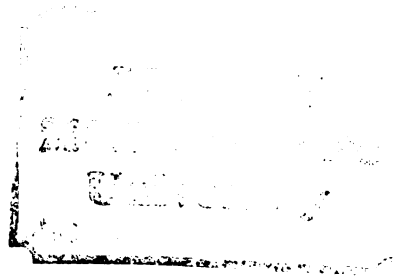


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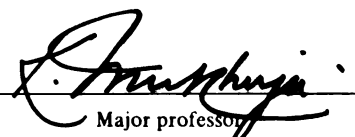


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THE EFFECT OF PLASTIC DEFORMATION ON THE
THERMAL STABILIZATION OF AUSTENITE-MARTENSITE
TRANSFORMATION

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THE EFFECT OF PLASTIC DEFORMATION ON THE
THERMAL STABILIZATION OF AUSTENITE-MARTENSITE TRANSFORMATION

By

Gie Hong Kang

A THESIS

Submitted to
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ABSTRACT

THE EFFECT OF PLASTIC DEFORMATION ON THE THERMAL STABILIZATION OF AUSTENITE-MARTENSITE TRANSFORMATION

By

Gie Hong Kang

The influence of plastic deformation on the thermal stabilization of austenite-martensite transformation has been studied in a 16Ni 1C steel. The studies include the effect of i) Extent of deformation prior to martensite formation ii) Rate of straining employed for deforming the specimens iii) Amount of martensite before stabilization and iv) straining subsequent to martensite formation.

Deformation prior to martensite formation decreases the M_s -temperature significantly. This decrease is very sensitive to the strain rate employed. The temperature interval over which the transformation is inhibited, following stabilizing treatment after partial martensite formation, decreases with increasing prior deformation. However, this trend varies with the percent transformation preceeding the stabilization treatment. In specimens strained after partial transformation, stabilization increases with increasing deformation. The transformation both before and after stabilization starts in general with a burst; and the size of a burst increases with deformation. The course of transformation after stabilization is not much different in deformed specimens and the microstructure indicates that martensitic plates formed in the deformed austenite are finer. The observed results appear to be dominated by strainaging effect.

ACKNOWLEDGEMENTS

The author is much indebted to Visiting Professor Dr. S.C. Das Gupta and Professor Dr. K. Mukherjee for their constant interest in this work, and for many ideas, comment and enlightening discussions. The author also wishes to express his deepest appreciation to his family.

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I. INTRODUCTION

Thermal stabilization of austenite-martensite reaction is an inhibition of the martensite formation resulting from an aging treatment. The course of formation of martensite in a steel during cooling may be influenced by the cooling procedure adopted. In general, if the cooling is arrested within the martensite transformation range and then allowed to age for a period of time, the transformation does not start on subsequent cooling, until some lower temperature is reached. There is a likelihood that at subsequent temperatures, the total amount of martensite is somewhat less than that produced by direct cooling to the temperature concerned. This phenomenon is referred to as stabilization. Figure 1 shows a schematic representation of the process.

The difference between the temperature of arrest and the restarting of the transformation, following aging treatment as represented in the diagram, is often taken as a measure of degree of stabilization.

The stabilization phenomenon will have influence on the amount of retained austenite in the martensitic steels. A cooling rate dependency exists in alloy steels quenched in water and in oil. The thermal stabilization of austenite in steels is thus of importance because of technological significance in heat-treatment and scientific implications relative

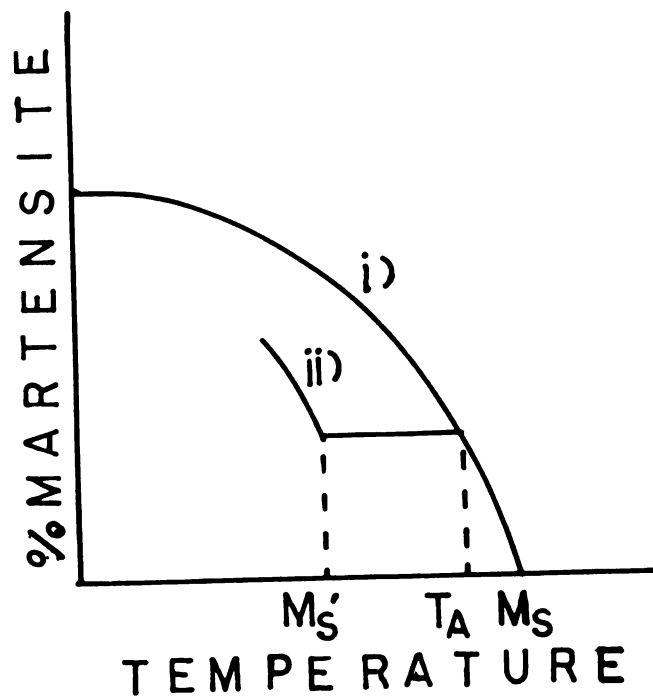
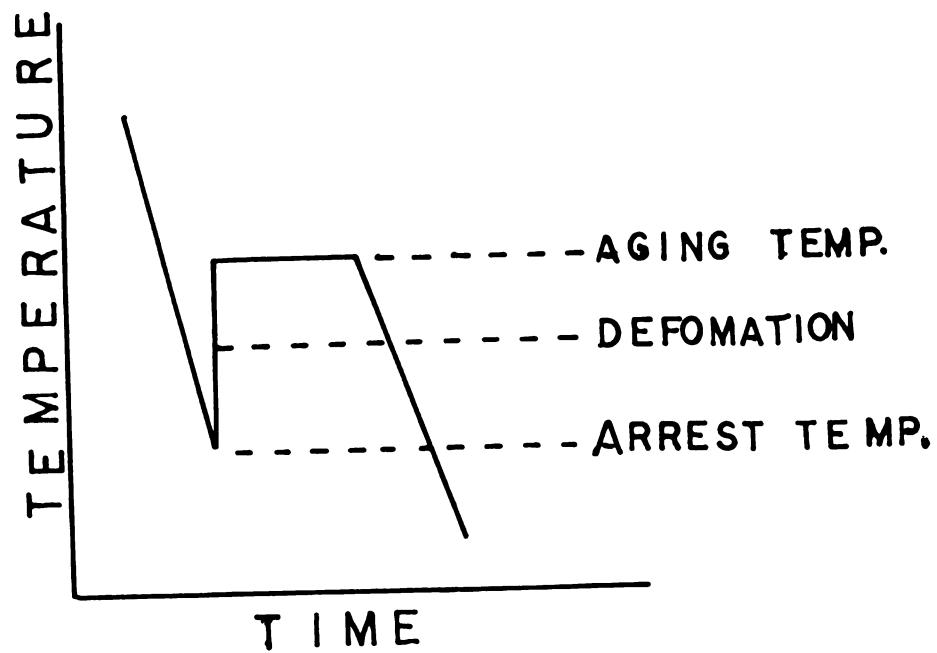


Figure 1. a) Schematic Plot of Temp. vs. Time Depicting the Stabilizing Treatment
 b) Course of Martensite Formation with Decreasing Temp. below M_S i) without interruption
 ii) with interruption

to the martensite transformation. Presence of retained austenite in a hardened steel is unwanted for various reasons. It may result in a softer material and may induce structural as well as dimensional instability.

2. THEORETICAL ASPECTS

2-1 Martensite Transformation

Phase transformations in solid state may be divided into two categories essentially:

- a) Nucleation and growth type (Diffusion controlled)
- b) Displacive or shear type (Diffusionless)

a) Nucleation and growth type transformation

In this type of transformations, a new phase grows at the expense of parent phase by relatively slow movement of atoms over multiple of atomic distances. Movements are independent and markedly vary with temperature. Reaction proceeds isothermally and amount of phase increases with time.

b) Displacive or shear type transformation

In this type of transformations, cooperative movements of many atoms are utilized instead of independent movement of individual atoms. Rearrangement takes place in an orderly displacive manner. Atomic movements are of minor order and in principle atoms do not change position with neighbors.

Martensite may form in a number of alloys of which the transformation in steel is most important and has been studied extensively.

2-2 Principal Characteristics of Martensitic Transformation

There are morphological, thermodynamic and kinetic features which can be considered as typical for martensitic

transformations.

(1) A plate-like morphology with a small ratio of thickness to other linear dimensions is the typical structural element resulting from martensitic transformations.

(2) The habit plane has a definite orientation to the crystalline axes of the initial and final phases.

(3) There is an orientational relationship between the lattice of the initial and final phases.

(4) The transformation is accompanied by a change in the form of the transformed region which manifests itself in a characteristic relief on the plane surface of the specimen at the site where a new phase plate appears. This macrodeformation is uniform and represents a combination of a simple shear along the plane of the plate and expansion or compression normal to this plane. The macrodeformation parameters are constant for each transformation.

