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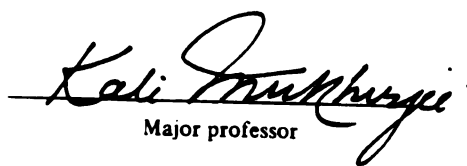
EFFECT OF PLASTIC DEFORMATION ON THE
STABILIZATION OF MARTENSITE IN AN FE-NI ALLOY

presented by

Eileen Jeannette Bryk

has been accepted towards fulfillment
of the requirements for

Master's degree in Metallurgy


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EFFECT OF PLASTIC DEFORMATION ON THE STABILIZATION
OF MARTENSITE IN AN FE-NI ALLOY

By

Eileen Jeannette Bryk

A THESIS

Submitted to
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ABSTRACT

EFFECT OF PLASTIC DEFORMATION ON THE STABILIZATION
OF MARTENSITE IN AN FE-NI ALLOY

By

Eileen Jeannette Bryk

A study of the effect of plastic deformation on the thermal stabilization of the austenite-martensite transformation has been conducted using an Fe-32.4Ni-.08C alloy. Experiments were designed to study the influence of i) Amount of plastic deformation prior to martensite formation, ii) Strain rate used in deformation and iii) Aging time. Other related studies included the effects of austenitizing temperature and of prior deformation on Ms temperature, and the effect of deformation on the temperature at which 55% martensite was obtained.

The degree of stabilization was minimum at 0% deformation, rose as deformation increased to 1%, and remained relatively constant thereafter. With a fixed 2% deformation, higher strain rates produced dramatically lowered stabilization. Also, studies indicate that longer aging time lowers the degree of stabilization.

ACKNOWLEDGEMENTS

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I. Introduction

The transient suppression of the austenite-to-martensite transformation which results from aging treatment is called thermal stabilization. Aging treatments, as well as the cooling procedures, affect the course of the transformation. The transformation from austenite to martensite may be arrested and the partially transformed specimen subjected to an aging treatment at or above the arrest temperature. Upon subsequent cooling, the transformation does not recommence, generally, at the arrest temperature, but at a temperature below the arrest temperature. Thermal stabilization is measured by the temperature interval needed to cause the transformation to recommence. Thus, the degree of stabilization, θ , is the difference between the temperatures of arrest and of the restarting of the transformation after a stabilization anneal. It may be noted that the degree of stabilization is sensitive to the time and temperature of the aging treatment. Also, it is likely that at a given temperature in the transformation range, a specimen which has undergone stabilization will have a lower martensite content than one which has been cooled without interruption.

As a result, stabilization (as well as cooling rate, in some cases) may affect the proportion of retained austenite in martensitic steels. It is desirable to avoid stabilization, and thus retained austenite, in hardened steels because the result may be a softer material subject to structural and dimensional instabilities. This could lead to mechanical failures and a reduction in safety. Thus stabilization is an important phenomenon not only because of its relationship to heat treatment but because of its scientific implications in the martensite transformation.

II. Martensitic Transformations

2.1. Solid-Solid Phase Transformations

Solid state phase transformations may be separated into two categories:

- (1) Nucleation and growth type (diffusion controlled)
- (2) Displacive or shear type (diffusionless)

In typical nucleation and growth transformations, the new phase grows at the expense of the parent phase as the interphase boundary slowly migrates. Neighboring atoms interchange position as they cross the interphase boundary, and they move independently at a rate which varies greatly with temperature. Transformations of this type may occur isothermally, and the amount of new phase increases with time. The transformed region usually has a different volume than that of the original phase.

Displacive or shear transformations are unique in that the new phase forms from the parent phase by cooperative movements of many atoms such that the region of transformation undergoes a change in shape and thus a transformation strain is produced. The change in shape occurs through the additive effect of the translational movements of the matrix. Homogeneous distortion, typical of martensitic transformation, results due to the shifting of

the matrix to maintain as close a coherence as possible with the growing plate. The cooperative movement of atoms occurs with a velocity which approaches $1/3$ the velocity of sound¹. Whereas nucleation and growth transformations may be completed isothermally, the shear transformation usually comes to a halt when the rate of heating or cooling is brought to zero, the transformation resuming only when the temperature change is recommenced. It may be noted that some incidences of isothermal shear (martensitic) transformations have been reported.

Surprisingly, martensitic transformations, which are of the shear type, may propagate at temperatures near absolute zero. Kulin and Cohen² studied this phenomenon in iron-nickel and iron-nickel-carbon alloys and noted that the interface mobility did not seem hindered by the conspicuous absence of thermal activation. Apparently, the interface motion is coordinated, not an atom-by-atom process as in nucleation and growth. In fact, each atom moves less than one lattice spacing relative to its neighbors as a result of transformation.

2.2. General Characteristics of Martensitic Transformations

- (1) A plate-like or a needle-like morphology is typical of martensites; the ratio of thickness to other dimensions is small. The plates become thin toward the extremities and so have a lenticular cross section.

- (2) There exists a definite orientation relationship between the crystal lattices of the original and new phases.
- (3) The growth rate of martensite plates is temperature-independent and is of the order 10^4 - 10^5 cm/sec.
- (4) If other variables (mechanical and thermal history, grain size) are fixed, the extent of transformation will be dependent on temperature. Martensite begins to form spontaneously on cooling at a temperature known as M_s . Upon further cooling, the percentage of material transformed increases until the transformation is completed; this occurs at a temperature known as M_f . In some cases, spontaneous transformation never reaches completion.
- (5) Martensitic transformations are usually athermal but may also be isothermal; both of these types of transformation may occur in the same material. Whereas the athermal mode exhibits a high nucleation rate with no temperature dependence, the isothermal mode has a rate of nucleation dependent on temperature.
- (6) Martensite plates have a regular internal structure; often plates are internally twinned or contain dislocations.
- (7) The transformation strain characteristic of martensitic transformations reveals itself in a macrodeformation, or relief, on the plane surface where the plate is formed. The transformation strain consists of a shear

component lying parallel to the interface (or habit) plane and a dilational component normal to it.

- (8) Martensitic transformations are reversible; the original atomic ordering may be restored and destroyed repeatedly. Consider a single crystal of the parent phase subjected to cooling and transformed into several crystals of martensite. When heated to some temperature which is always above M_s (except for thermoelastic martensites), the martensite will revert to a single crystal with the same size, shape, and orientation as the parent crystal. Upon subsequent transformations, martensite plates that form during cooling have the same size, shape, and location possessed in earlier transformations. Reversibility occurs in almost all martensitic transformations, although there are exceptions. Where reversibility is not seen, interfering secondary events are responsible. An example is the iron-carbon alloys, where the martensite phase is thermodynamically unstable, decomposing into stable phases before the reverse transformation begins.
- (9) Plastic deformation may have various profound effects on martensitic transformations, for example, it may induce martensite at a temperature too high for spontaneous transformation (ie, above M_s). The highest temperature at which martensite forms under stress spontaneously is M_d .

When stresses are applied within the transformation temperature range, the amount of transformation is increased and the reaction may go to completion. Different results are obtained when the plastic deformation is administered significantly above M_s , where the parent phase is stable. In such instances, transformation at any temperature is reduced, and the M_s temperature is depressed (or in rare instances, raised).

In single crystal experiments, the martensitic transformation may be hindered or aided by an appropriately oriented applied stress.

- (10) Cooling regimes may be administered so as to have a negative effect on the transformation. Consider a specimen cooled to some temperature within the transformation range and held for some period of time. The transformation will not usually begin as cooling recommences; the temperature must first be lowered by some increment. This phenomenon is called stabilization, and the extent to which it occurs in a specimen tends to increase with the amount of time for which the temperature is arrested. A general consensus has not been reached as to whether stopping the cooling schedule above the M_s temperature produces stabilization. At all lower temperatures, the percentage transformation tends to be lower than had the specimen been cooled directly to the given temperature.

2.3. Athermal and Isothermal Transformations

As mentioned previously, martensitic transformations are usually of an athermal-kinetic type of behavior. A fraction of the total volume becomes martensite at a temperature below M_s at a rate that is independent of temperature. It has been observed that more transformation occurs with decreased temperature and that its extent is a function of temperature. Bunshah and Mehl³ have found that martensite plates in an Fe-29.5Ni alloy propagate at about 10^5 cm/sec, and have a formation time of 0.05-0.5 μ sec. They also found that the propagation velocities of athermal and isothermal martensites were equal.

The isothermal component of martensite transformations, on the other hand, is either not operative or is obscured by the predominant athermal component and hence is not often observed. Due to the fact that martensitic transformations are strain sensitive and autocatalytic, athermal transformation may promote the nucleation of isothermal martensite when cooling is stopped below M_s . The transformation kinetics are completely different from that of the athermal transformation; a normal C-curve behavior is seen on a TTT diagram. This C-curve behavior would imply that the isothermal transformation is thermally activated. Quite the contrary: the plate formation time and linear growth velocity are the same as those of athermal martensite. Also, isothermal transformation increases the volume fraction of martensite due to the formation of new plates,

not because of growth of existing plates. With these observations, we can conclude that isothermal transformation is dependent on the thermally activated triggering of embryos. Growth does not require further thermal activation once embryos are triggered.

2.4. Autocatalysis and Partitioning Effects

After a martensitic transformation begins, either athermally or isothermally, its course may be affected by autocatalysis. When this phenomenon occurs, the initial rate of transformation is higher and preferred sites for nucleation form in the parent phase due to the perturbation around existing plates. A chain reaction may take place and plates may form end to end.

Further complicating the reaction is the partitioning of the parent phase, which contributes to the subsequent decrease in transformation rate. Nucleation occurs in progressively smaller volumes of austenite, therefore, the volume fraction becoming martensite per nucleation event is decreased and the size of newly formed fully grown plates decreases. A formal theory of this process is given by Fisher⁴.

The effects of autocatalysis and partitioning must be considered in the transformation rate equation. A semiempirical rate equation of the following form can be used⁵.

$$\frac{df}{dt} = (N_i + pf - N_u) (1 - f) v \exp(-\Delta W_a/RT) m q (1 - f)^{1+\frac{1}{m}} \quad (1)$$

where: f = fraction transformed

t = time

N_i = number of pre-existing nucleation sites

p = number of autocatalytic embryos per unit volume

N_u = number of martensitic plates per unit volume

v = lattice vibration frequency

ΔW_a = activation energy of nucleation at
temperature T

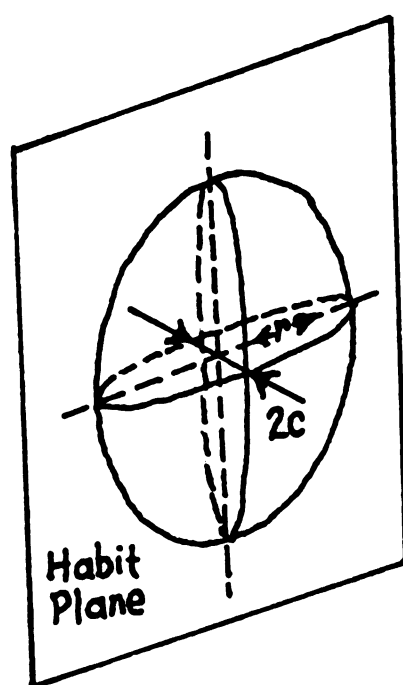
m = thickness-to-diameter ratio of plates

q = average volume per grain of austenite

This equation can be numerically integrated using metallographically determined values of q and m and unknown quantities p and ΔW_a which are obtained by curve fitting. The equation predicts experimental results well when less than 10% transformation has occurred⁵.

2.5. Theories of Martensite Nucleation

As mentioned previously, the kinetics of martensitic transformations are dependent mainly on nucleation, not growth, since each plate grows rapidly to its final size. We might begin our review of nucleation theories by considering a simple model of a martensite plate⁶. The plate is shown in Figure 1 and has an oblate spheroid shape.



$$\text{Volume} \approx \frac{4}{3} \pi r^2 c$$

$$\text{Surface Area} \approx 2\pi r^2$$

Figure 1. Schematic diagram of martensitic plate or embryo.

The plate radius is r and semi-thickness is c , where $r \gg c$. Its interfacial free energy per plate is:

$$V \Delta g_s^{P \rightarrow M} = 2\pi r^2 \lambda \quad (2)$$

Where V is the plate volume, $\Delta g_s^{P \rightarrow M}$ is the surface energy per unit volume, and λ is the interfacial energy.

