ORNL/Sub/97-SY009/1

Oxide Dispersion Strengthened Fe_3Al-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
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Progress 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 1100°C in the power generation industry. 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. 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-Fe_3Al 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_3Al 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-Fe_3Al 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.

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, 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_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 dispersoid strengthening (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. 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 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_3Al 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_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.

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_3Al 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_3Al 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.

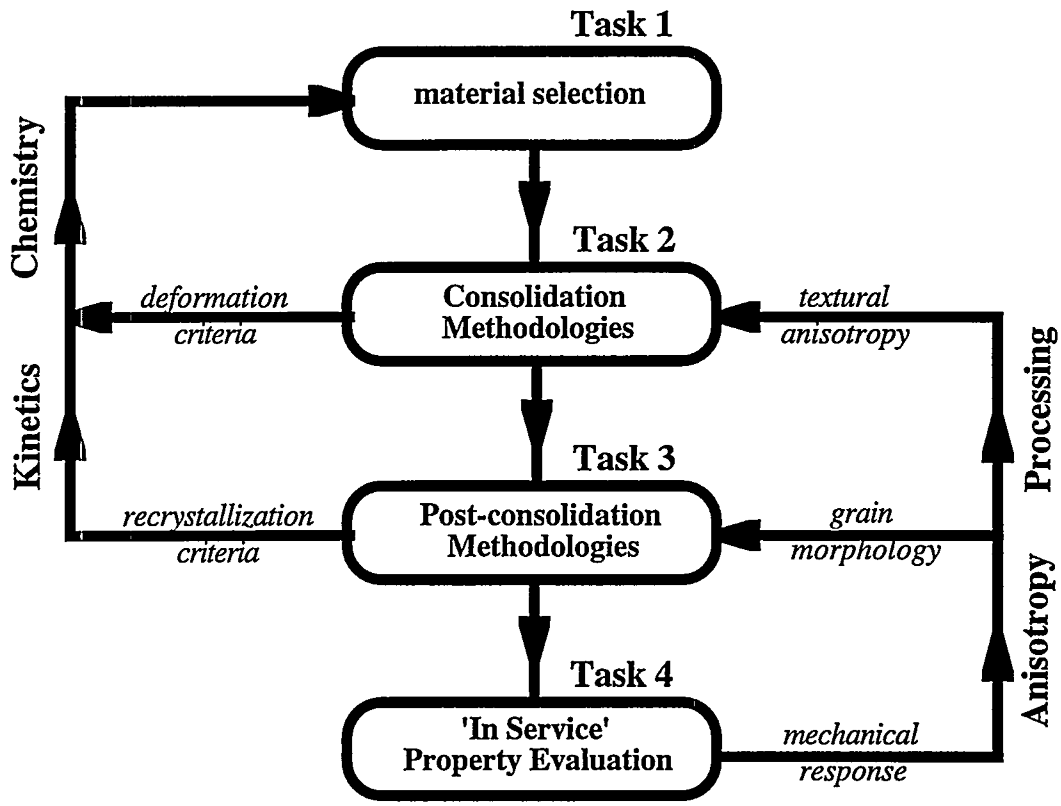


Figure 1. Schematic of the iterative research and development methodology for ODS-Fe_3Al based alloy tubes.

Progress Status and Report

This interim report describes the initial processing, microstructure and properties of an ODS-Fe_3Al 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-Fe_3Al 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_3Al + 0.5% Y_2O_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

Element	As-Received HM	As-Received 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%)

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.

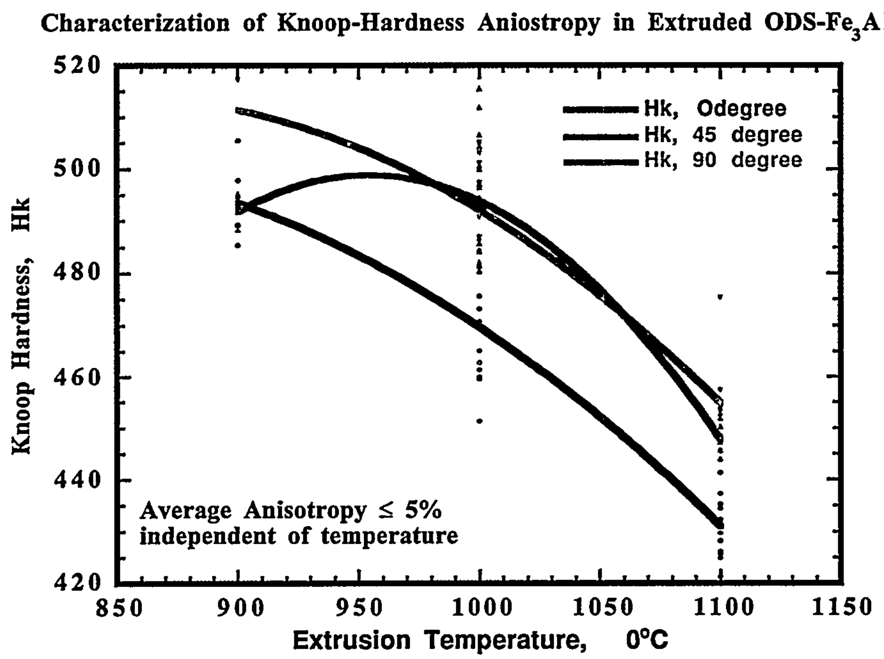
Table 2: Extrusion consolidation parameters for PMWY-2 powders

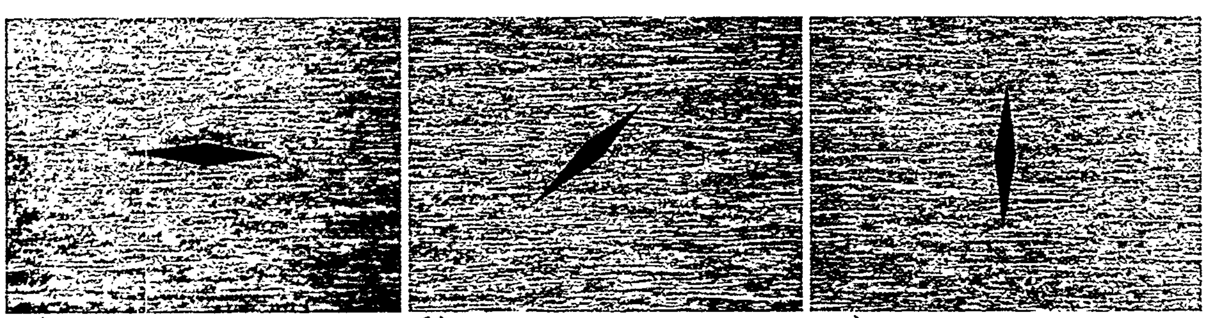
Extrusion Number	Extrusion Temperature	Die Size mm	Die Size inch	Area Reduction	Tonnage
5016[a]	900	16.3	0.64	9.8:1	266
5014[a]	1000	12.7	0.50	16.0:1	290
5013[a]	1100	12.7	0.50	16.0:1	180
5030[b]	1000	25.4	1.00	12.25:1	532

