DEVELOPMENT OF ODS HEAT EXCHANGER TUBING

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ABSTRACT

Due to extenuating circumstances Special Metals Corporation is terminating their role as prime contractor with this Vision 21 project. In response to this situation, a status report for the project as of this date, has been prepared and follows.

Significant work has been accomplished on three major tasks of this project – increasing the circumferential strength of MA956 tubing, joining of the MA956 alloy, and determination of the high temperature corrosion limits of the MA956 alloy. With respect to increasing the circumferential strength of a MA956 tube, the first rod extrusion campaign has been completed with microstructure analysis providing valuable information on the strengthening mechanism of this alloy. Also, based on the results obtained thus far extrusions of tubes are in process and creep testing to determine the “stress threshold” curves for this alloy continues. Regarding joining of the MA956 alloy, welds have been produced using the friction, explosive, magnetic impulse, and diffusion bonding techniques. Complete elevated temperature mechanical testing has not been conducted on joints produced using these methods, however room temperature tensile and shear testing has shown promising results on friction and explosive welds. And finally, laboratory high temperature corrosion testing of the material continues in both fluid-side and fire-side simulated environments.

Brief summary status statements from each of the subcontractors is appended to this report which additionally contains the expected funding needed to complete the project.

TABLE OF CONTENTS

<u>Section</u>	<u>Page #</u>
TITLE PAGE	1
DISCLAIMERS	2
ABSTRACT	3
INTRODUCTION	5
EXPERIMENTAL	5
RESULTS AND DISCUSSION	7
CONCLUSIONS	14

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. $T > 2000^{\circ}\text{F}$) 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: Special Metals (SM), 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 has been completed at SM. 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 with these extrusion samples.

Table 1
Matrix of Extrusion + Cold Work + Recrystallization Parameters

Extrusion Temp ($^{\circ}\text{C}$)	Extrusion Ratio	Amount of Cold Work (%)	Recrystallization Temp ($^{\circ}\text{C}$)	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 Bar Samples

Operation	Number Required	Number Complete	% Complete
Extrusion	180	180	100
Decanning	540	540	100
Cold Work	540	540	100
Annealing	540	540	100
Samples Prepared	540	540	100
Microstructure Analysis	540	540	100

Transverse samples were taken from each condition for evaluation.

Task 3: Joining

Friction Welding: Testing demonstrated the ability to friction weld the MA956 alloy. In an effort to determine how robust the friction welding process was with respect to joining MA956 tubing, a Design of Experiments was initiated in order to evaluate the effect of different process parameters on elevated temperature joint properties.

Transient Liquid Phase Bonding: TLP bonding trials were conducted on the MA956 alloy with preliminary success obtained on 2.5" OD tubing.

Explosive Welding: Explosive welding was shown to be a possible method for joining the alloy MA956 to itself and to the 601 alloy..

Magnetic Impulse Welding: Limited success was obtained using magnetic impulse welding on the MA956 alloy.

Task 4: Bending of MA956 Tubes

Preliminary work involving a suitable heat treatment for the non-recrystallized MA956 material in order to obtain some ductility before bending was completed.

Task 5: High Temperature Corrosion Limits of MA956

Both lifetime working side exposure testing (1100, 1200, and 1250°C) and laboratory fireside corrosion testing was initiated and is still in process.

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: Microstructural analysis of the rods that have been extruded + cold worked + annealed is complete and a variation in the grain structure has been observed. 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 microstructure morphology observed as a function of cold work for samples annealed 1300°C for six (6) hours.). Thus it would appear that while up to 40% CW can initiate recrystallization – it does not guarantee a large processed grain size. It is also important to note that shear bands occur for samples subjected to relatively large amounts of cold work, thereby creating transverse grain boundaries which may restrict secondary grain growth (Fig. 1(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.

Tube samples flow formed greater than 85% at Dynamic Machine Works in Bellerica, MA, and annealed at 1300°C/1 hour did recrystallized to an onion skin texture in the transverse direction. This work was funded outside of the Vision 21 project.

Thus far transmission electron microscopy (TEM) studies have been the most comprehensive for samples extruded at 1075°C using a 20:1 extrusion ratio (A3B series) and annealed at 1150 or 1300°C. The microstructures of these samples appear to be bimodal with regions that are fully recrystallized as well as areas where no recrystallization has occurred. Also, increasing the amount of cold work results in an increase of the volume fraction of recrystallized material, as shown in Figure 2 below.

(a) 0% CW

(b) 10% CW

(c) 20% CW

Figure 2. Effect of cold work on recrystallization for samples extruded at 1075°C using a 20:1 extrusion ratio and annealed at 1150°C for 6 hours.

Microstructures that have experienced creep at ORNL have also been examined with TEM. Figure 3 is a TEM micrograph of the microstructure of a sample exposed to a high stress for a short time (10 ksi / < ½ hour) at 900°C. As seen in this figure, the microstructure has the ability to develop and sustain a high density of dislocation network with the dislocations suitably pinned by dispersoids as shown in Figure 4, whereas Figure 5 shows grain boundary migration with segments pinned by large dispersoids. This gives initial evidence that the boundaries may be mobile at 900°C under high stress (10 ksi).

Figure 3. TEM micrograph of transverse oriented creep sample subjected to high stress for a short time (10 ksi / < ½ hour) at 900°C.

Figure 4. TEM micrograph of transverse oriented creep sample subjected to high stress for a short time (10 ksi / < ½ hour) at 900°C showing stable dislocation pinning by dispersoid particles.