Linear elasticity theory allows us to compute the strain energy for the plate:

$$V \Delta g_e^{P \rightarrow M} = \frac{4}{3} \pi r^2 c \left(\frac{Ac}{r} \right) \text{ (per plate)} \quad (3)$$

In this case, $V = \frac{4}{3} \pi r^2 c =$ plate volume, and (Ac/r) is the strain energy per unit volume. The factor A is of the form:

$$A = \{ \pi(2 - \nu)/8(1 - \nu) \} \mu r_o^2 + \frac{\pi}{4} \mu \epsilon_o^2 \quad (3a)$$

or

$$A = \mu(r_o^2 + \epsilon_o^2) \quad (3b)$$

where ν = Poisson's ratio, μ = shear modulus, and r_o and ϵ_o are shear and dilatational strains respectively that are associated with shape deformation. The elastic constants for the parent and martensitic phases are assumed equal.

Designating $\Delta g_c^{P \rightarrow M}$ as the chemical free energy per unit volume of martensite, we have:

$$V \Delta g_c^{P \rightarrow M} = \frac{4}{3} \pi r^2 c \Delta g_c^{P \rightarrow M} \text{ (per plate)} \quad (4)$$

Note that below T_0 , Δg_c^{P+M} is negative. The total free energy change due to the formation of a plate is:

$$\Delta W^{P+M} = -\frac{4}{3} \pi r^2 C \Delta g_c^{P+M} + 2\pi r^2 \lambda = \frac{4}{3} \pi r C^2 A \quad (5)$$

Classical nucleation theory allows us to determine the free energy of nucleation W^* corresponding to a critical radius r^* and critical semi-thickness C^* . To do this we set:

$$\begin{aligned} \text{and} \quad \frac{\Delta W^{P+M}}{r} &= 0 \\ \frac{\Delta W^{P+M}}{C} &= 0 \end{aligned} \quad (6)$$

This yields:

$$C^* = 2\lambda / \Delta g_c^{P+M} \quad (7a)$$

$$r^* = 2Ac^* / \Delta g_c^{P+M} \quad (7b)$$

$$V^* = \frac{4}{3} \pi r^{*2} C^* \quad (7c)$$

$$W^* = 32\pi A^2 \lambda^3 / 3 (\Delta g_c^{P+M})^4 \quad (7d)$$

$$C^{*2} / r^* = \lambda / A \quad (7e)$$

If the nucleation is completely random, each atom is a potential nucleation site and the random nucleation rate N is given by:

$$N = (N_O / V_m) \nu \exp (-W^* / kT) \quad (8)$$

where (N_O / V_m) = number of atoms per unit volume, ν is the lattice vibration frequency (10^{12} /sec).

The nucleation kinetics predicted by equation (8) should follow an isothermal C-curve behavior, with the maximum nucleation rate being at the knee of the C-curve.

To summarize, in the classical model, a martensite embryo becomes a critical size at some temperature and becomes a nucleus. The classical model of homogeneous nucleation may be used to compute V^* , c^* , r^* and W^* by using experimental values of Δg_C^{P+M} and theoretical values of A and λ .⁶

Various experimental results and theoretical postulations supply evidence that the homogeneous classical nucleation model does not apply to martensitic transformations. In fact, the evidence suggests that the reactions are heterogeneous.

Metallographic studies provide the strongest evidence that martensite nucleates at preferred sites and not in a random manner. Beta brass was selected for study⁷ on the merit that its martensitic transformations are conveniently reversed. It was found in repeated transformation cycles that the position and the sequence in which individual plates formed remained almost unchanged. It appears, from this experiment, that the parent phase contained preferred nucleation sites.

Cech and Turnbull⁸ studied the transformation kinetics of an Fe-29.2 Ni alloy, and their results also shed some light on this subject. Particles of the alloy, having

diameters of 37-100 μ , were austenitized and quenched to the martensitic transformation temperature range. Temperatures at which the martensitic transformation began varied widely among the particles. Some of the particles underwent no transformation even at the lowest temperatures attained. The researchers concluded that structural singularities, or heterogeneities, governed the nucleation in this alloy. Evidently, some of the particles contained fewer effective sites (heterogeneities) than others, and therefore required further supercooling. It is hypothesized that the probability that an effective heterogeneity exists increases with particle size. Experiments by Huizing and Klosterman⁹ on single crystal Fe-Ni spheres have yielded similar results.

In the classical models of nucleation, the Boltzmann probability factor [$\exp(-\Delta W^*/kT)$] appears in the nucleation rate equations of the type of Eq. (8). This practice has been criticized by Crussard¹⁰. The Boltzmann factor arises from the supposition that atoms constitute a system of separate oscillators possessing a characteristic frequency. Crussard disputes its use, contending that the statistical reinforcements of elastic waves supplies the energy for the thermally activated process being considered. Using quantum theory, he proposed an alternative probability factor, which predicts a finite rate of nucleation at 0°K and an operative homogeneous mode. The ramifications are that isothermal and athermal transformations may be explained in

this way and that heterogeneous nucleation need not be considered. However, as mentioned previously, experimental observations at very low temperatures suggest the homogeneous nucleation aspect included in Crussard's modification is deficient.

Numerous other theories of nucleation share Crussard's perspective that the nonclassical view is a valid one. In contrast with the classical nucleation model in which an embryo reaches a critical size at a given temperature and becomes a nucleus, Cohen¹¹ has proposed a "reaction-path" model, which is non-classical. In this model, within the volume where nucleation occurs, the lattice progresses through a series of states which leads the parent phase to become martensite. The primitive atom movements are thought to take place in a synchronized manner, the lattice strain generating a sequence of intermediate structures in any given region before propagating out like a strain wave. The series of states is visualized as a reaction path possessing an energy barrier between the initial and final states. From this perspective, activation of the embryos is due to fluctuating atomic configurations, not embryo size. The embryo associated with the reaction path model could be a strain center composed of lattice imperfections such as dislocation arrays. The strain center is assumed to be part of the way along the "reaction-path", and above M_s , it is a region of high free energy. Hollomon and Turnbull¹² have

added interfacial energy considerations to the reaction path model.

Frank's¹³ model of the austenite-martensite interface was used by Knapp and Dehlinger¹⁴ in their formulation of athermal martensite kinetics. They took into account the free energy balance of the embryo. The martensite embryo is viewed as a thin oblate spheroid with dislocation loops in the interface; it is thought that the loops contain mainly screw dislocations and that short edge components join the positive and negative screws. The embryo model is shown in Figure 2. Embryo growth in the $[110]_{\gamma}$ and $[225]_{\gamma}$ directions is achieved by the dilation (or growth) of the dislocation loops. Growth in the $[554]_{\gamma}$ directions entails the creation of new loops. As the synergetic motion of the loops allows growth of the martensite embryo, the dislocation interface sweeps through the austenite.

The energy required to create and expand the dislocation loops, the interfacial energy, and the strain energy, is supplied by the chemical driving force $\Delta g_c^{\gamma \rightarrow \alpha'}$. The embryo, as viewed by Knapp and Dehlinger, will be triggered when the chemical driving force is greater than the required interfacial and strain energies. This begins to occur when the total free energy change per unit volume becomes zero. By writing $\Delta W^{\gamma \rightarrow \alpha'}$ as $\frac{1}{V} \Delta W^{\gamma \rightarrow \alpha'}$ and maintaining our established notation for surface and strain energies, we write:

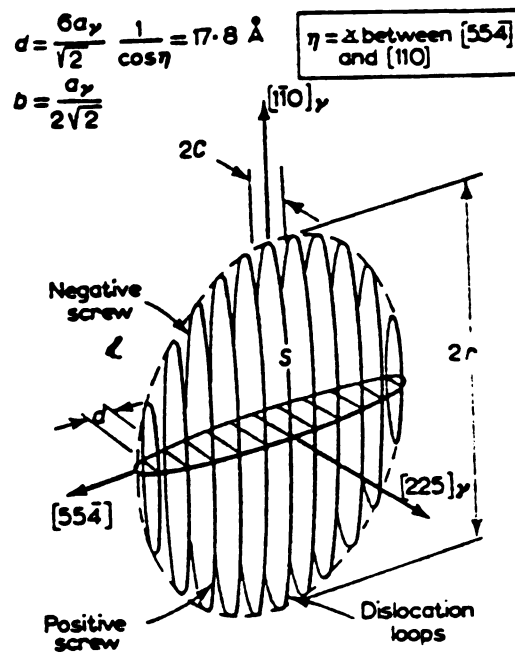


Figure 2. Knapp and Dehlinger model of the martensite embryo.

$$\Delta W^{\gamma+\alpha'} = -\Delta g_c^{\gamma+\alpha'} + \Delta g_e^{\gamma+\alpha'} + \Delta g_s^{\gamma+\alpha'} = 0 \quad (9)$$

For spontaneous triggering of the embryo, then,

$$\Delta g_c^{\gamma+\alpha'} = (\Delta g_e^{\gamma+\alpha'} + \Delta g_s^{\gamma+\alpha'}) \quad (10)$$

The last two terms in eqn. (10) can be written as Δg non-chem and equations (2) and (3) for an oblate spheroid may be used to yield:

$$\Delta g \text{ non-chem} = \frac{3\lambda}{2c} + \frac{Ac}{r} \quad (11)$$

where r , c , λ , and A retain their previous meanings. For a specified radius, Δg non-chem can be minimized relative to c by setting $\partial g \text{ non-chem} / \partial c = 0$. The minimum value of Δg non-chem is calculated to be

$$\Delta g \text{ non-chem}_{\text{minimum}} = \left(\frac{6\lambda A}{r} \right)^{1/2} = \frac{3\lambda}{c} \quad (12)$$

when

$$c = \left(\frac{3\lambda r}{2A} \right)^{1/2} \quad (13)$$

Equations (12) and (13) may be used to calculate Δg non-chem as a function of embryo size. Equation (12) shows that for a fixed value of c , $\Delta g \text{ non-chem}_{\text{minimum}}$ decreases with increasing radius, r . The implications are two fold: (a) the non-chemical energy barrier decreases as the embryo size increases, and (b) after the embryo starts to grow,

this barrier becomes smaller, enabling rapid interface movement. Below M_s , $\Delta g_c^{\gamma+\alpha'} > \Delta g_{\text{minimum}}^{\text{non-chem}}$ for the larger embryos, causing a net driving force which acts as a stress on the dislocation interface. The interface is thus swept outward, creating martensite from the embryo. With this model, the approximate size of the largest embryo at M_s may be found by using experimental and estimated values of the various parameters. Raghavan and Cohen¹⁵ developed this type of calculation further.

The model described above has been modified by Kaufman¹⁶ so that an equivalent dislocation loop (on the habit plane) encircling the oblate spheroid embryo is considered. The interfacial energy is within the dislocation loop. The loop's radius is r and its equivalent Burger's vector, ζ , is given by cb/d ; c is the semithickness, b is the Burger's vector of the lattice, and d is the distance between the loops in Knapp and Dehlinger's model (Figure 2). The loop has a line tension of $\Gamma = \mu\zeta^2/2$; μ is the shear modulus. If a net shear stress τ is imposed on the loop, the accompanying increase in free energy (starting from zero size) is

$$W^1 = 2\pi r\Gamma - \pi r^2 \zeta \tau \quad (14a)$$

where

$$\Gamma = \mu\zeta/2 \quad (14b)$$

The shear stress τ is equal to the chemical driving force ($-\Delta g_c$) minus the strain energy per unit volume (Ac/r). We then have

$$\tau = -\Delta g_c - \frac{Ac}{r} = -\Delta g_c - \frac{\lambda}{c} \quad (15)$$

where λ is the surface energy. Noting from eqn. (7e) that $c^2/r = \lambda/A$ and substituting from eqns. (15) and (14b) into eqn. (14a) yields:

$$W^1 = \frac{\pi b \lambda r^3}{d} \left[\frac{\mu b}{A d} + \Delta g_c \left(\frac{r}{A} \right)^{1/2} + 1 \right] \quad (16)$$

maximizing W^1 in terms of r , we have

$$r_c = \frac{9A\lambda}{\Delta g^2} \quad (17)$$

and

$$W_c^1 = \frac{9\pi A^2 \lambda^3}{2\Delta g^4} \quad (18)$$

In this case, r_c is the critical radius of the loop at which it may expand, thereby lowering its energy. This growth model, proposed by Kaufman, resembles aspects of mechanical twinning or kink band formation. Comparing with eqn. (7b),

$$r_c = \frac{9}{4} r^* \quad (19)$$

Because $r_c > r^*$, ΔW^* has a negative value at r_c .

A proposal has been made by Machlin and Cohen¹⁷ that embryos with radius r , where $r^* < r < r_c$ (r^* is the radius in the previous model) are activated during isothermal transformation. It may be noted that when $r < r^*$, W^* is of such a large magnitude that thermal activation is impossible

and also that embryos with $r > r_c$ are already triggered by the athermal process.

