

## UNIVERSITY OF DENVER

COLORADO SEMINARY

Denver Research Institute

University Park, Denver 10, Colorado

10 November 1959

MASTER

U.S. Atomic Energy Commission  
Chicago Operations Office  
P.O. Box 59  
Lemont, Illinois

Attention: Mr. Steven V. White, Director  
Research Contracts Division

Subject: Contract No. AT(11-1)-742

Gentlemen:

This informal letter report is the seventh of a series of monthly letter reports for the contract year, 15 March 1959 to 15 March 1960, describing the progress made on the research program, "Study of Factors Influencing Ductility of Iron-Aluminum Alloys", Contract No. AT(11-1)-742.

The objective of the program is to determine the effect of variations of aluminum content, heat treatment, and basic slip mechanism upon the room temperature ductility of Fe-Al alloys. Since alloys containing above 10% aluminum are characterized by an order-disorder transformation, heat treatment will provide the opportunity to study the effects of disorder, varying degrees of order, and incipient order upon the plastic flow mechanism. With a fundamental understanding of the deformation and fracture behavior of these alloys, it should then be possible to devise means to effect significant improvements in their room temperature ductilities by a combination of heat treatment and minor alloying additions.

It is estimated that approximately 60 percent of the proposed research has been completed over the first six and one-half months of the contract period.

Tensile Data

Additional heat treatment studies on 13.9 Alfenol tends to confirm earlier observations that reproducibility of ductility values of 8-9% are difficult to attain. It was conjectured that long holding times at temperatures of 600 to 650C might affect the FeAl domain size, which in turn might profoundly affect the Fe<sub>3</sub>Al ordered structure formed on further cooling. It is

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now apparent that this treatment does not result in improved ductility; if anything, ductility appears to have been impaired (specimens 135 to 142 in Table I). In view of these latest results, the future effort will be concentrated mainly on alloys with higher aluminum contents, and those containing molybdenum and yttrium additions. Preliminary results with heat treated 16-Alfenol and 13.9-Alfenol with molybdenum additions (Thermenol type) indicate that ductilities at least as good as those obtainable with the 13.9% binary alloy are possible. Refer to specimens 143 to 163 in Table I.

Specimen numbers 161 to 169 and 176 to 178 in the table are essentially 13.9 Alfenol containing varying amounts of yttrium, from 0.025% to 0.1%. These were given the standard recrystallization anneal of 725C for two hours, followed by an oil quench. The tensile data are rather disappointing, as it was anticipated that the extremely high heat of formation of yttrium oxide would result in substantially a complete deoxidation and, therefore, a more ductile condition. However, one alloy containing 0.1% yttrium (specimens 176 to 178) did exhibit a somewhat better ductility, suggesting that future work should include alloys with larger yttrium additions.

According to a recent phase diagram of McQueen and Kuczynski\*, alloys containing less than 12.5% aluminum exist either in a disordered or  $\text{Fe}_3\text{Al}$  ordered condition, depending upon the temperature. An alloy containing 12.25% aluminum was, therefore, included in this month's study to determine the effect of a fully disordered structure upon ductility. In comparing specimens 170 to 175 in Table I, it is quite apparent that it matters little as far as ductility is concerned, whether the alloy is in a fully ordered or disordered state. This evidence plus the impossibility of quenching in disordered structures in alloys of higher aluminum content, without the danger of microcrack formation, implies that some degree of order may be required to obtain maximum ductilities in these alloy systems.

#### Preparation of Alloys

During this report period the following series of 150 gram buttons were prepared by arc melting in a non-consumable electrode arc furnace.

16 Al, 0.025 Y, bal. Fe  
 16 Al, 0.05 Y, bal. Fe  
 16 Al, 0.075 Y, bal. Fe  
 15.8 Al, 3.2 Mo, 0.025 Y, bal. Fe  
 15.8 Al, 3.2 Mo, 0.05 Y, bal. Fe

\* "Order-Disorder Transformations in Iron-Aluminum Alloys"--Final Report, H.J. McQueen and G.C. Kuczynski, Office of Naval Research Contract NONR 1623(03), Project NR 031-529-May 1958.

These buttons will be reduced to 35 mil sheet in accordance with the hot and warm rolling schedules described in the progress report for September.

The addition of more than 0.025% Y to both the binary and ternary alloys results in the formation of a second phase in the grain boundaries in the as-cast buttons. Subsequent hot and warm working tends to remove these grain boundary precipitates, transforming them into stringers. This structural condition is described more fully in the following section on metallography.

