AECU-4400

UNIVERSITY OF DENVER
COLORADO SEMINARY

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University Park, Denver 10, Colorado

9 October 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 sixth 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 55 percent of the proposed research has been completed over the first six and one-half months of the contract period.

Tensile Data

Tensile tests were conducted on 13.9-Alfenol specimens in a number of heat treated conditions during September. The heat treatments and results of the testing are tabulated in Table I. In general, the

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Barry Steele
Authorizing Official
Date: 2-20-07

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treatments are duplications or minor variations on heat treatments which have produced favorable or promising degrees of ductility in past experiments. The range of elongation values is similar to that observed previously. The majority of specimens failed after 6 to 7 percent elongation. Some heat treatments were notably inferior producing consistently lower values, while other exceptional treatments unexpectedly resulted in elongations of 8 to 9 percent. Attempts to repeat these latter experiments have not been successful, suggesting that the structures responsible for increased ductility are extremely sensitive to minor divergence from the optimum heat treatments or else that the so-called optimum heat treatments were borderline in character.

The heat treatments reported presently are all initiated by a 2 hour anneal at 725C followed by air cooling. This treatment has been found to be satisfactory for recrystallizing the warm-rolled starting structure without inducing objectionable coalescence of grains at temperature or promoting micro-crack formation during cooling. Most of the specimens were then reannealed at 600C for a period of 12 hours to produce a reproducible equilibrium state of FeAl order in the material. Past and present experience indicates that the ductility can be affected by time and temperature employed for this FeAl ordering treatment, regardless of subsequent cooling rates, holding treatments or quenches. It is conjectured that the ordered FeAl domain size might profoundly affect the character of the Fe_3Al ordered structure produced upon later cooling. The 12 hour treatment at 600C was selected as standard for the present series of specimens in view of the fact that it was previously employed for three sets of specimens which fractured after 8 to 9 percent elongations.

Specimens 84 to 86 and 111 to 114, characteristic of quenching from the two preliminary treatments described above, deformed fairly consistently to 6 and 7 percent elongations. The attempts to improve on this degree of ductility by slow cooling the material into the upper Fe_3Al temperature range (as indicated by electrical resistivity curves reported previously) are represented by specimens 90 through 104. Various quenching media were utilized to "freeze in" with variable efficiencies the incipient states of Fe_3Al order. Rough generalization indicates that higher ductilities are obtained by fast quenches (ice water) from the higher temperature limit of the Fe_3Al transformation (535C). However, the best ductilities obtained in the present specimens were not superior to those characteristic of quenching from 600 or 725C.

Specimens 87 to 89, which have uniformly low ductility, were heated at 550C for 12 hours prior to slow cooling into the Fe_3Al transformation region. Comparison of this result with the relatively high elongations of specimens 105 to 107 suggests that varying the time and temperature

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of the FeAl soaking treatment might be a fruitful direction for future experimentation.

Specimens 123 to 130 were slow cooled to 450C and held for various lengths of time before quenching. This temperature is well inside the Fe_3Al region. The elongations, varying from 6 to 7 percent, were consistent and typical of the best described so far in the present series. Since holding time at 450C did not appear to affect the tensile properties, it is believed that the as-cooled specimen represented equilibrium conditions at this temperature. Ordered Fe_3Al domain growth, if it occurred at 450C, was not manifested by a change in the mechanical properties. This conclusion is further confirmed by specimen 119 which was heated directly to 450C and held for 24 hours. The elongation obtained on this specimen (6.5 percent) agreed with the rest of this 450C series.

Specimens 108 to 110 were given a heat treatment which was a variation on the other 450C heat treatments described in the preceding paragraph. The principal variable was the condition of the FeAl at the initiation of cooling, and the cooling rate was reduced to 7C per hour. The high ductilities can perhaps be explained by the unique combination of a critical FeAl domain size transforming to Fe_3Al at a critical cooling rate. Attempts to reproduce these critical conditions (specimens 131 to 134) have been unsuccessful to date.

