THE LATTICE PARAMETERS OF IRON-ALUMINUM
SILICON ALLOYS WITH THE DO_3 STRUCTURE

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Based on a M. S. thesis submitted to Iowa State University

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silicon alloys with the DO_3 structure

by

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The lattice parameter of iron-aluminum-silicon alloys
with the DO_3 structure*

Stephen Joseph Cowdery

Abstract

The room temperature lattice parameters of $Fe_{75}Si_xAl_{25-x}$ alloys for $x = 0$ to 25 and $Fe_{73}Si_xAl_{27-x}$ alloys for $x = 13.5$ to 24.3 were measured, as well as alloys of the composition $Fe_{68}Si_{10}Al_{22}$ and $Fe_{73.7}Si_{16.6}Al_{9.7}$, by x-ray techniques. Debye Scherrer powder photographs and a singly-iterative, weighted least-squares computer program were employed in this investigation. The values obtained were found to vary non-linearly from $5.6533\text{\AA}$ for Fe_3Si to $5.7935\text{\AA}$ for Fe_3Al for the $Fe_{75}Si_xAl_{25-x}$ alloys, and from $5.6569\text{\AA}$ for 10 wt % $Fe_{73}Al_{27-90}$ wt % $Fe_{73}Si_{27}$ to $5.7148\text{\AA}$ for 50 wt % $Fe_{73}Al_{27-50}$ wt % $Fe_{73}Si_{27}$ for the $Fe_{73}Si_xAl_{27-x}$ alloys. A polynomial fit was obtained for both curves. The lattice parameter of $Fe_{68}Si_{10}Al_{22}$ was found to be $5.7308\text{\AA}$ and that of $Fe_{73.7}Si_{16.6}Al_{9.7}$, the Sendust alloy, $5.6993\text{\AA}$. All values are corrected for refraction. Preliminary elastic constant measurements were also performed on a Fe_3Al single crystal at $77.4^\circ K$.

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INTRODUCTION

Ternary alloys of iron, silicon, and aluminum have elicited some attention in recent years because of the remarkable, and sometimes unusual, magnetic properties of certain alloys in this system (1).

In this investigation, alloys of the compositions $\text{Fe}_3\text{Si}_x\text{Al}_{1-x}$, for $x=0$ to 1, and $\text{Fe}_{73}\text{Si}_x\text{Al}_{27-x}$, for $x=13.5$ to 24.3, were used. These alloys are all long-range ordered at room temperature with a DO_3 -type unit cell (2), shown in Figure 1. This lattice is comprised of four interpenetrating face-centered cubic lattices denoted in the figure as I, II, III, and IV. In perfectly ordered, stoichiometric Fe_3Al or Fe_3Si , all I, II, and III sites will be occupied by iron atoms and all IV sites will be occupied by an aluminum or silicon atom. Fe_3Al and Fe_3Si form a continuous range of solid solutions between them and all alloys are ferromagnetic (2).

Iron-silicon alloys containing less than 5 weight percent silicon have enjoyed widespread application for their electrical and magnetic properties (1). These properties, as well as the lattice constants for iron-silicon alloys, have been fairly extensively investigated. Farquhar, Lipson and Weill (3) have measured these lattice parameters from 12.5 to 25.5 atomic percent silicon and report a value of $5.650\text{\AA}$ for Fe_3Si . Extrapolation of their results gives a lattice parameter of

Figure 1. The DO_3 unit cell

5.644Å for $\text{Fe}_{73}\text{Si}_{27}$.

Although they have less commercial usage, iron-aluminum alloys also exhibit excellent electrical and magnetic properties (1). Bradley and Jay (4), in addition to Taylor and Jones (5), report that the lattice constants of iron-aluminum alloys plateau from about 20 to 28 atomic percent aluminum at a value of 5.792Å. Taylor and Jones used alloys quenched from 250°C and reported a slight minimum at 24.3 atomic percent aluminum but it is debatable whether their values are good enough to discern as small of a drop as they claim. They link the constancy of the lattice parameters in this range to the onset of the DO_3 ordered structure.

Investigations of the ternary alloys of iron, silicon, and aluminum are less common. In 1932, Masumoto discovered that alloys containing 6 to 11 weight percent silicon and 4 to 8 weight percent aluminum have an especially large initial permeability and a very small hysteresis-loss (6). The largest initial permeability, 35100, occurred at a composition of 9.6 weight percent silicon and 5.4 weight percent aluminum. These alloys are extremely brittle and thus easily crushed - hence the name Sendust. Sendust is unusual in that the initial permeability in low fields decreased with increasing temperature up to about 200°C with the peak value occurring at about -100°C. The resistivity was maximized at a total silicon and aluminum content of about 10 weight per-

cent.

