Dispersion Strengthened FeCrAlY Alloys By Flake Powder Processing

by

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ABSTRACT

A novel, high yield, powder metallurgy technique involving high energy (attritor) milling was utilized to produce oxide dispersion strengthened FeCrAlY alloys without the concurrent formation of chromia or chromia-containing spinels. The technique utilizes attritor milling of atomized powder/organic surfactant slurries, in air, to produce flake-like shaped particles with large specific surface areas. It is the oxidation of these powder surfaces under ambient conditions that ultimately generates the dispersoid.

Consolidation of powder compacts was accomplished by hot extrusion at either 982°C or 1093°C. The dispersoid present in as-extruded bar was identified as 3Y2O3·5Al2O3 (yttrium-aluminum garnet) with an average particle size of about 300Å. As-extruded 982°C tensile and stress rupture properties were significantly improved when compared to the same composition without dispersoid.

Further improvement in stress-rupture strength was obtained by producing coarse grain structures with high aspect ratio. Two of the alloys produced responded to secondary grain growth in the as-extruded condition by both isothermal annealing and directional recrystallization.
INTRODUCTION

The increasing demand for high temperature materials with superior strength and surface stability has focused attention on oxide dispersion strengthened (O.D.S.) superalloys. The vast majority of O.D.S. alloys are produced by powder metallurgy techniques. Since conventional superalloys contain elements Ti, Al and Cr that readily form oxides which are difficult to reduce once formed, the possibility exists for excessive oxygen contamination by uncontrolled oxidation of the powder charge during processing. Prior investigations\(^{(1,2,3)}\) have utilized selective reduction as a means of controlling the oxygen content of selected O.D.S. alloys. However, in the case of superalloy compositions, reduction of unwanted oxides is not possible and therefore must be avoided. "Mechanical alloying"\(^{(4)}\) represents the first successful means of incorporating a uniform oxide dispersion into superalloy-type compositions by powder metallurgical techniques. Allen\(^{(5)}\) has demonstrated that controlled surface oxidation of prealloyed, inert gas atomized FeCrAlY powders is capable of producing O.D.S. alloys with a respectable combination of high temperature strength and oxidation resistance. Another technique, flake powder processing by attritor milling has been successfully applied to O.D.S. copper\(^{(6)}\) and nickel\(^{(7)}\) alloys which contained only one strong oxide forming element. The technique involves a surface oxidation effect similar to the SAP (sintered aluminum powder) method. However, instead of utilizing the oxide formed from the base metal as a dispersoid (Al\(_2\)O\(_3\) for SAP) the starting powder composition is preselected to yield, after processing, a stable oxide dispersoid.

Higher dispersoid content and improved distribution, compared to surface oxidation of as-atomized spherical powders, are possible if the powder particles possess a high surface area to volume ratio and a minimum dimension which is of the order of the desired interparticle spacing (0.1-0.01\(\mu\)m for O.D.S. alloys). These requirements are met by a flake-shaped particle which is easily produced by wet ball milling or attritor milling.

In the case of superalloys, the question of which surface oxides will form as a result of the milling process is difficult to answer. Clearly, the most desirable result would be to generate only the most stable oxides since the need for reduction of other oxides is thus eliminated. The iron-chromium-aluminum-yttrium (FeCrAlY) system was selected for this investigation with the expectation that its ability to form strongly adherent alumina scale would be of advantage from a processing standpoint. The objectives of the program were to generate an alumina dispersion in FeCrAl matrix by wet attritor milling without concurrently producing Fe or Cr oxides and then to assess the effect of the dispersion on high temperature properties; no attempt was made to optimize final alloy properties.

EXPERIMENTAL PROCEDURE

Argon atomized Fe-25Cr-4Al-1Y prealloyed powder of -60 mesh (-250\(\mu\)m size was obtained for this investigation (see Table I). A 450g trial batch of Table I