### Metallographic Studies

A metallographic examination was conducted on the DRI-prepared alloys described in the preceding reports. Specimens, representative of each alloy composition, were selected from the warm-rolled 0.035 inch thick sheet. Before polishing, they were subjected to a preliminary anneal at 725°C. for two hours so that the effect of grain-growth inhibitors, if any, could be subsequently ascertained.

The sheet specimens were mounted in lucite so that the rolling plane sections could be examined. Conventional polishing procedures were followed during the initial stages of preparation. The specimens were finished on a Syntron vibratory polishing machine with alumina abrasive. Etching was performed by brief immersion in the following solution: 25 ml. glycerin, 5 ml. HF and 0.5 ml.  $\text{HNO}_3$ .

Photomicrographs of the structures are presented in Figs. 2 and 3. Figure 2 illustrates the effect of small additions of molybdenum on 13.9-Alfenol. A slight refinement of grain size upon the addition of 1 percent molybdenum is observed, but there is no increased effect with higher additions. The molybdenum appears to be completely in solid solution at all levels of concentration.

The effects of yttrium additions are illustrated in Figure 3. The grain size appears to be noticeably refined at a concentration of yttrium as low as 0.025 percent. At 0.050 percent a second phase is distributed throughout the structure in the form of small spheroids, frequently located at the grain boundaries. At the 0.075 percent level, the second phase appears as stringers in addition to the isolated spheroids. The stringers are composed of angular fragments, indicating that this second phase is not ductile at the temperature of warm rolling (575°C). The segregation of yttrium probably occurred during initial solidification of the melts.

### Resistivity Measurements

A resistivity versus temperature curve for the 13.9% aluminum alloy containing 1% molybdenum was determined during this report period and is presented in Figure 1, together with three other resistivity curves for comparative purposes. Although the resistivity values of this alloy are too high in comparison with the alloy containing 3% molybdenum, the shape of the curve is similar to that of 16-Alfenol (No. 2), as one would predict if the molybdenum behaves similarly to aluminum. The abnormally high resistivities of this particular alloy may be attributed to inferior spot welds, or perhaps to excessive oxidation of the resistivity specimens.

Curves 1 through 4 in Figure 1 offer considerable evidence in support of the idea that molybdenum and aluminum are similar in their behavior and are, therefore, additive in their effect upon the order-disorder transformations. One important difference is perhaps the lower mobility of the heavier molybdenum atom, as evidenced by the much broader resistivity peaks of the alloys with greater molybdenum contents.

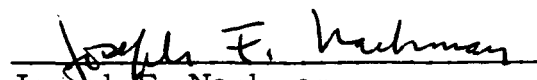
### Future Considerations

During the next report period heat treatment studies will be continued on the 16-Alfenol and 13.9 Al-3 Mo compositions. In addition, it is planned to investigate further the effects of yttrium and perhaps other rare earth additions upon the room temperature ductility of both binary and ternary iron-aluminum base alloys.

