Oxide Dispersion Strengthened Fe$_3$Al-Based Alloy Tubes: Application Specific Development for the Power Generation Industry

Research Sponsored by the US Department of Energy
Office of Fossil Energy
Advanced Research and Technology Development Program

Report Prepared by

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

Under
19X-SY009C

for
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831

Managed by
LOCKHEED MARTIN ENERGY RESEARCH CORP.

for the
DEPARTMENT OF ENERGY
Under contract DE-AC05-96OR22464

July 1999
DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, make any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.
DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.
Oxide Dispersion Strengthened Fe₃Al-Based Alloy Tubes: Application Specific Development for the Power Generation Industry

Research Sponsored by the US Department of Energy
Office of Fossil Energy
Advanced Research and Technology Development Program

Report Prepared by

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

Under
19X-SY009C

for
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831

Managed by
LOCKHEED MARTIN ENERGY RESEARCH CORP.

for the
DEPARTMENT OF ENERGY
Under contract DE-AC05-96OR22464

July 1999
Oxide Dispersion Strengthened Fe₃Al-Based Alloy Tubes:
Application Specific Development for the Power Generation Industry

Table of Contents

Progress Summary 1
Introduction 2
Program Particulars 2
Progress: Iteration 1 rod extrusions 4
As-extruded Mechanical Anisotropy 5
As-extruded Texture Anisotropy 5
Recrystallization Kinetics 6
Recrystallization Kinetics with Prestrain 7
Progress: Iteration 2 rod extrusions 9
As-extruded Texture Anisotropy 9
Recrystallization Kinetics 9
Progress: Iteration 3 tube extrusions 12
Hardness Properties & Recrystallization Kinetics 14
Recrystallized Grain Shapes & Morphologies 15
Summary and Conclusions 20
Acknowledgements 20
References 20
Appendix

New Alloys Distribution List
Oxide Dispersion Strengthened Fe₃Al-Based Alloy Tubes: Application Specific Development for the Power Generation Industry

**Progress Summary**

A detailed and comprehensive research and development methodology is being prescribed to produce Oxide Dispersion Strengthened (ODS)-Fe₃Al thin walled tubes, using powder extrusion methodologies, for eventual use at operating temperatures of up to 1100°C in the power generation industry. A particular 'in service application' anomaly of Fe₃Al-based alloys is that the environmental resistance is maintained up to 1200°C, well beyond where such alloys retain sufficient mechanical strength. Grain boundary creep processes at such high temperatures are anticipated to be the dominant failure mechanism.

Thus, the challenges of this program are manifold: 1) to produce thin walled ODS-Fe₃Al tubes, employing powder extrusion methodologies, with 2) adequate increased strength for service at operating temperatures, and 3) to mitigate creep failures by enhancing the as-processed grain size in ODS-Fe₃Al tubes.

Our research progress till date, has resulted in the successful batch production of typically 8 Ft. lengths of 1-3/8" diameter, 1/8" wall thickness, ODS-Fe₃Al tubes via a proprietary single step extrusion consolidation process. The process parameters for such consolidation methodologies have been prescribed and evaluated as being routinely reproducible. Such processing parameters (i.e., extrusion ratios, temperature, can design etc.) were particularly guided by the need to effect post-extrusion recrystallization and grain growth at a sufficiently low temperature, while still meeting the creep requirement at service temperatures. 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. Current efforts are now focused on material texture anisotropy, and the specifics of deformation strain environment in the extrusion process to control the recrystallized grain size, and more importantly the grain shape. The role of texture anisotropy is being evaluated, in particular, its contribution to producing near single crystal regions in the 1/8" thick tube cross-sections.


Introduction

Fe$_3$Al-based alloys are promising materials for high temperature, high pressure, tubing applications, on account of 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$_3$Al-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 dispersoid strengthening (ODS), are required to provide adequate strength at the higher service temperatures.

The target applications for ODS-Fe$_3$Al 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. The economic incentive is the low cost of Fe$_3$Al-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 Particulars

In December 1997, the University of California-San Diego (UCSD) was awarded a research subcontract to engage in a detailed and comprehensive research and development effort to produce thin walled ODS-Fe$_3$Al tubes, using powder extrusion methodologies, for eventual use at operating temperatures of up to 1100°C in the power generation industry. Grain boundary dominated creep processes at such high service temperatures are anticipated to be the dominant failure mechanism.

