Optimization of Oxide Dispersion Strengthened Fe_3Al Alloy Tubes: Application Specific Development for the Power Generation Industry

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Bimal K. Kad
University of California-San Diego, La Jolla, CA 92093-0085

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Program Summary

A detailed and comprehensive research and development methodology is being prescribed to produce Oxide Dispersion Strengthened (ODS)-Fe_3Al thin walled tubes, using powder extrusion methodologies, for eventual use at operating temperatures of up to 1000-1100°C in the power generation industry. The objectives of the research effort are to 1) produce thin walled ODS-Fe_3Al tubes with 2) increased hoop strength for service at 1000-1100°C operating temperatures, and 3) to mitigate creep failures by enhancing the as-processed grain size in ODS-Fe_3Al tubes.

Current single step extrusion consolidation methodologies typically yield 8ft. lengths of 1-3/8" diameter, 1/8" wall thickness, ODS-Fe_3Al tubes. The process parameters for such consolidation methodologies have been prescribed and evaluated as being routinely reproducible. Recrystallization treatments at 1200°C routinely produce elongated grains (with their long axis parallel to the extrusion axis), typically 200-2000μm in diameter, and several millimeters long. The dispersion distribution is unaltered on a micro scale by recrystallization thermal treatments, but the high aspect ratio grain shape typically obtained limits grain spacing and consequently the hoop creep response.

Current efforts are now focused on examining the processing dependent longitudinal vs. transverse (hoop) creep anisotropy, and exploring post-extrusion methods to improve hoop creep response in ODS-Fe_3Al alloy tubes. Improving hoop creep in ODS-alloy components requires an 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. In this report we examine the mechanisms of hoop creep failure and describe our efforts to improve hoop creep performance via 1) aggressive recrystallization heat treatments, 2) variations in recrystallization environment, and by 3) incorporating thermo-mechanical routes to altering the underlying grain shapes

Research sponsored by the US Department of Energy, Fossil Energy Advanced Research and Technology Development Program, DOE/FE AA 15 10 10 0, Work Breakdown Structure Element UCSD-2.

Introduction

Fe_3Al-based alloys are promising materials for high temperature, high pressure, tubing applications, due to their superior corrosion resistance in oxidizing, oxidizing/ sulphidizing, sulphidizing, and oxidizing/chlorinating environments. Such high temperature corroding environments are nominally present in the coal or gas fired boilers and turbines in use in the power generation industry. Currently, hot or warm working of as-cast ingots by rolling, forging or extrusion in the 650-1150°C temperature range is being pursued to produce rod, wire, sheet and tube products [1,2]. *A particular 'in service application' anomaly of Fe_3Al-based alloys is that the environmental resistance is maintained up to 1200°C, well beyond where such alloys retain sufficient mechanical strength.* Thus, powder metallurgy routes, incorporating oxide dispersions (ODS), are required to provide adequate strength at the higher service temperatures.

The target applications for ODS-Fe_3Al base alloys, in the power generation industry, are thin walled (0.1" thick) tubes, about 1 to 3 inches in diameter, intended to sustain internal pressures (P) of up to 1000psi at service temperatures of 1000-1200°C. Within the framework of this intended target application, the development of suitable materials containing Y_2O_3 oxide dispersoids, must strive to deliver both a combination of high mechanical strength at temperature, as well as 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. Thus post-deformation recrystallization, or zone annealing, processes are necessary to increase the grain size, and possibly modify the grain shape for the anticipated use. The economic incentive is the low cost of Fe_3Al-based alloys and its superior sulphidization resistance, in comparison to the competing Fe-Cr-Al base alloys and the Ni-base superalloys currently in service.

Program Status and Report

Current manufacturing methods yield typically 8 Ft. lengths of 1-3/8" diameter, 1/8" wall thickness, ODS-Fe_3Al tubes produced *via* a proprietary single step extrusion consolidation process. The process parameters for such consolidation methodologies have been prescribed earlier [3] and evaluated as being routinely reproducible. Static recrystallization studies till date show that elongated grains (with their long axis parallel to the extrusion axis), typically 200-2000μm in diameter, and several millimeters long can be obtained routinely, at heat-treatment temperatures of 1100-1200˚C. <u>*In this current project we address the metallurgical and microstructural processing issues that may improve and optimize the hoop creep response in ODS-alloy tubes*</u>. The project is iterative in nature, intended to systematically examine the various sub-processes for optimum performance and cost considerations.

This interim report describes our microstructure and property optimization of ODS-Fe_3Al alloy tubes, with a view to improving the high temperature creep response. In particular, we examine thermal-mechanical processing steps to affect and enhance secondary recrystallization kinetics and abnormal grain growth during post-extrusion processing to create large grains. Such procedures are particularly targeted to improve hoop creep performance at service temperatures and pressure.

Task 1. Materials Processing

The Fe_3Al+0.5wt% Y_2O_3 composition was optimized at Oak Ridge National Laboratory. Three separate batches are milled (identified as PMWY-1, PMWY-2 PMWY-3) and extruded. Figure 1 shows a set of tubes in the as-extruded and surface finished condition and Table 1 lists the extrusion processing parameters. Additional processing details are available elsewhere [3,4]. The tubes (typically 6-8 ft. length, 1-3/8" diameter and 1/8" wall thickness) are of sound quality and exhibit no cracking or damage after machining operations.

Figure 1. Assorted ODS-Fe_3Al tubes in the as-extruded (below), and surface finished (top) condition, as produced from an annular can (top left) consolidation methodology.

Table 1: Tube extrusion consolidation parameters for PMWY-1-3 powders

Material	Extrusion Temperature	Die Size inch	Mandrel Size inch	Area Reduction	Tonnage
PMWY-1[a]	1000˚C	≈1.375	1.00	≈16.0:1	NA
PMWY-2[a]	1000˚C	≈1.375	1.00	≈16.0:1	NA
PMWY-2[a]	1000˚C	≈1.375	1.00	≈16.0:1	NA
PMWY-3[a]	1000˚C	≈1.375	1.00	≈16.0:1	NA

[a]102 mm (4.0 in) billets for thin-walled tube extrusion

Milled Powder Chemistry

Table 2 lists the alloy powder chemistry before and after three separate milling conditions [5]. The powders are listed as PMWY-1, PMWY-2 and PMWY-3 in the order of decreasing total interstitial (C+N+O) impurity. As shown later this interstitial impurity plays a crucial role in the recrystallization kinetics and the resulting microstructures.

