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Study of Factors Influencing Ductility
Of Iron-Aluminum Alloys

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ABSTRACT

An investigation of the basic slip mechanism of iron-aluminum alloys containing 8 to 16 percent aluminum is described. The effects of order-disorder heat treatment variables, scavenging additions, processing procedures, and a correlative study of the microscopic modes of plastic flow and fracture as related to the observed tensile properties are discussed. Principal use was made of resistivity measurements and X-ray diffraction techniques for the detection of atomic order in this system of alloys.

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I. SUMMARY

A. An analysis of the basic slip mechanism of several iron-aluminum alloys indicates crystallographic type slip on the $\{110\}$ planes for Thermenol and the binary alloys (Alfenols) containing 10, 13.9, and 16 percent aluminum. The mechanism inhibiting banal glide in the binary alloys with increasing aluminum content appears to be in a transitional state at the 8 percent level, depending on orientation and the relative complexity of the operating stress systems. The superior ductility of the 8 percent alloy in comparison to the higher aluminum levels may be accounted for by the fact that fracture occurs by shear rather than cleavage in this composition range.

B. Surface preparation has been found to be a very important factor, indicating that these alloys are prone to notch sensitivity. The effects of earlier heat treatments were apparently masked by the notch effect. Electropolishing has been effective in minimizing this inherent notch sensitivity and has led to a greater reproducibility in tensile properties.

C. Heat treatment studies based upon resistivity vs. temperature curves indicate that room temperature tensile elongations up to 14% are possible. These latter results were obtained with fully recovered microstructures, indicating that recovered structures may be less notch sensitive than the recrystallized microstructure.

D. The similarity in shape of the resistivity curves of alloys containing molybdenum with those of higher aluminum content suggest that aluminum and molybdenum may have an additive effect. The broadening of the resistivity peaks by the addition of molybdenum may possibly be attributed to the low mobility of the molybdenum atom, which would tend to make the order-disorder transformation sluggish.

E. Outside of grain refinement, the beneficial effects of rare earth scavenging additions upon ductility have not been clearly established.

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II. INTRODUCTION

Interest in iron-aluminum alloys continues to be very active. These cheap non-strategic alloys offer many advantages of good structural strength at intermediate temperatures, excellent high temperature oxidation resistance, favorable strength-to-weight ratios over a wide range of temperatures, good to excellent magnetic properties, and relatively low thermal neutron absorption cross sections with the higher aluminum contents. In view of the above properties, iron-aluminum alloys would appear to be potentially useful in nuclear fuel cladding applications. To take advantage of most of these favorable properties it is desirable that the aluminum content be above 10%. However, at aluminum levels above 10% these alloys experience a rather drastic reduction in their room temperature ductilities.

The research described in this report was undertaken by the Denver Research Institute for the Division of Reactor Development of the Atomic Energy Commission in an attempt to gain an understanding of the effect of order-disorder 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 for effecting significant improvements in their room temperature ductilities by a combination of heat treatment and minor alloying additions.

This interim report describes the progress made on the research program under contract No. AT(11-1)-742 for the period 15 March 1959 through 31 April 1960.

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III. BACKGROUND INFORMATION

A. Iron-Aluminum Alloys

Investigations of the iron-aluminum system were reported as early as 1890 (1). Subsequent work by numerous investigators showed that iron base alloys containing 0-34 wt. percent aluminum consisted of a series of homogeneous solid solutions with many interesting physical and mechanical properties. In 1933, Sykes and Bampfylde (2) made a comprehensive study of the binary system of iron and aluminum up to 17 percent aluminum. Even though these alloys exhibited many interesting properties, commercial development did not follow, mainly because of the inherent difficulty of working iron alloys containing over 10 percent aluminum.

In 1952, interest by the Naval Ordnance Laboratory (NOL) in the magnetic properties of alloys containing 12-17 percent aluminum prompted investigations (3, 4, 5, 6) which resulted in a successful method for producing cold rolled sheet and strip as thin as 0.0003 inch. Further investigations resulted in the development of iron-aluminum base alloys exhibiting good stress-rupture properties at elevated temperatures. The binary compositions were named "Alfenol," while the heat-resisting modifications containing molybdenum and/or other additions were broadly termed "Thermenols."

Although the Alfenols and Thermenols have many interesting and useful properties, the basic problem of relatively low room temperature ductility (3-4 percent elongation) has served as a deterrent to their widespread use. The greater ease of fabricability of alloys containing less than 10 percent aluminum has led a number of investigators to concentrate their research efforts in this direction. Notable examples of these lower aluminum content alloys are Ferral (8-10 Al, 0-2 Ti, Bal. Fe) and DB-2 (7.5 Al, 5 Cr, 4 Nb, 0.75 Zr) developed by the Ford Motor Company (7) and the Martin Company (8) respectively.

Further work by Buehler and Dalrymple (9, 10) of the Naval Ordnance Laboratory has resulted in the development of a modified Thermenol with room temperature elongations of the order of 8 percent. This modification consisted of minor additions of Zr and C for grain refinement purposes, coupled with a suitable heat treatment. These promising results plus the unique observation by Thompson Ramo Wooldridge (11) of one isolated value of 14 percent elongation obtained in a tensile test with Thermenol offer evidence that iron-aluminum

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alloys containing over 10 percent aluminum are capable of considerable improvement in their room temperature ductilities.

B. Slip Mechanisms of Body-Centered Cubic Metals

Since the iron-aluminum alloys are solid solutions of the body-centered cubic structure, it would be appropriate to summarize briefly the plastic flow behavior of some metals possessing this structure. The room temperature plasticity of some of these metals exhibits peculiarities not observed in metals of the other common crystallographic structures.

The determination of the slip elements of body-centered cubic metals can be complex because of the multiplicity of slip systems which appear to operate. For example, glide in ingot iron is usually manifested by wavy slip traces, which cannot be directly correlated with any single crystallographic plane. Several theories have been proposed to account for the forked and bent slip lines in this metal. The mechanism originally accepted assumed that slip occurs in the $\langle 111 \rangle$ direction on planes of the types $\{110\}$, $\{112\}$ and $\{123\}$, which would explain curved slip traces on the basis of alternate glide on several of the many available slip systems. Brick and co-workers (13, 15) concluded that ingot iron slips in the octahedral direction on a plane which is not necessarily crystallographically significant and which is determined by (a) the orientation of the crystal relative to the stress axis, (b) the temperature and (c) the relative resistance to shear of the planes containing the $\langle 111 \rangle$ glide direction. Chen and Maddin (14) on the other hand, have proposed a mechanism of alternate $\{110\}$ slip which, although equivalent to operation of the $\{112\}$ and $\{123\}$ planes, would be favored on the basis of the free energy requirements.

The analysis of the microscopic slip mechanisms of several other body-centered cubic metals has been reasonably straightforward, and tabulations of the slip elements for various materials are available in the literature (16, 17). In general, the reported slip plane is either the $\{110\}$, $\{112\}$ or the $\{123\}$ and the slip direction is always the $\langle 111 \rangle$. In several instances, deformation at an elevated temperature brings a new type of slip plane into operation, although the slip direction remains the same and appears to be characteristic of the structure, being the direction of greatest atomic density.