[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.





a) b) c)

Figure 2. Knoop-hardness characterizations of the as-extruded ODS-Fe_3Al rods in the a) parallel (0°), b) inclined (45°), and c) perpendicular (90°) orientations.

As-extruded Texture Anisotropy

The ODS-Fe_3Al powder extrusions exhibited an extreme texture anisotropy, as observed earlier for cast and extruded Fe_3Al 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}<uvw> fiber texture is progressively stronger with extrusion temperature.

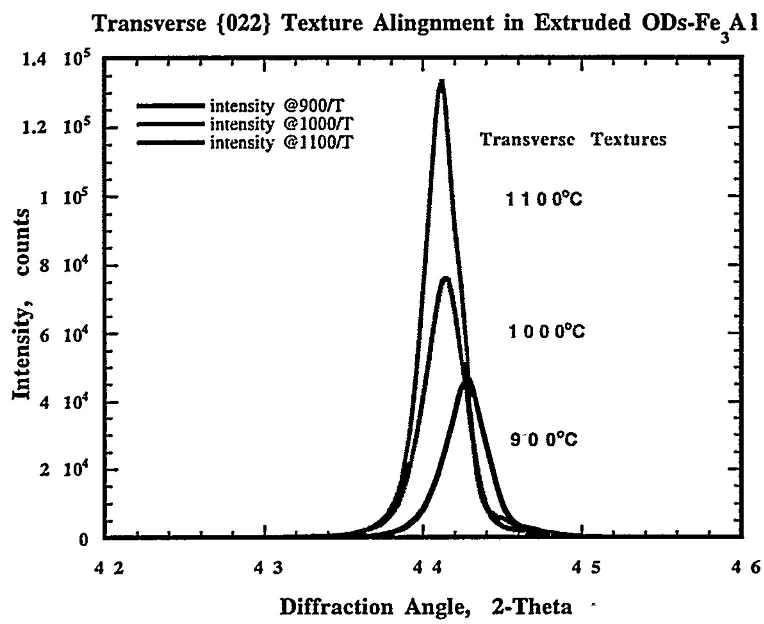


Figure 3. Comparison of {022) textural alignment with increasing extrusion temperature.

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.

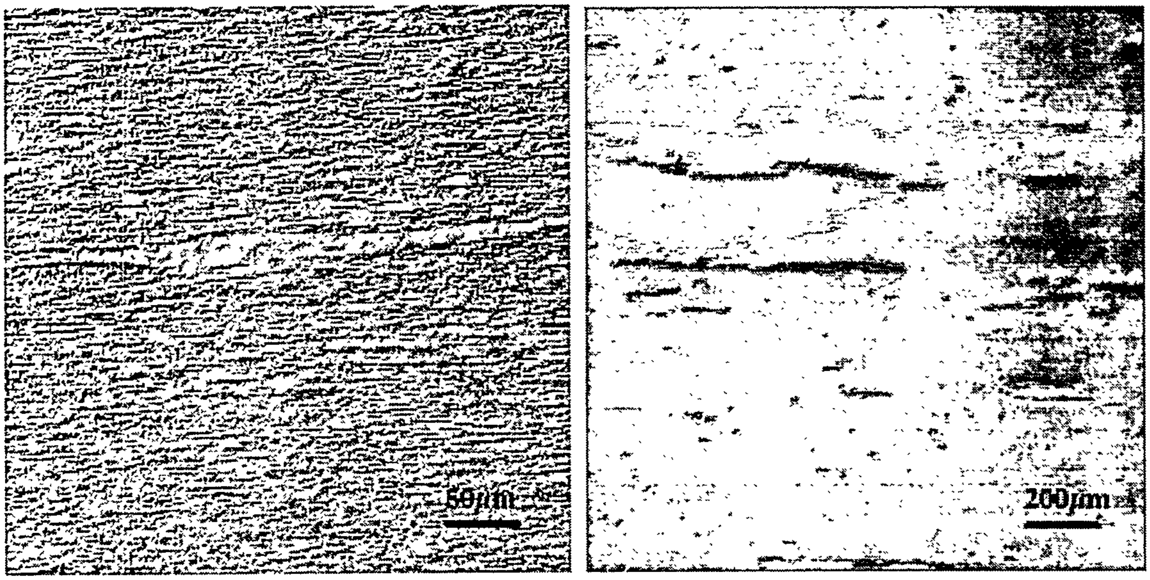


Figure 4. Optical micrographs of a) as-extruded rod, and b) heat-treated at 1300°C/1hr.

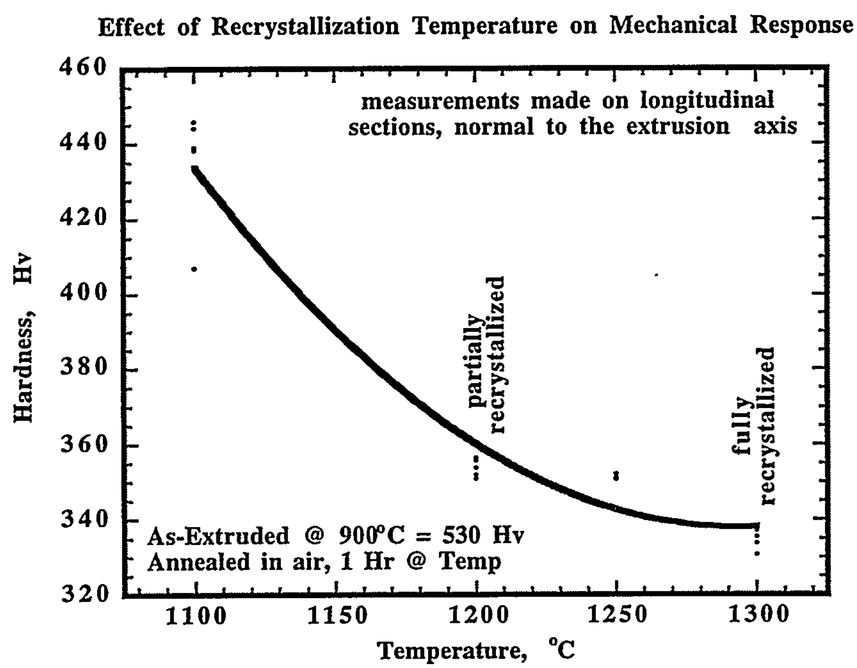


Figure 5. Vickers Hardness decay in response to the recrystallization heat-treatments.