Table 2: Chemical analyses of the as-received and milled powder batches [5]

Element	As-Received HM	PM	PMWY-1	PMWY-2	PMWY-3
Fe	Bal.	79.6			
Al	16.3	18.20			
Cr	2.4	2.18			
Zr	20 ppm	26 ppm			
O (total)	60 ppm	110 ppm	1800 *ppm*	1900 *ppm*	1400 ppm
O (in Y_2O_3)			1025 *ppm*	1053 *ppm*	1080 ppm
O balance			775 *ppm*	847 *ppm*	320 ppm
O *pickup*			**665 *ppm***	**737 *ppm***	**210 ppm**
N	18 ppm	7 ppm	1264 *ppm*	145 *ppm*	88 *ppm*
N *pickup*			**1257 *ppm***	**138 *ppm***	**81 *ppm***
C		24 ppm	667 *ppm*	360 *ppm*	303 *ppm*
C *pickup*			**643 *ppm***	**336 *ppm***	**279 *ppm***
H		16 ppm	115 *ppm*	40 *ppm*	29 *ppm*
C+N+O *pickup*			**2565 *ppm***	**1211 *ppm***	**570 *ppm***

(Bulk compositions are identified in wt%)

Recrystallization Kinetics

Samples from the extruded tubes were subjected to a standard static recrystallization heat treatment of 1200°C for 1hr in air. Figure 2 shows the longitudinal and transverse section microstructures from the PMWY-1 (left) and the PMWY-3 (right) chemistry. Note that PMWY-1 does not exhibit complete section recrystallization but PMWY-3 alloy produces complete section recrystallization in a reproducible manner.

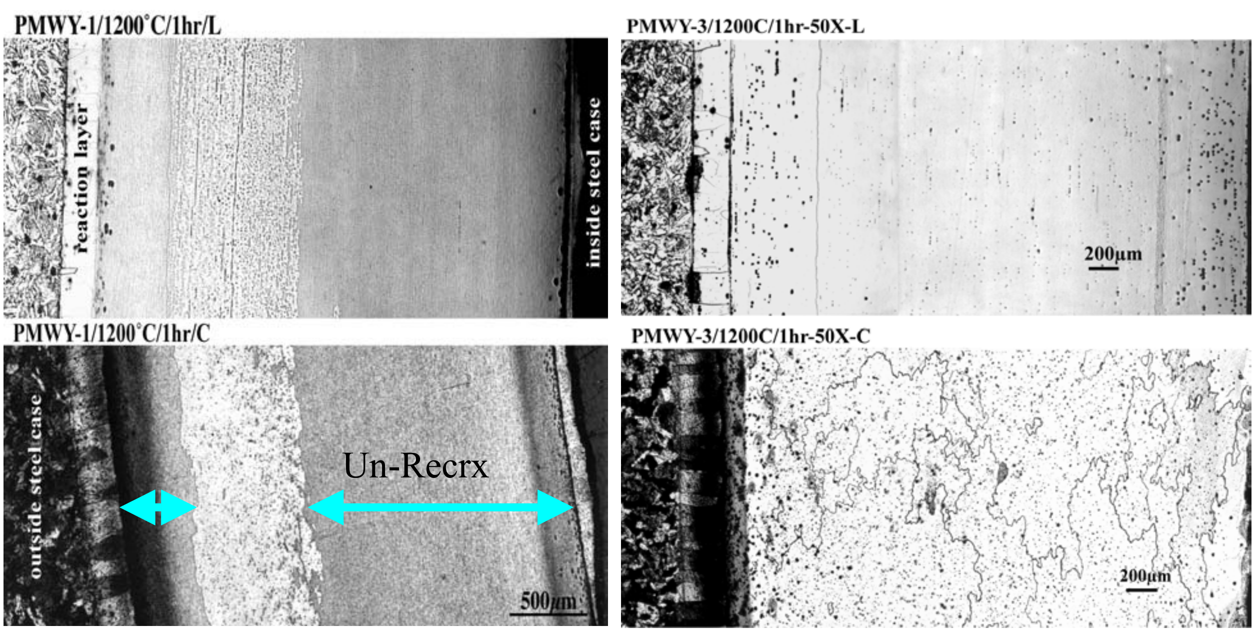


Figure 2. Recrystallized tube sections of (a) PMWY-1 (left) and (b) PMWY-3 alloy (right).

Task 2. Materials Characterization

TEM Microstructures

Bright Field TEM micrographs of specimens extracted from the heat-treated tubes are shown in Figure 3. The 3mm discs were extracted from the wall thickness of the tubing such that foil normal and the extrusion axis are co-incident. With the TEM thin foil perforation expected near the center of the discs, the microstructures shown below are then representative of the center of the tube thickness. We note that both PMWY-1 and PMWY-2 exhibit a fine-grained structure, Figure 3a, 3b, with a {110} texture. However, PMWY-3, Figure 19c, exhibits a coarse grain structure with a {111} recrystallized texture. The precipitate distribution in PMWY-3 is bi-modal with the coarser particles exhibiting a cell-type structure on the scale of 1 μm. This dimension is consistent with the as-extruded grain size and it is suggested that this particle distribution was originally present on the surface of the milled powders and upon consolidation was incorporated at the as-extruded grain boundaries. The Y_2O_3 precipitates are about 10-20nm in diameter and their distribution in PMWY-3 alloys is extremely homogenous at about 80-90nm spacing.