Figure 5. TEM micrograph of transverse oriented creep sample subjected to high stress for a short time (10 ksi / < ½ hour) at 900°C showing grain boundary migration under the applied stress and the drag exerted by coarse particles (as marked) in the microstructure.

Creep Testing: 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 Table 3 with the data plotted in Figure 6. The specimens were loaded at the initial value indicated in the table, and the load was increased by 1 ksi every 100 hours until failure. The ‘life’ values show the total exposure time and, in brackets, the time spent in the last load increment. It is important to note that the reason for performing this type of creep testing is that the excellent strength of this alloy at elevated temperature results in a relatively “flat” stress rupture curve. Thus extremely long times can be required to cause rupture if the applied stress is below a “threshold” value where essentially no creep occurs. In order to determine the approximate value of this threshold stress for a given alloy at a particular temperature, creep tests are conducted where the sample is incrementally loaded at 100-hour intervals. Testing of further specimens is in progress at different temperatures to establish the ‘threshold stress’ curve for this alloy.

Table 3
Results of Stress Threshold Testing for the MA956 Alloy

Temp. (°C)	Load (ksi)	Life	El. (%)	LM parameter
900	7-14	860 (44)	0	45.7
950	5-14	1224 (51)	—	47.8
1000	3-8	590 (59.2)	2.4	49.4

Figure 6. Larsen-Miller plot showing the good agreement between the stress rupture data obtained from this program and stress rupture data obtained from the literature.

In an effort to test MA956 tubing in the transverse direction, pieces of MA956 tubing were cut in half in the longitudinal direction and the half pieces hot worked into flat pieces of plate (Figure 7).

Figure 7. Schematic drawing showing how plate is produced from tube, thus allowing creep samples to be taken in the transverse direction.

The current compilation of data for the creep threshold curves for specimens cut from two tubes of different diameter, and from the flattened tube mentioned above are shown in Figure 8. The transverse direction creep threshold stresses determined for specimens cut from a flattened 6 cm OD x 5 mm wall tube are significantly lower than those determined for the longitudinal direction for specimens cut from a non-flattened tube. While this was expected - it is the basis for this project – the comparison is not straightforward since the grain sizes of the two heats of alloy are significantly different.

Figure 8. Longitudinal and transverse creep threshold curves for specimens cut from MA956 tubes. Note that the transverse samples were taken from a flattened 6 cm OD x 5 mm wall tube.

Task 3: Joining

Friction welding: A series of welds were made on the first lot of tubing supplied to EWI and metallographic analysis and tensile tests were conducted on samples welded with two different sets of welding parameters (noted as #I2 and #I3). For tensile testing, a strip sample was taken from various points around the diameter of the tube (noted as A, B, and C). Mechanical property data for each of the welds tested are listed in Table 4. These results appear promising compared to the parent alloy properties shown in the table, however the variation in mechanical properties around the circumference of the joint is causes for concern.

Table 4
Mechanical Property Data for Two Inertia Welds

Sample Identification	Ultimate Tensile Strength (ksi)	Yield Strength (ksi)	Elongation (%)
I2-A	99.0	85.8	4.4
I2-B	73.3	N/A	0.2
I2-C	97.1	93.3	2.2
I3-A	80.0	N/A	3.4
I3-B	99.4	80.1	7.7
I3-C	67.0	N/A	2.0
MA956*	93.5	80.2	10

* Typical properties of parent material

Friction welds between MA956 tubing and 601 tubing were also conducted with the mechanical properties of such a weld shown in Table 5 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 5
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.

In an effort to determine how robust a processing window was required to make an acceptable joint a small Design Of Experiments (DOE) task was initiated to evaluate the effect of different process parameters on the elevated temperature performance of Inertia Friction Welded (IFW) joints in MA956 alloy. Variables under investigation in the DOE study include initial surface velocity, or speed, and the welding force in the IFW process. Currently all welds have been made for the MA956/MA956 non-recrystallized couple and samples have been machined for testing. Based on these results other DOE studies for optimization may be conducted for the other material couples under investigation in the program. The process parameters of the current testing as well as weld features are indicated in Table 6 and photomicrographs of each sample are shown in Figures 9 through 12 below.

Table 6
Process Parameters for the Design of Experiments Being Conducted for Inertia Friction
Welding of the MA956 alloy

Sample	Speed (RPM)	Force (PSI)	Mechanically Mixed Zone width (mm)	Plasticized Zone width (mm)	HAZ Width thermal (mm)	Burnoff (mm)
5	425	25000	0.0425	0.50	1.10	1.77
6	625	25000	0.0825	0.54	1.45	6.35
7	625	35000	0.1125	0.48	1.80	8.63
8	425	35000	0.0150	0.40	1.10	2.80

Figure 9. Photomicrographs of inertia weld trial #5.

Figure 10. Photomicrographs of inertia weld trial #6.

Figure 11. Photomicrographs of inertia weld trial #7.

Figure 12. Photomicrographs of inertia weld trial #8.

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 was not 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 13 below).

Figure 13. Joint produced using transient liquid phase bonding.