In general, an increase in the temperature of deformation does not lead to the disappearance of slip elements which are operative at lower temperatures but new elements are permitted to operate at the

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higher temperatures. An example of this behavior is iron-silicon alloys of less than four percent silicon. At subatmospheric temperatures the only operative slip planes are the dedecahedral planes, whereas upon deformation at room temperature, the slip lines appear bent and wavy, indicating the advent of the noncrystallographic (or banal) mode of glide. The apparent "glide plane" in this type of deformation is closely associated with the plane of maximum shear stress containing the slip direction. The fact that glide can occur along a "plane" which is semi-independent of the orientation of the crystal indicates that the slip mechanism has become less inhibited and can more easily accommodate the applied stress.

The addition of an alloying element corresponds to a decrease in the temperature of deformation, in that some slip systems may become inhibited. In iron-silicon alloys of greater than four percent silicon, the only established slip plane is $\{110\}$ up to temperature as high as 500°C (12). It is well known that this composition marks a transition between ductile and brittle behavior in iron-silicon alloys and that atomic order can be detected at as low as five percent silicon.

C. Order-Disorder Phenomenon in Iron-Aluminum Alloys

It is well known that iron-aluminum alloys undergo order-disorder transformations upon slow cooling. X-ray diffraction studies by Bradley and Jay (18) established that the iron-aluminum system is characterized by two ordered structures, Fe_3Al (13.9% Al) and FeAl (33% Al). They observed sharp deviations in lattice parameters from Vegard's Law in alloys of 10 or more percent of aluminum, indicating the existence of atomic order to this lower aluminum composition.

More recently, McQueen and Kuczynski (19) have determined the phase relations between iron and aluminum between 20 and 34 atomic percent aluminum by dilatometric means. In general, the results of this research are in agreement with that of Bradley and Jay, except that the FeAl ordered structure was found to extend down to 23 at/0 instead of 25 at/0 aluminum as reported earlier by Bradley and Jay. Below 23 at/0 the disordered solution transforms directly to the Fe_3Al ordered structure.

Studies by Buehler and Dalrymple and those of Justusson, Zackay, and Morgan show that room temperature ductility is closely associated with the state of atomic order in these alloys containing in excess of 10 wt/0 aluminum and, as such, should be amenable to improvement by heat treatment.

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The research conducted under Contract AT(11-1)-742 included such studies as the determination of the basic slip mechanism of alloys containing 8 to 16 percent aluminum, heat treatment variables, effect of scavenging additions, processing procedures, and a correlative investigation of the microscopic modes of plastic flow and fracture as related to the observed properties. These studies are described in detail in the following sections of this report.

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IV. PREPARATION OF ALLOYS

The melting and fabrication procedure of the NOL Alfenols and Thermenols will only be described briefly, since complete descriptions can be readily obtained from the various Naval Ordnance Laboratory reports (4, 9).

A. Melting of NOL Alloys

The NOL alloys were melted by induction in MgO crucibles. Decarburization and deoxidation were carried out by wet and dry hydrogen treatments. The addition of aluminum was made just prior to pouring, and the melt was cast into a slab mold under a reduced pressure of helium. Casting under reduced pressures promotes the formation of equiaxed grains, which improve the hot workability of these alloys. After the ingot was allowed to solidify, it was stripped out of the mold while still red hot and placed in a furnace held at 1000° - 1050°C. Sufficient time was allowed for the ingot to heat through, after which the ingot could be hot-rolled directly or be allowed to furnace-cool to room temperature. This latter procedure eliminated or minimized the casting stresses that were induced by the chill casting.

B. Melting of DRI Alloys

The charge materials were essentially the same as those used in the preparation of the NOL alloys and consisted of electrolytic iron cathode chips obtained from Plastic Metals, 99.99 percent grade aluminum pig from Alcoa, and molybdenum of 99.9 percent purity from the WAH Chang Corporation. Special additives such as yttrium and lanthanum were obtained from Michigan Chemical Company and Lunex Company, respectively.

Charges of the above materials in 150 gm lots were arc-melted in a water-cooled copper crucible, non-consumable electrode furnace under an argon atmosphere. The buttons were turned several times and remelted to insure homogeneity. As soon as the buttons solidified and while still red hot, they were removed quickly, placed into a furnace at 1050° C, and then allowed to cool slowly with the furnace.

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C. Fabrication of Alloys into Sheet

The rolling procedure, except for minor variations, was essentially that employed by the Naval Ordnance Laboratory to reduce their iron-aluminum ingots to sheet. The buttons, weighing 150 gms and measuring approximately 3/8-inch thick were first allowed to soak in a furnace at 1050° C for 1/2 to 1 hour. Rolling was carried out on a Stanat 2-high/4-high combination rolling mill. Two passes of 0.010-inch each were used between each reheating period of five minutes. This schedule was continued until a thickness of 0.150-inch was attained. The furnace temperature was then reduced to 950° C and the rolling continued at the rate of two passes of 0.010-inch per reheating, until the final thickness of 0.125-inch was reached.

The final warm rolling of the 0.125-inch thick sheet was carried out at a temperature of about 575° C, with 0.005-inch passes between reheatings, to a final thickness of 0.035-inch.

No surface preparation of the buttons was attempted at this time, which may at least partially account for the poorer quality of the DRI sheet as compared to the sheet stock obtained from the Naval Ordnance Laboratory. Another departure from the NOL procedure was the lack of a strip heating furnace, which facilitates more uniform heating and rolling of the strip. This furnace is now being constructed for future work.

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D. Composition of Alloys Used in this Investigation

Table I lists the alloy compositions that were furnished by the Naval Ordnance Laboratory or made by DRI for this program.

TABLE I

<u>Alloy</u>	<u>Nominal Composition</u>	<u>Actual Composition</u>	<u>Source</u>
8 - Alfenol	8 Al - bal. Fe	---	NOL
10 - Alfenol	10 Al - bal. Fe	10.18 Al - bal. Fe	NOL
13.9 - Alfenol	13.9 Al - bal. Fe	13.88 Al - bal. Fe	NOL
13.9 - Alfenol	13.9 Al - bal. Fe	---	DRI
13.9 - Alfenol with 1 Mo	13.9 Al - 1 Mo - bal. Fe	---	DRI
13.9 - Alfenol with Y	13.9 Al - (0.024, 0.05, 0.0075, 0.1, 0.5) Y - bal. Fe	---	DRI
13.9 - Alfenol with La	13.9 Al - (0.1, 0.5) La bal. Fe	---	DRI
Thermenol Type	13.9 Al - (2, 3) Mo-bal. Fe	---	DRI
16 - Alfenol	16 Al - bal. Fe	16.18 Al - bal. Fe	NOL
Plain Thermenol	16 Al - 3.3 Mo-bal. Fe	15.86 Al - 3.29 Mo - bal. Fe	NOL
Modified Thermenol	16 Al - 3.3 Mo - 0.4 Zr - 0.1 C - bal. Fe	15.9 Al - 3.37 Mo - 0.37 Zr - 0.1 C - bal. Fe	NOL

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V. DETERMINATION OF PLASTIC FLOW AND FRACTURE MECHANISMS IN IRON-ALUMINUM ALLOYS

During the course of the past year a program was undertaken to determine the deformation mechanisms of the Alfenols as a function of aluminum content.

Tensile specimens machined from 0.035 inch thick sheet (representative of 8-, 10-, 13.9- and 16- Alfenol) were subjected to critical strain and annealed for an extended period of time at 1200° C to produce coarse grains. Grains varying from 1/8 to 1/2 inch in diameter were produced.

