DEVELOPMENT OF ODS HEAT EXCHANGER TUBING

Quarterly Technical Progress Report

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ABSTRACT

Work has begun under three major tasks of this project. With respect to increasing the circumferential strength of a MA956 tube, approximately 60 MA956 rods have been extruded using a 20:1 extrusion ratio and extrusion temperatures of 1000, 1075, 1150, and 1200°C. Also, creep testing is underway for the purpose of determining the "stress threshold" curves for this alloy. Regarding joining of the alloy MA956, work has begun on the friction welding, magnetic impulse welding, explosive welding, and transient liquid phase bonding aspects of this project. And finally, material is being prepared for the laboratory fire-side high temperature corrosion tests, with potential gas and deposits for a typical Vision 21 plant being reviewed for final determination of these variables in the test program.

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INTRODUCTION

This research is seeking to develop a MA956 heat exchanger tube which 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 are discussed below.

Task 2: Improvement of Circumferential Creep Strength of MA956 Tubes

The following matrix of tests shown in Table 1 below are currently being performed at HA. Extrusions using a 20: 1 extrusion ratio and extrusion temperatures of 1000, 1075, 1150, and 1200°C have been completed.

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	10
1200		15		
		25		

Creep testing is underway at ORNL for the purpose of determining the "stress threshold" curves of the MA956 alloy. A creep specimen cut from the axial direction of a nominal 1 inch diameter is currently being tested at 1000°C. The initial stress was 3 ksi, with incremental load increases leading to a present stress of 5 ksi being tested.

Task 3: Joining

EWI: Difficulties have been experienced in cutting the tubing to be used in preliminary friction welding trials (small cracks were produced on the cut surfaces) and thus special saw blades have been ordered to accomplish this task. Modeling activities have been completed for the design of a coil to be used in the magnetic impulse welding trials. Materials for the coil have been purchased with initial welds anticipated in mid April. And setups for four explosion welding trials are currently being prepared.

MTU: Recent work has included boriding trials of the MA956 material in which a fairly uniform boride layer 5-10 microns thick has been produced. Although this layer is substantially thinner than previous trials, it is still thicker than desired for diffusion bonding. In addition to the boriding trials, progress has been made in developing metallographic techniques that reveal the grain structure of the material.

Task 4: Bending of MA956 Tubes

No experimental work has been accomplished on this task during this reporting period.

Task 5: High Temperature Corrosion Limits of MA956

FWDC has been cutting specimens from ODS material received from Special Metals. Additionally typical gas and deposit analysis for a typical VISION 21 plant are being reviewed for final determination of their laboratory corrosion test.

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

The as-extruded rods produced using a 20:1 extrusion ratio and extrusion temperatures of 1000, 1075, 1150 and 1200°C all exhibit similar fine grained microstructures. The microstructure of the rods to be subjected to 0% cold work + different recrystallization treatments will provide information as to the importance of extrusion temperature at this extrusion ratio.

Due to the fact that the oxide dispersoids in an ODS alloy are typically stable up to the melting point of the alloy, the strength of these materials is relatively independent of exposure to elevated temperatures. Thus the slope of a typical stress to cause rupture curve for an ODS alloy is much less than that for a traditional wrought alloy. Figure 1 shows this difference by comparing the allowable stress for a 2 inch OD x 0.25 inch wall thickness tube made from a range of wrought versus ODS alloys[1]. One consequence of this relatively "flat" stress to cause rupture curve, is extremely long times can be required to cause rupture if the applied stress is below a "threshold" value below which 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. Currently ORNL has cut samples from the axial direction of nominal 1 inch diameter tube of the alloy MA956 and is testing a