Recrystallization Kinetics with Pre-Strain

A set of specimens were deformed in compression ($e \approx 8\%$, 16%), along the prior extrusion axis, at strain rates of $\approx 10^{-3}$ 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.

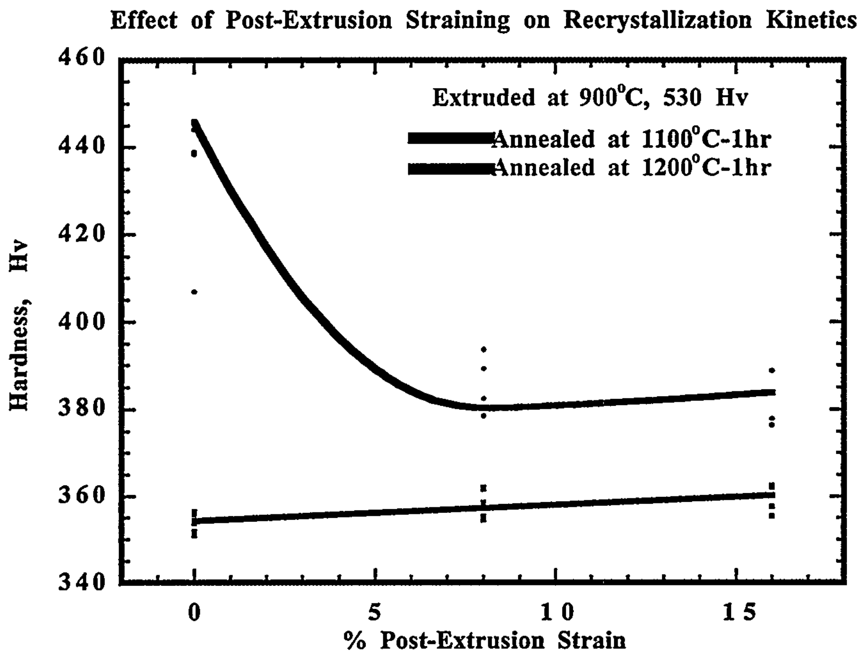


Figure 6. Hardness response of recrystallized specimens with and without pre-straining.

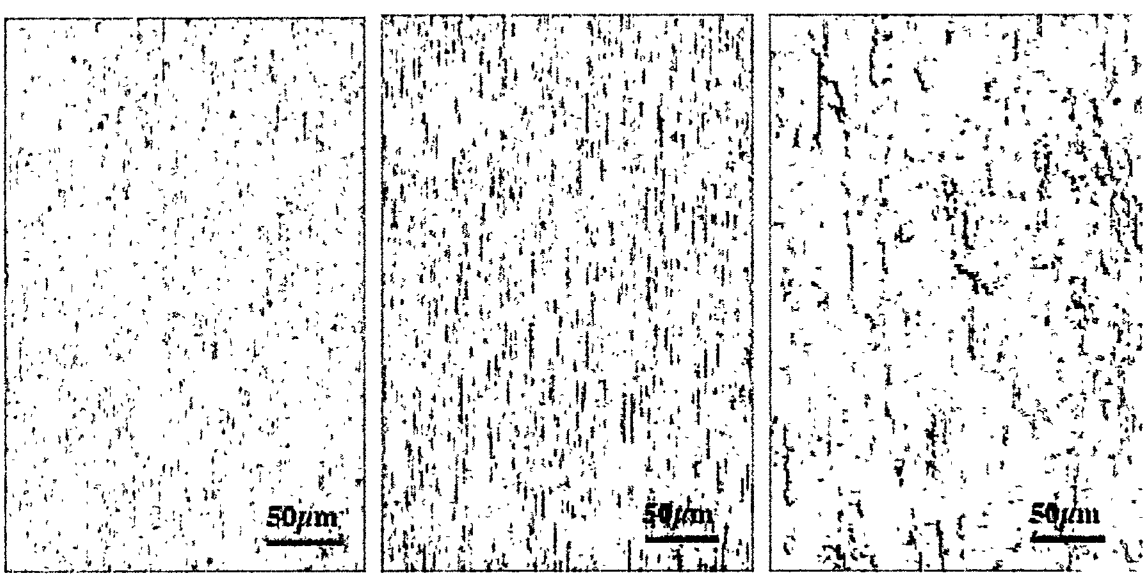


Figure 7. Comparison of a) as-extruded, b) heat treated at 1100°C-1hr, and c) pre-strained and heat treated at 1100°C-1hr.

