DEVELOPMENT OF ODS HEAT EXCHANGER TUBING

Quarterly Technical Progress Report

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ABSTRACT
Work continued on four major tasks of this project – increasing the circumferential strength of MA956 tubing, joining of the MA956 alloy, determining the bending limits of MA956 tubing, and determination of the high temperature corrosion limits of the MA956 alloy. With respect to increasing the circumferential strength of a MA956 tube, an additional 120 MA956 rods have been extruded (total of 180 rods) using 16:1 and 10:1 extrusion ratios and extrusion temperatures of 1000, 1075, 1150, and 1200°C. Also, approximately 40 cold work (0, 10, 20, 30, 40%) plus annealing treatments (1000, 1150, 1300°C) have been completed with the resulting sample microstructures presently being analyzed. Creep testing to determine the "stress threshold" curves for this alloy continues. Regarding joining of the MA956 alloy, work continued using friction welding, magnetic impulse welding, explosive welding, and transient liquid phase bonding, with encouraging results obtained from the friction, explosive, and transient liquid phase joining methods. Initial work on determining the bending limits of the MA956 tubing has shown that the recrystallized material shows good ductility but the un-recrystallized material does not. And finally, fluid-side high temperature corrosion testing of the material continues and the environment for the laboratory fireside corrosion testing has been established and testing initiated.

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INTRODUCTION

This research is seeking to develop a MA956 heat exchanger tube that will lead to the design and fabrication of a MA956 full-scale tube heat exchanger composed of the referenced alloy. The alloy MA956 is an oxide dispersion strengthened (ODS) material that possesses superior creep strength and corrosion resistance at very high temperatures (e.g. T > 2000°F) compared to traditional wrought or cast alloys. However, the creep properties are unidirectional (typically stronger in the longitudinal direction compared to the transverse direction), fabrication of components made from this alloy is relatively difficult, and the corrosion limits of the alloy MA956 in coal-fired environments are not known. Thus, the technical tasks being executed in this Vision 21 project are:

Task 1: Project Management
Task 2: Improvement of Circumferential Creep Strength of MA956 Tubes
Task 3: Joining
Task 4: Bending of MA956 Tubes
Task 5: High Temperature Corrosion Limits of MA956
Task 6: Generation of Data for Designers
Task 7: Implication of ODS Properties on Heat Exchanger Design
Task 8: Reporting

The members of the team conducting this research are: Huntington Alloys (HA), Foster Wheeler Development Corporation (FWDC), Oak Ridge National Laboratory (ORNL), University of California, San Diego (UCSD), Michigan Technological University (MTU), and the Edison Welding Institute (EWI).

EXPERIMENTAL

Experimental work associated with the tasks identified in the previous section is discussed below.

Task 2: Improvement of Circumferential Creep Strength of MA956 Tubes

The following matrix of tests shown in Table 1 is currently being performed at HA. The execution of this matrix will result in 540 different combinations of extrusion + thermomechanical + annealing parameters. Table 2 shows the work that has been completed thus far.

Table 1
Matrix of Extrusion + Cold Work + Recrystallization Parameters

Extrusion Temp (°C)	Extrusion Ratio	Amount of Cold Work (%)	Recrystallization Temp (°C)	Recrystallization Time (h)
1000	10:1	0	1000	0.5
1075	16:1	5	1150	1
1150	20:1	10	1300	6
1200		15		
		25		

Table 2
Work Completed on Extruded + Cold Work + Recrystallized Samples

Operation	Number Required	Number Complete	% Complete
Extrusion	180	180	100
Decanning	540	354	66
Cold Work	540	223	41
Annealing	540	175	32
Microstructure Analysis	540	54	10

Creep testing continues at ORNL for the purpose of determining the 'threshold stress' curves of the MA956 alloy. Results of this testing will be discussed in the next section.

Task 3: Joining
Friction Welding: In addition to the friction welds made last quarter (MA956 joined to MA956), welding trials of MA956 tubing joined to 601 tubing were performed.

Transient Liquid Phase Bonding: The development of a boriding technique that produces an approximate 4 micron thick boride layer has led to joints of the MA956 alloy bonded to itself.