The Sendust sample used in this investigation is described by Masumoto as being representative of the ternary alloys' magnetic properties. He reported, for an alloy containing 9.6 weight percent silicon and 5.4 weight percent aluminum, an initial permeability of 35100, a maximum permeability of 117500, and a hysteresis-loss of 28 ergs/cm³ per cycle. The coercive force was 0.022 oersteds, the residual magnetic induction, 3350 gauss, and the intrinsic magnetic induction in a field of 1200 oersteds was 1100 gauss. The specific electrical resistance at 20°C was 81.2 microhm cm.

J. Suwalski et al., recently performed a study of the order-disorder transformation in iron-silicon-aluminum alloys using Mössbauer effect spectroscopy (7). They selected two representative alloys, Fe₆₈Si₁₀Al₂₂ and Fe₇₃Si₁₆Al₁₁, called Alsifer 32 and Alsifer 27, respectively.

Alsifer 27, a composition very close to the Sendust composition, showed well-developed Zeeman patterns characteristic of a ferromagnetic material. Powdering of this alloy disrupts the order and line broadening, originating from a random arrangement of aluminum and silicon atoms, appears. Thermal homogenization leads to full recovery of the initial ordered state.

Alsifer 32, on the other hand, is reported to be paramagnetic. Their results showed the single central peak characteristic of paramagnetic materials. After crushing, how-

ever, a mixed spectra arose with a broadened Zeeman pattern joining the single central peak. This pattern persisted after thermal homogenization. A spectrum taken of the annealed specimen at liquid helium temperature showed a more pronounced Zeeman pattern.

Suwalski concludes from his Mössbauer data that the aluminum and silicon atoms in excess of 25 atomic percent tend to replace the iron atoms at the III sites. This is in agreement with a paper by Bradley and Jay (8) in which the atomic positions were ascertained from measurements of the intensities of the superlattice lines in their x-ray patterns. The sublattice with the III and IV sites fills up as more aluminum or silicon is added until the composition reaches FeAl.

It has been widely assumed that the lattice parameters vary linearly from Fe_3Si to Fe_3Al . Niculescu et al. have reported this in two separate papers (2). They base their contention on lattice parameter measurements of four alloys, between Fe_3Si and $\text{Fe}_3\text{Si}_{.75}\text{Al}_{.25}$, and an extrapolation of this line to the value commonly reported for Fe_3Al . With four experimental points and lattice parameters that are reported to only the third decimal place, there is some question as to whether they actually had a straight line or not (9). As will be seen, the change is not linear.

The purpose of the present investigation was to determine the lattice parameters of $\text{Fe}_3\text{Si}_x\text{Al}_{1-x}$ alloys from $x=0$ to 1,

the lattice parameters of $\text{Fe}_{73}\text{Si}_x\text{Al}_{27-x}$, for $x=13.5$ to 24.3 (i.e. from 10 to 50 weight percent $\text{Fe}_{73}\text{Al}_{27}$), and the lattice parameters of the Sendust composition ($\text{Fe}_{73.7}\text{Si}_{16.6}\text{Al}_{9.7}$) and Alsifer 32 composition ($\text{Fe}_{68}\text{Si}_{10}\text{Al}_{22}$) alloys. The study of the Fe_3Si - Fe_3Al series was undertaken to determine the rate at which the lattice expands from Fe_3Si to Fe_3Al . This was to provide a means for characterization of the compositions of alloys in this range using only the lattice parameter. This is in addition to all of the other information that can be obtained with a knowledge of the lattice parameter. The $\text{Fe}_{73}\text{Si}_{27}$ - $\text{Fe}_{73}\text{Al}_{27}$ series was included in order to bracket the Sendust composition and thus facilitate further investigations into alloys in this region.

An attempt was also made to redetermine the zero-field, adiabatic, elastic constants of a single crystal of Fe_3Al at 77.4°K . Previous elastic constant measurements made by Leamy, Gibson, and Kayser (10) in this laboratory were made with a crystal of lesser quality than is capable of being grown here now and with a vastly inferior ultrasonic apparatus. Because of the strong ferromagnetic domain walls in Fe_3Al single crystals, it was impossible to propagate signals through the crystal without placing it in a magnetic field at practical saturation with the old equipment. Even with a more powerful signal generator in the new equipment, it was difficult to propagate the pulse through the sample without

significant attenuation. Because of this, the results obtained in the present work are, in some respects, tentative. The results were also limited since they were taken at only one temperature. For these reasons, this part of the thesis is discussed in the Appendix.