There are still other models of heterogeneous martensite nucleation to be considered. Olson and Cohen¹⁸ proposed a model in which part of a wall of dislocations sustained faulting on close-packed planes, yielding the close-packed phase in the fcc to hcp transformation. In order for the fcc to bcc transformation to occur, a second shearing is required. An activation barrier is not included in their thermodynamic equations. However, it has been proposed by Magee¹⁹ that a barrier exists due to the thermally activated motion of the dislocations associated with the internal slip.

It might be mentioned at this point that the free energy change for nucleation of a martensite embryo within an expanding dislocation loop was calculated by Easterling and Thölen²⁰. In the case of internally twinned martensite, there was no apparent nucleation barrier. They chose 20 ergs/cm² for the interfacial energy of a martensite embryo; this contrasts greatly with the Kaufman and Cohen¹⁶ estimate of 200 ergs/cm² which would be a prohibitively large nucleation barrier in their model.

It has been suggested²¹ that heterogeneous martensite nucleation may be initiated at dislocation pile-ups, and also that an embryo at a pile-up location may experience the classical free energy barrier to nucleation²¹.

III. Prior Work

3.1. Isothermal Martensite Formation

Most martensitic transformations are classified as athermal; they are of a temperature-dependent nature in that the reactions proceed only when the temperature is changing. Fletcher, Averbach, and Cohen^{22,23} in 1948-49 found a time-dependent component to be present in some cases. Although up to 5% isothermal martensite formation occurred in their work on plain carbon and low alloy steels, the isothermal nature was not recognized as such. It was mistakenly thought to be a "tailing-off" effect that usually appears at the beginning and final stages of athermal transformations.

Kurdjumov and Maximova^{24,25} treated isothermal martensite as a separate phenomenon. They were able to achieve 25% isothermal formation of martensite in an Fe-0.6C-2Cu-6Mn alloy. They did this by rapidly cooling their specimens to -180°C which completely suppressed the transformation, then reheating and holding isothermally at various temperatures between -80° and -160°C. The transformation kinetics thus obtained had a C-curve behavior on a TTT diagram. At one of the experimental temperatures, the initial isothermal transformation rate was constant and

gradually decreased. There was a temperature at which the greatest amount of isothermal martensite was formed.

In other experiments, they quenched a 1.6C steel in alkaline iced water, forming approximately 20% martensite, and then cooled the specimens to liquid nitrogen temperature. Though only partial suppression of the transformation was introduced, this technique ensured that the amount of athermal martensite formed was fixed, and that upon reheating the isothermal transformation could be studied.

Das Gupta and Lement²⁶ encountered partial suppression of the transformation in a Fe-15Cr-0.7C steel. Some athermal martensite formed before the isothermal component was allowed to form. They observed that the initial rate of isothermal transformation increased with decreasing temperature, reached a maximum at -110°C and then decreased below this temperature. Specimens cooled to liquid nitrogen temperatures so that a constant initial amount of martensite was present were up-quenched and held at various subzero temperatures. Between about 3% and 8% isothermal martensite formed.

Machlin and Cohen²⁷, using an Fe-29Ni alloy, found also that the isothermal transformation followed partial athermal transformation. The isothermal transformation rate was determined to be a function of the amount of athermal martensite present, the temperature and time of isothermal holding, and the state of internal strain. The nucleation of new plates, as opposed to the growth of existing ones, is thought to be responsible for isothermal transformation.

Kulin and Speich²⁸ found that isothermal martensitic transformation occurs above the M_s temperature in an Fe-14Cr-9Ni alloy. A higher percentage of transformation occurred on holding isothermally at high temperatures than on quenching to low temperatures.

Cech and Holloman²⁹ worked with an Fe-23Ni-3.7Mn alloy and found that the rate of isothermal transformation increased with decreasing temperature, reached a maximum at -128°C , and decreased with further lowering of temperature.

It may be seen from the various studies mentioned that isothermal martensite formation is not dependent on whether athermal transformation has preceded it. At the same time, if athermal transformation has not consumed too large a portion of the parent phase, it may spur the nucleation of isothermal martensite when cooling is stopped below M_s . This is due to the strain sensitive and autocatalytic nature of martensitic transformations. With such a relationship between the two modes of martensitic transformation, it is clear that isothermal transformations are best studied in the absence of athermal martensite. As noted, however, sometimes this condition is impossible to meet. At the same time, one wishes to study isothermal kinetics without variable amounts of athermal martensite obscuring them. Quenching below the lowest isothermal level to be studied would yield a fixed quantity of athermal martensite, solving the problem. Temperatures could then be raised to study isothermal kinetics.

The isothermal kinetics yield a C-curve behavior whether there is no athermal martensite or a fixed amount is present. Maximum transformation rates occur at some intermediate temperature, typically between 100-150°K.

3.2. Burst Formation of Martensite

In some cases, the internal accommodation stresses from the martensitic plates give an autocatalytic effect known as "burst phenomenon". It manifests itself in a rapid, sudden transformation of a large percentage of the specimen at or below the M_s . Burst transformation may occur when transformation resumes following an aging treatment and thermal stabilization. It may also be seen in some cases after long isothermal holding treatments, after sufficient incubation period.

Upon metallographic examination, the martensitic plates are seen to be appreciably wider than normal plates. They form a zig-zag pattern in the specimen. Stresses produced by one plate aid in the activation of another nucleus, and so the burst transformation is like a chain reaction. The implication is that the burst transformation is one in which the stresses in the matrix are reduced. The plates formed in a burst transformation to some extent comprise a self-accommodating system of stress. They may be the reason that the plates are wider than normal.

Working with Fe-Ni-C alloys, Entwistle and Feeney³⁰ found that M_b , the temperature at which burst occurs, is dependent upon the heat treatment given to the austenite. The

percentage transformation in a burst was seen to be a function of austenite grain size and M_b .

There are several explanations offered for the burst phenomenon. Machlin and Cohen³¹ feel that the plastic deformation of the parent phase when martensite plates form creates embryos, or that existing embryos become supercritical at a given temperature.

Suzuki and Honma³² see burst transformation as analogous to deformation twinning in that it requires the dislocations encompassing the embryos to be able to multiply on favored habit planes. This mechanism of multiplication is not likely to happen on the $\{225\}$ type of habit planes. In those cases, the reaction proceeds by a mechanism similar to slip.

3.3. Stabilization

Harris and Cohen³³ performed the first organized study of stabilization using a 1.1C-1.5Cr steel. They found that stabilization does not occur unless the temperature of holding is below a certain temperature, T_s , below M_s . The degree of stabilization, θ , increased linearly as the temperature of holding was decreased.

Das Gupta and Lement³⁴ worked with an Fe-15Cr-0.7C steel and found that stabilization against athermal and isothermal transformation does not occur to a significant extent unless some transformation has preceded. In fact, when the amount of initial martensite exceeding what they referred to as the

"critical limit" was present before reaching the holding temperature, stabilization is more pronounced. They found that stabilization against isothermal transformation increases with cycling temperature and time spent at the cycling temperature. Also, they noted that for a fixed amount of initial martensite, a higher intermediate cycling temperature yielded a more permanent stabilization at the subzero reaction temperature.

Morgan and Ko³⁵ found that stabilization of austenite occurs during continuous cooling and also with isothermal holding both above and below M_s in steels with approximately 1% C and 0-5% Ni. The rate of stabilization was a function of the austenite composition, and it increased with both temperature and the presence of martensite. When stabilization above M_s occurred, M_s was depressed and the amount of austenite at a temperature near M_s was increased.

Edmondson³⁶ studied the effect of aging time and temperature on stabilization in an Fe-10Ni-1C steel. The extent of stabilization reached higher limiting values the lower the aging temperature. At relatively higher temperatures of aging, the value of θ decreased on prolonged aging after reaching a peak. The temperature dependence of θ is fairly pronounced in the Ni alloys.

Woodilla, Winchell, and Cohen³⁷ studied stabilization kinetics in an Fe-30.8Ni-.007C alloy. When they removed the interstitial elements (C,N) by moist-hydrogen treatment,

stabilization would not occur. When .007C was present, the activation energy for stabilization was commensurate to that of the diffusion of C or N in ferrite (rather than in austenite). They interpret this to mean that interstitial diffusion governing the stabilization occurs within the martensite embryos, not in the matrix of the parent phase. According to them, the kinetics imply that the interstitials diffuse from the embryo toward the surrounding matrix, immobilizing the austenite/martensite interface and causing stabilization.

In their work on the thermal stabilization of the athermal martensite transformation in Fe-Ni-C alloys, Kinsman and Shyne³⁸ found that aging temperature had a distinct effect on the character of stabilization; the results were markedly different for low vs. high aging temperatures. Within each of the lower aging temperatures used, θ increased, reached a maximum, then decreased with time. Increasing the aging temperatures decreased the maximum stabilization, $\theta_{\max}$, and the time of aging to reach $\theta_{\max}$. Their results are similar to those of Odaka and Okamoto³⁹, Glover⁴⁰, and Priestner and Glover⁴¹, who also found that after long aging treatment, beyond $\theta_{\max}$, θ increases again. In some cases the second increase of θ is attributed to the start of the bainite reaction. The transformation rate after stabilization was consistently found to be higher than that immediately preceding the transformation arrest and aging treatment. When the percentage of martensite present before

aging was increased, the degree of stabilization was more pronounced regardless of aging time and temperature. Stabilization did not occur when the carbon content was less than .002 wt%. Some experiments were done to test the "reversibility" of the stabilization. In some control cases, aging temperatures were held constant for the duration of aging treatment. In other cases, the aging temperature was changed during the treatment. For low temperature aging, the extent of stabilization approached a value typical of the last aging time and temperature used in the sequence, thus the stabilization was termed "reversible".

When high aging temperatures were used, the results were different. In partially martensitic specimens, the degree of stabilization as a function of time was constant for a sizeable interval, rising at longer aging times. The degree of stabilization at any aging time tended to be higher when the aging temperature was increased. Also, reversibility of thermal stabilization was not observed, in contrast with the reversibility of the low temperature results. In this case, the value of θ always approached the value associated with the highest aging temperature in the sequence.

Glover⁴⁰ investigated the effect of aging time and temperature of a partially martensitic 1.4%C steel on subsequent transformations. Higher aging temperature yields lower maximum values of θ . Regarding stabilization, Glover compares the influence of nickel content to that of Mn^{42} and

of Cr which was determined in a 1.5Cr-1C steel, and sees that Mn and Cr greatly diminish the aging temperature dependence of $\theta_{\max}$ (the maximum stabilization which occurs). In these cases, θ increases with aging temperature and reaches a limiting value which seems to be temperature-invariant. Comparing the effects of Ni³⁶ to those of Mn and Cr just mentioned, he makes an interesting hypothesis. In light of the fact that stabilization appears to involve carbon atoms, the information suggests that its sensitivity due to aging temperature is decreased when elements with an affinity for carbon are present and increased when elements which do not form stable carbides are present.

Guimarães and Shyne⁴³, using an Fe-31Ni-.01C steel, demonstrated that for a given temperature of arrest (prior to aging treatment) the value of θ is the same independent of the extent of prior deformation. Deformation consisted of up to 75% cold rolling.

Guimarães⁴⁴ conducted another investigation on the same alloy where some of the specimens were annealed and some had 25% deformation due to cold-rolling at room temperature. All of the specimens were cooled to an arrest temperature of -72°C, heated and aged, and subcooled to determine θ . The result: the extent to which the annealed and the deformed specimens underwent stabilization was equal, confirming the results of Guimarães and Shyne⁴³.

Priestner and Glover⁴¹ worked with an Fe-5Ni-1.43C steel whose Ms was near room temperature. Aging temperatures above 50°C were used and it was found that θ decreased with time, became negative, reached a minimum, and rose again.

3.4. Theories on Stabilization

There are numerous instances of stabilization reported in the literature; all might not be attributed to the same cause. Generally, theories of stabilization deal either with change in the austenite phase or with the change in effect that previously formed martensite has on the later transformation. It is widely thought that stabilization of austenite results from stress relaxation of the parent phase in the region of the martensite plates. The stress mentioned is thought to promote nucleation in unstabilized austenite, and its relaxation would have a stabilizing effect. Various investigators propose different mechanisms by which this relaxation takes place. The common element is that thermal stabilization is recognized as a thermally activated process controlled by interstitial diffusion (usually C or N) kinetics. Models may be categorized in three general groups: one³⁷ attributes the effect to solute atoms pinning (locking) the austenite/martensite interface, another⁴⁰ to the diminished autocatalytic effect of existing plates, and the last³⁵ to strain aging that would strengthen the parent matrix.