Respectfully submitted,



Frank C. Perkins  
Research Metallurgist



Joseph F. Nachman  
Project Supervisor

TABLE I

| Specimen No. | Composition                | Heat Treatment                           | % Elonga-<br>tion (1") | Ultimate<br>Strength<br>lb./in. <sup>2</sup> | Point of<br>Fracture |
|--------------|----------------------------|--|------------------------|--|----------------------|
| 135          | NOL Mat'l.<br>13.9 Alfenol | 2 hr. @ 725°C, air cooled. 24 hr. @      | 2.0                    | 101,000                                      | Outside gage         |
| 136          |                            | 650°C, cooled 30°/hr. to 500°C,          | 3.0                    | 116,000                                      | Center               |
| 137          |                            | oil quenched.                            | 4.0                    | 116,000                                      | Outside gage         |
| 138          | NOL Mat'l.<br>13.9 Alfenol | 2 hr. @ 625°C, air cooled. 18 hr. @      | 4.0                    | 115,000                                      | Outside gage         |
| 139          |                            | 650°C, oil quenched.                     | 5.0                    | 115,000                                      | Center               |
| 140          |                            |  | 5.5                    | 115,000                                      | Inside gage          |
| 141          | NOL Mat'l.<br>13.9 Alfenol | 2 hr @ 725°C, air cooled. 18 hr @ 650°C, | 3.0                    | 111,000                                      | Center               |
| 142          |                            | cooled 30°/hr to 475°C, oil quenched.    | 5.0                    | 111,000                                      | Center               |
| 143          |                            |  | 7.0                    | 98,000                                       | Outside gage         |
| 144          | NOL Mat'l.<br>16 Alfenol   | 2 hr. @ 725°C, oil quenched.             | 5.5                    | 92,000                                       | Outside gage         |
| 145          |                            |  | 5.0                    | 92,000                                       | Inside gage          |
| 146          |                            |  | 5.5                    | 92,000                                       | Inside gage          |
| 147          | NOL Mat'l.<br>16 Alfenol   | 2 hr. @ 725°C, air cooled. 24 hr. @      | 5.5                    | 102,000                                      | Outside gage         |
| 148          |                            | 650°C, cooled 30°/hr. to 500°C, oil      | 5.5                    | 97,000                                       | Outside gage         |
| 149          |                            | quenched.                                | 5.5                    | 97,000                                       | Outside gage         |
| 150          | NOL Mat'l.<br>16 Alfenol   | 2 hr. @ 725°C, air cooled. 18 hr. @      | 7.0                    | 91,000                                       | Inside gage          |
| 151          |                            | 600°C, cooled 30°/hr to 550°C, oil       | 7.5                    | 99,000                                       | Inside gage          |
| 115          |                            | quenched.                                | 6.0                    | 91,000                                       | Outside gage         |
| 116          | NOL Mat'l.<br>16 Alfenol   | 2 hr. @ 725°C, air cooled, 12 hr. @      | 6.0                    | 95,000                                       | Outside gage         |
| 117          |                            | 600°C. Oil quench.                       | 7.5                    | 107,000                                      | Outside gage         |
| 120          |                            | Same as 115                              | 4.0                    | 78,000                                       | Inside gage          |
| 121          | NOL Mat'l.<br>16 Alfenol   | 2 hr. @ 725°C, air cooled. 12 hr. @      | 4.5                    | 99,000                                       | Outside gage         |
| 122          |                            | 600°C. Water quench.                     | 4.0                    | 86,000                                       | Outside gage         |
|              |                            |  | 6.0                    | 84,000                                       | Outside gage         |

TABLE I (cont.)

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| Specimen No. | Composition | Heat Treatment                      | % Elongation (1") | Ultimate Strength lb./in. <sup>2</sup> | Point of Fracture |
|--------------|-------------|-------------------------------------|-------------------|--|-------------------|
|              | DRI Mat'l.  |                                     |                   |  |                   |
| 152          | 13.8% Al    | 2 hr. @ 725°C, air cooled. 24 hr. @ | 5.0               | 99,000                                 | Center            |
| 153          | 3.0% Mo     | 650°C, cooled 30°/hr to 500°C, oil  | 3.0               | 95,000                                 | Outside gage      |
| 154          | balance Fe  | quenched.                           | 5.0               | 100,000                                | Inside gage       |
|              | DRI Mat'l.  |                                     |                   |  |                   |
| 155          | 13.8% Al    | 2 hr. @ 725°C, oil quenched.        | 6.5               | 105,000                                | Outside gage      |
| 156          | 3.0% Mo     |                                     | 6.5               | 101,000                                | Outside gage      |
| 157          | balance Fe  |                                     | 7.0               | 108,000                                | Outside gage      |
|              | DRI Mat'l.  |                                     |                   |  |                   |
| 158          | 13.8% Al    | 2 hr. @ 725°C, air cooled. 12 hr. @ | 4.0               | 86,000                                 | Outside gage      |
| 159          | 3.0% Mo     | 600°C, oil quenched.                | 4.0               | 98,000                                 | Outside gage      |
| 160          | balance Fe  |                                     | 3.0               | 81,000                                 | Outside gage      |
|              | DRI Mat'l.  |                                     |                   |  |                   |
| 161          | 13.9% Al    | 2 hr. @ 725°C, oil quenched.        | 3.5               | 107,000                                | Inside gage       |
| 162          | 0.025% Y    |                                     | 3.5               | 108,000                                | Center            |
| 163          | balance Fe  |                                     | 3.5               | 115,000                                | Center            |
|              | DRI Mat'l.  |                                     |                   |  |                   |
| 164          | 13.9% Al    | 2 hr. @ 725°C, oil quenched.        | 2.0               | 104,000                                | Inside gage       |
| 165          | 0.05 % Y    |                                     | 3.0               | 101,000                                | Outside gage      |
| 166          | balance Fe  |                                     | 4.0               | 104,000                                | Center            |
|              | DRI Mat'l.  |                                     |                   |  |                   |
| 167          | 13.9% Al    | 2 hr. @ 725°C, oil quenched         | 3.0               | 112,000                                | Center            |
| 168          | 0.075% Y    |                                     | 2.5               | 119,000                                | Outside gage      |
| 169          | balance Fe  |                                     | 2.5               | 109,000                                | Center            |
|              | DRI Mat'l.  |                                     |                   |  |                   |
| 176          | 13.9% Al    | 2 hr. @ 725°C, oil quenched         | 5.5               | 108,000                                | Center            |
| 177          | 0.10% Y     |                                     | 5.0               | 107,000                                | Inside gage       |
| 178          | balance Fe  |                                     | 3.0               | 107,000                                | Inside gage       |