ALLOY AND SAMPLE PREPARATION

The samples used in this investigation were prepared in three groups - the Fe_3Si - Fe_3Al series, the $\text{Fe}_{73}\text{Si}_{27}$ - $\text{Fe}_{73}\text{Al}_{27}$ series, and the Alsifer 32 and Sendust composition alloys.

Master alloys of Fe_3Si and Fe_3Al were used as the melt stock for the Fe_3Si - Fe_3Al series. The Fe_3Al master alloy was prepared using a previously made, consumably arc-melted, alloy of iron plus 17 weight percent aluminum and arc-melted electrolytic iron. The Fe_3Si master alloy was made with arc-melted electrolytic iron also, plus semiconductor grade silicon. Both master alloys were arc-melted under helium with a tungsten electrode. The Fe_3Si alloy was crushed and the Fe_3Al alloy was cut into pieces with a sparkcutter to produce the melt stock for the rest of the series.

The masses of Fe_3Al and Fe_3Si needed to produce the $\text{Fe}_{3-x}\text{Si}_x\text{Al}_{1-x}$ alloys were calculated for x between 0 and 1 in 0.1 increments and carefully weighed. These alloys were arc-melted under helium into small buttons, then turned over and arc-melted in a small finger mold.

The arc-melted fingers were homogenized in a furnace heated to 1100°C and held there for one week before allowing the furnace to go back to room temperature. Because of the good oxidation resistance of these alloys, this heat treatment was done in air.

The $\text{Fe}_{73}\text{Al}_{27}$ master alloy was prepared in the same

fashion - arc-melting electrolytic iron and the previously prepared iron plus 17 weight percent aluminum alloy. The final finger of $\text{Fe}_{73}\text{Al}_{27}$ was spark cut into pieces and, as before, the cut faces were polished to avoid contamination. A button of $\text{Fe}_{73}\text{Si}_{27}$ was also prepared, as before, by arc-melting electrolytic iron and semiconductor grade silicon, and later crushed.

The masses of $\text{Fe}_{73}\text{Al}_{27}$ and $\text{Fe}_{73}\text{Si}_{27}$ needed to produce alloys of 10, 20, 30, 40, and 50 weight percent $\text{Fe}_{73}\text{Al}_{27}$ were calculated, measured, and the alloys arc-melted. The resultant fingers were homogenized by heating in air for seven days at 1031°C .

Alloys of the Sendust ($\text{Fe}_{73.7}\text{Si}_{16.6}\text{Al}_{9.7}$) and Alsifer 32 ($\text{Fe}_{68}\text{Si}_{10}\text{Al}_{22}$) compositions were also arc-melted under helium using electrolytic iron, the iron plus 17 weight percent aluminum alloy, and semiconductor grade silicon.

A second sample of Fe_3Al was also arc-melted because of possible contamination of the first Fe_3Al x-ray powder sample. The Fe_3Al and the Alsifer 32 samples were homogenized at 1044°C for five days while the Sendust sample was homogenized at 1025°C for five days (the Sendust sample was prepared prior to the other two).

For all three groups, a slab about 1/8 inch thick was spark cut out of the center of each of the fingers after the homogenizing anneal. The remaining external surface was

ground off and about 1/3 of the central portion of the remaining slab was powdered by crushing in a tool steel mortar and pestle.

The powders from the $\text{Fe}_3\text{Si}-\text{Fe}_3\text{Al}$ series were sealed in individual Vycor tubes and annealed at 650°C for 24 hours, cooled to 580°C and held for another 24 hours, and then step cooled 20°C per day until reaching room temperature. The $\text{Fe}_{73}\text{Si}_{27}-\text{Fe}_{73}\text{Al}_{27}$ series powders were placed in separate tantalum thimbles and the Alsifer 32 and Sendust composition alloys, along with the second Fe_3Al sample, were placed in separate tantalum envelopes. All were charged into a stainless steel retort and ion-pumped down to about 10^{-8} torr. The samples were heated to 650°C for three hours, cooled to 625°C and held for 24 hours, then cooled 25°C every 48 hours until reaching 300°C . After reaching 300°C , they were cooled 25°C every 72 hours until they reached room temperature.

The $\text{Fe}_3\text{Si}-\text{Fe}_3\text{Al}$ series samples were analyzed chemically for dissolved oxygen, nitrogen, and hydrogen which were found to average about 40 ± 10 , 9 ± 4 , and 3 ± 2 wppm, respectively. Carbon content was found to average about 30 wppm in the Fe_3Si and the Fe_3Al master alloys. Chemical analysis was not carried out for aluminum and silicon content. This was because only high purity charge materials were used and all materials were carefully weighed before and after arc-melting. Mass losses after melting were very small, all less than 0.15% of

premelted mass, and averaged about 0.04%.