Das Gupta and Lement³⁴ and Cohen, Machlin, and Paranjpe⁴⁵ hypothesize that solute atoms diffuse to strain centers in the parent phase. This lowers the driving force for transformation in regions that might have otherwise been preferred nucleation sites. Das Gupta and Lement³⁴ feel that the driving force for C-diffusion from martensite to strain embryos is given by the higher activity of carbon in martensite as opposed to that in austenite. Carbon atoms would be motivated to diffuse from the edges of martensite plates to nearby strain embryos.

Another possibility⁴⁶ is that a condition analogous to dislocation pinning may occur due to slight concentration build-up at the embryo or plate interface, immobilizing it.

Hollomon, Jaffe, and Buffum⁴⁷ suggest that the coupling between the austenite and martensite may be somewhat diminished by time-dependent yielding on relaxation.

Morgan and Ko³⁵ feel that stabilization may occur upon strengthening of the austenite due to formation of Cottrell atmospheres, which hinders displacements in martensitic transformations. Crussard⁴⁸ feels that precipitate formation may also have the same effect.

Kinsman and Shyne³⁸ have developed a model which is based on Knapp and Dehlinger's embryo model. They suggest that stabilization results from the inability of the semicoherent austenite/martensite nucleus interface to move due to migration of interstitial solute atoms to the dislocation array making up the interface. The kinetics of solute

segregation to the nucleus interface govern the time and temperature dependence of stabilization. Thus, deriving the time law for solute segregation enabled them to develop a time law for stabilization.

Glover and Smith⁴² feel that elastic stresses are relaxed due to (carbon and nitrogen) interstitial diffusion within the martensite, leading to the initial stages of tempering and causing stabilization.

Maximova et al⁴⁹ suggest that relaxation as well as the crystal defects that it produces cause stabilization; the defects interfere with plate growth.

Kurdjumov⁵⁰ suggests that a partially martensitic specimen may experience readjustment of the stresses and defects that would otherwise tend to enhance transformation. This process yields a more stable structure which is thus more difficult to transform.

3.5. Effect of Plastic Deformation on Martensitic Transformation

Elastic stress fields and externally applied plastic deformation influence martensitic transformations. It has been observed that a specimen will transform spontaneously at the M_s temperature under zero applied stress. When a stress field is applied, however, it affects the shape deformation of small regions of martensite, and alters the net driving force for their expansion. Whether the effect is positive or negative depends on both the nature of the stress field and the plate orientation. The M_s temperature may thus be

increased or decreased if it is a function of this net driving force. Frequently, the M_s temperature will be raised because the driving force for some of the habit plane variants will increase.

Sometimes, the M_s values may change by a much greater extent when deformation occurs, i.e. stresses beyond the elastic limit are applied. Transformation may occur above M_s , the temperature at which it occurs rising as the applied stress is increased. The highest temperature at which transformation may occur in this manner is M_d .

Stress applied to the parent phase above the M_d temperature may result in plastic deformation, and the matrix usually becomes mechanically stabilized. Subsequent cooling reveals a decreased M_s temperature in these cases.

It is possible that the defects introduced by plastic deformation obstruct the growth of the martensite phase. Sometimes the opposite occurs when small deformations are administered; the M_s may be raised due to the formation of internal stress concentrations promoting martensite nucleation or nuclei growth.

Reed⁵¹ subjected austenitic polycrystalline Fe-31Ni to plastic deformation at room temperature and found on subsequent cooling that the M_b temperature was suppressed. Bokros and Parker⁵² observed increased M_b temperatures in plastically deformed Fe-31.7Ni single crystals.

Brownrigg⁵³ studied the relationship between plastic deformation and isothermal martensite kinetics. Austenitic

specimens with small deformations (administered above M_d) exhibited slightly increased initial transformation rates, which progressively decreased as the amount of prior deformation increased. In all of the deformed specimens, at all of the isothermal holding temperatures, further progress of the transformation was retarded in comparison with undeformed specimens. It is suggested that the enhanced transformation rates are the result of a decreased activation energy for nucleation. Higher amounts of deformation inhibit (retard) the transformation; it is thought that the increased number of dislocations interferes with the mechanism of embryo propagation.

Many aspects of martensite kinetics are related to the interface resistance to martensite plate growth, suggest Magee and Paxton⁵⁴. The kinetics may also be related to the ease of dislocation motion required in embryo formation in heterogeneous nucleation. Whether nucleation or growth is the controlling step, the researchers feel that the transformation is profoundly affected by the resistance of the parent phase to dislocation motion.

Working with Fe-Ni and Fe-Ni-C alloys, Patel and Cohen⁵⁵ determined criterion that quantitatively predicted the effect of applied stress on the M_s temperature. A mechanical work term which is the product of the applied stress field and the transformation strain is added to the chemical free energy change which supplies the driving force for transformation. Thus the change in temperature at which the critical

thermodynamic driving force is attained and the transformation initiated may be computed.

Using several alloy steels (including Airkool-S, with 5% Cr), Breinan and Ansell⁵⁶ found that when austenitic specimens were deformed (above M_d) to obtain greater flow stresses, the M_s temperature was lower the higher the austenite yield strength was. The decrease in M_s was linear with increase in austenite flow stress. They suggested that this was caused by a reduction in dislocation mobility. Such a reduction might influence either the ease of nucleation or the ability of martensite plates to propagate. Further transformation was affected also, in the sense that the percentage transformation at 25°C was lower, the higher the austenite flow stress. When alloying increased the yield strength, M_s decreased in a similar way. Similar work was done by Ankara⁵⁷, who subjected an Fe-30Ni alloy to transformation cycling; this increased the austenite yield strength. The result was a decrease in M_s with increased yield strength. Ankara attributes this to a change in dislocation density.

Plastic deformation also affects the morphology of martensite subsequently formed. Bokros and Parker⁵² worked with single crystals of Fe-37Ni, and found a large difference in morphology between deformed and undeformed crystals. They found that previously strained crystals exhibited increased M_s temperatures and a decrease in the amount of transformation occurring in a burst. This is also an apparent

reduction in the number of habit plane variants operative during the burst transformation. Because transformation progresses along variants of favored groups, the morphology is simplified. It was found that substantial regions of the strained crystals underwent a burst transformation due to the operation of four habit-plane variants whose poles group about a common $[110]$ direction. These favored groups are those whose planes are almost perpendicular to the active slip plane, but the group whose poles cluster about the Burger's vector of the active slip system is not included. Regions consisting of a single group were of larger size the higher the amount of prior plastic strain.

Durlu and Christian⁵⁸ observed that prior plastic strain caused a decreased c/r (semithickness-to-radius) ratio for plates formed in a temperature range below M_b in an Fe-26.4Ni-.24 alloy. Datta and Raghavan⁵⁹ examined the plate dimensions of martensite formed at M_b in prestrained Fe-Ni alloys. They found that at any fixed transformation temperature, the c/r ratio linearly decreases as the amount of plastic deformation increases. Also, the variation of c/r with plastic strain is more pronounced at higher transformation temperatures.

Two types of martensite may form in austenitic Fe-Ni-C alloys during plastic straining, according to Maxwell, Goldberg, and Shyne⁶⁰. It is thought that the two martensites form by different mechanisms of transformation because of differences in morphology, distribution, and

temperature dependence. One type is the same as that which is formed spontaneously below M_s except that it is finer and less regularly shaped; it is called stress-assisted martensite. Strain-induced martensite formed on the $\{111\}_\gamma$ slip bands of the deformed austenite, and appeared as sheaves of parallel laths.

It has been illustrated that crystal defects and imperfections have a profound effect on martensite nucleation. Also, thermal stabilization is believed to occur because of the rearrangement of crystal defects and the pinning of martensite embryos. With this in mind, the objective of this research is to examine thermal stabilization in a appropriate steel, administering various amounts of deformation.

IV. Experimental Procedure

4.1. Material Composition and Preparation

The composition of the alloy chosen for this study is Fe-32.4%Ni-.08%C. Chemical analysis was done with spectrographic equipment; the carbon percentage was determined by wet chemistry.

The alloy was obtained in a cylindrical form, dimensions being 200 mm in length and 7 mm in diameter. To insure homogeneity, the material was first austenitized in an evacuated quartz tube. Thin rectangular pieces were needed; these were made by warm rolling in the austenitic condition with intermediate anneals. A manual shear cutter was used to obtain specimens of the following dimensions: 0.8 mm thick, 3 mm wide, and 45 mm long.

4.2. Austenitizing Conditions

After being sealed in evacuated quartz tubes, specimens were austenitized and then cooled to liquid nitrogen temperature; this was repeated twice with the intention of having a uniform microstructure, grain size, etc., before experimentation began. The austenitization was carried out at 800°C for 1 hour; these conditions were selected upon considerations of acceptable grain size and dissolution of carbides. Afterwards, specimens were air-cooled to room

temperature inside the quartz tubes. In a series of experiments, a single specimen was used repeatedly and subjected to this treatment so that consistent results could be obtained. Repeatedly austenitizing the specimen did not have a significant effect on the M_s temperature.

4.3. Method of Deformation

Deformation in the uniaxial tensile mode was given by mounting the austenitic specimens in a friction grip and applying a load with an Instron Tensile machine. A strain rate of $6.46 \times 10^{-3} \text{ sec}^{-1}$ was used, except for selected experiments where the effect of strain rates between 3.23×10^{-3} and $64.6 \times 10^{-3} \text{ sec}^{-1}$ were sought. The extent of deformation ranged from 0.5% to 4%; in a few experiments, 8% deformation was administered. It might be emphasized at this juncture that room temperature, at which the deformation was carried out, is above both M_d and M_s .

4.4. Determination of M_s and the Extent of Transformation

The progress of the martensitic transformation was studied by measuring the change in the specimen's electrical resistance using a Kelvin double bridge. In order to maintain an electrical resistance on the order of 10^{-2} to 10^{-3} ohms, the specimen size was manipulated to the approximate dimensions listed in Section 4.1. Because the M_s temperature of this alloy is below -65°C , a subzero temperature atmosphere which allows control of the specimen temperature is needed. For this reason, the specimen was

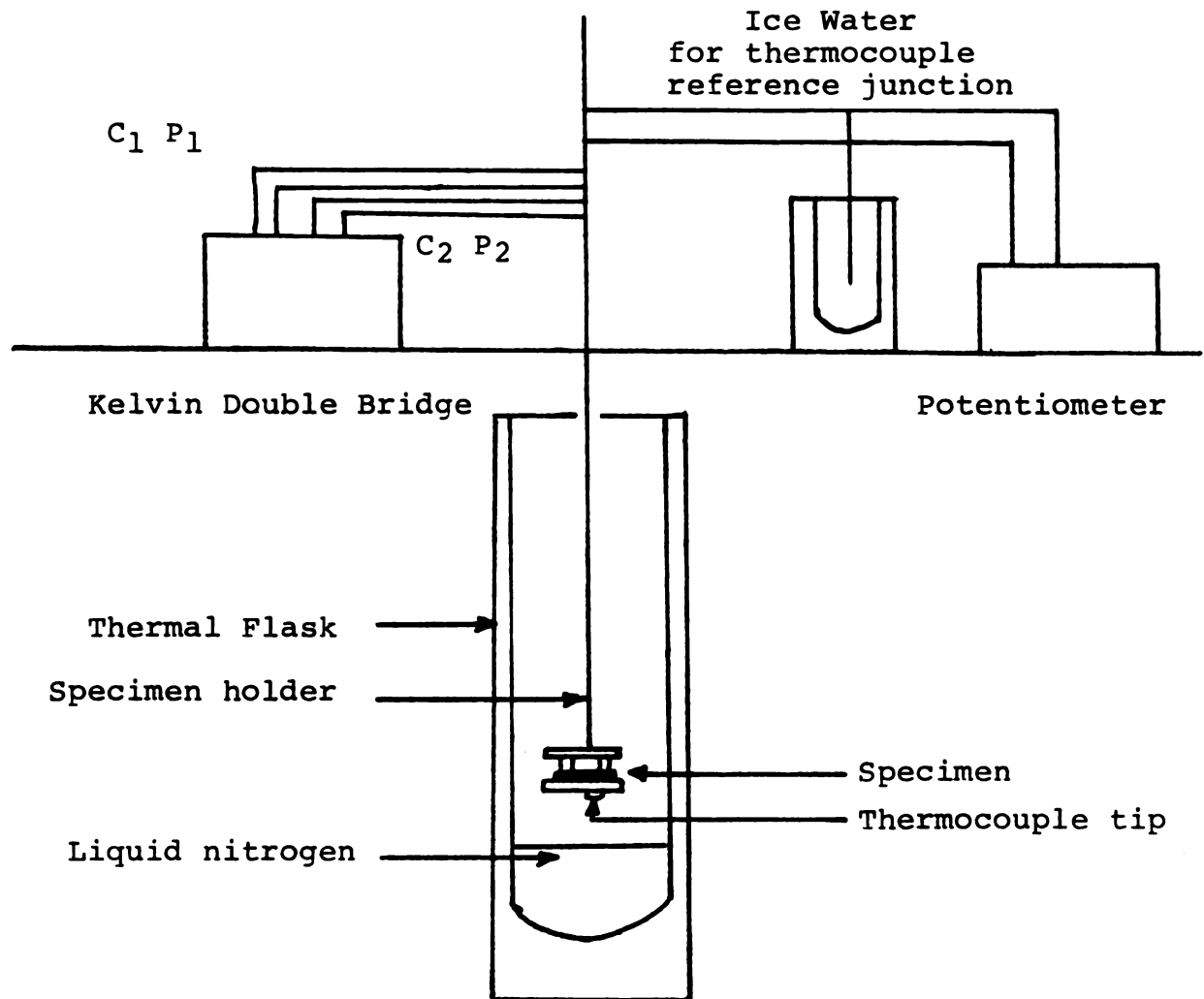


Figure 3. Experimental Apparatus

lowered manually to different levels above liquid nitrogen in a tall Dewar flask, as shown in Figure 3 at a cooling rate of about 1.5°C per minute.