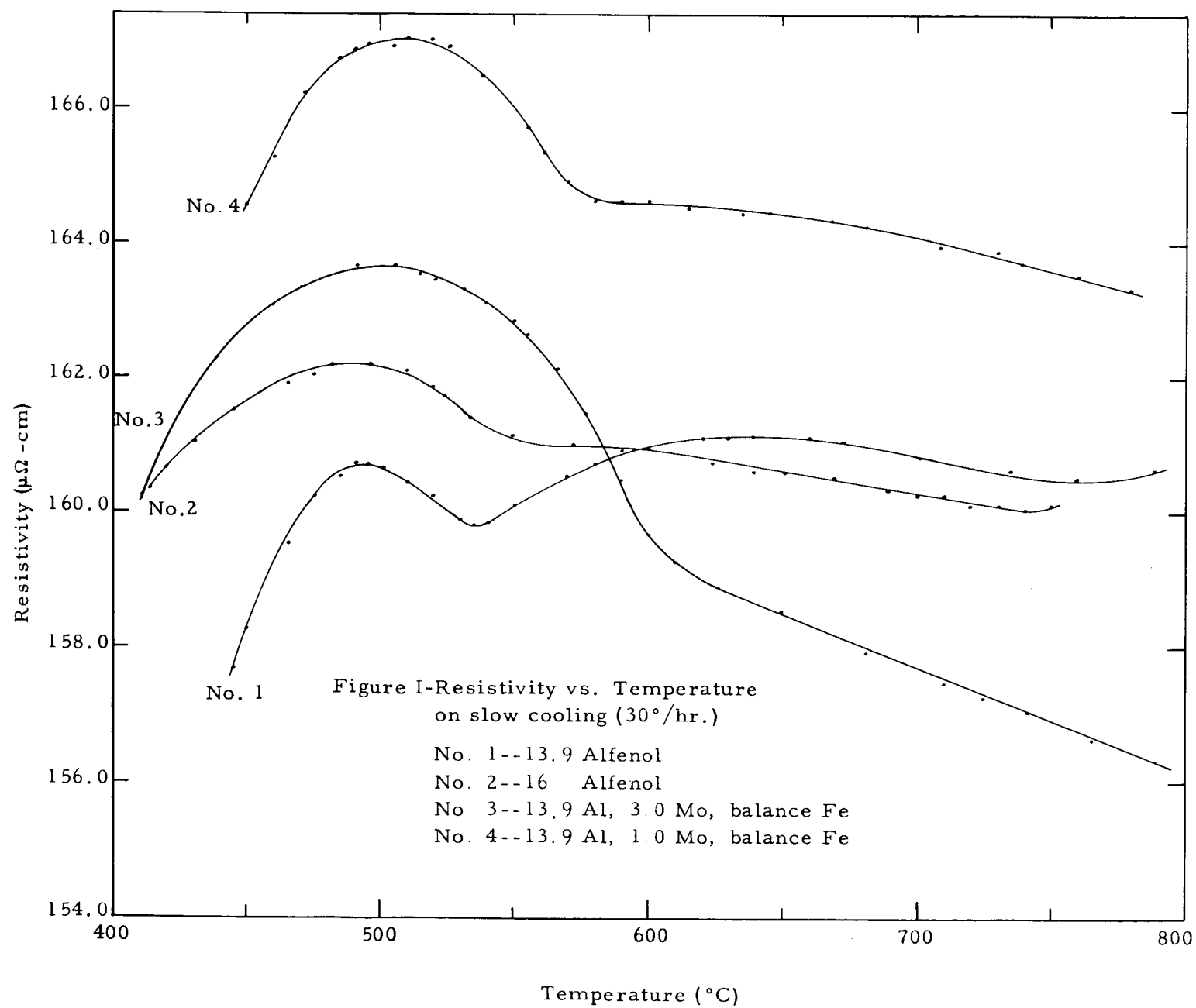
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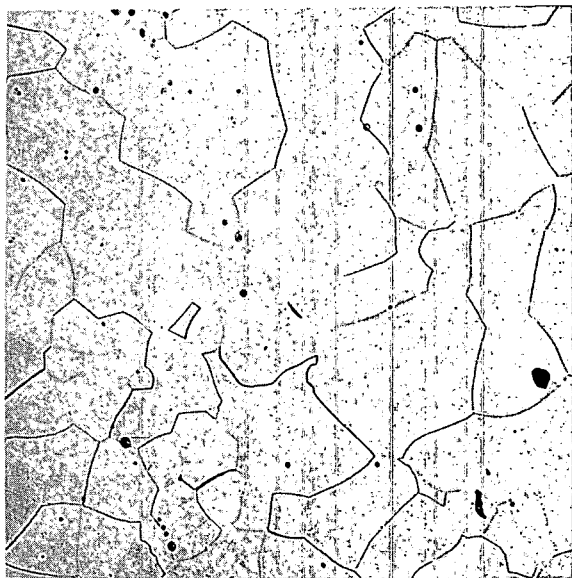