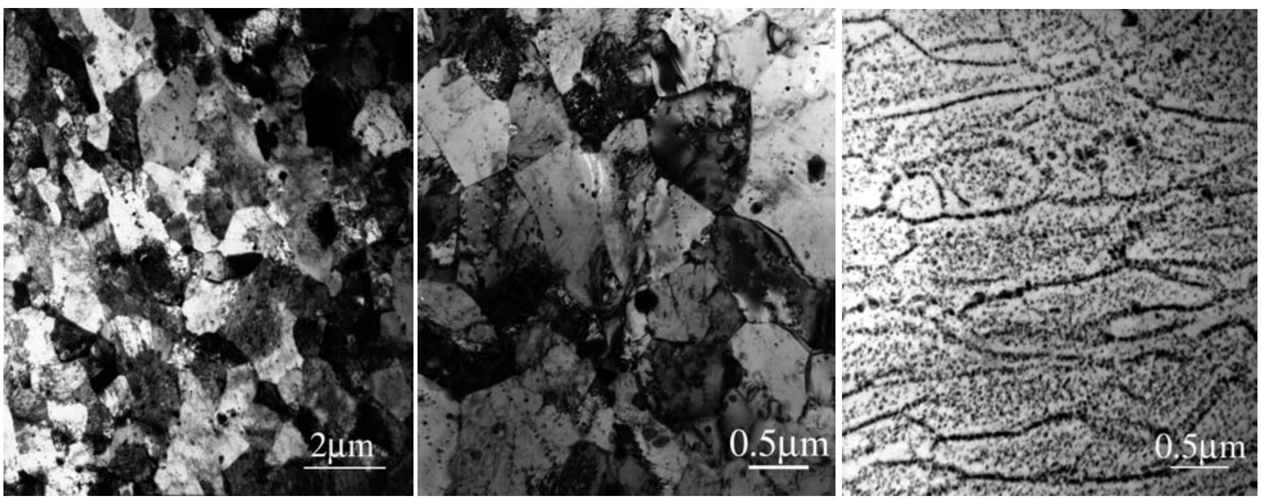


Figure 3. TEM micrographs of a) PMWY-1, b) PMWY-2 and c) PMWY-3 specimens extracted from the heat-treated tubes. The viewing direction is along the extrusion axis. PMWY-1 and PMWY-2 alloys exhibit the deformation processed and unrecrystallized {110} texture while PMWY-3 exhibits the recrystallized {111} texture [6].

A magnified view of the PMWY-2 sample, Figure 4, indeed shows that the grains are effectively pinned by precipitate particles. These precipitates (marked in red arrows) tend to be of the order of 0.25μm, i.e. much larger than the mean Y_2O_3 dispersions of about 10nm size. Precipitate chemical analyses confirm that they contain negligible amounts of yttrium and instead are rich in aluminum. This is reflected in the relative strengths of the aluminum peak in the matrix and precipitate spectra as illustrated in Figure 4. Looking back to the interstitial impurity analysis of Table 1, we note that both PMWY-1 and PMWY-2 have a significant level of oxygen, nitrogen pickup depending on the exact milling conditions employed. This impurity is in addition to the oxygen in Y_2O_3 and is interpreted as an overall increase in the precipitate volume fraction.

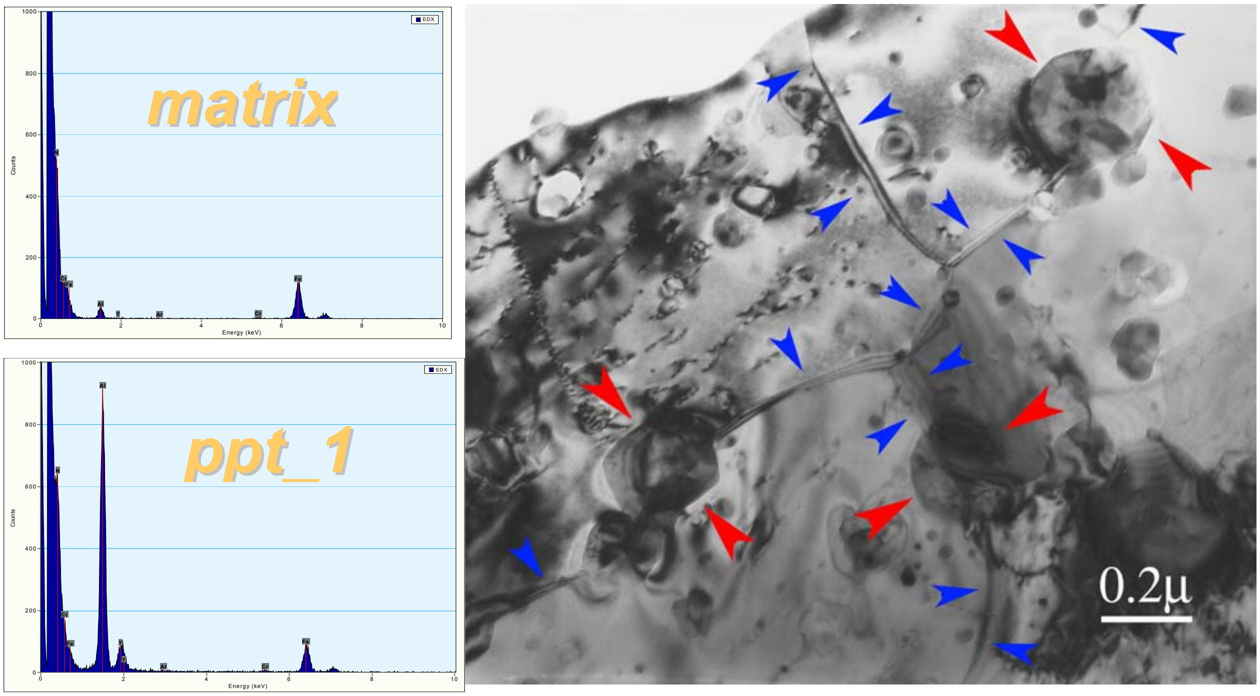


Figure 4. Impurity pickup at the milling stage results in the formation of large oxides and/ or nitrides of aluminum at the grain boundaries.

Task 3. High Temperature Mechanical Properties

The ductile to brittle transition temperature (DBTT) for ODS-Fe_3Al alloy tubes was assessed to be of the order of about 800^{o}C (REF_a) less than the anticipated service temperature range. Tests were thus limited to the 800-1100^{O}C range (i.e., in the creep regime) for all the three tubes. High temperature tensile and creep properties were evaluated for each of the three alloy chemistries in the longitudinal and transverse orientations. Standard ASTM E-8 miniature specimens are extracted from tube sections for tensile and creep tests. Longitudinal sections are spark machined directly from tubes and transverse sections are machined from flattened (hot pressed at 900^{o}C) tubes. The hot pressing temperature is limited to 900^{o}C to prevent any recrystallization at this step. Samples are initially heat-treated at 1200^{o}C for 1 hour. Tensile test were performed at temperature at a constant strain rate of 3 x 10^{-3} sec^{-1}.

