPF canister (No. S00404) were also tested for reference purposes. Results of the tests are summarized in the following table.

Table 4. Results of Electrochemical Corrosion Tests.

Material	Corrosion Rate (mpy)	Pitting Susceptibility
DWPF canister S00404 (304L)	< 0.1	No
MSC (Recycled 304)	< 0.1	No
CMI (Recycled 304)	< 0.1	Yes
Carbon steel (A 537)	5	Not tested

Metallography and SEM

Metallographic specimens were prepared from the as-received plates. The microstructure is consistent with that of a rolled austenitic stainless steel except for the excessive number of inclusions (Figures 1 and 2). An as polished sample from MSC was examined using a Scanning Electron Microscope (SEM) and an elemental analyses was performed using Energy Dispersive X-Ray (EDX) analyses. Results of this analyses indicated that the inclusions consisted mainly of manganese, chromium, sulfur, and oxygen (Figures 3a and b).

Discussion

The recycled material met all the requirements of ASTM A-240 Standard Specification for Heat-Resisting Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels. However; the inclusion content of the material from both manufacturers was much higher than would be expected in this type of material. The high inclusion content did not significantly effect the mechanical properties but dramatically decreased the corrosion resistance of both materials when tested in accordance with ASTM A 262 Practice A and C. A control standard was tested at the same time and passed both tests. Practice A and C are used for corrosion evaluation (CE) of material for the Savannah River Site and ASTM A 480 (if specified by customer). Practice A is only used for acceptance. This means that if a material fails Practice A, it would have to successfully pass Practice C before it could be released for use at SRS. Practice C is a severe test that uses boiling nitric acid and is intended to detect susceptibility to intergranular attack (IGA). Since both the CMI and MSC materials were not L grade (less than 0.03 wt% carbon) material, they may have been susceptible to IGA. Therefore, the observed corrosion may have been due to both IGA and the increased inclusion content. In any case the corrosion rates were extremely high and the sheet would not be recommended for service applications where excellent corrosion resistance is required.

Mechanical properties of the materials were satisfactory (met the requirement of ASTM A 240) although the tensile and yield strengths were higher than expected for annealed material. The high values are attributed to the cold work resulting from the rolling operation. The material supplied from both CMI and MSC were probably not annealed after rolling. Figure 4 shows the microstructure of an annealed Type 304 L plate from DWPF canister S00404. This plate (0.375" nominal thickness) was annealed after rolling. The grain size from the canister S00404 is much larger than that of the rolled sheet from either CMI or MSC. The higher than expected yield and tensile strength may cause some problems (cracking) if fabrication of the final product requires additional cold work (i.e. rolling of lips on drums).

In addition to the ASTM A 262 tests, a linear polarization (LP) test was also performed. This is an electrochemical corrosion test in which process water (untreated well water) was used as the corrosive media. This short term test was used to provide an estimate of the general corrosion rate of the specimens in the process water. This test more closely represents the actual environmental conditions which a burial box or drum would be exposed to in the field. Both MSC and CMI materials performed satisfactorily with corrosion rates of less than 0.1 mil per year (mpy). These results were comparable to the actual DWPF canister material. This result would be expected since the process water is not very corrosive especially to a nickel and chromium containing material. The A 537 plain carbon steel sample (0.24 wt% carbon max), which is the same material that was used to fabricate the Type III waste H Area storage tanks, experienced a slightly higher but still acceptable corrosion rate of 5 mpy. Corrosion rates less than 10 to 20 mpy are generally considered satisfactory. The actual cutoff will depend upon the particular application and component design. A cyclic potentiodynamic polarization (CPP) test was also performed on the MSC and CMI material in the process water. The CPP test is a common electrochemical test for evaluating the susceptibility to localized corrosion. The CMI material showed increased susceptibility to pitting corrosion compared to the MSC or DWPF cansiter samples. This result maybe due to the size and distribution of the inclusions. The more inclusions that intersect the surface of the material the more susceptible the material will be to corrosive attack, especially pitting. Inclusions were most likely manganese sulfide and titanium oxide. Chromium and iron observed in the EDX spectra were probably from the 304 matrix although some chromium oxide may have been present. Overpacks and burial boxes fabricated from these materials will have adequate corrosion resistance when exposed to ground water. However, the service conditions of new applications should be evaluated before the recycled material is approved for use.