TABLE I (cont.)

| Specimen<br>No. | Composition   | Heat Treatment                             | % Elonga-<br>tion (1") | Ultimate<br>Strength<br>lb./in. <sup>2</sup> | Point of<br>Fracture |
|-----------------|---------------|--|------------------------|--|----------------------|
| 170             | DRI Mat'l.    |  | 3.0                    | 89,000                                       | Outside gage         |
| 171             | 12.25 Alfenol | 2 hr. @ 725°C, oil quenched.               | 3.0                    | 88,000                                       | Inside gage          |
| 172             |               |  | 2.5                    | 92,000                                       | Center               |
| 173             | DRI Mat'l.    |  | 4.5                    | 95,000                                       | Center               |
| 174             | 12.25 Alfenol | 2 hr. @ 725°C, cooled 60°/hr to            | 3.5                    | 97,000                                       | Center               |
| 175             |               | 300°C, held 24 hr at 300°C, air<br>cooled. | 2.5                    | 96,000                                       | Outside gage         |

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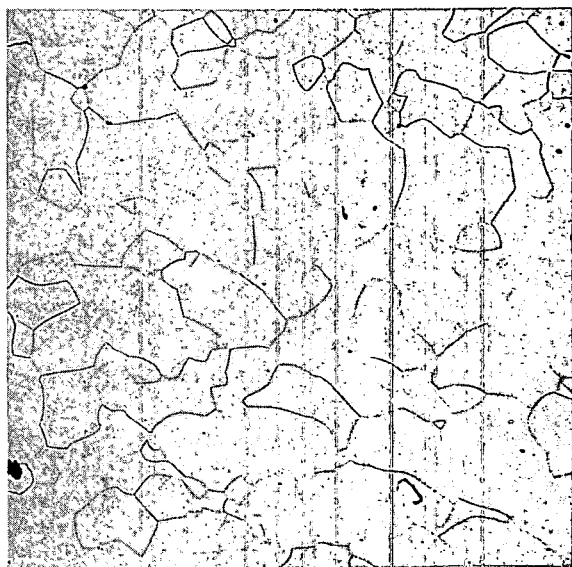




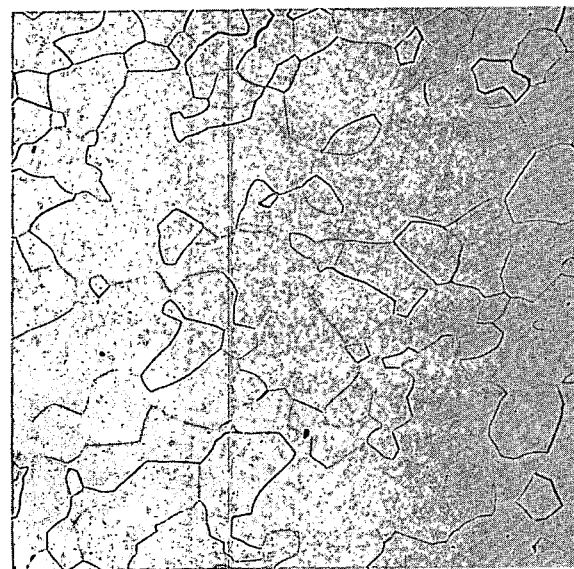
(a)



