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**SPECIFIC HEAT AND ELECTRICAL RESISTIVITY
OF FeAl ALLOYS**

**A Dissertation
Presented for the
Doctor of Philosophy
Degree
The University of Tennessee, Knoxville**

**Michael Delos Kass
August 1998**

Dedication

**This thesis is dedicated
to the memory of my son
Gregory Daniel Kass
and to the memory of my brother
Merrill Paul Kass**

Acknowledgments

There are many people to whom I am grateful for making this dissertation project very rewarding. Several of the Materials Science and Engineering faculty and staff were of great help and I would like to acknowledge Greg Jones and Doug Falcon for their assistance with the pulse-heating calorimeter. Deb Basak was instrumental in training me on the pulse-heating calorimeter. He also provided key help in understanding the computer programs used to operate the system and process the data.

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Abstract

The specific heat C_p and electrical resistivity ρ of four iron-aluminum alloys (30, 38, 43, and 48 at.% Al) were measured as a function of temperature and composition. The alloys were step-cooled from 1000°C to minimize vacancy concentration and induce maximum order. At room temperature the 30 at.% Al alloy was in the Fe_3Al phase region (D0_3 structure) while the other three were in the FeAl phase region (B2 structure). The resistivity at 25°C increased with increasing disorder (increasing Al content for Fe_3Al and increasing Fe content for FeAl), reaching a maximum value of $\sim 165 \mu\Omega\text{-cm}$ at the Fe_3Al -FeAl phase boundary.

A pulse-heating calorimeter was used to measure simultaneously C_p and ρ from 25 to approximately 1000°C. The samples were initially heated at 73°C/s (except for the 43 at.% Al which was heated at 90°C/s), then cooled back to 25°C at 1.5°C/s. They were then repulsed to examine the effect of the pulse treatment and of heating rate. For the 30 at.% Al alloy, ρ increased to a maximum at 485°C, at which a slight decrease occurred. This was taken as due to the D0_3 to B2 phase transformation. Then in the FeAl region, ρ decreased slightly to the upper temperature of measurement ($\sim 1200^\circ\text{C}$). The slope of the ρ -T curves decreased with decreasing Al content, becoming negative for the 30 at.% Al alloy in the FeAl region. The relatively high resistivities and the tendency for the ρ -T

curves to approximate a common value at high temperatures is similar to the saturation behavior of some amorphous and disorder transition alloys discovered by Mooij.

For the 30, 38 and 43 at.% Al alloys, the ρ -T curves were identical for the initial slowly cooled condition and after cooling from high temperature at 1.5°C/s. The curve for the 48 at.% Al alloy was about 30% higher after cooling at 1.5°C/s from high temperature. The ρ -T curves were the same for the heating rates that were used in this study.

The specific heat was determined for the alloys, and it showed a dramatic increase at high temperatures. The C_p -T curves were the same for the initial slowly cooled condition and for pulse treatment. For the 43 and 48 at.% Al alloys the C_p -T curves were independent of heating rate for the rates tested, but for the 38 at.% Al alloy, the temperature corresponding to the dramatic rise in C_p shifted to higher temperature with higher heating rate.

The C_p -T curves in the lower temperature linear region were extrapolated to higher temperature, and subtracted from the experimental C_p curve to determine the enthalpy of formation of defects responsible for the dramatic increase in C_p . The enthalpy of formation decreased from 135 kJ/mol at 30 at.% Al to 95 kJ/mol at 48 at.% Al. The vacancy concentrations n_v were calculated from these values by assuming triple defect formation. At 900°C n_v was constant (<0.2 at.%) from 30 to 38 at.% Al but increased

to ~ 1.2 at.% for 48 at.% Al. This is in agreement with the results from other experimental studies and the Chang-Neumann model.

For the 38 at.% Al alloy, C_p was affected by the heating rate but ρ was not. Therefore, it was determined that the β sublattice dominates the resistivity since vacancies form primarily on the α sublattice through the formation of triple defects.

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Nomenclature

a	constant
C	specific heat
c	heat capacity for an arbitrary mass
C_P	specific heat at constant pressure
C_V	specific heat at constant volume
C_P^+	dilation, electronic and lowest-order anharmonic contributions to specific heat
D	density
d	diameter
E	voltage
E_{KE}	voltage drop across the knife edges
E_S	voltage drop across the specimen
E_{SR}	voltage drop across a standard resistor
H	enthalpy
h	Plank's constant
I	current
I_S	specimen current
k_B	Boltzmann constant
L	length between voltage taps
L_{KE}	length between knife edges
L_T	total length of specimen

m	mass
m_s	specimen mass between voltage taps
m_T	total mass of specimen
N	Avagadro's number
N_V	number of vacancies
n_A^β	concentration of antisite A atoms on the β sublattice
n_V^α	concentration of vacancies on the α sublattice
n	sampling size
n_D	triple defect concentration
n_V	vacancy concentration
P	pressure
R	universal gas constant
S_e	standard error of estimate
S_f^V	entropy of vacancy formation
T	temperature
TCR	temperature coefficient of resistivity
T_D	temperature at which defects begin to form
V	volume
X	deviation from stoichiometry
x_i	independent variable
y_i	observed variable
z	concentration of triple defects

α_D	disorder parameter
ν	harmonic frequency
ν_D	cut off frequency
θ_D	Debye temperature
ρ	electrical resistivity
ρ_C	resistivity with no vacancies present
ρ_V	resistivity arising from a single vacancy
ρ_0	residual resistivity
P	power
ΔC_P^D	defect contribution to specific heat
ΔC_P^V	vacancy contribution to specific heat
ΔH_f^D	enthalpy of triple defect formation
ΔH_f^M	enthalpy of vacancy mobility
ΔH_f^V	enthalpy of vacancy formation
ΔQ	quantity of heat added to raise the temperature of an arbitrary mass by ΔT
dq	quantity of heat added to raise the temperature of a unit mass by dT

Abbreviations

bcc	body-centered cubic
emf	electromotive force
fcc	face-centered cubic
NIST	National Institute of Standards and Technology

Chapter 1

Introduction

The iron-aluminum intermetallic compounds Fe_3Al and FeAl are currently being investigated as high temperature materials since they exhibit excellent oxidation resistance and strength. The structure-property relationships for these materials are not well understood, but are known to be related to the compositional defect structure and the formation of thermal vacancies. The crystal structures of Fe_3Al and FeAl are designated D0_3 and B2 respectively and are based on a body-centered cubic lattice (bcc). Vacancies and antisite atoms are created in the FeAl B2 structure as a result of triple defect formation, the kinetics of which are not understood. Since the mechanical properties of these alloys are known to be influenced by the presence of point defects, such as vacancies, it is desirable and informative to have a knowledge of the compositional dependencies of the vacancy concentrations of FeAl and Fe_3Al as a function of temperature.

Rationale

The study of specific heat has contributed greatly to the knowledge of properties of materials. The atomic and molecular phenomena that occur in a solid during heating contribute to the specific heat. These include vibrational and rotational movements of atoms, excitation of electrons, magnetic effects, and the formation of lattice defects.

Magnetic and structural transformations are usually manifested as a discontinuity or abnormality in the specific heat versus temperature data. However, the observed specific heat includes contributions from all these sources and separation of the individual contributions can be difficult. The formation of lattice defects such as vacancies is believed to provide a significantly larger contribution to the specific heat than the dilation, electronic, or lowest-order anharmonic sources. This lattice defect contribution is believed responsible for the dramatic increase in specific heat that occurs at temperatures below melting for some refractory metals and alloys, including FeAl. There exist several techniques to separate the lattice defect formation to the specific heat. One of these is representing the dilation, electronic and lowest-order anharmonic contributions by baseline extrapolation. The difference in the observed specific heat and the baseline with temperature allows 1) determination of the temperature of defect formation, 2) derivation of the enthalpy of defect formation, and 3) the thermal defect concentration at a given temperature.

Another method of evaluating the structures of conducting materials is to measure the electrical resistivity as a function of temperature. Lattice defects scatter electrons thereby increasing the resistivity. This increase is normally proportional to the defect concentration. Therefore, resistivity measurements are a common method of evaluating the kinetics of defects and also their concentration.

Statement of the Problem

Preliminary specific heat and electrical resistivity investigations of several FeAl alloys showed that the ρ -T behavior did not correspond to the dramatic upswing in C_p observed at higher temperature. This upswing is believed to be caused by thermal defect or vacancy formation. In addition, the kinetics associated with vacancy/defect formation in FeAl are not understood. The literature contains few references concerning the kinetics associated with defect formation for these alloys. Studies involving positron lifetime measurements and Mössbauer spectroscopy all suggest that the vacancy diffusivity is slow at low temperatures (eg., 400°C) and very rapid at elevated temperatures (eg., 800°C).

It is important to note that the enthalpy of formation values reported in the literature for FeAl alloys represent the enthalpy associated with the vacancy generation only. They do not represent the enthalpy associated with the formation of the triple defect, which includes antisite atoms in addition to vacancies. As a result, the enthalpies of formation reported in the literature do not include the enthalpy contribution associated with antisite defects.

An interesting feature of Fe₃Al alloys are their unusual ρ -T behavior. The ρ -T curve exhibits a maximum at temperatures just below the D0₃ to B2 transition. One group of researchers attributed this maximum to the Curie temperature, while others believe that it is caused by a semiconductor effect in which additional electrons are excited into a conduction band.

Objectives of the Study

One of the advantages of pulse-heating calorimetry is that the high heating rate allows measurements to be made at high temperatures. As the length of time at high temperature increases, the materials composing the calorimeter begin to degrade. Because of rapid heating, the length of time at high temperature is too low to seriously degrade the calorimeter materials. In addition, the resistivity and specific heat data are determined simultaneously. This allows a direct comparison of the electrical and thermal behavior with temperature and because of the rapid heating, these measurements can be made on metastable states. The purpose of this study was to investigate thermal defect formation, structural kinetics, and compositional effects of Fe_3Al and FeAl by measuring specific heat and electrical resistivity as a function of temperature for one Fe_3Al alloy and three FeAl alloys. The results have provided insight into 1) the formation of thermal defects/vacancies, 2) the structural kinetics associated with the defect formation, and 3) the compositional effects associated with thermal defect formation for the FeAl phase.

Chapter 2

Theory of Specific Heat and Electrical Resistivity

Specific Heat

Definition of Specific Heat

According to a standard textbook (1), the heat capacity is the quantity of heat required to increase the temperature of a system or substance one degree of temperature. The molar heat capacity is the quantity of heat necessary to raise the temperature of one molecular weight of the substance one degree. The specific heat is sometimes defined as the ratio of the heat capacity to that of water at 15°C. However, in this dissertation specific heat will be defined as the heat capacity per unit mass in keeping with common usage. The heat capacity of a system of arbitrary mass is defined as

$$c \equiv \lim_{\Delta T \rightarrow 0} \left(\frac{\Delta Q}{\Delta T} \right) \quad (2-1)$$

where ΔQ is the quantity of heat added to raise the temperature by ΔT . Both c and ΔQ are associated with arbitrary mass. This relation is divided by the mass to obtain the expression for specific heat that is independent of mass

$$C \equiv \frac{c}{m} = \frac{dq}{dT} \quad (2-2)$$

where dq is the quantity of heat required to raise the temperature of a unit mass of the substance by an amount dT . In general dq will depend on the temperature of the system and other physical changes taking place in the substance during the temperature rise.

The physical properties of a thermodynamic system are interrelated by an equation of state, which involves the volume V , temperature T , and the pressure P . Since one of these variables can be expressed in terms of the other two, only two variables can have arbitrary values at the same time. Therefore as T is increased, only P or V can be kept constant. As a result, the specific heat is defined at constant pressure (C_P) and also at constant volume (C_V) by

$$C_P \equiv \left(\frac{dq}{dT} \right)_P \quad (2-3)$$

$$C_V \equiv \left(\frac{dq}{dT} \right)_V \quad (2-4)$$

Because it is very difficult to measure C_V directly on solid substances, C_P is the quantity that is normally determined by experimentation (2). At constant pressure, the change in enthalpy (dH) is equal to dQ , therefore

$$C_P \equiv \left(\frac{\partial H}{\partial T} \right)_P \quad (2-5)$$

Experimentally, C_P is determined by measuring the temperature and heat input to the specimen or by measuring the change in enthalpy of the specimen with changing temperature. The SI units for specific heat are J/g-K.

Historical Background

The classical theory of specific heat states that it is independent of temperature for a solid material (1, 2). The molar heat capacity was determined by Dulong and Petit in 1819 as

$$C_V \approx 3R \quad (2-6)$$

where R is the universal gas constant (8.3143 J/mol-K). This value is in good agreement with experimental values of most solids at room temperature, with the exception of boron, carbon, and silicon. The biggest difficulty with the classical theory is that it violates the third law of thermodynamics that the specific heat must tend to zero as the absolute temperature is approached.

With the advent of quantum theory, Einstein formulated a heat capacity equation by considering each atom in a solid as an independent harmonic oscillator. Each atom has the same fundamental frequency ν and is related to the specific heat at constant volume by

$$C_V \approx 3Nk_B \left(\frac{h\nu}{k_B T} \right)^2 e^{-h\nu/k_B T} \quad (T \rightarrow 0) \quad (2-7)$$

where N is Avagadro's number (6.022×10^{23} /mole), k_B is the Boltzmann constant (1.381×10^{-23} J/K), and h is the Planck constant (6.626×10^{-34} J s). In Einstein's model the specific heat approaches zero as $T \rightarrow 0$ and has a limiting value of $3R$ as $T \rightarrow \infty$ in accordance with the classical theory. However, subsequent experiments showed that

while the specific heat of solids decreases with temperature it does not decrease as fast as predicted by the Einstein model (1, 2).

A more sophisticated model was developed by Debye who considered that the atoms in a solid vibrate at a range of frequencies rather than at a single value. These range of frequencies are characterized by a distribution function and are cut off at a particular frequency ν_D , which is determined by the properties of the solid. According to the Debye model, the specific heat at constant volume is

$$C_V = 9Nk_B \left(\frac{T}{\theta_D} \right)^3 \int_0^{x_D} \frac{x^4 e^x}{(e^x - 1)^2} dx \quad (2-8)$$

where $x = h\nu/k_B T$, $x_D = h\nu_D/k_B T$, and θ_D is the Debye temperature defined by $h\nu_D/k_B$.

The Debye model has been found to fit the experimental low temperature specific heat better than the Einstein model (1, 2). At temperatures much less than the Debye temperature, the equation reduces to

$$C_V = \frac{12}{5} \pi^4 Nk_B \left(\frac{T}{\theta_D} \right)^3 \quad (2-9)$$

The Debye model shows that the specific heat approaches zero as T^3 in accordance with experimental observations.

Contribution of Lattice Defects

In many solids, especially refractory metals, a rapid rise in specific heat occurs before melting. This rapid rise has been attributed to lattice vacancies, since the vacancy concentration n_v increases rapidly according to

$$n_v = A \exp\left(-\Delta H_f^v / k_B T\right) \quad (2-10)$$

where ΔH_f^v is the enthalpy of vacancy formation, and A is equal to $\exp(S_f^v/k_B)$, where S_f^v is the entropy associated with vacancy formation. The vacancy contribution to specific heat ΔC_P^v can be determined from the increase in internal energy arising from vacancy formation and is given by

$$\Delta C_P^v = n_v R \left(\frac{\Delta H_f^v}{RT} \right)^2 \quad (2-11)$$

According to this equation, n_v can be determined from the experimental values of ΔC_P^v and ΔH_f^v . However it is not easy to separate the ΔC_P^v contributions from the measured value of C_P . In particular, it is especially difficult to establish the dilation, electronic and lowest-order anharmonic contributions (2).

One potentially useful technique to establish ΔC_P^v is to represent the dilation, electronic, and lowest-order anharmonic contributions (C_P^+) as a linear expression which can be extrapolated to high temperature as shown in Fig. 2-1 (2). The additional specific heat associated with the rapid rise is then attributed to lattice vacancies. The reliability of this method depends on validity of representing C_P^+ as a linear approximation. It is not

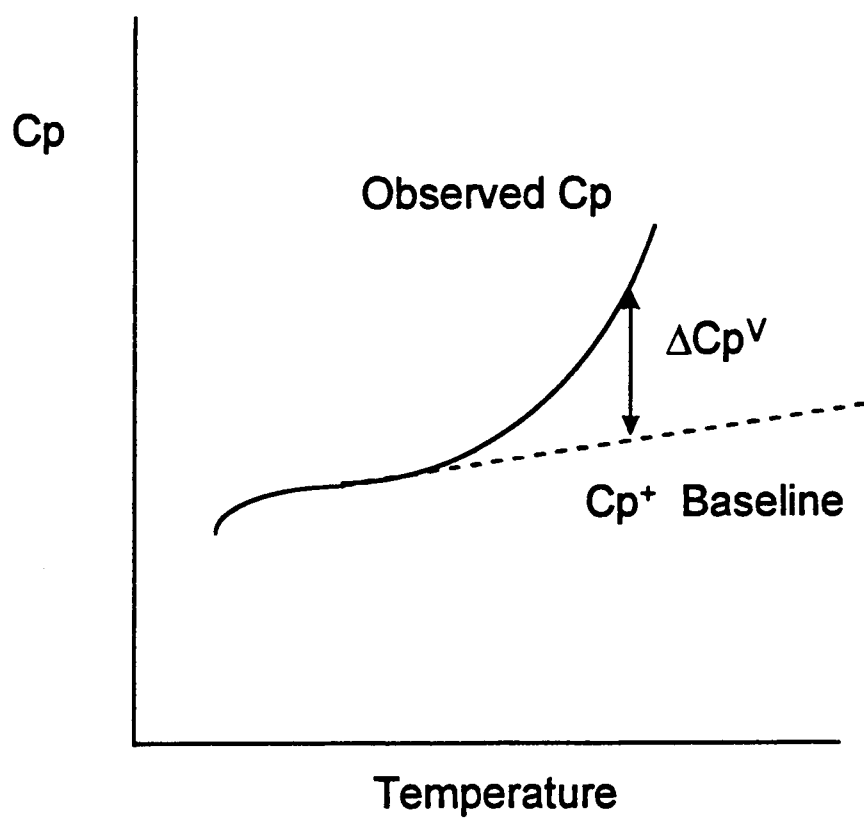


Fig. 2-1. Baseline extrapolation from the observed specific heat.

expected that C_P^+ will vary from the linear approximation by more than a few percent which is relatively insignificant when compared to the exceptionally large rise in C_P . For FeAl the formation of vacancies is accompanied by an antisite Fe atom in accordance with triple defect formation (discussed in a later section). Therefore (for FeAl), ΔC_P^V , ΔH_f^V , and n_V actually reflect the entire triple defect contribution rather than just the vacancy contribution and are labeled as ΔC_P^D , ΔH_f^D , and n_D in order to distinguish these terms from the actual vacancy contribution.

Electrical Resistivity

Definition of Electrical Resistivity

Electrical resistivity is defined as a measure of the extent to which electrons are deviated or scattered from a straight line path as they move under an external electric field. The electrical resistivity originates from perturbations in the movement of electrons produced by the presence of crystal imperfections. The contributions to electrical resistivity, therefore, come from electron-matter interactions that reduce the parallel flow of electrons. For most metals at room temperature, the resistivity mainly depends on the interaction of electrons with phonons. Phonons are the energy quantum associated with atom vibrational movement. At room temperature, the amplitude of the vibrating atoms is on the order of 5 to 10 % of the interatomic distance. Point defects such as vacancies, interstitial atoms or substitutional impurities are lattice features that contribute to the

electrical resistivity of a material. Other contributions come from dislocations and lattice strain. The contributions from each of these effects are considered additive and contribute to the total resistivity of a substance. The primary contributions to resistivity are phonons, chemical impurities, structural imperfections, and lattice strain. The contributions from chemical impurities are considered to be independent of temperature. Lattice strain (arising from cold working, etc.) is removed by annealing, and thus is not a temperature dependent contribution.

Temperature Dependence of Resistivity

The other two contributions (phonons and structural imperfections) are temperature dependent. Experiments and theory have shown that the phonon contribution reduces to zero as the temperature approaches absolute zero. Between 0 and $\sim 400^{\circ}\text{C}$, the phonon contribution predominates. In this temperature range, the resistivity increases linearly with temperature and is approximated by

$$\rho(T) = \rho_0 [1 + (\text{TCR}) T] \quad (2-12)$$

where ρ_0 is the residual resistivity, TCR is the temperature coefficient of resistivity, and T is the temperature. The residual resistivity is the resistivity measured at 0°C , but often is represented as the room temperature resistivity. The temperature coefficient of resistivity is on the order of $4 \times 10^{-3}/\text{K}$ in pure metals, and is a little less for alloys.

At higher temperatures vacancies begin to form in the lattice and, for ordered alloys, the structure may become disordered. The vacancy contribution predominates at

temperatures approaching melting. The increase in resistivity with temperature is considered to be proportional to the vacancy concentration. Therefore, the change in electrical resistivity can be used to evaluate the formation of thermal vacancies in a substance.

As shown previously in equation (2-10), the vacancy concentration increases rapidly with temperature. Since defects such as vacancies are typically present in low concentrations, it is assumed that the properties associated with defects (such as vacancies) are additive in proportion to their concentration. That is to say, the electrical resistivity effect of two vacancies in a given crystal is twice that of a single vacancy. Therefore, the electrical resistivity of a crystalline material is given by

$$\rho = \rho_c + N_v \rho_v \quad (2-13)$$

where ρ_c is the resistivity with no vacancies present and N_v is the number of vacancies. ρ_v is the contribution arising from a single vacancy and is assumed to be constant. This rearranges to become

$$N_v = (\rho - \rho_c) / \rho_v = \Delta\rho / \rho_v \quad (2-14)$$

The vacancy concentration n_v is the number of vacancies divided by the total number of lattice sites. The vacancy concentration can be related to $\Delta\rho$ by

$$a n_v = \Delta\rho \quad (2-15)$$

where a is a constant. The variation in n_v with temperature can now be determined from the above relation. However, the result cannot be quantified unless the value of the

constant a is known. Measurement of $\Delta\rho$ as a function of quench temperature is a common method of evaluating vacancy/defect formation in conducting materials. The technique is often referred to as resistometry.

Chapter 3

Review of Literature

Iron-Aluminum Phase Diagram

The iron-aluminum phase diagram has been intensely studied since the 1930's when it was discovered that two long-range order-phases, Fe_3Al and FeAl , occurred within the iron-rich segment of the equilibrium phase diagram. A recent equilibrium phase diagram of the iron-aluminum system is shown in Fig. 3-1 (4). In spite of numerous studies of Fe_3Al and FeAl , there is still disagreement as to the exact position of the boundary separating these two phases. One of the peculiarities of this system is the lack of a two-phase region between FeAl and αFe . A two-phase region is also missing between FeAl and Fe_3Al . It is important to note, however, that Fe_3Al and FeAl have very similar atomic structures. This is discussed in detail in a later section.

Although there are a number of phases present in the iron-aluminum system, only three are in the compositional range of interest for this proposal. They are (as listed in the diagram):

1. αFe - a disordered solid solution of aluminum in the body centered cubic (bcc) iron structure,

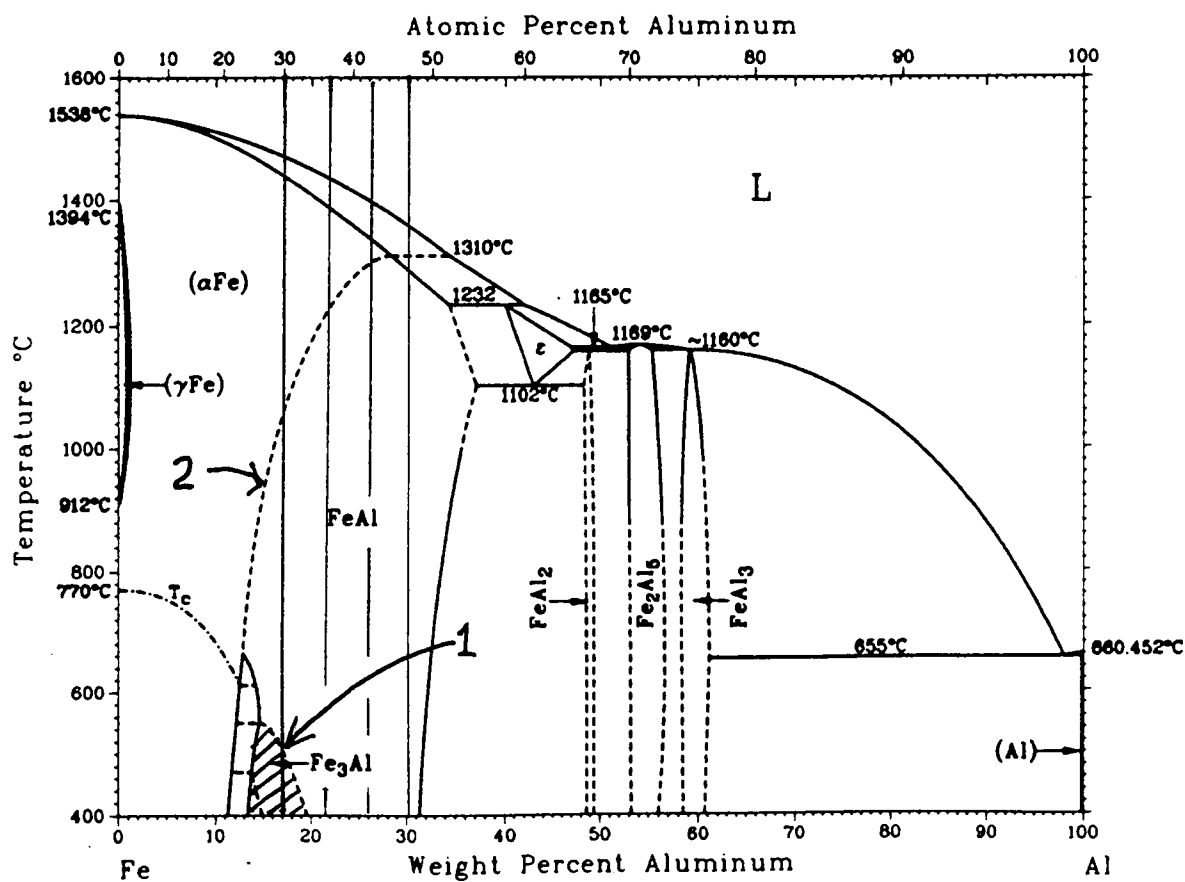


Fig. 3-1. Equilibrium phase diagram of the iron-aluminum system (4). The four vertical lines represent the alloy compositions investigated in this study.

2. Fe_3Al - an ordered DO_3 structure, and
3. FeAl - an ordered B2 structure, often denoted as α_2 .

Aluminum is soluble in iron up to 46 at.% Al, and in this range forms the superlattices Fe_3Al and FeAl as shown in the phase diagram. The Fe_3Al phase region is crosshatched for clarification. Fe_3Al undergoes a transformation to FeAl according to the boundary (labeled 1 in Fig. 3-1) separating the two regions. At even higher temperatures, FeAl transforms into the αFe solid solution according to the phase boundary (labeled 2 in Fig. 3-1). For example, an alloy containing 30 at.% Al (as shown in the diagram) will transform from the Fe_3Al phase to the FeAl at 485°C under slow heating. The FeAl phase will further transform to αFe around 1050°C .

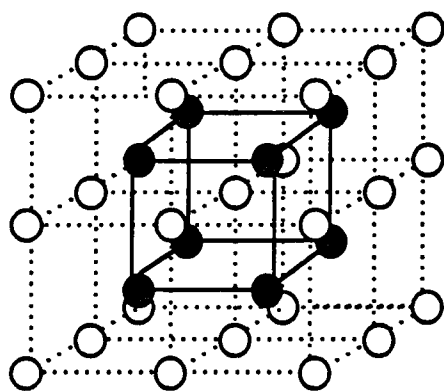
FeAl , which is our primary phase of interest, occurs over a wide range of solubility as shown in the phase diagram. This phase ranges from a minimum solubility at 22 at.% Al and 650°C to a maximum of 55 at.% Al at 1102°C . Because of this wide range of solubility, the formation of constitutional defects is especially important in determining the mechanical and physical properties.

The Curie temperature represents the boundary at which the alloy changes from magnetic to nonmagnetic as a function of composition and is labeled T_C in the Fig. 3-1. As shown in the diagram, the Curie temperature for αFe decreases slowly with the

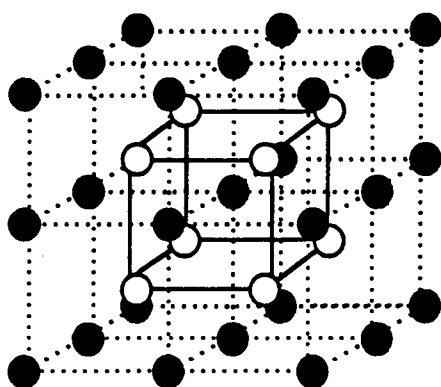
addition of aluminum to $\sim 500^{\circ}\text{C}$ near the Fe_3Al phase boundary and further additions cause further decrease (5).

Crystal Structures of Fe_3Al and FeAl

Both Fe_3Al and FeAl have ordered bcc crystal structures. The Bravias lattice is simple cubic consisting of two interpenetrating simple cubic sublattices with two atoms per unit cell. The ideal stoichiometric structures for Fe_3Al and FeAl are shown in Fig. 3-2 along with lattice site labeling for discussion. At stoichiometry (25 at.% Al) the ideal crystal of Fe_3Al (DO_3 structure) has the α sublattice occupied by the Fe atoms. The β sublattice has Al atoms located at alternate sites (labeled β_1 in the diagram), while the other sites (β_2) are occupied by the iron atoms. Bradley and Jay (6) studied the atomic positions of Fe and Al atoms for Fe_3Al between 25 and 37 at.% Al. They showed that with increasing Al content (up to 37 at.% Al) excess Al atoms begin to occupy the β_2 sublattice positions with Al atoms continuing to occupy the β_1 positions. However, once the structure becomes B2 (around 37 at.% Al), Al atoms will leave the β_2 positions to form a random arrangement of Al atoms on the β_1 and β_2 sites. Therefore, the B2 structure can be viewed as ordered on the α sublattice and disordered on the β sublattice.

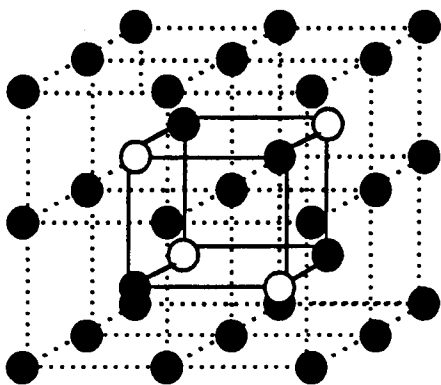


α sublattice: dashed lines
 β sublattice: solid lines
 β_1 sites: black atoms
 β_2 sites: crosshatched atoms



FeAl: B2 structure
 at stoichiometry

Fe atoms: black
 Al atoms: white



Fe_3Al : D0₃ structure
 at stoichiometry

Fe atoms: black
 Al atoms: white

Fig. 3-2. Diagram of the B2 and D0₃ crystal structures.

The crystal structure of FeAl has the Strukturbericht designation of B2, but is also known as the CsCl or L2₀ structure. This atomic arrangement is similar to the D0₃ structure in that only Fe atoms occupy the α sublattice sites. At the stoichiometric composition (50 at.% Al), all of the β sublattice sites are occupied by Al atoms.

However, with increasing Fe content, the arrangement of Al and Fe atoms at the β_1 and β_2 positions is random. This structure typically forms between elements of Group VIIIB (in our case Fe) and the elements of Group IIIA (in our case Al).

Structure Effects on the Hardness and Strength

It is well known that point defects, such as vacancies, have a significant impact on the mechanical properties of metallic materials (7). This is especially true for strongly-ordered intermetallics which remain ordered at high temperature (e.g., 1100°C).

Stoichiometric Fe₃Al has a D0₃ structure in which each lattice site is occupied by a specific atomic species. This highly ordered structure is responsible for the improved mechanical properties of Fe₃Al (8). Above ~530°C Fe₃Al transforms to the FeAl phase which has the B2 structure. This structure is very similar to the D0₃ structure except that the Fe and Al atoms occupy the β sublattice sites at random. The B2 structure is more ductile than the highly ordered D0₃ structure and is desirable for applications requiring toughness (9).

Most strongly-ordered alloys having the B2 structure exhibit a minimum in hardness and strength near the stoichiometric composition. In contrast, the hardness of FeAl was shown to increase monotonically with increasing aluminum content (9-14). Since Nagpal and Baker (10) showed that the thermal vacancy concentration also increases with Al content for FeAl, this behavior was attributed to the presence of vacancies. In fact, Chang et al. (14) showed that the hardness was proportional to the square root of the vacancy concentration. These excess vacancies enhance the hardness and yield stress of FeAl alloys, but also reduce the ductility and toughness as well (9-14).

Lattice Parameter Measurements and Defect Structure of FeAl

In 1932, Bradley and Jay (6) published the results of a detailed X-ray diffraction study of the iron-rich region (0 to 50 at.% Al) of the iron-aluminum system. This included a systematic investigation of the lattice parameter of annealed and quenched alloys measured at room temperature. This composition range includes what we now know as the Fe₃Al and FeAl phases. From 35 to 50 at.% Al (FeAl phase), Bradley and Jay found that the lattice spacing increased with Al content to a maximum at 50 at.% and then decreased with further increases in the Al concentration. Subsequent investigations (13, 15, 16) have confirmed this lattice spacing relationship with composition, those of Ho and Dodd (15) are shown in Fig. 3-3. This lattice parameter spacing behavior was seen by Chang and Neumann (17) and also by Ho and Dodd (15) as evidence that triple defects were formed in FeAl alloys.

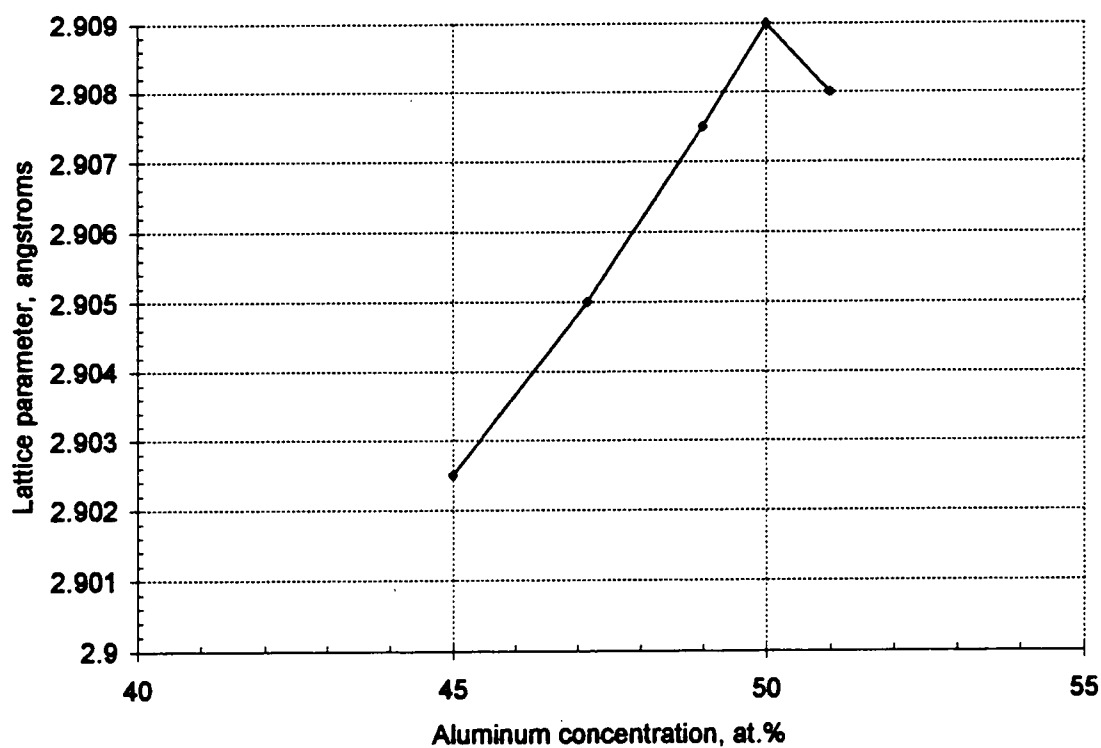
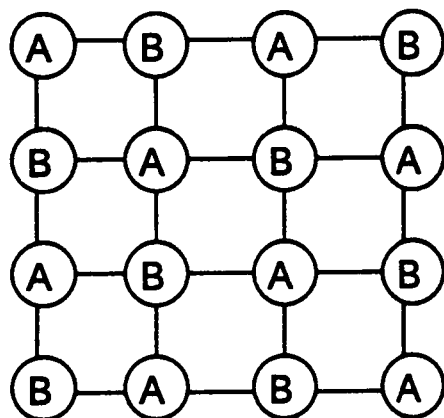


Fig. 3-3. Lattice parameters of FeAl at 25°C measured by Ho and Dodd (15).

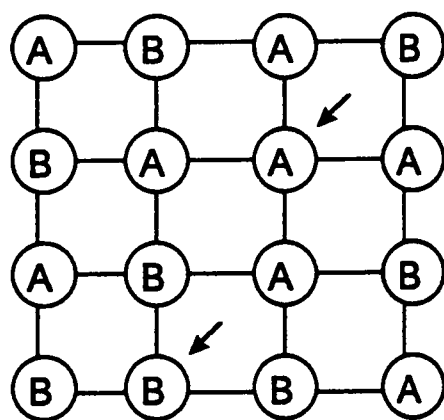
The triple defect concept was first established by Wasilewski (18) as the result of a detailed theoretical analysis of defect formation in B2 structures. He concluded that the structural symmetry requires that antisite and triple defects form as compositional defects in B2 structures. This was experimentally confirmed by later theoretical and experimental studies (15, 16, 17). A two-dimensional schematic diagram showing these two defect types is shown in Fig. 3-4 along with an ideal crystal for comparison. In the ideal crystal perfect ordering occurs when the A atoms occupy the α sites and the B atoms are at the β sites. An upset in the crystal order occurs if an A atom occupies a β site or, likewise, a B atom occupies an α site. These defects are known as antisite (or antistructure) defects. A triple defect occurs when an A atom jumps from an α site to a β site (thereby becoming an antisite atom) creating a vacancy on the α site. In order to maintain stoichiometry another vacancy is formed on an α site as shown.