Within the framework of this intended target application, the development of suitable materials containing Y$_2$O$_3$ 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.

In this current project we address manufacturing issues and development efforts towards our stated development goal. The challenges of this program are many-fold: 1) to produce thin walled ODS-Fe$_3$Al tubes, employing powder extrusion methodologies, with 2) adequate increased strength for service at operating temperatures, and 3) to mitigate creep failures by enhancing the as-processed grain size in ODS-Fe$_3$Al tubes. The detailed task structure is shown in Figure 1. The project is iterative in nature, intended to systematically examine the various sub-processes for optimum performance and cost considerations. This entails i) characterizing initial starting materials, ii) prescription of consolidation methodologies (single vs. multiple step) to create hollow tubes, and iii) prescribing zone annealing and recrystallization schedules to create large grain sized creep resistant tubes.
Progress Status and Report

This interim report describes the initial processing, microstructure and properties of an ODS-Fe₃Al alloy tubes. In particular, we examine the strength and texture anisotropies of these highly deformed as-extruded tubes, with a view to possibly affecting secondary recrystallization kinetics during post-extrusion processing to create large grains. Our work till date has focused on Tasks 1-3, and our progress in these tasked areas is outlined below, in iterative sequence. The progress reported here has been previously described in Quarterly Management Reports, and in the 12th and 13th Fossil Energy Conference Reports [6]. Thus, for the sake of brevity only the most pertinent results are presented here.

Our research has resulted in the successful batch production of typically 8 Ft. lengths of 1-3/8" diameter, 1/8" thick ODS-Fe₃Al tubes via a proprietary single step extrusion consolidation process. The process parameters for such consolidation methodologies have been prescribed and evaluated as being routinely reproducible. Such processing parameters (i.e., extrusion ratios, temperature, can design etc.) were particularly guided by the need to effect post-extrusion recrystallization and grain growth at a sufficiently low temperature, while still meeting the creep requirement at service temperatures. 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.
Progress: Iteration 1

The Fe$_3$Al + 0.5% Y$_2$O$_3$ composition mix was tentatively optimized at ORNL, and three separate batches were milled, identified as PMWY-1, PMWY-2 PMWY-3 in Table 1, details of which are available elsewhere [3]. PMWY-1 powder batch contained the maximum amount of interstitial impurities, and PMWY-3 the minimum. For the purposes of initial extrusion consolidation, the intermediate level impurity powder PMWY-2 was employed.

At the outset, experimental efforts were undertaken to consolidate the ODS powder, to produce sound stock material to be used for materials characterization and preliminary recrystallization studies. In this first iteration powder consolidation extrusions were carried out carbon steel billets.

Table 1: Chemical analyses of the as-received and milled powder batches

<table>
<thead>
<tr>
<th>Element</th>
<th>As-Received</th>
<th>PM</th>
<th>PMWY-1</th>
<th>PMWY-2</th>
<th>PMWY-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe</td>
<td>Bal.</td>
<td>79.6</td>
<td>1800 ppm</td>
<td>1900 ppm</td>
<td>1400 ppm</td>
</tr>
<tr>
<td>Al</td>
<td>16.3</td>
<td>18.20</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>2.4</td>
<td>2.18</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zr</td>
<td>20 ppm</td>
<td>26 ppm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>O (total)</td>
<td>60 ppm</td>
<td>110 ppm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>O (in Y$_2$O$_3$)</td>
<td>1025 ppm</td>
<td>1053 ppm</td>
<td>1080 ppm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>O balance</td>
<td>775 ppm</td>
<td>847 ppm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>O pickup</td>
<td>665 ppm</td>
<td>737 ppm</td>
<td>210 ppm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>N pickup</td>
<td>18 ppm</td>
<td>7 ppm</td>
<td>1264 ppm</td>
<td>145 ppm</td>
<td>88 ppm</td>
</tr>
<tr>
<td>N</td>
<td>18 ppm</td>
<td>7 ppm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C pickup</td>
<td>24 ppm</td>
<td>667 ppm</td>
<td>360 ppm</td>
<td>303 ppm</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>24 ppm</td>
<td>667 ppm</td>
<td>360 ppm</td>
<td>303 ppm</td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>16 ppm</td>
<td>115 ppm</td>
<td>40 ppm</td>
<td>29 ppm</td>
<td></td>
</tr>
<tr>
<td>C+N+O pickup</td>
<td>2565 ppm</td>
<td>1211 ppm</td>
<td>570 ppm</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Bulk compositions are identified in wt%)