One specimen each of the 8-, 10-, 13.9- and 16- Alfenols was metallographically polished and deformed in tension after quenching in oil from 725° C. Orientations of the individual crystals with respect to the specimen edges were determined by the X-ray diffractometer technique (see section V). Photomicrographs of the individual grains, aligned to correspond to the stereographic projections of the orientations obtained by the X-ray analyses, were then used for identification of the operative planes. (The normal drawn to a slip or twin trace will coincide with the stereographic pole of the plane responsible for the trace.)

Deformation of the specimens was interrupted early in the plastic range in the instance of the more ductile materials (8- and 10- Alfenol) so that the slip traces could be analyzed shortly after their initial development. At the earlier stages of plastic deformation the traces are straighter, the orientation of the crystal can be obtained more accurately, and the analysis is consequently more reliable. In the case of the 13.9- and 16- Alfenols, the initiation of plastic flow was followed very closely by fracture.

In the 10-Alfenol grains analyzed it was found that all of the slip traces were rectilinear and could be identified with {110} planes. The slip traces were bent and wavy near grain boundaries at the initiation of plastic flow, and upon further deformation the traces became wavy throughout all the heavily deformed regions. Fracture occurred by cleavage along a {100} plane, and twins were frequently present in the material adjacent to the fracture. In one 10-Alfenol tensile specimen, slip was confined to a single large grain which extended across the entire section area. A kink formed during the early stages of deformation, and flow was considerable prior to fracture.

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The 13.9- and 16- Alfenols exhibited the same type of crystallographic slip as the 10- Alfenol alloy, except the extent of flow was severely limited by the advent of fracture. Twinning after fracture was observed in the 13.0- Alfenol but it was not found in the 16- Alfenol.

Two grains were analyzed in the 8- Alfenol specimen. One grain exhibited extensive slip of a segmented and wavy character, the traces of which could not be correlated with any crystallographic plane. Continued deformation produced an increase in the density of the slip traces within this grain, and eventually fracture occurred by shear along the glide planes. No twins were observed in the vicinity of the fracture. A second grain in the 8- Alfenol specimen deformed along a set of straight slip traces which were identified with the $\{110\}$ plane. It would appear that the mechanism inhibiting banal glide in the Alfenols with increasing aluminum content is in a transitional state at the 8% aluminum level, being very sensitive to orientation and the relative complexity of the operating stress systems. The fact that fracture occurred by shear rather than by cleavage at this composition also may help account for the high ductility of 8-Alfenol in comparison with higher aluminum Alfenols.

This study was also extended to determine the deformation mechanisms of plain Thermenol (3.34% Mo, 15.8% Al, balance Fe). A coarse-grained sheet specimen quenched from 725° C in oil was metallographically polished and deformed by flexure. Two grains were analyzed, and it was found that all of the slip traces could be accounted for by planes of the type $\{110\}$.

It was also determined that cleavage occurred along a $\{100\}$ plane. No evidence of twinning was observed.

Typical deformation and fracture structures of the Alfenols are reproduced in Figure 1. The results of the study of deformation modes of the Alfenols as a function of aluminum content are summarized in Table II.

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A

B

Figure 1. Deformation and Fracture Structures of Alfenols.

- A. 8-Alfenol
- B. 10-Alfenol

75X

C.

D.

Figure 1. (Continued). Deformation and Fracture Structures of Alfenols.

C. 13.8-Alfenol

D. 16-Alfenol

75X

TABLE II

Summary of Deformation Studies of the Alfenols
and Thermenols

<u>% Al</u>	<u>No. of Grains</u>	<u>No Cor- relation</u>	<u>{110} Slip</u>	<u>{100} Cleavage</u>	<u>112 Twins</u>
8	2	3	1	-	-
10	4	-	8	1	3
13.9	1	-	2	3	1
16	3	-	6	3	-
Thermenol	2	-	6	1	-

The results of the above analyses are in agreement with the theoretical predictions of Flinn (27). According to this investigator, deformation of a superlattice structure at low temperatures, where diffusion and dislocation climb are inhibited by lack of thermal activation, will be restricted to actual crystallographic planes. The residual dislocations in the undeformed material should all be in stable or metastable orientations, since unstable configurations would previously have been eliminated by the actual formation of the superlattice. As dislocation pairs are generated under the applied stress during deformation, they should be confined to the actual slip planes in the absence of climb. Hence, he predicted that the wavy slip lines and choice of slip planes possible in a disordered B2 structure would not be observed in the ordered structure, and that slip would be confined to {110} planes. The slip analyses of the present study indicated the activation of only {110} planes in the composition range where ordering is known to occur.

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VI. ELECTRICAL RESISTIVITY MEASUREMENTS FOR DETECTION OF ATOMIC ORDER

The superlattice transformations which iron-aluminum alloys undergo upon slow cooling were used as the basis for the closely-controlled heat treatment schedules employed in an effort to improve the room temperature ductilities of the Alfenols and Thermenols. A convenient and sensitive method for determining the degree of atomic ordering in an alloy is the measurement of electrical resistivity.

A Kelvin double-bridge resistivity apparatus of 1 μ -ohm sensitivity was employed in determining the resistivity vs. temperature curves for the alloys studied. Specimens $0.035'' \times 1/2'' \times 6''$ were cooled $30^\circ/\text{hr.}$ from a temperature of approximately 800°C. Mild steel leads were spot welded to the ends of the specimens and served as current connections to the bridge. Potential leads were also spot welded to the specimens and provided a test length of $5-1/4$ inches. The electrical and thermocouple leads were brought out through one end of a horizontal tube furnace, and an argon flow of about 75 cc/min was introduced through the other end to prevent excessive oxidation of the steel leads. By shunting the furnace it was possible to reduce the temperature gradient along the specimen length to $2-1/2^\circ \text{C.}$ Resistivity vs. temperature curves obtained on slow cooling ($30^\circ/\text{hr.}$) the various alloys are illustrated in Figure 2.

The resistivity curves shown in Figure 2, particularly those for the binary compositions, agree rather well with curves determined by Bennett (20). Upon slow cooling the binary alloys to the critical Fe_3Al transformation temperature, an anomalous increase in resistivity is noted instead of the decrease that would normally be expected to occur on ordering. Bennett attributes this rise in resistivity to the onset of order and the formation of antiphase domains, which are out of step with one another. Once long range order begins to set in, the resistivity decreases with temperature until complete order is attained.

A phase diagram of the iron-rich iron-aluminum system was recently determined by McQueen and Kuczynski (19) with dilatometry and is shown in Figure 3. Fairly good agreement is obtained between the critical temperatures indicated on the resistivity curves and those shown on this phase diagram. As an example, the resistivity curve for 13.9-Alfenol shows a temperature of 750°C for the FeAl transformation and a temperature of 535°C for the Fe_3Al transformation. These are quite close to the critical temperatures of 730°C and 520°C which were determined by dilatometric means.

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Figure 3. Constitution Diagram of Iron-Rich Fe-Al Alloys in the Solid Solution Range as Determined by McQueen and Kucaynski

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It is interesting to note that the shape of the curve for 13.9 aluminum with 1 percent molybdenum is similar to that of 16-Alfenol (Curve #2). This resemblance of shapes suggests that molybdenum and aluminum may have an additive effect. The similar behavior of molybdenum and aluminum under these circumstances may be reasonable in view of their similar atomic radii. Greater broadening of the peaks on the resistivity curves may possibly be attributed to the lower mobility of the molybdenum atom. It would therefore appear that the presence of molybdenum would tend to make the order-disorder transformation more sluggish, allowing more latitude in the heat-treatment studies of these materials.