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Particle Size (Microns)</th>
<th>W/% Cr</th>
<th>W/% Al</th>
<th>W/% Y</th>
<th>W/% O</th>
<th>W/% C</th>
<th>V/% Dispersoid (1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-Rec'd</td>
<td>-250</td>
<td>25.0</td>
<td>4.1</td>
<td>1.5</td>
<td>0.03</td>
<td>-</td>
<td>--</td>
</tr>
<tr>
<td>A</td>
<td>-250</td>
<td>25.1</td>
<td>4.2</td>
<td>1.3</td>
<td>0.02</td>
<td>0.03</td>
<td>--</td>
</tr>
<tr>
<td>B</td>
<td>-150, +63</td>
<td>24.7</td>
<td>3.8</td>
<td>0.8</td>
<td>0.98</td>
<td>0.04</td>
<td>5.0</td>
</tr>
<tr>
<td>C</td>
<td>-150, +63</td>
<td>24.3</td>
<td>4.0</td>
<td>1.0</td>
<td>0.55</td>
<td>0.04</td>
<td>2.9</td>
</tr>
<tr>
<td>D</td>
<td>-63</td>
<td>24.2</td>
<td>1.4</td>
<td>0.4</td>
<td>2.25</td>
<td>0.10</td>
<td>10.6</td>
</tr>
</tbody>
</table>

(1) Assumed to be 100\% 3Y\(_2\)O\(_3\) 5Al\(_2\)O\(_3\) YAG (Yttrium-Aluminum-Garnet).
powder was attritor (Segvari Type 1-8) milled for 72 hours. At approximately 3-4 hour intervals, the attritor was stopped, the powder separated from the grinding balls, the attritor restarted and the powder added. This was done to insure that the entire powder charge was subjected to the attritor milling action. After milling, the powder charge was sized, hot outgassed in vacuum, and vacuum fusion oxygen analysis as well as specific surface area measurements (continuous flow method) made. Based on this data, powder for the actual alloys was prepared from approximately 450g charges of -60 mesh powder ground for 6-8 hours and sieved for size. Oversized flakes were recharged while these which were undersized were utilized for an alloy prepared from the milling fines.

The milled powders were loaded into mild steel extrusion containers and cold pressed to 60-70% density. After pressing, a top cover with evacuation tube was welded into place and the entire assembly leak tested with a mass spectrometer leak detector. The billets were vacuum outgassed at 538°C and sealed from the vacuum system under a dynamic vacuum of about 5µm Hg. A schematic flow diagram for the powder processing is given in Figure 1. Prior to extrusion, all billets were soaked at 1093°C/2 hours. Table II details the processing conditions along with pertinent extrusion data. All extrusions were checked for soundness by x-ray.

Standard metallographic techniques were used to evaluate the microstructure of extruded and extruded + heat treated specimens. A 10% oxalic acid solution was more effective for etching fine grain material while Kalling's etchant proved to be the most effective for determining coarser grain structures. The dispersoid crystal structure and composition were determined by x-ray diffraction (CoKα). Particle size, distribution and morphology were qualitatively determined by transmission electron microscopy. The vacuum fusion technique was used for oxygen analysis of as-extruded alloys. Selected specimens were examined directly by transmission electron microscopy. Thin foils were prepared by twin jet electropolishing using a 10% perchloric acid - 90% glacial acetic acid electrolyte.

Isothermal annealing and directional recrystallization were utilized for grain coarsening studies. Mechanical testing consisted of 982°C tensile testing of as-extruded alloys as well as 982°C and 1093°C stress-rupture testing of all alloys with various post extrusion treatments.

RESULTS AND DISCUSSION

Processing

Attritor milling has a marked effect on the spherical as-atomized powder. Figure 2 demonstrates that the powder shape has been altered to flake by the action of the attritor mill. Concurrent with this shape change, the oxygen content of the milled powder was observed to increase with decreasing particle size (see Figure 3). Oxygen content was also observed to increase with increasing specific surface area (Figure 4). Thus, the change in oxygen content is clearly related to a surface oxidation effect and not to bulk or Table II

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Extrusion Temp. ºC</th>
<th>Ram Speed in/min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>984</td>
<td>17.8</td>
</tr>
<tr>
<td>B</td>
<td>984</td>
<td>17.8</td>
</tr>
<tr>
<td>C</td>
<td>1093</td>
<td>17.8</td>
</tr>
<tr>
<td>D</td>
<td>990</td>
<td>17.8</td>
</tr>
</tbody>
</table>