The carbon steel cans measured 51-mm OD x 6.4 mm wall x 125 mm length (2-in x 0.25-in x 5-in). The cans were filled with powder, evacuated and sealed. A total of three cans were prepared for direct consolidation of powder into solid bars, the details of which are provided in Table 2.

Table 2: Extrusion consolidation parameters for PMWY-2 powders

<table>
<thead>
<tr>
<th>Extrusion</th>
<th>Die Size</th>
<th>Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number</td>
<td>Temperature</td>
<td>mm</td>
</tr>
<tr>
<td>5016$^a$</td>
<td>900</td>
<td>16.3</td>
</tr>
<tr>
<td>5014$^a$</td>
<td>1000</td>
<td>12.7</td>
</tr>
<tr>
<td>5013$^a$</td>
<td>1100</td>
<td>12.7</td>
</tr>
<tr>
<td>5030$^b$</td>
<td>1000</td>
<td>25.4</td>
</tr>
</tbody>
</table>

$^a$51mm (2.0 in) billets for solid extrusion; $^b$82mm (3.5 in) billet for tube extrusion.
**As-extruded Mechanical Anisotropy**

Knoop hardness measurements were made on longitudinal sections of extruded rods, in directions parallel (0° degrees), inclined (45° degrees) and perpendicular (90° degrees) to the elongated grains. Figure 2 shows the hardness response of the three extrusions, where the data scatter represents variations along the radial dimension of the extrusions. Both the 0° and 90° hardness measurements follow the expected decline with increasing extrusion temperature. The 45° indents show anomalous behavior with temperature. Nonetheless, the strength anisotropy was relatively small, of the order of about 5%, and the material was deemed mechanically isotropic.

![Characterization of Knoop-Hardness Anisotropy in Extruded ODS-Fe₃Al](image)

**As-extruded Texture Anisotropy**

The ODS-Fe₃Al powder extrusions exhibited an extreme texture anisotropy, as observed earlier for cast and extruded Fe₃Al base alloys [5]. A strong \{011\}<uvw> fiber texture was observed
along the extrusion axis, as recorded via X-ray diffraction peaks from transverse and longitudinal sections. A direct comparison of the \{022\} diffraction peaks taken as a general measure of stored energy, Figure 3, indicated diminishing line broadening with extrusion temperature. Several observations are apparent from such measurements: 1) the extrusions show an extreme anisotropy of \{011\} diffraction intensity in the longitudinal and transverse directions for each of the extrusions, and 2) the relative anisotropy increases with extrusion temperature, and 3) the \{011\}\textgreater fiber texture is progressively stronger with extrusion temperature.

![Transverse (022) Texture Alignment in Extruded ODs-Fe₃Al](image)

Figure 3. Comparison of \{022\} textural alignment with increasing extrusion temperature.

**Recrystallization Kinetics**

In this first iteration, recrystallization kinetics were studied only for the 900°C extrusion, of a lower extrusion ratio of 9.8:1. Samples were spark machined from the rod cross-section and heat-treated in air using a muffle furnace at 1100-1300°C for 1 hour. Figure 4 shows the respective optical micrographs of longitudinal sections of as-extruded, and the heat-treated specimen. Figure 5 shows the hardness response of the heat-treated samples where the as-extruded hardness of 530 DPH falls to about 340 DPH for the specimen treated at 1300°C. A large drop in strength occurred at 1100°C in the recovery stage, where no appreciable grain growth was observed. We also noted that a small hardness plateau was observed in the 1200-1250°C temperature range, beyond which the hardness continued to decrease, along with the observation of exaggerated grain growth. While complete recrystallization was obtained at 1300°C, this was accompanied by increased void formation in the solid rods. Thus, the processing parameters required a revision, in the following iteration, to enhance the kinetics, and reduce the recrystallization temperature.
Effect of Recrystallization Temperature on Mechanical Response

Figure 4. Optical micrographs of a) as-extruded rod, and b) heat-treated at 1300°C/1hr.