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VII. X-RAY IDENTIFICATION OF SUPERLATTICE STRUCTURES IN IRON-ALUMINUM ALLOYS

A. Order-Disorder Structures of Iron-Aluminum Alloys

The basic structure of iron-aluminum alloys is body-centered cubic, but the atoms of iron and aluminum may be distributed in several different ways. Three types of superlattice structures may be distinguished:

- (1) Random distribution. The atoms of iron and aluminum are distributed at random.
- (2) The FeAl Structure.
- (3) The Fe₃Al Structure.

Types (2) and (3) are defined by referring to Figure 4. In the FeAl structure, aluminum atoms occupy entirely B and D positions, and iron atoms occupy entirely A and C positions. The unit cell for the structure is then body-centered cubic with lattice spacing a and one iron and one aluminum atom per unit cell.

In the Fe₃Al structure, aluminum atoms occupy either B positions or D positions, but not both. Iron atoms occupy all other remaining lattice positions. The resultant unit cell is face-centered cubic with lattice spacing $2a$ and an underlying body-centered cubic structure.

These three body-centered cubic structures diffract X-rays in three very similar but distinguishable ways. In the first, or random structure, the diffraction pattern will be that of the normal body-centered cubic lattice, i. e., X-rays will be diffracted only by those planes whose Miller indices, when added together, give an even integer.

The FeAl structure will yield a diffraction pattern whose principal lines correspond to the diffraction lines of the random structure. However, by virtue of the fact that the aluminum atoms in the body-center lattice positions (positions B and D in Figure 4) diffract X-rays more efficiently than do iron atoms, a phase difference of $\lambda/2$ for planes whose Miller indices yield odd sums on addition will not be precise and these planes will diffract weakly. These weak lines permit distinction between random and FeAl type structures.

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Figure 4. Unit cell of Fe_3Al

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In the Fe_3Al structure, the principal lines of the diffraction pattern will have d spacings identical to the diffraction lines of the random structure. However, the Miller indices of the principal lines of the Fe_3Al structure will be twice the indices of the random structure, because its lattice parameter is twice that of the random structure. It is obvious then that the weak lines of the FeAl structure will, in the Fe_3Al structure, have Miller indices whose sums are even and, consequently, will diffract more strongly. Also, since the outer structure of the Fe_3Al unit cell is face-centered cubic, minor lines will appear which are normal for diffraction by a face-centered cubic cell, i. e., those planes will also diffract whose Miller indices are all odd numbers. These minor lines permit distinction between the FeAl or random structures and the Fe_3Al structure. Table III is a summary of the planes in the diffraction patterns of the three structures.

TABLE III

The Miller Indices for the Diffracting Planes
Of the Three Lattices Encountered in Iron-Aluminum Alloys

<u>Random Structure</u>	<u>FeAl Structure</u>	<u>Fe₃Al Structure</u>
		111(weak)
	100	200
110	110	220
		311(weak)
	111 (weak)	222
200	200	400
		331(weak)
	210 (weak)	420
211	211	422
		333(weak)
220	220	440
		531(weak)
	300 (weak)	600
310	310	620

B. X-Ray Technique

In order to correlate mechanical properties with order-disorder transformations in alloys, it is necessary that one identify the predominating superlattice structure of a specimen to be tested. An X-ray specimen run in conjunction with annealing treatment of test specimens certainly fulfills this requirement. It was first attempted to run X-ray powder specimens vacuum-sealed in vycor capsules in conjunction with annealing of tensile specimens. Because of the time and difficulty involved in their preparation, the use of powder specimens was discontinued. Large single crystals grown by the strain-anneal method were used in place of powder.

After subjecting the single crystal specimen to the heat treatment to be evaluated, the crystal was oriented by the X-ray diffractometer technique. The diffractometer orienting device mounted on a Norelco diffractometer is illustrated in Figure 5. Basically the apparatus permits rotation of the specimen about two perpendicular axes in the X-ray beam and independent 2θ settings of a Geiger or scintillation counter to detect X-ray reflections. By recording on a stereographic plot the angular settings of as few as three planar reflections it is possible to deduce the orientations and settings of all possible planes in the crystal from symmetry relationships.

The diffractometer device was next employed to detect the type of order in the crystal by determining the presence or absence of superlattice lines. By referring to the plotted stereographic orientation of the crystal, the proper angular settings of the specimen with reference to the beam and the detector were obtained.

Table IV lists the reflecting planes for the various superlattice structures of iron-aluminum alloys together with approximate interplanar spacings and Bragg angles for cobalt K α radiation. Interplanar spacings and Bragg angles vary slightly with annealing treatment and aluminum content.

100 197

Figure 5. Single-Crystal Orienting Device on a
Norelco Diffractometer (Less Shielding)

200 138

TABLE IV

The Reflecting Planes of the Superlattice Structures
of 13.9 Alfenol

Structure			Interplanar spacing ($\text{\AA}$)	Bragg Angle θ degrees	Reflecting Angle 2θ - degrees
<u>Random</u>	<u>FeAl</u>	<u>Fe₃Al</u>			
		111	3.33	15.75	31.15
	100	200	2.89	18.05	36.10
110	110	220	2.04	25.95	51.90
		311	1.75	30.85	61.70
	111	222	1.67	32.50	65.00
200	200	400	1.45	28.18	76.35
		331	1.33	42.30	84.60
	210	420	1.30	43.62	87.25
211	211	422	1.18	49.15	98.30
		333	1.12	53.00	106.00
220	220	440	1.03	60.65	121.30
		531	0.98	66.00	132.00
	300	600	0.96	68.75	137.50
310	310	620	0.92	77.25	154.50

The crystal to be analyzed is first oriented and its stereogram plotted using the reflecting planes common to all three structures. These include the 310 (or 620), the 220 (or 440), and the 200 (or 400). Once the stereogram is plotted, the angular settings for any reflecting plane can be read directly. The detector can then be set at the proper 2θ angle and the crystal positioned correctly in the X-ray beam. If the type of order is such that reflection will occur for a given plane, the reflection will be picked up by the detector. It can be seen that since not all reflecting planes are present in all three types of order, the predominating structure in the crystal can be determined by a process of elimination.

It should be pointed out that this method is not quantitative in nature because the intensity of the reflection received by the detector is directly proportional to the area of the crystal surface irradiated. In turn, the area of surface irradiated is dependent on the angle at which the crystal is tilted to the X-ray beam. The intensity of a reflection is therefore dependent on the orientation of the crystal, and only the presence of the predominating structure will be detected.

A special advantage of the technique is that the test crystal can be used repeatedly to evaluate different heat treatments. Of course, this utilization implies that the heat treatments be of sufficient severity to erase previous order structures.

C. Correlation of X-ray Analysis and Resistivity Curves

The determination of type of superlattice formation as a function of various heat treatments are tabulated in Table V. The heat treatments were selected with the view of corroborating data obtained using the described X-ray technique with the results of McQueen and Kuczynski, and with the resistivity data obtained on slow cooling of the Alfenols (Figure 2).

A point of particular significance revealed by the X-ray data on 13.9 Alfenol is the dependence of the sensitivity of the resulting structure upon the severity of the quenching medium, even from temperatures as low as 500°C. The data indicates that oil quenching does not preserve the elevated-temperature structure, as manifested by critical points on the resistivity curves or otherwise, to room temperature.