A comparison of the enthalpies of formation for various B2 structures led Neumann (19, 20) to suggest that both antisite atoms and triple defects were present in FeAl.

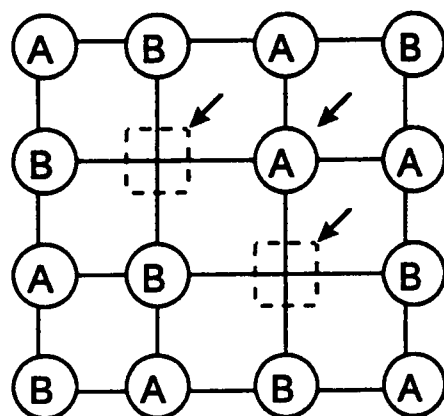
However, the decrease in lattice parameter with increasing Al content past stoichiometry (50 at.% Al) suggests the presence of constitutional vacancies since this decrease is most certainly due to vacancy relaxation. If Al antisite atoms were the dominant constitutional defect then the lattice parameter would increase since the Al atoms are larger than the Fe atoms. Field ion microscopy studies by Paris et al. (21) have further confirmed that triple defects predominate in FeAl alloys.



Ideal crystal showing perfect ordering of the A and B atoms on the α and β sites, respectively



Antisite defect containing one A atom on a β site and a B atom on an α site



Triple defect containing one A atom on a β site and two vacancies on α sites

Fig. 3-4. Schematic diagram showing antisite and triple defects.

Vacancy Formation and Compositional Dependence of FeAl

For FeAl alloys, a knowledge of the vacancy concentration with temperature is especially important since the mechanical properties of these alloys are very sensitive to point defects. The most often-used techniques to measure the equilibrium vacancy concentration (n_v) and the enthalpy of vacancy formation (ΔH_f^V) are resistometry and differential dilatometry. Other important methods are field ion microscopy and positron annihilation.

Constitutional Defect Formation

In 1977, Riviere (22) theoretically modeled the constitutional defect structure of FeAl assuming that excess Fe atoms occupy the Al sites. He derived a complicated expression to describe the concentration of vacancies on the Fe lattice as a function of Al content at a given state of disorder. The model predicts that the concentration of antisite Fe atoms increases with decreasing Al concentration for FeAl compositions less than stoichiometric (50 at.% Al). The vacancy concentration, however, begins to increase with Al content for FeAl alloys having compositions greater than 50 at.% Al. To confirm his model, Riviere measured the density of FeAl as a function of composition and compared the results to theoretically-calculated density values. His results confirmed that large quantities of constitutional vacancies were formed for FeAl alloys having Al concentrations greater than 50 at.% Al.

A simplified version of this model was derived from statistical thermodynamics by Chang and Neumann (17). Using the expression for configurational entropy and ignoring all second order or higher energy terms, they developed the following relationship between triple defect concentration and composition:

$$z^2 (z - 2X) = \alpha_D^3 \quad (3-1)$$

where z is the concentration of triple defects and X is the deviation from stoichiometry.

The disorder parameter (α_D) is the defect concentration at the stoichiometric composition at a given temperature. They further developed their model to describe the distribution of atoms and vacancies on the α and β sublattices

$$2 [n_V^\alpha]^2 [n_A^\beta] = \alpha_D^3 \quad (3-2)$$

Here, n_V^α is the concentration of vacancies on the α sublattice and n_A^β is the concentrations of antisite A atoms on the β sublattice. Therefore, for a given disorder parameter, the relationship between the concentrations of vacancies and antisite atoms can be determined. A graphical representation of this model is shown in Fig. 3-5. For compositions less than stoichiometric (A-rich region), antisite A atoms predominate, while vacancies dominate at compositions greater than stoichiometric (B-rich region). This model compares well with the experimental results of other researchers (10-14). However, it cannot be used quantitatively without an experimentally determined disorder parameter. Their model also does not explain the differences in the structure-property relationships that exist between different B2 structured alloys.

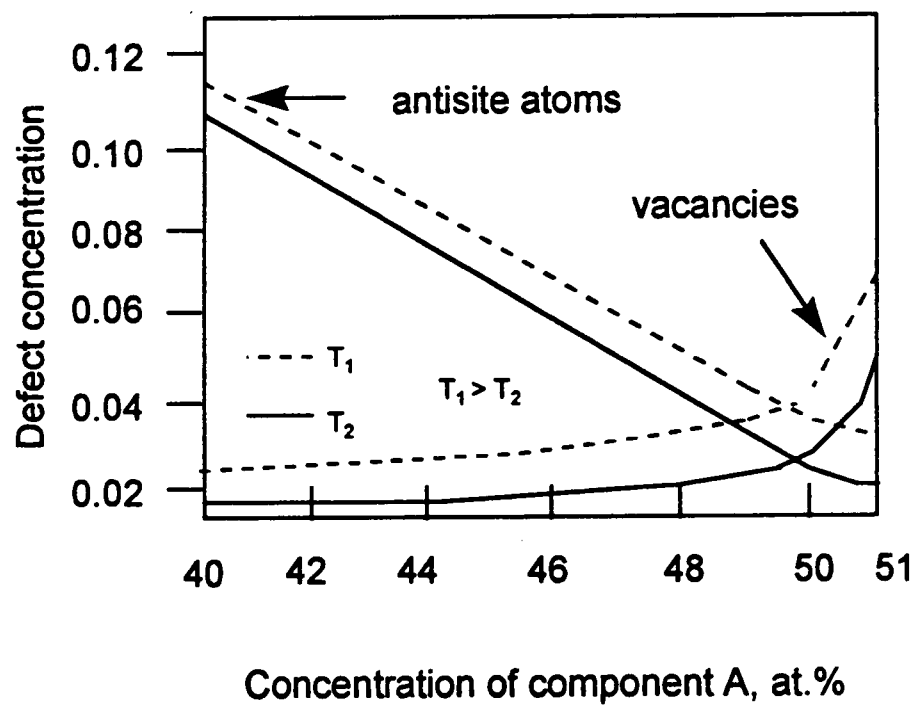


Fig. 3-5. Graphical representation of the Chang-Neumann model (17).

Thermal Vacancy Formation

A review of literature shows that the formation of thermal vacancies in FeAl alloys has not been extensively researched. In 1972 Troschkina and Kutcherenko (23) measured the specific heat on several FeAl alloys that were initially quenched from 800°C. Their results showed a dip in the specific heat around 400°C that was attributed to the removal of retained vacancies. They found that the size of this dip increased with increasing Al concentration. In 1975 Riviere and Grihle (24, 25) determined the enthalpy of vacancy formation (ΔH_f^V) in FeAl alloys by measuring the resistivity increase as a function of quench temperature. For FeAl alloys containing 38.5, 40, 44, and 50 at.% Al, they obtained a ΔH_f^V values of 90.7, 87.8, 80.1, and 74.3 kJ/mol respectively. They also found that the enthalpy of vacancy mobility (ΔH_f^M) for a 40 at.% Al alloy was approximately 155.3 kJ/mol. This decrease in ΔH_f^V with Al content was associated with the increase in n_v with increasing Al content for a given temperature. In a later investigation, Hasaka (26) theoretically modeled ΔH_f^V as a function of composition for FeAl using the Bragg-Williams approximation. Although his model does not compare well with Riviere and Grihle's results, it does show a decrease in ΔH_f^V with increasing Al concentration. Another theoretical study was performed by Fu et al. (27) who used the supercell approach to obtain a ΔH_f^V value of 93.6 kJ/mol for a stoichiometric FeAl alloy.

More recent values of ΔH_f^V and ΔH_f^M were obtained by Würschum et al. (28) by observing the equilibration process of the n_v by positron lifetime measurements. For

alloys having 37 and 39 at.% Al, ΔH_f^V was found to be 100.3 and 94.5 kJ/mol respectively. These results are in agreement with Riviere and Grihle's results although they are somewhat higher. For both alloys ΔH_f^M was approximately 164 kJ/mol which is in good agreement with Riviere and Grihle's result (24) and also agrees with the values obtained for bcc transition metals (e.g. Ta, Nb, W, Mo, etc.) as reported in a review paper by Schultz (29). However, the values of ΔH_f^V for FeAl (74 to 100 kJ/mol) are 2 to 3 times lower than for pure metals as reported in Schultz's paper. It is important to note that these enthalpies are for vacancies only and do not include the energy required for Fe antisite formation.

Thermal Vacancy Concentration Measurements

Paris et al. (21) first began studying the vacancy behavior in FeAl alloys by investigating the distribution of vacancies in a near-stoichiometric (49.5 at.% Al) alloy using field ion microscopy. They compared the structure of an alloy quenched from 1000°C to one that was annealed at 425°C for 24 hours. Their results showed that the Al atoms did not occupy the Fe-sites and that the majority of the vacancies occupied the Fe sites. This is consistent with triple-defect formation. They also noted that lower but significant number of vacancies (0.65×10^{-2}) occupied the Al-sites at 1000°C. Paris and Lesbats (16) then performed the first detailed study evaluating the n_v of FeAl with composition. Using differential dilatometry, they determined that, at a given temperature n_v for alloys between 46 and 51 at.% Al increased in accordance with the triple defect model developed by Chang and Neumann (17). Their results indicated that thermal vacancies

begin to form in FeAl between 400 and 500°C and that n_v at any given temperature increases with increasing Al content. They noted that the vacancy concentration reaches a maximum around 1100°C and then begins to decrease with temperature. This is probably associated with the B2 structure becoming more disordered.

Ho and Dodd (15) also used dilatometry to determine n_v in FeAl as a function of composition. A comparison of their results with those of Paris and Lesbats is shown in Fig. 3-6. Although the agreement is good between the two data sets, the results for Ho and Dodd are 2 to 4 times lower than those obtained from Paris and Lesbats' investigation. In addition, their results do show a dramatic increase at 50 at.% Al as predicted by the Chang and Neumann model (17). In either case the vacancy concentrations are very high compared with pure metals, typically 0.01%.

Defect Kinetics of the FeAl B2 Structure

The first investigation into the kinetics of the structural changes came from several studies of the effect of heat treatment of FeAl alloys on hardness (10, 12). For compositions between 33 to 50 at.% Al, Nagpal and Baker (10) found that the hardness was the same after either water quenching or air cooling from 1000°C. According to Würschum et al. (28), the unusually low ΔH_f^V values combined with a high ΔH_f^M were an indication of low vacancy diffusion for these alloys. Evidence that the atomic diffusion for FeAl is more complicated than for pure metals was provided by the Mössbauer

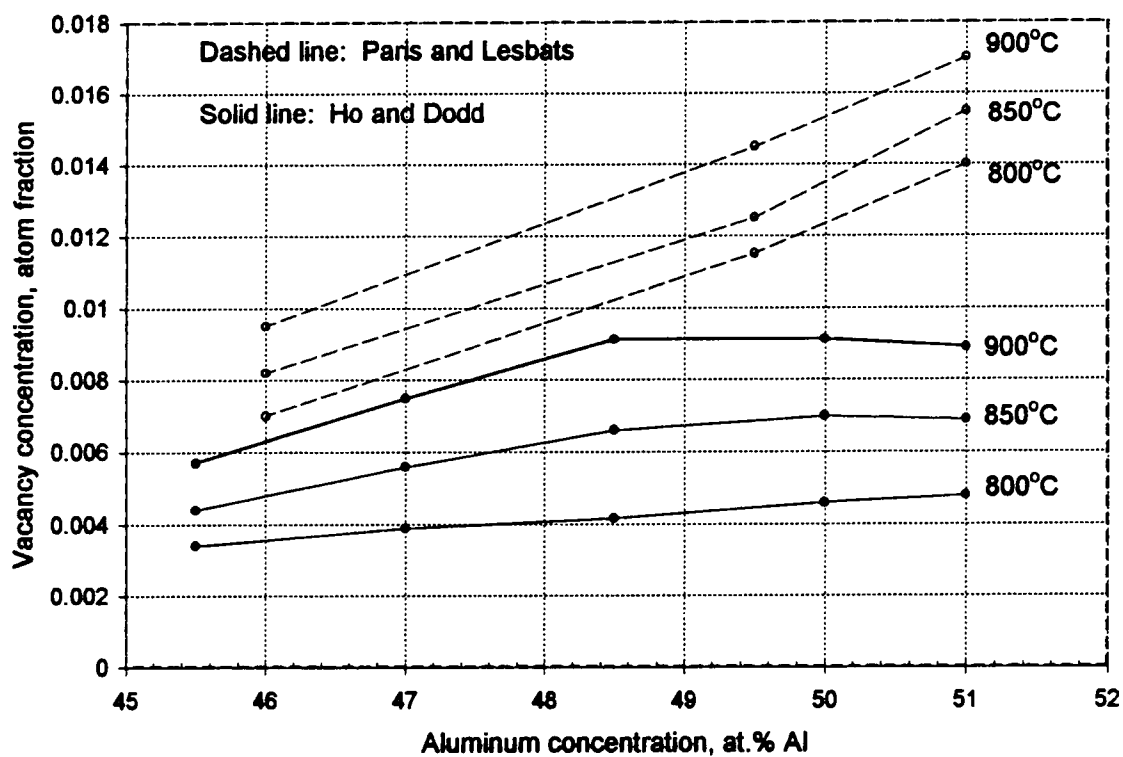


Fig. 3-6. Vacancy concentration in FeAl alloys as determined by dilatometry measurements of Ho and Dodd (15) and Paris and Lesbats (16).

spectroscopy studies of Vogl et al. (30, 31). They showed that Fe atoms diffuse by a double jump process illustrated in Fig. 3-7. In order for this jump process to occur, an Al atom must jump onto the Fe sublattice, and thereby create a vacancy on an Al site on the β sublattice as shown in Fig. 3-7(b). The Fe atom then jumps back to a position in the Fe (or β) sublattice as illustrated in Fig. 3-7(c). Vogl et al. (30) also showed that this jump mechanism was independent of composition and that the residence time between jumps increases with Fe content. As the Al content decreases, the number of sites available for diffusion to occur also decreases. This effect would explain why the residence time of the diffusing Fe atoms increases with increasing Fe concentration.

Another resistometry study was done Dinhut et al. (32) for a 40 at.% Al alloy. They quenched a series of 40 at.% Al alloys from 900°C and then measured the response of the electrical resistivity (ρ) with time for various annealing temperatures. The resistivity began to drop in value after 1 min. (t_i) above ~420°C, and took about 10 min. (t_f) to become constant. At 380°C, ρ began to drop after $t_i \cong 10$ min. and became constant at $t_f \cong 200$ min. These changes were attributed to the removal of vacancies. Extrapolation of t_i and t_f to higher temperatures suggests that only rapid cooling (e.g. water quenching) would retain thermal vacancies during cooling. The results of Dinhut et al. are puzzling when compared with the other kinetic studies. However, this discrepancy may be an indication that the highly ordered nature of the B2 structure causes the electrical resistivity to become less sensitive to the presence of vacancies.

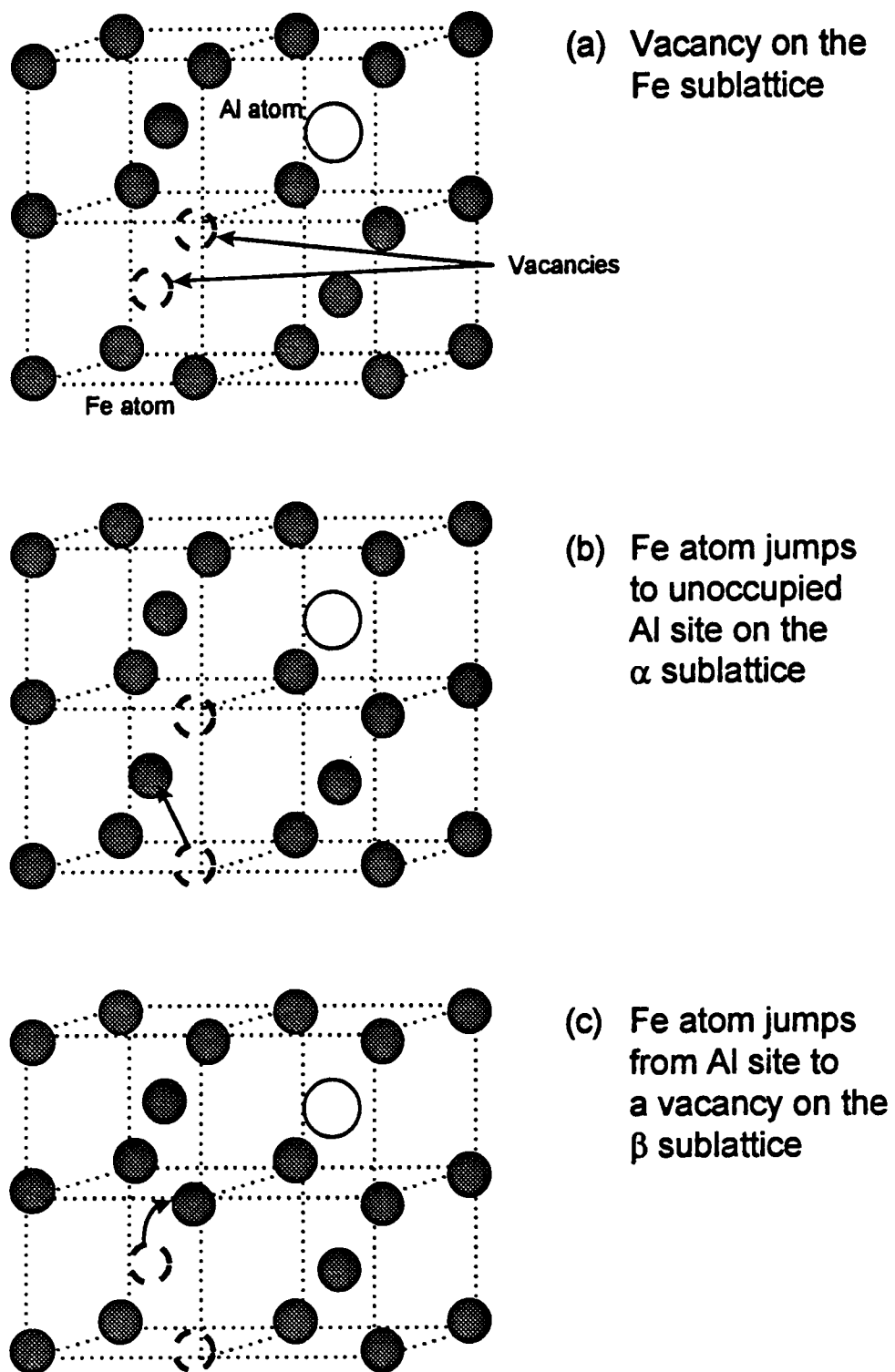


Fig. 3-7. Vacancy jump mechanism for FeAl determined by Vogl et al. (30)

D0₃-B2 Phase Change

A survey of the literature shows that a number of studies have been undertaken to investigate the D0₃-B2 ordering transition (33-42). In most, ρ was measured as a function of quenching temperature and annealing time. Because ρ of an ordered material is sensitive to defect concentration and degree of order, it is a widely used means of investigating and interpreting these phenomena. Several independent investigations (35-42) have shown that the kinetics associated with D0₃-B2 ordering are exceptionally fast and that only high quench rates (e.g., water quench) will retain the B2 structure at low temperature.

In a recent study Kim and Morris (43) determined the long range order parameter for the D0₃ and B2 structures for 28 and 32.5 at.% Al alloys by neutron powder diffraction. They found that D0₃ order begins to break down around 350°C and becomes completely disordered at the Fe₃Al-FeAl phase boundary. Below 350°C, the Fe diffusivity is very low, which is to be expected for a highly ordered structure. For the B2 structure they observed that disorder begins on the Fe (or α) sublattice around 500°C and is complete at the Fe₃Al-FeAl phase boundary. Kim and Morris defined disorder within the B2 structure as the decrease in Fe concentration on the α sublattice as vacancies are formed (also on the α sublattice). As a result, the temperature at which they define as the onset of disorder is also the temperature at which vacancies begin to form. Their results agreed with the positron lifetime measurements of Schaefer and Würschum (44) that 1)

structural vacancies do not form within the Fe_3Al D0_3 structure, and 2) appreciable quantities of thermal vacancies form at temperatures around 800°C .

Another interesting feature of Fe_3Al is its unusual ρ - T behavior. Numerous measurements (34, 36, 45-47) have shown that, for Fe_3Al alloys, the ρ - T curve exhibits a maxima at temperatures just below the D0_3 to B2 transition. One group of researchers (47) attributed this maxima to the Curie temperature, while others (36) believe that it is caused by a semiconductor effect in which additional electrons are excited into a conduction band. Recent investigations by Nishino et al. (46, 47), Kawamiya (48) and Shirai (49) have shown similar resistivity versus temperature behavior for types of intermetallic alloys.

Pulse-Heating Calorimetry and Resistivity Measurements

Pulse-heating calorimetry was developed as a means of overcoming heat losses associated with conventional calorimetry techniques. These inaccuracies and heat losses increase with temperature and are especially high at temperatures greater than 1000°C . In addition the materials used in conventional calorimetry equipment cannot withstand high temperature exposure. Because pulse-heating calorimetry reduces the time of exposure at high temperatures, the heat losses are less and the accuracy improved. Because of this, pulse-heating calorimetry is used extensively by the National Institute of Standards and Technology to determine the thermophysical properties of materials at

high temperatures. One important feature of pulse-heating calorimetry is that the specific heat and electrical resistivity of the specimen are determined simultaneously. This allows a direct comparison of these two properties as a function of temperature.

The first reported successful attempt to use pulse-heating calorimetry was by Worthington in 1918. He used a battery operated unit to determine the C_p values of tungsten and carbon. This technique has been modified and improved of the past 70 years, especially with the advent of high speed data acquisition systems and computer programming. Around 1965 Kollie (50) designed and built a pulse-heating calorimeter at the University of Tennessee in Knoxville. He used this system to determine the specific heats of high purity iron and nickel (51). More recently, Basak (52) designed and developed a programmable system which he used to measure the C_p and ρ of Ni_4Mo . He also determined the accuracy of the present system using pure nickel and found it to be within $\pm 1\%$ for resistivity measurement and $\pm 2\%$ for C_p determination (45). Basak observed a large rise in C_p for several iron aluminide alloys and successfully measured the electrical resistivity with temperature. It is this system that was used in the present study to investigate the defect formation of several iron aluminide alloys.

Chapter 4

Experimental Procedure

Specimen Preparation

The specimens used in this study were prepared in two batches. Four alloy samples having compositions of 30, 38, 43, and 48 at.% Al were cast in one setting and were labeled Batch #1. Another group of four alloy samples having the same compositions were prepared the next day and labeled Batch #2. Thus there were two specimens per alloy composition and eight specimens total. The 30 at.% Al alloy is in the Fe_3Al phase at room temperature while the other three compositions are in the FeAl phase. A flowchart showing each step used to fabricate the test specimens is shown in Fig. 4-1.

Alloy Fabrication

High purity iron and aluminum particles were weighed to 0.01 gram accuracy. The total mass targeted for each alloy was 30 grams. The components were loaded into a copper mixing crucible and placed inside an arc furnace. A bead of zirconium or tungsten was placed to one side of the crucible to serve as a getter for oxygen while the components were being melted. The furnace chamber was sealed and the atmosphere was evacuated to 0.05 torr. Next the chamber was flushed with Argon gas to 20 torr. The chamber was evacuated and flushed twice more with argon in order to minimize oxygen and

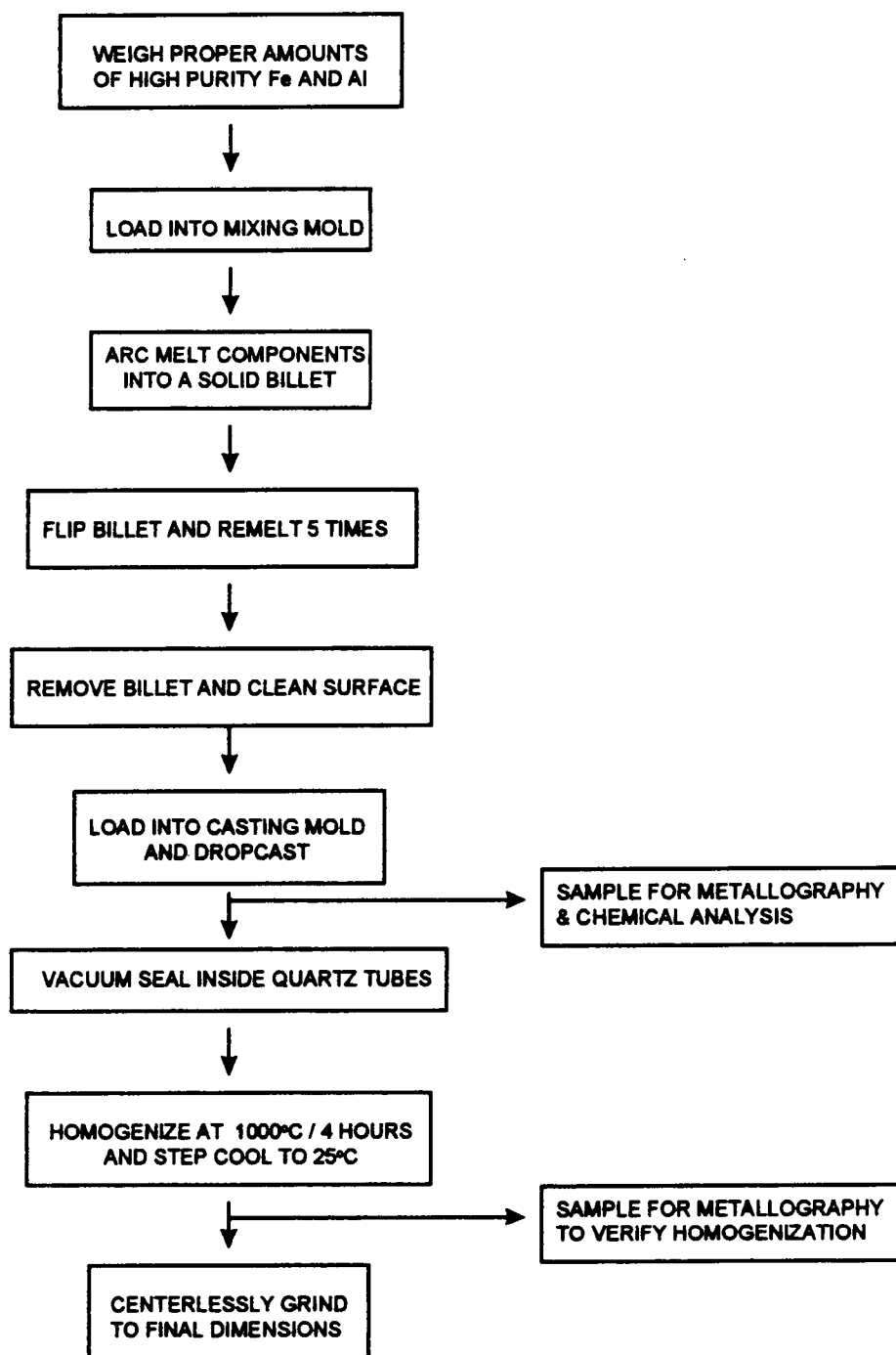
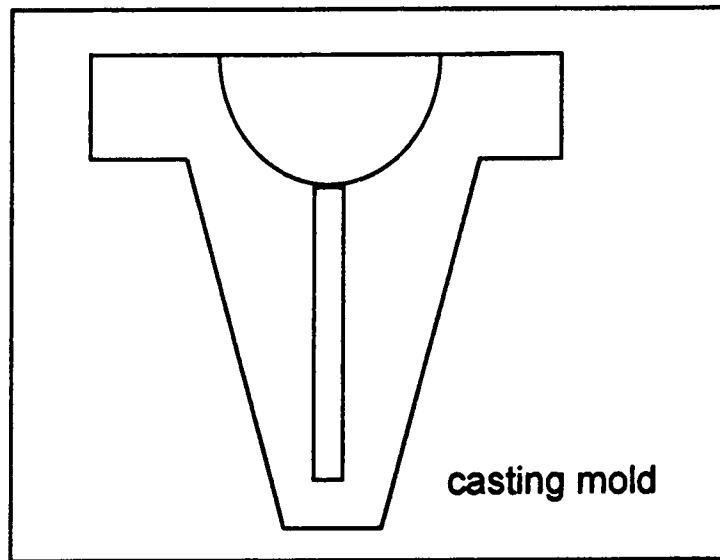


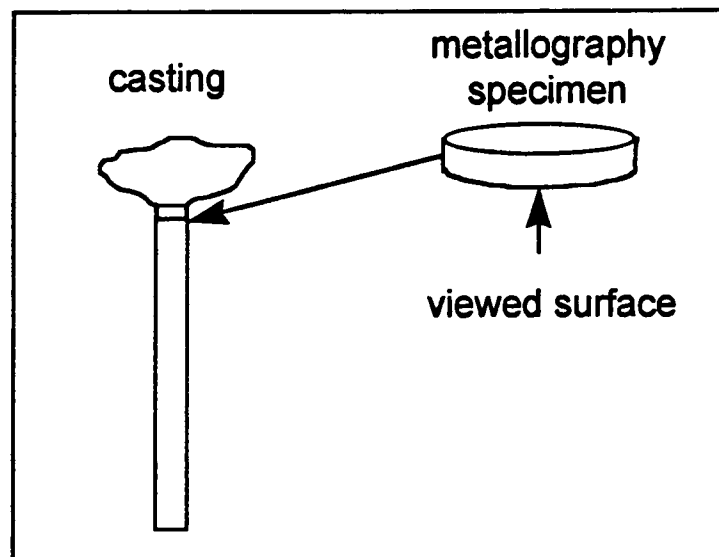
Fig. 4-1. Flow diagram of specimen fabrication process.

carbon. After pulling a vacuum for the third time, argon was added until the pressure was 5 torr.

The getter bead was melted first, then the Fe and Al pieces were arc melted under low power. During cooling, the melt solidified into a solid alloy billet, which was flipped over and remelted. This procedure was repeated five times to ensure that all of the material was thoroughly melted. The alloy billet was removed from the furnace and weighed to 0.01 gram accuracy to determine mass lost during the alloying process. The alloy samples were thoroughly cleaned using a wire brush to remove any surface contamination. Each billet was roughly 2.5 cm in diameter and approximately 1.7 cm thick. Next, the mixing crucible was removed from the furnace and was replaced with a copper casting mold that had an inner contour consisting of a hemispherical cavity which funneled into a long cylindrical casting chamber as shown in Fig. 4-2(a). The billet was loaded into the mold, the getter was replaced, and the chamber atmosphere was purged once again as described earlier. The billet was then melted until it dropped into the chilled mold. Because the amount of alloy material was greater than the capacity of the cylindrical casting chamber, excess material partially filled the funnel section of the mold to produce a casting as shown in Fig. 4-2(b). This excess material was cut from each cast rod using a diamond saw. The cast rods (with excess removed) measured approximately 0.64 cm in diameter and 5.72 cm long. A small piece was removed from the top end of each rod as shown. For the Batch #1 samples, these cut pieces were used for metallography with the cut section being the viewed surface as shown in Fig. 4-2(b).



(a)



(b)

Fig. 4-2. Schematic of casting crucible and cast alloy.

In the Batch #2 alloys, these cut pieces were sampled for elemental impurity analyses including carbon and oxygen.

Homogenization and Annealing

The eight specimens were individually placed inside quartz glass tubes. The tubes were flushed twice in argon to minimize oxygen, nitrogen, and carbon, evacuated to 8×10^{-6} torr pressure, and sealed. They were then placed inside a resistance furnace and heated at 1000°C for 4 hours to undergo homogenization. Following this heat treatment, the specimens were cooled to room temperature using stepwise procedure to minimize the retention of thermal vacancies in the cooled structure. The temperatures and corresponding hold times for the step-wise cooling procedure were 800°C for 1 day, 600°C for 3 days, 500°C for 5 days, 400°C for 7 days, 300°C for 7 days, and 200°C for 7 days. After cooling to room temperature the specimens were sampled once again for metallography (as described earlier) in order to determine whether homogenization was complete.

Final Preparation

The annealed specimens were centerlessly ground to diameters of 0.2 cm that were uniform to ± 0.002 cm over the entire specimen length. The diameter of each specimen was carefully measured at ten locations using a precision caliper. At each location a measurement was made in one direction (0°), then the specimen was rotated 90° and a second measurement was made. Therefore, a total of 20 measurements were made on

each specimen and the average of these measurements was used in the determination of the electrical resistivity and specific heat.

Before either ρ or C_p values could be determined, it was necessary to attach a thermocouple and a pair of voltage taps on each specimen surface. A 0.13 mm diameter Pt:Pt-13wt.% Rh thermocouple was spot welded to the specimen surface half way from the ends. The voltage taps were 0.13 mm diameter nickel wires that were spot welded on either side of the thermocouple (at a distance of approximately 1 cm). The voltage drop, E_{VT} , across the nickel wires was measured and the material between them defined that for which ρ and C_p was determined.

Metallography Procedure

Each alloy had a tiny section in the shape of a small disk removed at the top of the specimen for metallographic analysis, as was mentioned earlier. These sections were loaded into 1 inch diameter molds with the surface to be viewed facing down. The molds were filled with a quick-set epoxy which hardened overnight. After 24 hours the metallography samples were removed from the molds.

The samples were ground before being polished. The specimens were ground starting with a 20 grit sandpaper. Each sample was pressed against the sandpaper firmly and moved back and forth across the sandpaper surface 50 to 100 times while maintaining

constant direction. When finished the sample was rotated 90 degrees and resanded (using the same procedure) with a higher (finer particle size) grit sandpaper. After each run, the grit number was increased and this process was repeated. Final grinding was done using 600 grit sandpaper. The specimens were viewed using optical microscopy to ensure that there were no large scratch marks prior to polishing.

The metallography specimens were polished using a diamond paste technique. Diamond paste was directly applied to a polishing cloth without using water or any other aqueous media. Three polishing cloths were used to reach the final surface finish. The specimens were first polished with a 15 μ m particle size diamond paste, followed by 3 μ m, and finished with 0.25 μ m diamond particles.

The polished specimen surfaces were chemically etched using an mixture consisting of 50 vol.% acetic acid, 33 vol.% nitric acid, and 17 vol.% hydrochloric acid. This acid mixture was diluted with 1 to 2 parts water prior to etching. After etching, the surfaces were examined using an optical microscope and photographed.

Knife-Edge Measurements

A knife-edge apparatus built by Basak (45) was used to determine the room temperature resistivity and the length, L , between the voltage taps, is shown schematically in Fig. 4-3. A quartz rod with a sharp radius knife-edge glued to each end was used to define a distance, L_{KE} . This distance was determined using an optical comparator which was

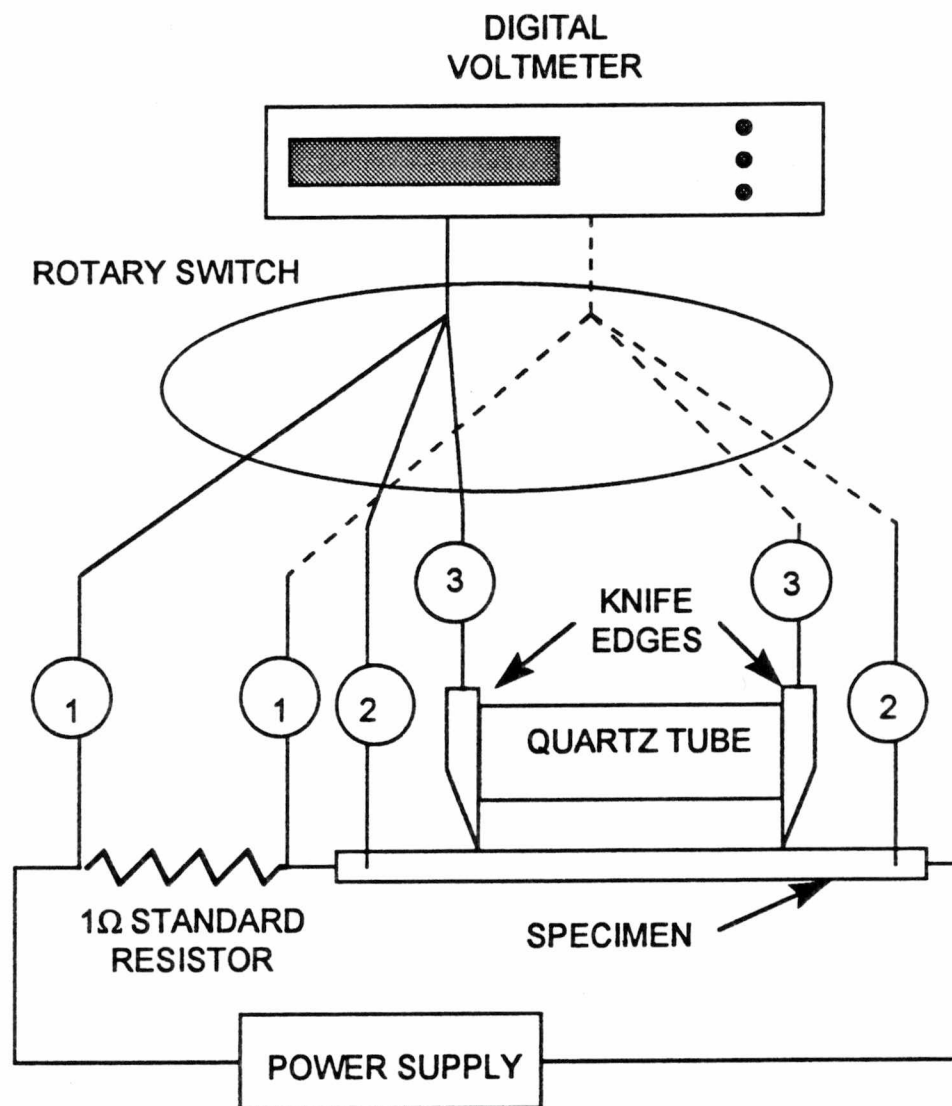


Fig. 4-3. Schematic representation of the knife-edge apparatus.

accurate to between +0.01% and -0.005% (50). The knife-edge was carefully placed on the specimen, which was in series with a 0.99994 Ω standard resistor and power supply. A current of approximately 0.1 amps was passed through the specimen for less than 20 seconds in order to obtain measurements without significantly increasing the temperature of the specimen. The voltage drops across the quartz tube knife-edges (E_{KE}), the standard resistor (E_{SR}), and the specimen (E_S) were measured using a precision digital voltmeter (see Fig. 4-3). The current was reversed and the measurement was repeated. The average distance L was calculated from

$$L = L_{KE} (E_S / E_{KE}) \quad (4-1)$$

This length, L , was determined to be accurate to within ± 0.01 mm.