As will be seen, the lattice constants obtained from these samples form a smooth curve when plotted against composition and the values for Fe_3Si and Fe_3Al correspond well to previously reported values for the alloys. Therefore, not even a systematic error is suspected in the composition.

The compositions, in weight and mole percent Fe_3Si and Fe_3Al , $\text{Fe}_{73}\text{Si}_{27}$ and $\text{Fe}_{73}\text{Al}_{27}$, and $\text{Fe}_{68}\text{Al}_{32}$, $\text{Fe}_{68}\text{Si}_{32}$, $\text{Fe}_{73.7}\text{Al}_{26.3}$, and $\text{Fe}_{73.7}\text{Si}_{26.3}$; and weight and atomic percent iron, aluminum, and silicon, for each alloy, are presented in Tables 1, 2, and 3.

Table 1. Compositions of Fe_3Si - Fe_3Al alloys

Wt % Fe_3Si	Wt % Fe_3Al	Mole % Fe_3Si	Mole % Fe_3Al	Wt % Fe	Wt % Si	Wt % Al	At.% Fe	At.% Si	At.% Al
0	100	0	100	86.13	0	13.87	75.00	0	25.00
10	90	9.96	90.04	86.08	1.44	12.48	75.00	2.49	22.51
20	80	19.91	80.09	86.03	2.87	11.10	75.00	4.97	20.03
30	70	29.88	70.12	85.98	4.31	9.71	75.00	7.47	17.53
40	60	39.87	60.13	85.93	5.74	8.32	75.00	9.96	15.04
50	50	49.86	50.14	85.88	7.18	6.94	75.00	12.46	12.54
60	40	59.87	40.13	85.84	8.62	5.55	75.00	14.97	10.03
70	30	69.89	30.11	85.79	10.05	4.17	75.00	17.46	7.54
80	20	79.91	20.09	85.74	11.49	2.77	75.00	19.98	5.02
90	10	89.95	10.05	85.69	12.92	1.39	75.00	22.48	2.52
100	0	100	0	85.64	14.36	0	75.00	25.00	0

Table 2. Compositions of $\text{Fe}_{73}\text{Si}_{27}$ - $\text{Fe}_{73}\text{Al}_{27}$ alloys

Wt % $\text{Fe}_{73}\text{Si}_{27}$	Wt % $\text{Fe}_{73}\text{Al}_{27}$	Mole % $\text{Fe}_{73}\text{Si}_{27}$	Mole % $\text{Fe}_{73}\text{Al}_{27}$	Wt %	Wt %	Wt %	At. %	At. %	At. %
90	10	89.94	10.06	84.37	14.11	1.52	73.00	24.28	2.72
80	20	79.90	20.10	84.42	12.55	3.03	73.00	21.58	5.42
70	30	69.87	30.13	84.47	10.98	4.55	73.00	18.87	8.13
60	40	59.85	40.15	84.53	9.41	6.06	73.00	16.16	10.84
50	50	49.85	50.15	84.58	7.84	7.58	73.00	13.46	13.54

Table 3. Compositions of Sendust and Alsifer 32 alloys

Wt % $\text{Fe}_x\text{Si}_{1-x}$	Wt % $\text{Fe}_x\text{Al}_{1-x}$	Mole % $\text{Fe}_x\text{Si}_{1-x}$	Mole % $\text{Fe}_x\text{Al}_{1-x}$	Wt % Fe	Wt % Si	Wt % Al	At. % Fe	At. % Si	At. % Al
Sendust $x = 73.74$									
63.21	36.79	63.07	36.93	85.00	9.60	5.40	73.74	16.56	9.70
Alsifer 32 $x = 68$									
68.59	31.41	68.75	31.25	81.28	6.01	12.71	68.00	10.00	22.00

EXPERIMENTAL PROCEDURES AND RESULTS

A total of 27 x-ray powder patterns were taken employing cobalt, iron, and chromium K_{α} radiation. All were taken with a 5.73 cm radius Debye-Scherrer camera using Kodak No-Screen x-ray film. All of the patterns were taken at room temperature.

The powdered samples were separated in a 270 mesh (0.053 mm) screen, placed in 0.2 mm capillary tubes, and centered in the camera by means of a microscope. The films were exposed for eight hours for the Fe_3Si - Fe_3Al series and for nine hours for the other patterns. Regardless of which x-ray tube was used, it was operated at 40 kV and 10 mA. All samples were rotated during irradiation and the appropriate filters were used to absorb the K_{β} wavelengths.