TABLE V

A. Results of X-ray Crystal Analyses of 13.9 Alfenol

<u>No.</u>	<u>Heat Treatment</u>	<u>Type of Quench</u>	<u>Type of Order</u>
44A	2 hrs @ 850°C	oil	FeAl
44B	2 hrs @ 850°C	ice water	Random
55A	2 hrs @ 725°C, cooled 30°/hr to 300°C	oil	Fe ₃ Al
55B	2 hrs @ 725°C, cooled 30°/hr to 535°C	ice water	FeAl
56A	2 hrs @ 725°C, cooled 30°/hr to 540°C	oil	Fe ₃ Al
56B	2 hrs @ 725°C, cooled 30°/hr to 490°C	ice water	Fe ₃ Al

B. Results of X-ray Crystal Analyses of 16 Alfenol

<u>No.</u>	<u>Heat Treatment</u>	<u>Type of Quench</u>	<u>Type of Order</u>
215A	1 hr @ 725°C, air cooled 16 hrs @ 600°C, cooled 30°/hr to 490°C	ice water	FeAl
216A	1 hr @ 725°C, air cooled 16 hrs @ 600°C, cooled 30°/hr to 510°C	ice water	Fe ₃ Al
217A	1 hr @ 725°C, air cooled 16 hrs @ 600°C, cooled 30°/hr to 525°C	ice water	FeAl

VIII EFFECT OF SCAVENGING ADDITIONS ON IRON-ALUMINUM ALLOYS

In general, body-centered-cubic metals are susceptible to embrittlement by the presence of small amounts of interstitial impurities. Because of the extremely high free energies of formation of their oxides and nitrides, the rare earth metals would seem ideal for use as scavenger additions to the iron-aluminum base alloys. The rare earths, particularly the heavy metals, have been successfully used at the Denver Research Institute to produce ductile vanadium (21). Recent work at the Institute indicates that the rare earth metals can also be successfully applied to other refractory metals, such as molybdenum. Table VI below lists the free energies of formation for selected rare earth metals and aluminum. From thermodynamic considerations the rare earth metals listed should serve as excellent deoxidizers.

A preliminary investigation of yttrium and lanthanum as scavenging agents was carried out during the past year. Alloys containing 0.025, 0.05, 0.075, and 0.1% each of lanthanum and yttrium were prepared by arc melting. A metallographic examination disclosed the formation of a second phase in the grain boundaries of the as-cast buttons containing more than 0.025% of either lanthanum or yttrium, indicating a limited amount of solid solubility of these rare earth metals in iron-aluminum base alloys.

A. Workability of Alloys Containing Lanthanum and Yttrium

The alloy buttons were rolled in accordance with the rolling schedule described earlier. Those alloys containing lanthanum were extremely hot short and were therefore discarded. The alloys containing yttrium rolled extremely well. This was no doubt, at least partially attributable to the fine as-cast grain size of the buttons containing yttrium.

TABLE VI

Free Energies of Formation of Various Metal Oxides

<u>Metal</u>	<u>Oxide</u>	<u>-Δ F₂₉₈ (Kcal) per gram-Atom of O₂</u>
Y	Y ₂ O ₃	145.2 (22)
La	La ₂ O ₃	136.5 (23)
Ce	Ce ₂ O ₃	138.5 (23)
Al	Al ₂ O ₃	126 (23)

200 701

B. Microstructure of Alloys Containing Yttrium

The photomicrographs in Figure 6 show the effect of yttrium additions upon the microstructure of 13.8-Alfenol recrystallized at 725° C for two hours. Specimens were mounted so that the rolling plane could be examined. Initial stages of sample preparation were carried out by conventional polishing procedures, followed by final polish on a Syntron vibratory polishing machine with alumina abrasive. The grain size appears to be noticeably refined at concentrations of yttrium as low as 0.025 percent. At 0.05 percent a second phase is distributed throughout the structure in the form of small spheroids, with some located at the grain boundaries. The second phase appears as stringers in addition to the isolated spheroids at the 0.075 percent level. These stringers are composed of angular fragments, indicating that this second phase is not ductile at the temperature of warm rolling (575° C).

C. Effect of Yttrium Additions on Grain Size

In order to more fully evaluate yttrium additions for the purpose of grain-growth inhibition a series of specimens of 13.9-Alfenol with and without yttrium additions was annealed at 1050° C for one hour and air cooled. The photomicrographs of the resulting microstructures are shown in Figures 7 and 8. Figure 7 illustrates the grain size obtained in 13.9-Alfenol after one hour at temperature. Figure 8 demonstrates the effect of yttrium additions up to 0.1 percent upon the heat-treated grain size. The 0.025 percent addition did not have any effect, probably because all the yttrium was in solid solution at the temperature of heat treatment. Larger additions of yttrium to the alloy produce a progressive decrease in the grain size.

It was found that the addition of 0.05 percent yttrium produces a final grain size, after one hour at 1050° C, equivalent to the grain size of a yttrium-free alloy heated to 725° C for two hours.

The effect of yttrium upon the room temperature ductility of iron-aluminum alloys will be discussed in the following section on heat treatment and tensile properties.

(a)

(b)

(c)

(d)

Figure 6. 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_3 100x

Figure 7. Microstructure of 13.8-Alfenol.

Heated 1 hour at 1050°C. Air cooled.

Rolling plane structure

Etchant - 25 ml. Glycerin, 5 ml. HF,

0.5 ml. HNO_3 . 100 X

550 034

(a)

(b)

(c)

(d)

Figure 8. Microstructures of 13.8-Alfenol Containing

(a) 0.025 γ

(b) 0.050 γ

(c) 0.075 γ

(d) 0.100 γ

Heated 1 hour at 1050°C. Air cooled. 100X

500 35

IX. STUDY OF HEAT TREATING VARIABLES AND RESULTANT TENSILE PROPERTIES

A. Heat Treatments Based on Resistivity vs. Temperature Curves

Initial heat treatment studies were influenced to a great extent by consideration of the mechanisms suggested by Zener (24) for the minimization of micro-cracks in metals during the course of deformation. According to this theory cracks can be propagated at grain boundaries and similar obstacles by the impact of slip lines. Zener suggested that in certain alloys the slip line momentum can be slowed down by the presence of fine dispersions of a second phase, with the result that very high elongations can be obtained. It appeared highly probable that this mechanism of micro-crack formation might be operating in iron-aluminum alloys of high aluminum content, especially when it was observed that slip in these materials is crystallographic in nature. It was therefore conjectured that a heat treatment might be devised using antiphase domains to slow down slip line momentums and increase ductilities. Antiphase domains might be expected to produce the same effects as fine dispersions of a second phase.

Some evidence has been obtained which indicates that the presence of some degree of order does have a beneficial effect on elevated temperature properties in the case of Thermenol (11). Curves of ultimate strength, yield strength and elongation as functions of temperature are characterized by abnormal peaks in the temperature range of approximately 370 to 540°C. These temperatures correspond quite closely to the peaks on the resistivity curves which mark the onset of order.