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complete oxidation of the powder charge. The data presented in Figures 3 and 4 allow the "calibration" of the alloy system and permit selection of dispersoid volume fraction by control of oxygen content. The milled powders, including the -230 mesh (-63μm) fines, were not pyrophoric and exhibited no additional oxygen pick-up (despite air storage) in the interim (~2 weeks) between milling and pressing into billets. The milling action produced particle thicknesses which were transparent to a 100 KV electron beam, thus this dimension was of the order of 0.1-0.3μm. An Al₂O₃ volume fraction of 0.03-0.04 was selected for this study and corresponds to about 0.65-0.85 w/o oxygen in the processed powder. Reference to Figure 3 indicates that this oxygen level corresponds to a -150 μm to +63μm size fraction. Powder from this size fraction was used to produce alloys B and C (see below).

The attritor milling technique utilized for this study yielded approximately 75% useful powder in the size range of primary interest (alloys B and C). Based on the data, it appears to be a straightforward step to modify the milling process to a continuous one by introduction of a recirculation system and milling the powder charge for preselected times to achieve the desired oxygen levels. Yields from this type of processing could logically be expected to be greater than 95%.

An important result of this study is the realization that a prealloyed powder such as FeCrAlY (2541) can be attritor processed as a slurry in air without the occurrence of significant Cr₂O₃ contamination. Samples of milled powders were tested for the presence of oxide(s) after various treatments which are described in Table III. The oxide was obtained by dissolving the treated powders in hot dilute HCl and centrifuging the residue. This residue was then identified by x-ray diffraction. As Table III indicates, no oxides were detected on as-attrited powder, however αAl₂O₃ and yttrium-aluminum-garnet (YAG) were detected after a 1093°C/3 hour treatment in a vycor encapsulation backfilled with 1/3 atmosphere of argon. The data tend to indicate the nature of the surface oxidation reaction. It is quite conceivable that the reaction takes place in two or more steps: first an adherent, amorphous alumina gel forms during milling and then transforms upon heating to crystalline alumina(10), further heating then results in the formation of YAG.

The alloys produced in this study are identified in Table I. Alloy A was produced from as-atomized, unmilled powder and was used as a "reference" alloy. Alloys B and C, nominally 4 w/o Al₂O₃, were made from a split batch of attrited powder. The only processing variable which differentiates these alloys is extrusion temperature (982°C versus 1093°C). Alloy D was produced from the -63μm milling fines. X-ray inspection confirmed the overall soundness of the extruded bars.

As-Extruded State

Metallographic inspection of the four as-extruded alloys revealed differences between them. For example, alloy A (as-atomized powder) possessed a fine (about 10μm) relatively equiaxed grain structure. The alloy

Table III

<table>
<thead>
<tr>
<th>Condition</th>
<th>Extracted Phases Present After Various Stages of Processing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Attrited + Vacuum Dry</td>
<td>None</td>
</tr>
<tr>
<td>Attrited + 550°C/2 hrs/Ar + 1093°C/3 hrs/Ar</td>
<td>αAl₂O₃ + trace 3Y₂O₃·5Al₂O₃ &amp; Cr₂3C₆</td>
</tr>
<tr>
<td>As-Extruded (Alloys B, C and D)</td>
<td>3Y₂O₃·5Al₂O₃ + trace αAl₂O₃ &amp; Cr₂3C₆</td>
</tr>
</tbody>
</table>

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contained an even distribution of the iron-yttrium intermetallics \((Y_2Fe_7)\) and \(YFe_5\). Alignment of the intermetallics parallel to the extrusion direction was apparent in the longitudinal section. Unprocessed prior atomized powder particles could be discerned by the needle-like distribution of the intermetallic particles.

Alloys B and C differed considerably from alloys A and D, but were similar to each other structurally. The Fe-Y intermetallics were present with what appeared to be a similar distribution to that of alloy A. Individual oxide particles cannot be resolved; however, the fine striated appearance characteristic of an oxide dispersion was evident. Although difficult to verify, it seems that the grain size of alloy B (extruded at 982°C) was somewhat finer than that of alloy C at 1093°C). The as-extruded grain sizes of both alloys (B&c) were about 5-10 microns. Both alloys B and C possessed an occasional "needle-like" area similar to alloy A. These areas are indicative of the incorporation of an unprocessed powder particle.