With this arrangement, temperatures in the range -25°C to $+195^{\circ}\text{C}$ were easily obtained. The specimens were mounted in a holder having knife-edged contact points for the current and potential leads to the Kelvin double bridge. A Leeds-Northrup potentiometer connected to a copper/constantin thermocouple in contact with the specimen was used to monitor temperature. An empirical equation (see Appendix) was used in conjunction with the temperature and resistance data to calculate the extent of transformation.

4.5. Stabilization Treatment

After being austenitized and given fixed amounts of deformation, specimens were transformed until specified percentages of martensite were present. Then stabilization, or aging treatment, was administered in a constant temperature bath at 50°C for 1 hour and specimens were remounted in the specimen holder. Subsequent cooling and temperature and resistance measurements revealed the course of the transformation as it recommenced. In selected studies, the aging time was varied from 5 minutes to 2 hours. Schematics of the time/temperature relationships in the stabilization experiments are shown in Figures 4a-d.

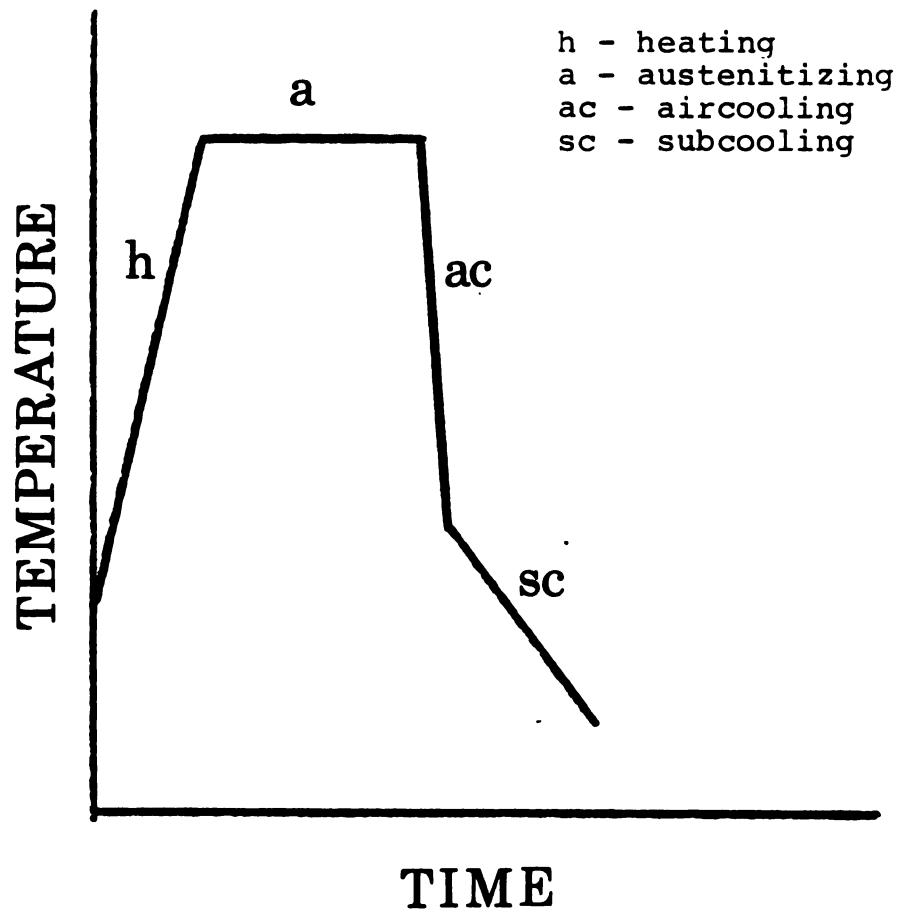


Figure 4a. Time/temperature sequence for stabilization treatment.

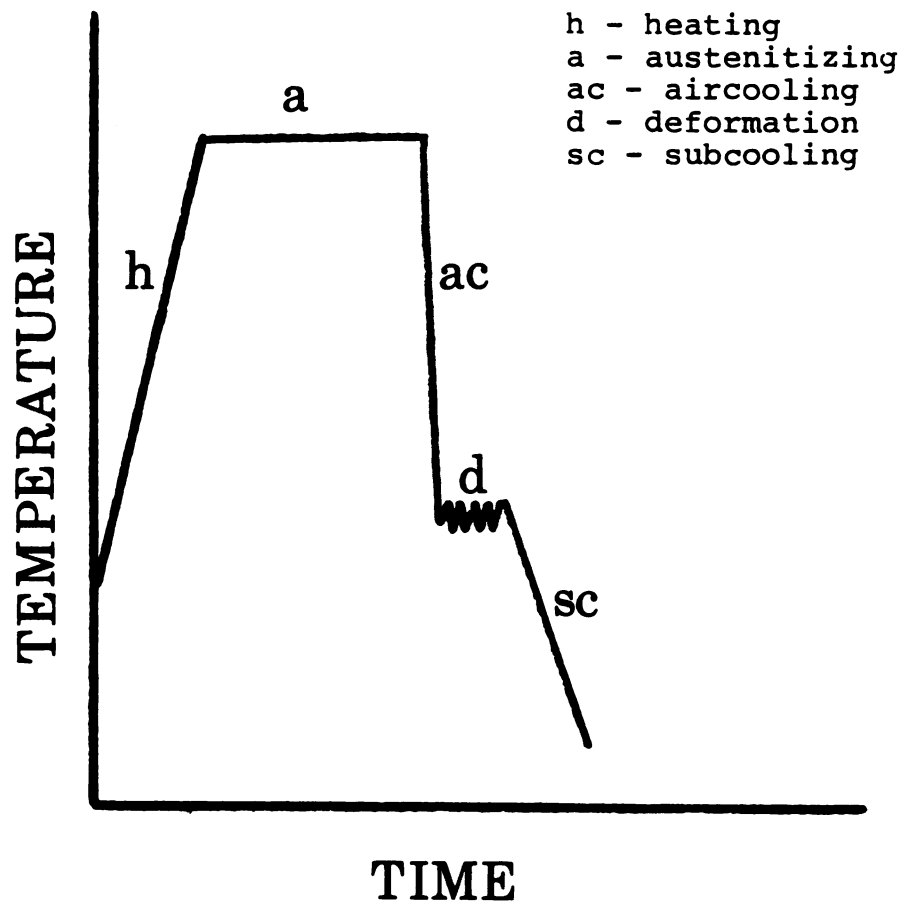


Figure 4b. Time/temperature sequence for stabilization treatment.

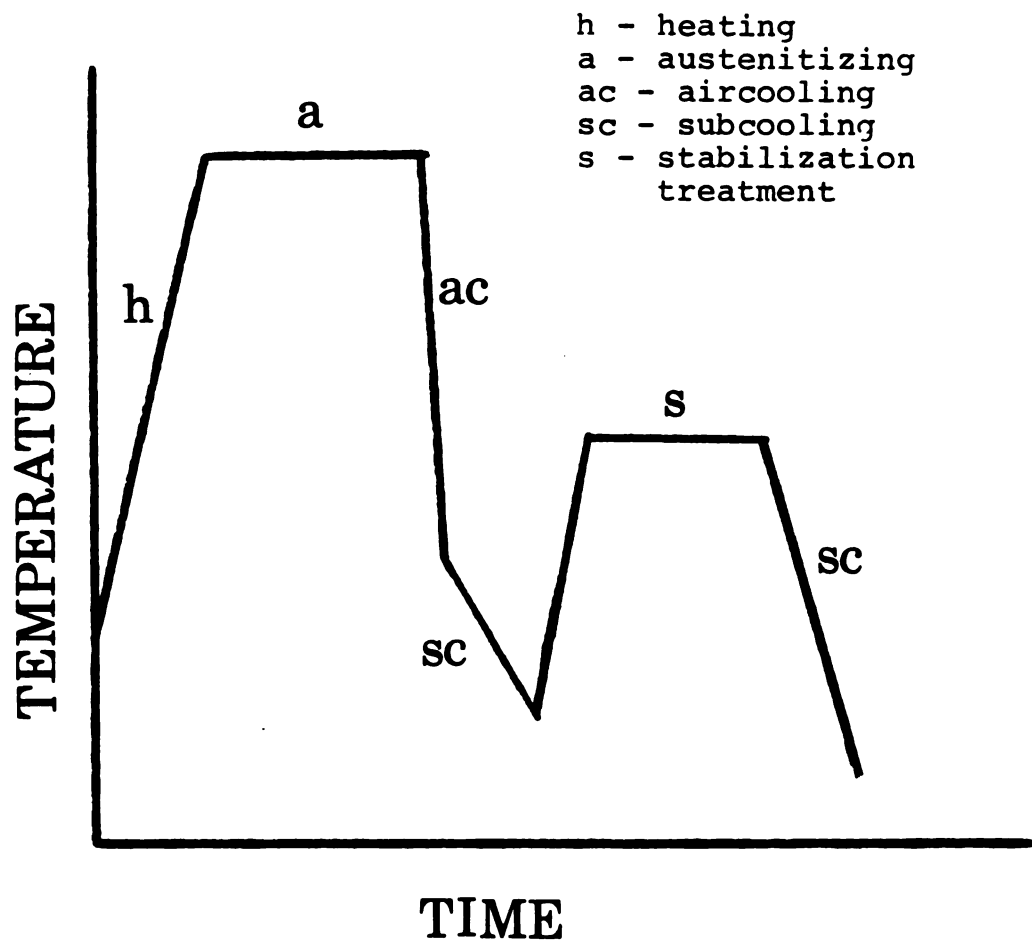


Figure 4c. Time/temperature sequence for stabilization treatment.