1. Heat Treatment Studies of 13.9 Alfenol (Fe₃Al composition)

With this view in mind a series of heat treatments for 13.9 Alfenol was devised, using the resistivity vs. temperature curve for this alloy as a basis (Fig. 2). Table VII lists these heat treatments and their resulting tensile properties. Results for 8-Alfenol and 10-Alfenol were included for comparative purposes only, since these compositions are not expected to respond to order-disorder type heat treatments. Sub-size tensile specimens measuring 1-3/4 inches long with a reduced section of 1/4 inch and a gage length of 1 inch were used for these studies (see Fig. 9). The best tensile elongations of 9 percent were obtained with samples annealed at 725° C for two hours, equilibrated at 600° C for 16 hours, slow cooled to 520° C, followed

500 100

TABLE VII

Tensile Properties of Alfenols (0.035" Thick)

<u>Alfenol Composition</u>	<u>Heat Treatment</u>	<u>% Elongation*</u>	<u>Ultimate Strength $\times 10^{-3}$ psi</u>
8	1 hr at 725° C, oil quenched	26.0	61
10.18	1 hr at 725° C, oil quenched	15.0	83
13.83	2 hr at 725° C, cooled 30°/hr to 535° C, O.Q.	6.8	110
13.83	2 hr at 725° C, cooled 30°/hr to 490° C, oil quenched	4.8	109
13.83	1 hr at 725° C, cooled 30°/hr to 520° C, oil quenched	4.3	108
13.83	2 hr at 725° C, air cool. 16 hr at 600° C, cooled 30°/hr to 520° C, oil quenched	9.0	117
13.83	2 hr at 725° C, air cool. 16 hr at 600° C, cooled 30°/hr to 490° C, oil quenched	7.5	119
13.83	2 hr at 725° C, air cool. 16 hr at 600° C, cooled 30°/hr to 535° C, oil quenched	7.5	119

* Av. of two spec. per condition

(a) Undersized tensile specimen

(b) Intermediate size tensile specimen

Figure 9. Specifications for (a) Undersized and
(b) Intermediate Size Tensile Specimens

by an oil quench. Since the quenching temperature of 520° C represents a position approximately midway up the resistivity peak, it would appear that the formation of antiphase domains might be responsible for this significant improvement in room temperature ductility. Repeated attempts to reproduce these data were unsuccessful. Many minor variations of this basic heat treatment were also investigated without success. Identical heat treatments produced tensile elongations that varied from 4 to 7 percent. It was concluded that some of this difficulty could perhaps be attributed to the kinetics of the transformation at temperatures near the critical. With high atomic mobility near the critical order-disorder temperature range it is possible that sensitivity is such that it is extremely difficult to quench in the optimum structure at the proper moment. Green and Brown (25) in their work on "age softening" of beta brass were also confronted with a considerable amount of scatter in their data on quenching from temperatures at the vicinity of the critical. They attributed the variation to the rapidly changing degree of long range order just below the critical temperature.

2. Studies of 16-Alfenol and Thermenol-Type Alloys

It was pointed out earlier that both aluminum and molybdenum tend to (a) broaden the peaks of the resistivity curves, (b) raise the FeAl to Fe₃Al transformation temperature, and (c) to be additive in these effects. Tendencies appeared favorable then that the transformation kinetics of high aluminum and molybdenum alloys might be somewhat sluggish and would provide greater control for quenching-in various ordered structures. Although tensile elongations of 6 and 7 percent were obtained for both the 16-Alfenol and Thermenol-type alloys, the results were somewhat disappointing in view of the pronounced effect that molybdenum additions have upon the character of the resistivity curves.

B. Relation of Specimen Size to Tensile Properties

In order to dispel any doubts as to the effect specimen size might have upon tensile properties, several intermediate-size tensile specimens (1-1/2 inch gage length) were prepared from 0.035 inch thick 13.9-Alfenol. These were given the standard recrystallization anneal of 2 hours at 725° C, air cooled, treated for 16 hours at 600° C, cooled at the rate of 30°/hr to 520° C, and oil quenched. Average elongation and ultimate strength values obtained were 6 percent and 129,000 psi respectively. These results are comparable to the data obtained with sub-size specimens prepared from the same material.

C. Importance of Surface Preparation

Although the importance of surface condition on the tensile ductility of brittle-type alloys has been emphasized in the literature (26), it was not until late in the present program that this factor was discovered to have a profound effect upon the room temperature tensile properties of iron-aluminum alloys. Because of poor thermal conductivity, the iron-aluminum alloys are known to be susceptible to the formation of micro-cracks which can propagate rapidly, inducing premature failure. This detrimental condition can be brought about by improper machining and/or by drastic quenching from elevated temperatures. In view of this effect, it is believed that it has not been possible to evaluate fully the effectiveness of the earlier heat treatments. The notch effect undoubtedly was the controlling factor, masking the true mechanical properties of the material by promoting premature fracture. The erratic nature of the previous heat treatment studies can probably be blamed upon the presence of notches at the surface.

D. Electropolishing Conditions

All subsequent tensile studies were made upon specimens that were electropolished after heat treatment. A cursory investigation of electropolishing time indicated that one-half hour with the following bath conditions would provide a suitable surface:

Electrolytic Bath - Concentrated H_3PO_4
 Temperature - 50° to 75° C
 Current Density - 50 Amps/dm²

Polishing under the above conditions allows the removal of approximately 2-1/2 mils from each surface. Longer polishing times tend to change the shape of the specimen and shorter periods do not consistently eliminate the notch effect.

Table VIII summarizes the available tensile data obtained after heat treatment of a variety of electropolished iron-aluminum alloy specimens. For clarity and convenience the table is further divided by alloy designation into six different parts. The most dramatic improvement in ductility as a result of electropolishing alone is best illustrated in Part A of Table VIII. Prior to the use of the electropolishing procedure, a tensile elongation of 3 to 3.5% was obtained for a heat treatment of 1-1/2 hours at 800° C in vacuum. After electropolishing it can be seen that a fourfold improvement in ductility is possible with the same heat treatment (specimens 1A to 3A). Similarly a significant improvement in room temperature

TABLE VIII

Part A - 12.25-Alfenol (0.035" thick)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength $\times$ 10^{-3} psi
1A	1-1/2 hr @ 800° C in vacuo, cooled on brass plate.	Recrystallized	12.5	110
2A			12.0	110
3A			10.5	115
4A	1-1/2 hr @ 800° C in air, cooled on steel plate.	Recrystallized	8.5	102
5A			10.0	98
6A			9.5	106
7A	2 hr @ 725° C, oil quenched.	Recrystallized	6.5	95
8A			6.0	96
9A	2 hr @ 725° C, oil quenched. 1 hr @ 500° C, water quenched.	Recrystallized	12.5	92
10A			8.0	85
11A	2 hr @ 725° C, oil quenched. 1 hr @ 500° C, cooled 60°/hr to 300° C, held 24 hr @ 300° C, furnace cooled to room temperature.	Recrystallized	10.5	106
12A			14.0	110
13A	2 hr @ 725° C, oil quenched. 1 hr @ 500° C, air cooled	Recrystallized	5.0	90
14A			10.5	90

TABLE VIII

Part B - 13.6 Alfenol (0.020" Thick)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength $\times$ 10^{-3} psi
1B	1 hr @ 725° C, water quenched. 0.014" stock	Recrystallized	11.5	93
2B			8.0	105
3B	2 hr @ 725° C, air cooled. 16 hr @ 600° C, cooled 30°/hr to 520° C, oil quenched. 0.014" stock.	Recrystallized	10.0	105
4B			8.5	105
5B	1/4 hr @ 650° C, oil quenched.	Partially Recovered	9.5	116
6B			8.0	119
7B			10.5	119
8B	1/2 hr @ 650° C, oil quenched.	Recovered	10.0	114
9B			9.0	114
10B			4.0	111
11B	3/4 hr @ 650° C, oil quenched.	Partially Recrystallized	7.5	105
12B			10.0	113
13B			9.0	113
14B	1 hr @ 650° C, oil quenched.	Partially Recrystallized	9.0	119
15B			6.0	107
16B			7.0	119