(5) The martensitic crystals have a regular internal structure. Often, the martensitic plate is internally twinned, faulted or dislocated.

(6) The growth rate of the martensitic crystals is high (10^4 - 10^5 cm S⁻¹) and does not display any noticeable temperature dependence.

(7) According to the operative nucleation kinetics, martensitic transformations are described as athermal or

isothermal. In the case of athermal transformation the nucleation rate is high and does not display any temperature dependence. In the case of isothermal transformation the rate of nucleation depends essentially on temperature.

(8) Isothermal and athermal transformations can occur in the same material.

(9) The amount of transformation is characteristic of temperature, provided other variables, such as grain size, are held constant. Transformation on cooling begins spontaneously at a fixed temperature (M_s), and as the temperature is changed, more and more material transformations, until the temperature (M_f) is reached, at which the change is complete.

(10) Martensitic reactions are reversible in the sense that an initial atomic configuration can be repeatedly obtained. A single crystal of the original phase may transform on cooling into several crystals of the new phase. The reverse change on heating will usually result in a single crystal of the same size, shape, and orientation as the original crystal. The reversibility is associated with a temperature hysteresis and the reverse reaction begins at a temperature above M_s (except in some thermoelastic cases). Moreover, in repeated transformations, the plates which form on cooling have the same size and shape, and appear in the same regions of the original crystal. This behavior probably applies in principle to all martensitic reactions. In Fe-C alloys, this, however,

is not normally observable since the process is interfered by the tempering phenomenon. By employing extremely rapid rate of heating, tempering phenomenon could be arrested and reversibility followed in Fe-C alloys.

(11) Plastic deformation is more important in martensitic transformations than in nucleation and growth changes. Application of plastic stress at any temperature in the transformation range usually increases the amount of transformation, and the reaction can often be completed by this means. In some transformations, elastic stresses have a similar effect. When single crystals are used, the direction of the applied stress is important, and some reactions may be inhibited as well as aided by a suitably orientated stress.

Deformation above M_s may also result in the formation of the product phase, even though the temperature is too high for spontaneous reaction. The highest temperature at which martensite may be formed under stress is called M_d . In general, the reverse reaction can be aided in the same way, and a suitable stress will induce transformation below the temperature at which it begins spontaneously.

If the original phase is cold-worked in a temperature range where it is stable, e.g., at temperatures sufficiently above M_s , the resultant deformation may either raise or lower the transformation start temperature depending upon the

degree of cold work. When the degree of cold work is relatively small M_s may be raised and with relatively larger deformation it may be depressed.

(12) The transformation is also inhibited in another way. If the specimen is cooled to a temperature in the transformation range, held there for a period of time, and then cooled again, transformation does not begin again immediately. Supercooling is then necessary to form any further martensite. At all subsequent temperatures the amount of transformation is less than that produced by direct cooling to the temperature concerned. This phenomenon may be referred to as permanent stabilization. The degree of stabilization increases with the time for which the specimen is held at the temperature. Slight variations in amount of transformation with cooling velocity are also presumably to be attributed to stabilization. There is no general agreement whether or not stabilization is produced by halting the cooling above the M_s temperature.

Isothermal Martensite Formation--In many ferrous systems, such as Fe-Ni, martensitic transformation can occur both athermally and isothermally. The isothermal transformation kinetics show a typical C-curve behavior indicative of a thermally activated reaction. However, the increase in

volume percent of martensite at any isothermal temperature is still due to the formation of new plates rather than the growth of the existing plates. Further, experimental results indicate^{1,2} that time of formation of individual isothermal plates is also $\sim 10^{-7}$ sec. This process, therefore, is related to thermally activated triggering of embryos, and once triggered the growth no longer requires any thermal activation.

During isothermal transformation, it is observed that the initial rate of transformation is greater. This higher initial rate is related to "autocatalysis" where formation of plates stimulates nucleation of other plates. The actual mechanism of this stimulation is not well understood. The subsequent decrease in transformation rate is related to partitioning of the austenite phase, i.e., volume fraction transformed per nucleation event is reduced since these nucleation events occur in progressively smaller volume of austenite. The transformation rate equation, therefore, must take into account both autocatalysis and partitioning effects. If f is the fraction of martensite formed, then the following type of semiempirical rate equation may be used.³

$$-\frac{df}{dt} = (n_1 + pf - N_u) (1-f) \nu \exp(-\Delta W_a/RT) m q (1-f)^{1+\frac{1}{m}} \dots (1)$$

where: f = fraction transformed

t = time

N_1 = number of pre-existing nucleation sites (estimated $10^7/\text{cc}$)

p = number of autocatalytic embryos per unit volume

N_u = number of martensitic plates per unit volume

ν = 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

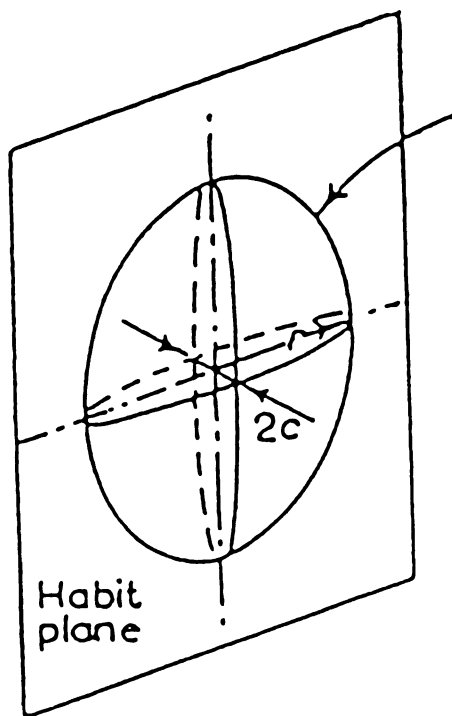
Equation (1) can be integrated numerically by substituting metallographically determined values of q and m and unknown quantities p and ΔW_a are determined by curve fitting. Experimental results agree well for up to ~10% transformation.³ Further manipulation of this equation is possible.³

2-3 The Nucleation of Martensite*

It has been emphasized earlier that kinetics of martensitic transformation is nucleation dominated. In order to consider nucleation and kinetics of this transformation, let us first consider a simple model of a martensite plate. We shall take an oblate spheroid shape⁴ as shown in Figure 2.

The radius of the oblate spheroid is r and the semi-thickness is c , where $r \gg c$. The interfacial free energy for

*The review section of nucleation is largely due to: K. Mukherjee, International Summer Course on Martensitic Transformation, Leuven, Belgium, 1982.



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

$$\text{Surface area} \cong 2\pi r^2$$

Figure 2. Schematic shape of a martensite plate (or an embryo)

such a plate is then:

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

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

Using linear elasticity theory, the strain energy of such a plate is given by:

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

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

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

$$\text{or } A = \mu (r_o^2 + \epsilon_o^2) \quad \dots\dots\dots(3b)$$

where ν = Poisson's ratio, μ = shear modulus and r_o and ϵ_o are the shear and dilatational strains associated with shape deformation. In the above equation, elastic constants for the parent and the martensitic phases are assumed to be the same.

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

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

We note that below T_o , $\Delta g_c^{P \rightarrow M}$ is negative. Thus the total free energy change associated with the formation of a plate is:

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

Using the classical homogeneous nucleation theory, we can determine the free energy of nucleation W^* corresponding to a critical radius r^* and a critical semithickness C^* . To do this we set:

$$\text{and } \left. \begin{array}{l} \frac{\Delta W^{P \rightarrow M}}{r} = 0 \\ \frac{\Delta W^{P \rightarrow M}}{C} = 0 \end{array} \right\} \dots\dots\dots(6)$$

We thus obtain:

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

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

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

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

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

If the nucleation is completely random then each atom is a potential nucleation site and thus the nucleation rate $\dot{N}$ is given by:

$$\dot{N} = (N_o/V_m) \nu \exp (-W^*/kT) \dots\dots\dots(8)$$

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

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

From the classical homogeneous nucleation model it is possible to calculate V^* , c^* , r^* and W^* by using experimental values of $\Delta g_c^{P \rightarrow M}$ and theoretical values of A and λ .⁴

But classical homogeneous nucleation model can not be applied in martensitic transformations. Recent experimental results as well as theoretical considerations have clearly established that martensitic nucleation is heterogeneous.