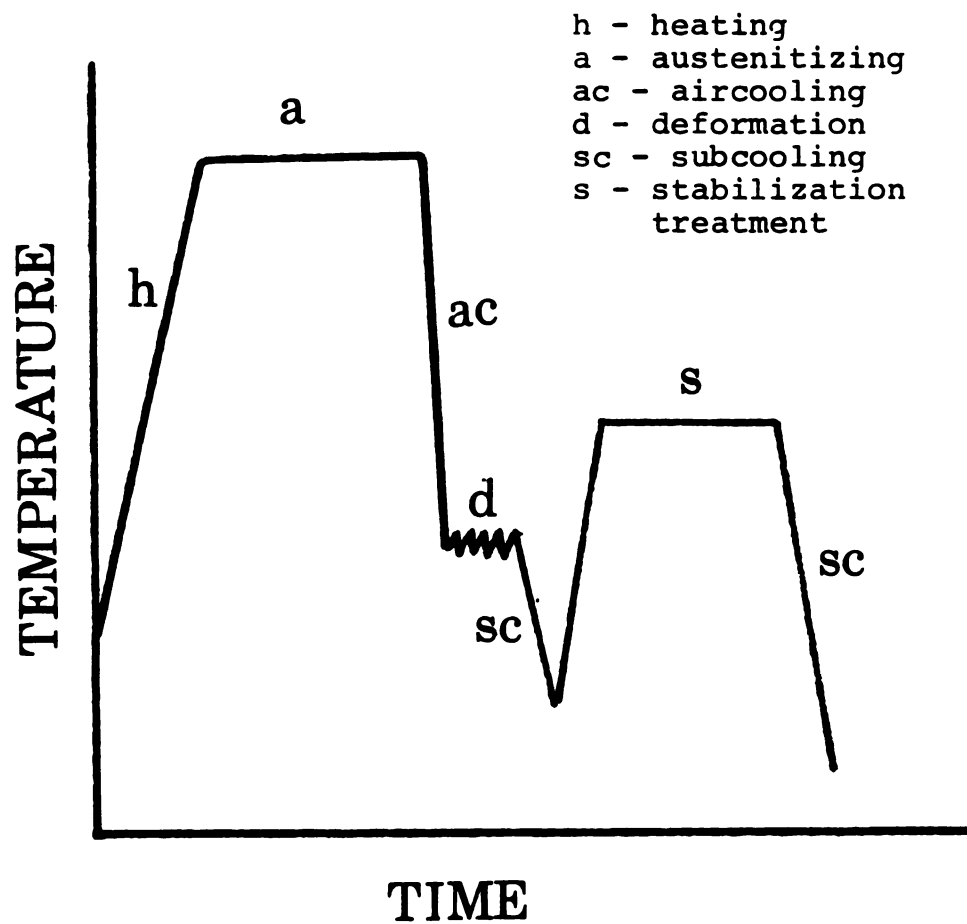


Figure 4d. Time/temperature sequence for stabilization treatment.

V. Results and Discussion

5.1. Effect of Austenitizing Temperature on Ms Point

Specimens of the alloy Fe-32.4 Ni-.08C were sealed in evacuated quartz tubes and austenitized for 1 hour at temperatures between 700°C and 900°C. They were mounted in the specimen holder in the undeformed state and subjected to subcooling. The time/temperature sequence used was that shown in Figure 4-a. Resistance and temperature measurements were used to determine Ms. It was found that higher austenitizing temperatures produced elevated Ms temperatures. This trend is shown in Figure 5. Sastri and West⁶¹ and Maki et al⁶² reported similar results. Others⁶³ have found the Ms increases as austenitizing temperature increases to a limiting temperature, and then in some cases decreases.

It is likely that as increased numbers of crystal defects are removed due to higher temperatures of treatment, the energy needed for complementary shear during transformation is reduced, thereby increasing the Ms. This was the inference drawn by Ankara⁵⁷ in his study of an Fe-30Ni alloy. He found that the effect of austenitizing temperatures on Ms was exaggerated by cooling immediately after rapid heating, retaining the highest possible amount of lattice defects.

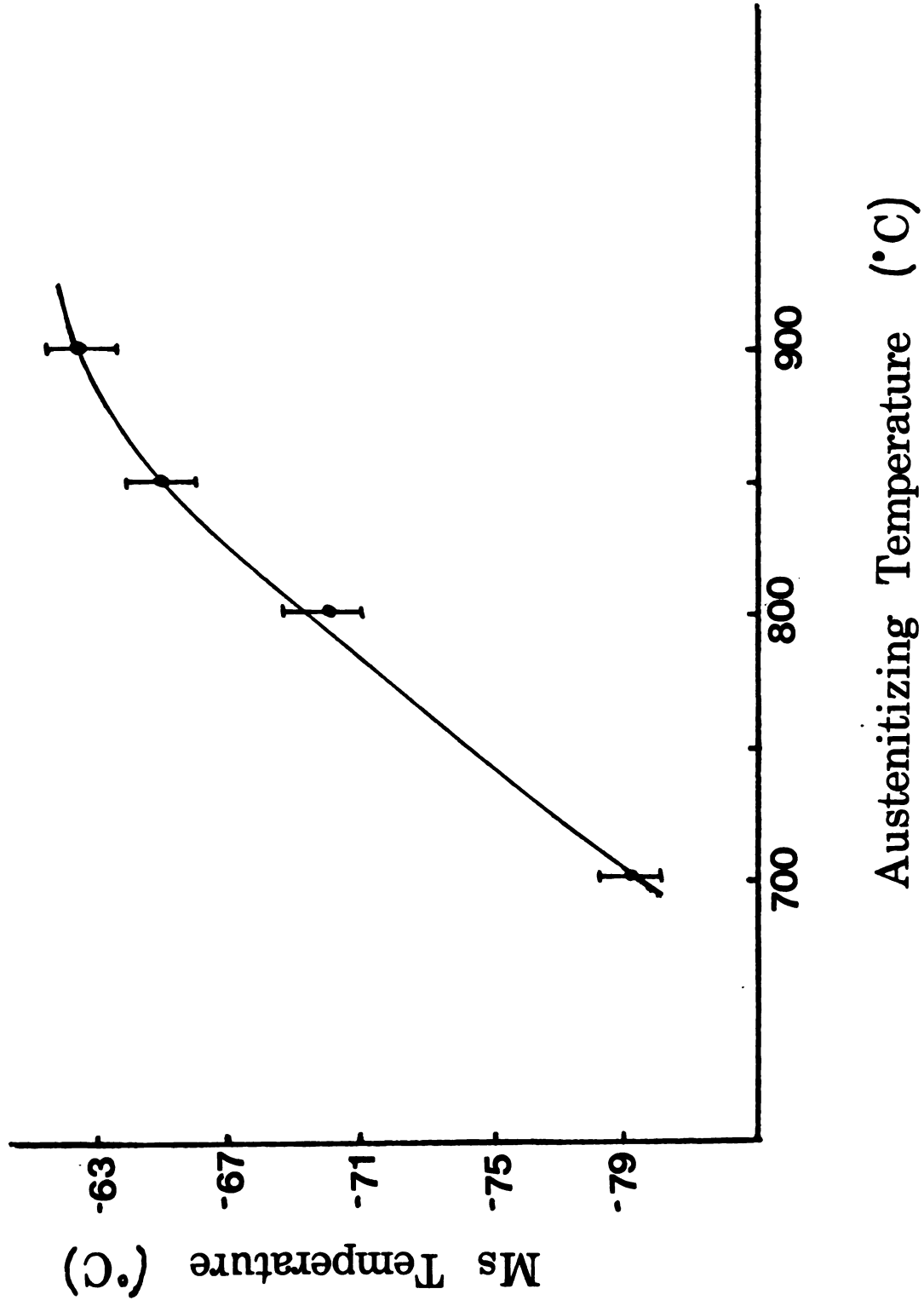


Figure 5. Effect of austenitizing temperature on Ms point.

The work of Entwisle and Feeney³⁰ in high Ni steels shows M_b rising with austenite grain size. One must not infer, they say, that the larger austenite grain size raises the M_b point; instead, that the increasing austenitizing temperature causes growth of austenite grains and increased M_b simultaneously.

A grain size relationship does seem to exist, however. A martensite crystal will stop growing when it reaches the grain boundary. Also, with smaller grain size, there is a greater buildup of backstress when plates form, making dislocation motion or shearing of the matrix difficult. In light of these facts, it is thought that small grain size, which will result from lower austenitizing temperatures, stabilizes the austenite (decreasing the M_s temperature).

5.2. Effect of Prior Deformation on M_s Point

Austenitized and air-cooled samples (austenitic) were deformed to various extents by tensile elongation. Amounts of deformation ranged from 0.5% to 8%. The samples were mounted in the specimen holder and subcooled, and M_s was determined. It was found that M_s was not substantially altered when 0% to 4% prior deformation existed; a depression of M_s was noted when 8% deformation was given, however. Average M_s values for differing amounts of deformation are shown in Figure 6.

Deformation is expected to have an effect on M_s , and these results may be explained by noting that opposing effects are in action. Existing martensite embryos may be

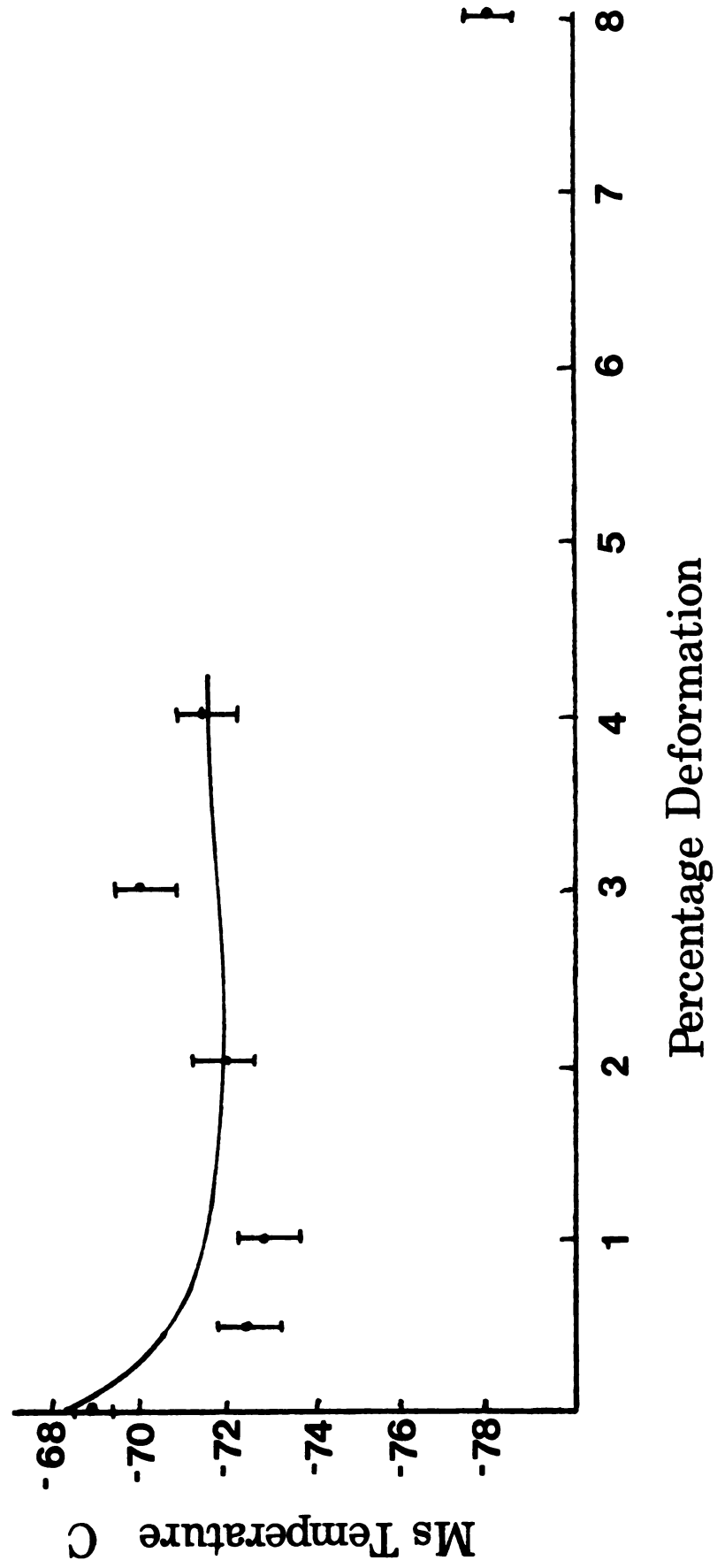


Figure 6. Effect of prior deformation on Ms point.

stimulated by internal stress from plastic deformation or from partial transformation, thus aiding transformation^{64,65}. This plastic deformation, on the other hand, strain-hardens the austenite matrix. Propagation of martensite plates is difficult due to the obstacles formed by dislocation accumulation. The M_s temperature is expected to decrease when this occurs.

It is our belief that the opposing effects of embryo stimulation and austenite strain hardening cancel each other (negate each other) when small amounts (0 to 4%) of deformation are administered, thus resulting in an M_s that is neither raised nor lowered. Austenite strain hardening effect predominated at higher deformation (8%), thereby depressing M_s .