<u>**Tensile Properties**</u>

High temperature tensile properties were examined for ASTM E-8 miniature specimens spark machined from the 1/8" shell thickness of the 1-3/8" O.D. extruded tubes. Tensile test were performed at temperature at a constant strain rate of 3 x 10^{-3} sec^{-1}. Figure 5a,b show the comparative tensile response of the recrystallized PMWY-1, PMWY-2 and PMWY-3 tubes in the longitudinal and transverse orientations. Employing tensile strength as a ranking measure at the 1000^{o}C test temperature, we note that for PMWY-3 alloy tube exhibits the best combination of yield and ultimate tensile strength. It is surprising that the performance range is rather narrow and belies the vast variations in creep response observed for the three alloys.

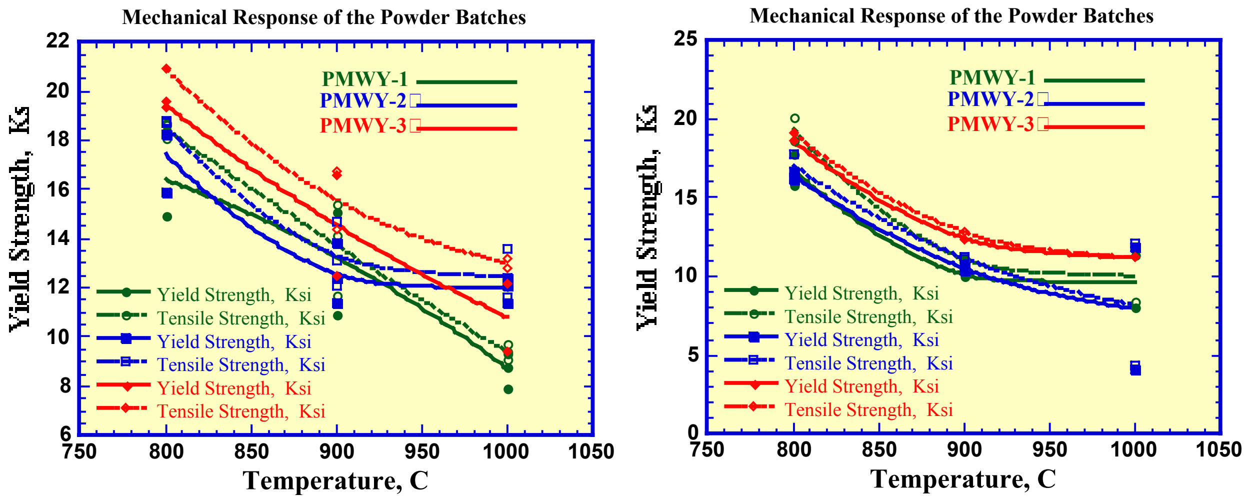


Figure 5. High temperature mechanical response of PMWY-1, PMWY- and PMWY-3 alloy tubes in the a) Longitudinal and b) Transverse orientations

Creep Properties

Longitudinal vs. Transverse Creep Anisotropy

Preliminary creep tests were carried out for the ODS-Fe_3Al alloy tubes as well as the commercially available MA-956. The Fe_3Al samples were subjected to a uniform 1200^oC/1-hr recrystallization heat treatment. Figure 6 shows the longitudinal (L) vs. transverse (T) creep anisotropy for both the Fe_3Al and the Fe-Cr-Al alloys. The MA-956 tests were conducted at 900^oC and the ODS-Fe_3Al tests were conducted at 1000^oC. We note the improved creep response of the Fe_3Al chemistry over the commercial MA-956 in both the L and T orientations.

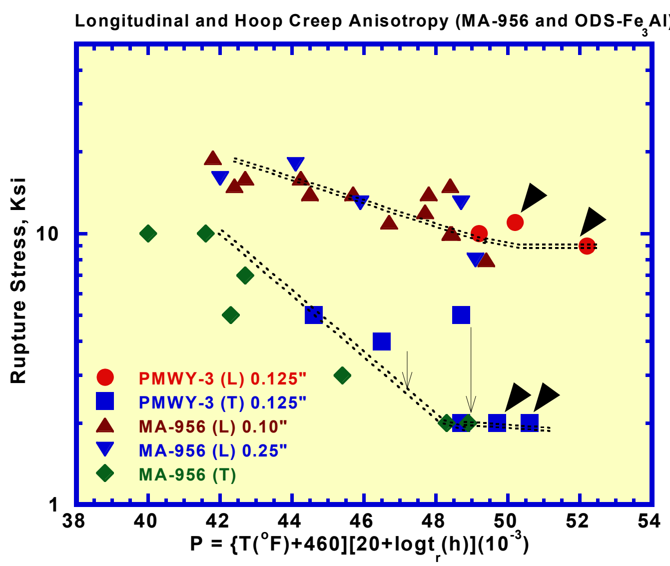
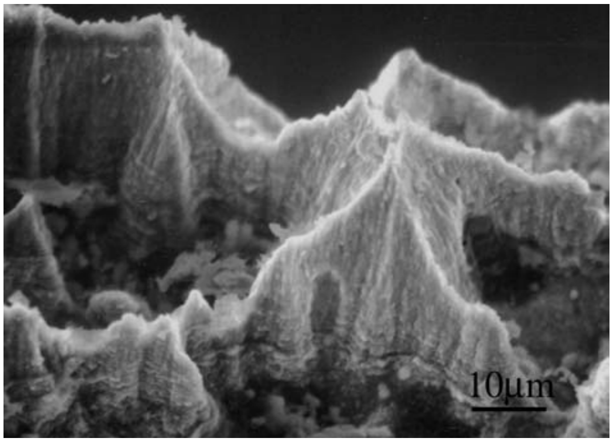


Figure 6. Longitudinal vs. transverse (hoop) creep anisotropy in ODS-Fe_3Al and MA956 alloy tubes.