Work continued on the MA956 diffusion bonding trials using the vacuum hot press once it was in operation. This included six exploratory bonding trials on 0.5 inch diameter bar, and initiation of an eight-run parametric study designed to identify the most important process variables in diffusion bonding of the MA 956 alloy. The six bonding trials included joining borided recrystallized rod to unrecrystallized rod, borided unrecrystallized rod to recrystallized rod, and recrystallized rod to unrecrystallized rod using a thin interlayer of elemental boron. Several of these samples showed significantly stronger evidence of grain growth across the prior joint interface than has been observed in prior trials. Figure 14 shows the bond line of the fifth bonding trial.

(a)

(b)

Figure 14. (a) Micrograph of borided, recrystallized MA956 (top) bonded to non-borided, non-recrystallized MA956. Box in (a) shows approximate region shown in (b). Samples etched with HCl.

As work continued on determining the most appropriate hot pressing parameters for joining the MA956 alloy results showed there may be little benefit to boriding one or both of the mating MA956 surfaces prior to bonding. Even with the low levels of boron added using the protocols developed earlier in the program, nearly all of the borided joints continue to exhibit residual borides at the joint interface, which may inhibit grain growth across the interface after bonding. Workpieces bonded with out boron, however, show a relatively clean interface and evidence of grain growth across this interface. Figure 15, for example, shows a ring of recrystallized MA956 tube bonded to a ring of unrecrystallized MA956 tube (2.5 inch OD x 0.25 inch wall). The two pieces were bonded by hot pressing at 1200°C for 2 hours, followed by 4 hours at 1340°C. Note that there is very little difference in the microstructure between the two pieces or between the joint and the surrounding matrix. Perhaps even more importantly, a number of grains clearly appear to have grown across the interface during bonding. Such a microstructure is likely to exhibit good elevated temperature tensile performance.

Figure 15. Micrograph of bond line microstructure produced by hot pressing recrystallized and unrecrystallized MA956 tubing.

In order to join MA956 tubes using the diffusion bonding process, MTU believes it will require joining two recrystallized tubes using an intermediate tapered ring of unrecrystallized material. This “3-body” joint would consist of two sections of recrystallized MA956 tubing machined at a 45° angle which fit into a surrounding “collar” of non-recrystallized MA956 containing two mating 45° angles. Figure 16 shows a diffusion bonded joint observed in the one of these 3-body coupons. This joint was produced using a modified pressing schedule incorporating an initial high-pressure, low-temperature deformation segment prior to diffusion bonding. This modified schedule improved the alignment of the three joint sections, although slight bulging did still occur in the mid-section of the sample. Metallographic examination of the cross-section of this sample revealed that joint bonding was much improved over prior samples. Micrographs of the joint are shown in Figure 17.

Figure 16. Diffusion bonded joint in a 3-body coupon.

Figure 17. Micrograph of hot press “3 body” joint of MA956. Note bond line running diagonally through image. Etched with HCl.

Explosive Welding: Parametric calculations for the explosion welding of a 0.375 inch thick MA956 plate, with an estimated room temperature yield strength of 80 ksi, indicated the potential for successful welding of the 4 inch x 6 inch plate samples would be marginal. Therefore, an initial setup for an MA956-to-601 weld trial was disassembled and the MA956 plate was milled down to 0.25-inch thickness to match the thickness of the 601 plate. The setup was then shot (explosion welded) with the MA956 plate accelerated down onto the stationary 601 plate. Ultrasonic inspection of the sample indicated a non-bond at the start-up of the joint 1-1.5 inch long with the remaining 4.5-5 inches of the joint being bonded. This preliminary examination indicated a successful weld between the material couple.

Setup for explosion welding the first sample of MA956-to-MA956 was also prepared and welded. Initially, the cladding or accelerated plate was milled down to 0.25-inch thickness, the same as had been done for the MA956-to-601 weld trial. The base or stationary MA956 plate, however, was not reduced in thickness, as its thickness does not

effect the explosion welding collision conditions. The explosion welding parameters used to weld this sample were the same as those used on the MA956-to-601 weld trial with a minor change in the setup to reduce the amount of non-bond experienced on the initial sample shot. Following this explosion welding trial, preliminary ultrasonic inspection revealed the sample to be completely bonded over the entire 4 inch x 6 inch surface area.

Two samples from the MA956 plate explosion welded to MA956 plate, and two samples from the MA956 plate joined to 601 plate were extracted for metallographic analysis and shear testing. Figure 18 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 7 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.

Figure 18. Optical micrographs of the interface of an explosion weld between (a) MA956 and MA956 plate and (b) MA956 and 601 plate.

Table 7
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 alloy

Magnetic Impulse Welding: Joining trials using 0.5 inch diameter tubes did show this process to be feasible to join the MA956 alloy, however higher impact velocities were needed. Thus a new 90 kJ system was installed and in preparation for weld trials, process-modeling was performed for design of tooling (coil & concentrator) necessary to weld the material couples under investigation. The tooling design was completed and the coil and concentrator fabricated by an outside vendor. However, a system “checkout” revealed an inefficient delivery of energy to the workpiece and thus the coil had to be modified.

In parallel to the equipment validation tasks, tooling/fixturing as well as joint preparation activities continued. MA956 tube material was prepared for welding by producing a lap joint configuration. The tubes were prepared by machining the ID of one tube and the OD of the mating tube so that the 2 tubes will fit together for welding with appropriate standoff distance for magnetic impulse welding. A sketch of the tube joint geometry is shown below in Figure 19. Initial weld trials are to be performed with 1/4t wall thickness (joint thickness 1/2t). A conservative approach is being used initially with this wall thickness to validate the process. Once successful, trials will be carried out with increased wall thickness. For initial weld trials with the MA956/601 couple, the 601 tube will be placed outside of the mating MA956 tube. Also, the fixturing and tooling required for welding the tubes has been designed and fabricated. Photos in Figure 20 show equipment setup for welding the MA956 tubes.