In classical model, an embryo attains a critical size at a temperature and becomes nucleus. As an alternative to this a "reaction-path" model has been proposed⁵ in which, through a coordinated movement of atoms within the nucleation volume, the lattice passes through a succession of intermediate structures in one region and propagates like a strain wave. Thus, activation is achieved by fluctuations in configuration rather than the size. The embryo associated with such a mechanism, therefore, could be a strain center in which local arrangement of atoms is part way along the reaction path. Lattice imperfections, such as dislocations, internal surfaces etc. could provide such centers. Above M_s , these regions represent centers for high free energy.

Using Frank's dislocation model⁶, Knapp and Dehlinger⁷ have treated kinetics of athermal martensite formation in steel. This model requires pre-existence of embryos and deals with the propagation rather than nucleation of an embryo.

In this model, the martensite embryo is an oblate spheroid surrounded by dislocation loops. These loops are primarily screw dislocations with short edge components joining the positive and negative screws. Growth along $[1\bar{1}0]_\gamma$ and $[225]_\gamma$ can occur by the expansion of the loops and growth along $[55\bar{4}]_\gamma$ requires generation of new loops. This model of the embryos is shown in Figure 3.

The formation and propagation of these loops are equivalent to the creation of an interface and the energy required for this process is supplied by the chemical driving force $\Delta g_c^{\gamma \rightarrow \alpha'}$. Similarly the strain energy associated with this embryo is also supplied by the chemical free energy change.

The embryo in this case is triggered as soon as the overall free energy change per unit volume becomes zero. Writing $\Delta W^{\gamma \rightarrow \alpha'}$ as $\frac{1}{V} \Delta W^{\gamma \rightarrow \alpha'}$ and using our previous convention regarding surface and strain energies, we write:

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

Thus for spontaneous triggering

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

We can write the last two terms in eq. (10) as $\Delta g_{\text{non-chem}}$ and using equations (2) and (3) for an oblate spheroid:

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

where r , c , λ and A have the same meaning as before. For a radius, $\Delta g_{\text{non-chem}}$ can be minimized with respect to c by

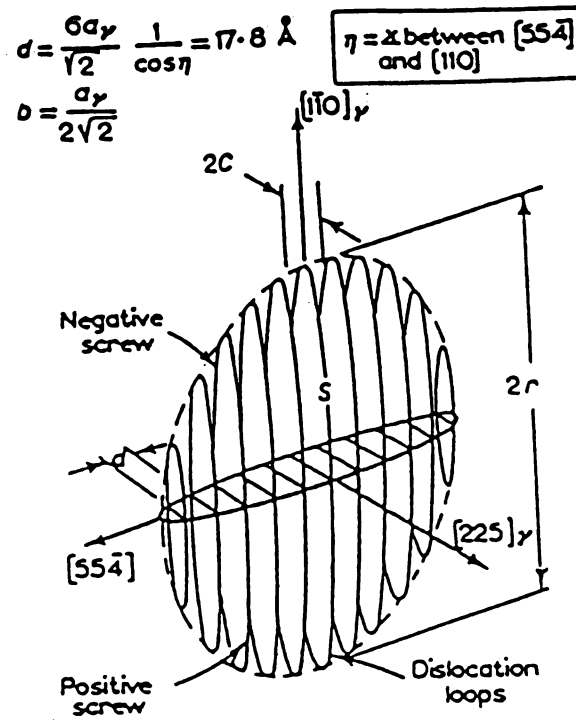


Figure 3. Knapp and Dehlinger Model of the Martensite Embryo.

setting $(\partial \Delta g_{\text{non-chem}} / \partial c) = 0$

$$\text{Thus } \Delta g_{\text{non-chem}}|_{\text{min}} = \left(\frac{6\lambda A}{r} \right)^{\frac{1}{2}} = \frac{3\lambda}{c} \dots\dots\dots(12)$$

$$\text{where } c = \left(\frac{3\lambda r}{2A} \right)^{\frac{1}{2}} \dots\dots\dots(13)$$

Using equations (12) and (13), the minimum in $\Delta g_{\text{non-chem}}$ now can be calculated as a function of embryo size. It can be seen from eq. (12), that for a fixed value of c , the minimum value of $\Delta g_{\text{non-chem}}$ decreases with increasing radius, r . That is, larger the embryo, smaller is the non-chemical energy barrier. It also means that as the embryo grows, non-chemical barrier becomes smaller and smaller, thus the propagation of the interface could be very rapid. Below M_g , $\Delta g_c^{\gamma \rightarrow \alpha} > (\Delta g_{\text{non-chem}})_{\text{min}}$ for the larger embryos and net chemical forces acts as a "stress" to move the dislocation interface. It is also possible to visualize the interaction of an applied stress with such an embryo both above and below the M_s . An estimate of the largest embryo (at M_s) can be made by using appropriate experimental and estimated values of various parameters.

The above model has been modified by Kaufman.⁴ In his model, an equivalent dislocation loop, encircling the oblate spheroid embryo, on the habit plane is considered. This circumferential dislocation loop then contains the interfacial energy of the embryo. The radius of this loop is r and its

equivalent Burger's vector, ξ , is given by cb/d , where 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 model (Figure 3). The line tension of this loop is then, $\Gamma = \mu \xi^2 / 2$, where μ = shear modulus. If the loop is subjected to a net shear stress τ , the corresponding increase in free energy is (starting from zero size)

$$W^l = 2\pi r \Gamma - \pi r^2 \xi \tau \quad \dots\dots\dots (14a)$$

$$\text{where } \Gamma = \mu \xi^2 / 2 \quad \dots\dots\dots (14b)$$

The shear stress τ is equal to chemical driving force ($-\Delta g_c$) minus strain energy per unit volume $\lambda c/r$. Thus

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

Where λ is the surface energy. We note from eq. (7e) that $c^2/r = \lambda/A$. Thus substituting from eq. (15) and (14b) in (14a), we have:

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

By maximizing W^l as a function of r , we obtain

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

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

where r_c is the critical radius of the loop at which it can lower its energy by expanding. This growth model is similar to mechanical twinning or kink band formation. Comparing

with eq. (7b):

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

Since $r_c > r^*$, ΔW^* is actually negative at r_c .

It has been proposed (17) that during isothermal transformation those embryos with radius r given by $r^* < r < r_c$ (r^* is the radius in previous model) are activated. For $r < r^*$, the magnitude of W^* is so large that thermal activation is not possible and the embryos with $r > r_c$ are already triggered by the athermal process.

In more recent years, some more models have been developed for heterogeneous nucleation of martensite. Olson and Cohen⁸ have studied a model in which a portion of a wall of dislocations underwent faulting on close-packed planes to produce the close-packed phase in the fcc to hcp transformation. A second shearing is required to produce the fcc to bcc transformation. No activation barrier occurs in their thermodynamic equations, but a barrier is postulated to result, however, from the thermally activated motion of the dislocations associated with the internal slip as first suggested by Magee.⁹ The free energy change for nucleation of a martensite embryo attached to and inside a growing dislocation loop has been computed by Easterling and Thölen.¹⁰ No nucleation barrier was found for internally twinned martensite. Their choice of 20 ergs/cm² for the interfacial free energy is

much smaller than the 200 ergs/cm^2 estimated by Kaufman and Cohen³ for a martensite embryo. Use of 200 ergs/cm^2 would yield an impossibly large barrier for nucleation in their model. Suezawa and Cook¹¹ suggest that a pile-up of dislocations can initiate heterogeneous nucleation of martensite and the classical free energy barrier to nucleation can exist for a martensite embryo at a pile up.

3. PREVIOUS WORK

3-1 Isothermal Formation of Martensite

Although martensite formation is more generally a temperature-dependent process, its time-dependent nature has also been revealed in some cases. It was observed as early as 1948-49 by Fletcher, Averbach and Cohen^{12,13} and as much as 5% isothermal martensite formation was noted in both plain carbon and low alloy steels. It was, however, considered then as a sort of tailing off effect of the main reaction.

Kurdjumov and Maksimava^{14,15} put forth a revolutionary claim to have suppressed the transformation completely in a 6Mn 0.6C steel by rapid cooling in liquid N₂ and as much as 25% martensite formed on reheating and holding isothermally at temperatures. Rate at one temperature was found to have an initial constant rate and followed by a gradual decrease over a greater interval of time. A temperature was found where greatest amount of isothermal martensite formed. Partial suppression of martensite was claimed by them in a 1.6C steel. By quenching first in alkaline iced water (about 20% martensite formed) and then further cooling to liquid N₂ temperature suppressed most of the transformation. On reheating isothermal transformation could be studied. Partial suppression of martensite formation was also indicated by Das Gupta and Lement¹⁶ in a 15 Cr 0.7C steel, although

on athermal amount proceeded the isothermal part. A maximum initial rate was noted at an intermediate temperature, (-110°C). At liquid N_2 temperature, starting with an athermal amount of about 15% as high as 27-28% amount formed on isothermal holding. Similar type of isothermal formation preceded by athermal amount was also noted by Machlin and Cohen¹⁷ in an Fe-Ni alloy.