Figure 5. Vickers Hardness decay in response to the recrystallization heat-treatments.

Recrystallization Kinetics with Pre-Strain

A set of specimens were deformed in compression (ε=8%, 16%), along the prior extrusion axis, at strain rates of $10^{-3}$ sec$^{-1}$. The specimens exhibited cracking along the compression axis, but were nonetheless heat-treated at 1100°C and 1200°C for one hour. Figure 6 shows hardness response due to the various thermal treatments. A small amount of prestrain was particularly helpful at the lower temperatures of 1100°C in promoting grain growth, but this effect was
essentially non-existent for treatments at 1200°C. Figure 7 shows a direct comparison of the longitudinal section microstructures of the as-extruded, as heat-treated, and pre-strained + heat-treated samples, where the latter exhibited a grain size of the order of 25μm. It was deemed likely that such pre-straining, or increasing the extrusion deformation strain, may be employed to accelerate the recrystallization kinetics, as attempted in iteration 2.

![Effect of Post-Extrusion Straining on Recrystallization Kinetics](image)

**Figure 6.** Hardness response of recrystallized specimens with and without pre-straining.

![Comparison of microstructures](image)

**Figure 7.** Comparison of a) as-extruded, b) heat treated at 1100°C-1hr, and c) pre-strained and heat treated at 1100°C-1hr.
Progress: Iteration 2

At the conclusion of the first iteration, the broad parameters of extrusion consolidation were identified. Furthermore, it was shown that the recrystallization kinetics of an as-extruded stock material, could be altered via post-extrusion straining techniques, as illustrated in Figures 6-7. However post-extrusion straining bears a certain cost, and thus a revised extrusion schedule was attempted on a second batch of extrusions to incorporate greater deformation strain.

In this second iteration, powder consolidation extrusions of PMWY-1, PMWY-2 and PMWY-3 material batches were carried out in carbon steel billets. Each of the carbon steel cans measured 51-mm OD x 6.4 mm wall x 125 mm length (2-in x 0.25-in x 5-in). The cans were filled with powder, evacuated and sealed. A total of three cans were prepared for direct consolidation at a 16:1 extrusion ratio, at 1000°C, the details of which are provided in Table 3.

<table>
<thead>
<tr>
<th>Material</th>
<th>Temperature</th>
<th>Die Size</th>
<th>Area Reduction</th>
<th>Tonnage</th>
</tr>
</thead>
<tbody>
<tr>
<td>PMWY-1a</td>
<td>1000</td>
<td>12.7</td>
<td>0.50</td>
<td>16.0:1</td>
</tr>
<tr>
<td>PMWY-2a</td>
<td>1000</td>
<td>12.7</td>
<td>0.50</td>
<td>16.0:1</td>
</tr>
<tr>
<td>PMWY-3a</td>
<td>1000</td>
<td>12.7</td>
<td>0.50</td>
<td>16.0:1</td>
</tr>
</tbody>
</table>

Table 3: Extrusion consolidation parameters for PMWY-1-3 powders

As-extruded Texture Anisotropy

A strong {011}<uvw> fiber texture was observed along the extrusion axis for each of the extruded rods of PMWY-1, PMWY-2, and PMWY-3 materials. A direct comparison of the {022} diffraction peaks for the transverse and longitudinal sections of the extruded rods is shown in Figure 8a-b. Results indicated that the textural alignment was the strongest for PMWY-1 and the weakest for PMWY-2. A further examination of the transverse versus longitudinal textures in each of the rods indicated that PMWY-1 exhibited the maximum anisotropy of ≈25 whereas this anisotropy was only of the order of 8-10 for the PMWY-2 extrusion. This variation in anisotropy is not well understood, particularly as the extrusion parameters were essentially identical for all the three bars. Nonetheless, these parameters are routinely tracked in an effort to establish correlations with recrystallization kinetics and characteristics.