The knife-edge apparatus was also used to measure ρ of the specimens at room temperature. The resistivity was calculated from

$$\rho = (\pi/4) (d^2/L) [E_S / I_S] \quad (4-2)$$

where d is the specimen diameter, E_S is the voltage drop across the specimen, and I_S is the current passed through the specimen and is equal to E_{SR} divided by the resistance of the standard resistor (0.99994 Ω).

The variable that has the greatest influence on the accuracy of ρ is the diameter, since it is small and is squared in the equation. Based on the accuracy of the quantities in the measurements, ρ was estimated to be accurate to $\pm 0.5 \mu\Omega\text{-cm}$ or about 0.7% for our specimens.

Operation of Pulse-Heating Calorimetry System

Principals of Operation

A schematic showing the calorimeter and the data acquisition system is shown in Fig. 4-4. The current and length of pulse are entered into the computer which then triggers the programmable power supply to provide the current pulse. The power supply is connected in series with the specimen and a standard resistor ($0.0100000 \pm 0.0000001 \Omega$). The specimen temperature increases by Joule heating. Three transient signals are acquired during the pulse as shown in Fig. 4-4. They are the voltage drop across the standard resistor (E_{SR}), the voltage drop across the nickel wire taps (E_{VT}), and the thermocouple emf (E_{th}). These three signals are amplified, converted from analog to digital format, and then stored in the computer memory with the accompanying time interval. These signals are processed by the computer such that the thermocouple emf is converted to degrees Kelvin (T) and the system current (I) is determined by $E_{SR}/0.0100000 \Omega$.

Pulse-Heating Operating Procedure

Each specimen was loaded individually into the specimen holder as shown in Fig. 4-5. The thermocouple wires and nickel leads were attached to the data acquisition circuitry described in Fig. 4-4. The specimen holder was placed inside the test chamber which was then sealed. The test chamber was depressurized using a oil mechanical pump to

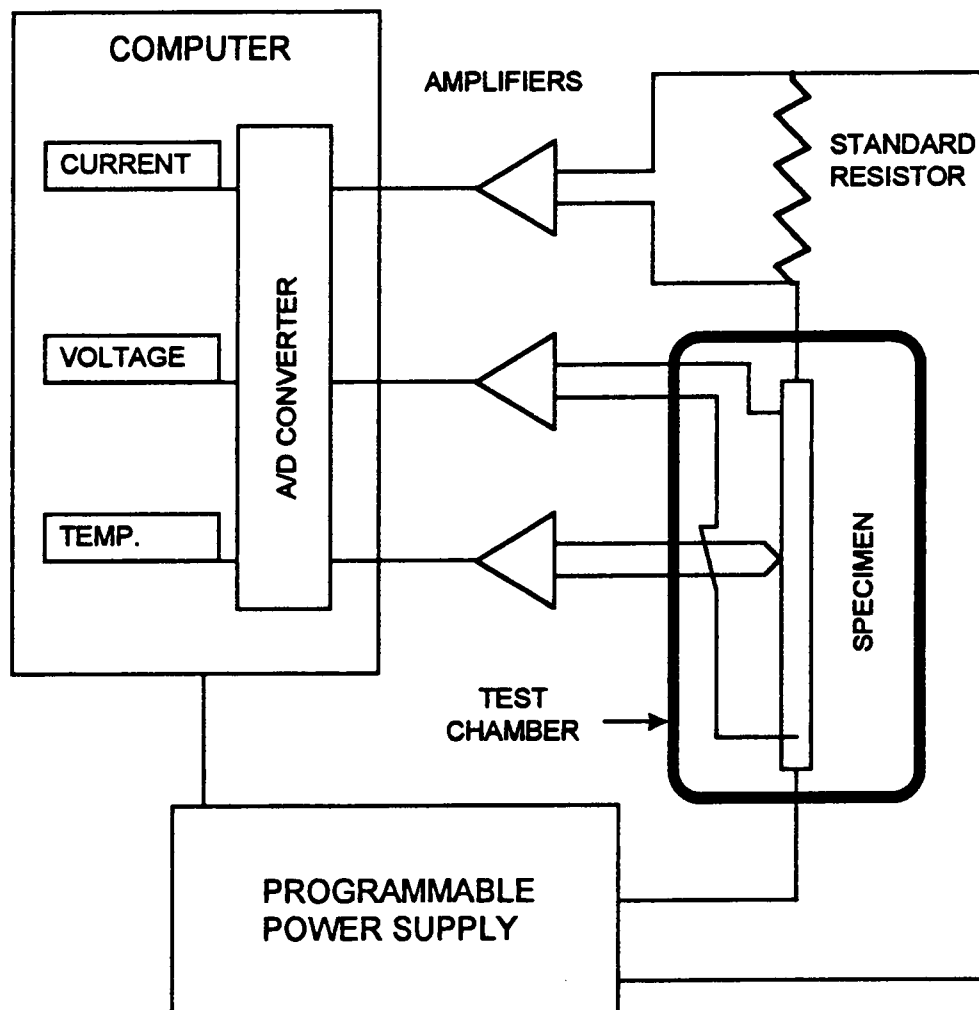


Fig. 4-4. Block diagram of the pulse heating calorimeter system.

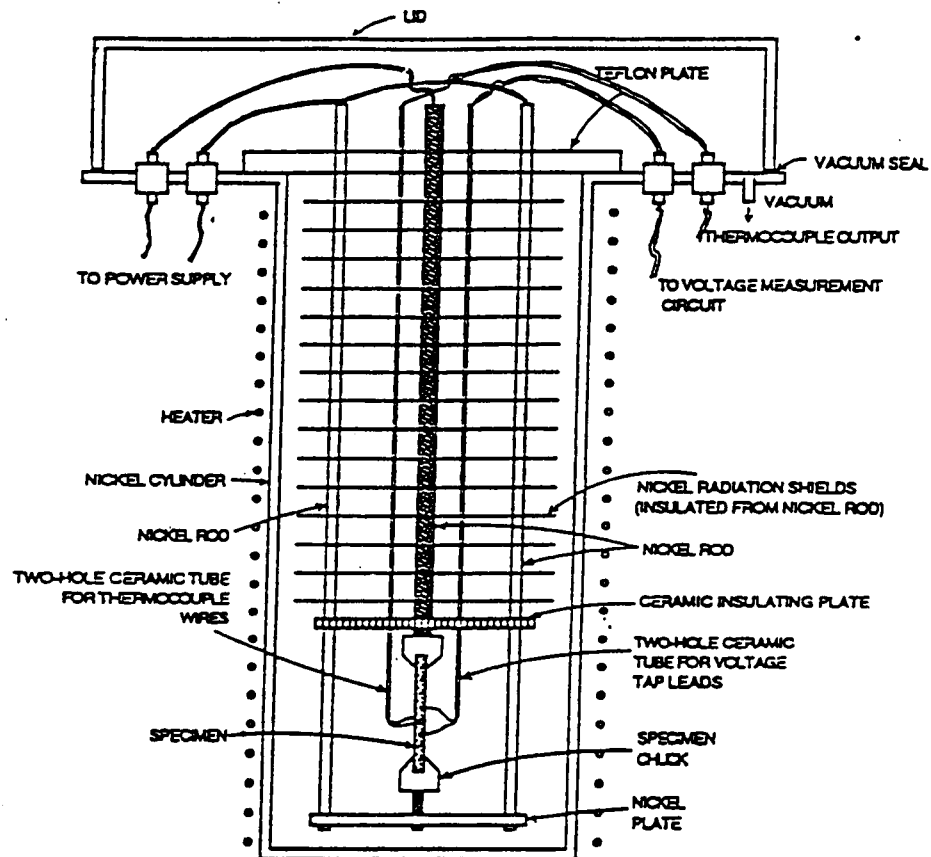


Fig. 4-5. Schematic diagram of the specimen holder.

below 10 torr. Once the pressure was below about 10 torr, a diffusion pump was activated. When the chamber pressure fell to approximately 2×10^{-5} torr, the specimen was ready to be tested.

The current and pulse duration parameters were entered by the operator into the computer. A typical current was 50 amps and the pulse interval was relatively short (10 to 20 seconds). The computer then prompted the power supply to send a specified current pulse (e.g., 50 amp) through the test specimen. The three signals (I , E_{VT} , and T) acquired during a test run are shown schematically as a function of time in Fig. 4-6. While the current (I) is constant throughout the pulse, both the voltage (E_{VT}) and temperature (T) increase with time. These three signals were recorded at 0.2 to 0.5 second intervals during the pulse, but only the temperature was recorded during the cooling period. The precision of the data collection system was evaluated using a precision voltage source which was set at levels similar to those of the actual readings. These measurements agreed with the input values within the resolution of the computer (i.e., ± 1 count).

Determination of Electrical Resistivity

The current (I) and voltage drop across the specimen (E_{VT}) were used to determine ρ as a function of temperature. The resistivity was calculated from the relation

$$\rho = [E_{VT}/I] (\pi/4) (d^2/L) \quad (4-3)$$

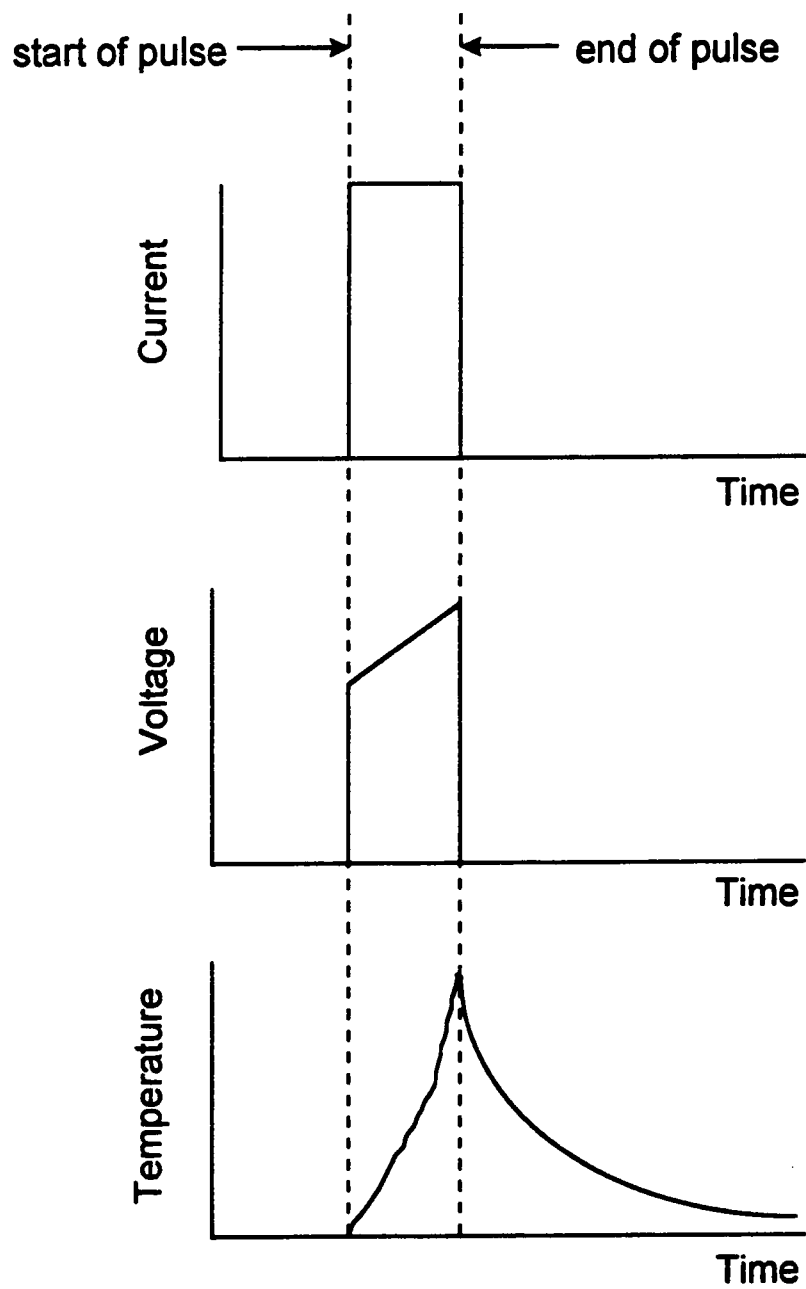


Fig. 4-6. Schematic showing relationship of the current, voltage, and temperature with time during the pulse.

where d is the specimen diameter averaged from 20 locations as discussed earlier and L is the distance between the nickel wire taps determined using the knife-edge apparatus. No corrections were applied for thermal expansion, which would increase the actual resistivity by 1 to 2% at high temperature. The temperature coefficient of resistivity (TCR) was determined from the ρ - T curves by dividing the slope associated with the initial rise in ρ by the initial resistivity.

Determination of Specific Heat

Before the specific heat could be calculated it was necessary to first determine the actual specimen mass between the voltage taps. The total mass (m_T) of each specimen was measured using a precision Mettler balance to 0.1 mg accuracy. Next the total specimen length (L_T) was measured using a precision caliper to 0.01 mm accuracy. The specimen density (D) was then calculated by

$$D = m_T(4/\pi)[1/(L_T d^2)] \quad (4-4)$$

The actual mass between the voltage taps (m_s) was then determined by

$$m_s = D (\pi/4)(L_T d^2) \quad (4-5)$$

The formula used to calculate C_p at each time interval was derived by balancing the different rates of energy. The power (P) is the rate of energy input to the specimen and is the product of the current (I) and the voltage (in our case E_{VT}).

$$P = \text{rate of energy input} = I E_{VT} \quad (4-6)$$

The amount of energy absorbed by the specimen is equivalent to the term $(m_s C_p dT)$.

Therefore, the rate of energy absorption can be expressed as

$$\text{rate of energy absorption} = m_s C_p (dT/dt)_{\text{HEATING}} \quad (4-7)$$

where m is the specimen mass, C_p is the specific heat, and dT/dt is the rate of temperature change with time. If there were no heat losses during testing, then the rate of energy input is equivalent to the rate of heat of absorbed. However, there always exists a finite heat loss associated during testing given by

$$\text{rate of energy loss} = -m_s C_p (dT/dt)_{\text{COOLING}} \quad (4-8)$$

From a power balance, the power input = rate of energy absorption + rate of energy loss.

By substitution this becomes

$$P = m_s C_p (dT/dt)_{\text{HEATING}} - m_s C_p (dT/dt)_{\text{COOLING}} \quad (4-9)$$

which rearranges to

$$C_p = (P / m_s) 1 / [(dT/dt)_{\text{HEATING}} - (dT/dt)_{\text{COOLING}}] \quad (4-10)$$

The temperature was determined by measuring the thermocouple emf and converting the emf signal to the absolute temperature scale. The National Institute for Standards and Technology has tabulated the emf signals with corresponding temperatures (absolute scale) for R-type thermocouples. These tables are published in NIST Monograph 175: Temperature-Electromotive Force Reference Functions and Tables for the Letter-Designated Thermocouple Types Based on the ITS-90 (International Temperature Scales of 1990). In order to maintain high accuracy Basak (45) previously fitted the tabulated data to a fourth order polynomial between 100 and 400°C and between 400 and 1100°C. His conversion polynomials were accurate to $\pm 0.1\%$. Because the

thermocouples used during this study were not calibrated, the actual error is estimated to be $\pm 2^{\circ}\text{C}$.

The current and voltage were measured directly by the test apparatus and the resulting power versus temperature data were fitted to a polynomial expression. The rate of change of temperature during the heating and cooling periods was calculated by individually fitting the temperature and time data for each period to a polynomial function. This function was differentiated with respect to time to determine (dT/dt) during heating and cooling. Then the heating and cooling (dT/dt) terms were plotted versus T and fitted to polynomial functions. Because the dependencies of $\dot{P}$, $(dT/dt)_{\text{HEATING}}$, and $(dT/dt)_{\text{COOLING}}$ on temperature were all described as polynomial functions, C_p could to be determined at any temperature using the above equation. In this study, C_p was determined at 50°C increments, which were sufficient for curve generation and baseline determination. Note that $(dT/dt)_{\text{COOLING}}$ is essentially constant for each run. However, $(dT/dt)_{\text{HEATING}}$ can be increased by increasing the heating rate applied to the specimen. Therefore, as the heating rate is increased, the heat losses associated with cooling become less significant since the expression $[(dT/dt)_{\text{HEATING}} - m_s C_p (dT/dt)_{\text{COOLING}}]$ dominates the C_p calculation.

Determination of ΔH_f^D , n_D , and n_V

In this dissertation, ΔH_f^D represents the enthalpy of triple defect formation which includes both the formation of vacancies (on Fe sublattice) and antisite Fe atoms (on the Al sublattice). Likewise, n_D is the concentration of triple defects that form in these alloys. Both the ΔH_f^D and n_D were determined using C_p -T data derived from pulse-heating calorimetry. In order to determine the defect contribution to specific heat (ΔC_p^D), it was first necessary to present the dilation, electronic, and lowest-order anharmonic terms to the specific heat (C_p^+) as a linear rise in temperature. This linear term was fitted to all experimental data points in the straight line region (typically between 500 and 900°C) using linear regression. This linear expression was then extrapolated to higher temperatures as shown in Fig. 2-1. The ΔC_p^D was determined by subtracting the linear extrapolated C_p^+ results from the empirically-derived C_p results at a given temperature in the high temperature region ($T \geq 800^\circ\text{C}$). This was performed for each specimen test.

The ΔH_f^D was determined from the following relationship:

$$\ln(\Delta C_p^D T^2) = \ln B - \Delta H_f^D (1/RT) \quad (4-11)$$

where B is the $\ln(\Delta C_p^D T^2)$ intercept. The values of $\ln(\Delta C_p^D T^2)$ were plotted against $1/T$ for each data set. The data fell onto a straight line as shown in Fig. 4-7. The slope of this line was measured and multiplied by the gas constant R (8.3143 J/mol-K) to obtain ΔH_f^D for each set of data.

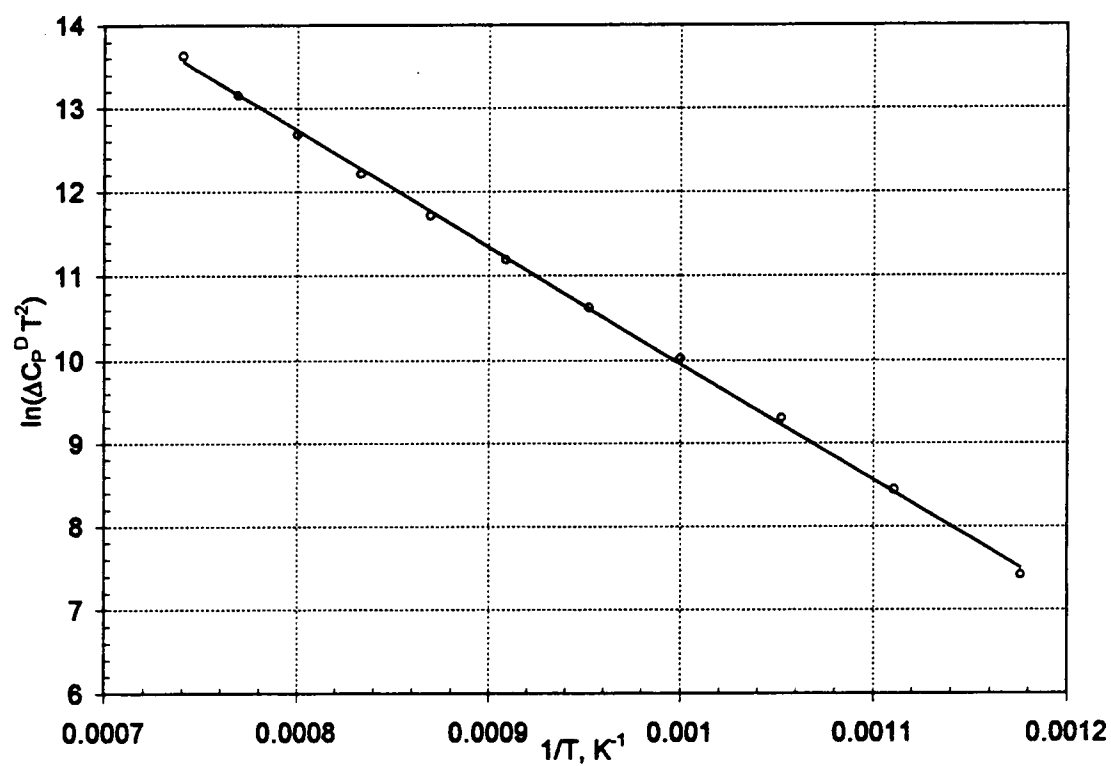


Fig. 4-7. Typical $\ln(\Delta C_p^D T^2)$ versus $1/T$ plot used to determine the enthalpy of triple defect formation.

The triple defect concentration, n_D , was determined by

$$n_D = \Delta C_P^D R (T/\Delta H_f^D)^2 \quad (4-12)$$

Since each triple defect consists of two vacancies on the α sublattice, the actual vacancy concentration n_V is determined by

$$n_V = 2 n_D \quad (4-13)$$

For each test run or data set, n_V was calculated at selected temperatures between 800 and 1000°C.

Test Protocol

Each of the annealed specimens was initially pulsed to ~1000°C at an average rate of 73°C/s $\pm$ 3°C/s, except for the specimen containing 43 at.% Al which was initially pulsed at 90°C/s. The specimens were cooled back to room temperature under vacuum at a rate of approximately 1.5°C/s. These results gave ρ and C_P as a function of temperature for the specimens in the annealed condition. Next the specimens were repulsed under the same operating conditions in order to determine whether ρ and C_P were affected by the thermal treatment of the initial pulse (heated to 1000°C then cooled at 1.5°C/s). The specimens were pulsed several times at different heating rates (50 to 170°C/s) to determine the influence of heating rate on ρ and C_P .

As part of the test protocol, data were taken with the current direction reversed to determine if an emf pickup was induced in the thermocouple. For the 30, 38, and 43

at.% Al alloys, the data were within the reproducibility of the measurements. The resistivity data for 48 at.% Al alloy differed slightly (e.g. up to 2%) with the current in opposite directions. These results were averaged to produce the ρ -T and C_p -T curves for the heat treated condition. The origin of the difference for this sample was not identified, although it was shorter than the other specimens.

Unfortunately, the 48 at.% Al alloy broke during a test run before the application of high heating rate. In addition it was discovered that heating past 1000°C caused the specimens to buckle in the holder. Therefore, the specimens were not heated to temperatures greater than 1000°C until the last run.

Chapter 5

Error Analysis

As part of this dissertation, the errors of estimate were determined for the calculated C_p -T baseline, ΔH_f^D and n_v values used in this dissertation. These errors are tabulated in Table 5-1 for each test run.

Prior Analysis

The errors associated with the experimentally determined ρ and C_p were previously estimated by Basak (45, 52). The variable that has the greatest influence on the accuracy for both of these measurements is the diameter, since it is small and is squared in the equations. Based on the accuracy of the quantities in the measurements, ρ obtained from the knife-edge apparatus was estimated to be accurate to $\pm 0.5 \mu\Omega\text{-cm}$, or about 0.7% for our specimens.

The accuracy of the pulse-heating system was evaluated by comparing electrical resistivity and specific heat data obtained for nickel from 25 to 1200°C. Kollie (51) previously had measured the resistivity of high purity nickel by a static method of equilibrating at a series of temperatures, and by using a pulse-heating apparatus similar

to that used for the present research. Kollie's original specimen was tested in the present device by Basak (45). The agreement with Kollie's data indicated an accuracy of 0.6%. Basak (45) also calculated the error of the experimental C_P data by combining the errors associated with the experimental and physical measurements, and estimated an error of $\approx 0.9\%$. The C_P data that Basak (45) obtained using Kollie's specimen agreed within 1% with those of Kollie from about 300 to 1200K, except just above the Curie temperature, where they agreed within 3%. The Curie temperature derived by Basak (45) from his measurements on Kollie's Ni specimen agreed to ± 1 K with Kollie's earlier value. Recall that only one thermocouple was on the specimen in the present research, and thus the axial temperature gradient is unknown. The agreement of the Curie temperature, and of ρ and C_P with the values of Kollie, is taken as an indication that the gradient is negligible.

Error Associated with Establishing the C_P -T Baseline

The error of the calculated values of ΔH_f^D and n_v are primarily determined by the error in establishing the C_P -T baseline which represents the dilation, electronic, and lower order anharmonic contributions. These baselines were determined by the least squares method for the linear region in the experimental C_P -T curve (typically between 270 and 500°C).

The standard error of estimate (S_e) for a simple linear regression is determined by

$$S_e = \sqrt{\frac{\sum [y_i - (b_0 + b_1 x_i)]^2}{n - 2}} \quad (5-1)$$

where the coefficients b_0 and b_1 are given by

$$b_1 = \frac{n\sum x_i y_i - (\sum x_i)(\sum y_i)}{n\sum x_i^2 - (\sum x_i)^2} \quad (5-2)$$

$$b_o = \frac{\sum y_i}{n} - b_1 \frac{\sum x_i}{n} \quad (5-3)$$

where y_i is the observed variable and x_i is the independent variable. The standard errors of estimate were calculated for each baseline run. The percent of error (normalized error) was determined by dividing S_e by the maximum value of C_p in the linear region in accordance with standard statistical practice. As shown in Table 5-1 there was some variance in the error between the runs. This is because the error associated with establishing the baseline was different for each run. For most of the runs, this error was less than 0.2%. However for the 38 at.% Al alloy heated at 162°C/s, the normalized error was 0.8%.

Error Associated with Determining ΔH_f^D

For each test run, ΔH_f^D was determined by multiplying the slope of the line of the $\ln(\Delta C_p T^2)$ versus $1/T$ data by the universal gas constant R . The $\ln(\Delta C_p^D T^2)$ versus $1/T$ data were fitted to a straight line using the least squares method and the error associated with the straight line was determined using the technique described above. This error is labeled slope error in this discussion. Because the slope is determined directly from the straight line, the slope error is equivalent to the error associated with the straight line fit. This error is shown in Table 5-1 for each test run. Since the experimental and baseline C_p - T curves were used in the determination of ΔH_f^D , their error contributions must be

included in the error estimate for ΔH_f^D . Therefore the error of estimate for ΔH_f^D is determined by

$$Error(\Delta H_f^D) = \sqrt{(Experimental)^2 + (Baseline)^2 + (slope)^2} \quad (5-4)$$

The experimental error used in this equation was previously determined by Basak (45) to be $\approx 0.9\%$. The estimate of error associated the enthalpy of vacancy formation for each test run is shown in Table 5-1. The scatter in values reflects the influence of the baseline error on each error estimate. Plots of the $\ln(\Delta C_p^D T^2)$ versus $1/T$ data for each test run are shown in Appendix A along with the calculated straight line fits.

Error Associated with Determining n_v

The defect concentration n_D is determined by $\Delta C_p^D R (T/\Delta H_f^D)^2$ and the vacancy concentration n_v equals $2n_D$. As a result the error of estimate contains the contributions from the experimental and baseline C_p - T curves and ΔH_f^D . However, because ΔH_f^D is squared in the calculation, its error must be doubled in determining the error estimate of n_v . Therefore the error estimate for the vacancy concentration is determined by

$$Error(n_v) = \sqrt{(Experimental)^2 + (Baseline)^2 + 2(\Delta H_f^D Error)^2} \quad (5-5)$$

As shown in Table 5-1, the estimate of errors of the calculated vacancy concentrations varied considerably between the test runs. The error associated with the 38 at.% Al alloy in the annealed condition is high because of high error associated with establishing both a baseline curve and a straight line of the $\ln(\Delta C_p^D T^2)$ versus $1/T$ data. This further

Table 5-1. Estimate of error (%) for the baseline, slope, ΔH_f^D , and n_v for each test run.

Alloy	Heat Treatment	Baseline Error (%)	Slope Error (%)	ΔH_f^D Error (%)	n_v Error (%)
30 at.% Al	annealed	0.03	1.5	2	3
38 at.% Al	annealed	0.2	3.2	3	5
43 at.% Al	annealed	0.06	1.0	1	2
48 at.% Al	annealed	0.2	1.2	2	2
38 at.% Al	heated to 1000°C, cooled at 1.5°C/s, and repulsed at 42°C/s	0.1	1.3	2	2
38 at.% Al	heated to 1000°C, cooled at 1.5°C/s, and repulsed at 52°C/s	0.05	1.8	2	3
38 at.% Al	heated to 1000°C, cooled at 1.5°C/s, and repulsed at 162°C/s	0.8	0.9	2	2
48 at.% Al	heated to 1000°C, cooled at 1.5°C/s	0.1	0.8	1	2

Chapter 6

Results and Discussion

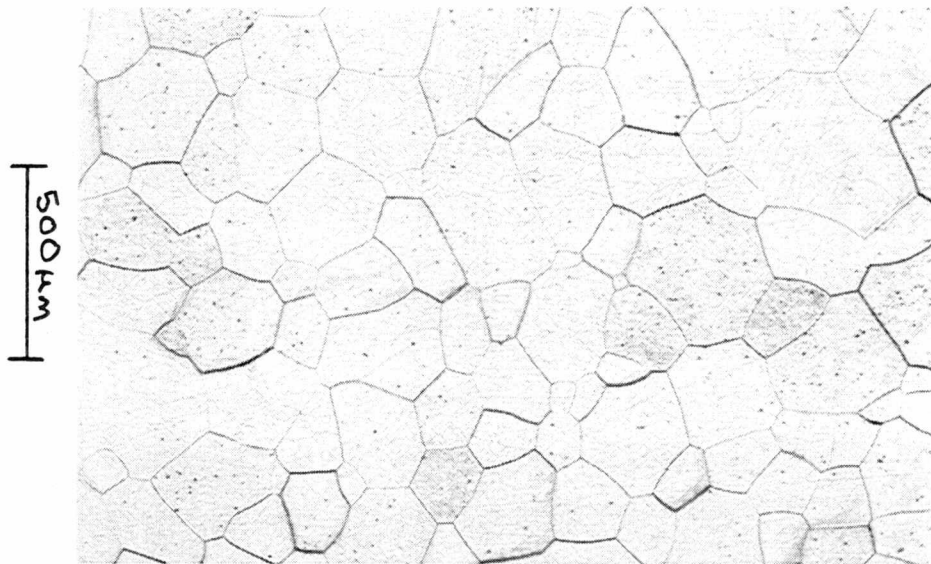
Specimen Chemical Analysis and Metallography

The micrographs of the "as-cast" grain structures for the Batch #1 alloys are shown in Fig. 6-1. The average grain size was determined by the line intercept method which was performed 5 times on each micrograph. The average grain size for each alloy is listed below in Table 6-1.

Table 6-1. Average grain size for each cast alloy.

Alloy	Avg. Grain Size (μm)
30 at.% Al	184 ± 15
38 at.% Al	159 ± 9
43 at.% Al	132 ± 11
48 at.% Al	122 ± 11

There is a noticeable decrease in the grain size associated with increasing Al concentration, though why this should be so is not clear. Coring was present which indicates that homogenization was required. Following homogenization and slow cooling, it was noted that the quartz glass annealing tubes had darkened uniformly to a yellow-brownish color. This suggests that a chemical reaction had occurred in the glass during heat treatment. There was no indication that the glass had reacted directly with the alloy specimens since the specimens were not stuck to the glass. It was also noted

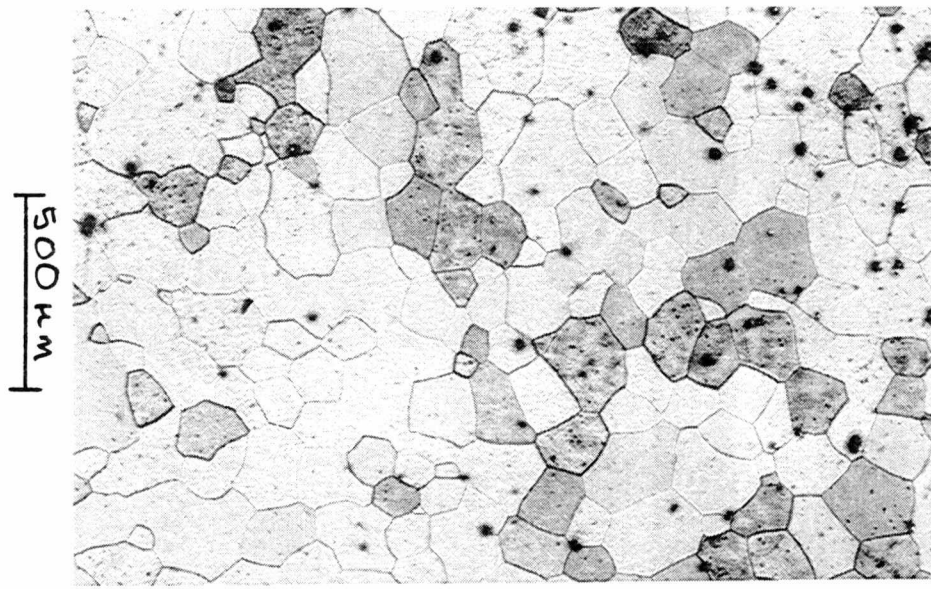


(a)

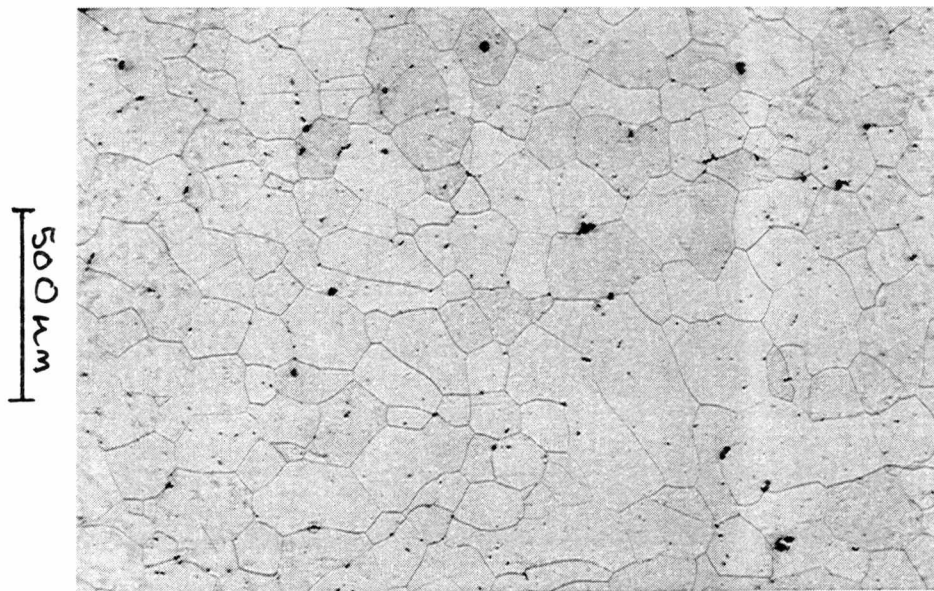


(b)

Fig. 6-1. Microstructures of cast alloys: (a) 30 at.% Al and (b) 38 at.% Al alloys (continued on the next page).



(c)



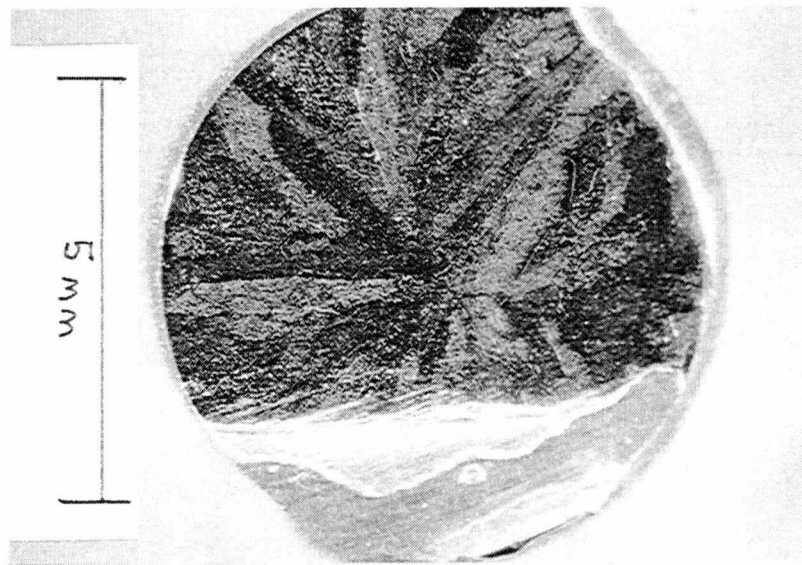
(d)

Fig. 6-1. (continued) Microstructures of cast alloys: (c) 43 at.% Al and (d) 48 at.% Al alloys.