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

Extrusion		Die Size		Area	
Material	Temperature	mm	inch	Reduction	Tonnage
PMWY-1[a]	1000	12.7	0.50	16.0:1	≈310
PMWY-2[a]	1000	12.7	0.50	16.0:1	≈290
PMWY-3[a]	1000	12.7	0.50	16.0:1	≈290

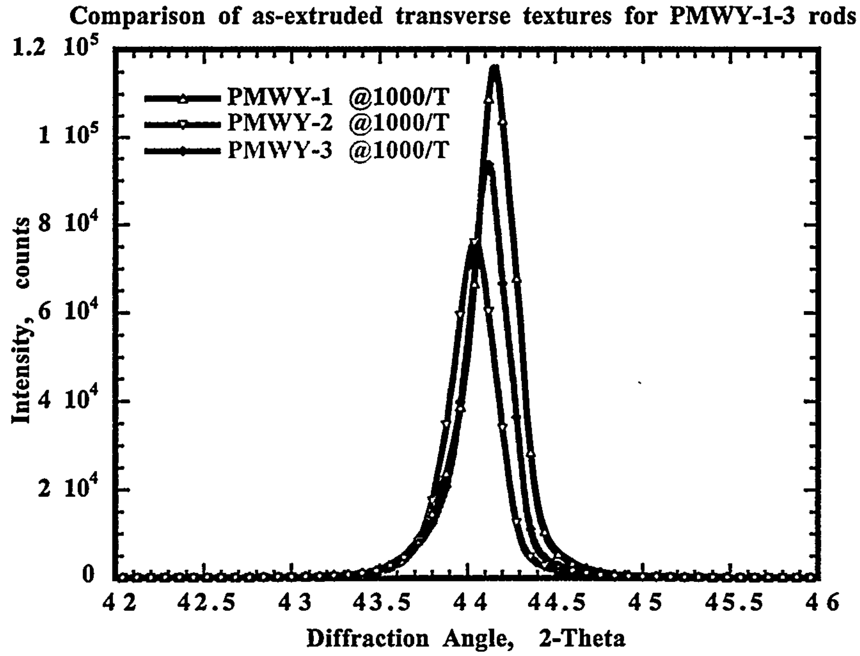
[a]51mm (2.0 in) billets for solid rod extrusion;

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



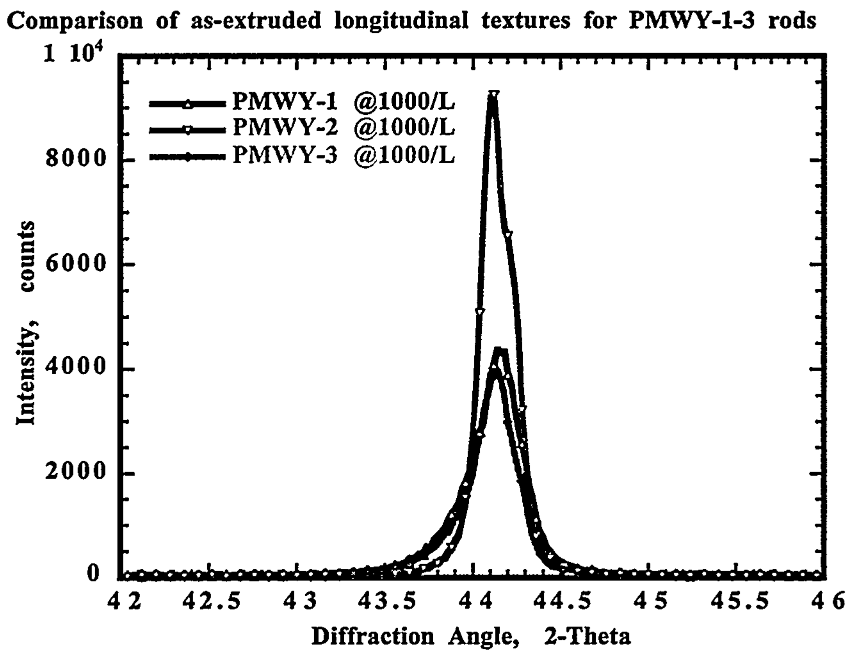


Figure 8. Comparison of {022} texture alignment in the a) transverse and b) longitudinal sections of PMWY-1, PMWY-2 and PMWY-3 extruded rods.

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.

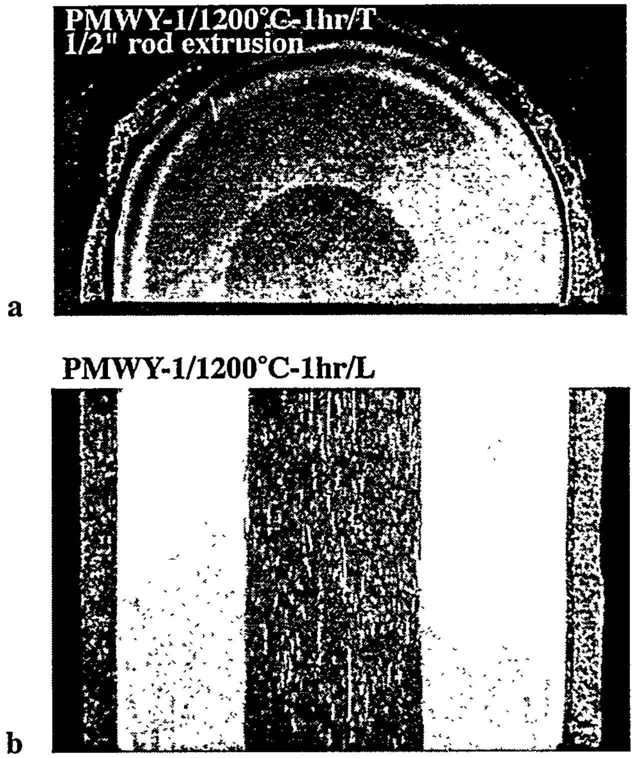


Figure 9. Optical micrographs of a) transverse, and b) longitudinal sections of the 1/2" extruded rod of PMWY-1 material, heat-treated at 1200°C-1hr.

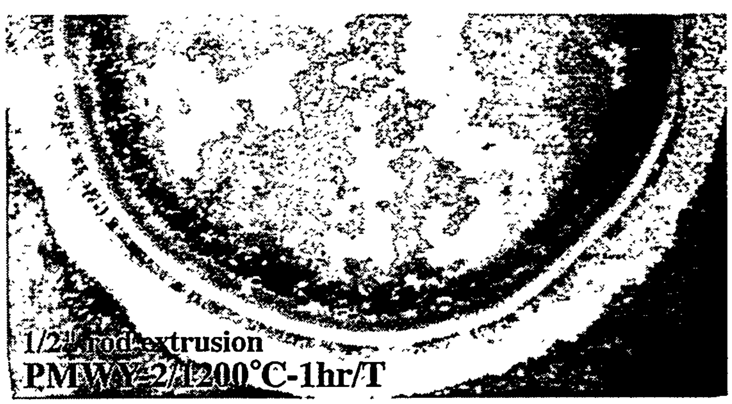


Figure 10. Optical micrographs of transverse section of the 1/2" extruded rod of PMWY-2 material, heat-treated at 1200°C-1hr.