Recrystallization Kinetics

A marked improvement in recrystallization kinetics was observed with this increased extrusion ratio. In essence all three rods could be recrystallized (grain size ≈ 1-2 mm) with the heat treatment schedule not exceeding 10 hours at 1200°C. For the sake of brevity, the grain growth behavior of PMWY-1 and PMWY-2 rods is shown in Figures 9 and 10, respectively. In the cross-section view
Comparison of as-extruded transverse textures for PMWY-1-3 rods

<table>
<thead>
<tr>
<th>PMWY-1 @1000/T</th>
<th>PMWY-2 @1000/T</th>
<th>PMWY-3 @1000/T</th>
</tr>
</thead>
</table>

Diffraction Angle, 2-Theta

Comparison of as-extruded longitudinal textures for PMWY-1-3 rods

<table>
<thead>
<tr>
<th>PMWY-1 @1000/L</th>
<th>PMWY-2 @1000/L</th>
<th>PMWY-3 @1000/L</th>
</tr>
</thead>
</table>

Diffraction Angle, 2-Theta

Figure 8. Comparison of {022} texture alignment in the a) transverse and b) longitudinal sections of PMWY-1, PMWY-2 and PMWY-3 extruded rods.

of Figure 9, there were only two grains in the cross-section of the extruded rods. However, for the PMWY-2 material, Figure 10, the exterior was essentially a single grain, but the central section was comprised of multiple grains, with grain size of the order of 1-2 mm. Furthermore, the grain shape morphology was extremely contorted, with interpenetrating segments.
Figure 9. Optical micrographs of a) transverse, and b) longitudinal sections of the 1/2" extruded rod of PMWY-1 material, heat-treated at 1200°C-1hr.

Figure 10. Optical micrographs of transverse section of the 1/2" extruded rod of PMWY-2 material, heat-treated at 1200°C-1hr.

The interpenetrating nature of the grains is further illustrated in Figure 10a-b, as extracted from transverse and longitudinal sections of the PMWY-2 sample. The grains essentially exhibit a cactus like structure with the net result of providing mechanical interlocking between adjacent grains. The etching contrast in the cross-section view of Figure 10a, indicates several such interpenetrating islands. Such features are considered important from the viewpoint of developing creep-resistant materials, and will be particularly examined in Task 4 of this program.
Progress: Iteration 3

Following the second iterative process, it was concluded that the modified extrusion parameters continued to yield sound extruded stock, with a marked increase in recrystallization kinetics. Thus large grain size grain sized material was obtained by routine heat-treatments in the 1100-1200° range, with hold times of less than 100 hours.

In this third iteration, the process parameters developed and refined in iterations 1 and 2 for rod stock, were applied to annular extrusions in an effort to directly produce tube stock. We note here that annular extrusions were a modification prescribed and accepted during the course of this research program. Typically tube products are produced via a two stage process, i.e., the first step involves creating a bulk ingot of a cylindrical cross-section, which is then re-worked to a hollow tube using different production methodologies. For specialty alloys and materials, this process typically involves drilling a center hole (i.e., gun drilling) before sizing the tube to required dimensions. In our modified process, it was proposed to combine the consolidation step with the tube drilling step via the use of an annular extrusion can. The relative manufacturing advantages and disadvantages of this single step process are illustrated briefly in Figure 12.

The benefits of this single step process are readily apparent, and four different extrusions were attempted as described in Table 4. Thus about 6-8 Ft. lengths of 1-3/8" diameter, 1/8" wall thickness, ODS-Fe₃Al tubes were produced via the single step extrusion consolidation process.
Comparison of Tube Extrusion Methodologies

**multiple step**

- Powder Can Extrusion
- Gun Drilling
- Mandrel Extrusion

**Advantages:**
- Clean inner surface
- Better dimensional tolerance

**Disadvantages:**
- multiple heating/extrusion steps
- poor product yield

**single step**

- Annular Powder Can Mandrel Extrusion

**Advantages:**
- energy efficient manufacturing
- improved product yield

**Disadvantages**
- annular can design complexities
- inside surface can layer removal

Figure 11: Comparison of single vs. multiple step extrusion consolidation methodologies.