Figure 19. Sketch of lap joint configuration for Magnetic Impulse Welding

Figure 20. Tooling/fixture set-up for magnetic impulse welding of tube test specimens.

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 8) show that the recrystallized material exhibited good ductility, particularly when deformed at 400°F, however the non-recrystallized material exhibited relatively poor ductility when annealed up to 1800°F for 24 hours. Actual tube bending tests have yet to be performed but are planned for this quarter.

Table 8
Results of Tube Flattening Tests

Tube Condition	Annealing Information		Hardness (HRC)	Flattening % at Test Temperature	
	T (°F)	Time (h)		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
CD/ANN	1900	1	---	---	40
CD/ANN	2000	1	---	---	52
CD/ANN	2150	1	---	---	62

Task 5: High Temperature Corrosion Limits of MA956

Laboratory Testing for Working Fluid Side: The exposure of samples of MA956 bar in air at 1300°C until failure was completed. The total mass change data are shown in Figure 21, and the observed and predicted lifetimes as a function of specimen thickness are shown in Figure 22. The lifetime calculations were based on a two-stage oxidation model developed using data for the MA956HT alloy, but using the oxidation rates (parabolic and linear stages) and transition oxide thickness measured for the MA956 alloy. The fit of the model appears to be good, except for the thinnest specimen. The only difference from the fit used for the MA956HT alloy was a change in the factor that qualifies the rate exponent in the 'linear' oxidation phase (f_{n3}) from 0.06 to 0.03 (see Fig. 22). Obviously, the oxidation lifetime of the MA956 alloy at 1300°C in air is significantly lower than that of the MA956HT alloy. The important difference between the two alloys appears to be that the rate of oxidation (in Stage 2 and Stage 3) is higher for the MA956 alloy. Since both alloys form the same oxide, the reason for this difference is not obvious. The oxidation rate of alumina-forming alloys can be significantly affected by doping with reactive elements such as Y, but the Y levels of the MA956 and MA956HT alloys are essentially the same (0.40 and 0.38 weight percent, respectively). Specimens have been prepared for examination of the structure and compositions of the alloy-oxide interface and the oxide grain boundaries by TEM.

Figure 21. Total mass gain data for the MA956 alloy at 1300°C, 100 cycles in air.

Figure 22. Comparison of oxidation lifetimes of the MA956 and MA956HT alloys at 1300°C in air as a function of specimen thickness. The data points indicate observed lifetimes. The solid line is the fit of a two-stage model developed from data on the MA956HT alloy.

Further specimens are being exposed at 1250, 1200, and 1100°C in air to determine lifetimes with five samples (out of a total of seven) of different thickness taken from MA956 bar stock having failed at 1250°C. The observed oxidation-limited lifetimes are shown in Figure 23 with data points for a similar set of samples of the MA956HT alloy included for comparison. Note that the data points with arrows indicate runs that are still in progress, and that the oxidation lifetime will be longer than presently indicated by those points. The lifetimes of MA956 and MA956HT at 1250°C appear to be more than double those at 1300°C. The coincidence of the 1250°C lifetimes for MA956 with the 1300°C data points for MA956HT indicate that the latter alloy has a 50°C advantage at these very high temperatures. Analysis of the kinetic data to generate the data needed for testing or modifying the lifetime model has not yet been completed. However, a procedure suggested by Monceau and Pieraggi¹ for deducing the ‘true’ parabolic rate constant, k_p , from experimental kinetic data has been examined using the oxidation data for MA956 and MA956HT. The ‘true’ parabolic rate constant is not influenced by any initial, rapid mass gain from transient oxide formation, which often can bias the value usually calculated. Initial results suggest that the use of the values of the true k_p in the life prediction calculations allows an improved fit to the observed lifetime data for MA956 and MA956HT at both 1250 and 1300°C.

Figure 6. Plot of oxidation lifetimes as a function of sample thickness for the MA956 and MA956HT alloys at 1250°C and 1300°C in air.

And finally, further modification of the lifing model has involved the replacement of the specimen thickness term by a ‘shape factor’ (volume/surface area). The lifetime data generated on MA956HT in an ORNL in-house program used disc-shaped specimens, whereas the MA956 specimens are parallelepipeds, so that there existed the possibility that the comparison of oxidation behavior was influenced by specimen shape. The range of V/A values of the specimens used so far is 0.03-0.07 cm (discs) and 0.02-0.08 cm (parallelepipeds). Preliminary calculations using the revised model indicate a reasonable fit to the experimental data at 1300°C, without the use of any ‘adjusting’ parameters, i.e., assuming strictly parabolic ($n=0.5$) and linear ($n=1$) segments to the kinetic curves.

Laboratory Testing for Fireside Environment: The laboratory testing using two different flue gases and three different deposits (see Table 9 and 10 below) at 2000°F continues. The exposure of the samples is terminated every 100 hours for deposit replenishment and at 500 hours one set of samples was removed for metallurgical examination. To date the test has completed 600 hours of the 1000-hour exposure and metallurgical examination of the 500-hour samples has been initiated.

Table 9
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 ₂ O	10	5
SO ₂	0.25	1.0
N ₂	Bal	Bal

Table 10
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 first utility with the appropriate locations for the air-cooled probes have been determined, the control hardware procured, and the probe design finalized and assembly initiated.