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.

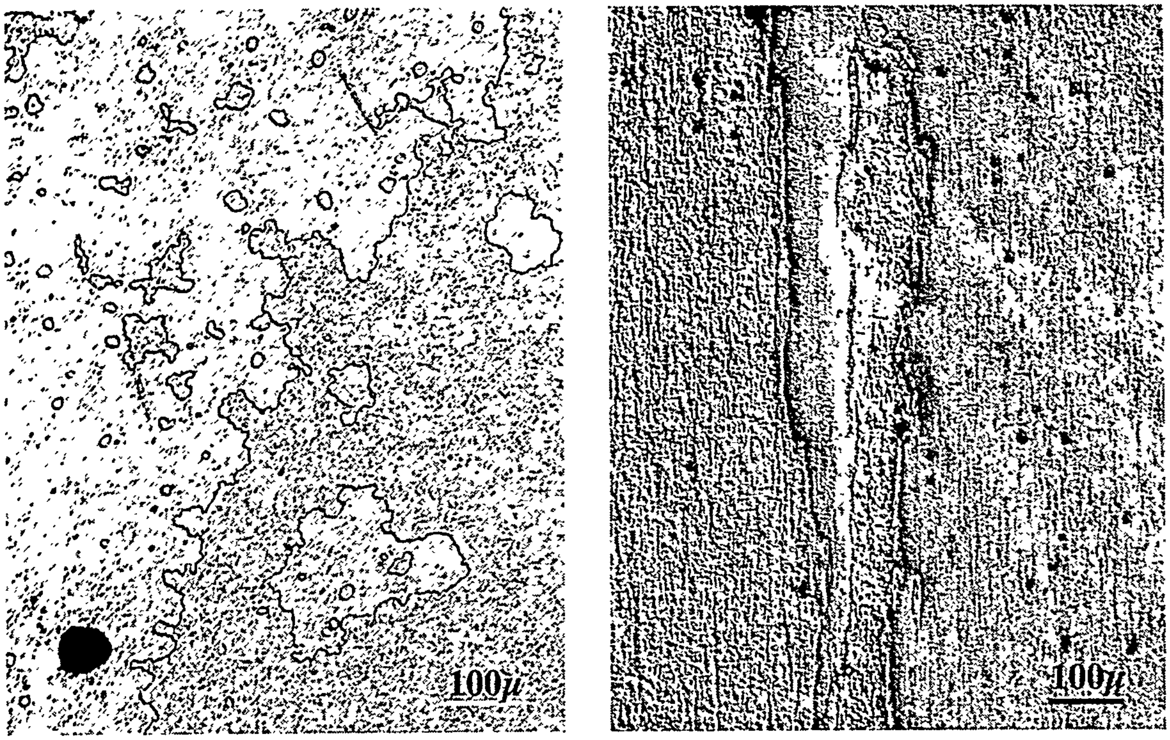


Figure 11. Optical micrograph of the interpenetrating recrystallized grain structure in a) transverse, and b) longitudinal sections of the extruded PMWY-2 rods. (Heat-Treatments conducted in the 1100°-1200°C range)

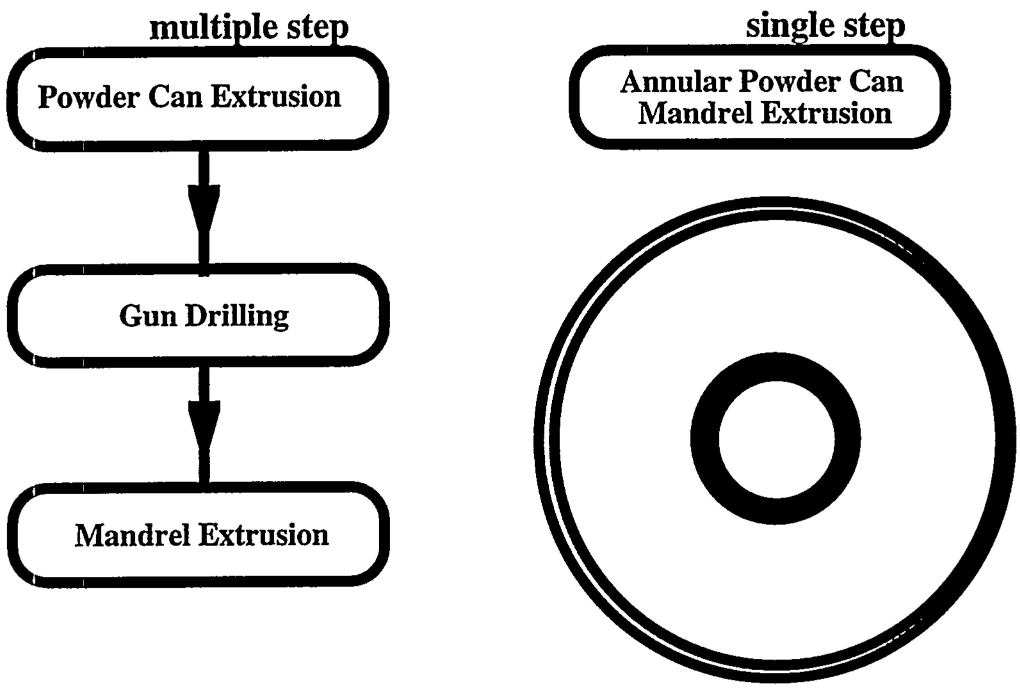
Progress: Iteration 3

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-Fe_3Al tubes were produced *via* the single step extrusion consolidation process.

Comparsion of Tube Extrusion Methodologies



Advantages:	**Advantages:**
Clean inner surface	**energy efficient manufacturing**
Better dimensional tolerance	**improved product yield**
Disadvantages:	**Disadvantages**
multiple heating/extrusion steps	**annular can design complexities**
poor product yield	**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

Extrusion		Die Size	Mandrel Size	Area	Area
Material	Temperature	inch	inch	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

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.