A few years later, Kulin and Speich¹⁸, Cech and Hollomon¹⁹ reported almost complete isothermal formation in Fe-Cr-Ni alloys and in Fe-Ni-Mn alloys respectively. Subsequently Shih, Averbach and Cohen²⁰ reported complete isothermal formation at -196°C in a different but similar composition as Cech and Hollomon. Further evidence of isothermal formation of martensite in ferrous alloy followed and in more recent year Wasaka and Wayman²¹ reported isothermal formation in a Fe-Ni-Mn alloy of slightly different composition from that of Shih, Averbach and Cohen.

It may be seen from the various work that athermal martensite is not essential for isothermal martensite. However, since martensite transformations are strain sensitive and autocatalytic, athermal martensite may stimulate the nucleation of isothermal martensite when the cooling is interrupted below M_s . Thus nature of isothermal formation is studied either in absence of athermal martensite or more

conventionally by introducing a fixed quantity of athermal martensite. Whether the reaction takes place in the absence of athermal martensite or in the presence of a fixed amount, the isothermal kinetics display a C-curve behavior, with the maximum rate coming at some intermediate temperature.

In almost all instances the isothermal reaction precedes by the nucleation of new plates, rather than by the growth of existing ones and the plates reach the full size within a small fraction of a second. Kurdjumov has concluded that nucleation rather than growth is the rate limiting step in isothermal formation and metallographic observations confirm this.

3-2 Stabilization of Austenite-Martensite Transformation

First systematic study was done by Harris and Cohen²² as early as 1949 in a 1.1C 1.5Cr steel. They observed that unless the temperature of holding is below a certain temperature below M_s (called σ_s) stabilization does not occur and the degree of stabilization occurring on holding below σ_s increases linearly as the temperature of holding is lowered.

Effect of stabilization on isothermal formation of martensite in a Fe 29Ni alloy was reported by Machlin and Cohen²³ in 1951. They tried to explain the process as resulting from relaxation of existing martensite embryos.

Working on a 15Cr 0.7C steel Das Gupta and Lement²⁴ observed that when the amount of martensite as formed by interrupting the cooling below M_s exceeds a critical amount the stabilization effect induced on subsequent cooling to lower temperature is more marked. However, stabilization effect eventually disappears on continued holding at such temperature by isothermal formation of martensite. Besides the authors further observed that higher the temperature of intermediate cycling the more permanent is the stabilization effect.

Morgan and Ko²⁵ reported for steels containing about 1C 0-5Ni that stabilization of austenite occurs during isothermal holding above and below M_s . The rate of stabilization varies with the composition of the austenite, increases with increasing temperature and is greatly increased by the presence of martensite. The stabilization above M_s causes a depression of M_s and increases the amount of austenite at a reference temperature near M_s .

Edmondson²⁶ examined stabilization in a 10Ni 1C alloy and suggested a theory for stabilization on the diffusion of interstitial solute atoms to preferred sites within the metal to explain the results obtained.

The work of Woodilla, Winchell and Cohen²⁷ showed that in a 30.8Ni 0.007C steel the activation energy for the

stabilization process is comparable to that for the diffusion of carbon (or nitrogen) in ferrite rather than in austenite. They suggested that the interstitial diffusion controlling the stabilization occurs within the martensite embryos rather than in the matrix of the parent phase.

Kinsman and Shyne²⁸ reported that a fixed population of martensite embryos is affected by the segregation of carbon to the embryo-austenite interface. The anchoring effect of the segregated carbon atoms inhibits the activation of the embryos into propagating martensite plates.

Thermal stabilization of the athermal martensite transformation was evaluated in Fe-Ni-C alloys in the isothermal temperature range of 0-200°C by Kinsman and Shyne.²⁹ They reported a distinct change in the characteristics of thermal stabilization at low and high temperature of aging. At low ageing temperature the extent of thermal stabilization varied with time, increasing to a maximum value and the maximum decreased with increasing aging temperature. The phenomenon behaved according to the authors in a manner consistent with a mechanism based on pinning of the interface of the martensite nucleus by a carbon atmosphere formed during aging. At higher temperature of aging beyond 80°C the extent of stabilization as a function of time was initially constant and then began to increase at long times. The authors report the

observations at high temperature of aging to follow some other mechanism.

Jin and Huang³⁰, working on a Fe 29Ni 4.3Ti observed stabilization of high strength austenite as well as that of annealed austenite and attributed this to the formation of G.P zones or short-range order of less than $10\text{\AA}$ in size. The initial rate of M_s suppression was larger in an ausaged and subsequently transformation-strengthened austenite than in a simply ausaged austenite.

Theories on Stabilization of Austenite-Martensite Transformation--Theories of stabilization attribute the phenomenon either to change within the untransformed austenite or to change in the effect which already formed martensite has on the subsequent progress of the transformation. There is a consensus of opinion that thermal stabilization of austenite is caused by relaxation of stress in the austenite around the existing martensite plates. This stress is believed to assist nucleation of fresh plates in an unstabilized austenite. However, how this relaxation takes place has been explained differently by different investigators. Thermal stabilization is a thermally activated process involving mostly interstitial solutes--kinetics being governed by diffusion of interstitials: C or N.

(1) Das Gupta and Lement²⁴ are of the view that relaxation of strain-embryos takes place by diffusion of C-atoms from the martensite present to the strain-embryos in austenite. The driving force for the latter process is provided by the difference in carbon activity which exists. In presence of martensite, activity of carbon in martensite is presumably higher than in austenite. Thus, there will be a tendency for the C-atoms to diffuse from the edges of the martensite plates into strain embryos in the vicinity.

(2) Morgan and Ko²⁵ proposed that C-atoms can diffuse during slow cooling or isothermal holding to form "clusters" or precipitates around the existing dislocation and thereby pinning the dislocations. This would harden the structure and thus make growth of austenite-martensite interface and accompanying deformation more difficult.

(3) Based on the model of Knapp and Deblinger, it has been proposed by Kinsman and Shyne²⁸ that thermal stabilization of austenite is a result of immobilization of the semi-coherent interface between martensite and austenite matrix because of segregation of interstitial solute atoms to the dislocation array comprising the interface. The time and temperature dependence of thermal stabilization must be directly related to the kinetics of solute segregation to the surface of the nucleus. Thus by developing a time law for interface

segregation the authors develop a time law for thermal stabilization.

(4) Glover and Smith³¹ suggest that stabilization may be due to relaxation of elastic stresses by diffusion of carbon and nitrogen inside the martensite, leading to first stage of tempering.

(5) According to Maksimova et al³², the relaxation process leaves structural imperfection in its wake and is thus doubly effective in causing stabilization because such imperfections impede the growth of martensite plates.

(6) According to Kurdjumov³³, a readjustment of the stresses and imperfections that ordinarily assist transformation is likely to occur in a transformed alloy even at room temperature. The structure that results from this is more stable and is more difficult to transformation.

3-3 Burst Formation

In certain cases under suitable condition the progress of martensite formation is accompanied with "burst" formation—a large volume of transformation taking place in an abrupt manner. The "burst" may occur either at M_s or at a temperature below M_s .

The "burst" may, also occur on long isothermal holding after an incubation period. Recommencement of transformation following a stabilizing treatment may, under certain conditions,

be accompanied by a burst.

The resultant transformation curves have been shown to be irregular in shape, especially when the size of the burst is relatively small. With progress of transformation the transformation-temperature curve tends to smooth out but may still remain undulating. When the size of the initial burst is large, the course of the subsequent transformation is more regular. The increment in percent martensite immediately following such a burst is small; this process has been named "pseudo-stabilization".

The process is autocatalytic in nature and metallographic examination show these plates forms in a zig-zag pattern in the specimen. The process is somewhat analogous to a chain reaction, the stresses produced by one plate assisting in the activation of the nucleus of another plate. This also means that the plates formed during the burst are such as to reduce the stress in the matrix, the whole group forming a partially self-accommodating system and this is probably why the individual plates are much wider than normal. Machlin and Cohen¹⁷ found that about 25% of the whole transformation in an iron-nickel alloy was produced in a burst of very short duration, accompanied by an audible click.

Entwisle and Feeney³⁴ suggested that where bursts occur, it appears that the first successful nucleating event triggers

off the whole burst, and that M_b truly represents the start of transformation. The burst temperature M_b is essentially a function of the thermal treatment to which the austenite has been subjected. The magnitude of the burst is a function of the austenite grain size and the temperature at which the burst has occurred.