CONCLUSIONS

At this early stage of the project, it is premature to make any technical conclusions, however the TEM results on samples that have undergone creep is providing valuable information on the mechanical behavior of MA 956. Using significant levels of tube reduction followed by a high temperature anneal does result in an onion skin texture in the transverse direction of tube reduction. Also, results from the friction, explosion, and TLP joining studies continue to show promise in this area.

REFERENCES

1. D. Monseau and B. Perragi, Oxid. of Met., vol. 50, no. 5-6, pp. 477-493, Dec. 1998.

RECOMMENDATIONS

Subtask 2.1: Effect of Extrusion and Thermomechanical Processing (TMP)

Parameters on Tubing Produced from “Standard” MA956 Powder

2.1.1: Development of an ODS Tube with “Pancake-Shaped” Grains in the Transverse Direction [Complete]

2.1.1.2: UCSD Microstructural Evaluation [95% Complete, recommended for completion.]

2.1.1.3: Produce 8 Tubes at ORNL Based on 2.1.1 and 2.1.1.2. [Hold pending assessment of results above.]

Subtask 2.1.2: Development of an ODS Tube with a Gradient Grain Morphology (GGM) in the Transverse Direction [Reassess based on results by flow forming and the desirability of a fine grain on the O. D. and I. D.] [This will impact on the activities at UCSD 2.1.2.2 and 2.1.2.3.2 plus tube production at ORNL 2.1.2.3.1.]

Subtask 2.2: Powder Processing Modifications of Current MA956 Powders [Delete this activity due to lack of resources at Special Metals and now deemed unnecessary for achieving project objectives since the achievement of the “onion-skinned” transverse microstructure has been demonstrated by flow forming.] [The work scope at UCSD and ORNL will be impacted.]

Subtask 2.3: Modifications to Current MA956 Powders [Given the success with the current powder product and the lack of resources at Special Metals it is recommended that this subtask be deleted.] [The work scope at ORNL and UCSD will be impacted.]

ESTIMATED BUDGET REDUCTION PENDING ADOPTION OF RECOMMENDATIONS

Subtask 2.1: Funding may be redirected towards use of flow forming. Therefore funding for this subtask may warrant reassessment.

Subtask 2.2: Deletion of this activity would reduce the total project funding by \$193,997 per original budget plan.

Subtask 2.3: Deletion of this activity would reduce the total project funding by \$200,564 per original budget plan.

Development of ODS Alloy for Heat Exchanger Tubing

I. G. Wright, B. A. Pint, and C. G. McKamey
Oak Ridge National Laboratory
March, 2003

Task 1. Review of the ODS alloy processing procedures from the COST-501 Program

Attempts were made to contact the relevant personnel associated with the development of ODS alloy tubing used in the COST-501 program. Those then employed by Dour Metal S.A., in Belgium, have returned to the Slovak Republic and are untraceable (B. Kazimierzak) or are deceased (J. M. Prignon). The British Gas personnel involved in constructing the heat exchanger (F. Starr and Q. Mabbutt) have been contacted and interviewed, but were involved with Dour Metal only in the final phases of producing tubes. A search of the literature found a number of publications by Dour Metal, which have been obtained.

Task 2a. Creep Threshold

Since the improvements sought in MA956 in this program are primarily in its high-temperature creep strength, particularly in the transverse direction, the concept of a 'creep threshold stress' developed in an ORNL internal program was extended for use as a relatively rapid procedure for quantifying such improvements, as well as for evaluating joints produced by other participants in the program. A peculiar characteristic of ODS alloys is that they are extremely sensitive to strain rate, such that when loaded at stresses beneath some threshold they undergo creep at an extremely low rate whereas, at loads above the threshold, rapid creep to failure ensues. The creep response of ODS alloys is governed not only by the ability of the oxide dispersion to inhibit dislocation movement in the alloy matrix, but also by void formation and coalescence in the alloy grain boundaries, with each mechanism contributing to an extent determined by its own strain rate sensitivity. The number of grain boundaries oriented normal to the principal stress direction, and the shape of the grains, therefore exerts a strong influence on the creep strength of these alloys; any reduction in the number of axially-oriented grain boundaries in tubes would be expected to lead to an improvement in transverse creep strength, which is a major goal of this program.

For these reasons, tensile tests are not a good measure of the effectiveness of microstructural modifications on the creep behavior of these alloys, and some form of creep test is required. The sequentially-loaded creep test adopted here provides a creep-type failure in a relatively short time (but still several hundreds of hours). The effort in this Task involved the establishment of 'creep threshold' values for the baseline MA956 alloy as a function of temperature. Comparison of the behavior of modified versions of the alloy with these baseline data is intended to provide a quantitative measure of the improvement accomplished. Figure 1 illustrates the temperature-dependence of the 'threshold stress' for two sets of 956 tubing (2.5 cm diam. x 2 mm wall, and 6 cm diam. x 5 mm wall), tested in the axial direction, as well as for the larger diameter tubing tested in the transverse direction (using samples taken from flattened tubing). Because of the sensitivity to alloy microstructure, the grain sizes of the alloys used to determine these thresholds are also shown.