Preparation and Fabrication of Alloys

Last month a series of 150 gram buttons, containing 13.9% aluminum and 1, 2, and 3% molybdenum, were prepared by melting in a non-consumable electrode arc furnace. In addition to the above, other alloys have been prepared during this report period, of the following compositions:

13.9% aluminum, balance iron

13.9% aluminum, 0.1 yttrium, balance iron

13.9% aluminum, 0.075 yttrium, balance iron

13.9% aluminum, 0.050 yttrium, balance iron

13.9% aluminum, 0.025 yttrium, balance iron

The selection of yttrium as an additive is based primarily upon the promising results that were achieved by its addition to molybdenum. A substantial hardness decrease along with refinement of cast grain structure was noted, indicating the efficiency of yttrium as a scavenger for the removal of interstitial impurities from molybdenum. Since oxygen, and perhaps other interstials, are known to contribute to the brittle behavior of the iron-aluminum alloys the addition of an effective scavenger could eliminate this source of embrittlement.

The study of the alloys containing the yttrium additions will be deferred until a suitable heat treatment has been devised for the 13.9% aluminum binary alloy.

All of the alloy buttons now on hand have been reduced to 35 mil sheet by rolling, conforming as much as possible to the NOL hot and warm rolling schedules. The rolling was carried out on a Stanat two high-four high combination rolling mill. Rolling schedules were, briefly, as follows:

Hot Rolling

The buttons, averaging about 3/8" in thickness, were soaked for approximately 1/2 to 3/4 of an hour at the rolling temperature of 1050C prior to rolling. An initial pass of 0.025" was given each button, and thereafter 0.010" passes were used with approximately a five minute reheating period between passes, until a thickness of 0.15" was attained. At this point, the temperature was reduced to 950C and rolling continued with 0.010" passes until a final thickness of 0.125" was obtained. The lower finishing temperature is desirable to refine the hot rolled grain size.

Warm Rolling

The 0.125" hot rolled material was warm rolled at 575C, using passes of 0.005", to a final sheet thickness of 0.035".

Tensile and resistivity specimens are now being machines from the above sheet material.

Resistivity Measurements

The resistivity vs. temperature curve for a 13.9% aluminum alloy containing 3% molybdenum was determined and is presented in the attached figure. It is interesting that the shape of this curve is similar to the 16-Alfenol curve presented in the last report. This suggests that the molybdenum and aluminum may have an additive effect upon the order-disorder transformations. The similar behavior of molybdenum and aluminum, under these circumstances, may not be too unreasonable in view of their similar atomic radii. Broadening of the Fe_3Al peaks on the resistivity curves may possibly be attributed to the low mobility of the heavy molybdenum atom.

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A comparison of the Thermenol curve, presented in the August report, and this present curve (13.9 Al, 3 Mo) clearly shows that the Fe_3Al peak on the Thermenol curve is considerably broader. The inflection points in both cases, however, appear to occur in the vicinity of 600C.

It is felt that the effect of molybdenum on the resistivity curves presents some very interesting possibilities for future heat treatment studies.

Future Considerations

It was believed at the beginning of the program that concentration of study on the 13.9-Alfenol, Fe_3Al composition, would manifest the greatest changes in ductility as a function of order-disorder phenomena. The optimum heat treatment conditions would then be applied to the higher aluminum ternary alloys which possess engineering properties of vital interest. It is now apparent that the use of order-disorder phenomena for greatly improving ductility in the 13.9 composition is not a simple or clearcut operation. Consequently, future studies will be concerned with higher alloy contents in addition to the 13.9 composition, in order to expedite progress towards the ultimate goal, viz. ductile Thermenol-type alloys.

During the next report period it is planned to continue heat treatment studies of 13.9-Alfenol, but perhaps on a reduced scale, and to initiate studies on the 16-Alfenol and 13.9 Al-3 Mo compositions. Tensile tests and x-ray investigations will be carried out on the heat treated specimens. If time permits two additional resistivity curves will be determined, one on the DRI 13.9-Alfenol and the other on the 13.9 Al-1 Mo alloy.