Interpretation of Burst--Suzuki and Honma³⁵ referred the burst transformation akin to deformation twinning. The propagation of such a transformation requires that the dislocation bordering the martensite embryos can multiply along certain favored habit planes. The mechanism of multiplication is such that this process is not favored on another common type of habit planes found in steel viz 225, the transformation then occurs by a process similar to slip.

Machlin and Cohen³⁶ did not agree with this view. They suggest that when martensite forms the austenite is plastically deformed and as a result, new embryos are created or the existing ones made supercritical at a given temperature. Simultaneous activation of a large number of such embryos result in a burst.

Cech and Turnbull³⁷ offered a completely different interpretation. They argue that since the burst phenomenon is more prevalent in Fe-Ni-C alloys and as these alloys are

relatively free from the heterogenities which nucleate martensite in these alloys, a large degree of supercooling is necessary to nucleate martensite. When transformation starts, it does so form many centers, thus causing a burst.

Philibert and Crussard³⁹ found C and N atoms responsible for stabilization of austenite and the subsequent burst transformation. They proposed a theory according to which the simultaneous activation of many embryos is made possible by the propagation of an elastic wave.

3-4 Effect of Plastic Deformation on Martensite Formation

Martensite transformations can be affected in a variety of ways by the application of stress and by plastic strain.

Patel and Cohen³⁹ demonstrated quantitatively how the initial nucleation of martensite is assisted by applied stress. A mechanical work term consisting of the product of the applied stress field and the transformation strain is added to the free energy change that provides the driving force for the transformation. Low temperature plastic deformation of the parent austenite prior to the martensite transformation influences both the nucleation and the growth of martensite. Nucleation is assisted by small amounts of plastic deformation, while extensive cold working suppresses nucleation.

The retarding effect of large plastic deformations on the subsequent martensitic transformation has been reported by

Kurdjumov.⁴⁰

Breinan and Ansell⁴¹ studied the effect of deformation on the forward transformation in an Aircool-S steel and found that the decrease in the martensite start temperature, M_s , with deformation applied above M_d (the highest temperature at which deformation can initiate the formation of martensite) correlated with the change in austenite yield strength. They concluded that M_s decreased with increasing yield strength as a consequence of reduced dislocation mobility, which may alternately affect either the nucleation of new martensite or the propagation of the martensite-austenite interface. Increasing the yield strength by alloying decreased M_s in the same manner. In a similar study, Ankara⁴² used transformation cycling of an Fe 30Ni alloy to increase the austenite yield strength and also found a decrease in M_s with increasing yield strength, which he attributed to a change in dislocation density.

According to Pope⁴³, the effect of plastic deformation introduced by rolling at room temperature on the austenite start temperature of an Fe 30.3Ni 0.005C alloy has been determined. The austenite start temperature increases monotonically with deformation. Microhardness measurement show that the austenite start temperature increases with the yield strength of the martensite.

Guimaraes and Shyne⁴⁴ reported that plastic deformation of austenitic specimens (Fe -28.45Ni -0.21C, Fe -31.15Ni -0.095C) at room temperature caused a rise in the M_s temperature. The effect was maximum, about 15°C, at an intermediate stage of deformation, 50% reduction by rolling. The so called mechanical stabilization of austenite could only be observed in specimens severely deformed (more than 8% reduction by rolling).

Maxwell, Goldberg and Shyne⁴⁵ reported that in the Fe-Ni-C alloys two distinctly different types of martensite formed concurrently with plastic deformation. The large difference in morphology, distribution, temperature dependence, and other characteristics indicate that the two martensites form by different transformation mechanisms. The first type, stress-assisted martensites, is simply the same plate martensite that forms spontaneously below M_s except that it is somewhat finer and less regularly shaped than that formed by a temperature drop alone. This difference is due to the stress-assisted martensite forming from cold-worked austenite. The second type, strain-induced martensite, formed along the slip bands of the austenite as sheaves of fine parallel laths.

Since imperfections play an important role in the nucleation process of martensite and thermal stabilization is

associated with same sort of relaxation process in this austenite around the existing martensite involving rearrangement of imperfections and immobilization of the embryo sites present, it is expected that plastic deformation before and after martensite formation would have considerable influence on the stabilization process. Keeping this in view it is proposed to have a new look at the stabilization process in a suitable steel with varying amount of deformation at different stages.

4. EXPERIMENTAL PROCEDURE

4-1 Material

The chemical composition of the steel investigated is as follows:

C%	-	Ni%	-	Fe%
1		16		Balance

Specimen preparation: Material was obtained in a rectangular bar form of an approximate dimension of 2.1" x 0.08" x 0.05". Experimental specimens ranging from 0.040" - 0.060" thickness were obtained by warm rolling in austenite condition in several steps. Austenitizing before rolling was carried out in evacuated and sealed quartz tubes. Decarburization was avoided as much as possible during warm rolling.

4-2 Austenitizing

Experimental specimens were austenitized for 2 hours at 1100 °C and then water-quenched to room temperature. The austenitizing temperature was selected on the basis of solution of carbides and reasonable austenite grain size. Prior to austenitizing treatment all specimens were given a stabilizing treatment by cooling to liquid nitrogen temperature. It may also be noted that in order to obtain comparable results a single specimen was repeatedly used while running a definite series of experiments. M_s -temperature did not materially alter on repeated austenitizing of the same specimen.

4-3 Determination of M_s -Point and Measurement of Extent of Transformation

M_s -point and the progress of transformation of austenite to martensite was determined by following the change in electrical resistance using a Kelvin double bridge. Specimens used for electrical resistance measurement were approximately of the following dimensions:

0.04" - 0.06" thickness

0.08" width

2.1" length

The specimens were gripped by a suitable holder having knife-edge contact points for the current and potential leads. The specimen dimensions were manipulated so as to maintain electrical resistance ranging between 10^{-2} and 10^{-3} ohms. Subzero temperature necessary for following the progress of transformation was obtained by holding the specimens at different levels of the airspace above liquid nitrogen kept in a tall thermal flask as shown in Figure 4. The specimens were lowered in steps to different levels manually and the rate of cooling was maintained approximately as 2°C per minute. A range of temperature between $+20^{\circ}\text{C}$ and -195°C could be followed by the set up. The temperatures were measured by a Leeds-Northrup potentiometer using a copper constant thermocouple placed in contact with the specimen.