TABLE VIII

Part B - 13.6 Alfenol (0.020" Thick) (Continued)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength $\times$ 10^{-3} psi
17B			10.0	126
18B	1/2 hr @ 650° C, cooled 60°/hr to 300° C, held 24 hr @ 300° C, air cooled.	Recovered	9.0	125
19B			6.0	117
20B			8.0	136
21B	24 hr @ 650° C, cooled 60°/hr to 300° C, air cooled	Fully Recrystallized	9.0	131
22B			10.0	148
23B			10.0	109
24B	24 hr @ 650° C oil quenched.	Fully	9.0	109
25B		Recrystallized	10.0	109
26B			8.0	117
27B	24 hr @ 650° C, cooled 30°/hr to 535° C, oil quenched.	Fully Recrystallized	10.0	115
28B			10.0	116
29B	24 hr @ 650° C, cooled 30°/hr to 520° C, oil quenched.	Fully	8.5	112
30B		Recrystallized	7.5	103
31B			9.0	107

TABLE VIII

Part B - 13.6 Alfenol (0.020" Thick) (Continued)

<u>Spec. No.</u>	<u>Heat Treatment</u>	<u>Microstructure</u>	<u>% Elongation</u>	<u>Ultimate Strength × 10⁻³ psi</u>
32B	24 hr @ 650° C, cooled 30°/hr to 490° C, oil quenched		9.0	108
33B		Fully	8.0	108
34B		Recrystallized	9.0	110
35B	1/2 hr @ 650°C, cooled 60°/hr to 550° C, oil quenched.		8.0	108
36B		Fully	8.5	112
37B		Recovered	10.0	118
38B	1/2 hr @ 650° C, cooled 60°/hr to 300°C, air cooled. 1/4 hr @ 500° C, oil quenched.		10.0	130
39B		Fully	10.0	118
40B		Recovered	12.0	117

TABLE VIII

Part C - 13.9-Alfenol (0.035" Thick)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength × 10 ⁻³ psi
1C	2 hr @ 725° C, oil quenched.	Fully Recrystallized	6.5	109
2C			8.5	114
3C			10.0	117
4C			6.0	117
5C			6.5	116
6C			8.0	120
7C	2 hr @ 725°C, cooled 30°/hr to 300°C, air cooled	Fully Recrystallized	9.0	127
8C			10.5	127
9C			11.0	129
10C	16 hr @ 600° C, oil quenched.	Fully Recovered	9.5	119
11C			9.5	119
12C			9.0	121
13C	1/4 hr @ 725° C, oil quenched.	Partially Recovered	6.0	118
14C			5.0	118
15C			9.5	120
16C			9.5	120

TABLE VIII

Part C - 13.9-Alfenol (0.035" Thick) (Continued)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength $\times$ 10^{-3} psi
17C	1/2 hr @ 725° C, oil quenched.	Fully	10.0	117
18C		Recovered	10.5	118
19C			9.5	118
20C	3/4 hr @ 725° C, oil quenched.	Incomplete	5.5	120
21C		Recrystallization	5.0	116
22C	1 hr @ 725° C, oil quenched.	Incomplete	9.5	116
23C		Recrystallization	7.0	117
24C	16 hr @ 600°C, cooled 60°/hr to 300°C, held 24 hr @ 300°C, air cooled.	Fully	8.5	135
25C		Recovered	9.5	136
26C	1/2 hr @ 725° C, air cooled.	Fully	4.5	116
27C		Recovered	8.0	124
28C	2 hr @ 725° C, air cooled. 16 hr @ 600° C, cooled 30°/hr to 520° C, oil quenched	Recrystallized	3.5	90
29C			7.0	114
30C			4.0	109
31C	2 hr @ 725° C, oil quenched.	Recrystallized	5.0	111
32C			4.0	113
33C			5.0	106

TABLE VIII

Part D - 16-Alfenol (0.035" Thick)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength × 10 ⁻³ psi
1D	2 hr @ 725 ° C, oil quenched.	Recrystallized	8.0	99
2D			5.0	86
3D			5.0	121
4D	2 hr @ 725°C, air cooled. 16 hr @ 600°C, oil quenched	Recrystallized	9.5	102
5D			6.5	102
6D			10.0	110
7D			12.0	128
8D			6.0	95*
9D			7.5	107*
10D	2 hr @ 725° C, oil quenched, 16 hr. @ 600° C, oil quenched. Electropolished on machined edges only.	Recrystallized	7.5	108
11D			6.0	83
12D	16 hr @ 600° C, air cooled	Fully Recovered	8.0	118
13D			11.0	123
14D			8.5	128
15D			10.5	123
16D	Cold rolled material, electropolished	Cold worked	2.0	110

* Not Electropolished

TABLE VIII

Part E - Plain Thermenol (0.035" Thick)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength $\times$ 10^{-3} psi
1E	2 hr @ 725° C, oil quenched.	Recrystallized	6.0	111
2E			8.5	96
3E			6.0	114*
4E			6.0	101*
5E			6.0	94
6E			4.0	74
7E			4.5	80
8E	1-1/2 hr @ 800° C, in vacuo, furnace cooled.	Recrystallized	8.0	95
9E			5.5	80
10E	16 hr @ 600° C oil quenched.	Recovered	10.7	125*
11E			8.7	117*
12E	2 hr @ 725° C, cooled 60°/hr to 300° C, held 24 hr @ 300° C, air cooled.	Recrystallized	4.5	105
13E			7.5	105
14E			7.0	113
15E			6.0	98
16E	2 hr @ 725° C, cooled 60°/hr to 460° C, oil quenched.	Recrystallized	7.5	106
17E			7.5	106

* Intermediate size tensile specimens, 1-1/2" gage length.

TABLE VIII

Part E - Plain Thermenol (0.035" Thick) (Continued)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength $\times$ 10^{-3} psi
18E	1/2 hr @ 675° C, oil quenched.	Partially	9.5	112
19E		Recovered	10.0	116
20E			9.0	100
21E	1/2 hr @ 725° C, cooled 60°/hr to 300° C, held 60 hr @ 300° C, air cooled	Recovered	6.0	100
22E			6.5	110
23E			10.0	107
24E	1/2 hr @ 725° C, cooled 60°/hr to 460° C, oil quenched	Recovered	5.0	92
25E			10.0	117
26E			10.0	120
27E	1/2 hr @ 725° C, oil quenched	Recovered	11.0	116
28E			14.0	132
29E			13.5	134
30E			11.5	115
31E			10.5	113
32E	1/2 hr @ 725° C, cooled 30°/hr to 600° C, held 10 min. @ 600° C, oil quenched.	Recovered	9.0	108
33E			7.5	95
34E			9.5	114

TABLE VIII

Part E - Plain Thermenol (0.035" Thick) (Continued)

<u>Spec. No.</u>	<u>Heat Treatment</u>	<u>Microstructure</u>	<u>% Elongation</u>	<u>Ultimate Strength × 10⁻³ psi</u>
35E	1/2 hr @ 725° C, cooled 30°/hr to 560° C, held 10 min. @ 560° C, oil quenched.	Recovered	10.0	119
36E			7.5	102
37E			8.5	119
38E	1/2 hr @ 725° C, air cooled.	Recovered	8.5	118
39E			6.0	101
40E			5.5	93
41E	1/2 hr @ 725° C, water quenched.	Recovered	9.0	123
42E			6.5	104
43E			7.5	113