(b)



(c)

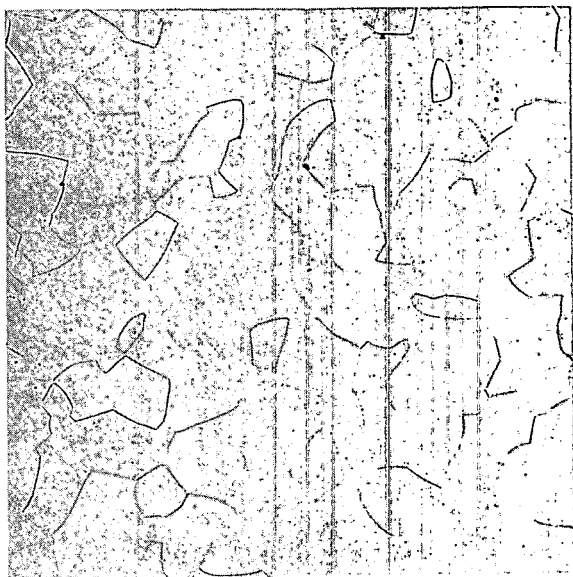


(d)

Figure 2. Microstructures of 13.8-Alfenol containing:

- (a) No addition
- (b) 1% Molybdenum
- (c) 2% Molybdenum
- (d) 3% Molybdenum

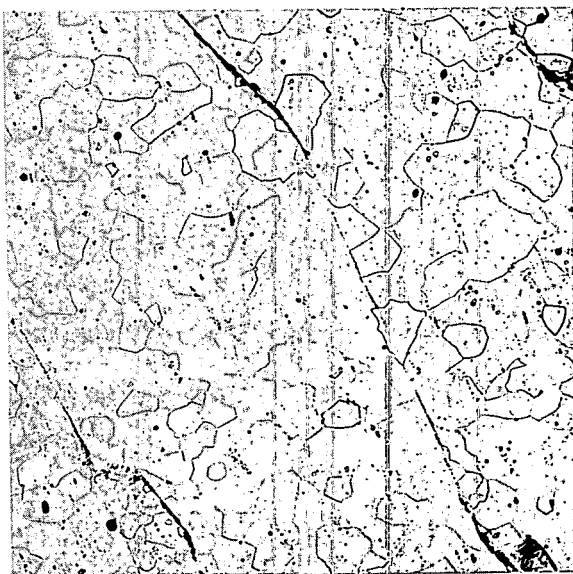
Etchant - 25 ml. Glycerin, 5 ml. HF, 0.5 ml. HNO<sub>3</sub> 100x



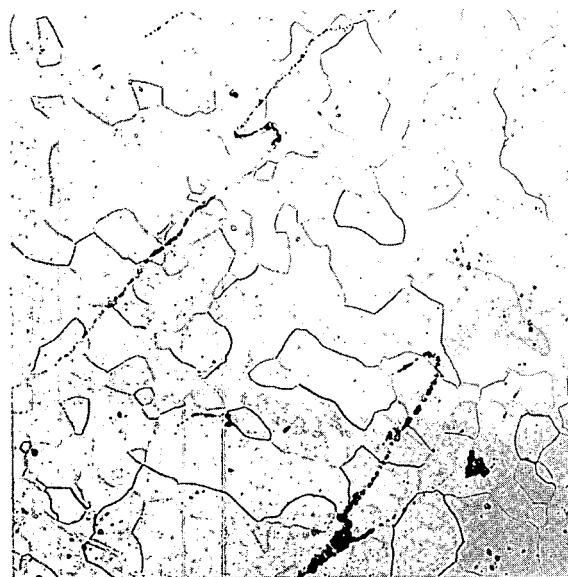
(a)



(b)



(c)



(d)

Figure 3. Microstructures of 13.8-Alfenol containing:

- (a) 0.025 yttrium
- (b) 0.050 yttrium
- (c) 0.075 yttrium
- (d) 0.100 yttrium

Etchant - 25 ml. Glycerin, 5 ml. HF, 0.5 ml. HNO<sub>3</sub> 100x