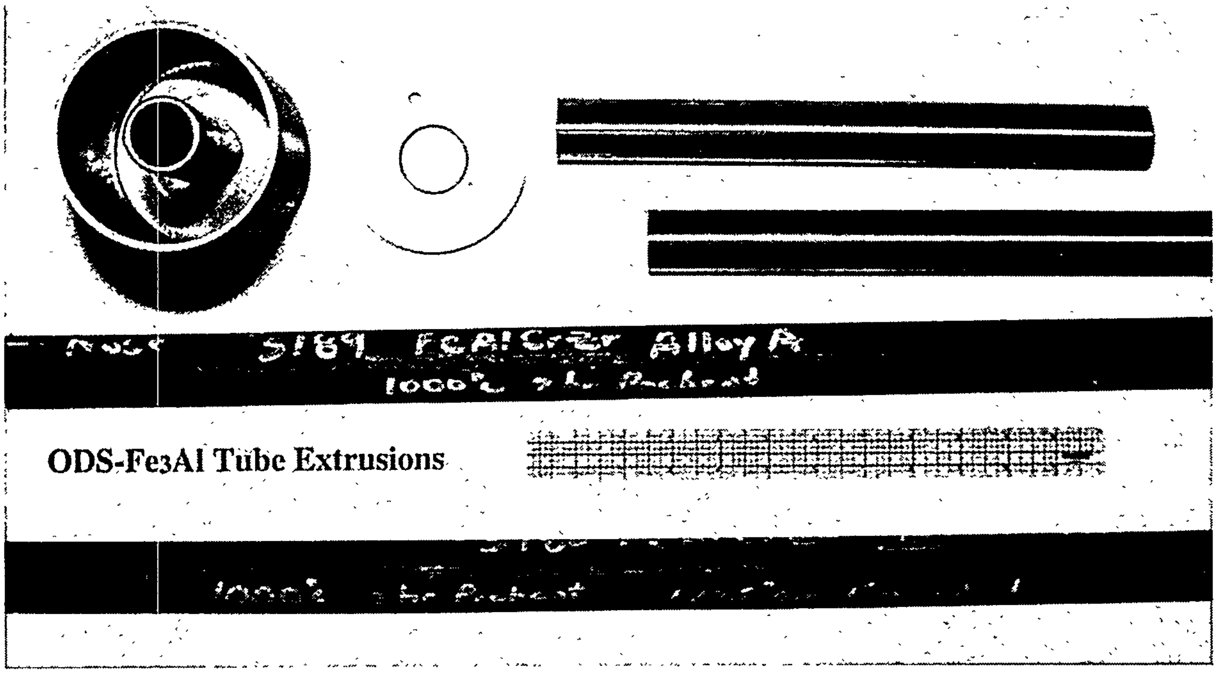


Figure 13. Assorted ODS-Fe_3Al tubes in the as-extruded (below), and surface finished (top) condition, as produced from an annular can (top left) consolidation methodology.

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 hardnesses 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.

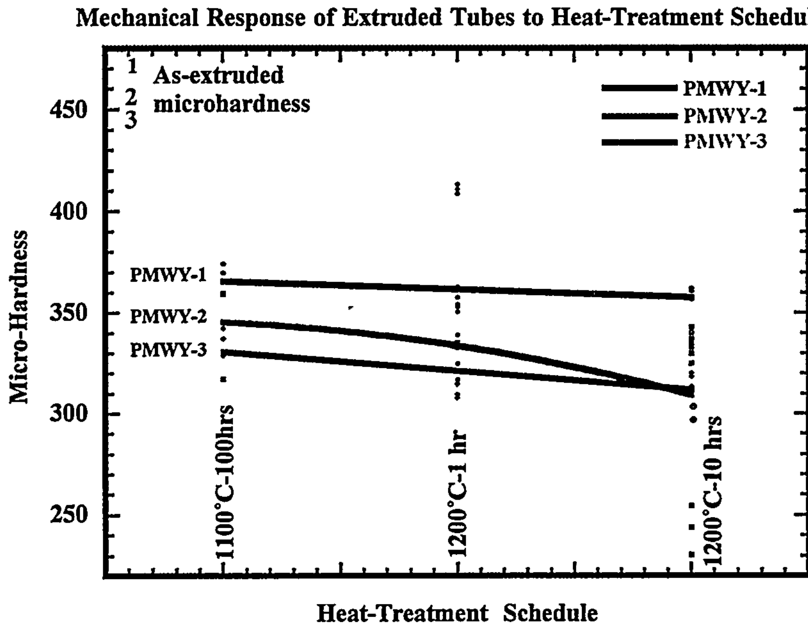


Figure 14. Material microhardness response in the as-extruded, as well as recrystallized state, for the PMWY-1, PMWY-2 and PMWY-3 extruded tubes. Note that the near flat hardness profile indicates a stable recrystallized state for all the powder batches.

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 200μm thick was observed on the exterior surface of the Fe_3Al tube, and was characterized by grain boundaries aligned roughly along the radial direction. In the Fe_3Al 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 circumfurential 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

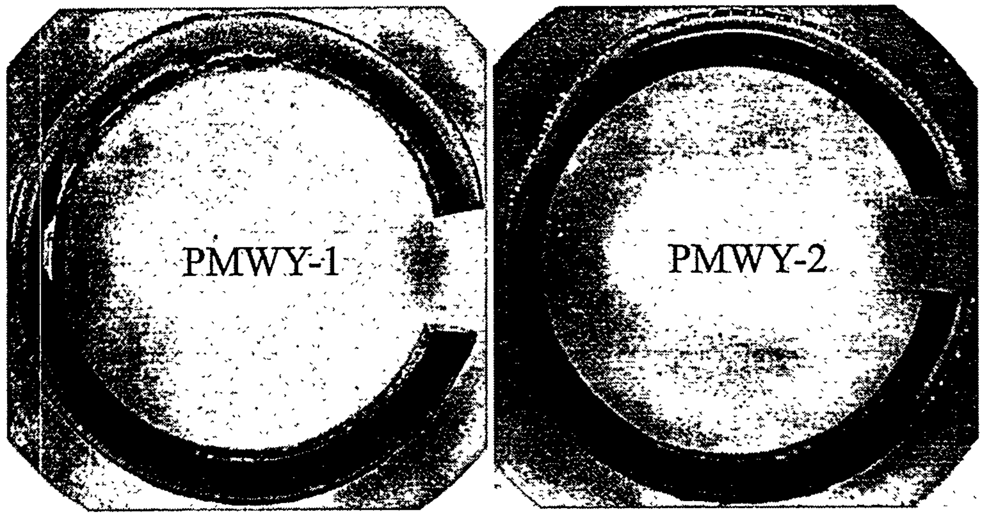


Figure 16. Cross-sectional macro views of the heat-treated a) PMWY-1, and 2) PMWY-2 tubes. Note a total of 1 to 3 circumfurentially aligned grains across the tube wall span.