All of the diffraction lines on all of the films were read all of the way through twice and their values averaged. From each set of lines straddling the incident and transmitted beam holes a value for the midpoint between the two lines was calculated. These values were then averaged to obtain a median value of the location of the incident and transmitted beams' points of penetration of the film. The distances along the film for each diffraction line were then adjusted to be the distances along the film from the incident beam hole. A factor to convert these distances into θ radians (the angle between the incident and diffracted beams),

corrected for film shrinkage during processing, was calculated by dividing $\pi/2$ radians by the distance between the points of penetration of the incident and transmitted beams.

To compute the lattice parameters, the radians per centimeter and the distance along the film from the incident beam hole for each line, along with their Miller indices, were inputted into a singly-iterative, weighted least-squares computer program written by Dr. F. X. Kayser. The systematic error was represented by the Taylor-Sinclair/Nelson-Riley function.

The lattice parameters for all 27 films are listed in Tables 4, 5, and 6, identified by the film number, alloy composition, and the radiation used. The values listed are corrected for refraction (11) and are believed to be precise to $\pm 0.0002\text{\AA}$.

One of the inputs to the least-squares program used is a weighting factor. This factor was used, in some cases, to put less weight on lines that were harder to read than others due either to their faintness or to their diffuseness. It was also used to selectively choose just those reflections in the back-reflection region with resolved K_{α_1} - K_{α_2} doublets giving six lines in the back-reflection region capable of being read very precisely. The reflection furthest out in the back-reflection region was typically greater than $80^\circ \theta$. For those patterns taken with iron or chromium K_α radiation, all of

the fundamental reflections were weighted. This was because, for iron radiation, only two reflections were able to be resolved, and for chromium radiation, only one. The highest value of θ found for iron and chromium radiations was only about 75° . With this lesser number of lines, it was felt best to include the unresolved fundamental reflections, as well as the resolved, since these were the most intense lines and could thus be read better than the superlattice reflections.

Tables 4, 5, and 6 are denoted to reflect this difference in the lines chosen to calculate the lattice parameters. It is felt that this latitude to choose which lines are to be considered is an important factor in the precisions claimed for these measurements. The slow cooling rates used in the preparation of these alloys is also instrumental. These cooling rates insured that a very high degree of order was present in each specimen upon reaching room temperature. The high purity of the samples and the care taken during the reading of the films are also important factors in the claimed precisions of $\pm 0.0002\overset{\circ}{\text{\AA}}$.

Table 4. Lattice parameters of $\text{Fe}_3\text{Al-Fe}_3\text{Si}$ alloys for Co-K_α , Fe-K_α , and Cr-K_α radiation, corrected for refraction

Film number	Alloy composition	Lattice parameter ($\text{\AA}$)		
		Co-K_α	Fe-K_α^a	Cr-K_α^a
SJC-1	100 wt% Fe_3Si (sample 1a)	-	5.6533	-
SJC-2	100 wt% Fe_3Si (sample 1b)	-	5.6533	-
SJC-10	10 wt% Fe_3Al -90 wt% Fe_3Si	5.6659	-	-
SJC-11	20 wt% Fe_3Al -80 wt% Fe_3Si	5.6803	-	-
SJC-16	20 wt% Fe_3Al -80 wt% Fe_3Si	5.6800	-	-
SJC-9	30 wt% Fe_3Al -70 wt% Fe_3Si	5.6932	-	-
SJC-8	40 wt% Fe_3Al -60 wt% Fe_3Si	5.7079	-	-
SJC-7	50 wt% Fe_3Al -50 wt% Fe_3Si	5.7210	-	-
SJC-6	60 wt% Fe_3Al -40 wt% Fe_3Si	5.7358	-	-
SJC-5	70 wt% Fe_3Al -30 wt% Fe_3Si	5.7502	-	-
SJC-4	80 wt% Fe_3Al -20 wt% Fe_3Si	5.7648	-	-
SJC-3	90 wt% Fe_3Al -10 wt% Fe_3Si	-	5.7786	-
SJC-12	90 wt% Fe_3Al -10 wt% Fe_3Si	5.7792	-	-
SJC-14	90 wt% Fe_3Al -10 wt% Fe_3Si	-	-	5.7784
SJC-13	100 wt% Fe_3Al (sample 1)	-	-	5.7934
SJC-15	100 wt% Fe_3Al (sample 1)	5.7935	-	-
SJC-17	100 wt% Fe_3Al (sample 2)	5.7935	-	-

^aAll fundamental reflections weighted.