Explosive Welding: Shear testing and examination of the interface of MA956/MA956 and MA956/601 joints produced by explosion welding were completed.

Magnetic Impulse Welding: Further work on this task awaits the arrival of a new magnetic impulse welder.

Task 4: Bending of MA956 Tubes
Tube flattening tests have been performed on the recrystallized and un-recrystallized tubing supplied to FWDC.

Task 5: High Temperature Corrosion Limits of MA956
The exposure of samples of MA956 bar stock in air at 1200 and 1250°C has begun. Also, the test environment for the laboratory fireside corrosion testing has been established, testing initiated, and work on fabricating the boiler probe for in-field testing has begun.

Task 6: Generation of Data for Designers
No experimental work has been accomplished on this task during this reporting period.

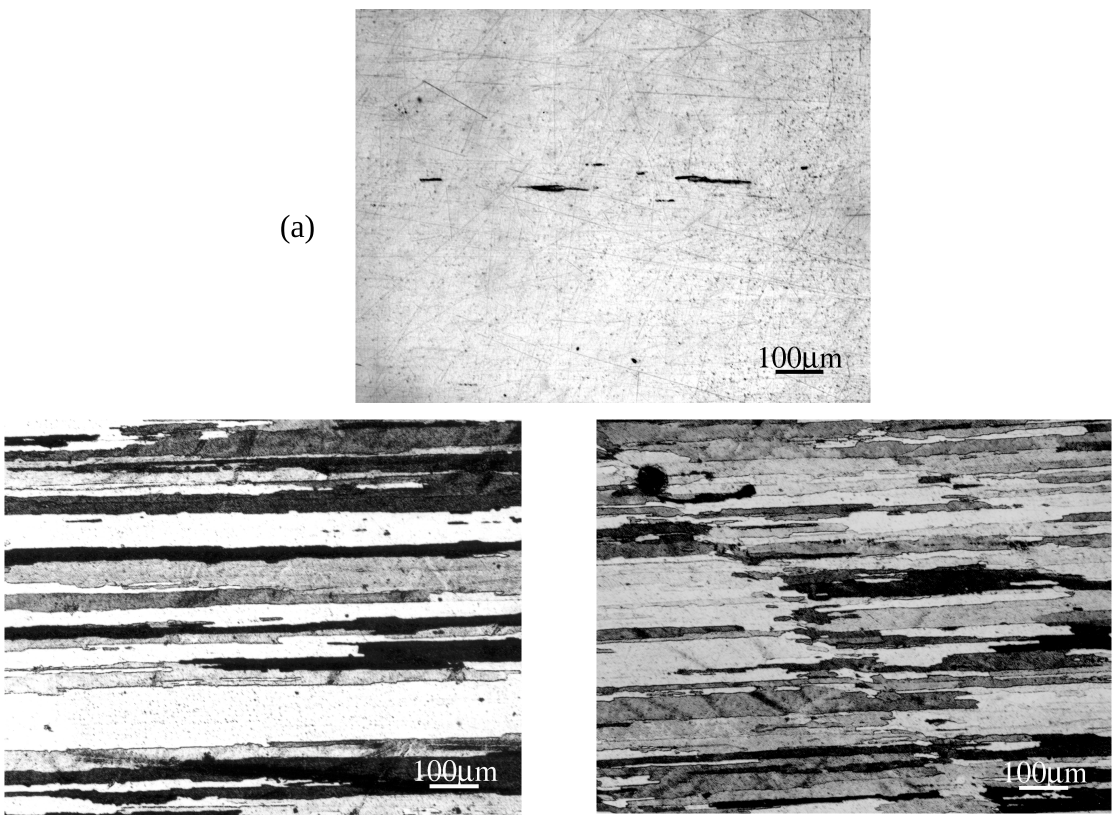
Task 7: Implication of ODS Properties on Heat Exchanger Design
No experimental work has been accomplished on this task during this reporting period.

RESULTS AND DISCUSSION

Task 2: Improvement of Circumferential Creep Strength of MA956 Tubes
Microstructural analysis of the 54 MA956 rods that have been extruded + cold worked + annealed has shown a variation in the grain structure and hardness of the samples.