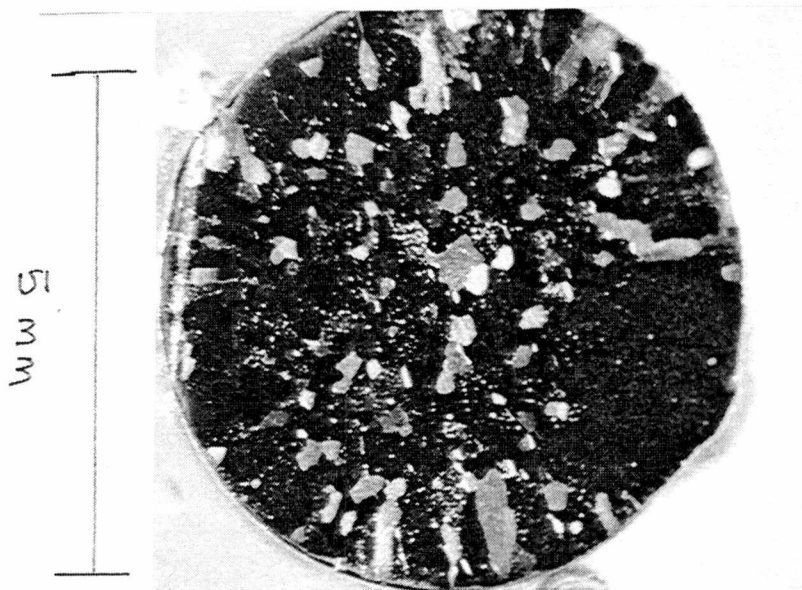
that the surfaces of the alloy specimens had also darkened and their surface texture had become rougher. In addition, very large grains were visible on the specimen surfaces. This may be indication that some vaporization had occurred on the specimen surfaces resulting in thermal etching at the grain boundaries. If vaporization did occur, then it might account for the darkening of the quartz glass if metal atoms were able to diffuse into the glass structure. The machined surfaces did not have the dark coloration.

The microstructures after homogenization and slow cooling treatments are shown in Fig. 6-2. Coring is no longer visible and substantial grain growth had occurred in the 30, 43, and 48 at.% Al alloys. These results indicate that the specimens were homogenized by the heat treatment. The two 30 at.% Al alloys were found to be ferromagnetic at room temperature while the other alloy compositions were not.

The results of the elemental impurity analysis are shown in Table 6-2. The oxygen content was measured using neutron source activation and is considered accurate to within 3%. Nitrogen was determined by chemical separation, while the remaining elements were determined by spark spectrographic analysis. The spectrographic analysis results showed that the only elements present in measurable quantities were copper, nickel, and silicon. The levels of these impurities present in the alloys are considered low. The levels of Al and Fe in the specimens were not determined and are assumed to be the starting amounts of both these elements prior to melting.

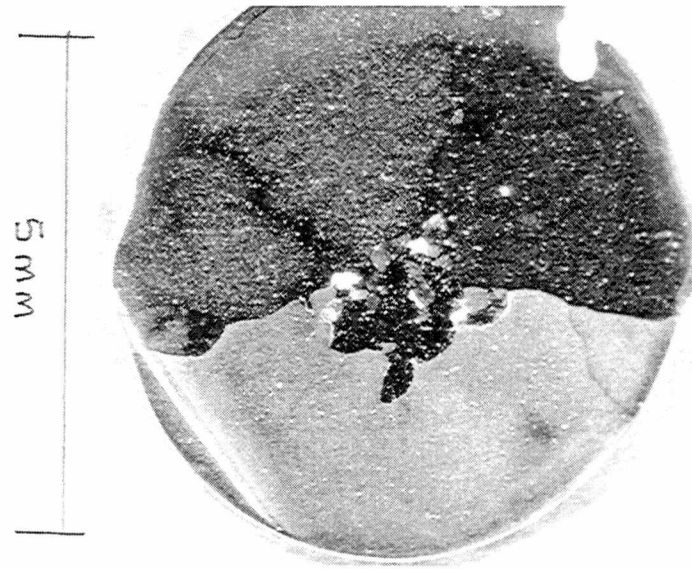


(a)

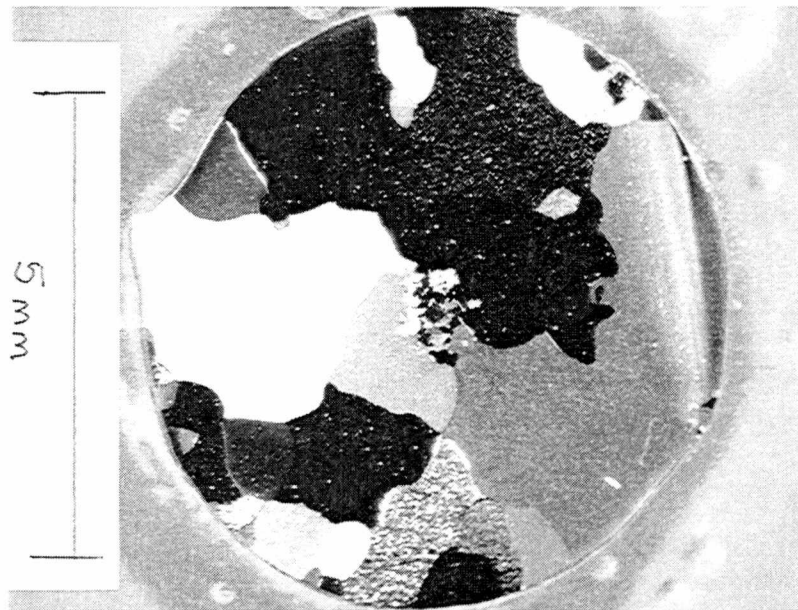


(b)

Fig. 6-2. Microstructures of alloys after homogenization and step cooling:
(a) 30 at.% Al and (b) 38 at.% Al alloys (continued on next page).



(c)



(d)

Fig. 6-2. (continued) Microstructures of alloys after homogenization and step cooling: (c) 43 at.% Al and (d) 48 at.% Al alloys.

Table 6-2. Impurity analysis for Batch #2 cast alloys
(impurity levels are expressed as at. ppm).

Element	30 at.% Al	38 at.% Al	43 at.% Al	48 at.% Al
Oxygen	31.6	28.6	51.6	57.7
Nitrogen	11.1	5.4	3.4	6.9
Copper	10	10	14	11
Nickel	10	6	<8	490
Silicon	41	52	88	75

Electrical Resistivity Measurements

Electrical Resistivity Data at 25°C

Fe₃Al Alloy. Data from other sources (39, 40, 42, 45, 47) show that ρ increases with increasing Al content for Fe₃Al alloys (Fig. 6-3). The data point for our 30 at.% Al alloy falls in the trend band. This increase in ρ with increasing Al content corresponds to the reduction in the degree of order as the composition deviates from the stoichiometric Fe₃Al composition (25 at.% Al). At 25°C the position of the phase boundary between FeAl and Fe₃Al is unknown. However, a currently assessed phase diagram (4) positions the boundary at 36 at.% Al at 400°C, which is shown in Fig. 6-3. The trend of the resistivity increase with Al content indicates that, at the phase boundary, the resistivity of FeAl and Fe₃Al are close to the same value. It is important to note that the results for Fe₃Al in Fig. 6-3 are for a wide variety of heat treatments, which are listed in Table 6-3. The significance of this is commented on in the following section.

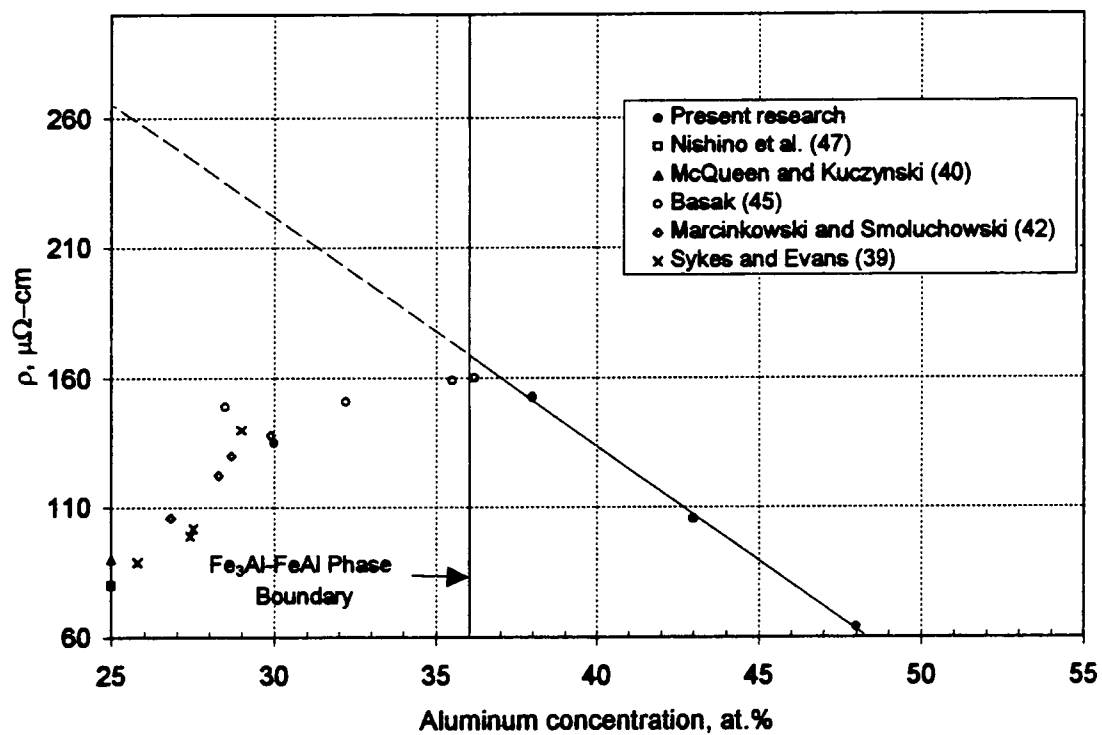


Fig. 6-3. Electrical resistivity of Fe_3Al and FeAl at 25°C .

Table 6-3. Summary of heat treatments for references used in Fig. 6-3.

Reference	Heat Treatment
Basak (45)	Heated to 1000°C then cooled at 1.5°C/s
Nishino (47)	Heated to 1000°C then slow cooled
McQueen and Kuczynski (40)	Heated to 1100°C for 24 hours, annealed at 250°C for 28 hours
Marcinkowski and Smoluchowski (42)	Heated to 300°C for one hour then quenched
Sykes and Evans (39)	Heated to 700°C for 20 minutes then cooled at 30°C/hour

FeAl Alloys. The electrical resistivities at 25°C for the annealed alloys are presented in Fig. 6-3 as a function of Al concentration. The decrease in ρ is associated with increasing improvement in proper site occupation as the composition approaches stoichiometric FeAl. This is illustrated by the schematic diagrams in Fig. 6-4. In these diagrams, it is assumed that the vacancy concentration is negligible. As the Al content increases, the number of clusters of Fe-Fe nearest neighbor bonds decreases. If these act as sources of electron scattering, then the resistivity will decrease. A comparison of the electrical resistivities of the FeAl (B2) structure and the Fe₃Al (D0₃) structure for compositions between 25 and 36 at.% Al can be made by extrapolating the ρ data for FeAl in Fig. 6-3. Note that the extrapolated resistivity of the FeAl B2 structure, for a 25 at.% Al alloy, is about three times higher than that of the D0₃ structure. However, this extrapolation is discussed relative to the ρ -T extrapolation in a later section.

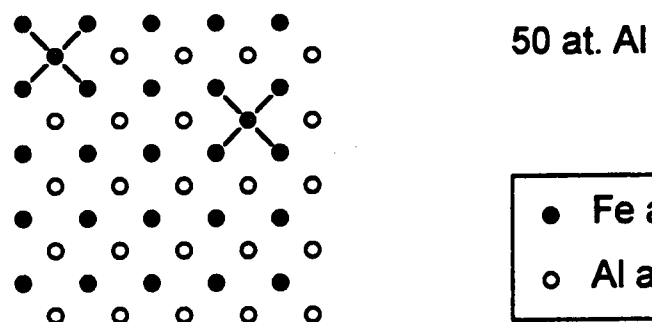
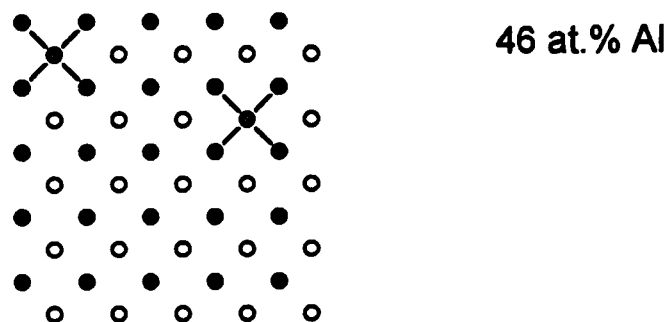
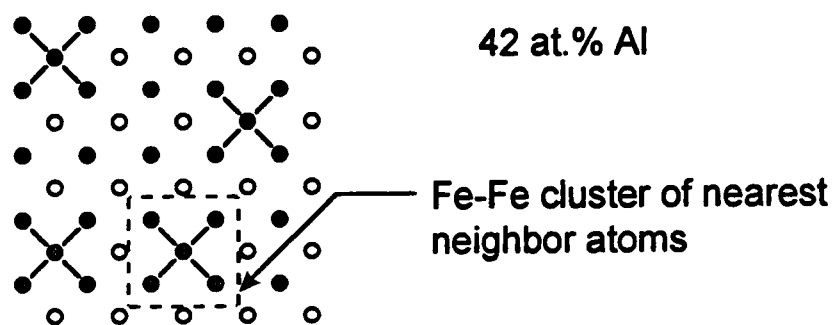


Fig. 6-4. 2-D schematic representation of the crystal structures of FeAl as a function of composition.

Further explanation of the low temperature resistivity behavior for Fe_3Al and FeAl is provided by the Mott s-d electron scattering model for transition metals and alloys (53). In the Mott model, the high resistivity associated with the transition metals is due to interactions of the 4s electrons with the holes of the 3d shell (state). Fe is a transition element in which the 3d electron state is only partially filled (contains 4 holes) as shown in Fig. 6-5. The 4s shell is the lowest energy band and, therefore, provides the conducting electrons.

According to the Mott model, the s-electrons scatter due to fluctuations in the local environment by structural disorder, and this scatter causes a conducting s-electron to jump into a vacant d-state, thereby increasing the resistivity. Increasing the structural disorder increases the scattering, and therefore increases the probability that an s-electron will jump into a vacant d-state, and thus resistivity increases with increased disorder. This relationship corresponds to the data shown in Fig. 6-3. The resistivity (at 25°C) for both Fe_3Al and FeAl increases with increased disorder which is caused by deviations from their stoichiometric compositions (25 at.% Al for Fe_3Al and 50 at.% Al for FeAl). For the FeAl structure, the resistivity increase resulting from the scatter of electrons by the Fe-Fe clusters is also a valid explanation. Mott's model is considered to be relatively independent of temperature (although it does predict that the Fermi level will increase with temperature) and cannot therefore be used to describe the ρ -T behavior for these alloys (53, 54).

Shell	K	L		M		N	
Subshell	1s	2s	2p	3s	3p	3d	4s
13 Al	2	2	6	2	1		
21 Sc	2	2	6	2	6	1	2
22 Ti	2	2	6	2	6	2	2
23 V	2	2	6	2	6	3	2
24 Cr	2	2	6	2	6	4	1
25 Mn	2	2	6	2	6	5	2
26 Fe	2	2	6	2	6	6	2
27 Co	2	2	6	2	6	7	2
28 Ni	2	2	6	2	6	8	2
29 Cu	2	2	6	2	6	10	1
30 Zn	2	2	6	2	6	10	2

Fig. 6-5. Electron states for the first period of transition metals plus aluminum.
Unfilled states are double framed.

Pulse-Heating Electrical Resistivity Data

Fe₃Al (30 at.% Al). The temperature dependence of ρ for the Fe₃Al alloy (30 at.% Al) is shown in Fig. 6-6. The resistivity increases to a maximum at 485°C, where there is a slight discontinuity at which ρ decreases. It then continues to decrease slightly to the limit of measurement (~1150°C). The shape of the curves closely resembles that reported for this and similar Fe₃Al compositions (34, 36, 45, 47). Note that our specimens were heated much faster (between 50 and 70°C/s) than those of previous studies (e.g., 3°C/min).

For a stoichiometric Fe₃Al alloy, Nishino et al. (47) attributed the maximum to the ferromagnetic-paramagnetic change, which has a Curie temperature close to 485°C. However for the 30 at.% Al alloy, the Curie temperature is reported to be close to 200°C (4). Typically an inflection in the ρ -T curve occurs at the Curie temperature for ferromagnetic materials. However, such an inflection is not present in the data shown in Fig. 6-6. Cahn and Feder (37) have argued that the maximum is the result of two competing phenomena. At temperatures below the maximum, phonon scattering predominates which causes ρ to increase, while at higher temperatures the resistivity decrease associated with increased conduction electrons becomes the overriding factor. This resistivity maximum has been reported for other D0₃ and L1₂ intermetallics having a transition metal component (48, 49).

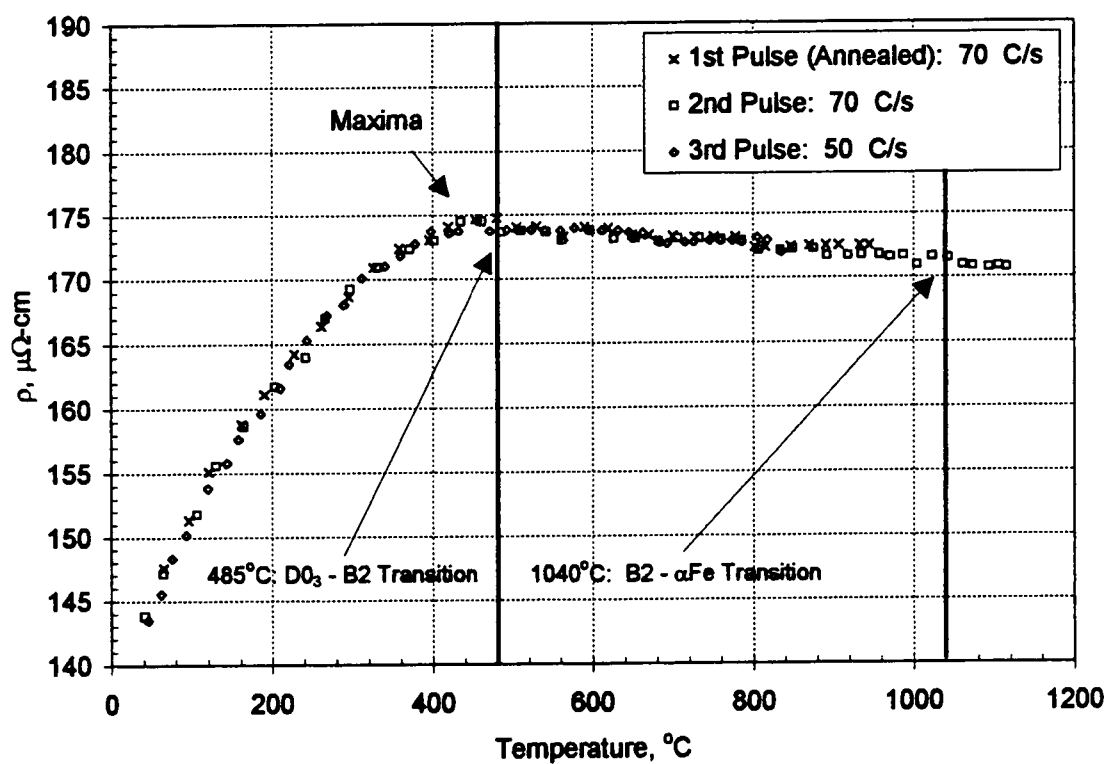


Fig. 6-6. Resistivity of the 30 at.% Al alloy as a function of temperature and heating rate.

The aforementioned investigations based their interpretations that the ρ -T maximum occurs before the structure changes from $D0_3$ to B2, and hence the comments do not address the decreasing resistivity in the higher temperature range (see below). For the 30 at.% Al alloy, the $D0_3$ -B2 transition temperature occurs near 480°C in the evaluation of Kattner and Burton (4) which is in good agreement with the discontinuity at 485°C in Fig. 6-6. Thus this feature is taken to be associated with this phase change. At high temperature, the 30 at.% Al alloy changes from the B2 structure to the disordered α Fe solid solution. Kattner and Burton give this temperature as 1070°C, although there exists considerable uncertainty in the value of this transition. There is no clear indication in the data in Fig. 6-6 of a phase change in this range. In contrast, iron undergoes a fcc to bcc change at 1400°C which causes a change in resistivity of about 2% (51).

Once the alloy transformed to the B2 structure (at about 485°C), the resistivity decreases slightly with temperature. Similar behavior has been reported for stoichiometric Fe_3Al (45). This effect is discussed in the section with the behavior of the higher Al alloys, which have only the B2 structure.

We now turn to an examination of the kinetics of the structural changes. There are two considerations here: 1) the initial degree of order and the accompanying lattice defect concentration, and 2) the formation of these defects on rapid heating and retention during cooling. Keep in mind that the initial annealing of the specimen was designed to induce maximum order and minimum (essentially zero) vacancy retention.

It is to be noted that the data in Fig. 6-6 are identical for either the initial annealed condition (prior to pulse) or cooled at about 1.5°C/s from 950 to 1100°C . Also note that the data are identical for heating rates from 50 to 70°C/s . In addition, the resistivity at 25°C for the Fe_3Al alloys in Fig. 6-3 is for a variety of heat treatments, most involving aging treatments or relatively slow cooling. The data at 25°C from our research, for the annealed condition and after cooling from high temperature at about 1.5°C/s , also agree with the results in Fig. 6-3.

The similarity of the ρ -T curves for various heat treatments implies that the transformation from the ordered B2 structure to the D0_3 is quite rapid. This is confirmed by aging studies of similar Fe_3Al alloys by Branski and Rudman (41), McQueen and Kuczynski (40) and Sykes and Evans (39). They show that cooling from the B2 region (e.g., 1000°C) at about 1°C/s or slower will result in the D0_3 structure. Much faster cooling (e.g., water quenching) is required to retain the B2 structure. For Fe_3Al alloys for which this has been done, the resistivity at 25°C versus quenching temperature curve is similar in shape to that in Fig. 6-6, but the values are lower by about $15\mu\Omega\text{-cm}$.

Kim and Morris (43) showed recently that for a similar composition the D0_3 structure begins to disorder between 300 and 350°C . However, there is no anomaly on the ρ -T curve associated with this feature.

These results show that the ρ -T curve is characteristic of the structure at each temperature. That is, on heating rapidly (e.g., 70°C/s) the kinetics of disordering are sufficiently rapid to maintain the equilibrium degree of order. Such rapid kinetics are probably related to the highly-ordered structures of these alloys and are discussed in further detail in a later section.

FeAl (30, 38, 43, and 48 at.%Al). The ρ -T data for the higher Al alloys (38, 43, and 48 at.% Al) are shown in Fig. 6-7 through 6-9. According to the phase diagram of Kattner and Burton (4), these compositions all lie in the B2 structure region for our temperature range of measurement (up to ~1000°C). A phase change to the disordered α Fe structure occurs above the B2 region. Kubaschewski (55) indicates a more complex behavior involving phase changes within the temperature range of measurements. However, the curves in Figs. 6-7 through 6-9 do not show any indication of a high temperature phase change.

The data for the initial annealed condition are compared in Fig. 6-10. The curve for the 30 at.% Al alloy is included since it has the B2 structure above 485°C. The resistivity at low temperature decreases with Al content (see also Fig. 6-3), but the curves cross over, so that at high temperature the resistivity increases with increasing Al content. The temperature coefficient of resistivity (TCR) is plotted versus Al content in Fig. 6-11. It is interesting to note that the TCR values are low and decrease with decreasing Al content. The significance of this is discussed in detail in a later section.

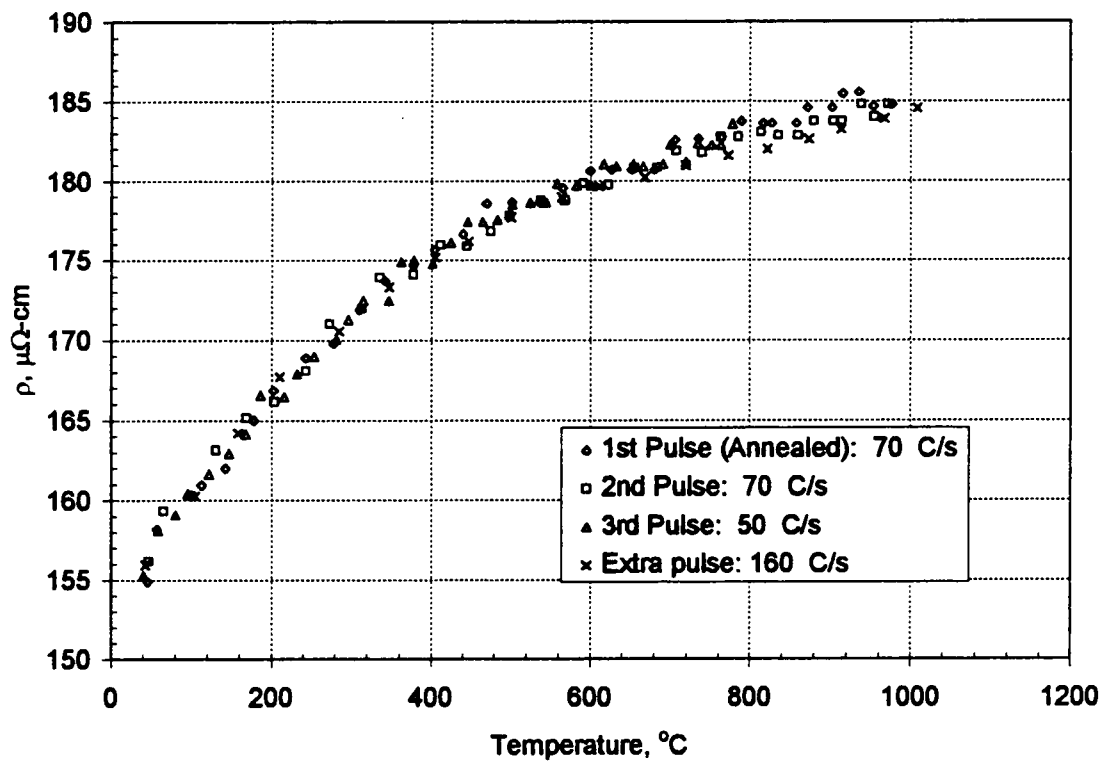


Fig. 6-7. Resistivity of the 38 at.% Al alloy as a function of temperature and heating rate.

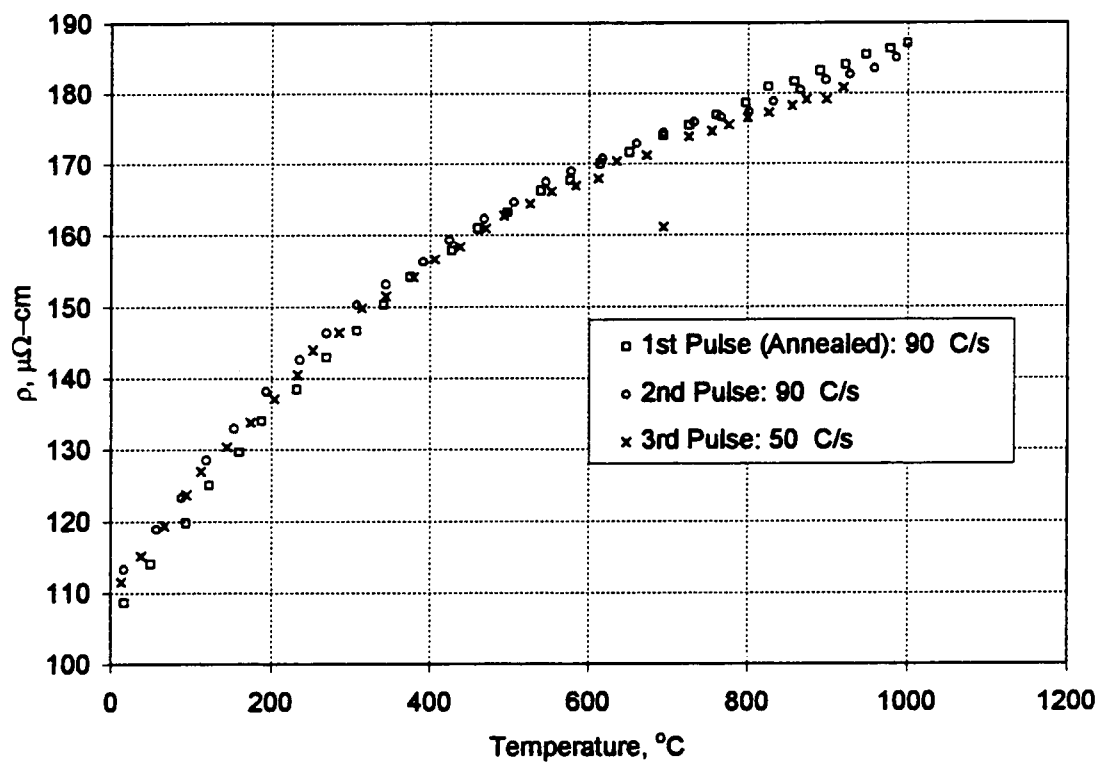


Fig. 6-8. Resistivity of the 43 at.% Al alloy as a function of temperature and heating rate.

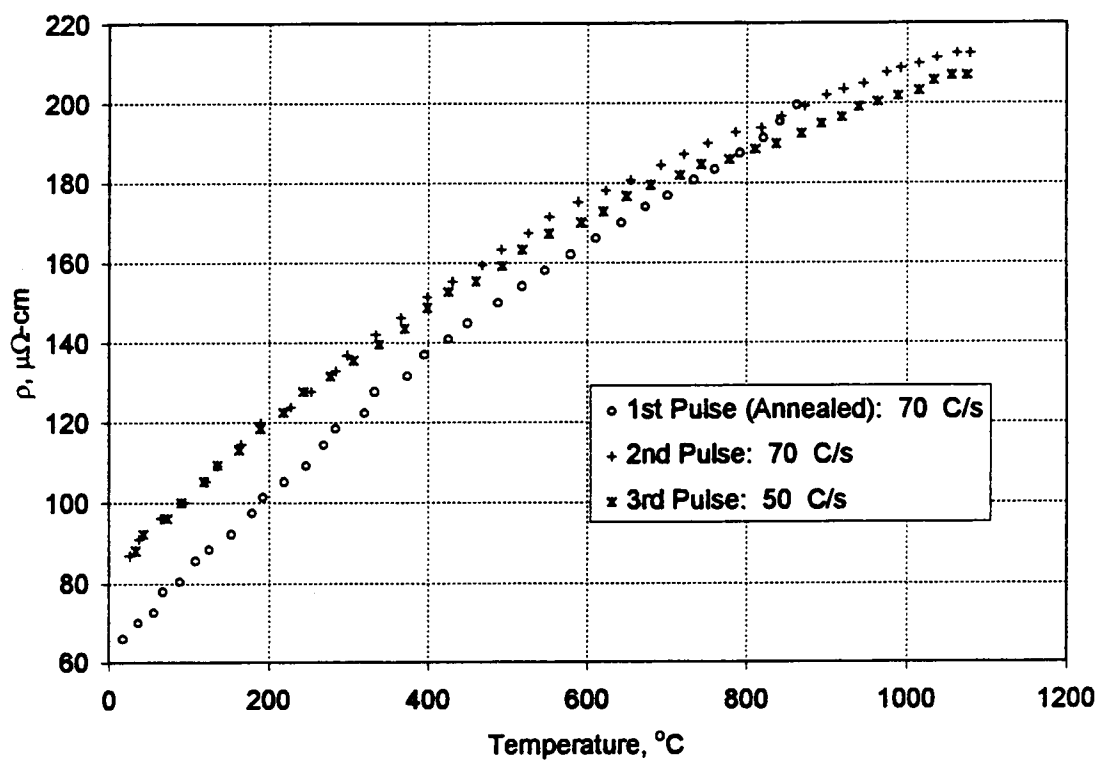


Fig. 6-9. Resistivity of the 48 at.% Al alloy as a function of temperature and heating rate.

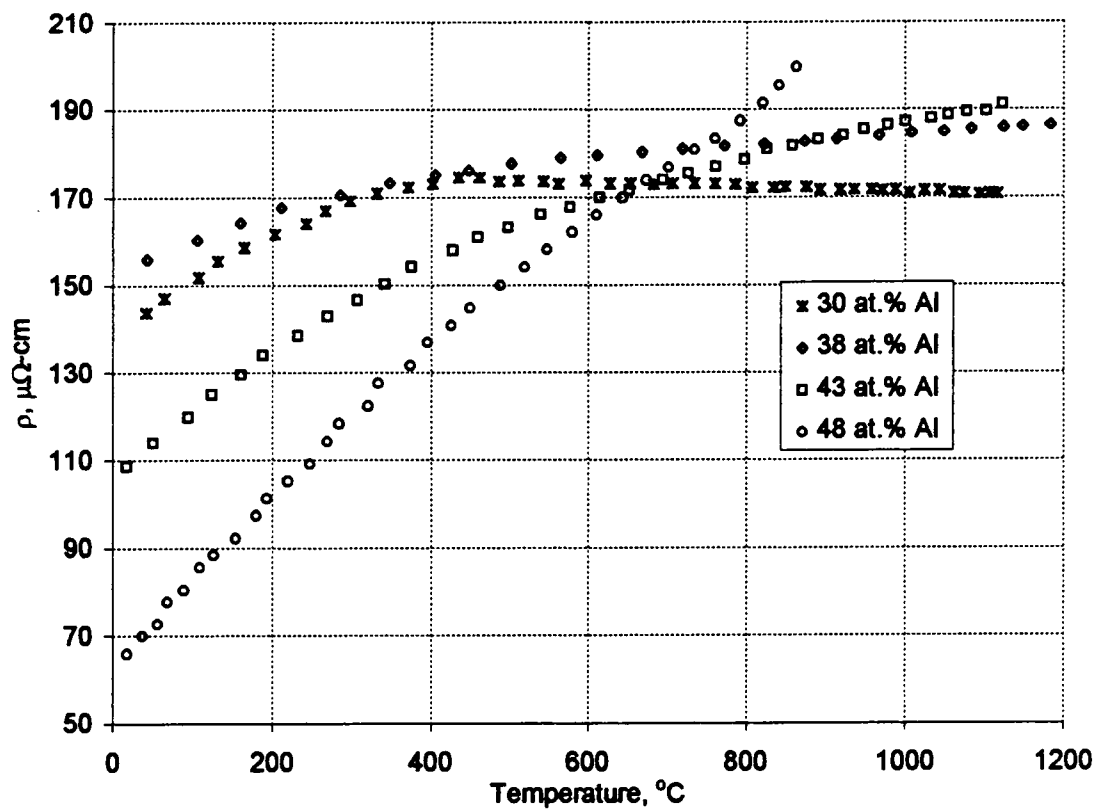


Fig. 6-10. ρ -T curves for annealed Fe_3Al and FeAl alloys.

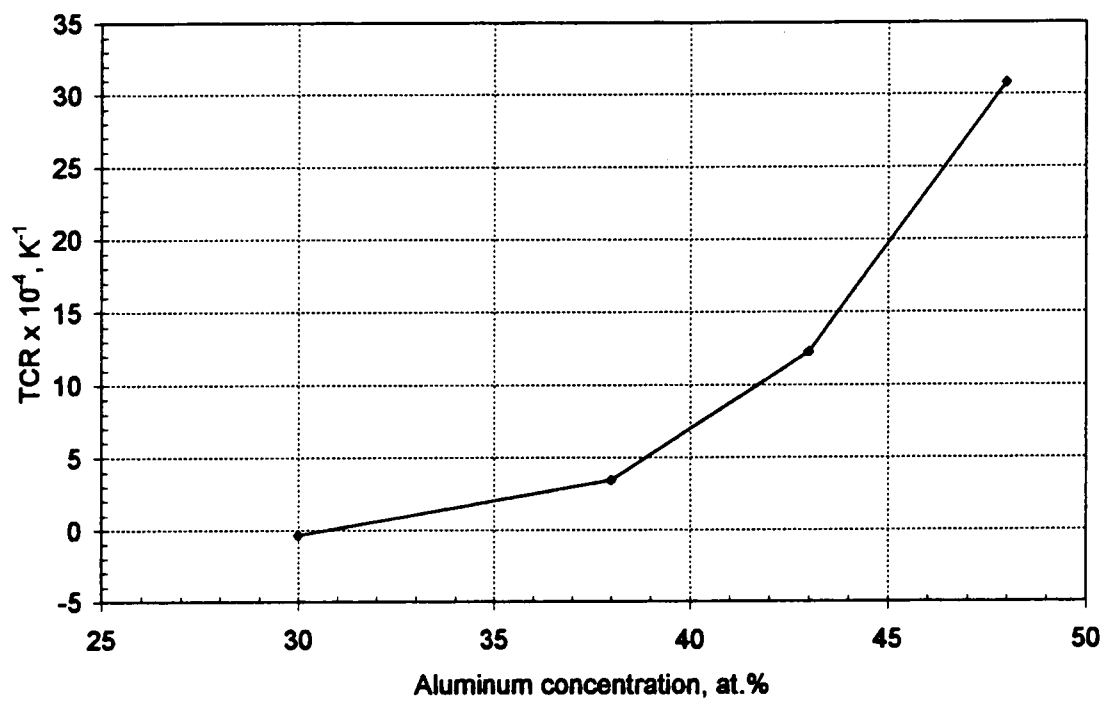


Fig. 6-11. Approximate TCR for annealed FeAl alloys.