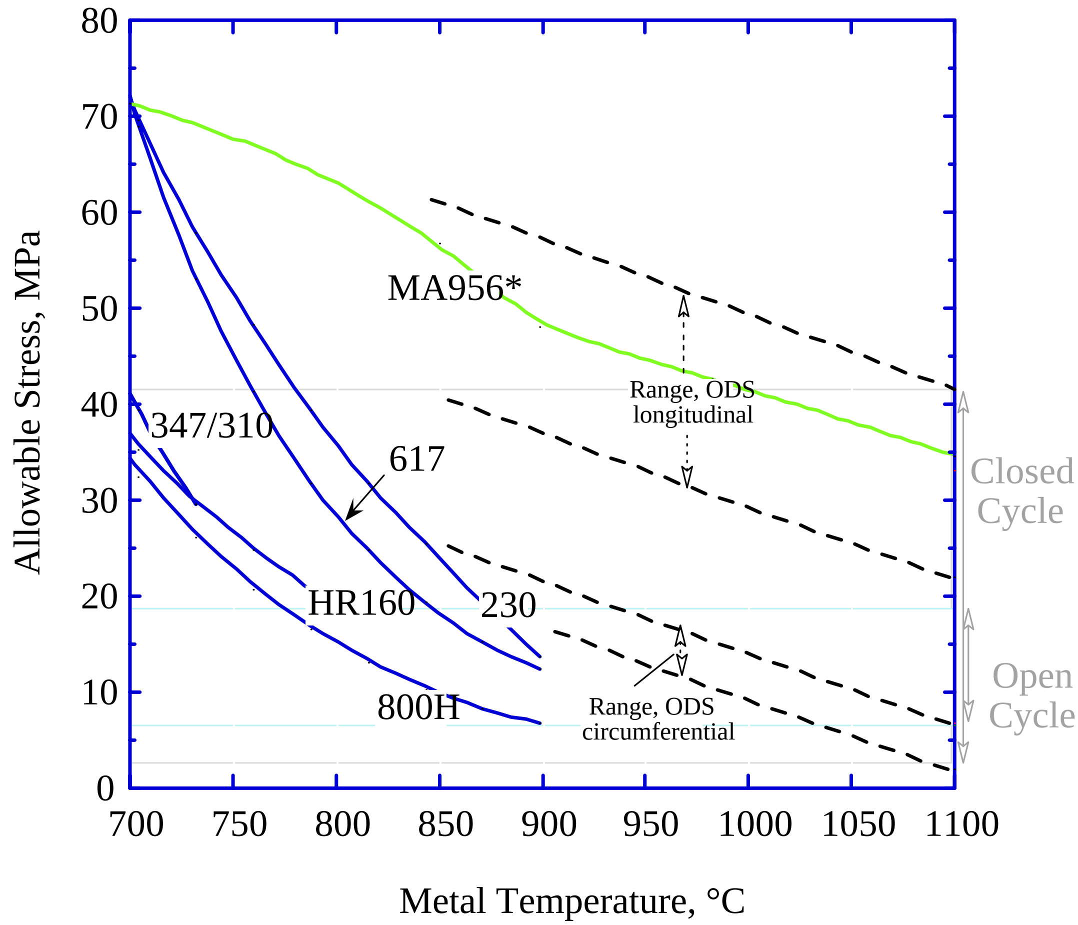

Figure 1. Allowable Stresses for Qualified and Pending ASME Boiler and Pressure Vessel Code High-Temperature Alloys (*data for MA956 and other ODS alloys were based on 2/3 10,000 hr creep rupture strength)[1].

sample at 1000°C. The initial load on this sample was 3 ksi with testing at 5 ksi in progress.

In addition to generating the stress threshold curves discussed above, ORNL has performed a literature search on a European COST-501 program that investigated the use of ODS alloys for heat exchanger tubing, plus other European work associated with ODS alloy development. As of this date, a total of 23 papers have been identified and are currently being obtained and reviewed[2-24]. Notes on pertinent findings will be reported in the future.

Task 3: Joining

Although ODS alloys can be successfully fusion welded, such joints have a significantly reduced high temperature load-bearing capability. This is because fusion locally destroys the controlled distribution of the dispersed phase and disrupts the continuity of the microstructure, which are the essential features that provide high-temperature creep strength. Furthermore, fusion welding can result in cracking at grain boundaries. Solid-state joining processes are more successful than the melt-based techniques and thus four solid state joining techniques are being investigated in this work: friction welding, magnetic impulse welding, explosive welding, and transient liquid phase bonding (TLP).

EWI has experienced difficulties in cutting MA956 tubing for preliminary friction welding trials. Attempts to cut the material have resulted in small cracks on the cut surfaces, a problem not uncommon with this material, and thus special saw blades have been ordered to alleviate this problem. Regarding the efforts on magnetic impulse welding of the MA956 alloy, modeling activities have been completed for the design of a coil to use in this process and machining of the coil is in process with anticipated joining trials to begin in April.

Regal Technologies (a subcontractor to EWI) is currently making final preparations for four preliminary explosion welding trials using plate material instead of tubing. These four welds will attempt to bond alloy MA956 to alloy MA956 and alloy MA956 to alloy 601. One set of welds will be produced using low pressure and kinetic energy to make the bond and one set using high pressure and kinetic energy. The welds will then be characterized with respect to the weld interface morphology, extent of work hardening, and response of the weld microstructure during subsequent heat treating as a function of explosion parameters. These initial four explosion welds are planned for mid April.