Table 5. Lattice parameters of $\text{Fe}_{73}\text{Al}_{27}$ - $\text{Fe}_{73}\text{Si}_{27}$ alloys for Co-K_{α} and Fe-K_{α} radiation, corrected for refraction

Film number	Alloy composition	Lattice parameter (Å)	
		Co-K_{α}	Fe-K_{α} ^a
SJC-20	10 wt% $\text{Fe}_{73}\text{Al}_{27}$ -90 wt% $\text{Fe}_{73}\text{Si}_{27}$	-	5.6569
SJC-27	10 wt% $\text{Fe}_{73}\text{Al}_{27}$ -90 wt% $\text{Fe}_{73}\text{Si}_{27}$	-	5.6571
SJC-28	20 wt% $\text{Fe}_{73}\text{Al}_{27}$ -80 wt% $\text{Fe}_{73}\text{Si}_{27}$	-	5.6705
SJC-22	30 wt% $\text{Fe}_{73}\text{Al}_{27}$ -70 wt% $\text{Fe}_{73}\text{Si}_{27}$	5.6848	-
SJC-25	30 wt% $\text{Fe}_{73}\text{Al}_{27}$ -70 wt% $\text{Fe}_{73}\text{Si}_{27}$	-	5.6847
SJC-23	40 wt% $\text{Fe}_{73}\text{Al}_{27}$ -60 wt% $\text{Fe}_{73}\text{Si}_{27}$	5.6995	-
SJC-26	40 wt% $\text{Fe}_{73}\text{Al}_{27}$ -60 wt% $\text{Fe}_{73}\text{Si}_{27}$	-	5.6997
SJC-24	50 wt% $\text{Fe}_{73}\text{Al}_{27}$ -50 wt% $\text{Fe}_{73}\text{Si}_{27}$	5.7148	-

^aAll fundamental reflections weighted.

Table 6. Lattice parameters for Sendust and Alsifer 32 alloys for Co-K_{α} radiation, corrected for refraction

Film number	Alloy composition	Lattice parameter (Å)
SJC-19	85 wt% Fe, 5.4 wt% Al, 9.6 wt% Si	5.6993
SJC-18	81.3 wt% Fe, 12.7 wt% Al, 6.0 wt% Si	5.7308

DISCUSSION

The lattice parameters for the Fe_3Si - Fe_3Al series are shown graphically in Figure 2. Close investigation of this curve shows that it is not a straight line. This result is contrary to that reported by Niculescu et al., who, on the basis of only four points carried to only the third decimal place, predicted a linear dependence for the lattice parameter versus composition (2).

A least-squares determination of the polynomial function best fitting the data was obtained using a double precision computer program written by Dr. F. X. Kayser. The solution is of the form:

$$a_0 = A + Bx + Cx^2 + Dx^3.$$

Values for A, B, C, and D, with x equal to weight and mole percent Fe_3Al , along with the standard deviations, are listed in Table 7.

Table 7. Coefficients for polynomial fit of Fe_3Si - Fe_3Al data for weight and mole percent Fe_3Al , with standard deviations

x	A	B $\times 10^{-3}$	C $\times 10^{-6}$	D $\times 10^{-9}$	Std. dev. $\times 10^{-4}$
wt % Fe_3Al	5.65319	1.2916	1.8765	-7.5369	3.6544
mole % Fe_3Al	5.65319	1.2842	1.9374	-7.4066	3.6513

Figure 2. The lattice parameters of $\text{Fe}_3\text{Si}_{1-x}\text{Al}_x$ alloys as a function of composition

One possible contribution to the deviation from linearity for the lattice parameter versus weight percent Fe_3Al curve can be found in the phase diagrams of iron-silicon and iron-aluminum. The DO_3 ordered structure for Fe_3Si is believed to persist to the temperature of the solidus, whereas Fe_3Al must go through a disordered region upon cooling from the melt before the transition to the DO_3 ordered structure (12). For Fe_3Si , this results in an alloy with a Bragg-William order parameter very near 1 and virtually free of antiphase boundaries (12). This is not the case with Fe_3Al . Equilibrated Fe_3Al alloys possess numerous antiphase boundaries and is therefore not nearly as perfectly ordered as Fe_3Si (12). The transition from the imperfectly ordered Fe_3Al to the nearly perfectly ordered Fe_3Si could be somewhat responsible for the curvature in Figure 1.