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 Fe_3Al tube. In the Fe_3Al tube, an average of three to five grains were observed over the wall thickness. Both the exterior and interior extremities of the Fe_3Al 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 Fe_3Al 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.

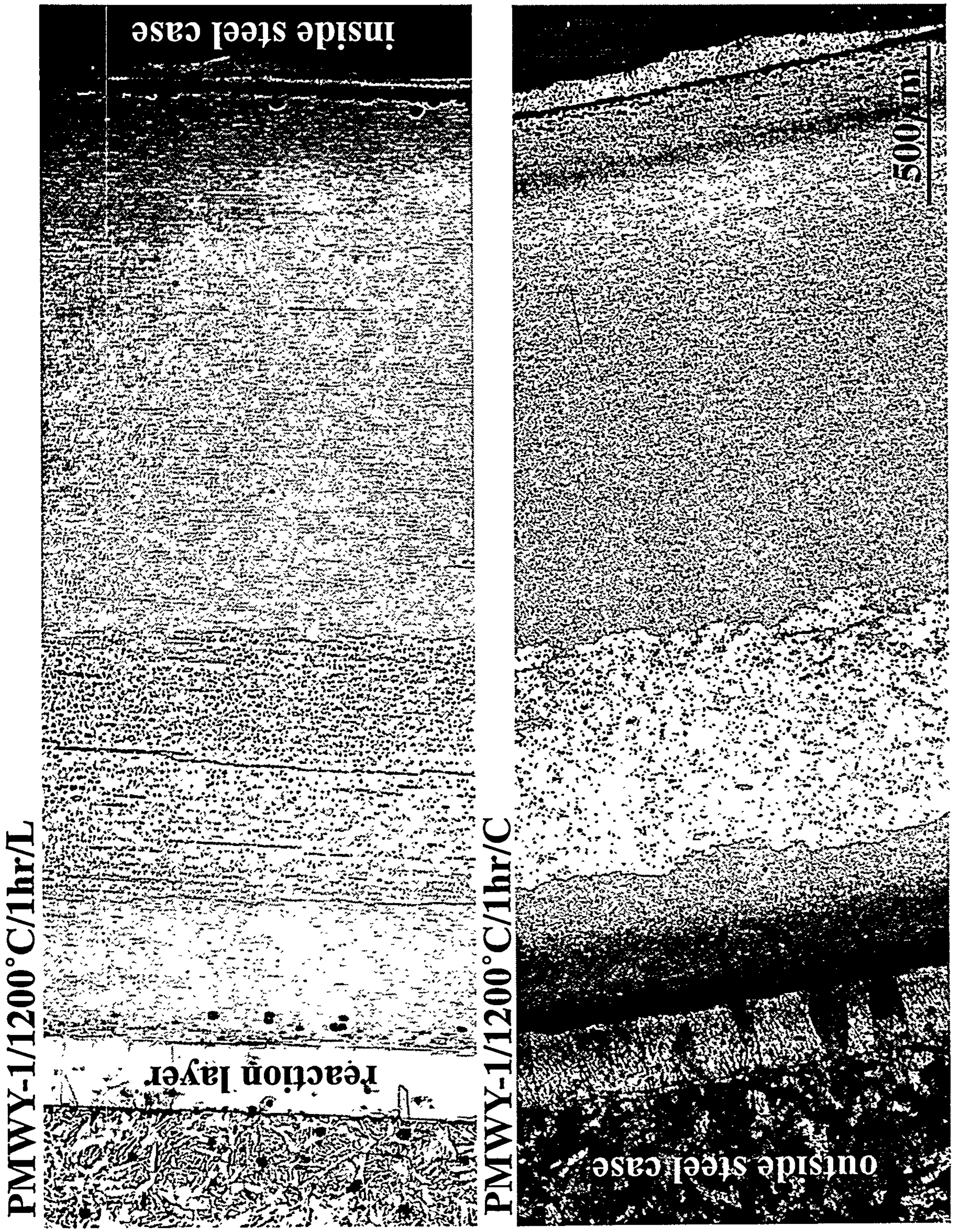


Figure 16. Longitudinal and transverse views of the PMWY-1 tube heat-treated at 1200°/1hr.