Table 4: Tube extrusion consolidation parameters for PMWY-1-3 powders

<table>
<thead>
<tr>
<th>Extrusion</th>
<th>Temperature</th>
<th>Die Size</th>
<th>Mandrel Size</th>
<th>Area Reduction</th>
<th>Area Tonnage</th>
</tr>
</thead>
<tbody>
<tr>
<td>PMWY-1&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1000°C</td>
<td>1.375</td>
<td>1.00</td>
<td>≈16.0:1</td>
<td>NA</td>
</tr>
<tr>
<td>PMWY-2&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1000°C</td>
<td>1.375</td>
<td>1.00</td>
<td>≈16.0:1</td>
<td>NA</td>
</tr>
<tr>
<td>PMWY-3&lt;sup&gt;a&lt;/sup&gt;</td>
<td>1000°C</td>
<td>1.375</td>
<td>1.00</td>
<td>≈16.0:1</td>
<td>NA</td>
</tr>
</tbody>
</table>

<sup>a</sup>102 mm (4.0 in) billets for thin-walled tube extrusion
Figure 13 shows a set of tubes in the as-extruded as well as in the surface finished condition. The surface finishing is intended to remove only the outside steel case, and no attempt was made to remove the inside steel case. The tubes were of sound quality, and exhibited no cracking and/or damage after routine machining operations.

Figure 13. Assorted ODS-Fe₃Al tubes in the as-extruded (below), and surface finished (top) condition, as produced from an annular can (top left) consolidation methodology.

**Hardness Properties & Recrystallization Kinetics**

The microhardness measurements on longitudinal sections of the as-extruded, and the recrystallized tubes, are illustrated in Figure 14. The as-extruded hardesses were of the order of 450-480 DPH, with PMWY-1 material being the hardest and PMWY-3 the softest. In previous iterations, it was ascertained that large recrystallized grains could be produced by heat-treatments in the 1100°C-100hrs to 1200°C-10 hours range. Thus, in this iteration, the heat-treatment schedules were focused to three sets of conditions as; i) 1100°C for 100 hrs, ii) 1200°C for 1-hr, and iii) 1200°C for 10 hrs.

The heat-treated tubes yielded nearly flat (i.e., stable) hardness profile for the three heat-treatment schedules, with the exception of PMWY-2 tubes heat-treated at 1200°C-1hr exhibiting some softening at the extended hold time. This flat profile was concomitant with a recrystallized state, similar to that observed previously for 1/2" rod extrusions (see Figure 5, iteration 1). For example PMWY-1 had the highest level of interstitial impurities and PMWY-3 the least, which correlated with PMWY-1 exhibiting a higher micro-hardness in the as-extruded and the heat-treated
conditions. This correlation with interstitial impurity (introduced during the milling process) points to a possible avenue of additional strength improvements.

Mechanical Response of Extruded Tubes to Heat-Treatment Schedules

![Graph showing microhardness response to heat-treatment schedules for PMWY-1, PMWY-2, and PMWY-3 tubes.](image)

Figure 14. Material microhardness response in the as-extruded, as well as recrystallized state, for the PMWY-1, PMWY-2 and PMWY-3 extruded tubes. Note that the near flat hardness profile indicates a stable recrystallized state for all the powder batches.

Recrystallized Grain Shapes & Morphologies

The hardness measurements of Figure 14 indicated that the materials were recrystallized for all the three heat-treatments, which was also confirmed via optical metallographic observations for all the specimens. For the sake of brevity, we report here a single set of longitudinal and transverse micrographs for the PMWY-1, PMWY-2 and PMWY-3 tubes heat-treated at 1200°C for 1 hr. A generic view of the entire cross-section of the sectioned tubes is shown in Figure 15, indicating large scale grain growth along the tube circumference.

Figure 16a-b shows the longitudinal and transverse views of the PMWY-1 tube respectively, over the entire wall-thickness, including the exterior (at left) and interior (at right) steel-case. A reaction zone about 200um thick was observed on the exterior surface of the Fe3Al tube, and was characterized by grain boundaries aligned roughly along the radial direction. In the Fe3Al tube, an average of three grains were observed over the wall thickness, as illustrated via etching contrast differences in the longitudinal and cross-section views. Most importantly, these grains were extended along the circumferential direction, akin to rings fitting over each-other. In addition, these circular grains exhibited the familiar interpenetrating features (see Figure 11) in the vicinity of their grain-grain interfaces. This grain alignment is particularly noteworthy, for pressurized tubes, where the tangential loading is twice as severe as the longitudinal loading. Such a tangential grain
morphology, ensures that the grain boundaries are essentially parallel to the direction of maximum loading. Aside from the millimeter scale processed grain sizes, such a grain shape morphology is expected to have a beneficial effect on creep-performance. This will be the subject of attention during 'in-service property' evaluations for these tubes in Task 4.

