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

Quarterly Technical Progress Report
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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^{o}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 first quarter of performance, program activities were initiated for Tasks 1 and 2 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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§ 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.

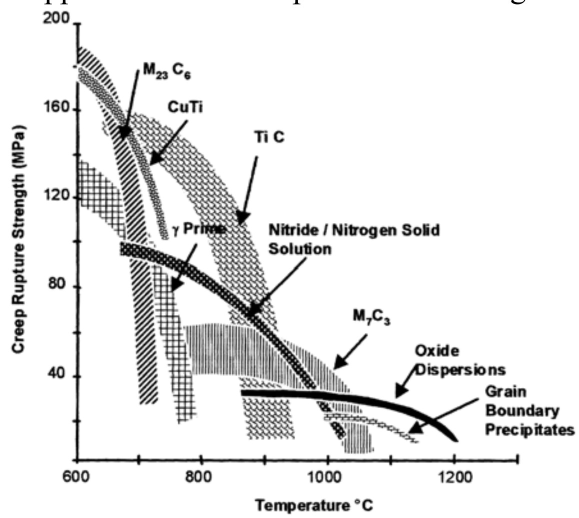

Figure 1. The creep performance envelope as a function of strengthening phase [1].

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^oC. 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.

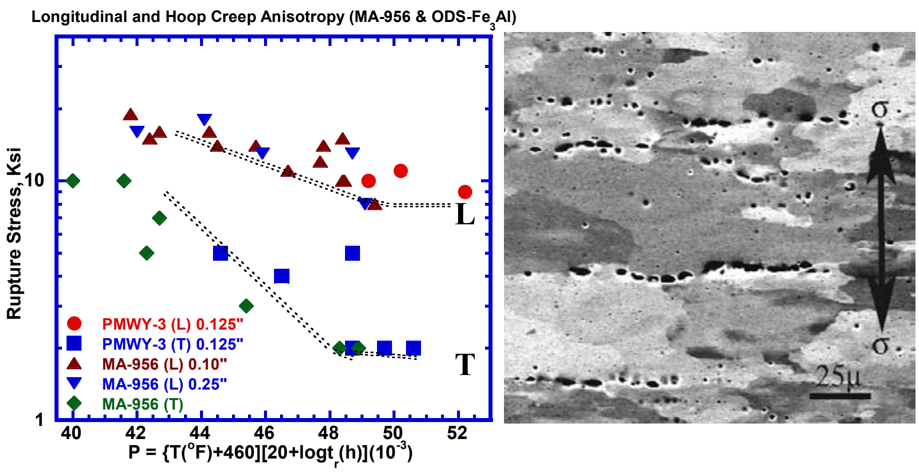

Figure 2. Longitudinal (L) vs. transverse (T) creep anisotropy in Fe_3Al (PMWY3) and MA-956 tubes. b) Creep cavitation observations in hoop creep loading tests.

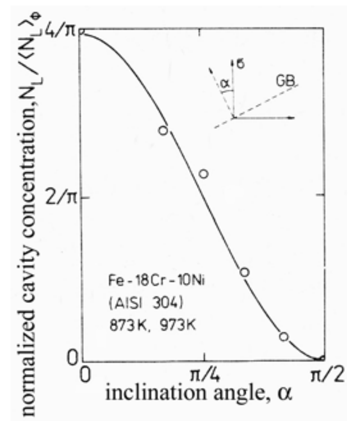

Figure 3. Creep cavity density as f_n(GB orientation) with respect to the loading axis [7].

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 first quarter of performance period of October 1st 2003 – December 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 next quarter and will be engaged in iteratively. Samples of both the ODS-Fe_3Al and FeCrAl (MA956) materials have been flattened at cross-rolled. The ODS-Fe_3Al tubes are initially 1¼" OD, 1/8" wall thickness and the ODS-FeCrAl tubes are 2½" OD, ¼" wall thickness. Three different roll flattening processes are employed as 1) roll-longitudinally, 2) roll transverse to flatten and 3) roll flatten to 20-25% reduction in thickness. Samples are now being cut in transverse (hoop) orientations for high temperature creep testing. Full details of these creep tests will be reported in the next performance period.

§ 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 30th 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.

The alloy materials ODS-Fe_3Al and FeCrAl (MA956) required for our program were acquired and prepared. FeCrAl alloy tubes of 2½" OD, ¼" wall thickness were acquired from Special Metals Corporation in the unrecrystallized condition. In addition smaller dimension tubing of 1¼" OD, 1/8" wall thickness were prepared using a single step extrusion consolidation process at the Oak Ridge National Laboratory. The exact procedure is encapsulating the powder in an annular 4" OD can and preheating it in a furnace at about 400^{o}C while being evacuated via a mechanical pump. After about 2-4 hours the cans are pinch sealed under nominal vacuum. The cans are then furnace soaked for 2-4 hours in the 1000-1100^{o}C ranges. The cans are extruded at a roughly 16:1 extrusion ratio with a 7/8" OD internal mandrel. Previously extruded ODS-Fe_3Al alloys were made available from a research program at UCSD conducted in collaboration with Oak Ridge National Laboratory. It is expected that the material inventory at hand is sufficient for the entire duration of this project. In preparation for Task 2 about 8-12" lengths of tubes were slit along their length. The 1¼" OD tubes were slit longitudinally into 2 (180^{o} section) pieces, and the 2½" OD tubes were slit longitudinally into 3 (120^{o} section) pieces. These curved sections were wrapped in stainless steel covers and preheated in an air furnace at 900^{o}C for 1 hour and then pressed flat in a single stroke. This task activity was conducted in collaboration with ORNL.

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.

Materials produced in Task 1.1 and 1.2 were sectioned and examined for microstructural details. No recrystallization was observed in either alloy materials as a result of this 900oC thermal-mechanical treatment. This flattened strip is the required material for the initial matrix of parametric cross-rolling studies. Based on the post-forging microstructural evaluation, and in the interest of narrowing experimental windows, all further cross-rolling studies are to be conducted at 900^{o}C. Residual curvature in the forge-flattened specimens was eliminated via subsequent rolling as described here. 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. . Additional levels of (cross-rolling) strains will be evaluated in the next quarter. The rolled flat samples were removed from their stainless steel wraps and prepared for the recrystallization treatments. Additional levels of (cross-rolling) strains will be evaluated in the next quarter. The cross-rolled specimens are recrystallized to create abnormal grain growth in such ODS-alloy coupons. The heat treatments are 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. It is likely that surface layers are affected in rolling schedule 1 and 2 but no changes were perceptible at the level of optical resolution.

These initial samples have been spark machined to extract ASTM E-8 standard specimens from the transverse orientation. These are currently being evaluated in transverse (hoop) creep tests and compared to the base line hoop creep behavior of as-extruded tubes. Initial results point to improved creep response. Results of a complete matrix of tests will be reported in the next performance period.

§ 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 will yield much of the preliminary test data fro cross-rolling strains necessary for the success of this program.

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). It is unknown if lower levels of strain (say fro example 10-15%) will be sufficient to effect some grain organization. This will be examined in the next iteration of cross-rolling studies. Looking ahead to the next quarter – we note promising improvement in hoop creep response in the cross-rolled specimens

§ 4. Conclusions

The current research program was initiated on October 1st 2003 and is just concluding its first quarter of performance. No conclusions are offered at this early juncture.

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