MTU is investigating an innovative approach to bond the ODS tubes by using a combination of the TLP and diffusion bonding processes. First, carefully controlled boriding diffusion pretreatments are being studied in order to introduce boron and silicon into the joint interface. This boriding approach will then be coupled with demonstrated diffusion bonding techniques to produce a hybrid process capable of accommodating wider ranges in joint fit-up and achieving more complete grain growth across the prior interface than can be attained using the diffusion process alone. Work accomplished up to this date includes boriding trials of the MA956 material in which a fairly uniform boride layer 5-10 microns thick has been produced. Although this layer is substantially thinner than previous trials, it is still thicker than desired for diffusion bonding. In addition to the boriding trials, progress has been made in developing metallographic techniques that reveal the grain structure of the material.

Task 4: Bending of MA956 Tubes

The effect of strain experienced during the bending of tubes is of critical importance to designers of heat exchangers. Therefore, FWDC will bend MA956 tubes imposing 5, 10, 15, 20, and 25% strain on the material and then expose them to 1204^oC (2200^oF) for 100 hours. Analysis of the tube microstructure before and after the exposure will be conducted in order to determine the maximum amount of strain that can be induced into a MA956 tube without recrystallization occurring during operation. Presently, FWDC is waiting to receive tubing in order to start testing under this task. The tubing has been ordered and the first shipment is due to arrive at HA in April, after which it will be shipped to FWDC.

Task 5: High Temperature Corrosion Limits of MA956

Although the high-temperature corrosion resistance of alloy MA956 at very high temperatures in oxidizing atmospheres is well known, there is very little experience with the performance of this material in actual boiler environments at the high temperatures required in an expected Vision 21 plant environment. Thus this task involves complementary laboratory and field exposures in environments that are expected to be encountered by the external and internal surfaces of MA956 tubes in service.

FWDC has been cutting specimens for their laboratory corrosion testing. Also, a review of possible gas and deposit analyses for a typical VISION 21 plant is in progress for final determination of these variables in their laboratory corrosion test.

CONCLUSIONS

No technical conclusions are available at this time. Work has been initiated under Tasks 2, 3, and 5, with Task 4 waiting on material delivery.

REFERENCES

1. Wright, I. G. and Stringer, J., “Materials Issues for High-Temperature Components in Indirectly-Fired Cycles”, ASME Paper No. 97-GT-300 (1997).
2. B. Kazimierzak and J. M. Prignon, “Development of ferritic ODS materials for application above 1100°C,”
3. B. Kazimierzak, M. Prignon, C. H. Lecompte-Mertens, and D. Coutsouradis, “Iron-based ODS alloys with improved properties,” Tube International (UK), 9 (39), 319-321 (1990).
4. B. Kazimierzak, M. Prignon, C. H. Lecompte-Mertens, and D. Coutsouradis, ”Fe-base ODS alloys with improved mechanical strength,” Pp. 131-142 in Proc. COST

501/505 Conf. on *High-Temperature Materials for Power Engineering, 1990*; Kluwer Academic Publishers, Dordrecht, The Netherlands (1990).

5. B. Kazimierzak, M. Prignon, C. H. Lecompte-Mertens, and D. Coutsouradis, "Iron-base ODS alloys with improved mechanical strength," Anti-Corrosion Methods and Materials, 37 (10), 4-6 (1990).
6. B. Kazimierzak, M. Prignon, C. H. Lecompte-Mertens, and D. Coutsouradis, "Oxide dispersion-strengthened iron-based PM alloys combine high strength and oxidation resistance for high-temperature applications," Industrial Heating, 57 (10), 42-44 (1990).
7. B. Kazimierzak, M. Prignon, F, Starr, L. Coheur, C. H. Lecompte, D. Coutsouradis, and M. Lamberigts, "Development of ferritic ODS tubes for heat exchangers operating above 1100°C (retroactive coverage)," Pp. 137-145 in Proc. Conf. on *Structural Applications of Mechanical Alloying*, Myrtle Beach, South Carolina, 27-29 Mar. 1990; ASM International, Materials Park, Ohio (1990).
8. B. Kazimierzak, M. Prignon, C. H. Lecompte-Mertens, and D. Coutsouradis, "Iron-base ODS alloys with improved mechanical strength," Powder Metallurgy International, 23 (1), 41-44 (1991).
9. B. Kazimierzak, M. Prignon, C. H. Lecompte-Mertens, and D. Coutsouradis, "Oxide dispersion-strengthened alloys based on iron and with improved mechanical strength," Roestvast Staal, no. 3, 43-47 (Mar. 1991).
10. B. Kazimierzak, J. M. Prignon, and R. I. Fromont, "An ODS material with outstanding creep and oxidation properties above 1100°C," Pp. 363-372 in Proc. Conf. on *Synthesis, Processing, and Modelling of Advanced Materials*, Paris, France, 11-13 Sep. 1991; Trans Tech Publications Ltd., Aedermannsdorf, Switzerland (1993).
11. B. Kazimierzak, J. M. Prignon, and R. I. Fromont, "An ODS material with outstanding creep and oxidation properties above 1100°C," Pp. 9 in Proc. Conf. on *PM Aerospace Materials*, Lausanne, Switzerland 4-6 Nov. 1991; MPR Publishing Services Ltd., Shrewsbury, England (1992).
12. B. Kazimierzak, J. M. Prignon, and R. I. Fromont, "An ODS material with outstanding creep and oxidation properties above 1100°C," Materials and design (UK), 13 (2), 67-60 (1992).
13. J. J. Huet, "Dispersion-strengthened ferritic steels for fuel elements in fast reactors," ATB Metallurgie, 10 (4), 126-130 (1970).
14. J. P. Breyer, J. M. Diez, V. Leroy, J. J. Huet, and L. Habraken, "Ferritic steel alloys containing Ti and Mo as major alloying additions," CRM Journal, Sept. 1973, (36), 67-75 (1973).
15. J-J. Huet, P.H. VanAsbroeck, and V. Leroy, "Dispersion-strengthened ferritic steels as fast reactor structural materials," Am. Nucl. Soc. Trans., 17, 200 (1973).
16. J-J. Huet and V. Leroy, "Dispersion-strengthened ferritic steels as fast reactor structural materials," Nuclear Technology, 24, 216-224 (1974).
17. M. Snykers and J-J. Huet, "Dispersion-strengthened ferritic alloys for high-temperature application," Pp. 237-241 in *Creep Strength in Steel and High-Temp. Alloys*, Metals Society, London (1974).
18. J-J. Huet, L. DeWild, and V. Leroy, "Ferritic steel strengthening for nuclear application," Metallurgie XIX (1/2), 37-41 (1979).