Longitudinal creep performance of PMWY-1 and PMWY-2 alloys (not plotted here) is poor compared to PMWY-3. Selected samples of PMWY-2 however exhibit performance approaching that of PMWY-3, and we note that such samples exhibit a high recrystallized cross-section comparable to PMWY-3 chemistry. Figure 7 shows comparative fracture surface features for the PMWY-1 (left) and PMWY-2 (right) alloys. PMWY-1 exhibits a large array of ductile lobes each fully necked down to a point (Figure 7a) and stands about 20-30μm tall. The PMWY-2 as well as PMWY-3 fracture features are dissimilar and indicative of transgranular creep failure. PMWY2 and PMWY 3 materials were also observed to sustain numerous elongated voids.



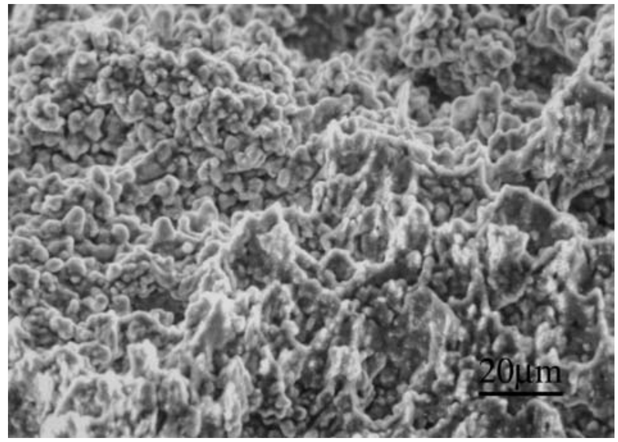


Figure 7. Creep fracture in longitudinal orientations. a) PMWY-1 and b) PMWY-2 alloys.

Task 4. Enhancing Transverse Creep Response

Our program efforts are directed towards exploring metallurgical and microstructural means to enhancing and optimizing hoop creep response. The effect of chemistry as induced via milling impurities was explored earlier, and PMWY-3 alloy was selected as promising alloy chemistry based primarily on its large recrystallized grain size. Selected efforts at improving hoop-creep are enumerated below.

<u>Transverse creep deformation and failure</u>

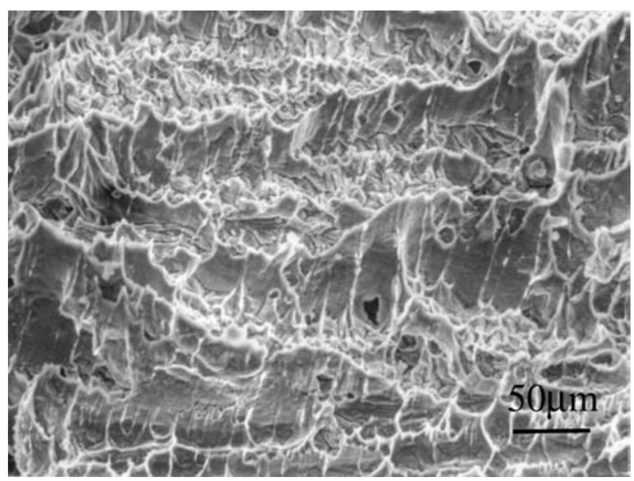


Figure 8. Transverse creep failures by void formation and subsequent coalescence.

The limiting hoop creep response in ODS-alloy heat exchanger tubes is a prominent hurdle to meeting ARM's target performance envelope. A mechanistic understanding of hoop creep failures thus is an important pre-requisite to aid any subsequent microstructural modifications and/or redesign efforts at the system, sub-system or component level. Figure 8 shows a typical transverse creep failure as observed in fully recrystallized PMWY-3 alloy tested at 1000^{o}C. Significant ductile voids are observed and it is presumed that failure is preceded by void formation and coalescence. A better insight into the origin of voids is obtained by examining the specimen surface immediately below the creep failure. Figure 9a,b shows creep void formation in crept ODS-Fe_3Al and MA-956 alloys respectively. ODS-Fe3Al test was conducted at low-stress for long-term exposure and shows limited number of voids. On the other hand samples cut from flattened MA-956 tube were subjected to a short-term test at high stress and consequently the voids formation is severely exaggerated. In either case the origin of such voids is predominantly at the grain boundary. Corresponding TEM studies of thin foils extracted from regions immediately below the creep fracture are illustrated in Figure 10.

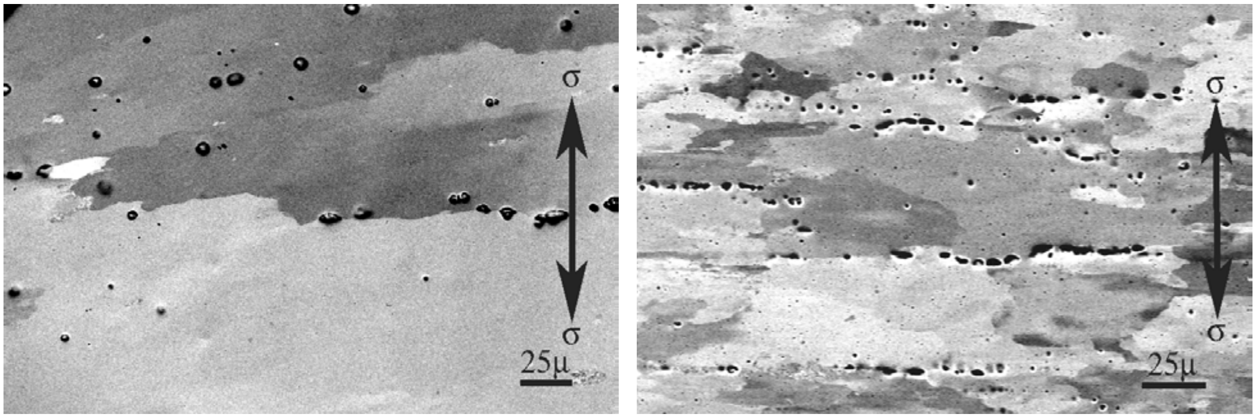


Figure 9. Creep void formation and coalescence in a) PMWY-3 crept at 3Ksi at 1000°C, and b) MA-956 crept at 10Ksi at 900°C. Voids form predominantly at the grain boundaries.

