Cross-Roll Flow Forming of ODS Alloy Heat Exchanger Tubes For Hoop Creep Enhancement

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Abstract

Mechanically alloyed oxide dispersion strengthened (ODS) Fe-Cr-Al alloy thin walled tubes and sheets, produced via powder processing and consolidation methodologies, are promising materials for eventual use at temperatures up to 1200°C in the power generation industry, far above the temperature capabilities of conventional alloys. Target end-uses range from gas turbine combustor liners to high aspect ratio (L/D) heat exchanger tubes. Grain boundary creep processes at service temperatures, particularly those acting in the hoop direction, are the dominant failure mechanisms for such components. The processed microstructure of ODS alloys consists of high aspect ratio grains aligned parallel to the tube axis, a result of dominant axial metal flow which aligns the dispersoid particles and other impurities in the longitudinal direction. The dispersion distribution is unaltered on a micro scale by recrystallization thermal treatments, but the high aspect ratio grain shape typically obtained limits transverse grain spacing and consequently the hoop creep response. Improving hoop creep in ODS-alloy components will require understanding and manipulating the factors that control the recrystallization behavior, and represents a critical materials design and development challenge that must be overcome in order to fully exploit the potential of ODS alloys.

The objectives of this program are to 1) increase creep-strength at temperature in ODS-alloy tube and liner components by 100% *via*, 2) preferential cross-roll flow forming and grain/particle fibering in the critical hoop direction. *Recent studies in cross-rolled ODS-alloy sheets (produced from flattened tubes) indicate that transverse creep is significantly enhanced via controlled transverse grain fibering, and similar improvements are expected for cross-rolled tubes*. The research program outlined here is iterative in nature and is intended to systematically i) prescribe extrusion consolidation methodologies via detailed test matrices, ii) examine and identify post-extrusion forming methodologies to create hoop strengthened tubes, which will be iii) evaluated at 'in-service' loads at service temperatures and environments. This research program is to be conducted in collaboration with the DOE's Oak Ridge National Laboratory and the vested industrial partners Special Metals Corporation. In this second quarter of performance, program activities were initiated for Tasks 2, 3 and 4 and are reported herein. The completion of Task 1 ensures sufficient materials are now available for the remainder of this program.

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Table of Contents

§ 1. Executive Summary

Oxide dispersion strengthened (ODS) ferritic alloys based on FeCrAl and intermetallic Fe_3Al alloys are promising materials for high-temperature, high-pressure tubing, liner and shell applications on account of their creep strength at very high temperatures and excellent corrosion resistance in oxidizing, oxidizing/sulphidizing and oxidizing/chlorinating environments compared to available high-temperature alloys. Requirements for such a combination of properties are found in advanced systems being developed for utilization of fossil fuels, such as the **DOE**'s **Vision 21** and **FutureGen** programs and in improved gas turbines being developed for power generation.

The creep strength of conventional high-temperature alloys decreases rapidly with increasing temperature, as shown in Fig. 1, since the thermodynamic stability of the various available strengthening phases also decreases with increasing temperature[1]. Also shown in Fig. 1 is the significant increase in temperature capability afforded when a dispersion of inert oxide particles is used as the strengthening phase. A major feature of oxide dispersion-strengthened alloys is that the most successful route for their preparation appears to involve powder metallurgical processing[2-6]. Further, the critical need to maintain the fine size, volume fraction, and uniform distribution of the oxide particles in the alloy matrix, as well as the need to develop specific grain shapes, results in some significant differences in alloy fabricability and in the application of joining procedures, compared to conventional cast and wrought alloys. Hence, while ODS alloys offer a significant increase in temperature capability, they have a limited formability envelope, their mechanical properties are very anisotropic, and they cannot be joined by conventional fusion welding processes. Thus, the exploitation of the full capabilities of ODS alloys is limited until these critical hurdles are addressed and overcome.