5.3. Effect of Deformation on $T_{55\%}$

A series of experiments were done in which 55% martensite was needed prior to stabilization treatment. Austenitic specimens were given between 0% and 4% deformation and subcooled. It was found that as the amount of deformation increased, the temperature at which 55% martensite had formed (denoted as $T_{55\%}$) also increased. The relationship between deformation and average $T_{55\%}$ is illustrated in Figure 7. This data was compiled from experiments whose time/temperature sequences followed those of Figures 4a-d. A typical plot of resistance vs. temperature is shown in Figure 8. This diagram shows that

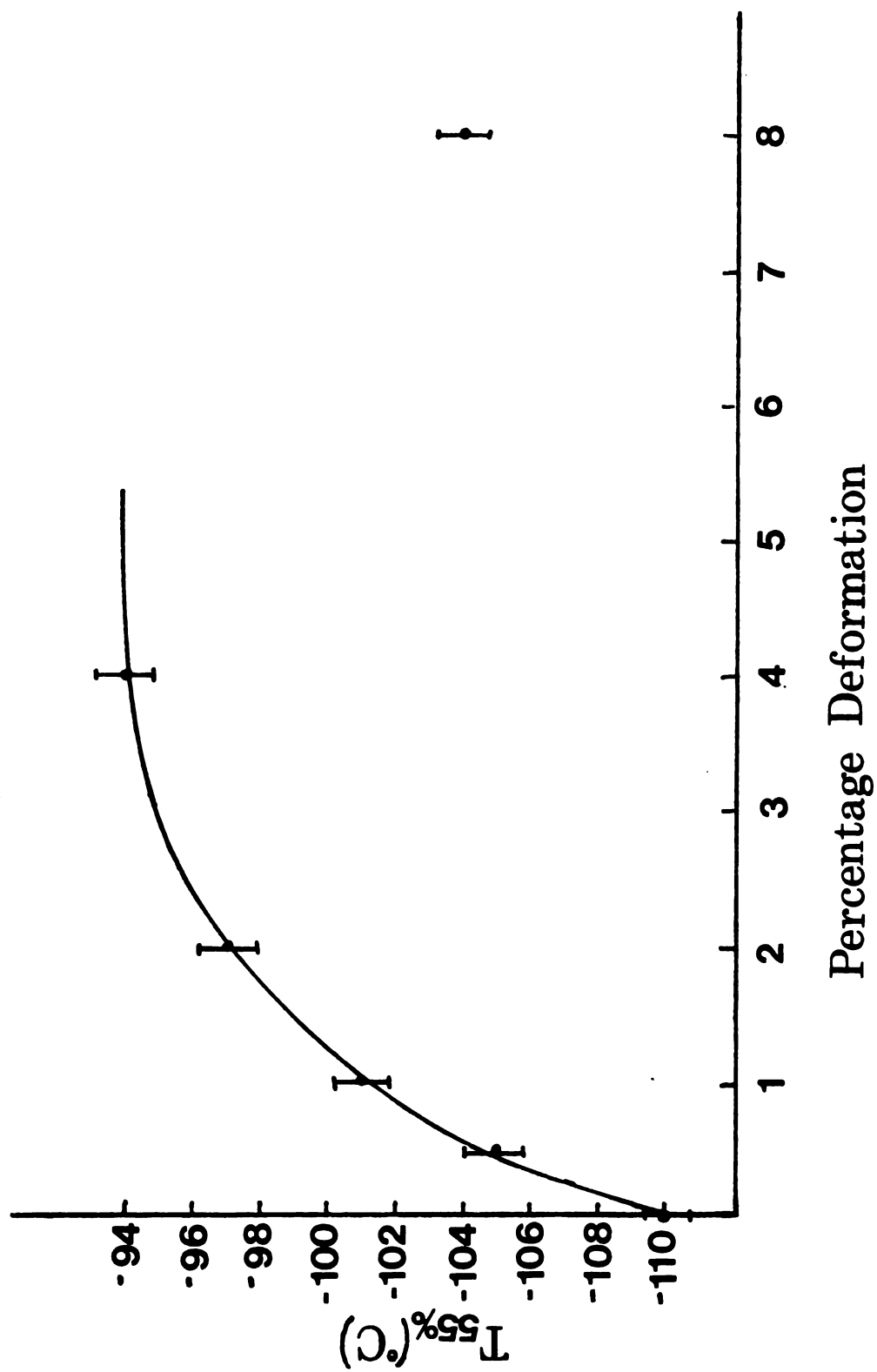


Figure 7. The effect of prior deformation on $T_{55\%}$.

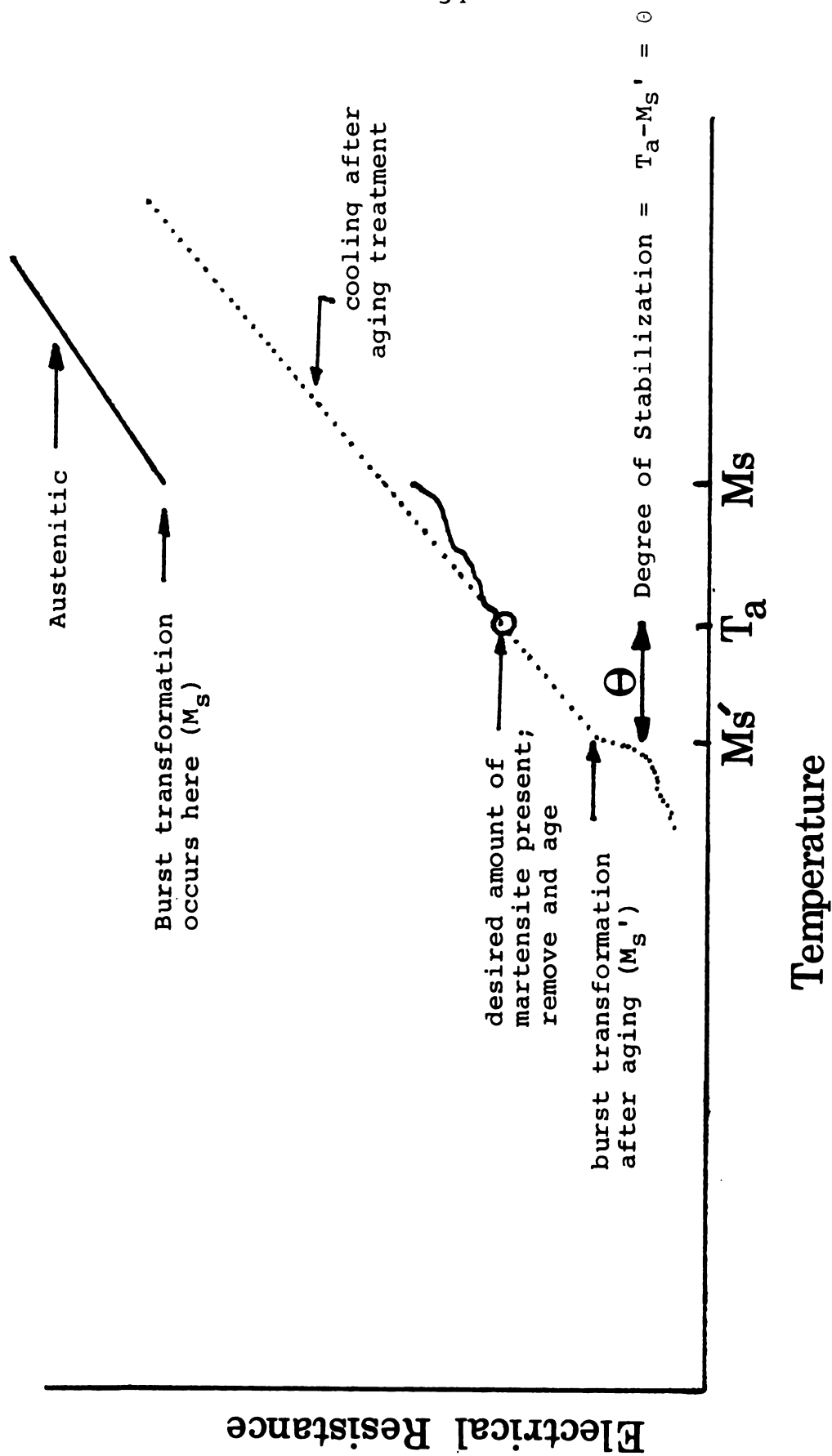


Figure 8. Electrical resistance vs. temperature plot.

martensite formation commences in this alloy with a large burst, and manifests itself by a large decrease in electrical resistance. Burst formation was accompanied by an audible click and was of the order of 15% to 50% martensite. Amounts of prior deformation did not have a marked effect on burst size.

The formation of martensite may be assisted by plastic deformation. Existing embryos may be stimulated by the resulting internal or external stress^{64,66}. It is suggested that the deformation administered did not have a significant effect on M_s temperatures (except at 8% def.) because the deformation did not alter the potency of the largest embryos which would be the first to become martensite plates. Possibly the smaller, less potent embryos were stimulated such that they would transform at higher temperatures. The statistical distribution of embryo size and potencies, then, could have been condensed and the lower end of the potency range shifted upwards with plastic deformation. This would result in an increased amount of transformation (at temperatures somewhat lower than M_s) resulting from increased deformation. Thus, the temperature at which 55% martensite is formed ($T_{55\%}$) will increase with plastic deformation.

5.4. Effect of Deformation on Stabilization

Specimens were austenitized, deformed to various extents at a rate of 0.02 cm sec^{-1} and subcooled. When prescribed amounts of 45% or 55% martensite were obtained, specimens

were given stabilization (aging) treatment at 50°C for 1 hour and again subcooled. The time/temperature sequences used are shown in Figures 4c,d. The variation in the extent of stabilization as a function of percentage prior deformation is shown in Figure 9, where 55% martensite was present prior to aging. Without deformation, the stabilization was minimum; it increased with deformation until 1% deformation was given, and above this it remained relatively constant.

Attempts to study the effect of varying amounts of deformation and 45% martensite prior to aging on the stabilization phenomenon were not successful. The temperatures at which specimens achieved 45% martensite ($T_{45\%}$) and the difference in temperature between T_{Ms} and $T_{45\%}$ varied widely. This presents inconsistencies in experimental conditions.

It was found that as deformation was increased, θ increased and then remained constant above 1% deformation. It is suggested that higher deformation gives more opportunity for C to diffuse, and therefore increased stabilization may occur due to C destroying the embryo potency. It is thought that the maximum effect of this mechanism has occurred by 1% deformation.

5.5. Effect of Strain Rate on Stabilization

Austenitized and air-quenched specimens were deformed at room temperature to 2% elongation with various strain rates. The specimens were then subcooled until approximately 50% martensite was formed whereupon they were removed, aged at

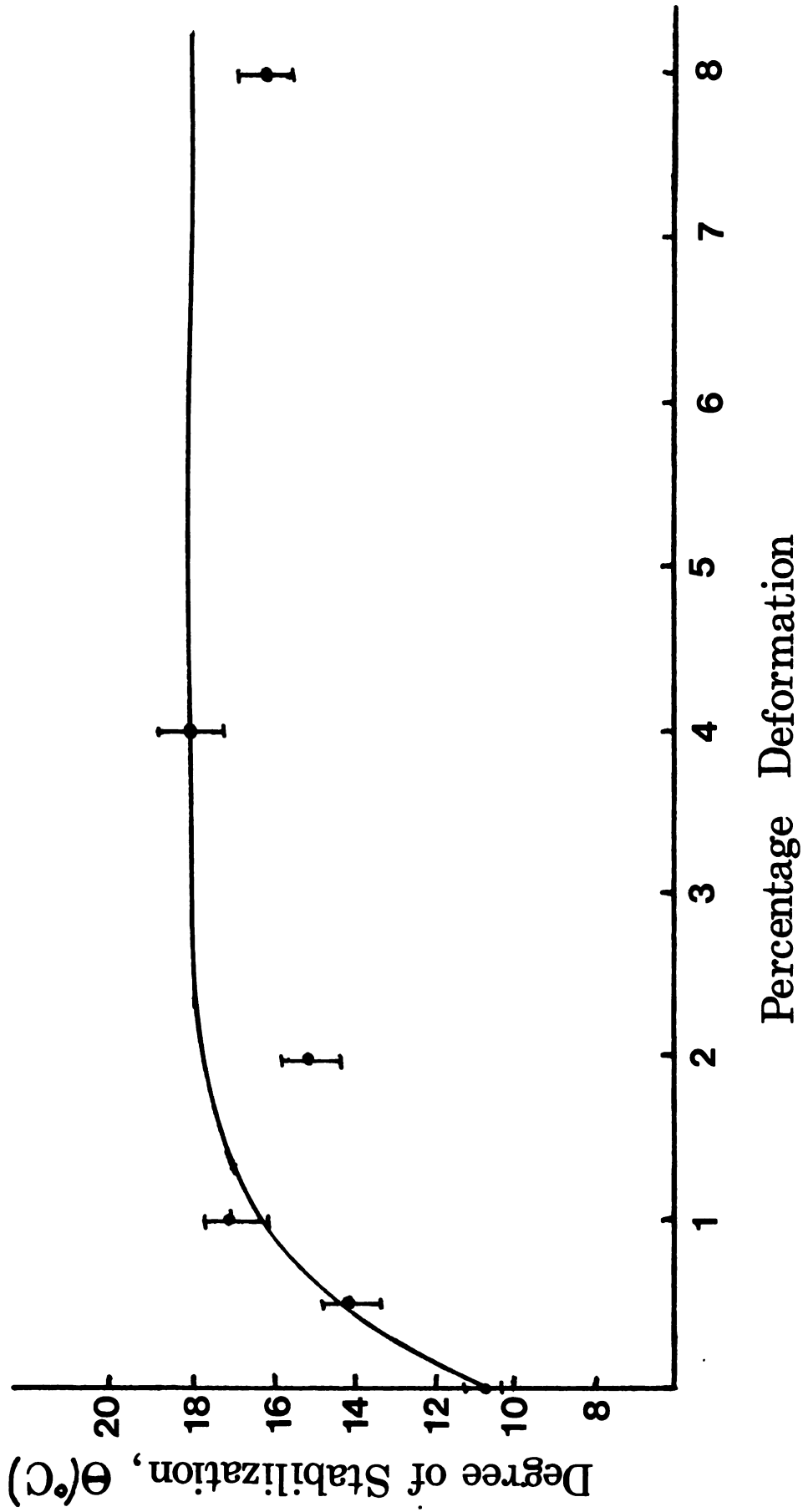


Figure 9. Degree of stabilization as a function of amount of prior deformation.