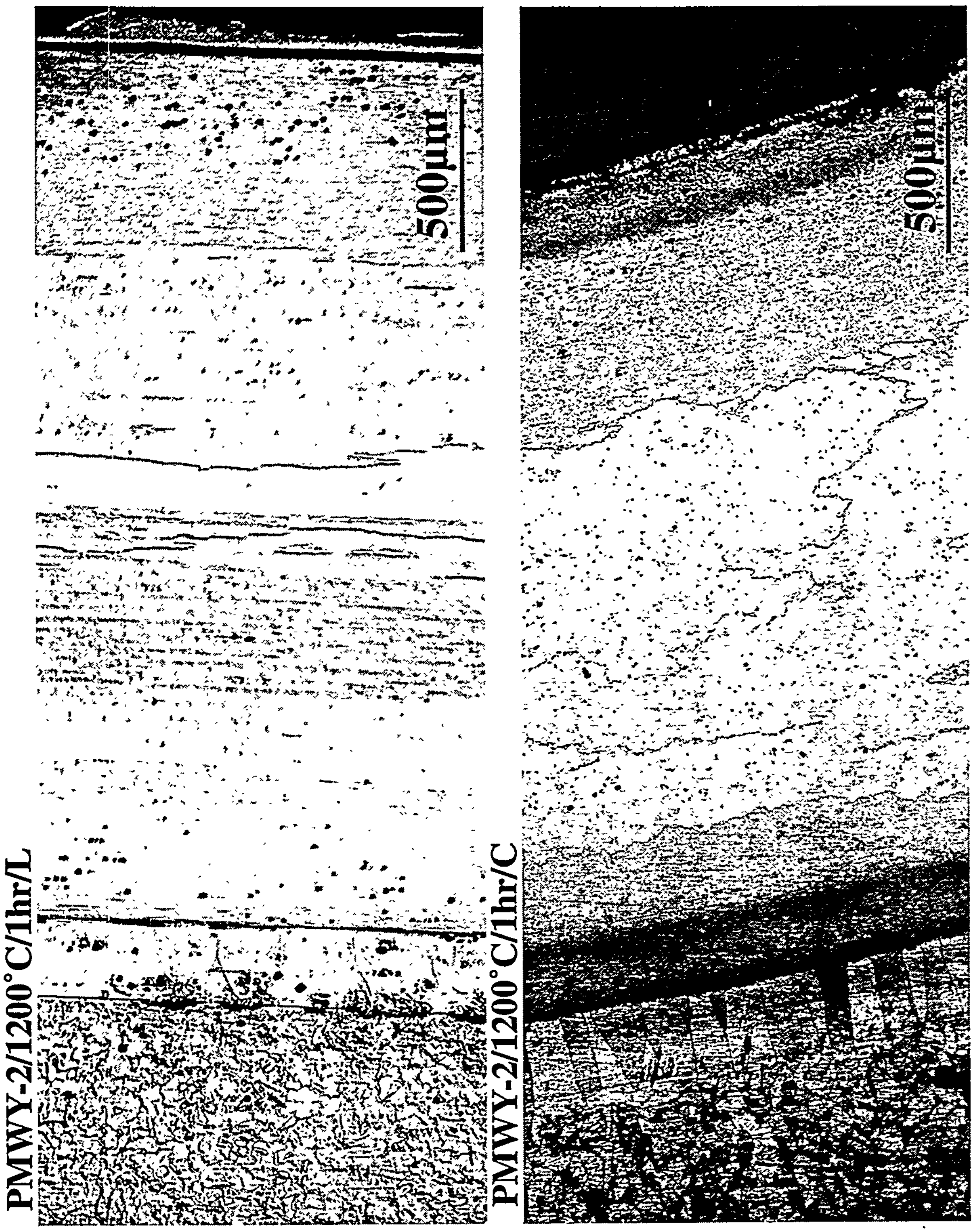


Figure 17. Longitudinal and transverse views of the PMWY-2 tube heat-treated at 1200°/1hr.

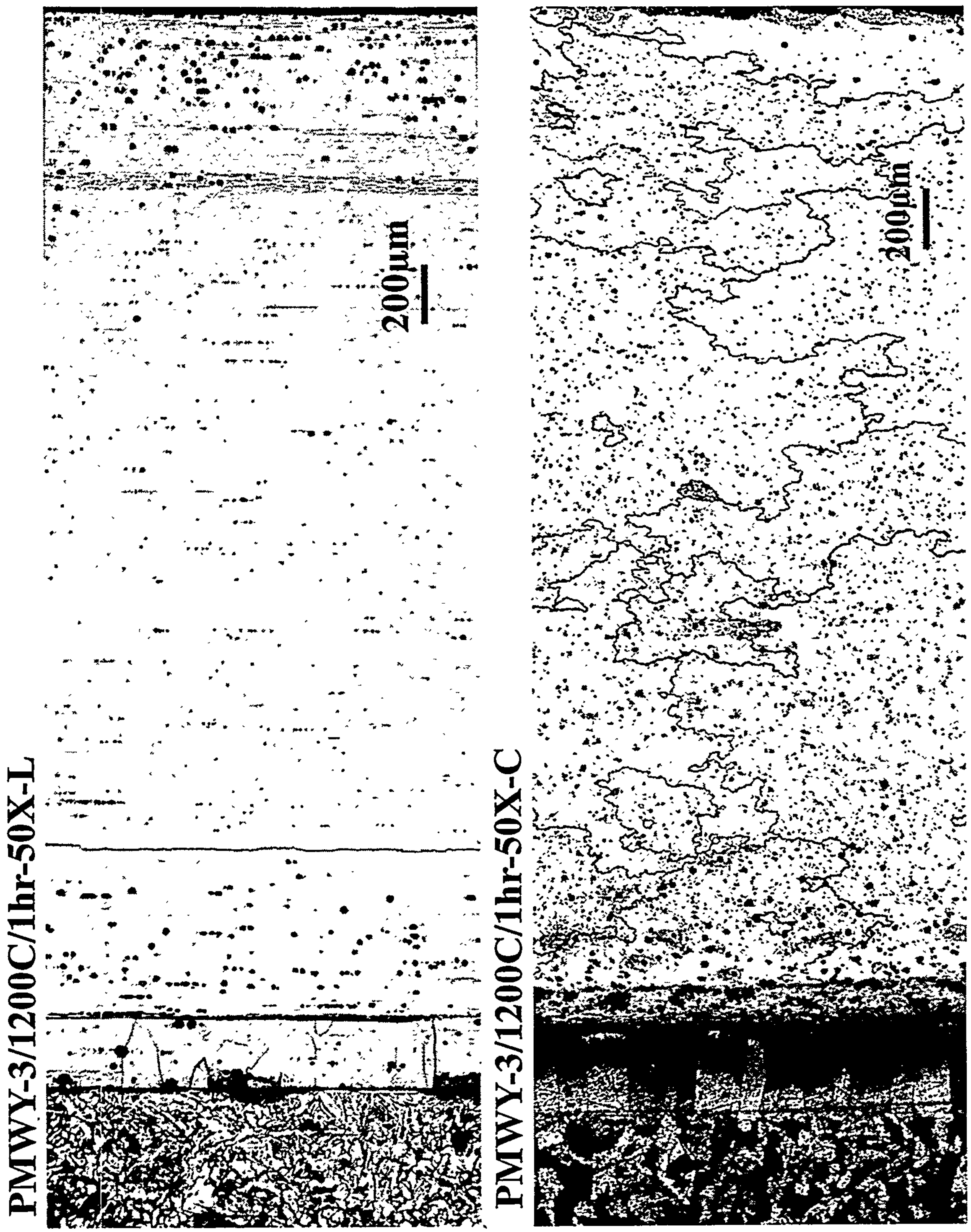


Figure 18. Longitudinal and transverse views of the PMWY-3 tube heat-treated at 1200°/1hr.

Summary and Conclusions

The oxide dispersion strengthened (ODS) Fe_3Al-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_3Al tube, for our 'in-service performance evaluation' to be undertaken in Task 4 of this research program. The creep performance ODS-Fe3Al 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_3Al 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

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