For the 38 and 43 at.% Al alloys, there is little difference between the ρ -T data for the initial annealed condition and for cooling from high temperature (e.g. 1000°C) after the first pulse (Figs. 6-7 and 6-8). However, the 48 at.% Al alloy shows a measurable increase in the resistivity after an initial pulse to high temperature (Fig. 6-9). This difference becomes less at high temperature. (The difference between the second and third pulse data is probably due to specimen buckling.) For all of the alloys tested, there was no effect of heating rate on resistivity for the heating rates tested. This effect is examined further in the following discussions on the specific heat results.

Analysis of the FeAl Defect Structure

To assist in the explanation of the resistivity behavior of these alloys as a function of composition and temperature, we have provided two-dimensional schematic diagrams of the atomic arrangement for two FeAl alloys. Both low and elevated temperature arrangements of Fe and Al atoms on the B2 lattice structure are shown in Fig. 6-12 for a 37.5 at.% Al alloy and in Fig. 6-13 for a 47.2 at.% Al alloy. The location of excess Fe atoms on the Al sublattice was randomly selected for the low temperature structures. For the purposes of discussion, the room temperature diagrams in Figs. 6-12 and 6-13 represent the annealed structures as ideal crystals in which excess Fe atoms occupy Al sublattice positions. Comparison of the low temperature structures shows that the degree of order is higher for the 47.2 at.% alloy, which is closer to stoichiometry than the 37.4 at.% Al alloy, and therefore ρ is lower. This is consistent with our earlier

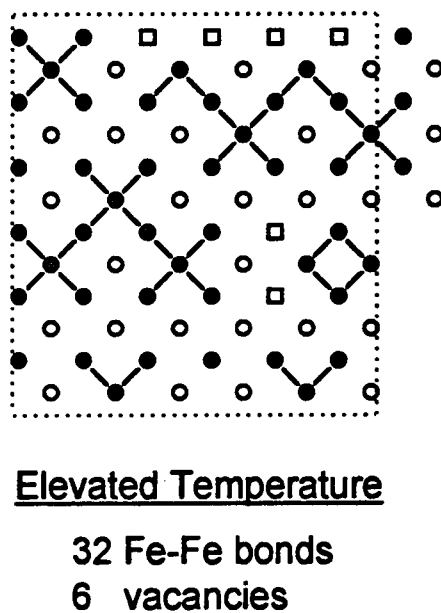
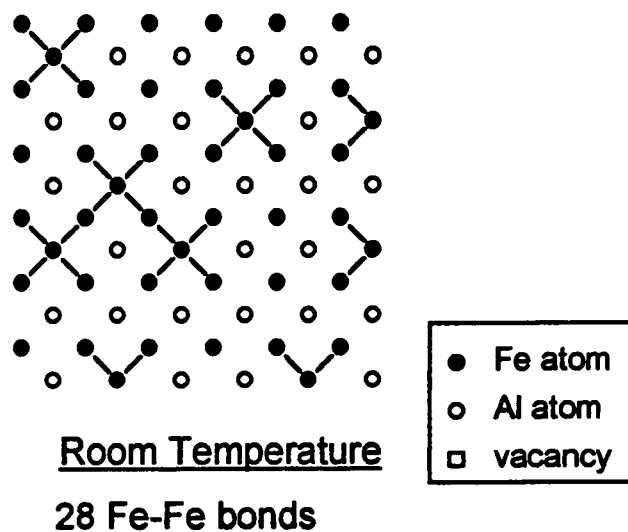
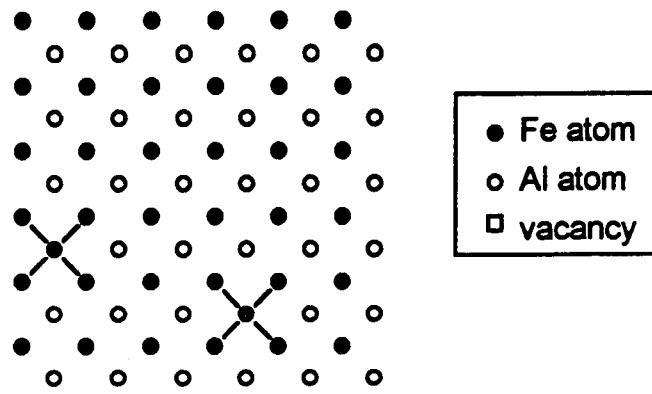
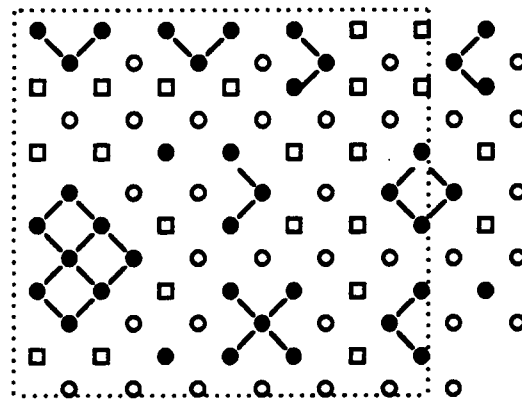


Fig. 6-12. 2-D schematic representation of the crystal structures of FeAl (37.5 at.% Al) at low and elevated temperatures.



Room Temperature

8 Fe-Fe bonds



Elevated Temperature

30 Fe-Fe bonds

22 vacancies

Fig. 6-13. 2-D schematic representation of the crystal structures of FeAl (47.2 at.% Al) at low and elevated temperatures.

assessment of the low-temperature resistivity behavior (Fig. 6-3). The elevated temperature diagrams were determined using two simplifying assumptions:

1. Triple defects are the only defect type formed. Several studies (15-17) have shown that for FeAl triple defects predominate at elevated temperature. As a result, Fe atoms jump to the Al sublattice creating a vacancy. This is demonstrated in the diagrams by arrows that show the direction of the Fe atom movement. To maintain composition, an additional vacancy is located on the Fe sublattice adjacent to the antisite Fe atom.
2. The number of vacancies adjacent to any Fe atom is limited to two. This assumption prevents the occurrence of an Fe atom being surrounded completely by vacancies. The presence of divacancy formation in these structures was noted by a previous investigation (30).

The structural diagrams depicted in Figs. 6-12 and 6-13 show that the number of vacancies formed at elevated temperatures rises with increasing Al concentration and for the composition near stoichiometry (47.2 at.% Al) is quite large. This analysis corresponds to experimental and theoretical studies of vacancy formation of FeAl. Chang et al. (14) calculated the vacancy concentration as a function of temperature and Al content. There is a marked increase in the vacancy concentration as the stoichiometric 50 at.% Al is approached. Xiao and Baker (12) obtained similar results. They also determined the Fe antisite atom concentration and showed that it decreases

almost linearly as the Al content increases, approaching close to zero at stoichiometric FeAl. Our analysis also corresponds to the theoretical model developed by Chang and Neumann (17) for B2 phases exhibiting triple defect formation. Further evidence that the thermal vacancy concentration was exceptionally high for the near stoichiometric alloy (48 at.% Al) can be obtained by comparing the resistivities in the annealed state to the resistivity data obtained following the first pulse. For the 38 and the 43 at.% Al alloys, the resistivity data were unchanged. However, for the 48 at.% Al alloy (see Fig. 6-9), the resistivity increased 30% from 63 to 86 $\mu\Omega$ -cm.

It is important to note that if the resistivity was influenced by electron scattering at vacancies, then the rise in resistivity should be proportional to the vacancy increase with temperature. However, the resistivity curves for these alloys do not show a dramatic upswing associated with vacancy formation. Instead, the slope decreases with increasing temperature. Kim and Morris (43) determined that for the B2 phase, atomic disorder (or vacancies) begins to form on the α sublattice around 500°C. However, the ρ -T curves for these alloys do not contain any anomaly that corresponds with this feature. This is discussed in more detail in the later section on specific heat.

Comparison with Disordered or Amorphous Transition Metals and Alloys

Before further discussing the ρ -T curves for Fe₃Al and FeAl, a brief description of the ρ -T behavior for disordered or amorphous transition metals and alloys is needed. Disorder is defined as random atom arrangement within a crystal lattice. The ρ -T behavior of

disordered or amorphous transition metals or alloys is peculiar in that it almost always contains the following general features (54, 56, 57):

1. the residual resistivities (the resistivity at 0°C, denoted as ρ_o) are high ($\rho_o > 40 \mu\Omega\text{-cm}$),
2. the temperature coefficients of resistivity (TCR) are small and are always positive for $\rho_o < 150 \mu\Omega\text{-cm}$, and negative for $\rho_o > 150 \mu\Omega\text{-cm}$, and
3. the alloys exhibit resistivity saturation at high temperatures.

As we will demonstrate in the following discussion, each of the above features occurs for FeAl even though it has an ordered B2 structure.

The residual resistivities were determined for each FeAl alloy by extrapolation of the ρ -T curves in Fig. 6-10 to 0°C. For the 30 at.% Al alloy, only the region that was in the FeAl phase was used for the extrapolation. Note that this extrapolation gives a resistivity value at 25°C that is considerably less than the one determined from the Fig. 6-3 extrapolation. The TCRs were determined for 25°C and are plotted against ρ_o to obtain the Mooij plot for FeAl shown in Fig. 6-14. The Mooij plot is a graphical representation of the empirical relationship of TCR with ρ_o and is associated with the second feature listed above (54, 57). For disordered or amorphous transition metals and alloys, the TCR always decreases as the ρ_o increases and is zero at approximately $150 \mu\Omega\text{-cm}$. As

shown in Fig. 6-14 for FeAl, the ρ_o values are high ($\rho_o > 40\mu\Omega\text{-cm}$) and the TCR decreases with increasing ρ_o similar to that of disordered or amorphous transition metals or alloys (54). The TCR values for FeAl are also positive for $\rho_o < 150\mu\Omega\text{-cm}$ and negative for $\rho_o > 150\mu\Omega\text{-cm}$.

The third feature is the resistivity saturation phenomena which is depicted in Fig. 6-15. As the temperature increases, the resistivities for each curve all converge to a single value (usually around $150\mu\Omega\text{-cm}$). The resistivity saturation phenomena is not understood but it is theorized that d-band conduction or electron-phonon hopping may be responsible (54).

For FeAl, the ρ -T curves in Fig. 6-10 resemble the resistivity saturation phenomena but differ in that they do not converge to a single value. Instead they cross each other at elevated temperature. This crossover effect can be explained as the result of thermal vacancy formation, which increases the resistivity. This additional contribution to resistivity increases the slope of the ρ -T curve causing the crossover and preventing each curve from approaching a single value. It is known that the formation of thermal vacancies increases with Al concentration. When this effect is taken into account, the curves in Fig. 6-10 more closely resemble the resistivity saturation behavior illustrated in Fig. 6-15.

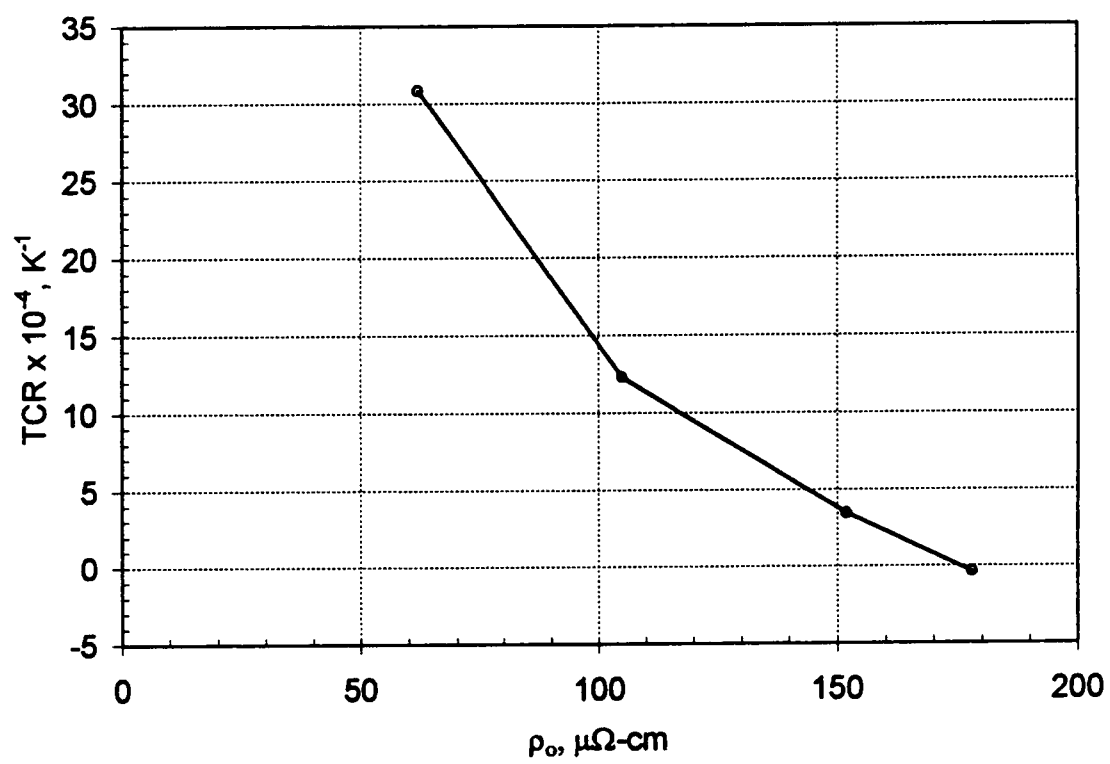


Fig. 6-14. Mooij plot for FeAl.

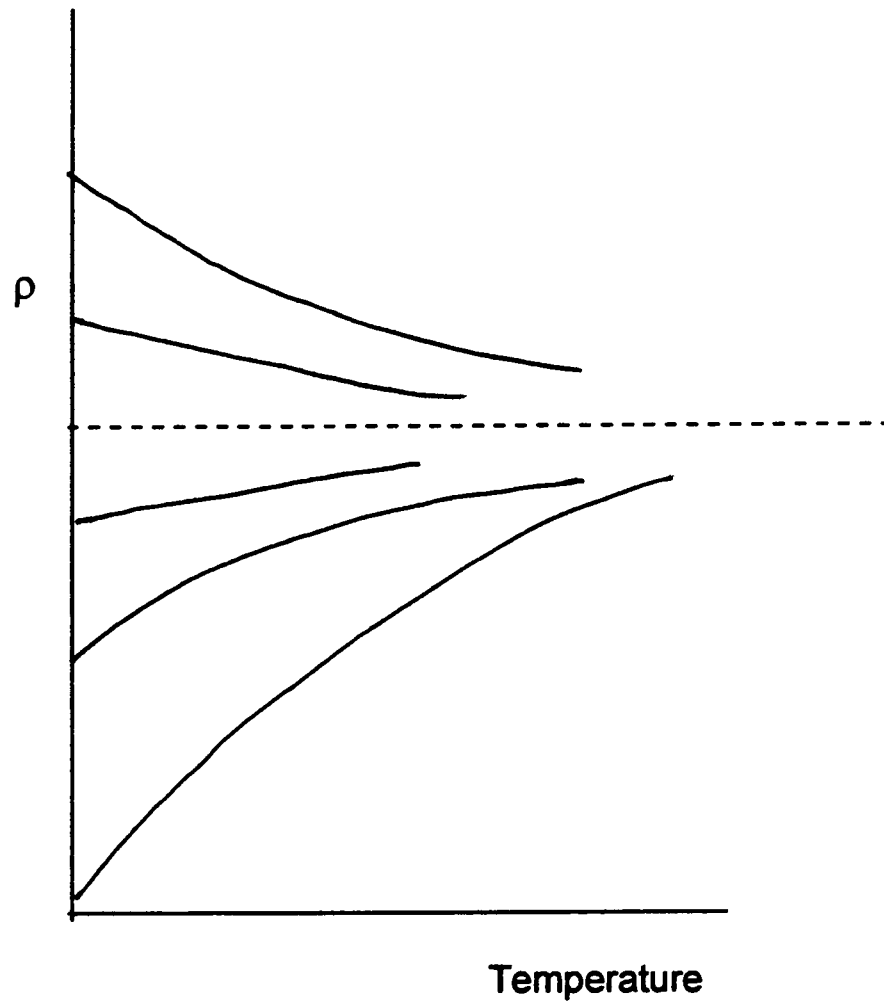


Fig. 6-15. Resistivity saturation in a disordered or amorphous transition metal or alloy.

The general features of the ρ -T curves for FeAl correspond to those observed for disordered metals and alloys containing a transition element. Because the FeAl B2 structure consists of a highly ordered α -sublattice and a disordered β -sublattice, these results indicate that the resistivity for this alloy is dominated by the disorder in the β -sublattice. If the vacancies on the α -sublattice contributed significantly to ρ , then ρ should increase in proportion to n_v . However, this is clearly not so. As a result, the ρ -T behavior is unaffected by heating rate (see Figs. 4-7 through 4-9). In addition, the resistivity is not especially sensitive to retained thermal vacancies in the cooled structure, except for the 48 at. % Al alloy, in which the retained thermal vacancy concentration was exceptionally high.

This study represents the first time that the resistivity saturation phenomena has been noted for an ordered B2 structure. Triple defect formation is likely responsible for this behavior since vacancies do not form on the β sublattice. The important discovery of the saturation phenomena in FeAl alloys has three important consequences:

1. Resistivity measurements were made to high temperatures, which for amorphous structures is limited because recrystallization cannot be prevented.
2. The saturation of the resistivity at high temperatures means that FeAl alloys have use for applications where an approximately constant resistivity is required.

3. Resistometry studies of FeAl at high temperature will have to consider the resistivity saturation phenomena when using resistivity measurements to study defect concentrations and kinetics.

Specific Heat Measurements

Baseline Extrapolation

The general shape of the experimental C_p -T curves was similar to those obtained for refractory metals such as tungsten. Between 275 and 475°C (550 and 750K), the C_p rise with temperature was linear, but at higher temperatures the C_p rise was much faster. The linear region is believed to represent the C_p contributions from dilation, electronic, and lowest-order anharmonics. This region was extrapolated to higher temperatures by performing a linear regression. The baseline is important for two main reasons: 1) the thermal defect contribution of the C_p can be determined by subtracting the experimental value from the baseline value at a given temperature and, 2) the temperature at which thermal defects begin to form for a given alloy and test condition can be determined as the point at which the experimental C_p -T curve deviates from the baseline curve. This temperature is designated as T_D in the following discussion.

Effect of Composition

The experimental C_p - T curves for the alloys in the annealed condition are shown in Fig. 6-16. The heating rate for these alloys averaged $73 \pm 3^\circ\text{C/s}$, except for the 43 at.% Al alloy which was heated at 90°C/s . The results show that the dramatic upswing in C_p occurs at decreasing temperature with increasing Al content. The extrapolated baseline linear region for each alloy is shown in Figs. 6-17 through 6-20 along with the respective experimental C_p - T curve. Using these curves, T_D was determined and is listed in the Table 6-4 below for each alloy. The values for T_D are considered approximate because of the errors associated with baseline extrapolation and visual determination of the separation point.

Table 6-4. Defect formation temperatures for the annealed alloys.

Alloy	T_D ($^\circ\text{C}$)
30 at.% Al	680
38 at.% Al	530
43 at.% Al	530
48 at.% Al	480

As shown in Table 6-4, T_D for the 38, 43, and 48 at.% Al alloys does not change significantly with composition. These three alloys are entirely within the FeAl phase or B2 structure. The T_D is in agreement with the findings of Paris and Lesbats (16) and Kim and Morris (43). In a neutron powder diffraction study, Kim and Morris (43) found that disorder begins on the α sublattice of the B2 structure around 500°C . They define disorder as the concentration of Fe atoms on the α sublattice, with the remainder being vacancies. Therefore, by their definition, disorder describes the vacancy defect

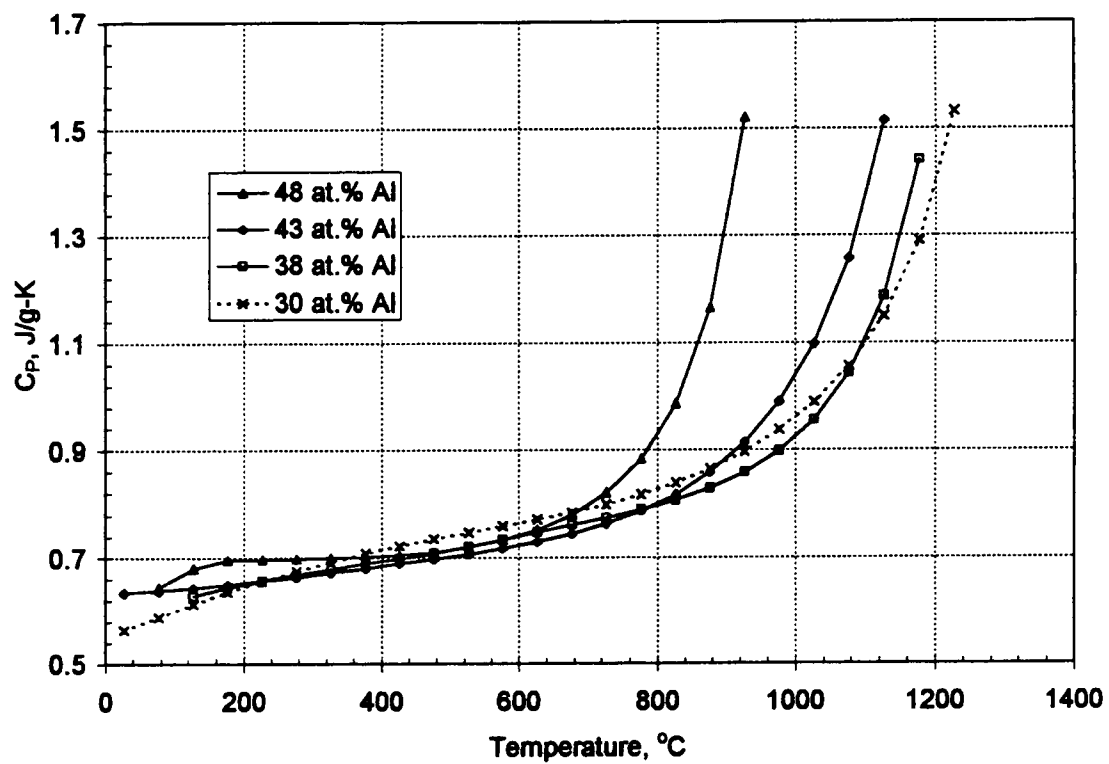


Fig. 6-16. C_p -T curves for annealed FeAl and Fe₃Al alloys.

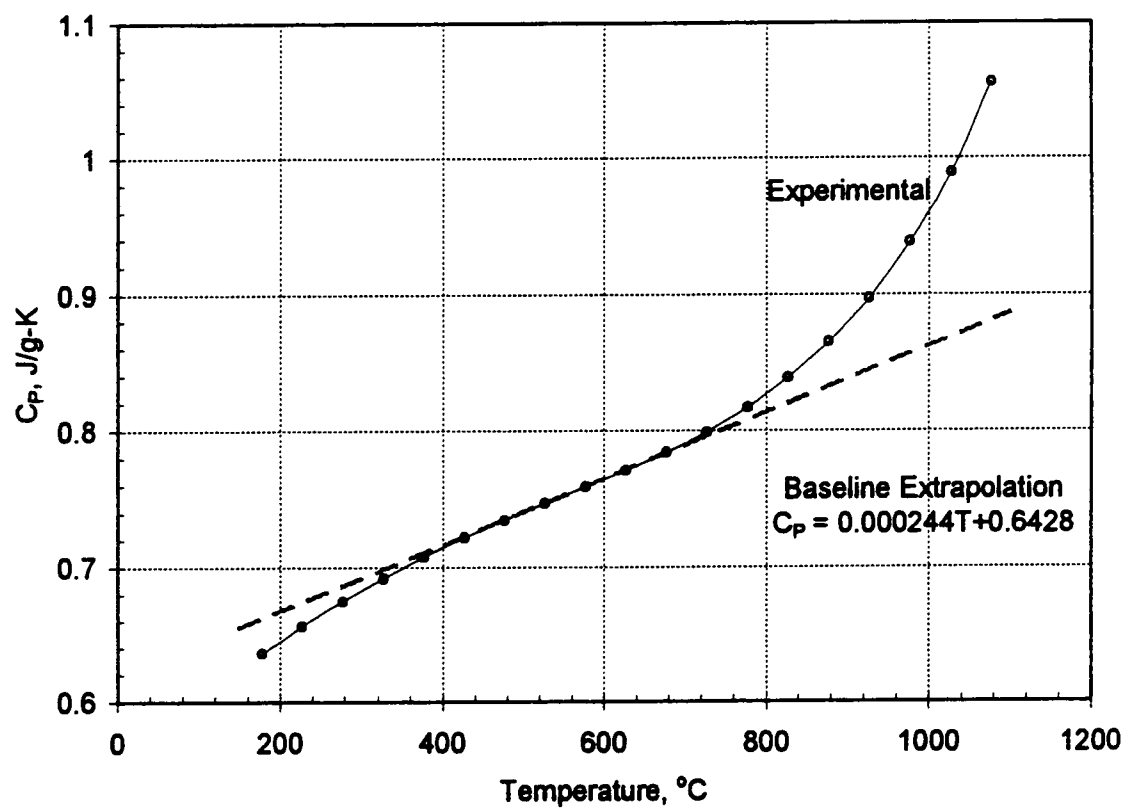


Fig. 6-17. C_p -T curve for the 30 at.% Al alloy in the annealed condition.

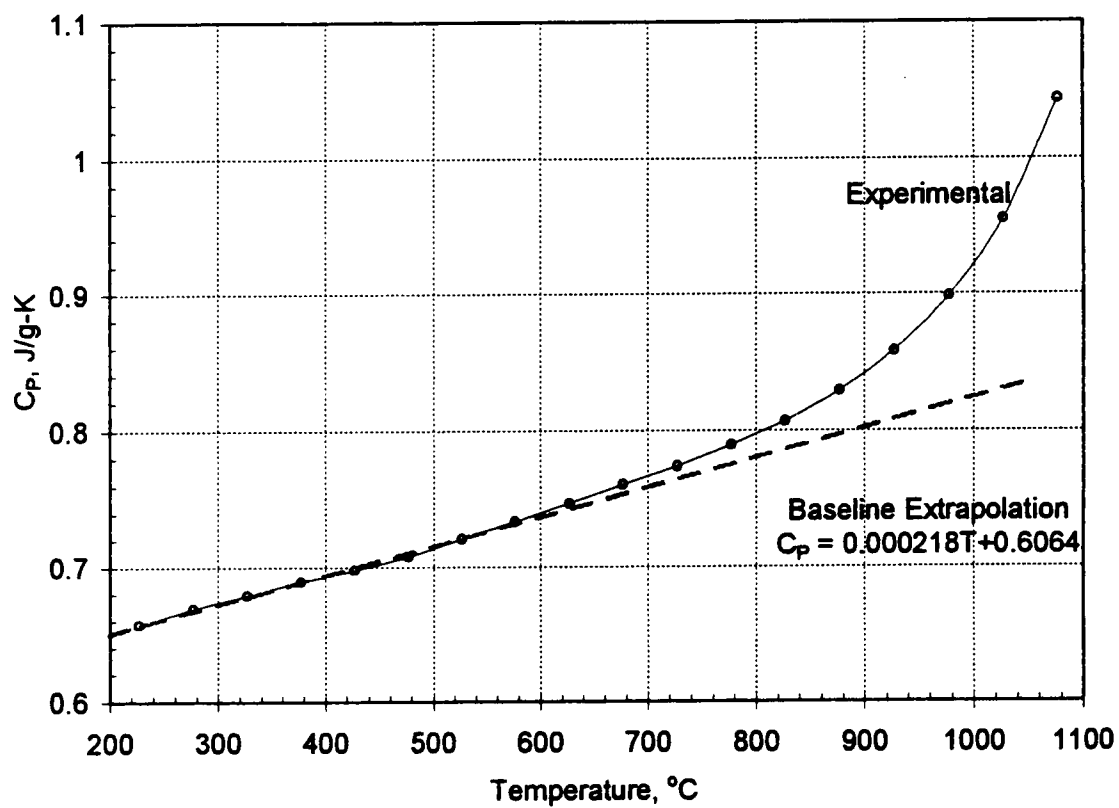


Fig. 6-18. C_p -T curve for the 38 at.% Al alloy in the annealed condition.

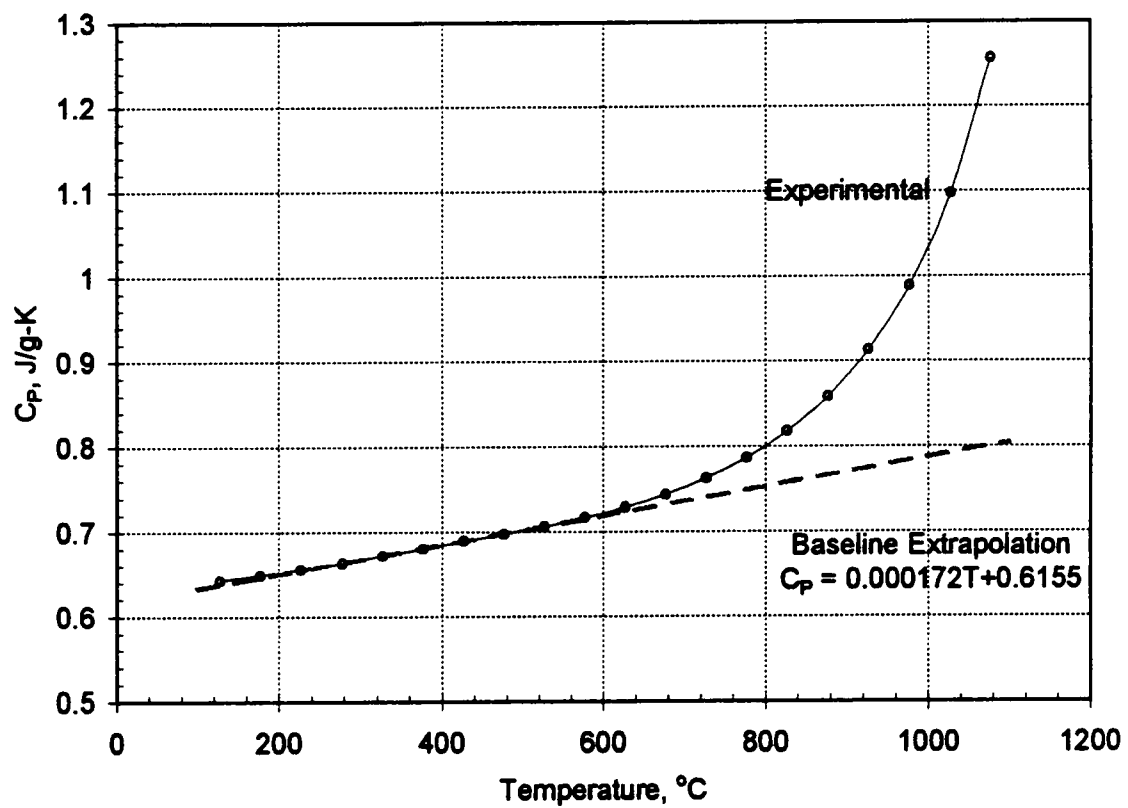


Fig. 6-19. C_p -T curve for the 43 at.% Al alloy in the annealed condition.

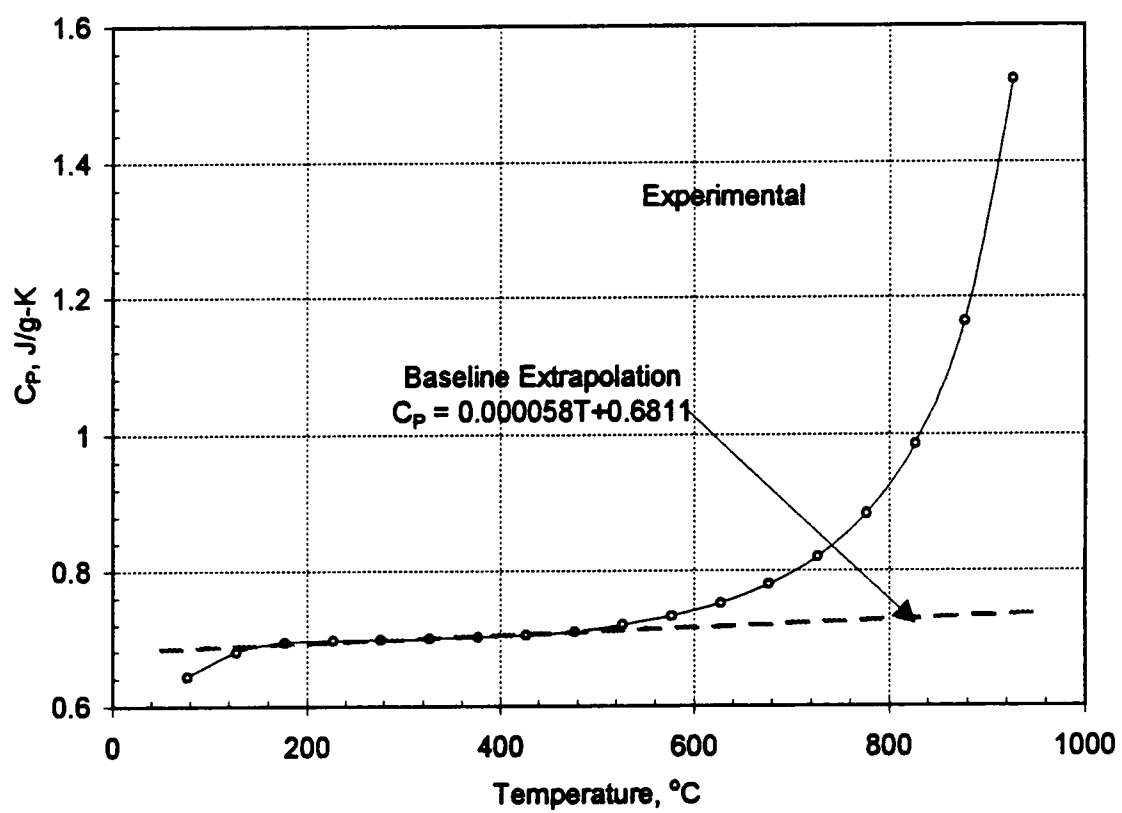


Fig. 6-20. C_p -T curve for the 48 at.% Al alloy in the annealed condition.

formation for these alloys. Our results are in good agreement with their data. In contrast, lattice parameter measurements by Ho and Dodd (15) and Pike et al. (13) have indicated that thermal vacancies begin to form in FeAl around 700°C. However this technique may not be sensitive enough to detect the onset of these defects.

It is interesting to note that the T_D value of 680°C for the 30 at.% Al alloy occurs well within the FeAl phase or B2 structure. According to a recent equilibrium phase diagram by Kattner and Burton (4), the D0₃-B2 transition for this composition occurs near 480°C. This agrees with the findings of Feder and Cahn (36), Shaefer and Würschum (44), and Kim and Morris (43) that thermal defects do form within the Fe₃Al phase or D0₃ structure. Another interesting feature is that the experimental C_P -T curve for this alloy does not show a discontinuity corresponding to the D0₃-B2 transition. This may be due to the fact that the crystal structures for D0₃ and B2 are very similar and that the transition is gradual.

Effect of Thermal Treatment

During the initial pulse the annealed specimens were heated to approximately 1000°C and then cooled at 1.5°C/s. Although it has been well established that vacancies will form in these alloys at this temperature, it is not known whether this cooling rate is fast enough to retain them. The effect of this thermal treatment was evaluated by comparing the treated C_P -T curves with the annealed C_P -T curves.

The C_p - T curves for each alloy composition in the annealed and thermally-treated conditions are shown in Figs. 6-21 through 6-24. Only the 48 at.% Al alloy showed a change in the C_p - T curve as a result of the thermal treatment (Fig. 6-24). The dramatic upswing in C_p for the annealed condition was shifted to higher temperature by the thermal treatment (the extrapolated baseline for this curve is shown in Fig. 6-25). The T_D results for the annealed and treated conditions were determined to be about 480°C and 600°C respectively. This indicates that a large concentration of thermal defects was retained in the low temperature structure during cooling. Since these defects were already present, they did not need to be reformed during heating. If the heating rate was low, the retained vacancies would anneal out at low temperature producing a dip in the C_p - T curve, as shown in Fig. 6-26. However, because the heating rate was high, the energy release associated with retained defect annealing was suppressed to high temperature. Thus, the dramatic upswing in C_p was suppressed to higher temperature. The C_p - T curves for the lower Al alloys (30, 38, and 43 at.% Al) were identical to that obtained for the initial annealed condition. This suggests that the cooling rate was not sufficiently high to retain significant concentrations of thermal vacancies for these alloys, which is a further indication that the defect concentrations for these compositions are not nearly as great as the 48 at.% Al alloy.