Regarding the effect of recrystallization temperature, samples annealed at 1000°C did not exhibit any recrystallization, independent of the amount of cold work imposed on the sample. However, samples annealed at 1150°C did show the onset of primary recrystallization, the amount being dependent on the amount cold work. When annealed at 1300°C, all the samples exhibited primary and secondary recrystallization, with the final recrystallized grain morphology being a function of the cold work for a given sample. Figure 1 shows the variation in microstrucutre morphology observed as a function of cold work for samples annealed 1300°C for six (6) hours. Although enough data is not yet available to allow for an understanding and prediction of the effect of the various production variables on the final component microstructure, this knowledge will be critical in producing a tube with the desired microstructure and thus desired mechanical properties.

(a)



(b) (c)

Figure 1. Variation in microstructure for MA956 rods extruded at 1000°C using a 20:1 extrusion ratio followed by (a) 0% CW, (b) 10% CW, and (c) 30% CW, and then annealed at 1300°C for 6 hours.

Results of the creep testing of specimens cut in the axial direction from the walls of a nominal 1 inch diameter MA956 tube (Heat # WBD0643) are shown in Figure 2. As shown on this Larsen-Miller plot, the data being generated in this program is in agreement with data in the literature. The next step in this task will be to generate data from the transverse direction of the tube.

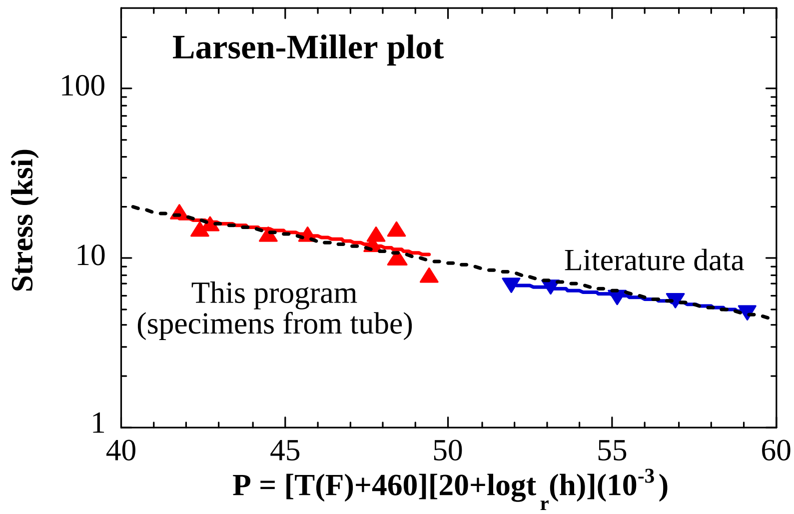


Figure 2. Larsen-Miller plot showing the good agreement between the stress rupture data obtained from this program and stress rupture data obtained from the literature.

Task 3: Joining

Friction welding: During the 3rd quarter of this project friction welds were made on the MA956 to MA956 tubing with some of the joints showing tensile properties approaching those of the base material. The 4th quarter resulted in friction welds between MA956 tubing and 601 tubing with the mechanical properties of such a weld shown in Table 1 below. As shown in this table, the joint strength approaches that of the 601 alloy and thus shows promise that friction welding may serve as a suitable technique for joining MA956 to a traditional wrought heat resistant alloy. However, the very low ductility of these joints also shows that more work is needed in this area.

Table 1
Mechanical Property Data for the MA956 Tubing Joined to 601 Tubing Using Friction Welding

Sample Identification	Ultimate Tensile Strength (ksi)	Yield Strength (ksi)	Elongation (%)
MA956/601-1	39.6	37.6	< 1
MA956/601-2	26.4	19.8	< 1
MA956/601-3	43.2	42.4	< 1
MA956/601-4	40.5	40.3	< 1
601*	107.5	42.1	47

* Typical properties of 601 hot finished bar solution annealed at 2000°F.