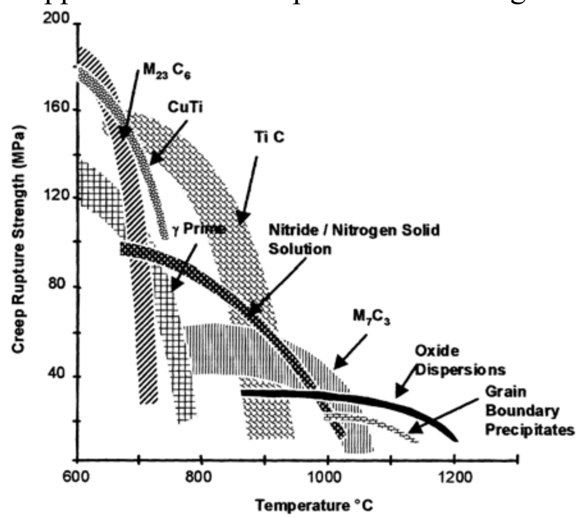

Figure 1. The creep performance envelope as a function of strengthening phase [1].

Our current program target is envisaged as a demonstration of the applicability of ferritic and Fe_3Al-based ODS alloys in the high temperature heat-exchanger tubing as proposed under the proposed **DOE** and **NETL Vision-21** program metrics, intended to sustain internal pressures (P) of up to 1000psi at service temperatures of 1000-1200°C. Within the framework of this target application, the development of suitable mechanically alloyed ferritic FeCrAl and intermetallic Fe_3Al alloy materials and processes must strive to deliver a combination of high mechanical strength at temperature and prolonged creep-life in service. Such design requirements are often at odds with each other as strengthening measures severely limit the as-processed grain size detrimental to creep life. The extrusion consolidation processes currently employed cause material flow in the longitudinal direction, resulting in extreme dispersoid and powder surface impurity fibering in the axial direction in ODS materials. Thus, elongated grains are produced aligned parallel to the longitudinal direction, with a fine grain spacing in the hoop direction. The basic problem of limited hoop creep is illustrated in Figure 2a,b within the context of the existing underlying grain structure. Fortunately ODS-alloys do exhibit intrinsic creep strength sufficient to meet design requirements albeit that this performance is only exhibited in the longitudinal direction. Ultimate failure in transverse (hoop) creep involves creep cavity concentration, Figure 2b, which strongly depends on the dominant grain boundary orientation with respect to the loading axis, Figure 4[7]. Such fibering, unless altered by post-flow forming, is expected to thwart attempts to arrive at the large transverse grain size[3,8] considered essential for improved creep performance in the hoop direction. Clearly what is required is to devise a means of effecting material flow in directions other than longitudinal that would reorient the primary fibering axis of dispersoids and impurities in the hoop direction.

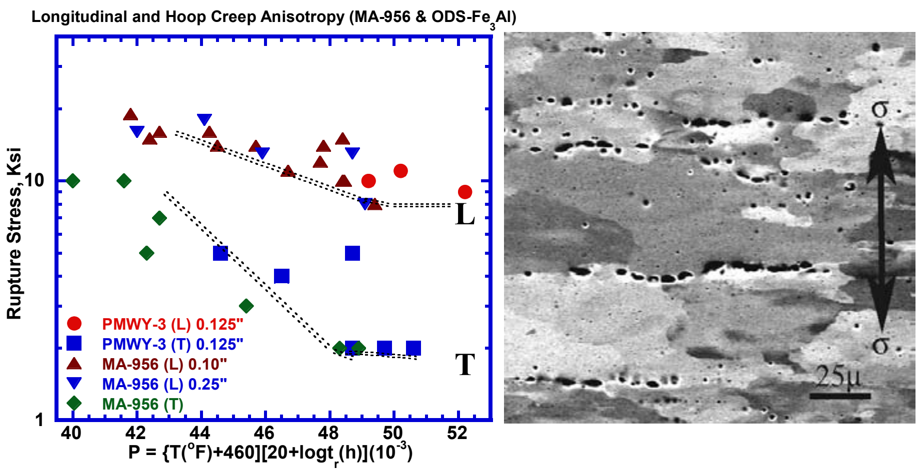

Figure 2. Longitudinal (L) vs. transverse (T) creep anisotropy in Fe_3Al (PMWY3) and MA-956 tubes. b) Creep cavitation observations in hoop creep loading tests.