The Type 304 material supplied by CMI and MSC would not be acceptable for use in fabricating a DWPF canister. The procurement specification for the Beneficial Reuse Demonstration did not require that 304-L be produced. DWPF canisters are fabricated from Type 304-L. In addition, the residual stresses of the cold worked material may result in distortion of the rolled plates during welding. Even the slightest distortion may result in rejection of the canister because of the very stringent parallelism requirements between the canister bottom and the nozzle. A complete anneal will alleviate this condition. The service conditions of new applications should be thoroughly evaluated prior to approving this material for use. MSC has recently installed a new vacuum induction melting furnace which should be able to produce L grade material and dramatically reduce the inclusion content. Material produced from this furnace will be sent to WSRC for evaluation when it is available.

Conclusions and Recommendations

Based on the results of the mechanical and metallurgical tests the conclusions and recommendations are as follows:

- Recycled stainless steel sheet from MSC and CMI met the requirements of ASTM A-240 for a Type 304 stainless steel as required by Procurement Specification G-SPP-K-00005 Rev. 4.
- Inclusion content of the CMI and MSC stainless steel was excessive and degraded the corrosion resistance of the materials.
- Both manufacturer's material failed ASTM A-262 Practice A and C and therefore, they should not be used in very corrosive, oxidizing environments.
- Corrosion rates were less than 0.1 mil per year for the CMI and MSC material in process water.
- Cyclic polarization showed a slight tendency for the CMI to pit in process water.
- CMI and MSC material possesses adequate corrosion resistance to perform satisfactorily as a burial box, overpack, or drum when these containers are exposed to ground water. However, service conditions of new applications should be evaluated before the containers are approved for use.

References

1. ASTM A 240 - 94a, Standard Specification for Heat-Resisting Chromium and Chromium-Nickel stainless Plate, Sheet, and Strip for Pressure Vessels.
2. ASTM A 480 - 94b, Standard Specification for General Requirements for Flat-Rolled Stainless and Heat-Resisting Steel Plate, Sheet, and Strip.
3. Procurement Specification G-SPP-K-00005 Rev. 4.
4. ASTM A 370 - 92, Test Methods and Definitions for Mechanical Testing of Steel Products.
5. ASTM E 190 - 92, Standard Test Methods for Guided Bend Test for Ductility of Welds.

6. Section IX of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code, Section QW-462, 1994.

7. ASTM A 262 - 93, Standard practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels.

8. Engineering Standard 05951 Rev. 0, Corrosion Evaluation: Stainless Steels And Other Corrosion Resisting Alloys, SRS Engineering Standards Manual WSRC-TM-95-1.

Figure 1. Photomicrograph of CMI recycled stainless steel sheet showing numerous inclusions (Magnification 100 X, Etch 10% Oxalic acid electrolytic).