Figure 10a shows bulk grain deformation activity in PMWY-3 alloy sample crept at 1000°C (corresponding to Figure 9). The Y_2O_3 distributions are evenly distributed and are effective at dislocation pinning. However, the observed dislocation density is extremely small as if the structure is well annealed. On the other hand in the high stress deformation of MA-956 alloy, Figure 10b, we note ample dislocation activity in the bulk matrix. Dislocations are uniformly pinned by the dispersoids, which contribute effective creep strengthening. However, this matrix deformation is observed when the boundary is well cavitated and ripe for failure. It is envisioned that in the conventional low-stress operating regime, the role of bulk deformation is severely limited. Thus failure may be highly localized in the vicinity of the grain boundary, and occur at limited creep strains. Looking ahead to Figure 12 this is indeed experimentally observed.

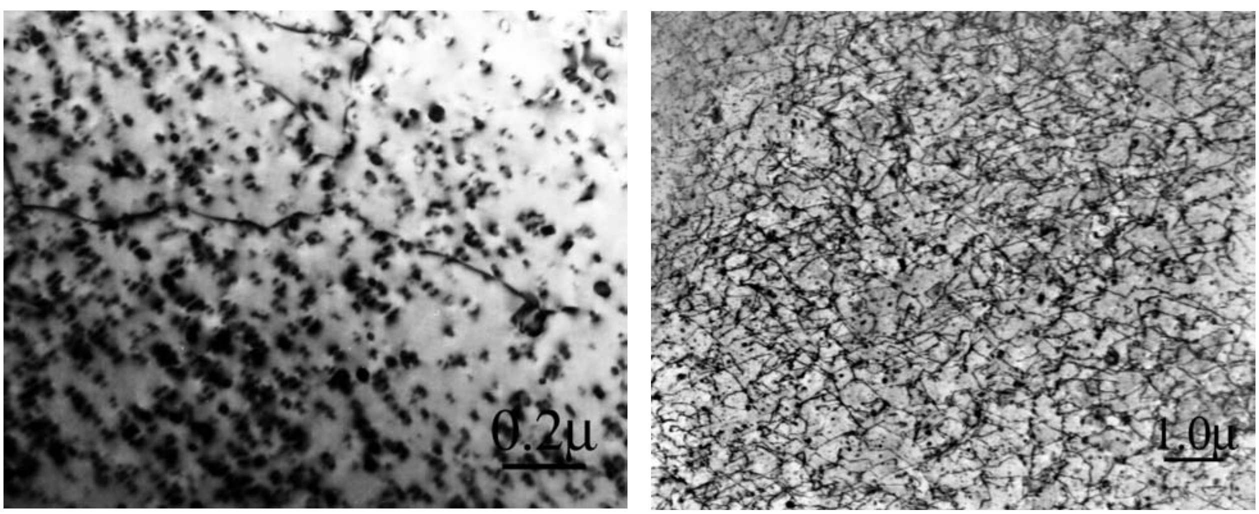


Figure10. TEM micrographs of bulk deformation in a) PMWY-3, and b) MA-956 alloy in the vicinity of failure, corresponding to the results presented in Figure 9.

Transverse creep in PMWY-2 alloys

For the data presented in Figure 6 all ODS-Fe_3Al alloy samples were recrystallization heat-treated at 1200^{o}C for 1 hour. In an effort to promote further recrystallization, aggressive re-heat treatments were attempted at 1250^{o}C and 1300^{o}C for a period of 1-10 hours for the PMWY-1 and PMWY-2 alloys. (Heat-treatment performed on bare ASTM E-8 test coupons; sample thickness =1.0 mm). Transverse oriented samples were then tested in the 900^{o}C -1000^{o}C temperature range. Table 3 lists a set of transverse test results for the PMWY-2 alloy specimens. The samples exhibit extreme stress sensitivity and no appreciable life is expected at 2Ksi at temperatures 900^{o}C and above. Furthermore, no improvement was observed following the aggressive re-heat treatment.

Table 3: Transverse creep in PMWY-2 alloys as a function of thermal treatments

Sample	Heat Treatment	T^{o}C	Ksi	Life	rate/day	L-M
#03, B2T	**HT:1250/10hr, Air**	**900**	**1**	**254**		**47.32**
#03, B2T	**HT:1250/10hr, Air**	**900**	**2**	**10.5**		**44.40**
#4, B2T	**HT: 1250/10hr, Air**	**1000**	**1**	**241**		**51.30**
#09, B2T	**HT:1300/1hr, Air**	**900**	**2**	**25**		**45.19**
#11, C2T	**HT:1300/1hr, Air**	**900**	**2**	**5**		**43.72**
#12, C2T	**HT:1250/10hr, Air**	**900**	**2**	**5**		**43.72**
#13, C2T	**HT: 1200/1hr, Air**	**900**	**2**	**9**		**44.26**

Transverse creep in PMWY-3 alloys

Alternate recrystallization heat-treatments and thermo-mechanical processing schemes are explored for the PMWY-3 alloy chemistry as it exhibits the best performance among the family of ODS-Fe_3Al alloys under study as well as in comparison to the commercial MA-956 alloy, Figure 6. Such effects were explored for

1) *Aggressive recrystallization heat treatment*,
2) *Variations in recrystallization environment*, and
3) *Thermo-mechanical routes to altering the underlying grain shape*

Table 4 lists the complied results of all processing variations prescribed above and are discussed below. In each case spark machined samples were heat-treated bare with or without a protective atmosphere. Samples are tested at a constant load and temperature and held for a minimum of 1000 hours at each test condition before incrementing load. The accumulated test time is recorded for the peak load and all prior load values. The observed creep is tabulated in days. For example, Test #19 has 3191 hours of exposure at 2Ksi, 1968 hours at the 3Ksi increment and about 963 hours at the 4Ksi increment, and the creep rate is recorded as 2e-5/day at the 4Ksi test condition. It is surmised that a 1000hour exposure is sufficient for predicting long-term survivability at that specific test condition on account of the high stress exponent for failure. This is observed in such ODS-alloys particularly in the transverse orientation creep tests.