Transient Liquid Phase Bonding: Past boriding work has resulted in the successful production of samples with uniform borided layers 3 to 5 microns thick. Since the vacuum hot press has not been in operation, three sets of samples were prepared for HIP. These include recrystallized/recrystallized, non-recrystallized/recrystallized, and non-recrystallized/non-recrystallized samples of the MA956 alloy, where both pieces in the bonding couple had been borided, as well as 3 bonding couples with only one borided piece. The joining of the non-recrystallized to recrystallized material was performed in order to cause grain growth across the interface during the joining process. Initial joining runs have resulted in joints that appear void-free with a few residual particles and limited grain growth across the interface (see Figure 3 below). Work continues on refining the boriding technique and additional joining trials using the vacuum hot press are planned.

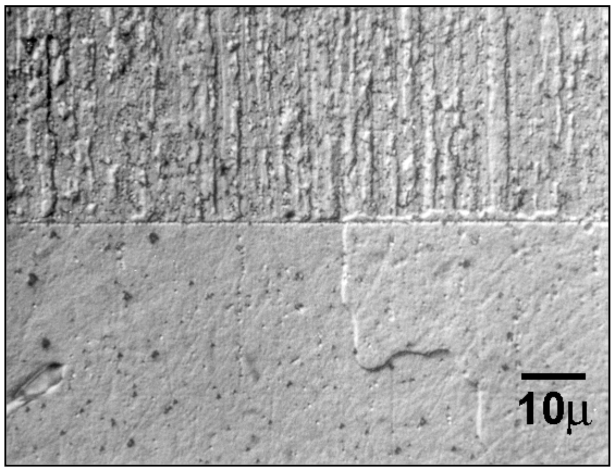
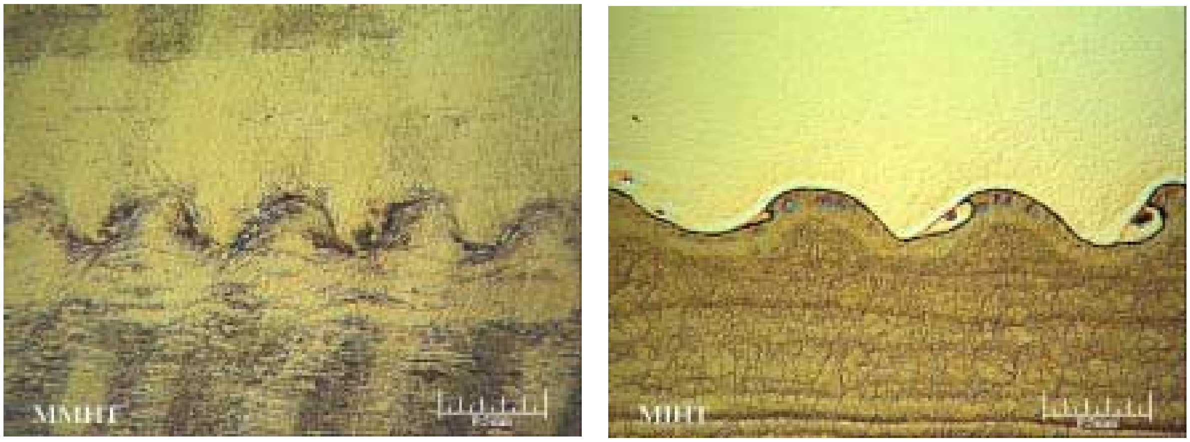


Figure 3. Joint produced using transient liquid phase bonding.

Explosive Welding: Two samples from MA956 plate explosion welded to MA956 plate, and two samples from MA956 plate joined to 601 plate were extracted for metallographic analysis and shear testing. Figure 4 shows the interface of the MA956/MA956 and MA956/601 welds after a post explosion weld heat treatment at 1000°C for 1 hour. As shown in these micrographs, the wave-shaped interface characteristic of a successful explosion weld is present. Table 2 shows the results of the shear testing performed on the as-welded samples and the post weld heat-treated samples. The heat-treated MA956/MA956 sample showed a slight decrease in bond strength as a result of the heat treatment whereas the MA956/601 sample showed an increase in shear bond strength after the heat treatment. However, the measured shear strength for the heat treated MA956/601 sample may be artificially high due to the relatively high ductility of the annealed 601 material. The next step will consist of weld trials on the tube materials of the MA956 and 601 alloys using nearly identical explosion welding parameters as for the plate samples.