The lattice parameters for the $\text{Fe}_{73}\text{Si}_{27}$ - $\text{Fe}_{73}\text{Al}_{27}$ series, along with the lattice parameter of the Sendust sample, are plotted in Figure 3. The abscissa in Figure 3, weight percent $\text{Fe}_x\text{Al}_{1-x}$, was chosen so that the curve from Figure 2 could also be shown. Here x refers to the iron atom-fraction, therefore Fe_3Al has an $x=0.75$, $\text{Fe}_{73}\text{Al}_{27}$ has an $x=0.73$, and the Sendust sample an $x=0.7374$. It is interesting to note on this figure that Fe_3Al and $\text{Fe}_{73}\text{Al}_{27}$ have, when the $\text{Fe}_{73}\text{Si}_{27}$ - $\text{Fe}_{73}\text{Al}_{27}$ line is extrapolated to 100% $\text{Fe}_{73}\text{Al}_{27}$, essentially the same lattice parameter. As the line moves to the silicon-

Figure 3. The lattice parameters of $\text{Fe}_{73}\text{Si}_x\text{Al}_{27-x}$ and Sendust alloys as a function of composition

rich side, the two lines diverge. This result is in agreement with data published by Taylor and Jones (5) on iron-aluminum alloys and with a value for $\text{Fe}_{73}\text{Si}_{27}$ extrapolated from the data of Farquhar, Lipson, and Weill (3).

The curvature of the line representing the $\text{Fe}_{73}\text{Si}_{27}$ - $\text{Fe}_{73}\text{Al}_{27}$ series is more readily apparent. The polynomial function best fitting the data (10 weight percent $\text{Fe}_{73}\text{Al}_{27}$ to 50 weight percent $\text{Fe}_{73}\text{Al}_{27}$), determined in the same way as for the Fe_3Si - Fe_3Al data is of the form:

$$a_0 = A' + B'x + C'x^2 + D'x^3.$$

Table 8 lists A' , B' , C' , and D' for x equal to weight and mole percent $\text{Fe}_{73}\text{Al}_{27}$ plus their standard deviations.

Table 8. Coefficients for polynomial fit of $\text{Fe}_{73}\text{Si}_{27}$ - $\text{Fe}_{73}\text{Al}_{27}$ data for weight and mole percent $\text{Fe}_{73}\text{Al}_{27}$, with standard deviations

x	A'	B' 10^{-3}	C' 10^{-6}	D' 10^{-8}	Std. dev. 10^{-5}
wt % $\text{Fe}_{73}\text{Al}_{27}$	5.64462	1.1964	5.3929	-2.5000	1.1952
mole % $\text{Fe}_{73}\text{Al}_{27}$	5.64460	1.1915	5.3183	-7.8608	1.3704

The lattice parameter of the Alsifer 32 alloy, although listed in Table 6, is not plotted in Figure 3. It was left off of Figure 3 for the sake of clarity, since it contains 5 atomic percent less iron than the other alloys plotted. It is interesting to note, however, that, contrary to results

obtained by Suwalski et al., (7), the Alsifer 32 ($\text{Fe}_{68}\text{Si}_{10}\text{Al}_{22}$) alloy used in this investigation was ferromagnetic, both the homogenized finger and the powder.

The results of this investigation have been the establishment of reliable and reproducible lattice parameters, in the fourth decimal place, for a number of iron-rich, iron-aluminum-silicon alloys. The alloys that were chosen for this work are known for their magnetic properties and bracket the alloy that seems to have the best soft magnetic properties, the Sendust alloy. In anticipation of further research in this area, it is now possible to confirm an alloy's composition from its lattice parameter.

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APPENDIX

The zero-field, adiabatic elastic constants of a single crystal of Fe_3Al were taken at 77.4°K. As was mentioned in the Introduction, this was undertaken to check the results of Leamy, Gibson, and Kayser (10), previously obtained in this laboratory. These measurements were also performed to see whether a signal could be passed through the ferromagnetic domain walls in Fe_3Al single crystals without the presence of an external magnetic field.

It was decided to measure the elastic constants at liquid nitrogen temperature for two reasons. The first because the bond used, Fisher Nonaq stopcock grease, works very well at liquid nitrogen temperature and is easy to apply and remove and, secondly, because temperature control is very easy with liquid nitrogen, thus eliminating the need for a thermocouple.

As mentioned earlier, the crystal used in the previous investigation was of lesser quality than is now capable of being grown. The Fe_3Al single crystal used in the present investigation was produced by arc-melting calculated weights of arc-melted electrolytic iron and the iron plus 17 weight percent aluminum master alloy. These were first arc-melted in the form of a button and then flipped over and remelted in the form of a finger. The finger was cut in half and placed in a one inch outer diameter by two inches high, flat-bottomed, stabilized-zirconia crucible and, under partial

pressure of helium, grown by the Bridgman method. After growth, slices of approximately 1/8 inch thick were spark cut from the top and bottom in order to eliminate the grown in concentration gradients. The cut faces were ground and polished to avoid contamination. Subsequent metallographic examination revealed that it was a single crystal but that it contained a network of low-angle boundaries.