50°C for 1 hour, and cooled again. The effect of different strain rates on stabilization has been presented in Figure 10. The time/temperature sequence followed for these experiments is illustrated in Figure 4-d.

An increased degree of stabilization with decreased strain rate was observed in the alloy Fe-32.4Ni-.08C. This is in line with the proposed mechanism of C pinning the martensite embryos. The potency of a martensite embryo is destroyed when carbon diffuses to the site, and it is known that C diffusion is a time and temperature dependent function. With a slower rate of deformation, it will take a longer time interval to reach a given amount of deformation, allowing more time for the carbon to diffuse to the embryos and destroy their potency. Destroyed potency would increase the stabilization and the results show this.

5.6. Effect of Aging Time on Stabilization

Austenitized specimens were deformed to 2% elongation at a strain rate of $6.46 \times 10^{-3} \text{ sec}^{-1}$, and subcooled until approximately 50% martensite was present. Aging treatment at a constant 50% was administered for various durations of time. The treatment is shown in Figure 4-d. Data was analyzed so that the difficulties presented by differing $T_{50\%}$ and $T_{Ms}-T_{50\%}$ values would be eliminated. The data showed that as the time of aging was increased, stabilization decreased. While this is contrary to what is expected, the trend was consistent. Results are reported in Table 1.

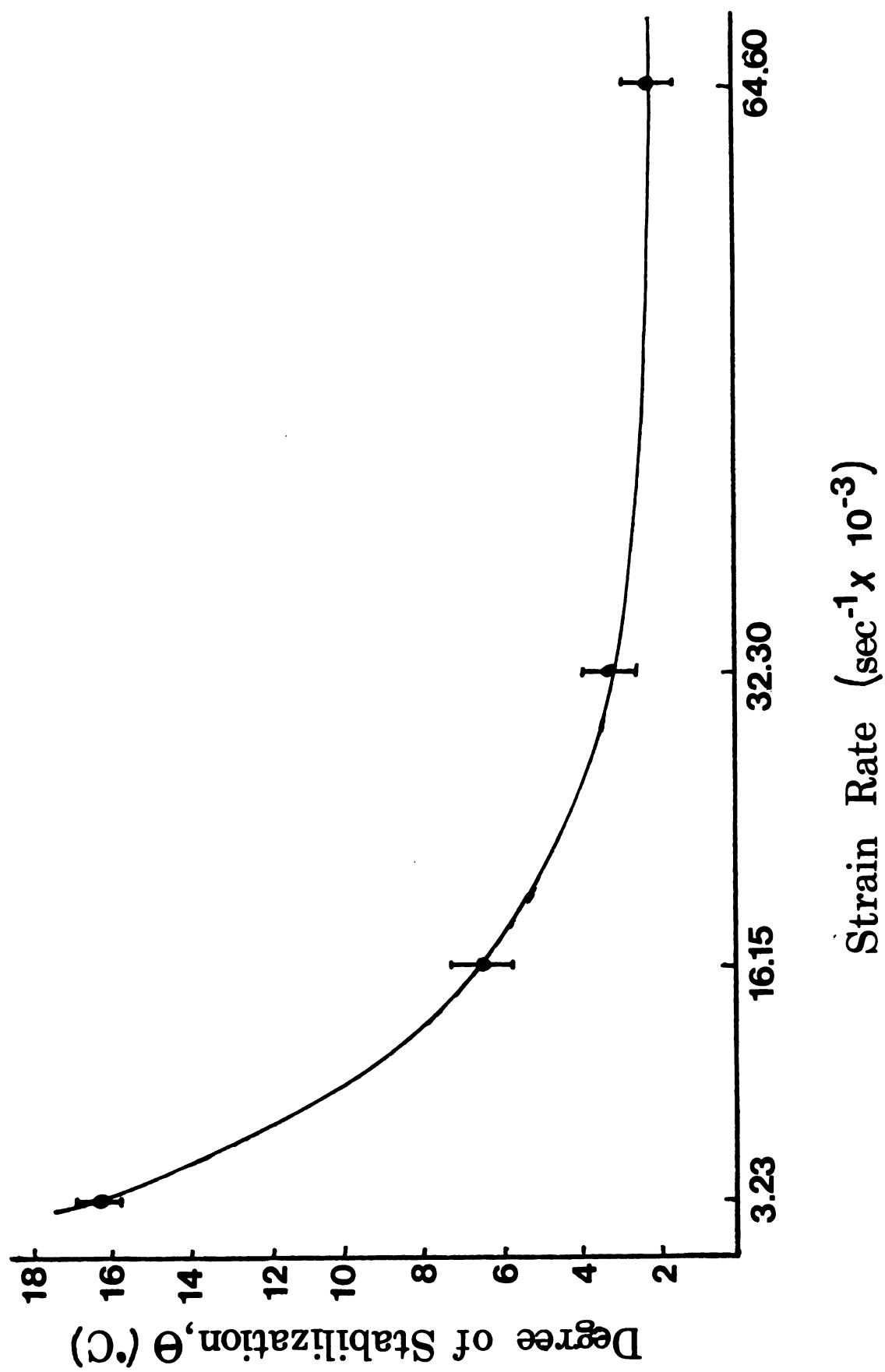


Figure 10. The degree of stabilization as a function of strain rate.

Table 1. Degree of stabilization as a function of time and energy.

% martensite	T _a arrest temperature	Ms'	aging time (min.)	θ degree of stabilization (°C)
49.2	-83.2	-98.1	5	14.9
50.5	-84.5	-92.2	120	7.7
53.1	-103.3	-112.9	20	9.6
49.0	-104.4	-1.07.7	60	3.3

It is desirable to analyze and compare data in which Ms-T_a and T_a values are relatively constant. In order to facilitate this, the first two data are analyzed separately from the second two data. While a complete set of acceptable data including several different aging times is not available, the two sets of data suggest a decrease in the degree of stabilization with increased aging time.

As mentioned, stabilization typically increases with aging time. However, there are several cases in the literature in which stabilization increases, reaches a maximum, and then decreases as a function of aging time. If this is the trend which the data (in Table 1) follows, then it is possible that the shortest aging period used represented the portion of the aging period vs. stabilization curve in which stabilization was decreasing. Had it been feasible to study the effect of shorter aging periods, stabilization might have been found to first increase and reach a maximum before decreasing.

Okamoto and Odaka⁶⁷ studied the effect of aging time on stabilization in an Fe-1.63Cr-1.06C steel. In specimens aged at 100°C and at 200°C, stabilization increased, reached a maximum, and then decreased. According to Nishiyama⁶⁸, sufficiently high aging temperatures allowed the interstitial solute atoms to cluster, forming precipitates after their segregation to nucleation sites. As a result of the clustering, the interstitial content of the austenite phase is lowered, consequently increasing M_s . According to Nishiyama, this is why the degree of stabilization decreases after reaching a maximum value. It might be noted that Glover reported similar results for a 1.4%C steel.

The results of Priestner and Glover⁴¹ are striking. Studying an Fe-5Ni-1.43C steel, they found that aging above 50°C caused stabilization (θ) to increase with time, reach a maximum, then decrease, even making θ negative, before

increasing again. They attribute the negative θ to softening caused by overaging. A strain-aging model was used by these authors to account for their overall results.

Kinsman and Shyne³⁸ found that with relatively low aging temperatures (0°C to 80°C), three Fe-Ni-C alloys also exhibited a decreasing θ after reaching a maximum. They observed that the kinetics of thermal stabilization are similar to those of precipitation hardening. Because in some cases stabilization increases to a maximum before decreasing, they feel that this behavior is analogous to overaging in solid state precipitation. According to them, the fact that stabilization occurs by the pinning of the martensite/austenite interface means that the degree of stabilization must be a function of the excess concentration of segregated solute atoms at the interface. The time/temperature dependence of interface carbon segregation was determined, and it was found that the interface carbon concentration increases to a maximum value and then decreases at longer times, as does stabilization in many cases. This would seem to support their contention.

It has not been possible, however, to conclusively determine the cause of the increasing and then decreasing stabilization in this research. As mentioned, data analysis was quite difficult. Determining the cause of the observed behavior would entail many further experiments and possibly the use of more sophisticated equipment.

VI. Conclusions

1. Martensitic transformation begins in the Fe-32.4Ni-.08C alloy with a burst producing between 15% and 50% martensite.
2. As the austenitizing temperature is increased from 700°C to 900°C, the Ms temperature increases.
3. Plastic deformation prior to transformation of austenite has no effect on Ms until high amounts of deformation are given.
4. In deformed specimens, after forming 55% martensite, the degree of stabilization increases with deformation up to 1% deformation and then remains relatively constant at higher deformations.
5. For specimens deformed 2%, transformed to 55% martensite and aged, the slower the rate of strain, the higher the degree of stabilization.
6. Specimens deformed 2% and transformed to 50% martensite had a higher degree of stabilization if the aging period was shorter.

APPENDIX

APPENDIX

Using resistance and temperature data, the amount of martensite present at any point may be calculated. The following procedure is used:

- 1) An austenitic specimen is cooled from room temperature to liquid nitrogen temperature (-195°C). Many simultaneous readings of resistance and temperature are taken. After maximum transformation has occurred (at -195°C), the specimen is heated and the change of resistance with respect to temperature is noted.

With this information, the following parameters are calculated:

Average resistance drop per 1°C in austenite = R_{γ}

Average resistance drop per 1°C in martensite = R_{α}

Resistance drop per 1% martensite formation = R_M

Using x-ray diffraction, the maximum amount of martensite was found to be 80%. Thus, R_M could be calculated in the following way:

$$R_M = \frac{R_{Ms} - R_{195} - R_{\alpha}(Ms+195)}{80}$$

In this case, R_{Ms} and R_{195} are the resistances at M_s and at -195°C , respectively. To eliminate the size effect between specimens, all of the above parameters

were divided by R_{MS} and the ratios R_γ/R_{MS} , R_α/R_{MS} and R_M/R_{MS} were calculated. These ratios were found to be relatively constant from specimen to specimen despite size differences.

2) Having obtained the necessary parameters, the calculations performed in stabilization experiments are:

- i) An austenitic specimen is cooled to the M_s temperature, and the average value of R_γ is calculated.
- ii) The resistance measured immediately prior to transformation is denoted as R_B and the corresponding temperature, T_B . The first data measured after transformation commences consists of a resistance R_{A1} and temperature T_{A1} . Resistance at M_s (R_{MS}) may be calculated: $R_{MS} = R_B - R_\gamma(T_B - M_s)$.
- iii) The percentage of martensite at T_{A1} is then determined:

$$\% \text{ martensite} : \frac{1 - R_{A1}/R_{MS} - R_\gamma/R_{MS}(M_s - T_{A1})}{R_M/R_{MS}} = X_1$$

- iv) The amount of transformation at a lower temperature (T_{A2}) may be determined using this formula: % martensite at $T_{A2} = X_2 =$

$$\frac{X_1 + R_{A1}/R_{MS} - R_{A2}/R_{MS} - \{R_\gamma/R_{MS}(\frac{X_1}{100}) + R_\alpha/R_{MS}(1 - \frac{X_1}{100})\} [T_{A2} - T_{A1}]}{R_M/R_{MS}}$$

- v) In this manner, the amount of martensite present with successive temperature decreases may be calculated.

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