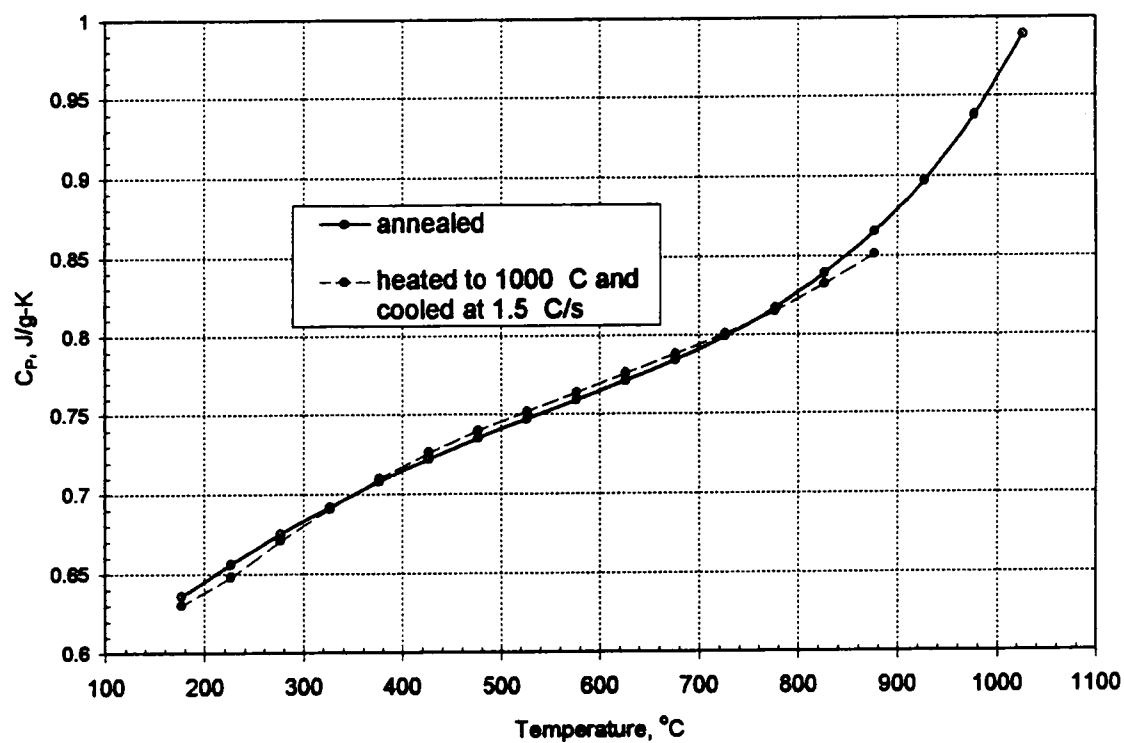


Fig. 6-21. C_p -T curves for the 30 at.% Al alloy as a function of thermal treatment.

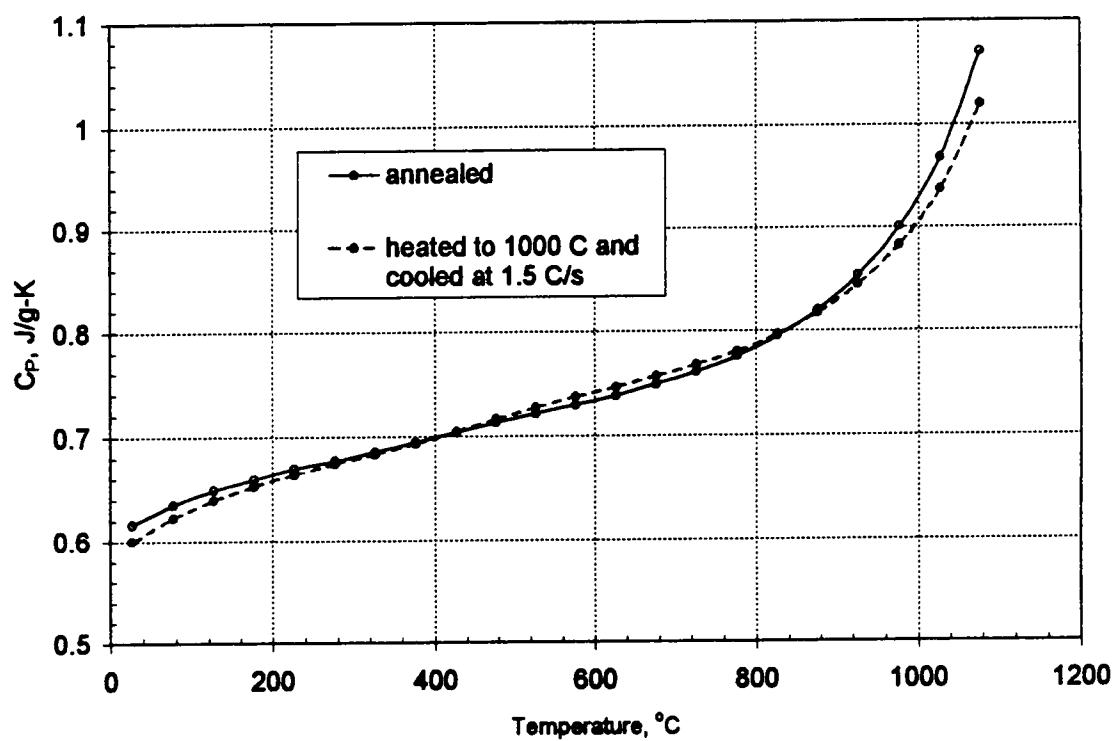


Fig. 6-22. C_p -T curves for the 38 at.% Al alloy as a function of thermal treatment.

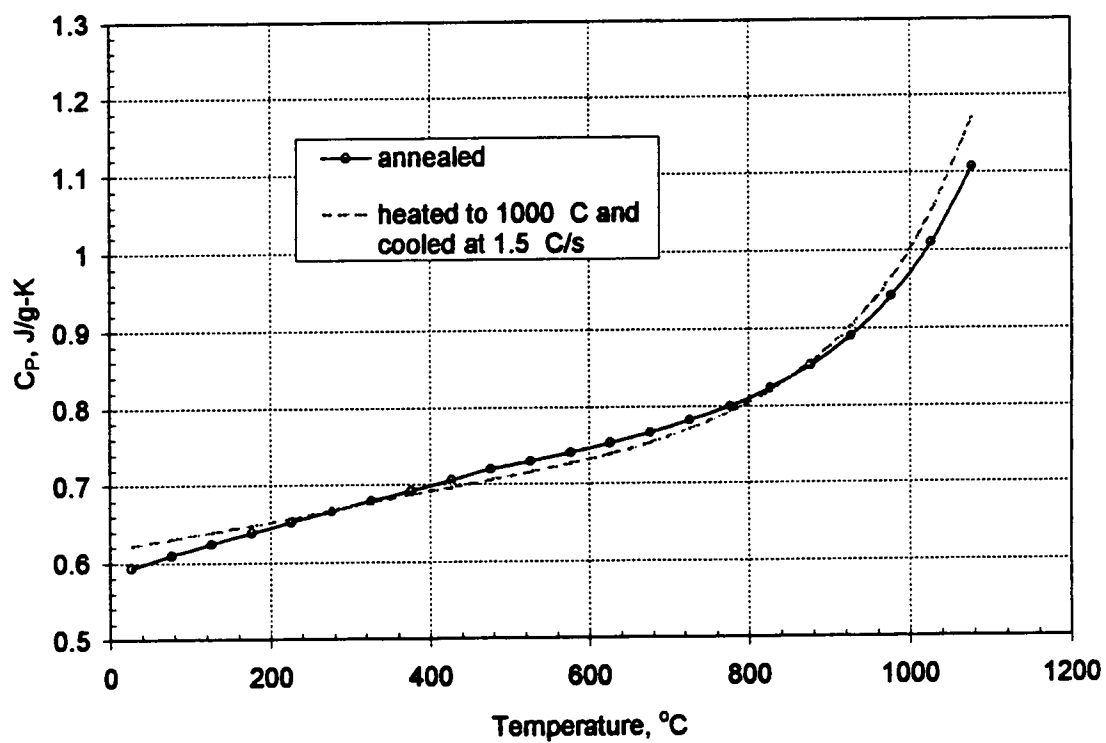


Fig. 6-23. C_p -T curves for the 43 at.% Al alloy as a function of thermal treatment.

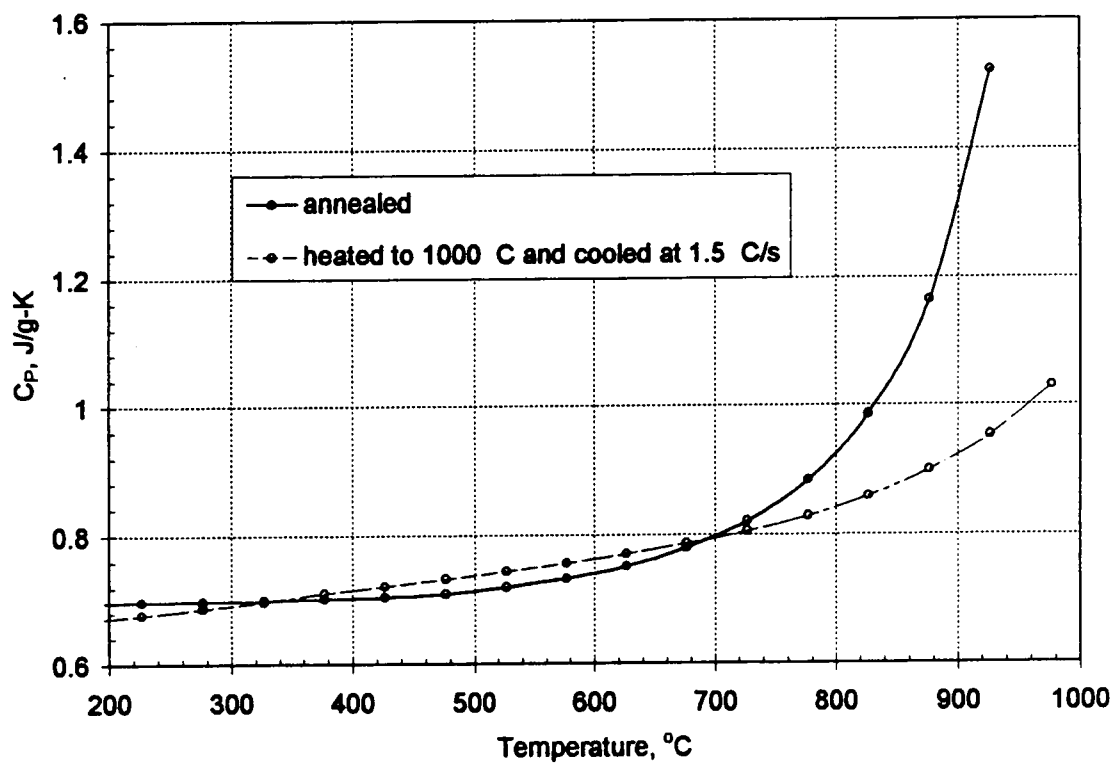


Fig. 6-24. C_p -T curves for the 48 at.% Al alloy as a function of thermal treatment.

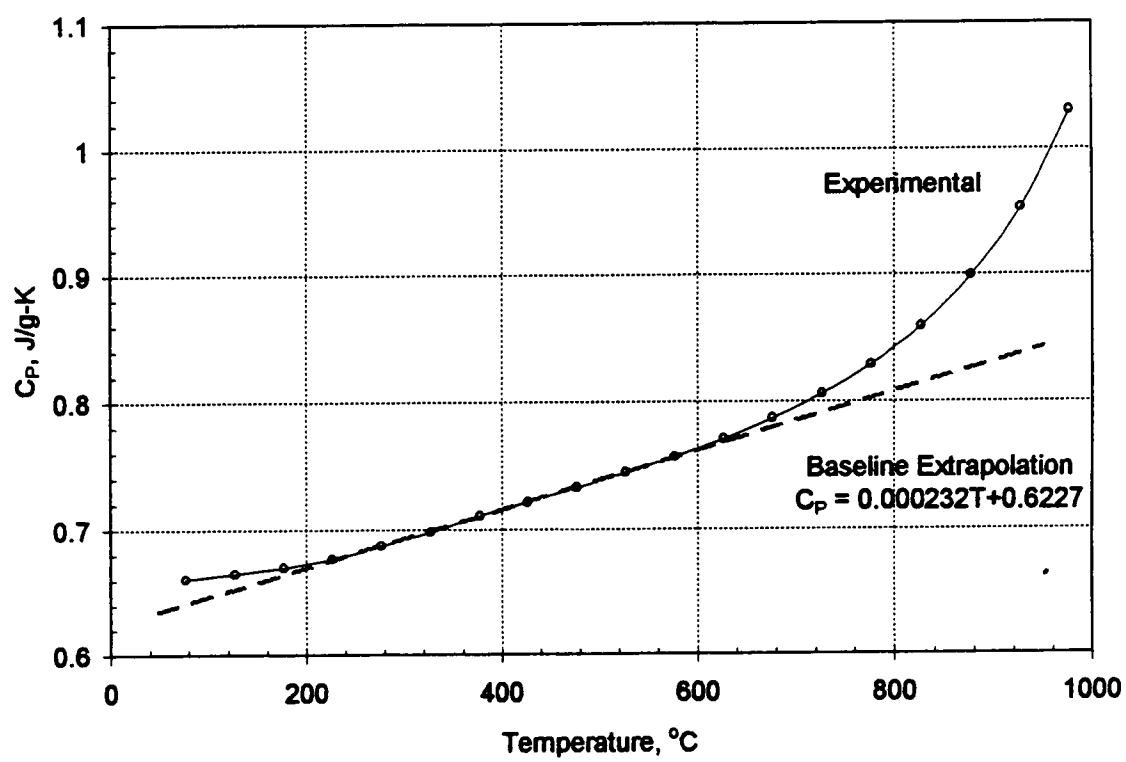


Fig. 6-25. C_p -T curve for the 48 at.% Al alloy heated to 1000°C and cooled at 25°C/s.

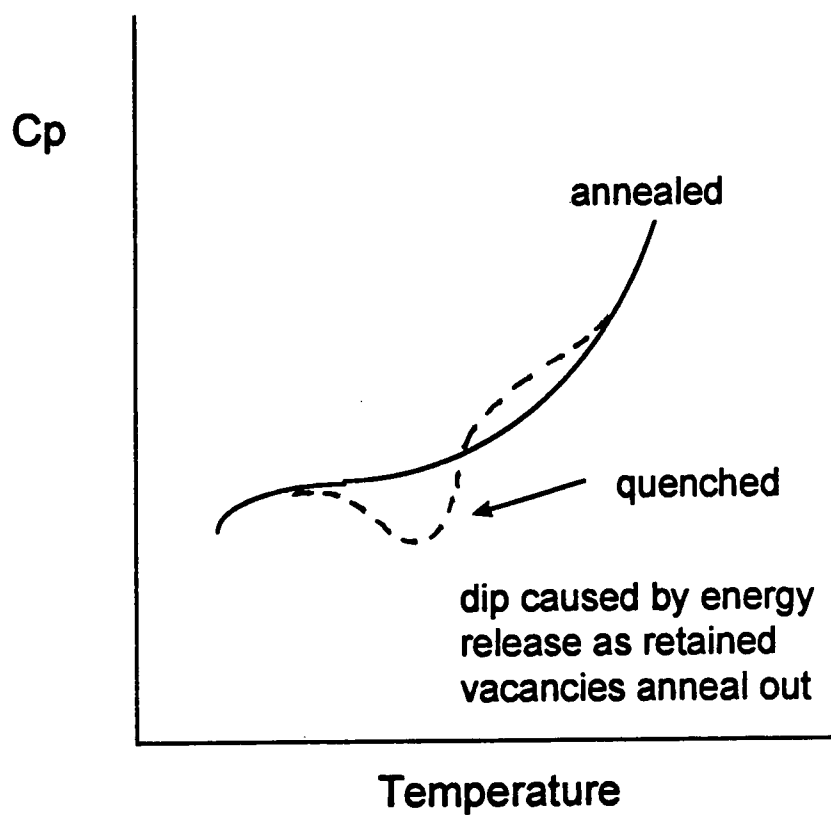


Fig. 6-26. Energy release associated with annealing of retained vacancies during slow heating.

Effect of Heating Rate

The three FeAl alloys (38, 43, and 48 at.% Al) were pulsed at various heating rates in order to determine the influence of heating rate on the C_p -T curve. These results are shown in Figs. 6-27 through 6-29. There was no observable change in the C_p -T curves with heating rate for the 43 and 48 at.% Al alloys for the rates tested as shown in Figs. 6-28 and 6-29. However, for the 38 at.% Al alloy (Fig. 6-27) the temperature corresponding to dramatic upswing in C_p increased with increasing heating rate. For this alloy the extrapolated baselines for these curves were determined (see Figs. 6-30 through 6-33) and T_D for each run is listed below in Table 6-5.

Table 6-5. Temperature of defect formation for the heating rates tested for the 38 at.% Al alloy.

Heating Rate ($^{\circ}\text{C/s}$)	T_D ($^{\circ}\text{C}$)
42	480
52	530
73	530
162	630

These results clearly show that the dramatic upswing in C_p was suppressed to higher temperatures for the 38 at.% Al alloy. However, the curves do not cross over at high temperature, as would be expected as shown in Fig. 6-34. The vacancy concentration at T_1 is the same for both heating rates. The curve for the high heating rate shows a large rise in C_p as T_2 (temperature near melting) is approached. During slower heating the curve exhibits a more gradual rise which crosses over the high heating curve, as shown in

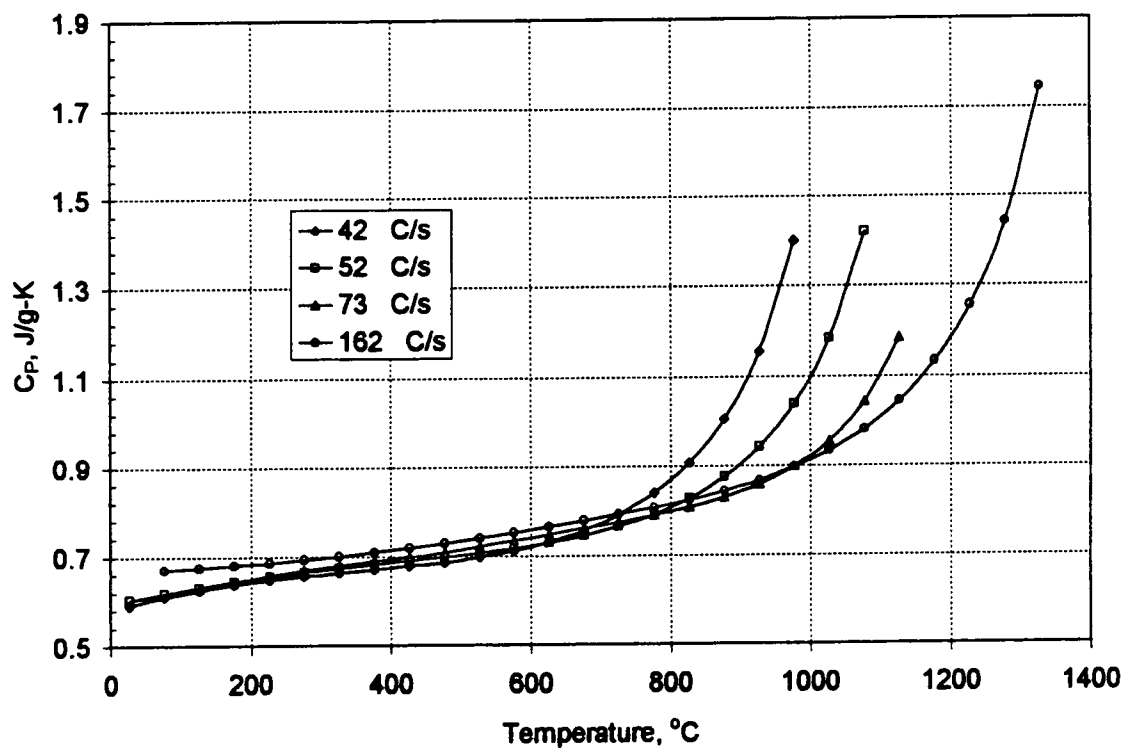


Fig. 6-27. C_P - T curves for the 38 at.% Al alloy as a function of heating rate.

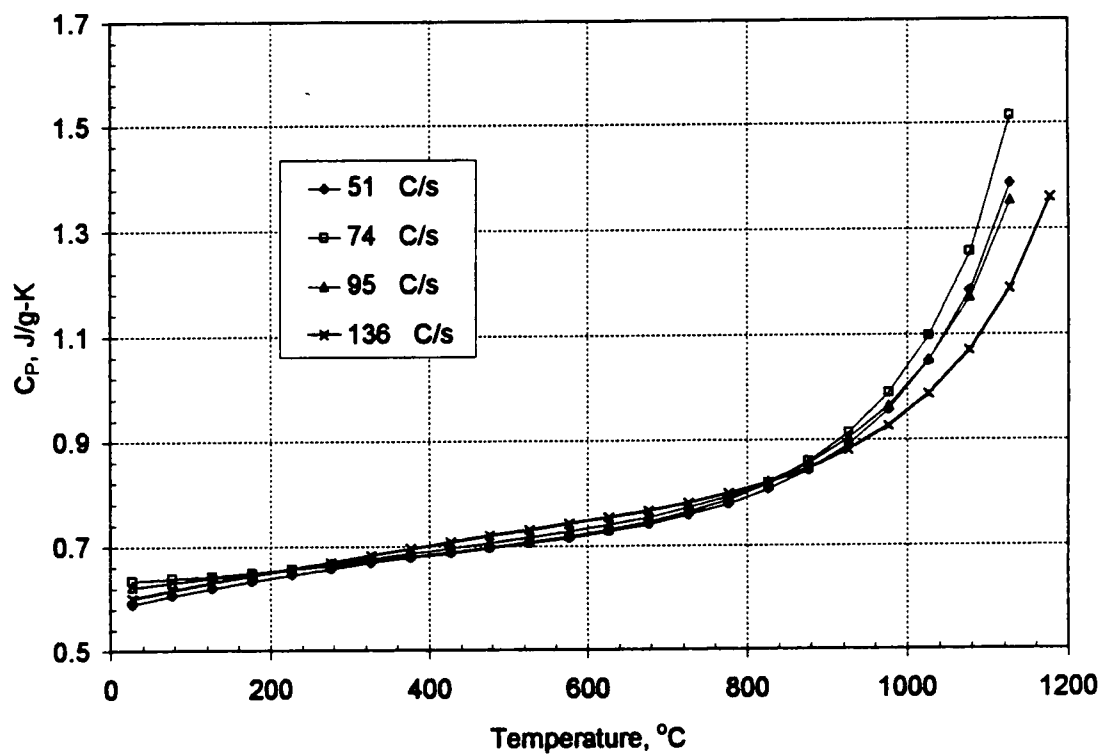


Fig. 6-28. C_p -T curves for the 43 at.% Al alloy as a function of heating rate.

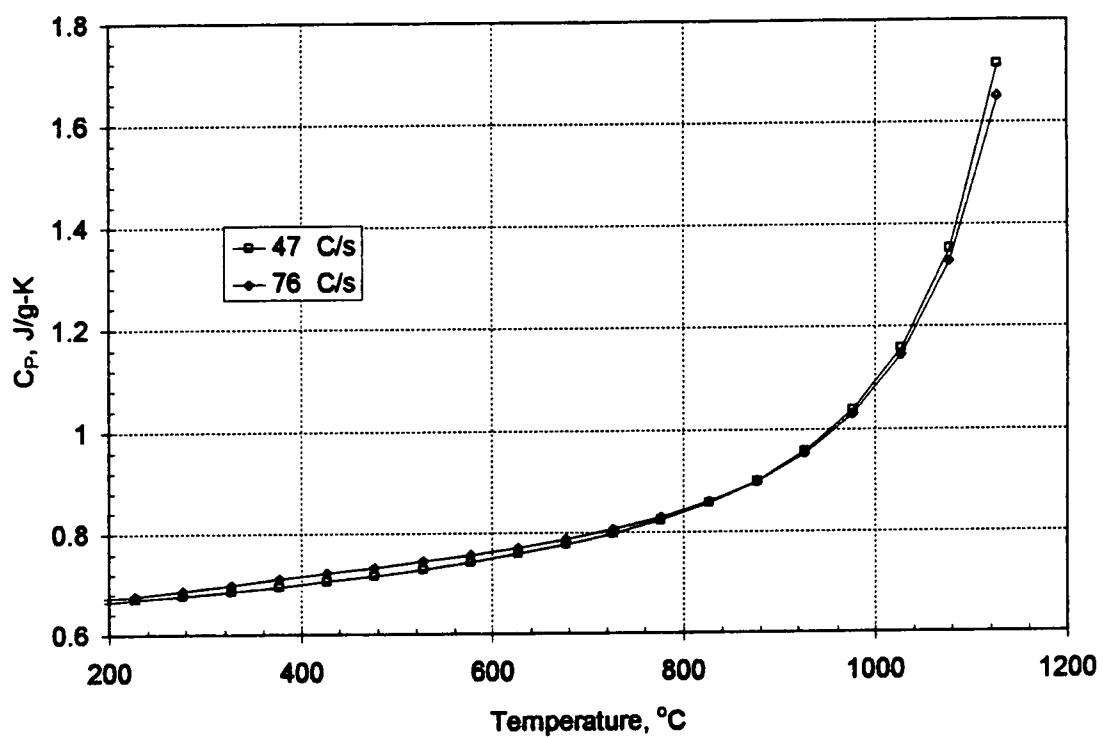


Fig. 6-29. C_p -T curves for the 48 at.% Al alloy as a function of heating rate.

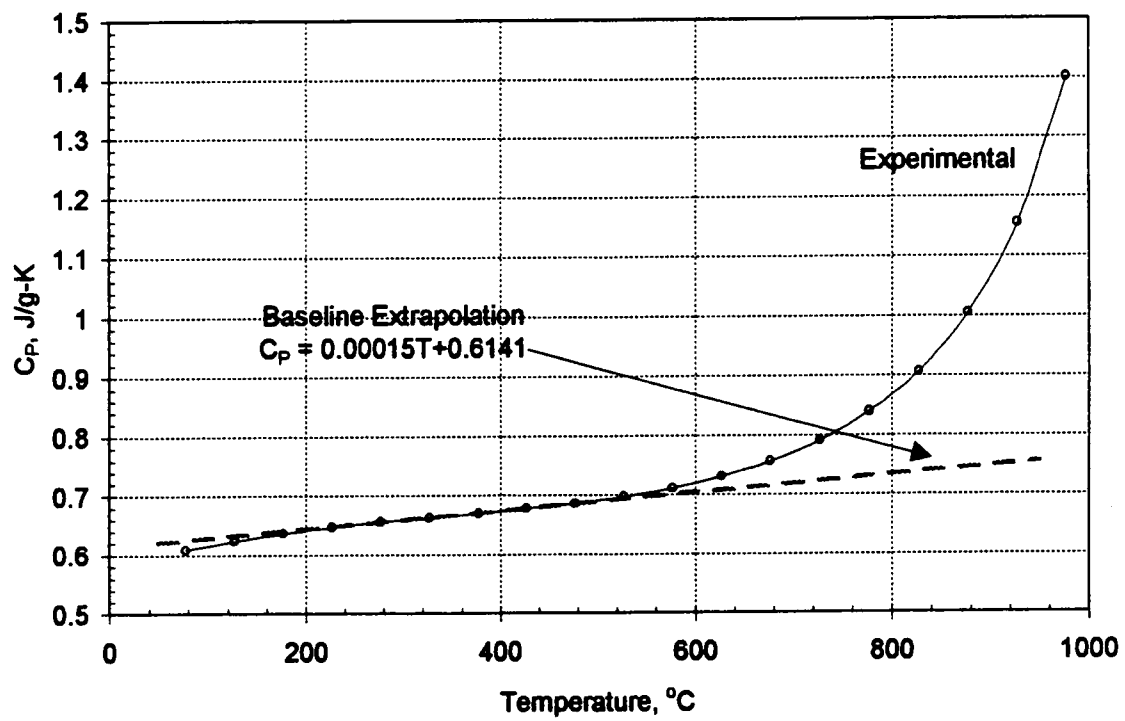


Fig. 6-30. C_p -T curve for the 38 at.% Al alloy heated at 42°C/s.

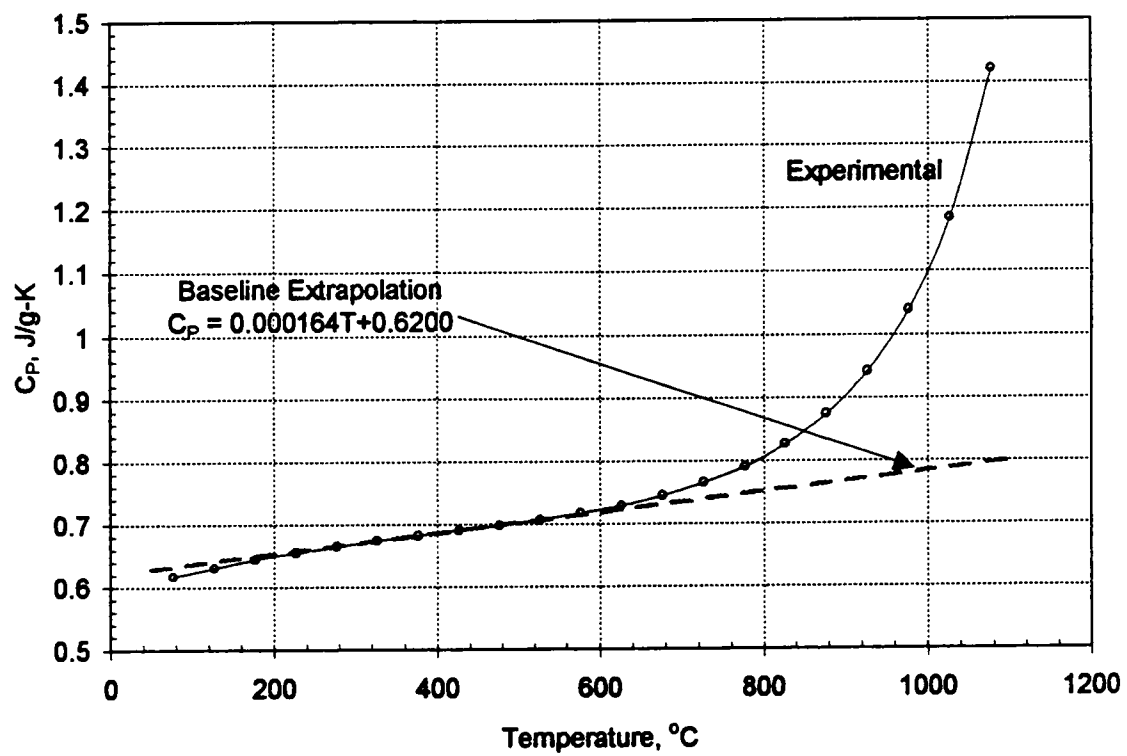


Fig. 6-31. C_p -T curve for the 38 at.% Al alloy heated at 52°C/s.

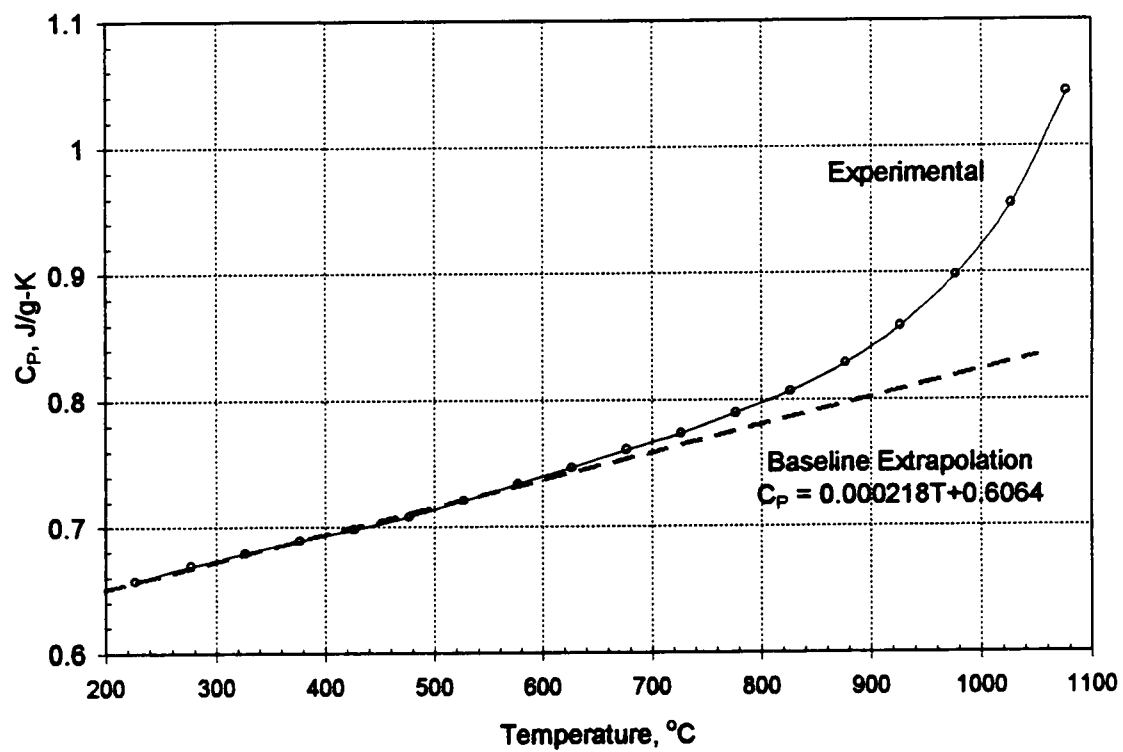


Fig. 6-32. C_p -T curve for the 38 at.% Al alloy heated at 73°C/s.

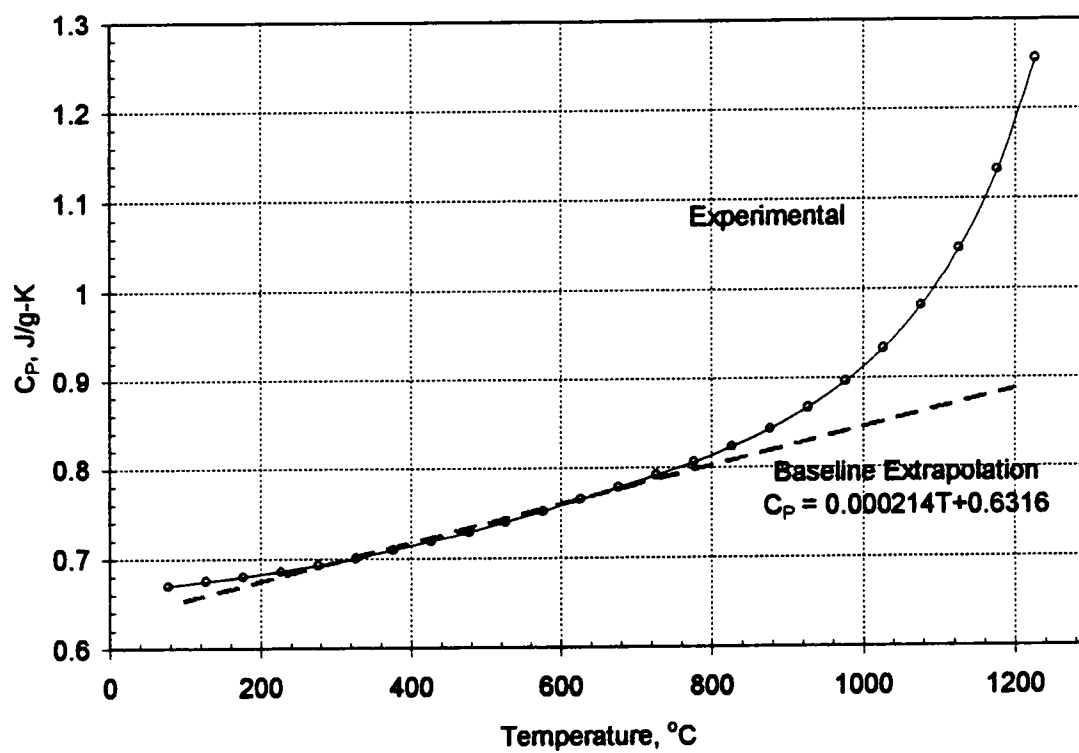


Fig. 6-33. C_p -T curve for the 38 at.% Al alloy heated at 162°C/s.

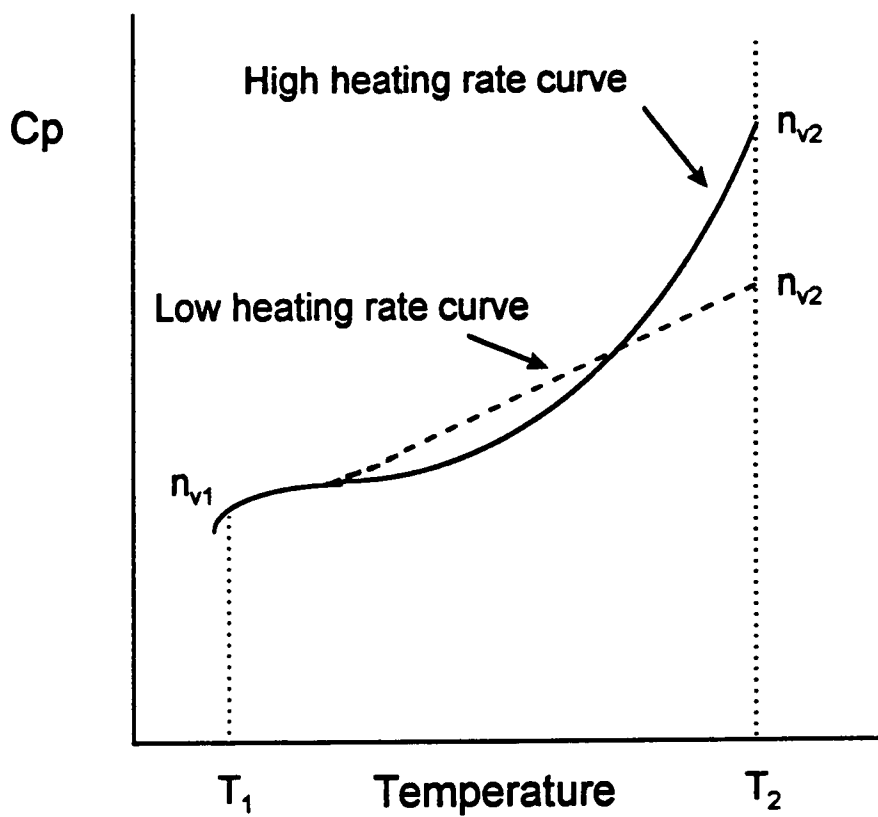


Fig. 6-34. Schematic showing the theoretically expected behavior as the heating rate is increased.