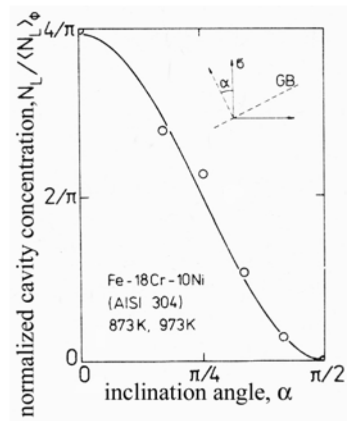

Figure 3. Creep cavity density as f_n(GB orientation) with respect to the loading axis [7].

Thus, our research objective is to modify tube-processing methodologies by incorporating cross-roll forming to create the underlying microstructure that will meet or exceed the design 'in-service' creep-life requirements of such ODS-alloy heat exchanger tubes. We are examining microscopic, microstructural and morphological issues with a view to addressing optimum material design for macroscopic components for a well prescribed 'in-service' loading criteria. A set of program tasks were outlined in the initial submission and a list of anticipated milestones were submitted to NETL in Fall 2003. This quarterly report summarizes our research activity in the second quarter of performance period of January 1st 2004 – March 31st 2003.

In the first quarterly performance period (October 1st – December 31st 2003) program work was initiated on Tasks 1 and 2 as proposed. Task 1 was completed in this period and will provide all program materials for the remaining tasks of this program. Task 2 consisting of cross-rolling flat segments of tubes is continuing into the current quarter and will be engaged in iteratively. Samples of both the ODS-Fe_3Al and FeCrAl (MA956) materials have been flattened and cross-rolled. Preliminary tests of cross-rolled specimens show a marked improvement in hoop creep response evaluated at 1000°C. Task 3 was initiated in the current quarter with the evaluation and identification of suitable helical rolling equipment as required for cross rolling tubes. A Medart Size ‘0’ helical rolling equipment installation is proposed. Preliminary evaluation of cross rolling (Task 3.1) has been successfully demonstrated for MA956 alloy tubes at 900°C. Future work will repeat this test for the ODS-Fe_3Al alloy tubes.

§ 2. Experimental Task Structure

The experimental work reported here is described in the context of the task structure outlined below. For the duration of this program activity through September 30^{th} 2005 and required reporting (monthly or quarterly) we will refer to this task structure for clarity and precise reference.

Task 1: Extrusion Consolidations, Tube and Sheet Forms

1.1 ODS-Powder materials –milling studies, impurity evaluation
1.2 Annular ODS-Alloy tube and sheet extrusions

Task 2: Rolling Studies for Optimum Fibering

2.1 Single vs. cross-rolling evaluation, Parametric studies
2.2 Correlate cross-rolling strains and overall grain re-orientation

Task 3: Post-Extrusion Cross-Roll Rolling of ODS-tubes & shells

3.1 Helical/cross rolling for grain fibering
3.2 Computer model verification for torsional flow predictions

Task 4: Microstructure and Creep Performance Evaluation:

4.1 Recrystallization annealing: static and gradient
4.2 Microstructure characterization & evaluation
4.3 Transverse creep and stress-rupture response

§ 3. Experimental Program Activity

Task 1.1 and 1.2: Annular ODS-Alloy tube and sheet extrusions: Initial materials (for this program) prepared and characterized in the tube and sheet form and are available for the remainder of this program. This task was completed in the prior performance period.

Task 2.1: Single vs. cross-rolling evaluation, parametric studies: Flat sections of initial uniaxially rolled/extruded coupons to be cross-rolled *via* parametric evaluations of cross-grain fibering of the underlying grain structure.

Three separate rolling schemes were employed: 1) Rolling longitudinally in 0.01" steps till the sample was measurably flat, 2) Rolling transversely to the tube axis in 0.01" steps till the sample was measurably flat, and 3) Rolling transversely to effect a net 20-25% thickness reduction in the starting wall thickness. In the rolling schedule 3, this large deformation was accomplished in steps of 4-5% reduction per pass with the sample reheated to $900^{o}C$ for 15 minutes in the air furnace.