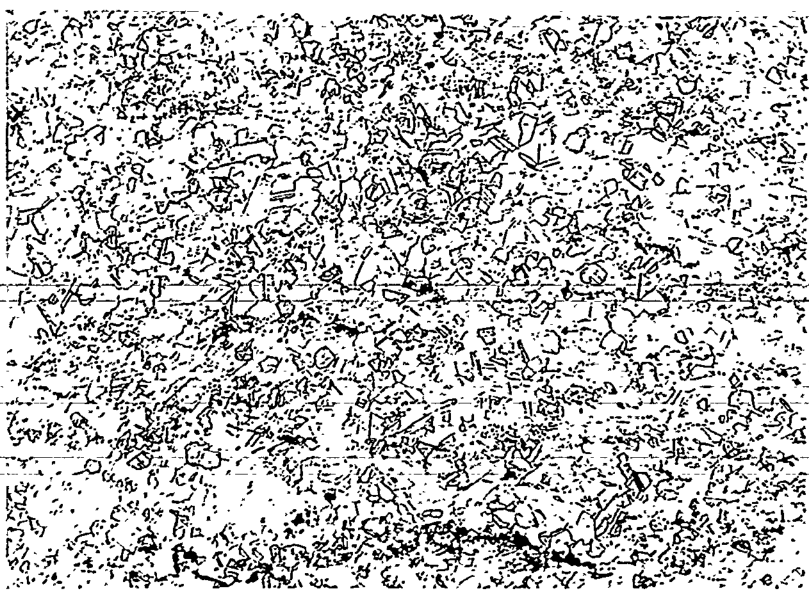
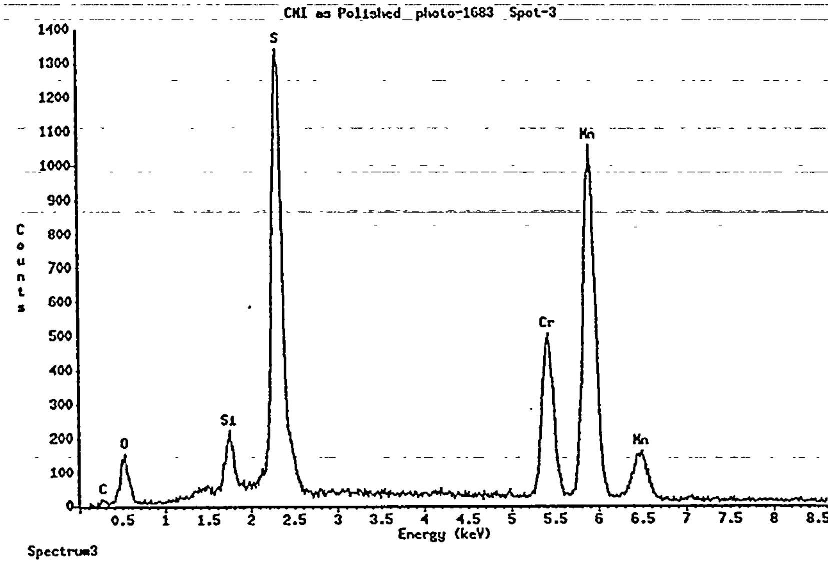
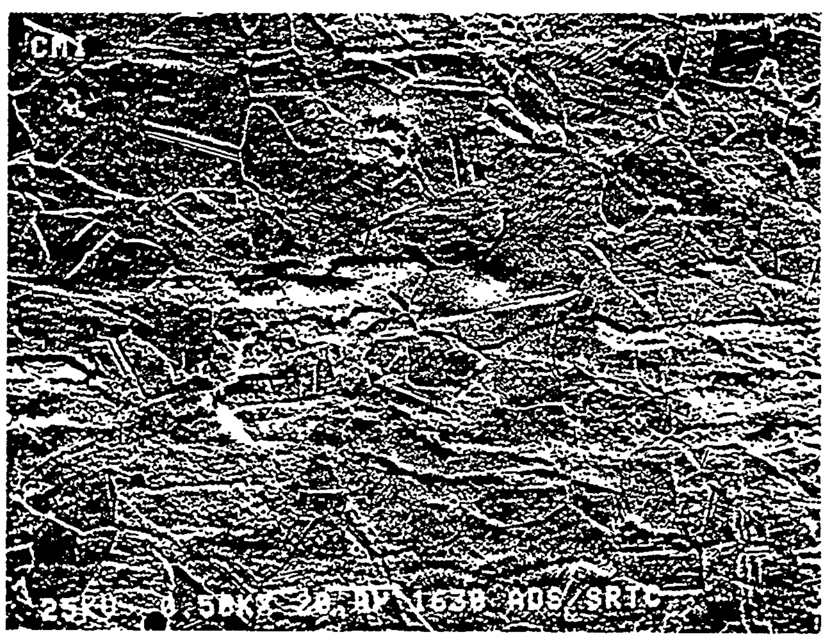


Figure 2. Photomicrograph of MSC recycled stainless steel sheet showing inclusions (Magnification 100 X, Etch 10% Oxalic acid electrolytic).



(a)



(b)

Figure 3. a) Elemental analysis of CMI recycled material. b) SEM photograph showing pits where inclusions were located before metallographic preparation. Etching removed inclusions leaving the pits.

Figure 4. Photomicrograph of the DWPF canister S00404 (0.375" nominal wall thickness). The material of construction is annealed Type 304 L stainless steel (Magnification 100 X, Etch 10% Oxalic acid electrolytic).

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