Respectfully submitted,

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Project Supervisor

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Attachments

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TABLE I

Tensile Tests on 13.9-Alfenol

Specimen No.	Heat Treatment	% Elongation (1")	Ultimate Strength lb./in.^{2}	Point of Fracture
84	2 hr. at 725°C. Oil quench.	3.0	113,000	Outside gage.
85	Same as 84.	7.0	102,000	Inside gage.
86	Same as 84.	7.0	110,000	Inside gage.
111	2 hr. at 725°C. Air cooled. 12 hr. at 600°C., Water quench.	7.0	118,000	Center
112	Same as 111.	6.0	103,000	Center
113	2 hr. at 725°C. Air cooled. 12 hr. at 600°C., Oil quench.	6.0	123,000	Inside gage.
114	Same as 113.	6.0	111,000	Outside gage.
90	2 hr. at 725°C. Air cooled. 12 hr. at 600°C., Cooled 30°C./hr. to 535°C., Ice water quench	7.5	116,000	Inside gage.
91	Same as 90.	7.0	116,000	Center
92	2 hr. at 725°C. Air cooled. 12 hr. at 600°C., Cooled 30°C./hr. to 520°C., Ice water quench	5.0	112,000	Inside gage.
93	Same as 92.	3.5	98,000	Center
94	Same as 92.	4.0	110,000	Center
95	Same as 92.	6.0	117,000	Center
105	2 hr. at 725°C. Air cooled. 12 hr. at 600°C., Cooled 30°C./hr. to 490°C., Ice water quench	7.0	114,000	Center
106	Same as 105.	7.0	120,000	Center
107	Same as 105.	5.0	119,000	Center

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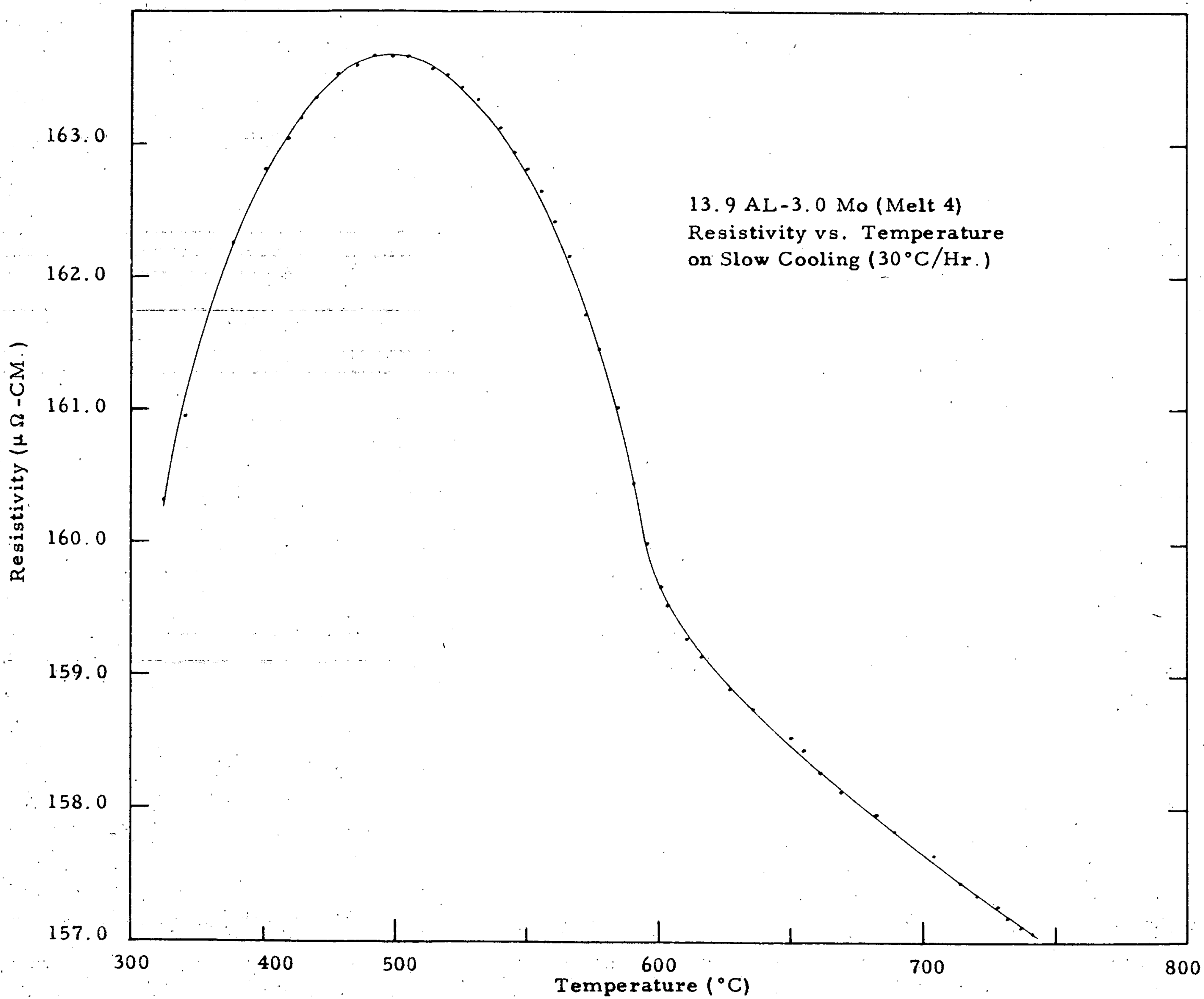
TABLE I (Continued)