Table 4: Transverse creep response in PMWY-3 alloys as a function of thermal and thermal-mechanical processing conditions. (* tests continuing as of 3/10/03)

Sample	Heat Treatment	T^{o}C	Ksi	Life	rate/day	L-M
#06, C2T	**HT:1300/1hr, Air**	**900**	**2**	**643**		**48.17**
#20, C2T	**HT: 1300/1hr, Air**	**900**	**2**	**2110**	**7e-5**	**49.26**
#20, C2T	**HT: 1300/1hr, Air**	**900**	**3**	**146**		**46.81**
#08, C2T	**HT:1250/10hr, Air**	**900**	**2**	**1093**		**48.66**
#08, C2T	**HT:1250/10hr, Air**	**900**	**3**	**1093**	**7e-4**	**46.37**
#05, C2T	**HT:1250/10hr, Air**	**1000**	**1**	**208**		**51.15**
#10, C2T	**HT: 1200/1hr, Air**	**900**	**2**	**1325**		**48.83**
#10, C2T	**HT: 1200/1hr, Air**	**900**	**3**	**299**	**4e-5**	**47.47**
#15, C2T	**HT: 1200/1hr, Air**	**1000**	**1**	**3796***	**1e-4**	**54.04***
#19, C2T	**HT: 1175/1hr, Argon**	**900**	**2**	**3191***		**49.64***
#19, C2T	**HT: 1175/1hr, Argon**	**900**	**3**	**1968***		**49.20***
#19, C2T	**HT: 1175/1hr, Argon**	**900**	**4**	**963***	**2e-5**	**48.54***
#21, C2T	**CR-25%, 1200/1hr,**	**900**	**3**	**1272***	**1e-8**	**48.80***
#21, C2T	**CR-25%, 1200/1hr,**	**900**	**4**	**268***	**1e-8**	**47.37**
#22, C2T	**CR-25%, 1200/1hr**	**1000**	**1**	**813***	**4e-5**	**52.51**

Aggressive recrystallization heat treatment

Figure 11 shows a direct comparison of creep response for PMWY-3 alloys heat-treated at different temperatures in air. The exact data is from Test #6 and #10 (indicated in Table 4) as tested at 2Ksi stress at 900^{o}C. The 1300^{o}C heat-treatment was conducted by re-heating a test sample previously recrystallized at 1200^{o}C. It is noteworthy that aggressive thermal exposure did not improve creep-life in PMWY-3 much in agreement with our earlier observations for PMWY-2 alloys. A faster creep rate was observed for the 1300^{o}C treatment. Repeated tests, Table 4, indicate that creep response deteriorates with increasing temperature for the recrystallization treatment.

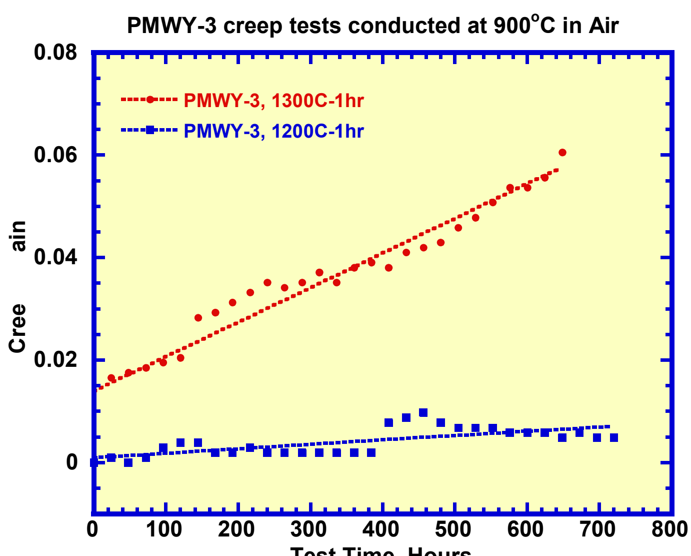


Figure 11. Comparison of creep rates for different recrystallization heat-treatments.

Figure 12 shows a comparison of the transverse creep failures observed for the test#6 and test#10 (as plotted in Figure 8). Note that test#6 failed at 2Ksi whereas test#10 failed at the incremented 3Ksi stress. Both failures appear to have originated in the vicinity of the grain boundary and failed via void formation and coalescence. The ductile lobes have a scale of the order of 60μm in test #6 (HT; 1300°C-1hr) compared to about 200μm for test #10 (HT: 1200°C-1hr). Such lobe detail would suggest that significant plastic deformation occurred in the test #6 which is indeed corroborated by the 4% creep strain observed, Figure 8. This observation is in agreement with all PMWY-2 transverse creep tests, Table 3, that fail at appreciable strains with significant fine lobe formation. In contrast, PMWY-3 samples with improved creep life exhibit limited creep-strain prior to failure, despite their lower over-all creep rates.

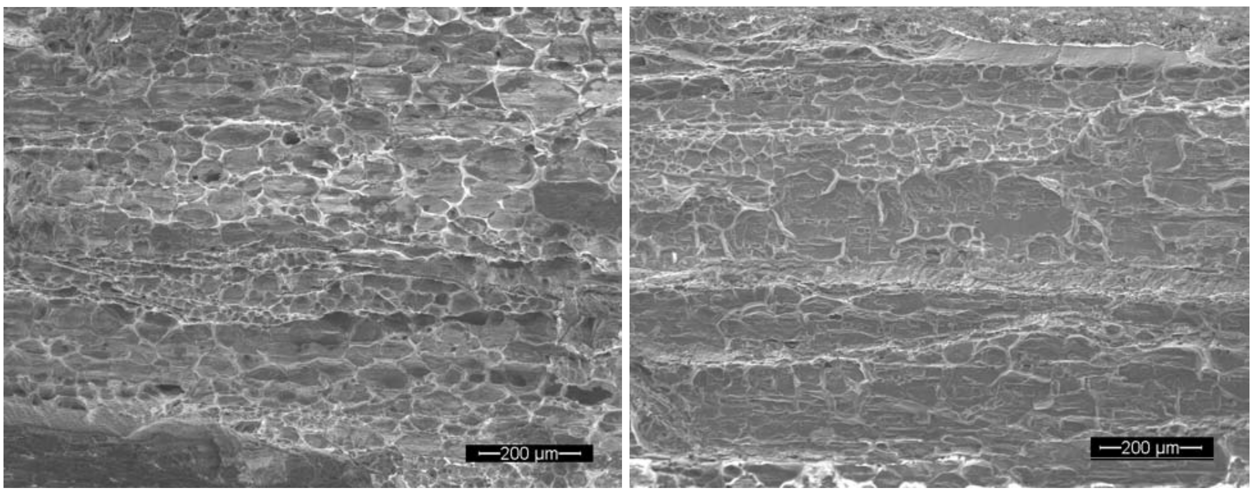


Figure 12. Transverse creep failures in PMWY-3 alloy tube samples for a) 1300°C-1hr, b) 1200°C-1hr heat-treatments in air. Failure occurred at 2Ksi in (a) and at 3Ksi in (b).