Task 2b. Novel Transient Liquid Phase Joining Technique

Work has been initiated to investigate the applicability of a transient liquid phase (TLP) joining approach developed by N. S. Bornstein (ex-United Technologies Research Center) for use with a chromium oxide-forming ODS alloy in the HiPPS program. Since alloy MA956 forms a more thermodynamically stable

Figure 1. Summary of creep threshold data for MA956

and tenacious aluminum oxide scale, joining it is expected to present a greater challenge. For a successful joint, the TLP alloy must fully wet the surfaces to be joined, allowing the formation of a metallurgical bond at a temperature well below the melting point of the constituent phases of the parent alloy, and then rapidly diffuse away from the joint so that the melting temperature of the joined area is restored to that of the parent alloy, while the amount of residual TLP materials near the joint is sufficiently low that the mechanical properties are not compromised. A model TLP alloy based on a precious metal was cast and rolled to thin sheet (50-75 μm thick) for initial trials. A joint made in MA956 tubing appeared to be sufficiently promising that further efforts were made to produce thinner sheet stock of the model TLP alloy, which was used to produce joints that will be subjected to detailed analysis to derive information useful for optimizing the technique.

Task 3. Environmentally-Dictated Temperature Limitations

The oxidation behavior of MA956 and 956H is being studied in air over the temperature range 1000 to 1300°C, with emphasis on quantifying the rate of oxidation and expected lifetime as a function of temperature. This requires understanding the relationships among the form of the kinetic curves, the scale morphology, and the depletion of Al in the alloy matrix. Specimens are exposed in individual, lidded alumina crucibles to allow measurement of total oxygen uptake as well as the extent of scale spallation; the exposures are made in cycles of 100 h (500 h at 1000°C), with weighing and inspection at the end of each cycle. Multiple specimens of different thickness are being exposed to determine the actual oxidation-limited lifetimes, and to provide information on the changes associated with the different stages of oxidation.

Figure 2 summarizes the kinetic data for the base alloy, MA956, and for a higher-Al modification, MA956H. The clear indication is that 956H has a consistently slower oxidation rate than 956. The form

Figure 2. Summary of oxidation kinetics from cyclic oxidation tests in air

of the oxidation kinetics was found to be the same for both alloys in the range 1200 to 1300°C, and involves four distinct stages:

- Stage 1 is a 'transient' oxidation stage during which the main protective oxide scale is established;
- Stage 2 is a period of protective scale formation (parabolic oxidation; rate constant k_2) with essentially no scale spallation;
- Stage 3 involves essentially linear kinetics (rate constant k_3) due to the influence of scale spallation; and
- Stage 4 is acceleration of the oxidation rate to breakaway.

Stages 2 and 3 are considered the most important, and were addressed in a separate effort at ORNL to develop a mathematical model of the oxidation behavior.

The temperature dependence of the rate constants for these stages were determined from the Arrhenius plots shown as Figs. 3 and 4. These indicated different activation energies for the two alloys for Stage 2 suggesting some difference in the rate-controlling mechanisms, whereas the activation energies for Stage 3 were similar, with 956 exhibiting a consistently faster rate. The scale thickness at which the transition from stage 2 to 3 occurred ($\ell_{2,3}$) also appeared to be temperature-dependent. The current expression for oxidation-limited lifetime based on this multi-stage model (from ORNL studies) is:

Figure 3. Arrhenius plot for Stage 2 parabolic kinetics

Figure 4. Arrhenius plot for Stage 3 linear kinetics

$$\text{Time to breakaway (t}_b\text{)} = \left\{ \left[\frac{S \cdot 10^{-4} \cdot \rho_A \cdot A_{\square} \cdot e^{-Q_{\square}/(1.987 \cdot T)}}{3600 \cdot A_2 \cdot e^{-Q_2/(1.987 \cdot T)}} \right]^2 + \left\{ \frac{1}{3600 \cdot M} \cdot \left(\frac{V}{A} \right) \cdot \left(\frac{\rho_M}{A_3 \cdot e^{-Q_3/(1.987 \cdot T)}} \right) \cdot [(C_{B_0} - C_{B_b}) - M \cdot S \cdot 10^{-4} \cdot (A/V) \cdot (\rho_A/\rho_M) \cdot A_{\square} \cdot e^{-Q_{\square}/(1.987 \cdot T)}] \right\} \right\} \text{ hours}$$

where: A = the area exposed to oxidation (cm²)

V = the volume of alloy being oxidized (cm³)

C_{B₀} = the initial mass fraction of Al in the alloy

C_{B_b} = Al mass fraction at which a protective Al₂O₃ can no longer form

ρ_A is the density of alumina = 3.965 gcm⁻³ (used in the absence of an actual value for a thermally-grown oxide)

ρ_M = the density of the alloy (= 7.2 gcm⁻³)

S converts mass of Al₂O₃ formed to mass gain of oxygen (= 0.470)

M = converts mass gain of oxygen to mass of Al consumed (= 1.1246)

A₂ and A₃ are the Arrhenius constants for k₂ and k₃

A_□ is the Arrhenius constant for ρ_{□,3}

Q₂, Q₃, and Q_□ are the activation energies for k₂, k₃ and ρ_{□,3}, respectively

T is the metal temperature (°K)

The value for C_{B_b} has proven difficult to measure experimentally, since a number of changes occur in the alloy beneath the protective scale immediately ahead of breakaway. Nevertheless, measurements made so far suggest values near to zero (approx. 0.001-0.002) when breakaway not influenced by mechanical processes such as gross scale spallation. This value is significantly lower than that used by other researchers (approx. 0.024); it is not yet known if C_{B_b} changes with temperature. Lifetimes predicted for MA956 as a function of temperature (using the new value of C_{B_b} and assuming no temperature-dependence) in the two-stage model are shown as dashed lines in Fig. 5, while actually observed times to breakaway oxidation for specimens of different thickness are shown as individual data points. The model slightly over-predicts the observed lifetimes at 1200-1300°C, especially for the lower V/A values (thinner specimens); this also appears to be the case at 1100°C, although the exposures are incomplete. It remains to be seen how well the predictions compare with the actual lifetimes at the lower temperatures that are more likely to be experienced in practical use.