Alloy D (produced from minus 63µm "fines") displayed the most refined as-extruded structure of the four alloys produced. A considerable size reduction of the Fe-Y intermetallic phases was apparent as well as the absence of the needle-like areas present in the other three alloys. A fine background of oxide particles was also evident.

In all alloys except alloy "A" the dispersoid was identified as \(3Y_2O_3 \cdot 5Al_2O_3\). A small amount of \(Cr_23C_6\) was also present (see Table I). Considerable line broadening was observed in the x-ray diffraction patterns which indicates a fine < 500Å particle size. The change in major oxide phase from \(Al_2O_3\) to YAG after extrusion may be due to an additional reaction during extrusion or to a solid-state reaction similar to that observed by Benjamin et al. (13)

Tension testing was done on an Instron tensile testing machine at a constant cross head speed of 0.02 cm/min. except for alloy A which was tested at 0.002 cm/min. The data are summarized in Table IV. Results of stress-rupture testing of as-extruded alloys are presented in Figures 5 and 6. Alloy B (5 v/o YAG) displayed an eighteen-fold increase in stress for 100 hour rupture life compared to unmilled alloy A. Alloy C (2.9 v/o YAG) was about 11 times stronger than alloy A on the same basis. The difference in stress rupture strength between alloys B and C appears to be related to differences in volume fraction dispersoid after extrusion and extrusion temperature (see Table I).

### Grain Coarsening Behavior and Properties

#### Isothermal Annealing

Secondary recrystallization (14) (or exaggerated grain growth) in O.D.S. materials leads to anisotropic grain size with high aspect ratio \((L/d)\). In most cases the ratio of longitudinal diameter \((L)\) to transverse diameter \((d)\) should generally be greater than 3 to produce substantial improvement in high

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Vol. % Oxide</th>
<th>UTS (MPa)</th>
<th>0.2 Y.S. (MPa)</th>
<th>% El.</th>
<th>% R.A.</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td></td>
<td>17.1</td>
<td>3.9</td>
<td>166.0</td>
<td>95.5</td>
</tr>
<tr>
<td>B</td>
<td>5.0</td>
<td>42.7</td>
<td>36.5</td>
<td>36.4</td>
<td>94.7</td>
</tr>
<tr>
<td>C</td>
<td>2.9</td>
<td>46.2</td>
<td>40.0</td>
<td>41.2</td>
<td>94.7</td>
</tr>
<tr>
<td>D</td>
<td>10.6</td>
<td>62.7</td>
<td>60.0</td>
<td>13.0</td>
<td>10.0</td>
</tr>
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</table>

982°C Tensile Data For As-Extruded Alloys
temperature properties. (15) Under ideal conditions the grain aspect ratio may exceed ten. Annealing in air at 1316°C, near the incipient melting point (\(\sim 1360°C\)) of FeCrAlY (2541), was used to determine secondary recrystallization response. Figure 7 shows photomicrographs of longitudinal sections of alloys B and C after annealing at 1316°C for 1.5 hours.

The key structural change for alloy B was an increase in longitudinal and transverse grain sizes. The longitudinal dimension, although difficult to determine accurately, was in the 150-200\(\mu m\) range while the transverse grain diameter is about 40\(\mu m\) (\(L/d \sim 4.5\)). The fine, background oxide dispersoid was evident, but appeared to be absent in localized regions throughout the bar. A few clusters of much smaller grains are also present throughout the specimen volume. Although frequently associated with regions apparently lacking dispersoid, these fine grain areas were also observed in regions where dispersoid was present.

Alloy C (Figure 7) responded to the isothermal anneal in a manner similar to alloy B. An elongated grain structure was also present after annealing. The transverse grain diameter was about 35 microns while the longitudinal grain dimension was about 200 microns (\(L/d \sim 5\)). Fine, relatively equiaxed grains were observed to be completely surrounded by much larger grains. As in alloy B, the iron-yttrium intermetallic particles have coarsened and generally appear to be located at grain boundaries. Regions apparently lacking dispersoid were also observed for alloy C. Alloys A and D which contained 0 and 14\% dispersoid respectively, did not show a significant change in grain size or shape as a result of the isothermal anneal.

Alloys B and C which had been isothermally annealed in air at 1316°C/2 hours were stress rupture tested at 982°C. The results were plotted in Figures 5 and 6.