Fig. 6-34. At T_2 the vacancy concentrations n_{v2} for both heating rates should be identical and the areas under both curves should likewise be identical. This effect is not observed for this alloy. The reason for this is unclear but may be related to the defect mobility and/or the need to go to higher temperature. These results suggest that the vacancy/defect mobility for this particular alloy is low. This is in agreement with the results of Würschum et al. (28) that FeAl alloys of 37 and 39 at.% Al have exceptionally slow vacancy diffusion. As previously discussed, Vogl et al. (30) showed through Mössbauer spectroscopy that the diffusion is a two-step process involving an Al vacancy site, and that time constants increase with increasing Fe content. Because the probability of finding a vacant Al site decreases with increasing Fe content, the mobility also decreases with increasing Fe content. It is this low vacancy diffusivity that is responsible for the heating rate effects observed for the 38 at.% Al.

Defect Formation and Concentration

The enthalpy of vacancy/defect formation was determined by plotting $\ln(\Delta C_F^D T^2)$ versus $1/T$ for each test run. The data were fitted by a least squares method to a straight line. The slope of this line was multiplied by the universal gas constant R to obtain ΔH_F^D for each test condition. It is important to restate that the formation of vacancies occurs via triple defects. In other words, an antisite Fe atom is created along with two vacancies. Therefore, the enthalpy of formation ΔH_F^D is actually the enthalpy of triple defect formation.

The $\ln(\Delta C_P^D T^2)$ versus $1/T$ plots for each annealed alloy are shown in Fig. 6-35. The range of data for 38, 43, and 48 alloys increase by an order of magnitude with increasing Al concentration for a given temperature. However, the data for the 30 at.% alloy lies in the same range of values as the 38 at.% Al alloy, even though their slopes are different. The calculated ΔH_f^D values are shown as a function of composition in Fig. 6-36 along with the experimental ΔH_f^V (note that this is not ΔH_f^D) results of Riviere and Grihle (25) and Würschum et al. (28) and the theoretical result of Fu et al. (27). Our results are shown with the associated error. Comparison of the data shows that ΔH_f^D increases with decreasing Al concentration, and roughly parallels the ΔH_f^V values of Riviere and Grihle and Würschum et al. Note that the literature values of Riviere and Grihle, and Würschum et al. are for resistometry and positron lifetime measurement techniques, respectively. These techniques measure vacancy formation and concentration. They do not include the Fe antisite atom formation and concentration that occurs with triple defects. Therefore, our values are higher since ΔH_f^D includes the enthalpy to form antisite Fe atoms in addition to enthalpy of vacancy formation. Subtraction of their ΔH_f^V results from ΔH_f^D data indicates that the enthalpy of antisite Fe formation is approximately 25 kJ/mol.

From the ΔH_f^D values, the triple defect concentration n_D was determined for each alloy composition. There are two vacancies that are formed with each triple defect (Fig. 3-4). Thus, the vacancy concentration n_V was determined by multiplying n_D by 2.

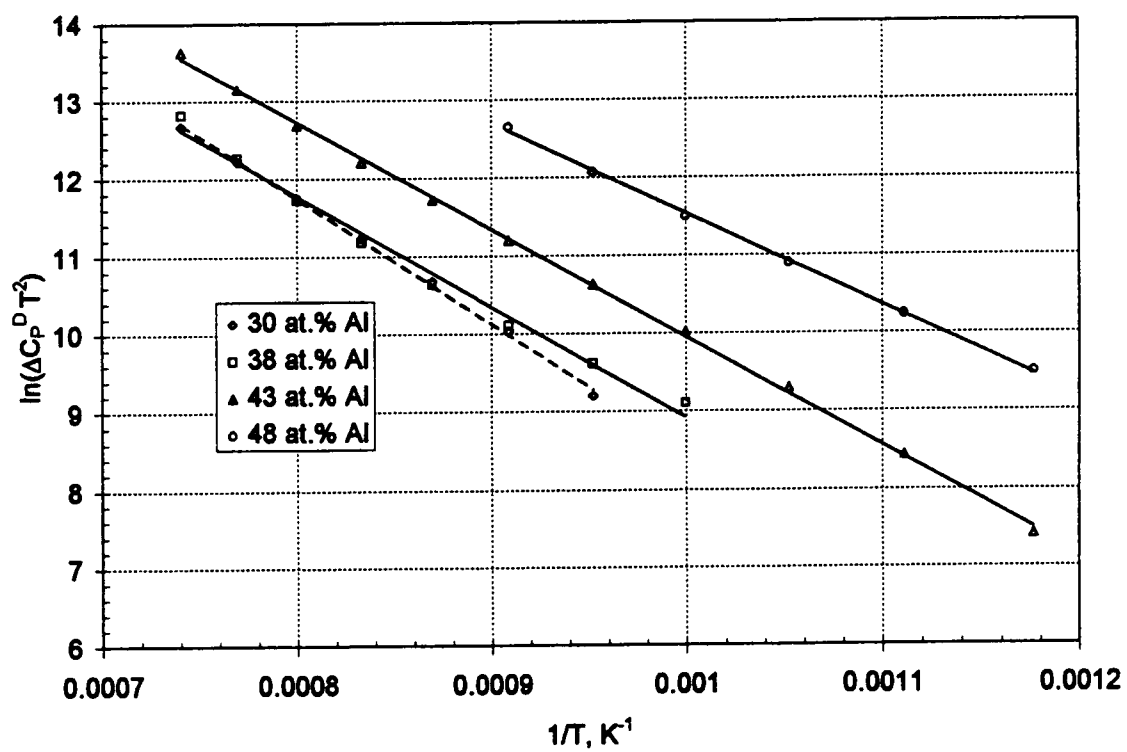


Fig. 6-35. Curves used to determine ΔH_f^D for each annealed alloy.

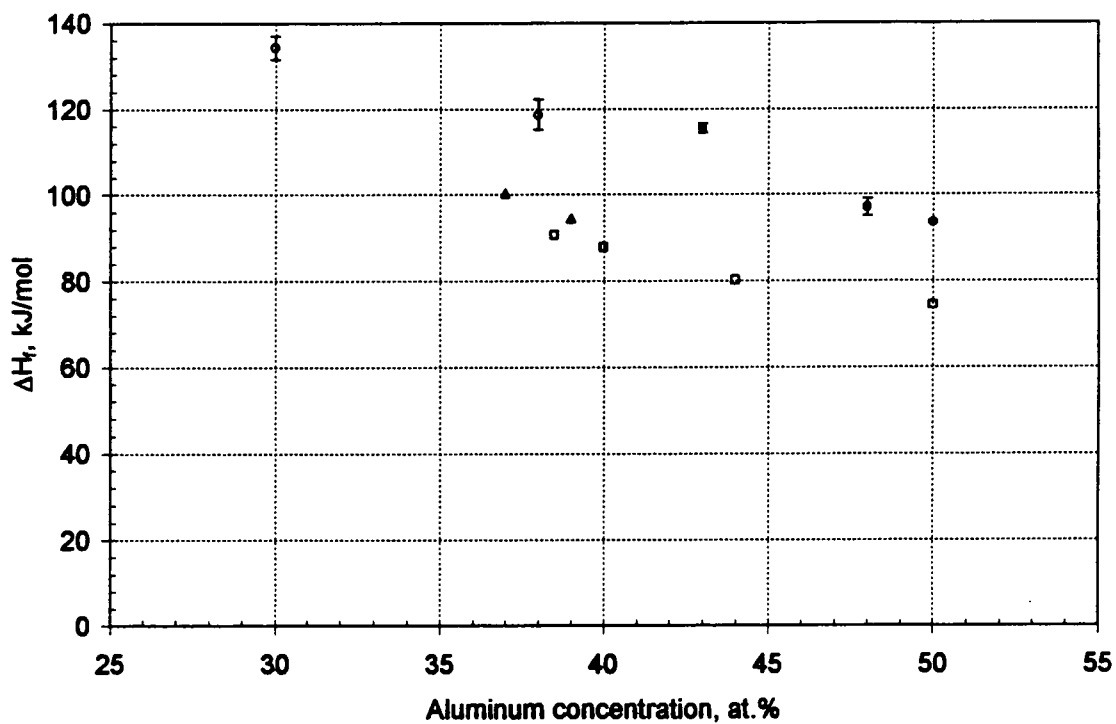


Fig. 6-36. Enthalpy of vacancy/defect formation as a function of composition. Riviere and Grihle's data are marked by squares (25), Würshum et al. by triangles (28), and Fu et al. by a solid circle (27).

Our results are shown in Fig. 6-37 along with the results of Ho and Dodd (15) and Paris and Lesbats (16) in Fig. 6-38. The results show that the vacancy concentrations for the 30 and 38 at.% Al alloys are very low, in accordance with the prediction of the Chang and Neumann model (17). For the higher Al compositions, n_v increases with increasing Al concentration in a manner similar to the data of Paris and Lesbats and corresponds to the Chang and Neumann model. The results are in good agreement with the range of data obtained by Ho and Dodd and Paris and Lesbats.

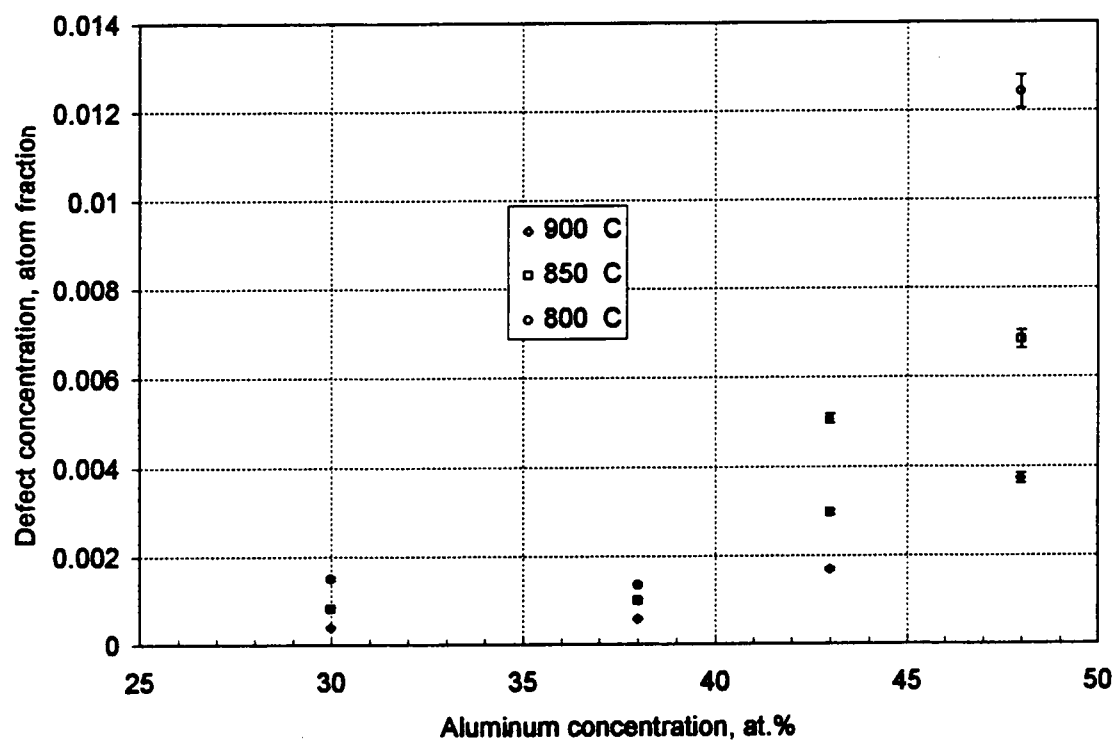


Fig. 6-37. Thermal defect concentrations for FeAl alloys determined by pulse-heating calorimetry.

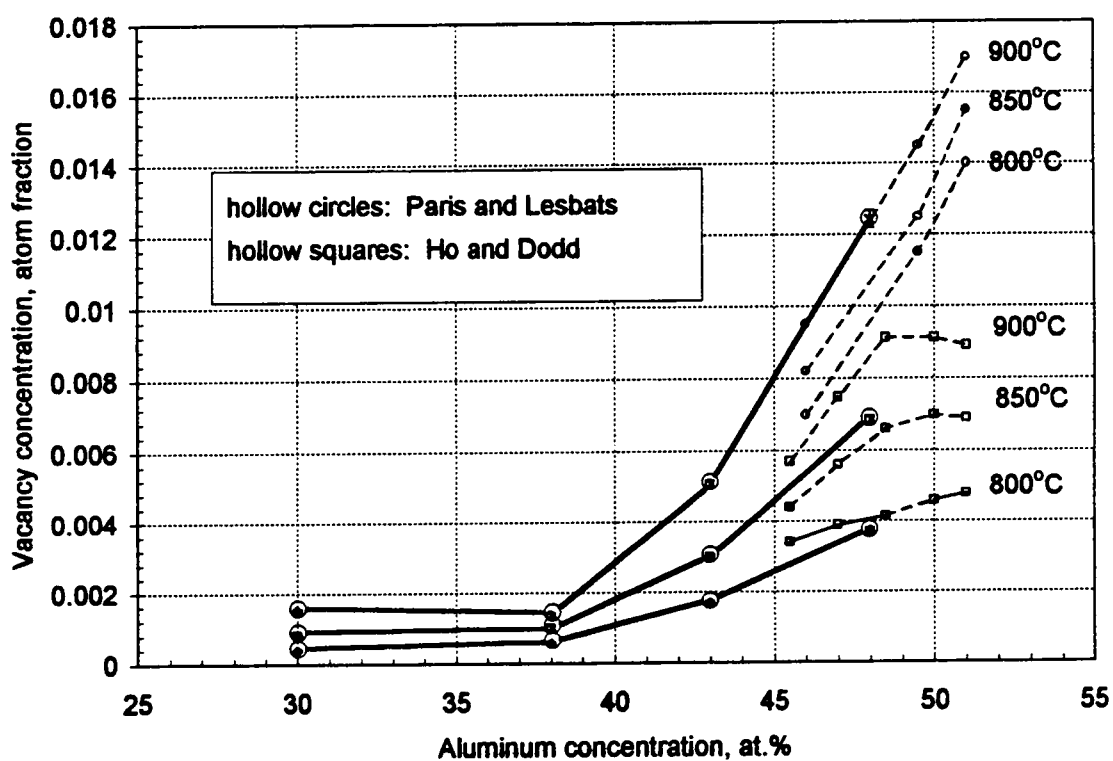


Fig. 6-38. Comparison of vacancy concentration results. Data determined by pulse-heating calorimetry are shown along with data of Paris and Lesbats (16) and Ho and Dodd (15).

Chapter 7

Conclusions

For both the Fe_3Al (D0_3 structure) and FeAl (B2 structure) alloys annealed to minimize vacancy content and maximize the degree of order, the electrical resistivity at 25°C increases with deviation from stoichiometry. This shows that the resistivity is controlled by the degree of order.

For the 30 at.% Al Fe_3Al alloy, the electrical resistivity was identical for an initial step-cooled condition and for cooling at 1.5°C/s from high temperature (e.g., 1000°C). The resistivity was the same for heating rates from 50 to 70°C/s . There was a slight decrease at 480°C , corresponding to the change from the D0_3 to the B2 structure. This resistivity change did not correspond to the Curie temperature for this alloy. In the B2 structure range ρ decreased slightly (see below). However, the specific heat showed a very marked increase, but beginning around 680°C , in the B2 temperature range, and is attributed to the formation of defects (see below). The C_P results show that the D0_3 structure has a low defect content, but in the higher temperature range in the B2 structure defects do form. However, the ρ - T curve is not affected by heating rate or annealing, which shows that the scattering of electrons is not affected by the lattice vacancies.

The 38, 43, and 48 at.% Al alloys were all in the B2 structure range. The ρ -T curves for the 38 and 43 at.% Al alloys were identical for the initial slowly cooled condition and after cooling from high temperature at 1.5°C/s. The curve for the 48 at.% Al alloy was about 30% higher after cooling from high temperature, which is taken to indicate that more vacancies are present as the stoichiometric composition is approached. After cooling from high temperature subsequent ρ -T curves were independent of heating rate for all alloys (including 30 at.% Al). None of the curves showed any indication of structural changes. However, the C_p -T curves showed a marked increase in C_p at high temperatures. It is concluded that the resistivity for FeAl alloys is determined by the antisite occupation on the β sublattice and not vacancies.

The enthalpy of formation values of defects derived from the C_p data are similar to those obtained by other methods, and the derived vacancy concentrations are similar to those reported in the literature.

One of the most important findings in the present research is the behavior of the ρ -T curves of the B2 structure (including the high temperature data of the 30 at.% Al alloy). As the Fe content increases, the slope of the ρ -T curves decreases, becoming slightly negative for the 30 at.% Al alloy. Also, these curves showed a tendency to approach a constant (or saturation) value, a behavior found for amorphous and disordered transition alloys. The causes of this characteristic are unknown.

Chapter 8

Recommendations

The pulse-heating apparatus is an extremely useful tool for studying physical phenomena in metals and alloys. It has the ability to measure simultaneously the electrical resistivity and specific heat to high temperatures (e.g., 1200°C) as a function of heating rate.

Although not utilized in the present research, the system can be used to measure the electrical resistivity and power during isothermal treatment and during controlled heating and cooling. The recommendations listed below suggest measurements using the pulse-heating device on Fe-Al alloys to provide additional information about their behavior.

Measurements on other alloy systems would also provide information about interpretation of the ρ -T and C_p -T curves and defect structures for ordered alloys.

1. The electrical resistivity and specific heat of the alloys should be measured using higher heating rates. The power supply is now being altered to increase the maximum current to about 200 amps. This should increase the heating rate to approximately 400°C/s. These data will establish better the baseline for the calculation of the enthalpy of defect formation.
2. Data should be taken to higher temperatures. This is especially important in interpreting the specific heat results for the 38 at.% Al alloy, which didn't go to sufficient temperature to establish the high temperature behavior (Fig. 6-27).

3. After the appropriate measurements indicated under 1 and 2 have been made, the specimens should be sent to Dr. Deb Basak at NIST. Dr. Basak has agreed to make measurements of the electrical resistivity and specific heat using his pulse-heating device which heats from 25°C to above the melting point in microseconds. This will supply data at extremely high rates. Note that in his method the specimen is melted and thus any desired prior measurements must be completed first.
4. The pulse-heating apparatus should be used to measure the electrical resistivity and specific heat of other intermetallic alloys chosen to provide insight into structural defects. For example, NiAl, CoAl, CuZn, AuZn and FeCo are all B2 alloys which have antisite defects or triple defects.
5. Measurements should be made to examine further the resistivity saturation phenomenon.

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Appendices

Appendix A

Curves Used to Determine the Enthalpy of Defect Formation

Contents—Appendix A

Figure

- A-1 $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 30 at.% Al alloy in the annealed condition.
- A-2 $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 38 at.% Al alloy in the annealed condition.
- A-3 $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 34 at.% Al alloy in the annealed condition.
- A-4 $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 48 at.% Al alloy in the annealed condition.

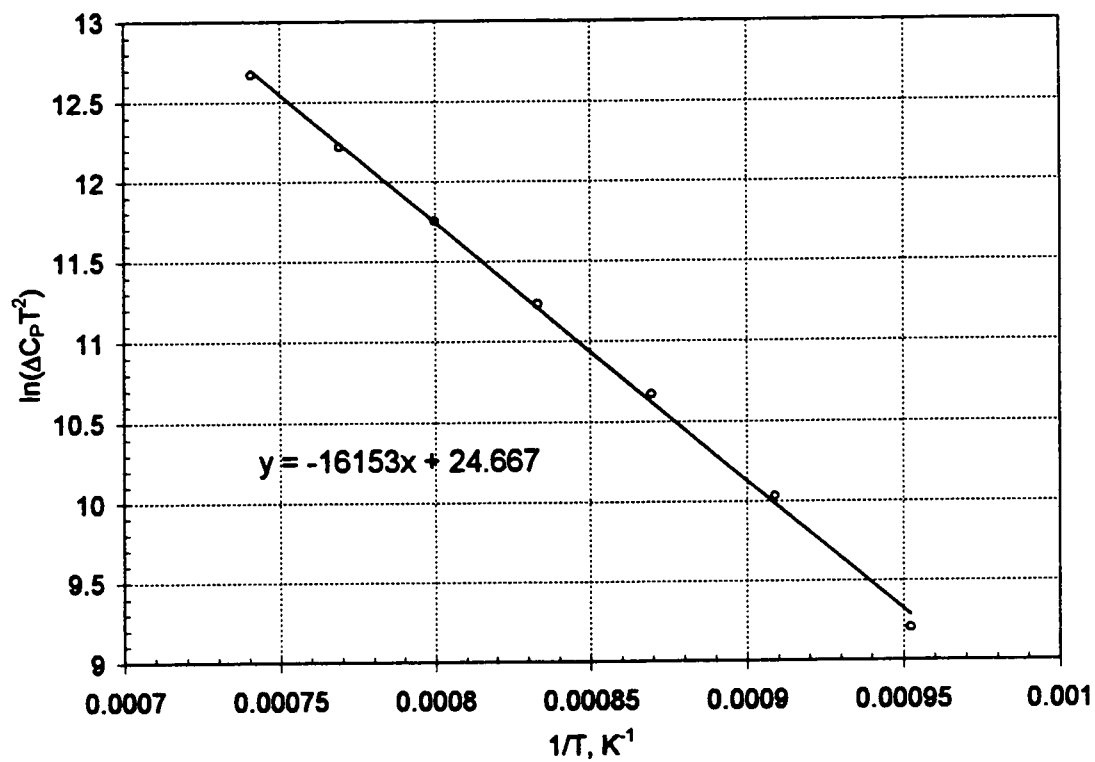


Fig. A-1. $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 30 at.% Al alloy in the annealed condition.

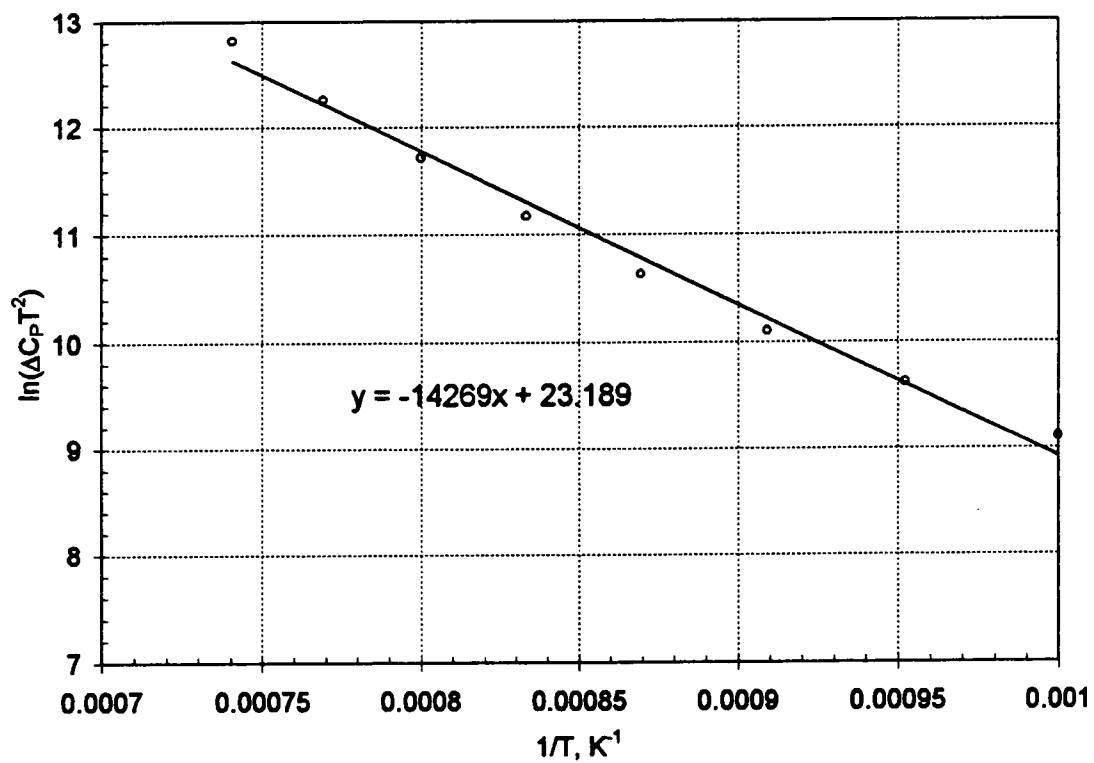


Fig. A-2. $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 38 at.% Al alloy in the annealed condition.

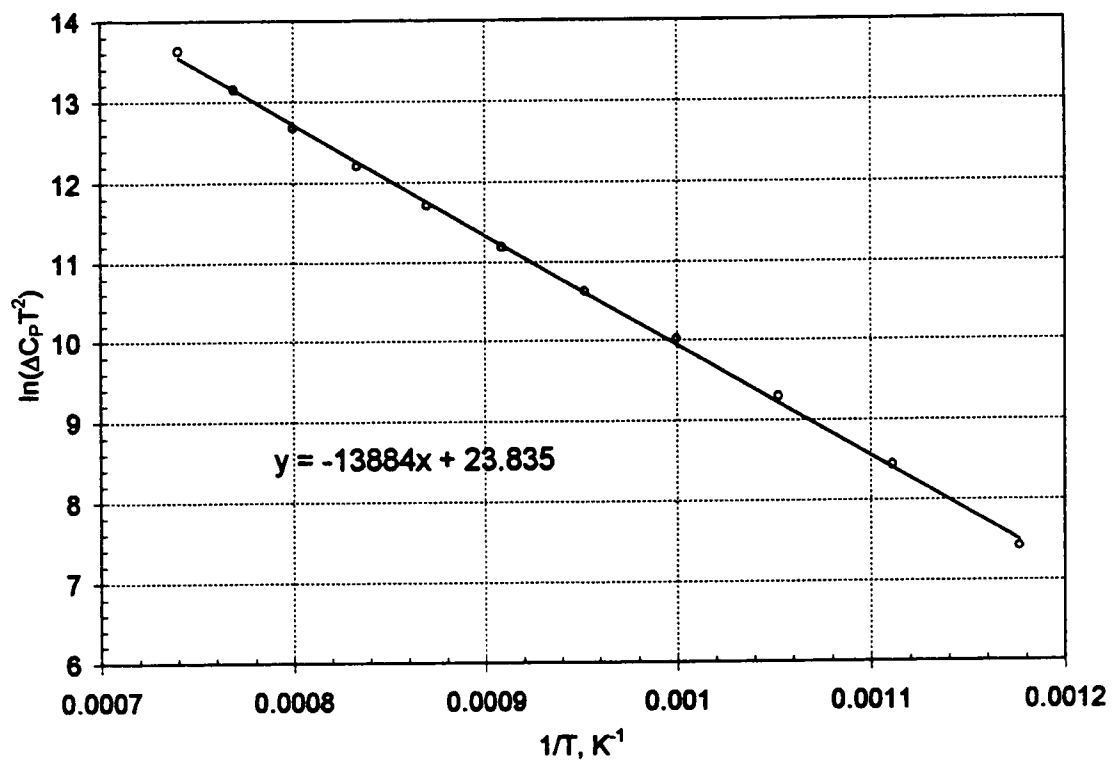


Fig. A-3. $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 43 at.% Al alloy in the annealed condition.

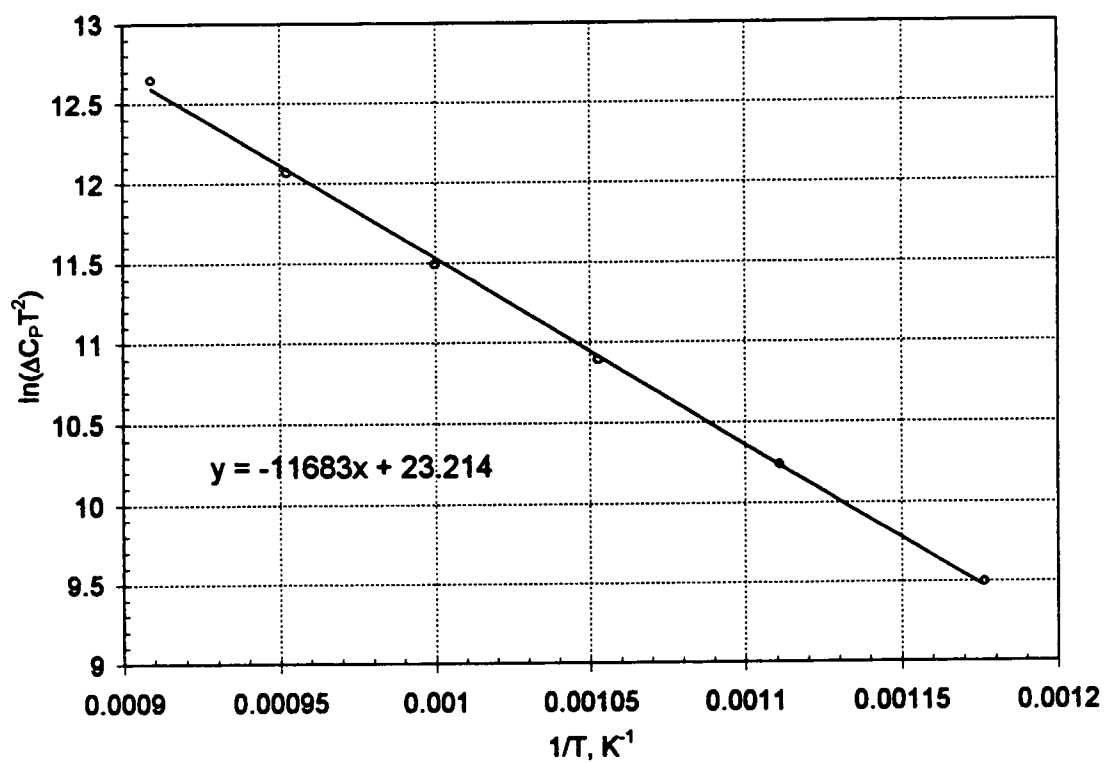


Fig. A-4. $\ln(\Delta C_p T^2)$ versus $1/T$ data for the 48 at.% Al alloy in the annealed condition.

Appendix B
Tabulated Data Files

Contents—Appendix B

Table

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B-26	Heating data for F0122T3
B-27	Heating data for F0124T2
B-28	Heating data for F0124T3
B-29	Heating data for F0124T4
B-30	Heating data for F0124T5
B-31	Heating data for F0124T6

Table B-1. Legend.

Run Number	Specimen	Current (Amps)	Cable Position
F1210T1	38-Batch #1	35	crossed
F1210T2	38-Batch #1	35	crossed
F1210T3	38-Batch #1	35	uncrossed
F1213T1	38-Batch #1	40	crossed
F1213T2	38-Batch #1	50	crossed
F1213T3	38-Batch #1	50	crossed
F1215T1	38-Batch #1	80	crossed
F1230T2	38-Batch #1	50	crossed
F103T1	38-Batch #2	50	crossed
F103T2	38-Batch #2	50	uncrossed
F0109T1	43-Batch #1	55	uncrossed
F0110T2	43-Batch #1	55	crossed
F0112T1	43-Batch #1	48	uncrossed
F0112T2	43-Batch #1	40	uncrossed
F0112T3	43-Batch #1	65	uncrossed
F0116T1	48-Batch #1	50	uncrossed
F0116T2	48-Batch #1	50	uncrossed
F0116T3	48-Batch #1	50	uncrossed
F0116T4	48-Batch #1	50	uncrossed
F0122T1	48-Batch #1	50	crossed
F0122T2	48-Batch #1	40	uncrossed
F0122T3	48-Batch #1	60	uncrossed
F0124T2	30-Batch #1	50	uncrossed
F0124T3	30-Batch #1	50	uncrossed
F0124T4	30-Batch #1	40	uncrossed
F0124T5	30-Batch #1	50	crossed
F0124T6	30-Batch #1	60	uncrossed

Table B-2. Power-T coefficients.

Run	Polynomial Coefficients					
	Degree 0	Degree 1	Degree 2	Degree 3	Degree 4	Degree 5
F1210T1	10.7977	0.0188507	-2.1611E-5	1.20138E-8	-2.61239E-12	0.0
F1210T2	7.87126	0.0358778	-5.72265E-5	4.38222E-8	-1.29142E-11	0.0
F1213T1	14.4016	0.0222492	-2.3282E-5	1.11551E-8	-1.89813E-12	0.0
F1213T3	22.0402	0.0358124	-4.12759E-5	2.37702E-8	-5.43534E-12	0.0
F1215T1	74.997	.00514841	-7.08237E-7	0.0	0.0	0.0
F1230T2	17.3681	0.0456961	-5.56918E-5	3.2095E-8	-7.01043E-12	0.0
F103T1	17.5768	0.0443562	-5.44783E-5	3.1982E-8	-7.18651E-12	0.0
F103T2	16.6441	0.0473912	-5.90714E-5	3.47676E-8	-7.77377E-12	0.0
F0109T1	8.22192	0.0436634	-3.01986E-5	7.93434E-9	0.0	0.0
F0110T2	8.73844	0.0445986	-3.37514E-5	9.75732E-9	0.0	0.0
F0112T1	6.71253	0.0341083	-2.5648E-5	7.3101E-9	0.0	0.0
F0112T2	4.49619	0.0242603	-1.80251E-5	5.01788E-9	0.0	0.0
F0112T3	13.4815	0.0558637	-3.88167E-5	9.99656E-9	0.0	0.0
F0116T1	2.44881	.00387054	4.04968E-5	-5.02612E-8	1.90412E-11	0.0
F0116T2	1.3619	0.0245929	-1.20021E-5	2.31381E-9	0.0	0.0
F0116T3	1.25001	0.0256812	-1.46685E-5	3.56696E-9	0.0	0.0
F0116T4	1.48953	0.0238818	-1.2083E-5	2.35092	0.0	0.0
F0122T2	.0596894	0.0183996	-1.70523E-5	1.29517E-8	-4.74025E-12	0.0
F0124T2	23.1525	-.0446697	.000221513	-3.59801E-7	2.49546E-10	-6.36055E-14

Table B-3. dT/dt -T during heating coefficients.

Run	Polynomial Coefficients					
	Degree 0	Degree 1	Degree 2	Degree 3	Degree 4	Degree 5
F1210T1	34.1499	0.072611	-.000149866	1.36667E-7	-5.89993E-11	0.0
F1210T2	34.1733	0.0702417	-.000140272	1.26358E-7	-5.63777E-11	0.0
F1213T1	51.6648	0.0667037	-0.00015214	1.36653E-7	-5.48384E-11	0.0
F1213T3	123.614	-0.23199	.000705155	-1.02823E-6	6.90483E-10	-1.81633E-13
F1215T1	296.782	-0.608251	0.00172507	-2.29662E-9	1.39183E-9	-3.24034E-13
F1230T2	99.0046	-0.105767	0.00039992	-6.36996E-7	4.45038E-10	-1.22982E-13
F103T1	113.864	-0.181749	0.00056994	-8.42717E-7	5.67746E-10	-1.50097E-13
F103T2	107.083	-0.124202	.000386151	-5.92056E-7	4.09894E-10	-1.12771E-13
F0109T1	47.0854	0.212545	-.000325056	2.45883E-7	-8.74452E-11	0.0
F0110T2	41.4122	0.233237	-.000347213	2.5566E-7	-8.93708E-11	0.0
F0112T1	26.5992	0.19322	-.000273616	1.94681E-7	-7.07203E-11	0.0
F0112T2	30.9189	0.0881099	-.000125476	8.79017E-8	-3.46983E-11	0.0
F0112T3	74.5465	0.257896	-.000386672	2.87911E-7	-1.02101E-10	0.0
F0116T1	186.129	-1.18649	0.00368167	-5.11154E-6	3.44938E-9	-9.41851E-13
F0116T2	9.48755	0.169108	6.34626E-6	-3.10941E-7	3.20677E-10	-1.13054E-13
F0116T3	-48.3777	0.50133	-.000744393	5.35766E-7	-1.63861E-10	0.0
F0116T4	-34.8556	0.423069	-.000596968	4.14365E-7	-1.25408E-10	0.0
F0122T2	5.92998	0.140109	-.000167615	1.15604E-7	-4.56693E-11	0.0
F0124T2	96.0619	-.0117362	-1.71628E-5	-1.69109E-9	-1.26499E-13	0.0

Table B-4. $dT/dt-T$ during cooling coefficients.