(a) MA956/MA956 (b) MA956/601

Figure 2. Optical micrographs of the interface of an explosion weld between (a) MA956 and MA956 plate and (b) MA956 and 601 plate.

Table 2
Shear Test Results of Explosion Welds Made on
MA956 plate to MA956 plate and MA956 Plate to 601 Plate

Sample	Shear Strength (ksi)
MA956/MA956	82.0
MA956/MA956 + Heat Treatment*	77.2
MA956/601	79.5
MA956/601 + Heat Treatment*	88.7**

* Heat treatment performed at 1000°C for 1 hour

** Value may be artificially high due to ductility of the 601 after the 1000°C/1 hour heat treatment

<u>Magnetic Impulse Welding:</u> A new machine with 4 times the energy of the present machine will be installed and operational in November. This machine will allow for greater energy input in the welding trials.

Task 4: Bending of MA956 Tubes
In an effort to determine preliminary tube bending limits for the MA956 alloy, tube-flattening tests were performed on recrystallized and non-recrystallized material. The results of these tests (Table 3) show that the recrystallized material exhibited good ductility, particularly when deformed at 400°F, however the non-recrystallized material exhibited relatively poor ductility even after being annealed at 1800°F for 24 hours. Actual tube bending tests will be performed this next quarter.

Table 3
Results of Tube Flattening Tests

Tube Condition	Annealing Information		Hardness	Flattening % at Test Temperature	
	T (°F)	Time (h)	(HRC)	Ambient	400°F
CD / ANN	Standard HA Procedure		25	37.6	54.0
CD	N.A.	N.A.	35	0 (cracked)	0 (cracked)
CD/ANN	1700	1	26-28	5.9	6.5
CD/ANN	1700	24	26-28	4.4	8.2
CD/ANN	1800	1	26-28	4.0	5.3
CD/ANN	1800	24	25	6.2	9.0

Task 5: High Temperature Corrosion Limits of MA956

Laboratory Testing for Working Fluid Side: Work continues on the lifetime prediction of the MA956 alloy in air at very high temperatures. Testing at 1300°C has been completed and testing at 1200 and 1250°C is currently being performed.

Laboratory Testing for Fireside Environment: Laboratory testing using two different flue gases and three different deposits has been initiated. The composition of the flue gases and deposits are shown Tables 4 and 5 below. A char analysis produced by the FWDC partial gasification unit was used as the basis for the deposit compositions.

Table 4
Flue Gas Compositions to be Used in Laboratory Fireside Testing

Species	Amount (vol %)	
	Gas Mixture 1	Gas Mixture 2
O_2	4	2
CO_2	15	15
H_2O	10	5
SO_2	0.25	1.0
N_2	Bal	Bal

Table 5
Deposit Compositions to be Used in Laboratory Fireside Testing

Species	Amount (wt%)		
	Ash 1	**Ash 2**	**Ash 3**
Si Dioxide	14.6	11.6	7.6
Al Dioxide	6.0	6.0	6.0
Ti Dioxide	0.3	0.3	0.3
Fe Oxide	1.3	1.3	1.3
Ca Oxide	3.3	3.3	3.3
Mg Oxide	0.3	0.3	0.3
Na Oxide	0.4	1.4	2.4
K Oxide	0.3	1.3	2.3
S Trioxide	1.2	2.2	3.2
P Pentoxide	0.3	0.3	0.3
KCl			1.0
Carbon	72.1	72.1	72.1

Field Exposure Testing: The location for field probe has been determined, the probe control hardware is currently being procured and the probe design is being finalized.

CONCLUSIONS

No technical conclusions are available at this time, however the change in grain morphology as a function of the extrusion + TMP parameters for the MA956 rods, and the joining obtained results thus far, are encouraging. Work will continue under Tasks 2, 3, 4, and 5.

REFERENCES

None.