Specimen No.	Heat Treatment	% Elongation (1")	Ultimate Strength lb./in.^{2}	Point of Fracture
99	2 hr. at 725°C. Air cooled. 12 hr. at 600°C., Cooled 30°C./hr. to 535°C., Oil quench	6.0	112,000	Outside gage.
100	Same as 99.	6.5	114,000	Center
101	Same as 99.	6.5	112,000	Center
102	2 hr. at 725°C. Air cooled. 12 hr. at 600°C. Cooled 30°C./hr. to 520°C., Oil quench	5.0	116,000	Outside gage.
103	Same as 102.	6.5	116,000	Center
104	Same as 102.	3.5	114,000	Outside gage.
96	2 hr. at 725°C. Air cooled. 12 hr. at 600°C. Cooled 30°C./hr. to 490°C. Air cooled	3.0	104,000	Center
97	Same as 96.	4.0	113,000	Center
98	Same as 96.	4.0	117.000	Center
87	2 hr. at 725°C., Air cooled. 12 hr. at 550°C., Cooled 30°C./hr. to 490°C. Ice Water quench	2.0	103,000	Outside gage.
88	Same as 87.	3.0	126,000	Outside gage.
89	Same as 87.	3.0	108,000	Outside gage.
123	2 hr. at 725°C., Air cooled, 12 hr. at 600°C., Cooled 30°C./hr. to 450°C. Oil quench.	6.0	114,000	Inside gage.
124	Same as 123.	6.0	112,000	Inside gage.
125	Same as 123, but held 30 min. at 450°C.	6.5	114,000	Center
126	Same as 125.	6.0	108,000	Inside gage.

TABLE I (Continued)

Specimen No.	Heat Treatment	% Elongation (1")	Ultimate Strength lb./in.^{2}	Point of Fracture
127	Same as 123, but held 2 hr. at 450°C.	7.0	112,000	Center
128	Same as 127.	7.0	110,000	Center
129	Same as 123, but held 8 hr. at 450°C.	7.0	114,000	Center
130	Same as 129.	6.5	111,000	Inside gage.
119	2 hr. at 725°C., Air cooled, 24 hr. at 450°C., Air cooled.	6.5	--	Center
108	2 hr. at 725°C., Air cooled, Heated to 540°C and slow cooled 7°C./hr. to 450°C. Air cooled.	8.5	118,000	Center
109	Same as 108.	8.5	116,000	Inside gage.
110	Same as 108.	9.0	110,000	Inside gage.
131	Same as 108.	4.0	118,000	Outside gage.
132	Same as 108.	7.0	122,000	Center
133	2 hr. at 725°C., Air cooled. Heated to 540°C. and slow cooled 7°C./hr. to 450°C. Oil quenched.	7.0	119,000	Inside gage.
134	Same as 133.	4.0	111,000	Outside gage.


13.9 AL-3.0 Mo (Melt 4)
Resistivity vs. Temperature
on Slow Cooling (30°C/Hr.)
Resistivity (μΩ-CM.)
163.0
162.0
161.0
160.0
159.0
158.0
157.0
300
400
500
600
700
800
Temperature (°C)

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