Figure 17a-b shows the longitudinal and transverse views of the PMWY-2 tube, over the entire wall-thickness, including the exterior (at left) and interior (at right) steel-case. A reaction zone about 225µm thick was observed on the exterior surface of the Fe₃Al tube. In the Fe₃Al tube, an average of three to five grains were observed over the wall thickness. Both the exterior and interior extremities of the Fe₃Al tube were characterized by the familiar tangentially oriented grains. These grains were nearly continuous over the entire circumference of the tube. However, the middle region was populated by an assortment of inter-penetrating grains none of which extended over the entire circumference.

Figure 18a-b shows the longitudinal and transverse views of the PMWY-3 tube, over the entire wall-thickness, including the exterior (at left) and interior (at right) steel-case. A reaction zone about 225µm thick is observed on the exterior surface of the Fe₃Al tube. Unlike PMWY-1 and PMWY-2 tubes, there were no regions of circumferentially aligned grains. Though large jagged and interpenetrating grains were observed throughout the transverse and longitudinal sections, often spanning the entire wall thickness, they did not extend circumferentially. Additionally, PMWY-3 tubes heat treated at 1100°C-100hrs and 1200°C-10hrs (not shown here) also exhibited similar grain morphologies.

The kinetics of secondary grain growth are somewhat counter-intuitive as powder PMWY-1, with the highest level of impurity particles, consistently yielded near single-crystal cross-section. However, we were unable to observe a similar level of secondary grain growth activity in the cleanest powder PMWY-1. Additionally, the level of voids observed at the interior/exterior steel case interface were the most severe for PMWY-3 and the least for PMWY-1. Such simple correlations with impurity criteria will be the basis of future processing design considerations.
Figure 16. Longitudinal and transverse views of the PMWY-1 tube heat-treated at 1200°/1hr.
Figure 17. Longitudinal and transverse views of the PMWY-2 tube heat-treated at 1200°/1hr.
Figure 18. Longitudinal and transverse views of the PMWY-3 tube heat-treated at 1200°/1hr.
Summary and Conclusions

The oxide dispersion strengthened (ODS) Fe$_3$Al-based alloy powders (PMWY-1, PMWY-2, PMWY-3) were successfully consolidated into solid rods and tubes by the single step hot extrusion methodologies. These extrusion consolidation practices are now being fine-tuned, and we anticipate batch producing about 50-100 feet length of ODS-Fe$_3$Al tube, for our 'in-service performance evaluation' to be undertaken in Task 4 of this research program. The creep performance ODS-Fe$_3$Al materials has been initially studied in flat-rolled samples [4], and deemed acceptable within the prescribed performance envelope. Such creep performance will be evaluated specifically for the tube specimens in the next phase of the research program. The following conclusions are possible based on our studies till date:

1. The ODS-Fe$_3$Al rods and tubes can be fully consolidated at 1000°C and at extrusion ratios of about 16:1, while exhibiting reasonable post-extrusion recrystallization kinetics.

2. All tubes are fully recrystallized, with grain sizes of the order of 200-5000µm, for heat-treatments of temperature-time combinations between 1100°C-100hrs and 1200°C-10hrs.

3. The circumferentially aligned grain morphology in PMWY-1 and PMWY-2 tubes are of particular interest from the perspective of specific loading of internally pressurized tubes, where tangential stress is twice as severe as the longitudinal stress.

4. Secondary grain growth kinetics exhibit an inverse relationship with the total interstitial impurity content of the milled powder batches, with the purest batch PMWY-3 exhibiting the most sluggish grain growth.

Acknowledgements

The support and guidance of Dr. I.G. Wright and Dr. V.K. Sikka is gratefully acknowledged.