Variations in recrystallization environment

Efforts to control recrystallization environment were prompted by the observations of creep response dependence on recrystallization temperature. It is reasoned that variations in aluminum loss in the thin test specimens at the respective high recrystallization temperatures may account for the creep response. Thus specimens were heat-treated in an argon atmosphere at 1175°C for about 1 hour. Such inert treatment ensures that the minimum level of aluminum loss will be incurred at the 900-1000°C test temperatures.

Table 4 shows one such incremental load test #19 currently in progress. Test #19 has about 3191 hours of total exposure at 2Ksi, 1968 hours of exposure at 3Ksi increment and finally about 963 hours of exposure at the 4Ksi increment. No failures have been observed till date – and consequently further microstructural insights are awaited.

Variations in thermo-mechanical processing

Thermo-mechanical processing efforts have been prompted by the extreme longitudinal vs. transverse (hoop) creep anisotropy in ODS-Fe_3Al and MA-956 alloys. Clearly there are different mechanisms of creep failure at work in the orthogonal orientations. Longitudinal creep limit is dictated by ODS strengthening of the matrix while transverse creep strength is limited by the grain boundary void formation and coalescence. Such boundary void formation is further enhanced by the presence of large impurity oxides and unrecrystallized stringers aligned along the primary extrusion axis (i.e., normal to the hoop loading axis). Thus the strength anisotropy is linked with the underlying grain structure of the processed tubes.

With respect to the end-use application of internally pressurized tubes, the hoop stress required is twice the longitudinal stress. This application specific requirement is at odds with (and inverse of) the intrinsic material anisotropy illustrated in Figure 6. What is clearly desired is an exploration of likely thermo-mechanical methodologies to alter the underlying grain structure. Such a process is routine in plates and sheets, which are cross-rolled to effect isotropic in-plane response. Cross rolling of tubes is a challenge and we seek to identify the threshold of cross-rolling strains that can produce a 100% improvement in creep response over the current limit.

Flattened tube segments (of as-extruded canned tubes) were cross-rolled at 900^{o}C in multiple passes to a total of 25% thickness reduction. Samples in the transverse orientation (to the original tube) were spark machined from the rolled plate and ground to remove the canned layer. They were further pre-oxidized at 900^{o}C for 30 minutes prior to the recrystallization heat-treatment of 1200^{o}C for 1 hour in air. Table 4 lists two continuing tests #21 and #22 for cross-rolled specimens. We note that initial transient creep rates are of the order of 1e-8/day for the 900^{o}C test at 4Ksi and 4e-4/day for the 1000^{o}C test at 1Ksi. The observed creep rates are an order of magnitude better than those recorded for any other recrystallization treatment. No failures have been observed till date – and consequently further microstructural insights are awaited. Further plans to perform recrystallization under inert atmospheres are underway and will be evaluated for hoop creep performance in the near future.

Summary and Conclusions

High temperature creep response in ODS-Fe_3Al tubes is reported for the longitudinal and transverse orientations. The kinetics of grain growth are affected by interstitial impurity content, which limit the extent of recrystallized regions observed in the tube wall. Consequently, the microstructures exhibiting the best creep response are ones of high purity that undergo complete recrystallization.

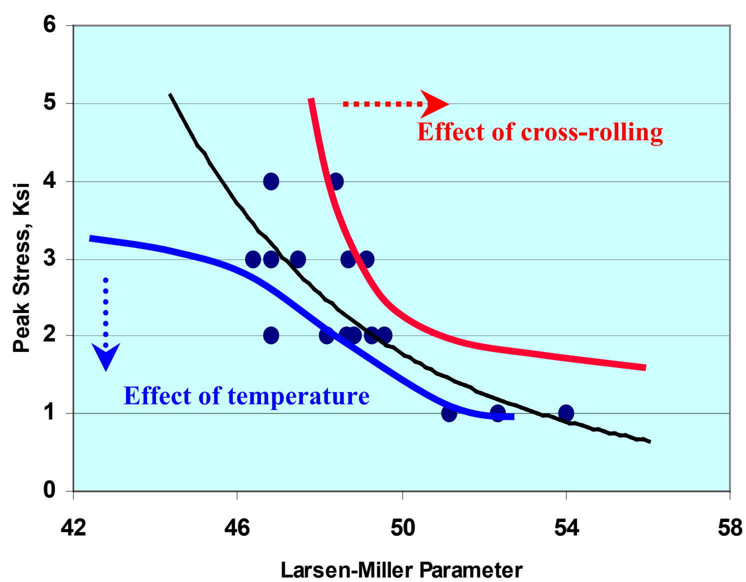


Figure 13. Processing dependent hoop creep response.

Figure 13 is a composite illustration of the effect of various thermal-mechanical treatments on the ensuing hoop creep response in PMWY-3 alloys. The black curve is a median representation of all test results presented in Table 4 and best fit for Test #19 (inert recrystallization). The blue curve indicates creep performance decay at aggressive thermal treatments and environments. The red curve is a partial construct of the beneficial effects of cross-rolling that is expected to alter the underlying grain structure and consequently arrive at a more optimum matching of intrinsic material property with the end-use stress requirement. The results of this study are summarized as:

- Powder batch milling appears to be the single most pervasive processing component dominating microstructural and material response. It is suggested that coarse nitrides and oxides of aluminum (formed during milling) inhibit recrystallization grain growth in PMWY-1 and PMWY-2 alloys.

- Creep response of the respective powder batches is proportional to the underlying grain structure produced via heat-treatments. Thus powder batch PMWY-3 with its completely recrystallized tube wall section offers the best creep response.

- Recrystallization environment plays a significant role in affecting hoop creep response. For equivalent recrystallization treatments, an argon environment produced significant improvements in creep life.

- Thermo-mechanical processing to alter the underlying grain shape and structure has a directly beneficial effect on improving hoop creep response. These enhancements are beyond those achieved by recrystallization in inert atmospheres based on the negligible creep rates observed till date.

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