If one compares isothermally annealed to as-extruded material it is apparent that a substantial improvement in high temperature stress rupture strength has been achieved as a result of the isothermal annealing treatment and the associated microstructural features which were developed. For example, alloy C as-extruded which had a stress for 100 hour life at 982°C of 15.2 MPa has been increased to 40.7 MPa.

The fact that alloys B and C display grain growth as a result of isothermal annealing is of particular interest and advantage. Allen(5) had considerable success with O.D. FeCrAlY alloys which had been grain coarsened by extensive thermo-mechanical processing followed by directional recrystallization. His investigation did not produce an O.D. alloy which would grain coarsen as a result of isothermal annealing. The only other alloy systems produced to date which respond similarly are of the "mechanical alloy" genus.

Directional Recrystallization

Directional recrystallization is another method for the solid state generation of oriented microstructures and has proven to be effective in furthering the high temperature strength of O.D. alloys. (16) The technique is especially useful for generating a grain structure with high aspect ratio (\(L/d\)) in O.D. alloys which have been previously processed in such a way that they will not respond (exaggerated grain growth) to isothermal annealing. The directional recrystallization apparatus used in this investigation is similar to that used by Allen.(16)

Extruded bar specimens approximately 0.95 cm \(\phi\) by 20 cm long were directionally recrystallized at rates of 5cm \(\text{h}^{-1}\) and 18-20cm \(\text{h}^{-1}\). The maximum temperature in the hot zone was 1343°C for all runs. Figure 8 shows macro-etched specimens of alloys B and C which had been directionally recrystallized at 18cm \(\text{h}^{-1}\). As can be seen, significant grain growth has
The overall grain structure is one of a bundle of thin, pencil-like grains of varying lengths, many growing the entire length of the bar. The grain aspect ratio is estimated to be in the range of 15-30 for both alloys and both recrystallization rates. The longitudinal structures of the isothermally annealed material, contained fine, equaxed regions which were found to be relatively free of dispersoid. In most cases, these fine grain regions were surrounded or engulfed by a single, much larger grain. The iron-yttrium inter-metallic phases have coarsened and are associated mostly with grain boundary regions. Due to the difficulty of finding a suitable grain boundary etchant, it was nearly impossible to identify and follow individual grains in a metallographic section.

Dispersoid distribution was qualitatively determined by transmission electron microscopy in alloys B and C which had been directionally recrystallized at 1343°C/18cm hrt. Figure 9 shows a typical transverse structure of both alloys. Oxide formation was excellent. No evidence of agglomerated oxides was observed. The observed "in situ" particle size was found to range from about 100Å - 2000Å with the majority of the particles in the 200-300Å size range. A larger (>1μm) blocky phase was also observed in the thin foils and was randomly distributed throughout the matrix. This phase is thought to be Y-Fe intermetallics. A unique feature of the dispersoid distribution was the occurrence of "stringers" or lines of dispersoid (as near circled area in Figure 9). In many cases, the lines were observed as nearly parallel pairs with a spacing of about 0.1-0.3μm. The overall matrix dislocation structure appeared to be typical of a recrystallized ferritic alloy. Localized fine grained regions were also observed in the foils.

Alloys B and C processed by directional recrystallization displayed a further increase in stress-rupture strength (Figures 5 and 6). In fact, the strongest material produced in this study had been directionally recrystallized. Directional recrystallization resulted in grain structures with very high aspect ratios (15-30). Furthermore, the occurrence of fine equaxed grain regions which were observed in the isothermally annealed alloys was substantially decreased. The improvement in stress-rupture strength as a result of directional recrystallization is most probably due to the grain size increase and elimination of a major fraction of the fine, equaxed grains. Although a substantial improvement in stress-rupture properties compared to as-extruded material was observed, the improvement was only about 15 percent greater than that obtained by isothermal annealing. Similar results have been obtained for mechanically alloyed nickel base alloys. (17)

SUMMARY AND CONCLUSIONS

It has been demonstrated that a powder metallurgy technique involving attritor milling can be utilized to generate an inert oxide in an alloy matrix containing significant amounts of chromium and aluminum without the formation of chromia-containing spinels. Oxide formation apparently occurs on the surface of each individual powder particle and is only surface related. The milling procedures employed resulted in a small fraction of unmilled material included in the consolidated bar and ultimately to fine grain regions after recrystallization.