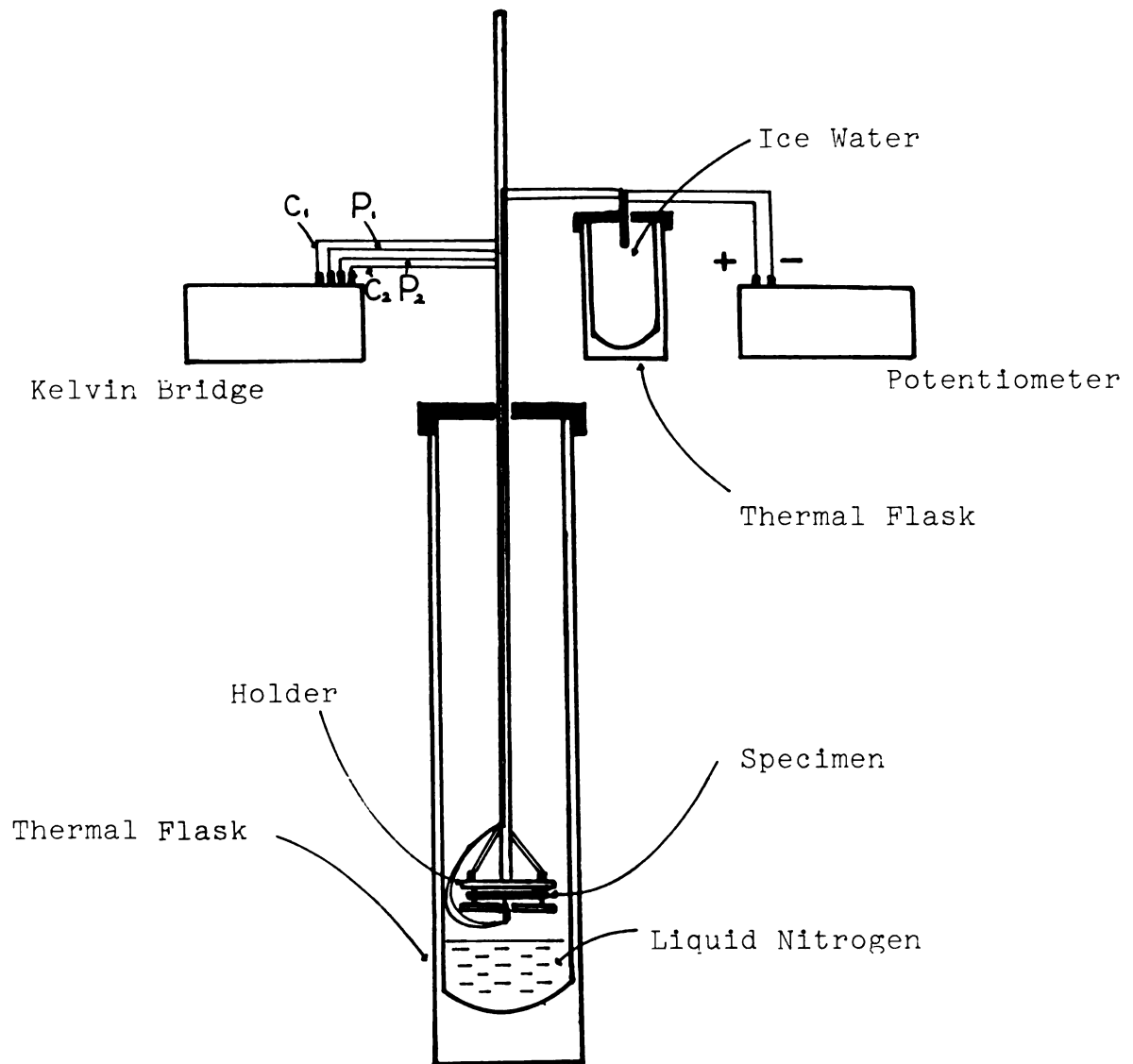


Figure 4. Experimental Apparatus

4-4 Method of Deformation

Specimens were deformed by tensile elongation in an Instron Tensile Machine using a friction grip. The rate of loading was maintained normally at 0.05 cm per min. and was varied between 0.2 cm to 0.02 cm per min. in selected cases. The extent of deformation ranged between 0.5% to 4.0% usually.

4-5 Stabilization Treatment

Specimens containing a definite amount of martensite and deformed in various manners were reheated to $+39^{\circ}\text{C}$ and aged for 1 hour before finally sub-cooling to follow the course of transformation. In selected specimens time of aging was varied from 5 min. to 4 hours. Schematic drawings of the heat treatments involved are shown in Figure 5.