Run	Polynomial Coefficients					
	Degree 0	Degree 1	Degree 2	Degree 3	Degree 4	Degree 5
F1210T1	-18.2284	0.094982	-0.00017867	1.26221E-7	-3.21522E-11	0.0
F1210T2	-22.2679	0.116637	-0.000221466	1.62924E-7	-4.37677E-11	0.0
F1213T1	-4.99843	0.0304562	-5.57116E-5	2.13679E-8	0.0	0.0
F1213T3	-27.8198	0.136745	-0.000229639	1.42002E-7	-3.1188E-11	0.0
F1215T1	-406.935	1.96501	-0.00356957	3.01248E-6	-1.24081E-9	2.01837E-13
F1230T2						
T<700°C	-4.30267	0.029694	-6.67456E-5	3.78641E-8	0.0	0.0
T>600°C	-27.8852	0.0991983	-0.00115522	3.46177E-8	0.0	0.0
F103T1						
T<800°C	-4.44256	0.0374739	-9.78658E-5	8.16935E-8	-2.23253E-11	0.0
T>600°C	-4.44256	0.0374739	-9.78658E-5	8.16935E-8	-2.23253E-11	0.0
F103T2						
T<800°C	-14.7538	0.0792166	-0.00135949	6.74802E-8	0.0	0.0
T>630°C	-3.9752	0.040291	-6.62392E-5	2.04004E-8	0.0	0.0
F0109T1						
T<700°C	-1.01769	0.016444	-5.22135E-5	-5.22135E-5	3.11515E-8	0.0
T>600°C	-27.1222	0.0928885	-0.00108384	3.1114E-8	0.0	0.0
F0110T2						
T<700°C	-8.37529	0.0157558	-5.23875E-5	3.29512E-8	0.0	0.0
T>600°C	-20.1546	0.072798	-9.10825E-5	2.71361E-8	0.0	0.0
F0112T1						
T<700°C	1.0357	.00546093	-3.61006E-5	-3.61006E-5	2.53104E-8	0.0
T>600°C	-12.0531	.0476264	-6.72809E-5	2.05692E-8	0.0	0.0
F0112T2						
T<675°C	-1.14552	0.018234	-6.06343E-5	4.08929E-8	0.0	0.0
T>500°C	-10.0296	0.0425203	-6.56496E-5	2.2532E-8	0.0	0.0
F0112T3						
T<800°C	0.740284	.00684102	-3.5341E-5	2.15541E-8	0.0	0.0
T>600°C	-15.4474	0.0606771	-7.92494E-5	2.23453E-8	0.0	0.0
F0116T1						
T<700°C	1.84583	-1.774E-5	-2.41032E-5	1.3848E-8	0.0	0.0
T>500°C	5.83588	-0.0107819	-1.09465E-5	4.42135E-9	0.0	0.0
F0116T2						
T<700°C	-16.6902	0.149816	-0.000466119	5.80236E-7	-2.64025E-10	0.0
T>500°C	-4.25775	0.0283621	-5.37737E-5	1.69603E-8	0.0	0.0
F0116T3						
T<700°C	-.528719	0.0163476	-5.98683E-5	4.03256E-8	0.0	0.0
T>500°C	-4.73043	0.0288994	-5.36956E-5	1.68817E-8	0.0	0.0
F0116T4						
F0122T2						
F0124T2						

Table B-4 (continued).

Run	Polynomial Coefficients					
	Degree 0	Degree 1	Degree 2	Degree 3	Degree 4	Degree 5
F0116T4						
T<700°C	0.941792	.00750409	-4.2941E-5	2.97623E-8	0.0	0.0
T>500°C	-3.24965	0.0217564	-4.6006E-5	1.53567E-8	0.0	0.0
F0122T2						
T<700°C	-.596372	0.0153402	-5.41822E-5	3.34045E-8	0.0	0.0
T>500°C	5.92998	0.140109	-.000167615	1.15604E-7	-4.56693E-11	0.0
F0124T2	11.6987	-0.069253	.000141489	-1.35079E-7	4.1993E-11	0.0

Table B-5. Heating data for F1210T1.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.9887	314.51	155.59
1.483	337.97	156.71
1.9773	350.83	158.61
2.4716	375.93	160.16
2.966	398.04	161.83
3.4603	424.1	162.91
3.9546	446.18	164.46
4.449	472.68	165.17
4.9433	495.01	167.08
5.4376	517.86	167.8
5.9319	541.26	169.47
6.4263	566.16	170.3
6.9206	586.15	171.13
7.4149	609.88	171.97
7.9093	630.29	172.8
8.4036	657.04	173.64
8.8979	671.7	174.35
9.3922	693.67	175.31
9.8866	715.85	175.31
10.381	741.08	176.14
10.875	764.19	177.11
11.37	782.58	176.98
11.864	805.72	177.81
12.358	820.72	177.94
12.853	838.67	178.65
13.347	864.71	178.65
13.841	885.4	179.62
14.336	903.29	179.62
14.83	924.41	179.62
15.324	944.07	179.75
15.819	954.14	180.32
16.313	982.59	180.45
16.807	998.19	180.45
17.302	1011.2	181.29
17.796	1027	181.29
18.29	1048.3	181.42
18.784	1057.9	182.26
19.279	1075.8	182.26
19.773	1088.5	182.26

Table B-6. Heating data for F1210T2.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
9.89E-01	313.176	155.211
1.48299	373.951	156.595
1.97731	356.193	158.261
2.47164	411.458	160.164
2.96597	402.339	162.192
3.4603	422.879	162.426
3.95463	440.521	164.334
4.44896	468.826	165.88
4.94329	494.462	166.837
5.43762	523.654	167.671
5.93194	538.628	169.215
6.42627	563.168	170.174
6.9206	583.702	171.008
7.41493	612.781	171.842
7.90926	636.503	172.676
8.40359	651.396	173.511
8.89792	671.702	174.345
9.39224	699.756	174.345
9.88657	722.347	175.308
10.3809	739.213	175.179
10.8752	761.045	176.143
11.3696	780.837	176.978
11.8639	805.27	176.978
12.3582	826.483	177.813
12.8525	841.708	177.813
13.3469	863.034	178.648
13.8412	887.059	178.648
14.3355	907.894	179.482
14.8299	924.415	179.482
15.3242	944.928	179.482
15.8185	959.5	180.451
16.3129	970.689	180.451
16.8072	999.027	180.317
17.3015	1014.45	180.317
17.7958	1024.56	181.286
18.2902	1043.12	181.152
18.7845	1062.68	181.286
19.2788	1073.02	182.122
19.7731	1088.49	181.152

Table B-7. Heating data for F1210T3.

Time (s)	T (mV)	ρ ($\mu\Omega\text{-cm}$)
9.89E-01	0.205	155.473
1.48299	0.342	156.711
1.97731	0.508	158.378
2.47164	0.693	160.76
2.96597	0.845	161.712
3.4603	1.069	163.38
3.95463	1.216	164.213
4.44896	1.475	165.88
4.94329	1.904	167.054
5.43762	1.968	168.133
5.93194	2.139	169.465
6.42627	2.314	171.008
6.9206	2.534	171.842
7.41493	2.861	172.676
7.90926	2.93	173.511
8.40359	3.218	174.345
8.89792	3.325	174.474
9.39224	3.682	175.179
9.88657	3.921	176.013
10.3809	4.038	176.978
10.8752	4.326	176.847
11.3696	4.546	177.813
11.8639	4.438	177.813
12.3582	5.073	178.516
12.8525	5.332	178.648
13.3469	5.493	179.482
13.8412	5.698	179.35
14.3355	5.918	180.317
14.8299	6.167	180.317
15.3242	6.382	181.018
15.8185	6.558	181.152
16.3129	6.782	180.451
16.8072	6.948	181.018
17.3015	7.197	182.122
17.7958	7.349	182.122
18.2902	7.612	182.122
18.7845	7.803	182.417
19.2788	7.964	183.364
19.7731	8.257	182.957

Table B-8. Heating data for F1213T1.

Time (s)	T (K)	ρ ($\mu\Omega$ -cm)
9.89E-01	320.402	155.448
1.48299	335.839	157.163
1.97731	375.933	159.984
2.47164	410.838	161.549
2.96597	432.899	163.011
3.4603	468.826	165.204
3.95463	496.115	165.935
4.44896	524.189	168.346
4.94329	553.513	169.81
5.43762	585.23	170.542
5.93194	615.679	172.118
6.42627	642.793	172.85
6.9206	671.222	173.47
7.41493	699.756	175.047
7.90926	730.609	175.78
8.40359	760.12	176.512
8.89792	783.493	177.245
9.39224	815.494	177.245
9.88657	841.708	178.825
10.3809	869.041	178.093
10.8752	887.497	179.558
11.3696	920.619	179.558
11.8639	939.966	180.291
12.3582	970.266	180.291
12.8525	987.126	181.024
13.3469	1014.45	181.024
13.8412	1036.29	181.024
14.3355	1061.54	181.142
14.8299	1084.93	181.875
15.3242	1105.39	181.757
15.8185	1118.27	182.609
16.3129	1143.86	182.609
16.8072	1169.22	182.609
17.3015	1182.59	182.846
17.7958	1200.09	183.7
18.2902	1216.32	183.461
18.7845	1232.47	183.461
19.2788	1248.53	184.929
19.7731	1263.36	184.075

Table B-9. Heating data for F1213T2.

Time (s)	T (K)	ρ ($\mu\Omega\text{-cm}$)
5.49E-01	306.599	155.001
8.24E-01	323.905	156.576
1.09851	342.758	159.04
1.37313	372.095	160.391
1.64776	401.078	162.081
1.92239	431.701	163.436
2.19702	446.18	165.125
2.47164	473.251	166.484
2.74627	504.112	167.757
3.0209	530.901	169.12
3.29552	573.164	170.038
3.57015	572.24	171.134
3.84478	607.872	172.228
4.11941	630.78	173.411
4.39403	656.066	173.912
4.66866	679.238	174.912
4.94329	708.953	175.594
5.21791	731.92	176.093
5.49254	759.195	176.87
5.76717	776.8	177.183
6.04179	803.092	178.053
6.31642	830.792	178.551
6.59105	853.017	178.551
6.86568	875.473	179.142
7.1403	900.679	179.733
7.41493	915.52	179.639
7.68956	946.21	180.324
7.96418	966.541	180.136
8.23881	984.688	180.324
8.51344	1008.79	181.507
8.78806	1029.03	180.916
9.06269	1050.75	181.412
9.33732	1070.66	181.412
9.61195	1091.23	181.907
9.88657	1108.92	182.098
10.1612	1125.69	182.594
10.4358	1147.34	182.594
10.7105	1166.55	183.184
10.9851	1178.76	183.184

Table B-10. Heating data for F1213T3.

Time (s)	T (K)	ρ ($\mu\Omega\text{-cm}$)
0.988657	330.2653	157.5
1.482986	377.9074	160.478
1.977315	413.9322	162.923
2.471643	470.4175	165.874
2.965972	508.3556	167.909
3.460301	557.0513	170.128
3.954629	605.9641	172.318
4.448958	648.469	173.593
4.943287	684.3131	175.187
5.437616	734.2603	176.592
5.931944	780.3785	177.866
6.426273	824.6847	178.457
6.920602	860.3789	178.861
7.41493	904.5068	179.639
7.909259	947.4058	180.136
8.403588	986.7057	180.632
8.897916	1027.791	181.412
9.392244	1065.454	181.317
9.886573	1094.373	181.812
10.3809	1136.101	182.498
10.87523	1167.336	182.993
11.36956	1194.033	183.583
11.86389	1229.093	183.775
12.35822	1258.576	184.27
12.85255	1285.139	184.366
13.34687	1306.016	185.054
13.8412	1327.203	185.054
14.33553	1356.441	185.151
14.82986	1379.184	185.248
15.32419	1392.348	185.345
15.81852	1412.673	186.035
16.31285	1428.926	186.231
16.80718	1446.571	186.133
17.3015	1458.541	186.231
17.79583	1469.481	186.329
18.29016	1463.51	225.731
18.78449	1453.28	260.458
19.27882	1440.214	205.21
19.77315	1432.112	260.458

Table B-11. Heating data for F1215T1.

Time (s)	T (K)	ρ ($\mu\Omega$ -cm)
0.549254	315.9898	155.937
0.823881	378.4328	160.262
1.098508	432.3003	164.209
1.373135	484.1481	167.686
1.647762	558.6091	170.535
1.922389	621.0606	173.314
2.197016	678.2786	175.169
2.471643	720.9362	176.201
2.746271	774.9632	177.715
3.020898	837.419	178.962
3.295524	884.5188	179.639
3.570152	941.1649	180.208
3.844779	992.4968	180.959
4.119406	1045.501	181.591
4.394033	1095.179	181.978
4.66866	1146.947	182.622
4.943287	1186.798	183.235
5.217914	1240.279	183.906
5.492541	1281.444	184.555
5.767168	1321.696	184.892
6.041795	1356.81	185.458
6.316422	1397.019	185.841
6.591049	1421.169	186.093
6.865676	1456.092	186.415
7.140303	1479.252	186.408
7.41493	1502.043	186.535
7.689557	1535.531	186.336
7.964184	1559.447	186.063
8.238811	1591.758	184.792
8.513438	1590.339	181.078
8.788065	1632.575	185.019
9.062692	1643.619	193.132
9.337319	1639.159	189.433
9.611946	1639.159	194.03
9.886573	1649.141	192.086

Table B-12. Heating data for F1230T2.

Time (s)	Temp (K)	ρ ($\mu\Omega$ -cm)
0.988657	339.2393	155.043
1.482986	385.4776	158.697
1.977315	425.3085	161.216
2.471643	474.2681	164.364
2.965972	519.4755	166.254
3.460301	556.5316	168.861
3.954629	602.0411	170.752
4.448958	656.5511	172.551
4.943287	696.4288	173.902
5.437616	741.0795	175.07
5.931944	782.5775	177.052
6.426273	822.0755	177.682
6.920602	862.1493	178.313
7.41493	910.4966	178.943
7.909259	947.4058	179.667
8.403588	988.3857	180.108
8.897916	1026.633	180.833
9.392244	1060.725	180.833
9.886573	1097.916	181.463
10.3809	1136.894	182.093
10.87523	1168.825	182.093
11.36956	1204.193	183.257
11.86389	1228.709	183.45
12.35822	1257.816	184.08
12.85255	1289.957	183.983
13.34687	1313.716	184.08
13.8412	1336.262	184.71
14.33553	1363.288	184.516
14.82986	1379.843	185.341

Table B-13. Heating data for F103T1.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
9.89E-01	332.416	153.688
1.48299	376.592	157.517
1.97731	426.398	160.037
2.47164	465.977	163.274
2.96597	509.441	165.165
3.4603	557.051	167.775
3.95463	598.007	170.387
4.44896	644.655	171.559
4.94329	684.313	172.73
5.43762	731.92	174.805
5.93194	777.627	175.343
6.42627	819.373	176.512
6.9206	854.349	177.236
7.41493	903.29	177.866
7.90926	942.449	179.034
8.40359	981.406	179.128
8.89792	1011.21	179.664
9.39224	1052.71	180.294
9.88657	1092.04	180.484
10.3809	1129.91	181.02
10.8752	1161.6	181.651
11.3696	1195.12	182.186
11.8639	1220.1	182.186
12.3582	1255.92	182.377
12.8525	1276.31	183.008
13.3469	1308.26	183.543
13.8412	1331.51	183.64
14.3355	1356.44	183.64
14.8299	1374.49	184.271
15.3242	1400.23	184.271
15.8185	1408.38	184.999
16.3129	1430.38	184.174
16.8072	1450.83	184.465
17.3015	1466.32	184.368

Table B-14. Heating data for F103T2.

Time (s)	Temp (K)	ρ ($\mu\Omega$ -cm)
0.988657	321.7182	152.51
1.482986	365.5483	155.898
1.977315	405.978	158.647
2.471643	453.6541	161.091
2.965972	500.943	163.532
3.460301	537.6776	165.882
3.954629	588.6887	167.598
4.448958	621.0606	169.667
4.943287	669.3019	170.749
5.437616	710.3712	172.639
5.931944	752.4305	173.36
6.426273	800.004	174.621
6.920602	834.7346	175.159
7.41493	881.5368	175.882
7.909259	917.6818	176.419
8.403588	961.9626	177.049
8.897916	998.1908	177.679
9.392244	1027.047	177.679
9.886573	1069.849	178.216
10.3809	1102.257	178.309
10.87523	1136.101	178.846
11.36956	1161.604	179.475
11.86389	1193.644	179.57
12.35822	1216.708	180.105
12.85255	1251.499	180.735
13.34687	1275.932	180.735
13.8412	1299.798	180.735
14.33553	1322.068	181.365
14.82986	1345.96	181.365
15.32419	1370.12	182.09
15.81852	1388.769	182.09
16.31285	1408.747	182.09
16.80718	1417.976	182.09
17.3015	1438.479	181.46

Table B-15. Heating data for F0109T1.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.768956	290.0897	108.564
1.153434	323.1769	114.023
1.537911	366.8907	119.794
1.922389	396.1371	124.998
2.306867	433.4981	129.594
2.691345	460.9347	134.012
3.075823	505.2016	138.43
3.460301	542.7343	142.916
3.844779	580.7428	146.669
4.229256	614.7798	150.283
4.613734	648.469	154.187
4.998212	700.1363	157.875
5.38269	732.8566	160.903
5.767168	770.9164	163.117
6.151646	812.7843	166.149
6.536124	849.9948	167.706
6.920602	887.9348	170.003
7.305079	924.8456	171.646
7.689557	966.965	173.948
8.074035	999.4449	175.429
8.458512	1033.901	176.909
8.842991	1070.662	178.56
9.227469	1098.721	180.87
9.611946	1131.099	181.611
9.996424	1163.49	183.094
10.3809	1195.12	184.011
10.76538	1221.175	185.406
11.14986	1251.118	186.237
11.53434	1273.362	187.069
11.91881	1305.717	187.811
12.30329	1327.203	188.644
12.68777	1351.057	189.296
13.07225	1374.86	189.478
13.45673	1395.925	191.056

Table B-16. Heating data for F0110T2.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.76896	302.067	113.968
1.15343	332.988	119.399
1.53791	369.432	123.88
1.92239	404.851	129.104
2.30687	438.149	133.594
2.69135	478.774	138.827
3.07582	513.121	142.451
3.4603	556.012	146.211
3.84478	593.152	150.046
4.22926	629.791	153.076
4.61373	674.535	156.774
4.99821	712.639	158.992
5.38269	741.546	162.028
5.76717	778.085	164.169
6.15165	826.033	166.546
6.53612	860.379	168.848
6.9206	900.679	170.988
7.30508	931.988	172.551
7.68956	975.252	173.374
8.07404	1008.37	175.024
8.45851	1040.74	176.422
8.84299	1081.37	177.82
9.22747	1115.56	178.732
9.61195	1151.21	179.559
9.99642	1182.2	180.957
10.3809	1203.5	182.44
10.7654	1236.22	183.181
11.1499	1263.74	184.665
11.5343	1288.83	185.317

Table B-17. Heating data for F0112T1.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.76896	286.347	111.52
1.15343	310.943	115.124
1.53791	340.65	119.39
1.92239	367.427	123.661
2.30687	385.478	126.957
2.69135	418.116	130.32
3.07582	447.353	133.829
3.4603	477.65	137.121
3.84478	506.29	140.486
4.22926	525.15	143.929
4.61373	559.128	146.454
4.99821	587.164	149.821
5.38269	617.674	151.505
5.76717	652.76	154.197
6.15165	678.758	156.64
6.53612	710.371	158.41
6.9206	743.317	160.938
7.30508	766.401	162.712
7.68956	799.095	164.398
8.07404	826.842	166.084
8.45851	857.366	166.927
8.84299	885.833	167.954
9.22747	908.762	170.393
9.61195	946.21	171.236
9.99642	967.812	161.114
10.3809	998.609	173.862
10.7654	1027.79	174.61
11.1499	1049.11	175.55
11.5343	1073.02	176.49
11.9188	1099.04	177.237
12.3033	1128.79	178.179
12.6878	1146.16	179.121
13.0723	1171.57	179.121
13.4567	1192.48	180.811

Table B-18. Heating data for F0112T2.

Time (s)	Temp (K)	ρ ($\mu\Omega$ -cm)
0.98866	288.69	111.224
1.48299	318.935	115.375
1.97732	338.674	118.385
2.47164	362.178	122.319
2.96597	384.829	126.493
3.4603	413.932	128.584
3.95463	437.554	132.603
4.44896	464.834	135.616
4.94329	491.813	137.715
5.43762	518.939	141.644
5.93194	542.734	143.746
6.42627	565.648	146.857
6.9206	589.603	148.676
7.41493	619.568	150.881
7.90926	648.957	153.898
8.40359	674.054	155.809
8.89792	700.136	158.127
9.39224	727.327	160.038
9.88657	755.584	160.939
10.3809	777.627	161.945
10.8752	797.276	164.064
11.3696	827.74	165.969
11.8639	854.793	166.077
12.3582	878.902	168.09
12.8526	907.113	169.096
13.3469	924.415	171.221
13.8412	947.064	172.004
14.3355	978.542	172.116
14.8299	1003.45	173.348
15.3242	1021.34	174.242
15.8185	1044.76	174.242
16.3129	1069.44	175.364
16.8072	1086.06	176.257
17.3015	1108.52	177.495

Table B-19. Heating data for F0112T3.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.6591	279.414	111.972
0.98866	327.384	118.522
1.31821	369.432	123.712
1.64776	412.077	130.066
1.97732	460.935	135.232
2.30687	504.112	140.297
2.63642	540.735	144.74
2.96597	590.618	149.247
3.29552	636.503	153.134
3.62508	679.238	156.963
3.95463	726.013	160.168
4.28418	775.423	163.242
4.61373	819.824	165.888
4.94329	858.253	167.841
5.27284	912.577	170.425
5.60239	959.925	172.382
5.93194	998.191	173.71
6.2615	1043.53	175.742
6.59105	1086.06	177.074
6.9206	1123.7	178.261
7.25015	1171.18	179.448
7.57971	1205.74	180.928
7.90926	1235.46	402.039
8.23881	1272.61	457.679
8.56836	1298.37	230.288
8.89792	1335.52	41.641
9.22747	1364.02	176.1
9.55702	1394.83	361.668
9.88657	1417.61	271.298

Table B-20. Heating data for F0116T1.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.768956	290.8659	65.957
1.153434	310.1969	70.018
1.537911	328.8266	72.649
1.922389	340.6497	77.798
2.306867	361.6362	80.391
2.691345	381.0524	85.622
3.075823	398.6763	88.309
3.460301	426.3985	92.205
3.844779	452.4916	97.4
4.229256	465.9767	101.296
4.613734	492.3653	105.192
4.998212	520.0122	109.144
5.38269	542.2085	114.342
5.767168	557.5708	118.363
6.151646	593.6588	122.329
6.536124	606.3659	127.534
6.920602	647.101	131.576
7.305079	668.3406	136.93
7.689557	698.8061	140.842
8.074035	722.3475	144.754
8.458512	761.9695	149.971
8.842991	792.4475	154.044
9.227469	820.7248	158.043
9.611946	852.6618	162.047
9.996424	884.0806	165.967
10.3809	916.3849	169.888
10.76538	946.6373	173.899
11.14986	974.4074	176.7
11.53434	1007.124	180.721
11.91881	1033.488	183.34
12.30329	1064.721	187.367
12.68777	1093.568	191.298
13.07225	1114.759	195.434
13.45673	1135.705	199.579

Table B-21. Heating data for F0116T2.

Time (s)	Temp (K)	ρ ($\mu\Omega$ -cm)
0.768956	299.1748	86.829
1.153434	310.9433	90.906
1.537911	337.9667	96.101
1.922389	368.0961	100.101
2.306867	393.7145	105.301
2.691345	409.0977	109.201
3.075823	438.7425	114.461
3.460301	462.6574	119.789
3.844779	500.3957	123.76
4.229256	526.2173	127.601
4.613734	557.5708	132.809
4.998212	571.7259	136.787
5.38269	607.3699	141.924
5.767168	638.9634	146.059
6.151646	671.7018	151.354
6.536124	703.8376	155.268
6.920602	741.0795	159.35
7.305079	765.5708	163.268
7.689557	800.4584	167.274
8.074035	826.4827	171.284
8.458512	861.7069	175.115
8.842991	896.4969	177.915
9.227469	928.2044	180.626
9.611946	965.6941	184.456
9.996424	994.5079	187.171
10.3809	1024.565	189.888
10.76538	1059.092	192.609
11.14986	1091.23	193.817
11.53434	1116.277	196.643
11.91881	1145.762	199.265
12.30329	1172.664	202.099
12.68777	1194.033	203.518
13.07225	1218.942	204.939
13.45673	1247.382	207.676
13.8412	1265.561	208.77
14.22568	1289.205	209.973
14.61016	1311.55	211.397
14.99464	1335.891	212.598
15.37911	1352.46	212.486

Table B-22. Heating data for F0116T3.

Time (s)	Temp (K)	ρ ($\mu\Omega$ -cm)
0.768956	306.5993	88.171
1.153434	316.7278	92.253
1.537911	346.1142	96.151
1.922389	362.8533	100.049
2.306867	391.7946	105.246
2.691345	409.0977	109.258
3.075823	435.7679	113.16
3.460301	462.6574	118.487
3.844779	491.2595	122.457
4.229256	516.2495	127.668
4.613734	550.4881	131.576
4.998212	579.721	135.555
5.38269	611.78	139.538
5.767168	643.6751	143.525
6.151646	671.7018	148.745
6.536124	698.3309	152.659
6.920602	733.3246	155.35
7.305079	766.8623	159.183
7.689557	792.4475	163.268
8.074035	825.5839	167.186
8.458512	865.5979	169.977
8.842991	893.4426	172.682
9.227469	922.6901	176.607
9.611946	953.2901	179.317
9.996424	989.5609	181.84
10.3809	1016.939	184.65
10.76538	1051.896	185.862
11.14986	1083.718	188.48
11.53434	1110.037	189.789
11.91881	1141.173	192.406
12.30329	1166.159	194.922
12.68777	1192.089	196.539
13.07225	1213.7	199.16
13.45673	1236.529	200.47
13.8412	1261.843	201.886
14.22568	1289.205	203.304
14.61016	1307.513	205.928
14.99464	1329.73	207.13
15.37911	1348.547	207.13

Table B-23. Heating data for F0116T4.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.768956	308.1014	86.92
1.153434	311.6886	91.001
1.537911	344.7184	94.852
1.922389	365.5483	98.801
2.306867	392.4353	102.755
2.691345	404.2239	108.014
3.075823	436.3639	111.918
3.460301	464.8338	117.123
3.844779	492.3653	119.851
4.229256	523.6542	123.76
4.613734	551.4278	128.903
4.998212	572.6507	134.111
5.38269	601.0336	138.09
5.767168	636.5033	140.695
6.151646	661.2996	144.754
6.536124	696.4288	150.049
6.920602	725.0733	152.579
7.305079	746.1107	156.573
7.689557	787.9748	160.572
8.074035	818.111	163.354
8.458512	850.4395	167.186
8.842991	885.8332	169.799
9.227469	914.3082	172.501
9.611946	948.2595	175.115
9.996424	976.8552	177.729
10.3809	1012.036	180.342
10.76538	1040.736	183.052
11.14986	1076.59	185.667
11.53434	1101.856	186.974
11.91881	1125.292	188.48
12.30329	1160.425	190.997
12.68777	1184.538	192.406
13.07225	1210.998	195.024
13.45673	1230.167	196.539

Table B-24. Heating data for F0122T1.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
7.69E-01	320.987	85.578
1.15343	337.258	89.561
1.53791	353.588	93.552
1.92239	380.399	97.501
2.30687	404.224	101.401
2.69134	427.003	105.301
3.07582	454.815	110.501
3.4603	473.816	114.461
3.84478	509.983	118.425
4.22926	536.621	122.393
4.61373	563.168	126.299
4.99821	590.618	130.205
5.38269	616.677	134.181
5.76717	652.273	138.09
6.15165	682.974	143.525
6.53612	707.154	146.212
6.9206	750.202	150.049
7.30508	770.456	154.206
7.68956	804.363	156.819
8.07403	827.74	160.824
8.45851	866.923	163.439
8.84299	895.188	166.054
9.22747	926.052	170.155
9.61195	959.925	171.464
9.99642	993.251	174.173
10.3809	1026.63	176.7
10.7654	1053.53	179.317
11.1499	1081.37	180.626
11.5343	1107.31	183.244
11.9188	1139.19	184.65
12.3033	1168.82	185.959
12.6878	1186.8	188.579
13.0723	1210.69	189.888
13.4567	1245.17	192.609

Table B-25. Heating data for F0122T2.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
1.482986	318.9354	115.375
1.977315	338.6741	118.385
2.471643	362.1775	122.319
2.965972	384.829	126.493
3.460301	413.9322	128.584
3.954629	437.5543	132.603
4.448958	464.8338	135.616
4.943287	491.8126	137.715
5.437616	518.9386	141.644
5.931944	542.7343	143.746
6.426273	565.648	146.857
6.920602	589.6028	148.676
7.41493	619.5675	150.881
7.909259	648.9571	153.898
8.403588	674.0543	155.809
8.897916	700.1363	158.127
9.392244	727.3266	160.038
9.886573	755.5836	160.939
10.3809	777.6265	161.945
10.87523	797.2756	164.064
11.36956	827.7402	165.969
11.86389	854.7932	166.077
12.35822	878.9024	168.09
12.85255	907.1129	169.096
13.34687	924.4147	171.221
13.8412	947.0642	172.004
14.33553	978.5418	172.116
14.82986	1003.454	173.348
15.32419	1021.335	174.242
15.81852	1044.762	174.242
16.31285	1069.442	175.364
16.80718	1086.062	176.257
17.3015	1108.515	177.495

Table B-26. Heating data for F0122T3.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.659105	279.4142	111.972
0.988657	327.3838	118.522
1.31821	369.4323	123.712
1.647762	412.0773	130.066
1.977315	460.9347	135.232
2.306867	504.1115	140.297
2.63642	540.7353	144.74
2.965972	590.6178	149.247
3.295524	636.5033	153.134
3.625077	679.2376	156.963
3.954629	726.0125	160.168
4.284182	775.4226	163.242
4.613734	819.8238	165.888
4.943287	858.2526	167.841
5.272839	912.5765	170.425
5.602392	959.9248	172.382
5.931944	998.1908	173.71
6.261497	1043.531	175.742
6.591049	1086.062	177.074
6.920602	1123.698	178.261
7.250154	1171.176	179.448
7.579706	1205.741	180.928

Table B-27. Heating data for F0124T2.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
0.768956	315.2508	144.71
1.153434	342.0563	147.941
1.537911	370.7651	152.607
1.922389	406.6033	155.614
2.306867	445.1228	158.704
2.691345	470.985	161.797
3.075823	511.4992	164.807
3.460301	542.2085	167.152
3.844779	572.6507	169.323
4.229256	608.8748	170.917
4.613734	647.101	172.604
4.998212	676.8395	174.02
5.38269	706.6807	173.929
5.767168	734.2603	174.682
6.151646	764.648	174.682
6.536124	782.5775	173.838
6.920602	811.0662	174.02
7.305079	846.5232	173.838
7.689557	867.3644	174.02
8.074035	901.5498	174.02
8.458512	922.2588	174.02
8.842991	956.6096	173.267
9.227469	976.8552	173.176
9.611946	1007.124	173.176
9.996424	1030.682	173.358
10.3809	1057.949	173.176
10.76538	1075.778	172.423
11.14986	1100.65	172.423
11.53434	1125.69	172.423
11.91881	1147.737	172.333
12.30329	1172.664	172.513
12.68777	1190.223	172.423
13.07225	1207.984	171.76
13.45673	1227.941	171.76

Table B-28. Heating data for F0124T3.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
7.69E-01	337.258	144.71
1.15343	368.765	148.096
1.53791	402.968	151.776
1.92239	434.096	155.695
2.30687	462.657	158.787
2.69134	501.49	161.797
3.07582	533.977	164.894
3.4603	568.639	167.065
3.84478	597.098	169.411
4.22926	632.657	171.007
4.61373	666.512	172.513
4.99821	698.806	173.176
5.38269	728.265	173.929
5.76717	752.894	174.682
6.15165	784.409	173.838
6.53612	811.88	174.02
6.9206	835.182	173.929
7.30508	869.924	173.929
7.68956	899.46	174.02
8.07403	927.774	174.02
8.45851	952.438	173.267
8.84299	980.564	173.176
9.22747	1005.96	173.267
9.61195	1035.06	173.267
9.99642	1063.5	173.267
10.3809	1082.5	172.423
10.7654	1104.59	172.333
11.1499	1129.51	172.423
11.5343	1150.42	172.333
11.9188	1175.32	172.423
12.3033	1196.67	172.333
12.6878	1217.79	171.76
13.0723	1230.55	171.76
13.4567	1254.85	171.76

Table B-29. Heating data for F0124T4.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
7.69E-01	318.935	143.474
1.15343	335.128	145.527
1.53791	348.895	148.325
1.92239	366.22	150.191
2.30687	393.075	153.822
2.69134	416.888	155.788
3.07582	431.1	157.654
3.4603	458.747	159.623
3.84478	483.143	161.595
4.22926	494.462	163.463
4.61373	516.788	165.331
4.99821	541.788	167.199
5.38269	563.168	168.024
5.76717	584.721	170.112
6.15165	613.781	171.047
6.53612	633.151	171.87
6.9206	651.396	172.804
7.30508	671.222	173.738
7.68956	694.144	173.625
8.07403	705.26	173.738
8.45851	729.672	174.559
8.84299	745.18	173.738
9.22747	766.401	173.851
9.61195	780.378	173.851
9.99642	797.276	173.851
10.3809	814.14	173.738
10.7654	833.481	173.738
11.1499	851.329	173.964
11.5343	868.6	173.851
11.9188	885.395	173.738
12.3033	905.81	173.738
12.6878	919.324	173.625
13.0723	936.195	173.512
13.4567	956.184	172.804
13.8412	966.118	172.692
14.2257	988.386	172.804
14.6102	999.445	172.804
14.9946	1019.76	172.916
15.3791	1033.08	172.916
15.7636	1047.55	172.916
16.1481	1059.5	172.804
16.5326	1078.21	173.142
16.917	1091.23	172.916
17.3015	1108.52	171.982

Table B-30. Heating data for F0124T5.

Time (s)	Temp (K)	ρ ($\mu\Omega\text{-cm}$)
7.69E-01	314.511	143.811
1.15343	337.258	147.113
1.53791	379.744	151.776
1.92239	403.596	155.532
2.30687	437.554	158.621
2.69134	476.524	161.712
3.07582	515.711	163.969
3.4603	540.209	166.977
3.84478	570.698	169.234
4.22926	604.959	170.917
4.61373	644.165	172.333
4.99821	675.015	172.995
5.38269	707.628	174.499
5.76717	734.728	174.499
6.15165	759.195	173.656
6.53612	784.409	173.747
6.9206	815.494	173.656
7.30508	835.182	172.995
7.68956	870.365	173.747
8.07403	900.679	173.085
8.45851	926.483	173.085
8.84299	955.759	172.904
9.22747	979.3	173.085
9.61195	1007.12	173.085
9.99642	1033.49	172.995
10.3809	1059.09	172.904
10.7654	1080.16	172.152
11.1499	1107.31	172.152
11.5343	1122.9	172.243
11.9188	1148.53	172.243
12.3033	1165.77	171.67
12.6878	1191.7	171.67
13.0723	1208.76	171.76
13.4567	1230.93	171.76
13.8412	1244.41	171.58
14.2257	1261.16	171.67
14.6102	1278.12	170.917
14.9946	1296.87	171.58
15.3791	1316.25	171.49
15.7636	1334.78	170.917
16.1481	1347.07	170.828
16.5326	1367.26	170.738
16.917	1379.48	170.828
17.3015	1389.13	170.738

Table B-31. Heating data for F0124T6.

Time (s)	Temp (K)	ρ ($\mu\Omega$ -cm)
6.59E-01	307.351	143.844
9.89E-01	358.922	148.201
1.31821	390.511	152.549
1.64776	435.768	156.468
1.97731	472.119	160.885
2.30687	513.661	163.552
2.63642	560.58	166.71
2.96597	592.646	168.53
3.29552	630.286	170.499
3.62508	670.263	171.837
3.95463	715	172.393
4.28418	740.613	172.242
4.61373	773.217	172.317
4.94329	809.256	172.242
5.27284	849.194	171.686
5.60239	879.781	171.686
5.93194	910.93	171.611
6.2615	949.966	171.13
6.59105	981.828	171.055
6.9206	1008.37	170.905
7.25015	1045.09	170.905
7.57971	1075.78	170.349
7.90926	1102.26	170.199
8.23881	1132.29	170.349
8.56836	1162.7	170.199
8.89792	1186.8	170.199
9.22747	1215.55	170.274
9.55702	1233.54	169.643
9.88657	1263.74	169.569
10.2161	1286.27	169.495
10.5457	1308.64	169.569
10.8752	1333	169.569
11.2048	1358.95	168.864
11.5343	1375.59	169.42
11.8639	1403.07	168.864
12.1934	1418.34	168.864
12.523	1439.93	168.234
12.8525	1462.79	167.678
13.1821	1481.41	167.604

Vita

Michael Kass was born in Trona, California on September 14, 1962. He attended public schools in California, Nevada, and Nebraska. He graduated from Wisner-Pilger High School in Wisner, Nebraska in May, 1980. He entered the University of Nebraska, Lincoln in 1980 and graduated with a Bachelor of Science in Agricultural Engineering. During his junior year at the University of Nebraska, Mr. Kass became interested in materials science. He entered the Master's program in Ceramic Engineering at Clemson University, South Carolina in 1985 and officially received the Master's degree in August 1988. Following graduation, he was employed by the Development Division of the Oak Ridge Y-12 Plant in Oak Ridge, Tennessee. After working for one year, he enrolled at the University of Tennessee, Knoxville to pursue a Doctorate of Philosophy in Metallurgical Engineering. While pursuing the degree, he continued to work full-time at the Y-12 site, and in 1995, he transferred to the Engineering Technology Division of the Oak Ridge National Laboratory. The doctoral degree was received August, 1998.

He is presently working on a variety of projects for the Oak Ridge National Laboratory. Mr. Kass has also been active in several professional societies including the American Ceramic Society and has published over 20 publications.