References

Appendix

(New Alloys Distribution List)
NEW ALLOYS DISTRIBUTION

ALLISON GAS TURBINE DIVISION
P.O. Box 420
Indianapolis, IN 46206-0420
P. Khandelwal (Speed Code W-5)
R. A. Wenglarz (Speed Code W-16)

AMAX R&D CENTER
5950 McIntyre Street
Golden, CO 80403
T. B. Cox

BABCOCK & WILCOX
Domestic Fossil Operations
20 South Van Buren Avenue
Barberton, OH 44023
M. Gold

BETHLEHEM STEEL CORPORATION
Homer Research Laboratory
Bethlehem, PA 18016
B. L. Bramfitt
J. M. Chilton

BRITISH COAL CORPORATION
Coal Technology Development Division
Stoke Orchard, Cheltenham
Gloucestershire, England GL52 4ZG
J. Oakey

CANADA CENTER FOR MINERAL & ENERGY TECHNOLOGY
568 Booth Street
Ottawa, Ontario
Canada K1A OG1
R. Winston Revie
Mahi Sahoo

COLORADO SCHOOL OF MINES
Department of Metallurgical Engineering
Golden, CO 80401
G. R. Edwards

DOE
DOE OAK RIDGE OPERATIONS
P. O. Box 2008
Building 4500N, MS 6269
Oak Ridge, TN 37831
M. H. Rawlins

DOE
Federal Energy Technology Center
3610 Collins Ferry Road
P.O. Box 880
Morgantown, WV 26507-0880
D. C. Cicero
F. W. Crouse, Jr.
R. A. Dennis
N. T. Holcombe
W. J. Huber
T. J. McMahon
J. E. Notestein

DOE
Federal Energy Technology Center
626 Cochrans Mill Road
P.O. Box 10940
Pittsburgh, PA 15236-0940
A. L. Baldwin
G. V. McGurl
U. Rao
L. A. Ruth
T. M. Torkos

DOE
OFFICE OF FOSSIL ENERGY
FE-72
19901 Germantown Road
Germantown, MD 20874-1290
F. M. Glaser

DOE
OFFICE OF BASIC ENERGY SCIENCES
Materials Sciences Division
ER-131 GTN
Washington, DC 20545
H. M. Kerch
ELECTRIC POWER RESEARCH INSTITUTE
P.O. Box 10412
3412 Hillview Avenue
Palo Alto, CA 94303
W. T. Bakker
J. Stringer

EUROPEAN COMMUNITIES JOINT RESEARCH CENTRE
Petten Establishment
P.O. Box 2
1755 ZG Petten
The Netherlands
M. Van de Voorde

FOSTER WHEELER DEVELOPMENT CORPORATION
Materials Technology Department
John Blizard Research Center
12 Peach Tree Hill Road
Livingston, NJ 07039
J. L. Blough

IDAHO NATIONAL ENGINEERING LABORATORY
P.O. Box 1625
Idaho Falls, ID 83415
R. N. Wright

LAWRENCE BERKELEY LABORATORY
University of California
Berkeley, CA 94720
Ian Brown

LAWRENCE LIVERMORE NATIONAL LABORATORY
P.O. Box 808, L-325
Livermore, CA 94550
W. A. Steele

LEHIGH UNIVERSITY
Materials Science & Engineering
Whitaker Laboratory
5 E. Packer Avenue
Bethlehem, PA 18015
J. N. DuPont

NATIONAL MATERIALS ADVISORY BOARD
National Research Council
2101 Constitution Avenue
Washington, DC 20418
K. M. Zwilsky

OAK RIDGE NATIONAL LABORATORY
P.O. Box 2008
Oak Ridge, TN 37831
M. P. Brady
P. T. Carlson
J. M. Crigger (4 copies)
R. R. Judkins
C. T. Liu
M. L. Santella
J. H. Schneibel
V. K. Sikka
R. W. Swindeman
P. F. Tortorelli
I. G. Wright

PACIFIC NORTHWEST LABORATORY
P. O. Box 999, K3-59
Battelle Boulevard
Richland, WA 99352
R. N. Johnson

SHELL DEVELOPMENT COMPANY
WTC R-1371
P.O. Box 1380
Houston, TX 77251-1380
W. C. Fort