TABLE VIII

Part F - Modified Thermenol (0.035" Thick)

Spec. No.	Heat Treatment	Microstructure	% Elongation	Ultimate Strength $\times$ 10^{-3} psi
1F	1 hr @ 725° C, oil quenched	Incomplete	6.0	105
2F		Recrystallization	9.0	126
3F			10.0	123
4F	1 hr @ 725° C, oil quenched-Not elec- tropolished.	Incomplete	7.0	121
5F		Recrystallization	7.0	114
6F	Furnace heat to 1050° C, hold 1 hr @ 1050° C, cool 30°/hr to 600° C, held 10 min. @ 600° C.	Recrystallized	†5.0	80
7F			†3.0	79
8F			†3.5	79
9F			*5.5	90
10F			*5.5	91
11F			*5.0	83
12F	2 hr @ 800° C, oil quenched.	Recrystallized	7.5	114
13F			7.5	116
14F			7.5	116
15F	1 hr @ 800° C, oil quenched.	Recrystallized	8.0	124
16F			9.0	129
17F			8.0	128
18F	1/2 hr @ 725° C, oil quenched.	Recovered	7.0	157
19F			8.0	150
20F			8.0	144

† Water quenched

* Oil Quenched

tensile properties was noted also for 16-Alfenol in the same heat-treated condition, but differing only in surface preparation. Refer to Part D and specimen Numbers 4D through 7D. Note that specimens 8D and 9D, which were not electropolished, have been included for comparison. In scanning the entire table it should be noted that in almost all instances high ultimate strengths are associated with high ductility values, providing further evidence that premature fracture can be caused by the presence of micro-cracks.

E. Effect of Microstructure on Ductility

Even though fairly high average tensile elongations were obtainable with electropolished surfaces, it was quite evident that there was still considerable scatter among completely recrystallized specimens with identical heat treatments. It was found in general that a fully recovered microstructure (recognized metallographically by incipient recrystallization) promoted a greater degree of consistency between identically heat treated specimens. This was particularly true for the thicker specimens (0.035"). Refer to specimens with fully recovered microstructures in Part C of the table and note the consistent 9 to 10% tensile elongations.

A further check on the assumption that an equiaxed recrystallized grain structure may be undesirable was made with Alfenol of approximately the same composition as above (13.6 Al), but of a thinner gage (Part B). The lower recrystallization temperature of this material necessitated a lower annealing temperature (650°C) in order to duplicate the microstructures of the preceding materials. It should be noted that these elongations tended to approach 9 to 10 percent regardless of microstructure. A possible explanation for the apparent reduced notch-sensitivity of the 0.020" material, as compared to the 0.035" specimens, may be the tendency of the thinner sections to minimize thermal gradients during quenching.

Improved ductilities were obtained also for 16-Alfenol and Plain Thermenol with fully recovered microstructures. See Parts D and E of Table VIII. Up to 11 and 14 percent elongations were obtained with 16-Alfenol and plain Thermenol, respectively. This is the highest ductility value obtained on iron-aluminum alloys containing 16 percent aluminum, with the exception of a single value of 14 percent reported by Thompson-Ramo Wooldridge in their work on Thermenol for the Navy Bureau of Aeronautics, as mentioned in an earlier section.

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Experimentation with modified Thermenol indicated that consistency in elongations could be expected with this alloy, also. Modified Thermenol contains a small amount of a dispersion of zirconium carbide and is characterized by a very fine grain structure, maintaining this structure to relatively high temperatures. Because of the exceptional high-temperature grain size stability of this alloy, several heat treatments were carried out at 1050° C (Part F-spec. 6F through 11F); however, inferior properties resulted, tending to indicate that an equiaxed, recrystallized grain structure is prone to notch sensitivity effects.

F. Effect of Scavengers on Tensile Properties

It was expected that the addition of yttrium, with its extremely high free energies of formation of the oxides, would result in substantial improvements in the room temperature ductilities of iron-aluminum alloys. The expected improvements were not realized, however, and may be due in a great measure to the second phase that was found in alloys containing more than 0.025 percent yttrium. Specimens 28C through 33C typify the results that can be expected with yttrium additions, with tensile elongations ranging from 3.5 to 7 percent.

G. Some Effects of Order and Disorder Upon Tensile Properties

To determine the properties of slow cooled(ordered) structures, a number of specimens of 13.9-Alfenol were cooled in such a manner that the amount of Fe_3Al order present would be comparable in all samples. Refer to Part C (specimens 7C to 9C and 24C to 25C) in the table. It is apparent from the elongations and strengths reported that notch effects were absent. The significant increase in ultimate strength noted in this series of specimens can be attributed to the effect of Fe_3Al order, in agreement with order-disorder theory (27). In the absence of serious notch sensitivity, it would be expected that order would decrease the mobility of dislocations and contribute toward a high work - hardening rate. The ductilities of 13.9-Alfenol, as measured by elongation at fracture, were comparable to the ductilities obtained by quenching this alloy from above the Fe_3Al transformation. The relatively high elongations of the ordered material were surprising, in view of previous tests on ordered specimens which had not been electropolished. These latter specimens exhibited elongations in the range from 2 to 7 percent. However, under conditions of equal and high notch sensitivity, it would be expected that an ordered alloy characterized by high strength and work hardening rate would fracture

at lower total elongations than a disordered alloy which could flow more readily at the lower stress levels.

It has been noted with interest that widely divergent heat treatments of 13.6 and 13.9-Alfenol seem to produce elongations which fall into a narrow band of 9 to 10 percent. A possible explanation for this fact may be the tendency for the Fe_3Al composition to form a metastable "foam-type" structure, which is common for the DO_3 crystal structure type (28). Should this occur, growth of the antiphase domains would be greatly inhibited. Heating of these type structures in the vicinity of the critical temperature may offer some promise as a means for obtaining varying domain sizes. Reference should be made to Part B-spec. Numbers 38B to 40B. Although these results are preliminary, it is felt that this approach can be a fruitful area for further investigation.

Unlike 13.9-Alfenol (Fe_3Al composition), Thermenol, quenched from the FeAl order region, would not be expected to be complicated by the presence of a "foam-type" structure. Since the FeAl ordered structure is capable of forming only two different antiphase domains, growth of these domains can take place without interference. In general the lowest ductility values of 6 to 10 percent were obtained with slow-cooled or fully-ordered alloys (Part E- spec. 21E to 23E). The most significant improvements in ductility of 11 to 14 percent were obtained with Thermenol in the fully recovered condition, quenched from the FeAl order region (spec. 27E through 31E).

In view of the foregoing discussion, it would appear that perhaps some degree of order may be desirable in order to attain maximum ductilities in iron-aluminum alloys containing more than 10 percent aluminum.

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X. FUTURE CONSIDERATIONS

Although significant improvements in room temperature ductilities of the iron-aluminum alloys have been made, it is quite clear from the foregoing discussions that much still remains to be learned regarding order-disorder transformations and mechanical properties.

The research effort for the coming year will consist of the following five phases:

- Phase I - Continuation of Order-Disorder Heat Treatment Studies
- Phase II - Investigation of the Effects of Microstructure
- Phase III - Effect of the Removal of Interstitials upon Room Temperature Ductility
- Phase IV - Study of the Effect of Surface Preparation
- Phase V - Correlation of Mechanical Properties with Order-Disorder Structures.

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