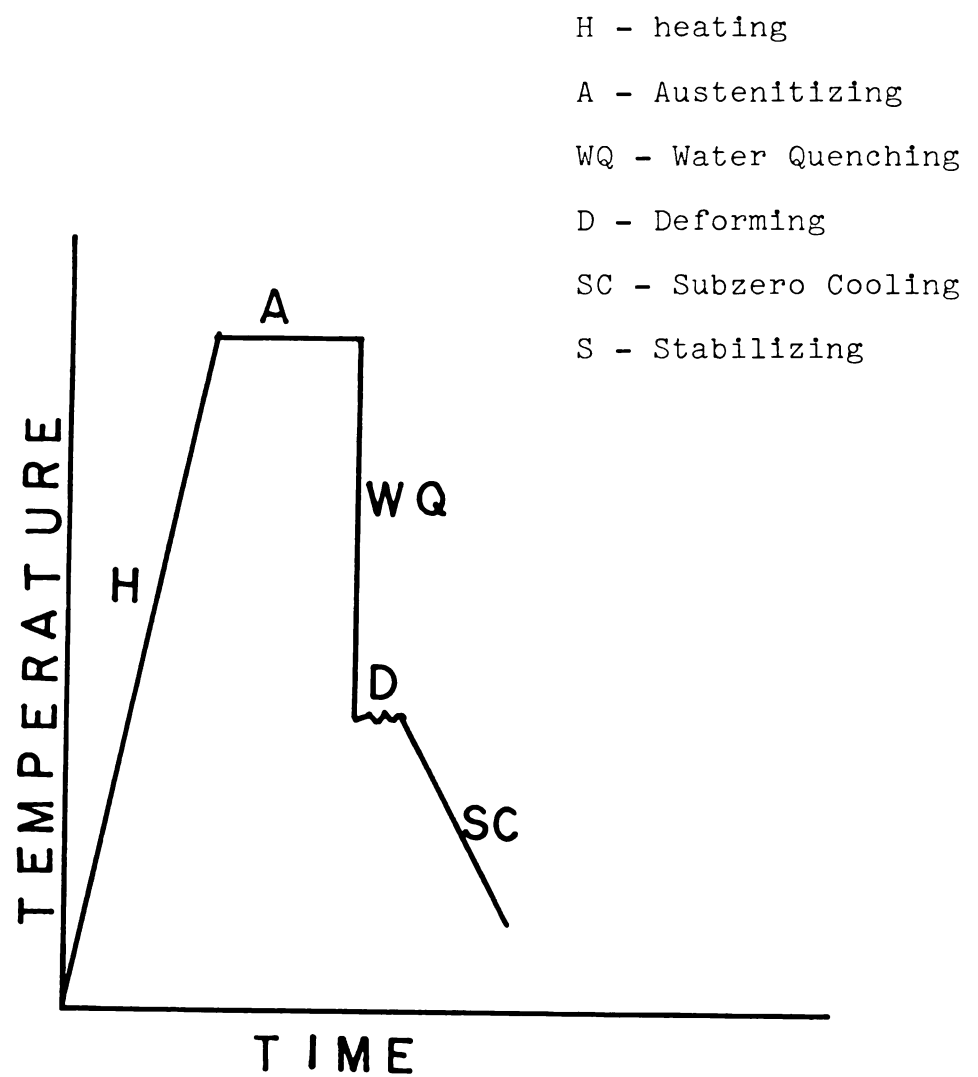


Figure 5-a Schematic Diagram Depicting the Treatment

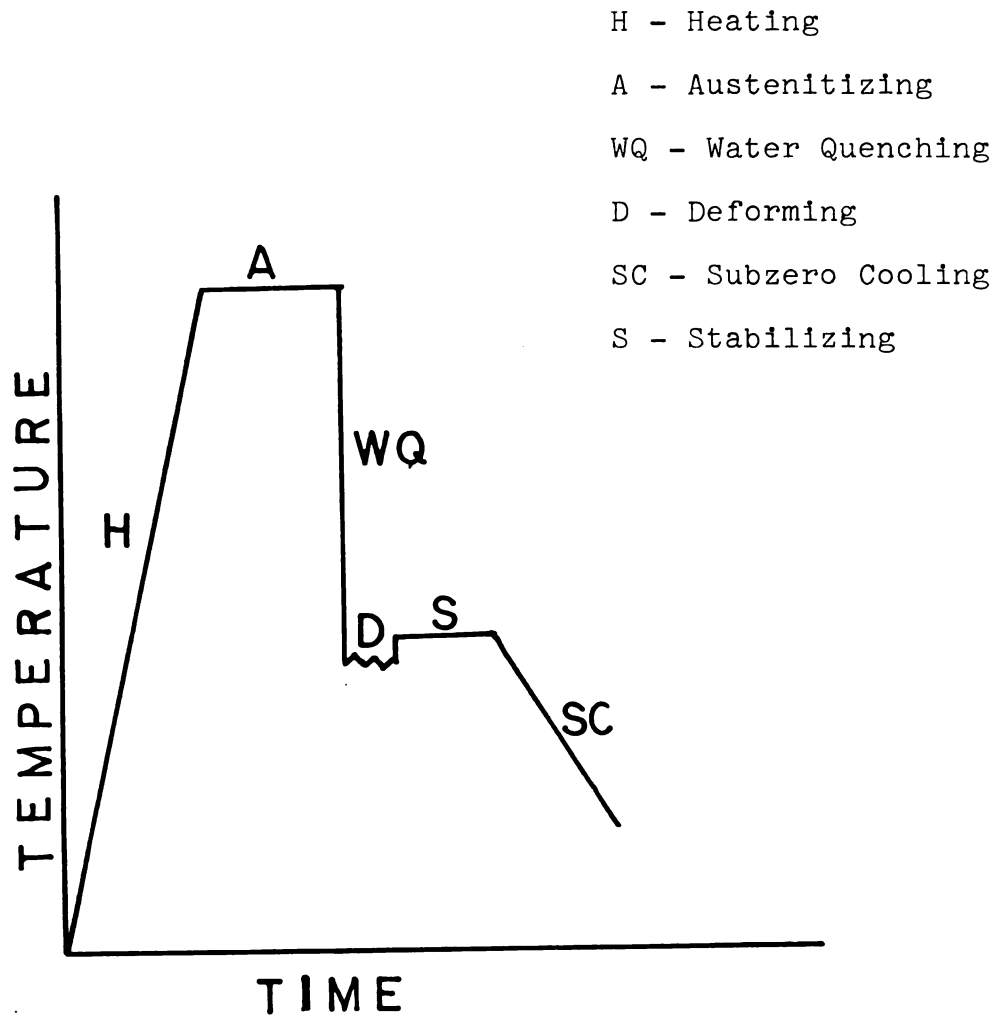


Figure 5-b Schematic Diagram Depicting the Treatment

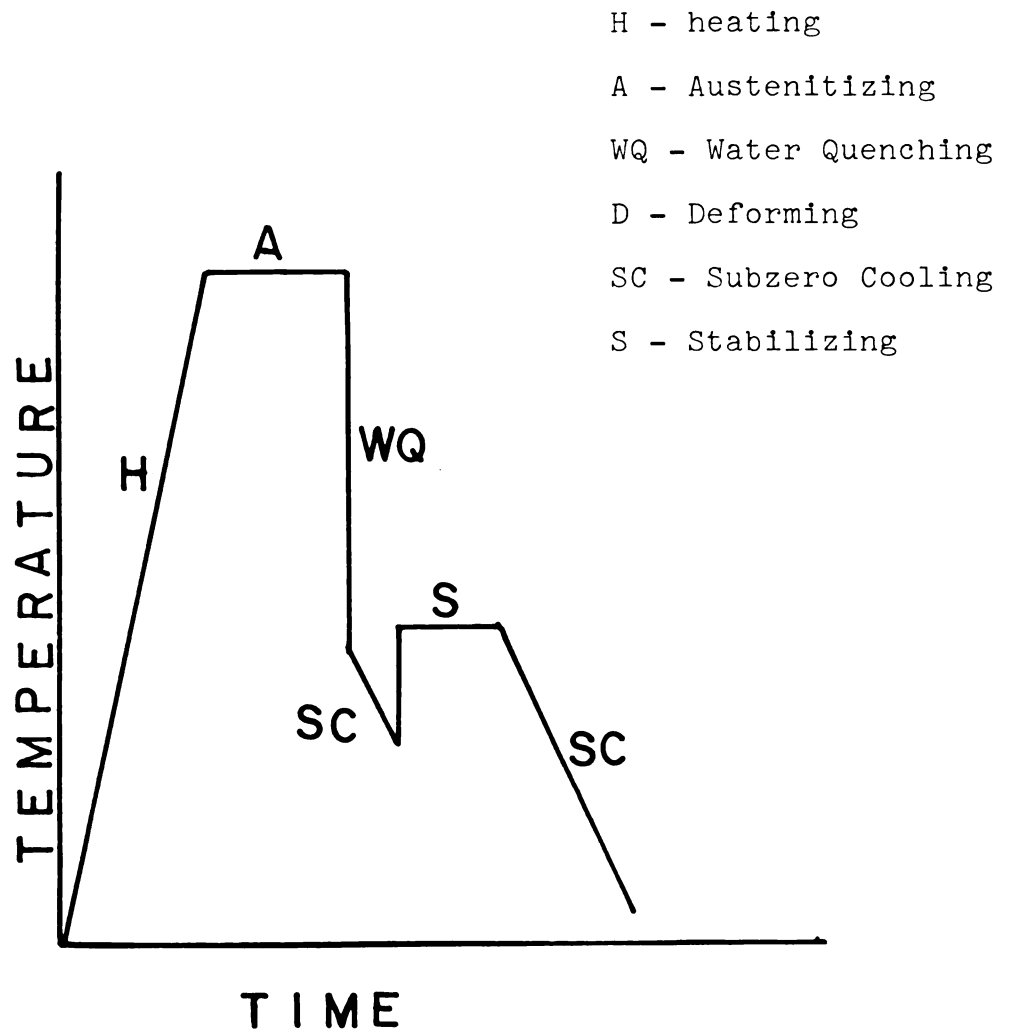


Figure 5-c Schematic Diagram Depicting the Treatment

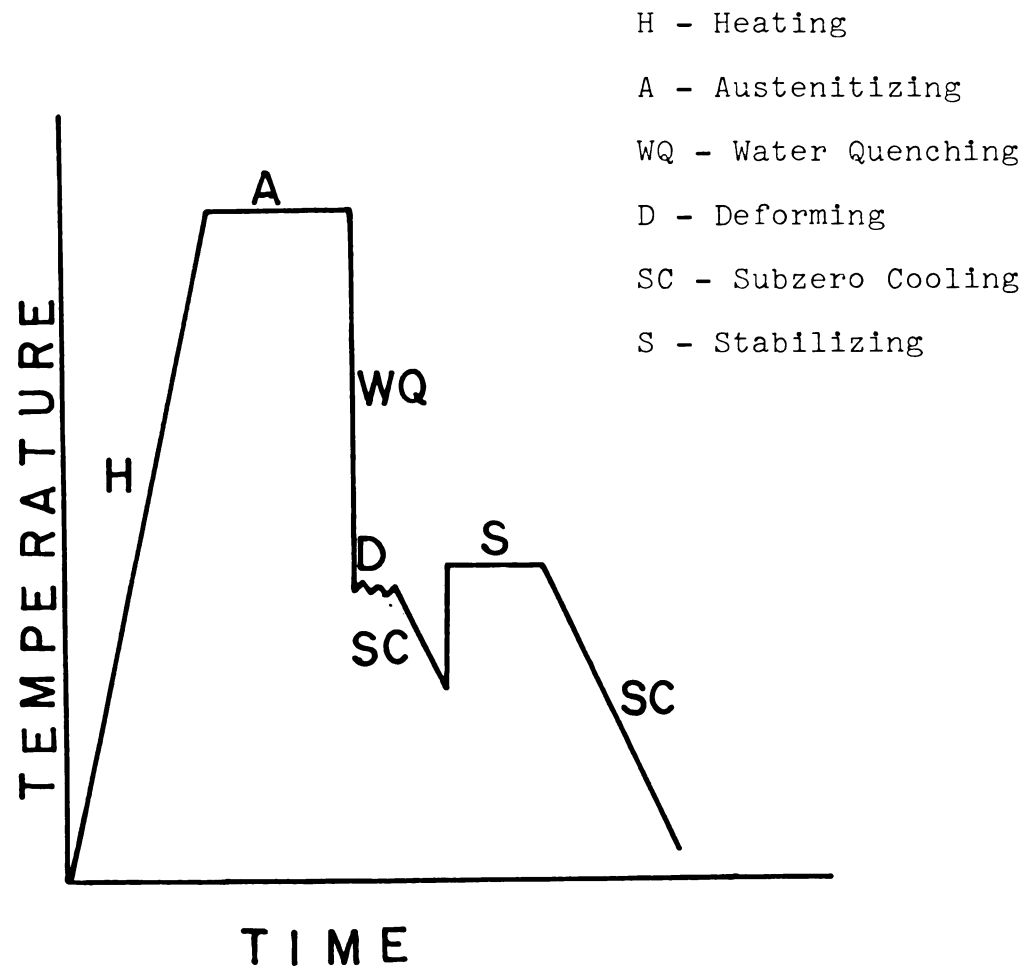


Figure 5-d Schematic Diagram Depicting the Treatment

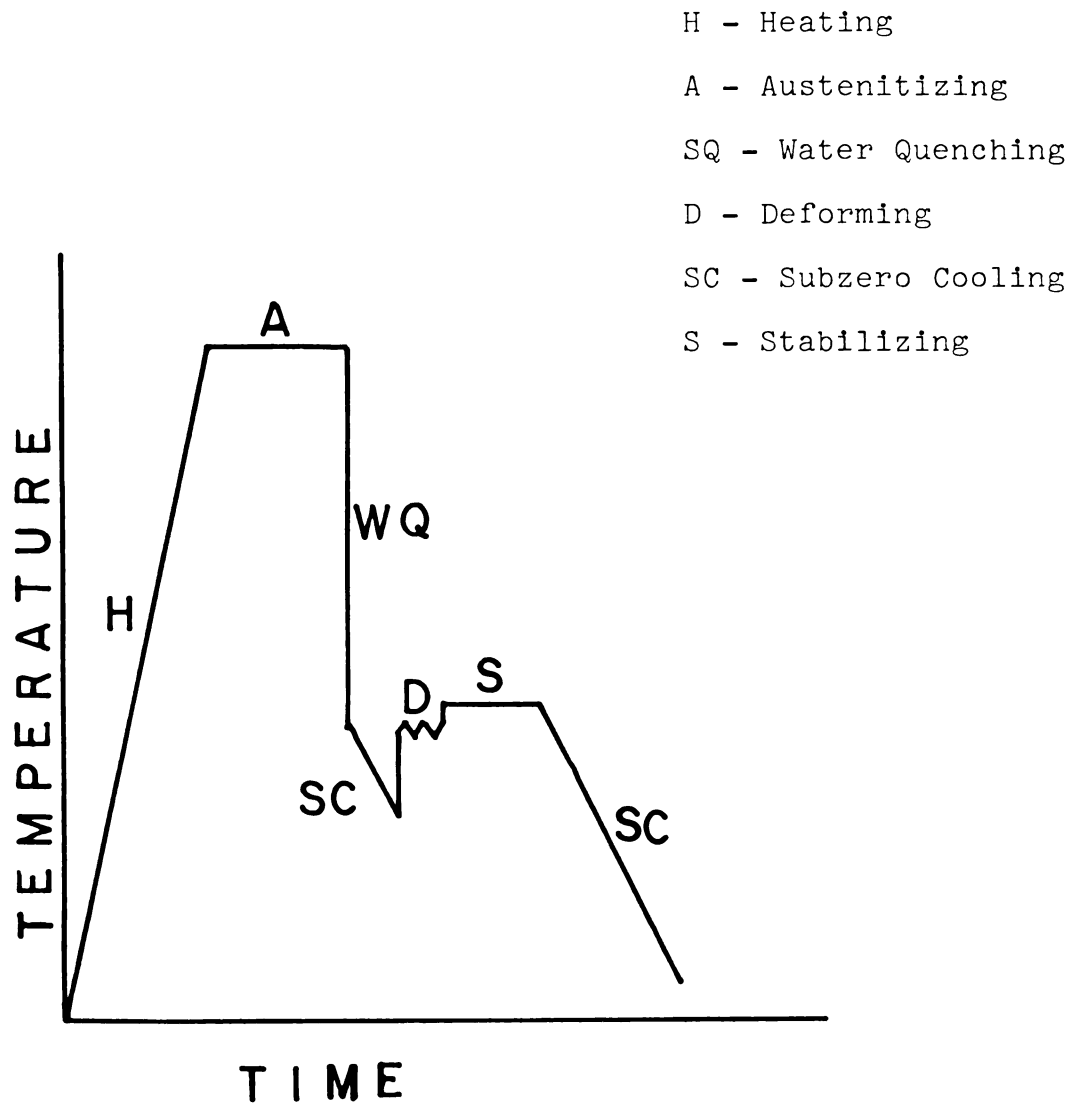


Figure 5-e Schematic Diagram Depicting the Treatment

5. RESULTS

5-1 Effect of Strain Rate on M_s -Point

Austenitized and quenched specimens were deformed at room temperature to a constant 2.0% tensile elongation at different rates of straining. Cross-head speed of 0.2 cm/min., 0.05 cm/min. and 0.02 cm/min. were used. The effect of different straining rates on the M_s -point has been presented in Table 1. The treatment followed is shown in the schematic diagram (Figure 5-a). An increasing depression of M_s -point with decreasing rate of straining may be noted. Figure 6 shows the variation of M_s -point as a function of rate of strain prior to martensite formation.

5-2 Effect of Prior Deformation on M_s -Point

Austenitized and quenched (above the M_s temperature) specimens were deformed to various extents by tensile elongation prior to martensite formation. The extent of deformation ranged from 0.5% to 4.0% (strain). The effect of prior deformation on the M_s -point has been studied (1) by cooling subsequent to deformation and (2) by stabilizing at 39°C for 1 hour before transformation. In both cases the M_s -point is substantially depressed and with increasing deformation the extent of depression of M_s increases consistently.