Two of the alloys produced in this investigation represent the only other O.D. materials which respond to isothermal and gradient annealing by secondary recrystallization in the as-extruded condition. Only materials processed by mechanical alloying respond in a similar manner.

The alloy structures are characterized by a fine (less than 300Å) uniform dispersoid of discrete 3Y2O3·5Al2O3 (yttrium-aluminum garnet). Iron-yttrium intermetallic phases as well as small amounts of Cr23C6 were present in the
alloys studied. The YAG dispersoid possesses excellent high temperature stability (i.e. resistance to particle coarsening) and is clearly suitable as a dispersoid.

Specifically, the following conclusions can be made as a result of this study:

1. Flake powder processing is capable of generating uniform, inert oxide dispersions in FeCrAlY (2541) without chromia contamination.
2. Oxide formation takes place as a result of surface reaction(s).
3. FeCrAlY alloys produced by this technique are capable of secondary and directional recrystallization in the as-extruded condition.
4. The dispersoid present in the consolidated alloys is 3Y$_2$O$_3$·5Al$_2$O$_3$ (yttrium-aluminum garnet).
5. Alloys with improved stress-rupture strength have been produced utilizing this technique.
6. Large grain size and high aspect ratio have a major beneficial effect on the high temperature strength of O.D. FeCrAlY alloys.

ACKNOWLEDGEMENTS

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REFERENCES

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AS RECEIVED POWDER (60 MESH) → ATTRITOR MILL → DECANT AND AIR DRY → SIZE → CAN AND OUTGAS → CONSOLIDATE

RECYCLE CHARGE

OVER UNDER FINES

FIGURE 1. SCHEMATIC FLOW DIAGRAM FOR POWDER PROCESSING

FIGURE 2 EFFECT OF ATTRITOR PROCESSING ON PARTICLE SHAPE

AS ATOMIZED FeCrAlY (2541) POWDER 100x

ATTRITED FeCrAlY 6-8 HRS 50x

FIGURE 3 OXYGEN CONTENT VERSUS PARTICLE SIZE FOR ATTRITED POWDERS

FIGURE 4 OXYGEN CONTENT VERSUS SPECIFIC SURFACE AREA FOR ATTRITED POWDERS
**Figure 5. Stress Rupture Plot for Alloy B**

- **ALLOY A**
  - 982°C
  - X AS-EXTRUDED
  - + ANNEALED 1366°C/15HR
  - △ D.R. 5cm/HR
  - □ D.R. 16cm/HR
  - ○ UNPROCESSED
  - 1093°C
  - ◇ D.R. 18 cm/HR

- **ALLOY B**
  - 982°C
  - X AS-EXTRUDED
  - + ANNEALED 1366°C/15HR
  - △ D.R. 5cm/HR
  - □ D.R. 16cm/HR
  - ○ UNPROCESSED
  - 1093°C
  - ◇ D.R. 18 cm/HR

**Figure 6. Stress Rupture Plot for Alloy C**

- **ALLOY A**
  - 982°C
  - X AS-EXTRUDED
  - + ANNEALED 1366°C/15HR
  - △ D.R. 5cm/HR
  - □ D.R. 16cm/HR
  - ○ UNPROCESSED
  - 1093°C
  - ◇ D.R. 18 cm/HR

- **ALLOY C**
  - 982°C
  - X AS-EXTRUDED
  - + ANNEALED 1366°C/15HR
  - △ D.R. 5cm/HR
  - □ D.R. 16cm/HR
  - ○ UNPROCESSED
  - 1093°C
  - ◇ D.R. 18 cm/HR

**Figure 7. Structure After Isothermal Annealing at 2400°F/1.5 HRS (Long Section) Etchant: 10% Oxalic**

- **ALLOY B**
  - 0.1 mm

- **ALLOY C**
  - 0.1 mm

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FIGURE 8. ALLOYS B AND C DIRECTIONALLY RECRYSTALLIZED AT 2450°F/7 HR
ETCHANT: STRONG FeCl₃

FIGURE 9. TRANSMISSION ELECTRONMICROGRAPH OF ALLOY B DIRECTIONALLY RECRYSTALLIZED AT 1343°C/18 cm HR⁻¹ (TRANSVERSE SECTION)
50,000x