Task 3: Post-Extrusion Cross-Roll Rolling of ODS-tubes & shells: This task includes the purchase and installation of a <u>used</u> helical rolling mill at UCSD to effect cross-rolling in cylindrical tube shapes rather than the flat specimens as cross-rolled in Task 2.1. In February 2004 the PI took traveled to Pittsburgh PA; Cleveland OH; Toledo OH; and Detroit MI to inspect used machinery available either at operating facilities or at equipment dealers showrooms. A total of 7 helical rolling machines were inspected as follows; 2 in Pittsburgh, 4 in Toledo and 1 in Detroit. All machines were similar in size and capabilities equivalent to a MEDART size '0' 2-roll rotary straightener. The equipment (Medart '0' machine) located at Bar Processing Corp, Michigan was selected for further contract and delivery negotiations. These discussions are expected to close in the next performance period with the equipment installation at UCSD slated in mid summer.

Task 3.1: Helical/cross rolling for grain fibering: As a prelude to the equipment installation at UCSD, preliminary and subscale trials were performed using a lower capacity machine. Due to the high intrinsic strength of the as received MA956 tube and the power limitation of the rolling apparatus, MA956 tubes could not be rolled at ambient temperatures. In a compromise measure MA956 tubes were preheated to 900^{o}C and incrementally cross-rolled in 10-12 steps. The exact procedure involves loading the preheated tube onto a snug fitting mandrel and cross rolling with the sample reheated in the furnace for a minimum of 15 minutes between each step. Figure 1 illustrates the operation of one such pass. We note that preheating was employed to overcome machine limitations. We expect that such preheating cause recovery, which may delay subsequent recrystallization response. Looking ahead to the observations of Task 4.1 this is of particular concern for MA956 alloy tubes because of the lower overall processed grain size obtained. Successful installation of the UCSD equipment will allow for cross rolling at reduced temperatures.

Figure 1. Helical cross rolling of MA956 tubes at 900^{o}C

Task 4.1: Recrystallization annealing: static and gradient:

Flat samples cross-rolled in Task 2.1 are recrystallized via heat treatments of 1-hour at 1200^{o}C in air for ODS-Fe_3Al and a 1-hour at 1375^{o}C in air for FeCrAl (MA956). Microstructures reveal elongated grain shapes in the transverse orientation only for the sample cross-rolled 20-25% in the transverse orientation. However the recrystallized transverse grain sizes of 200-400μm in ODS-Fe_3Al samples are consistently larger than the 50-100 μm transverse dimensions in MA956 alloy. This despite the significantly higher recrystallization temperature employed for MA956 alloys. Further attempts will focus only on rolling schedule 3 in Task 2.1.

The recrystallized samples were spark machined to extract ASTM E-8 standard specimens from the transverse orientation. These are currently being evaluated in transverse (hoop) creep tests in Task 4.2 and compared to the base line hoop creep behavior of as-extruded tubes. Initial results point to a 50% improved creep response and complete results will be reported later.

§ 4. Results and Discussion

The experimental program is proceeding at the originally prescribed timetable. The initial material preparation steps are well characterized and known from prior experience. Additional tasks under way have begun to yield the preliminary test data for cross-rolling strains necessary for the success of this program as applied to cross-rolling of tubes. In the cross-rolling trials we note that significant grain alignment was recorded in the transverse direction, whereas none was observed in rolling schedule #2 (see section on Task 2.1). Initial tests conducted on these cross-rolled specimens have produced a consistent improvement in hoop creep performance. The best level achieved till date is a 50% improvement in response.

Task 3 is well underway via the purchase of requisite Medart equipment to be installed at UCSD. Preliminary trials for cross-rolling tubes have been successful and no problems have been encountered till date. Further cross rolling tasks will follow after the UCSD equipment installation is completed and the machine brought on line for testing.

§ 5. Conclusions

The current research program was initiated on October 1st 2003 and is now concluding its second quarter of performance. We have till date recorded evidence of hoop creep improvement in flat coupons that were cross-rolled. Our objective now is to embed such cross rolling deformation to tubes. Initial efforts at cross rolling tubes have been successfully demonstrated.

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