The crystal was heat-treated at 1040°C in air for five days and then aligned by means of the back-reflection Laue method. Two pairs of {110} faces and one pair of (100) faces were cut into the crystal by means of a spark cutter. Again, the cut faces were ground and polished in order to avoid contamination.

The resulting parallelepiped was electropolished and placed on a Picker x-ray diffractometer in order to try to determine how close the cut planes were to the true crystallographic planes. The crystal was placed in the diffractometer such that the plane being investigated was nearly parallel to the incoming beam slit. The sample was then rotated independently of the x-ray tube and detector until a maximum was observed. The location of this maximum corresponded to where the beam was parallel to the crystal face, i.e. where the normal to the cut plane was perpendicular to the beam. A 2θ scan was then performed and the diffractometer was centered on a major reflection (e.g. the (220)

reflection off of a (110) face). The sample was then given an omega scan until a maximum for the particular reflection was obtained. This maximum corresponded to the alignment of the normal of the crystallographic plane perpendicular to the x-ray beam. Thus the angular difference between the normal to the cut face and the normal to the crystallographic face measures the misalignment of the crystal.

The misalignment for two of the faces, one (110) and the (100), was found to be less than $3/4^\circ$, a value well within the mosaic spread of the crystal, which was on the order of 1° . The third face, the $(\bar{1}10)$, was found to be about $1\ 1/2^\circ$ misaligned. The crystal was then ground to bring the faces into perpendicularity. Subsequent measurements on a microscope showed that the faces were perpendicular to within $1'$. The face that was $1\ 1/2^\circ$ misaligned was brought much closer, probably on the order of the other two faces.

An ordering anneal at 575°C for three hours with the temperature reduced 25° every 24 hours after that until room temperature was reached was then performed.

The elastic constants of this Fe_3Al single crystal were measured using acoustical techniques. The transit times for ultrasonic waves propagated from the transducer-containing face to the opposite face and back were measured by the pulse-echo-overlap method (13,14).

X cut and Y cut, ten-MHz quartz transducers were bonded

to the (110) and the ($\bar{1}10$) faces of the specimen. If t_L is the transit time, through the crystal and back, for a longitudinal wave, t' the transit time for a shear wave polarized in the $[\bar{1}10]$ direction, and t for a shear wave polarized in the [001] direction, then the corresponding velocities are

$$V_L = 2w/t_L$$

$$V' = 2w/t'$$

$$V = 2w/t,$$

where w is the thickness of the crystal in the direction of wave propagation.

The corresponding adiabatic elastic constants are then

$$C_L = pV_L^2 = \frac{1}{2}(c_{11} + c_{12} + 2c_{44})$$

$$C' = pV'^2 = \frac{1}{2}(c_{11} - c_{12})$$

$$C = pV^2 = c_{44},$$

where p is the theoretical density and c_{11} , c_{12} , and c_{44} are the elastic constants of a crystal with cubic symmetry. The results of this investigation are shown in Table A-1.

The elastic constants listed above were calculated using a value for the density of 6.6439 gm/cm^3 , obtained by using a value of $5.7935\text{\AA}$ for the lattice parameter of Fe_3Al .

The elastic constants obtained are still tentative because of the high attenuation encountered for the C and C_L modes. It has been shown that a signal can be transmitted through the sample without an external magnetic field present

Table A-1. The adiabatic elastic constants of Fe_3Al from the (110) and $(\bar{1}10)$ faces for the present work and for that of Leamy, Gibson, and Kayser (10) at 77.4°K in units of 10^{12} dyn/cm²

(110) face	w^a (cm)	t (μsec)	V	C	C (ref. 10)
C_L	1.334	4.2^b	0.6	2.7	2.922
C'	1.334	14.195	0.188	0.235	0.237
C	1.334	-	-	-	1.377
$(\bar{1}10)$ face	w^a (cm)	t (μsec)	V	C	C (ref. 10)
C_L	0.987	3.2^b	0.6	2.5	2.922
C'	0.987	10.537	0.187	0.233	0.237
C	0.987	-	-	-	1.377

^aAt 20°C.

^bRead off of the oscilloscope screen.

for the C_L mode, but the attenuation is so high that the second echo is barely discernible. Therefore, the transit time for the C_L mode was simply read off of the oscilloscope screen. The attenuation was even higher in the C mode, making it impossible to propagate a signal through the crystal. Therefore, no meaningful value of C (and consequently c_{44}) could be obtained. Attenuation was low in the C' mode and a good value was obtained. This value agrees fairly well with that obtained by Leamy, Gibson, and Kayser (10).