19. M. Lamberigts, V. Leroy, L. Habraken, C. Driesen, and J. J. Huet, “Dispersion-strengthened ferritic alloys with high damping capacity for high-temperature applications,” Pp. 385-393 in Proc. Conf. on *Behaviour of High-Temperature Alloys in Aggressive Environments*, Petten, The Netherlands, Oct. 1979 (1980).
20. J-J. Huet and CH. Lecompte, “Ferritic steels for fast reactor canning material and turbine applications,” Proc. ASM Conf. on *Ferritic Steels for High-Temperature Applications*, Warren, PA, 6-8 Oct 1981.
21. J-J. Huet, L. Coheur, CH. Lecompte, A. Magnee, and C. Driesen, “Powder metallurgy oxide dispersion-strengthened (ODS) ferritic material with high damping capacity for rotating parts at intermediate temperature,” Pp. 173-178 in Proc. *Int. Powder Metallurgy Conference*, Florence, Italy, 20-25 June, 1982; Assoc. Italiana di Metallurgia, Milan, Italy (1982).
22. CH. Lecompte-Mertens, A. Magnee, L. Coheur, J-J. Huet, and C. Driesen, “ODS ferritic steels with high damping capacity,” Pp. 737-745 in Proc. Conf. on *High-Temperature Alloys for Gas Turbines*, Liege, Belgium, 4-6 Oct. 1982; D. Reidel Publishing Co., Dordrecht, The Netherlands (1982).
23. J-J. Huet, L. Coheur, L. DeWild, J. Gedopt, W. Hendrix, and W. Vandermuelen, “Fabrication and mechanical properties of ODS ferritic alloy canning tubes for fast reactor fuel pins,” pp. 329-334 in Proc. Topical Conf. on *Ferritic Alloys for use in Nuclear Energy Technologies*, J. W. Davis, Ed., Met. Soc. AIME (1984).
24. J-J. Huet, “Preparation and Properties of oxide dispersion-strengthened ferritic alloys,” Pp. 197-213 in Proc. Conf. on *Sintered Metal-Ceramic Composites*, New Delhi, India, 6-9 Dec. 1983; Elsevier Science Publishers, Amsterdam (1984).
25. J-J. Huet, L. Coheur, A. DeBremaecker, L. DeWild, J. Gedopt, W. Hendrix, and W. Vandermuelen, “Fabrication and mechanical properties of oxide dispersion strengthening ferritic alloy canning tubes for fast reactor fuel pins,” Nuclear Technology, 70, 215-219 (1985).
26. J-J. Huet, “Preparation and Properties of oxide dispersion-strengthened ferritic alloys,” Metal Powder Report, 40 (3), 155-158 (1985).
27. L. DeWild, J. Gedopt, S. DeBurbure, A. Delbrassine, C. Driesen, and B. Kazimierzak, “Paper 50. Pilot scale fabrication of ODS-ferritic alloy components for fast breeder reactor fuel pins,”, pp. 271-276 in Materials for Nuclear Reactor Core Applications, BNES, London (1987).