Figure 5. Actual and predicted oxidation-limited lifetimes for MA956

Similar data for MA956H are shown in Fig. 6, for which fewer specimens have reached the breakaway condition. Nevertheless, the model over-predicts the lifetimes for 956H at 1300°C. Examination of the mode of failure of these alloys indicates some effect of specimen shape, such that failures tend to initiate at specimen corners. Efforts to further refine the model involve approaches to provide a more rigorous description of the influence of specimen shape.

Figure 6. Actual and predicted oxidation-limited lifetimes for MA956H

A major finding from this study is that, while it was expected that the oxidation-limited lifetime of MA956 would be shorter than 956H because of its lower Al content (4.5 vs 5.7%), the shortfall was greater than expected from a simple reduction in Al reservoir. The difference in predicted lifetimes is illustrated in Fig. 7. As noted in Fig. 3, there appears to be a difference in the diffusion process that controls the oxide growth rate in Stage 2 in the two alloys. Inspection of the detailed composition of the

Figure 7. Comparison of predicted oxidation-limited lifetimes for MA956 and MA956H

alloys does not suggest any obvious reason for this. As a means of understanding the reasons for this important difference, specimens have been exposed to provide scale thicknesses suitable for examination by transmission electron microscopy. Cross sections including the oxide-metal interface have been made and the detailed scale structures and compositions, especially of the oxide grain boundaries, will be measured.

March 19, 2003

Gaylord Smith
Special Metals Corporation
3200 Riverside Drive
Huntington, WV 25705

Dear Gaylord:

As we discussed earlier today, I have enclosed a brief status report on the Incoloy® MA 956 joining work currently being done at Michigan Technological University under Special Metals P.O. Number 222976. If you have any additional technical questions about this work, please feel free to contact me. If you have additional questions about contractual issues, please contact Mike Gilles at either (906) 487-2225 or mgilles@mtu.edu.

Sincerely,

Marv McKimpson

Marvin McKimpson, Ph.D.
Principal Investigator
(906) 487-1825 (voice)
mmckimp@mtu.edu (email)

— Subcontractor Status —
Michigan Technological University

The objective of the work at Michigan Technological University under the Vision 21 program “Development of ODS Heat Exchanger Tubing” is to develop a novel hybrid transient liquid phase/diffusion bonding process for joining Incoloy[®] MA956 tubing. The University was tasked to do three things:

- ❖ Develop a suitable transient liquid phase (TLP) bonding process for joining Incoloy[®] MA956 tube to MA956 tube so that the materials can be used in a recrystallized condition. This work was originally scheduled to require 12 months and cost \$148,700. (Task 3.2.1—ODS Tube-to-ODS-Tube TLP Bonding)
- ❖ Develop a suitable transient liquid phase (TLP) bonding process for joining Incoloy[®] MA956 tube to a heat-resistant header alloy such as Inconel[®] Alloy 601. This work was originally scheduled to begin in Month 4 of the project and continue through Month 15. It was to cost \$119,400. (Task 3.2.2—ODS Tube-to-Heat Resistant Alloy TLP Bonding)
- ❖ Produce 10 Incoloy[®] MA956 tube-to-MA956 tube joints and 10 Incoloy[®] MA956 tube to heat-resistant header alloy (Inconel[®] Alloy 601) joints for elevated temperature tensile and creep testing at Oak Ridge National Laboratory. This work was to cost \$24,500 for production of the MA956-to-MA956 tube joints and \$28,800 for production of the MA956-to-header alloy joints. Production of each set of samples was anticipated to require about 6 months. (Task 3.4.1 and 3.4.2)

At present, Task 3.2.1 — ODS Tube-to-ODS-Tube Bonding, is nearly complete. A diffusion bonding protocol has been developed which enables recrystallized MA956 tubes to be joined

Figure 1. Two recrystallized Incoloy[®] MA956 tubes joined using the “3-body” diffusion bonding process developed under this program (left) and a micrograph of the joint showing grain growth across the interface (right).

with grain growth across the joint interface. As shown in Figure 1, the process involves joining two as-machined Incoloy[®] MA956 surfaces using an intermediate tapered collar of unrecrystallized material. After bonding, the joint is given a conventional recrystallization anneal to promote grain growth across both recrystallized/unrecrystallized interfaces.

Status

As of February 28, 2003, Michigan Technological University has incurred costs totaling \$163,624.50 under the program. This total includes \$32,931.97 in unpaid expenses incurred prior to the Special Metals bankruptcy filing on 27 March 2002. Task 3.2.1 of the program is nearly complete; the only work remaining is to perform room temperature tensile tests on the bonded joints.* Work is currently underway on development of the ODS tube-to-heat resistant alloy joints to be fabricated under Task 3.2.2 of the program. It is anticipated that these joints will be produced using techniques similar to those developed under Task 3.2.1. Both Tasks 3.2.1 and 3.2.2 are expected to be completed within 6 months.

Under the original program, fabrication of the 20 test joints (10 joints/set) called out under Tasks 3.4.1 and 3.4.2 was scheduled to require 1 year (6 months/set). At that time, however, the required test coupon geometry had not yet been fully determined. If these test joints can be machined out of longer versions of the tubular joints shown in Figure 1, they can be fabricated more quickly than called out in the original program. In this case, these 20 joints can be fabricated and shipped within 2 to 3 months after completion of Task 3.2.2.

As per the current subcontract with Special Metals, total additional cost for completion of Tasks 3.2.1, 3.2.2, 3.4.1 and 3.4.2 will be \$157,694.† (Note, however, that wage rates, fringe benefit rates and federally-approved overhead rates at Michigan Technological University have changed since the original subcontract was issued. If a new contract is executed with Michigan Tech, some of these costs may be subject to renegotiation.)

* As called out in the original Statement of Work, studies were carried out on boriding and transient liquid phase bonding of the ODS material. All of the boriding techniques investigated, however, produced significant residual borides along the joint line. To avoid these detrimental Fe borides, no transient liquid phase was used for the final bonding protocol developed. Using the tapered collar of unrecrystallized material also avoided the need for conducting bonding trials on both recrystallized and unrecrystallized materials. Accordingly, the work performed under Task 3.2.1 developed the required technical information, although the actual tests performed did not conform exactly to those called out in the original SOW.

† This represents the original program value of \$321,318 minus the \$163,624 expended through 2/28/03. It does **not** include the \$32,932 in unpaid invoices still owed Michigan Tech due to the Special Metals bankruptcy filing.

Development of ODS Heat Exchanger Tubing

Project Status Update

By
University of California-San Diego
Bimal K. Kad, P.I.

The University of California-San Diego is engaged as a subcontractor to Special Metals Corporation (SMC) in the research and development of DOE funded Vision-21 specific materials technologies for heat exchangers. UCSD discharges its task responsibilities in coordination with SMC and Oak Ridge National Laboratory (ORNL). The following is a brief summary of our task performance status update through March 31st, 2003.

Tasks	Description	Status
2.1.1.2	microstructure analysis of rods	540 x 2 =1080 samples 95% completed
<p>This task is in co-ordination with SMC. All samples for analysis are provided by SMC for UCSD to analyze. Metallographic samples generated in the 540 conditions are being examined in the longitudinal (L) and transverse (T) orientations for a total of 1080 unique samples. The last batch of samples from SMC was made available on March 1st 2003 and we are currently in the process of completing this task</p>		

2.1.1.3	extrude tubes & microstructure	tube shell extrusions in-process, 30% completed
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This task is in co-ordination with SMC and ORNL. UCSD initiated the task with designing and producing tube shells for extrusions. These were subsequently shipped to SMC in June 2002 for powder filling and encapsulation. SMC shipped the cans to ORNL for extrusions in July 2002. However, the extrusion press at ORNL was undergoing repairs and not available for the balance of 2002. The press is now operational, extrusion are planned for the March/April 2003 time frame. Tube shell extrusion cans for each subsequent extrusion tasks have been manufactured by UCSD.

2.2.2	microstructure analysis of rods	0% complete
2.2.3	extrude tubes & microstructure	20% complete
2.2.4	rupture testing- microstructure analysis	25% complete for ORNL

This task is in co-ordination with SMC and ORNL. No samples received from SMC for task 2.2.2. Tube shells are available (made by UCSD) for task 2.2.3. ORNL is currently testing existing 2-1/2" tube made available by SMC under task 2.2.4. UCSD is providing TEM microstructure analysis of deformation and failure behavior in these tests.

2.3.2	microstructure analysis of rods	0% complete
2.3.3	extrude tubes & microstructure	20% complete

This task is in co-ordination with SMC and ORNL. No samples received from SMC for task 2.3.2. Tube shells are available (made by UCSD) for task 2.3.3

3.5.3	elevated temperature testing deformation & failure characterization	TEM study of ORNL tests 35% complete
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UCSD has a support role under task 3.5.3 assisting ORNL with microstructural analyses. Our analyses include understanding the deformation behavior in longitudinal and transverse oriented specimens in as processed MA956 tubes as well as joined segments of tubes. UCSD is mapping the mechanisms responsible for the (L) vs. (T) strength and creep anisotropy and the origins of failure in L, T, oriented loadings. In addition, UCSD has performed non-fusion joining trials of unrecrystallized 2-1/2" OD, 1/4" thick wall MA-956 tubes. Such joints have been tested for integrity via room temperature bend tests.

Task Completion Forecast

The lack of timely availability of the extrusion press at ORNL for program requirements have led to significant delays. The press was under repair for much of 2002 and was only brought back on line in early 2003. Task 2.1.1.3 extrusions will begin in late March 2003 and we anticipate the remaining extrusion tasks 2.2.3 & 2.3.3 to follow shortly thereafter.

Fiscal Update:

The original fixed subcontract costs are \$302,413. Through the performance period of March 31st, 2003 our total expenses are \$165,183.00. This includes \$14,878.20 of work billed in the first quarter of 2002 but unpaid due to SMC bankruptcy proceedings. It is estimated that completion of the remaining tasks as defined will require \$150,000.00. This entails a project performance period extension through December 31st 2004.

Bimal K. Kad, Ph.D.
March 20th, 2003