Table 1. Effect of Strain Rate on M_s Temperature for 2% Elongation

Austenitizing Temperature	Quenching	Strain Rate (sec ⁻¹)	M_s Temperature
1100 °C for 1 hr.	Water	0.02	-29
1100 °C for 1 hr.	Water	0.05	-19
1100 °C for 1 hr.	Water	0.2	-11

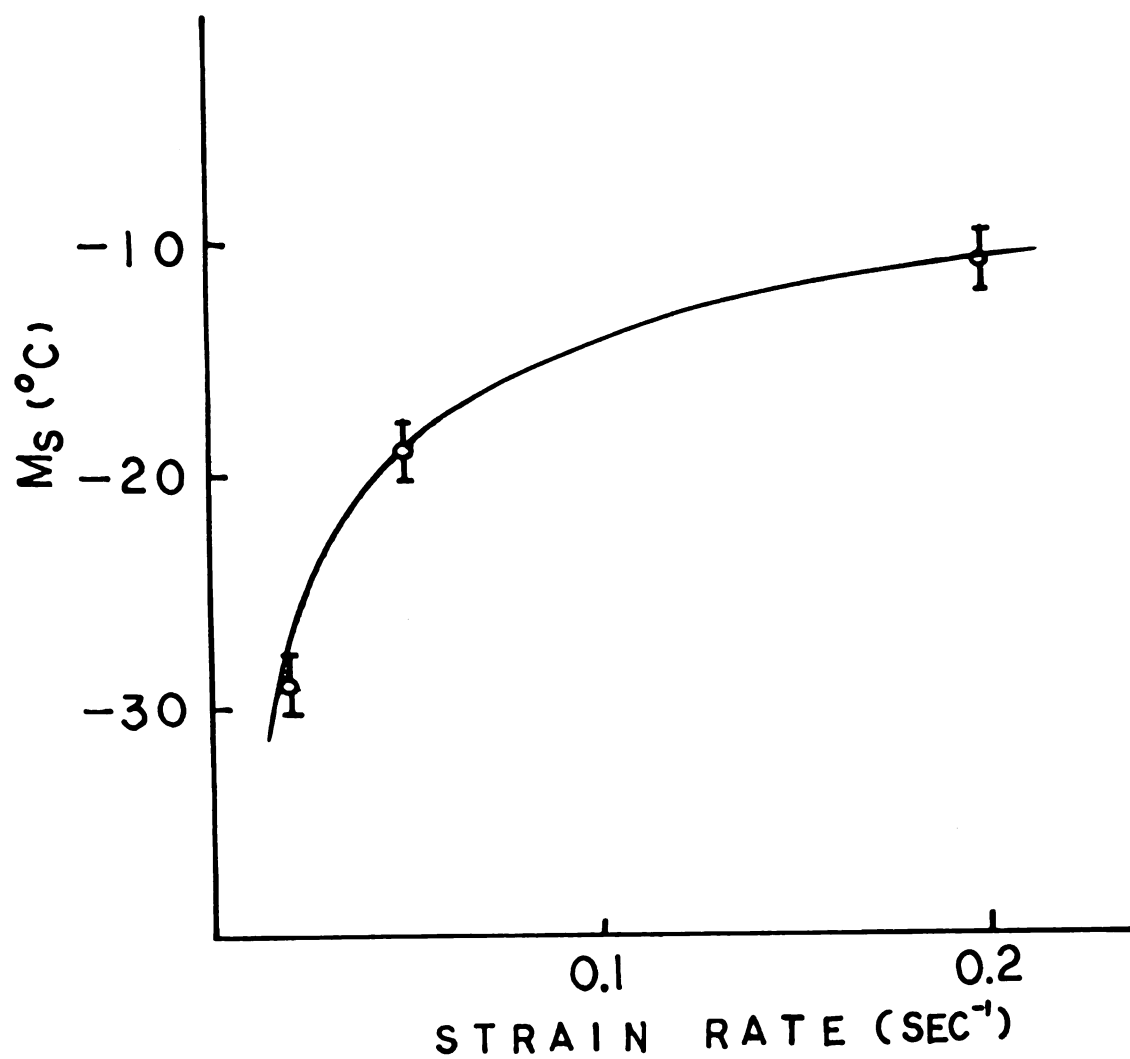


Figure 6. Variation of M_s -Point as a Function of Strain Rate: 2% Elongation

However, the degree of lowering of M_s in specimens having stabilizing treatment at 39°C for 1 hour is smaller than that in specimens having no stabilizing treatment as shown in Figure 7. The rate of decrease of M_s with deformation appears to be the same in both cases. Resistance change followed as a function of temperature for specimens deformed to various extent has been plotted in Figure 8. Large change in resistance with martensite formation, indicating burst formation, can be noted in both underformed and deformed specimens. The size of burst increases with the extent of prior deformation. It may be further noted that although the undeformed specimen shows two successive relatively smaller burst with beginning of transformation the deformed specimens start transforming with one large burst. The course of transformation as followed by resistance measurement does not change with varying extents of prior deformation. Similar trend of the course of transformation is noted in specimens deformed and stabilized at 39°C for 1 hour prior to transformation (treatment b). The treatment followed is shown in the schematic diagram (Figure 5-a and 5-b).

5-3 Effect of Time of Stabilizing Treatment on the Extent of Stabilization

Specimens with and without prior deformation (2%) were transformed to have 30% martensite and subsequently stabilized

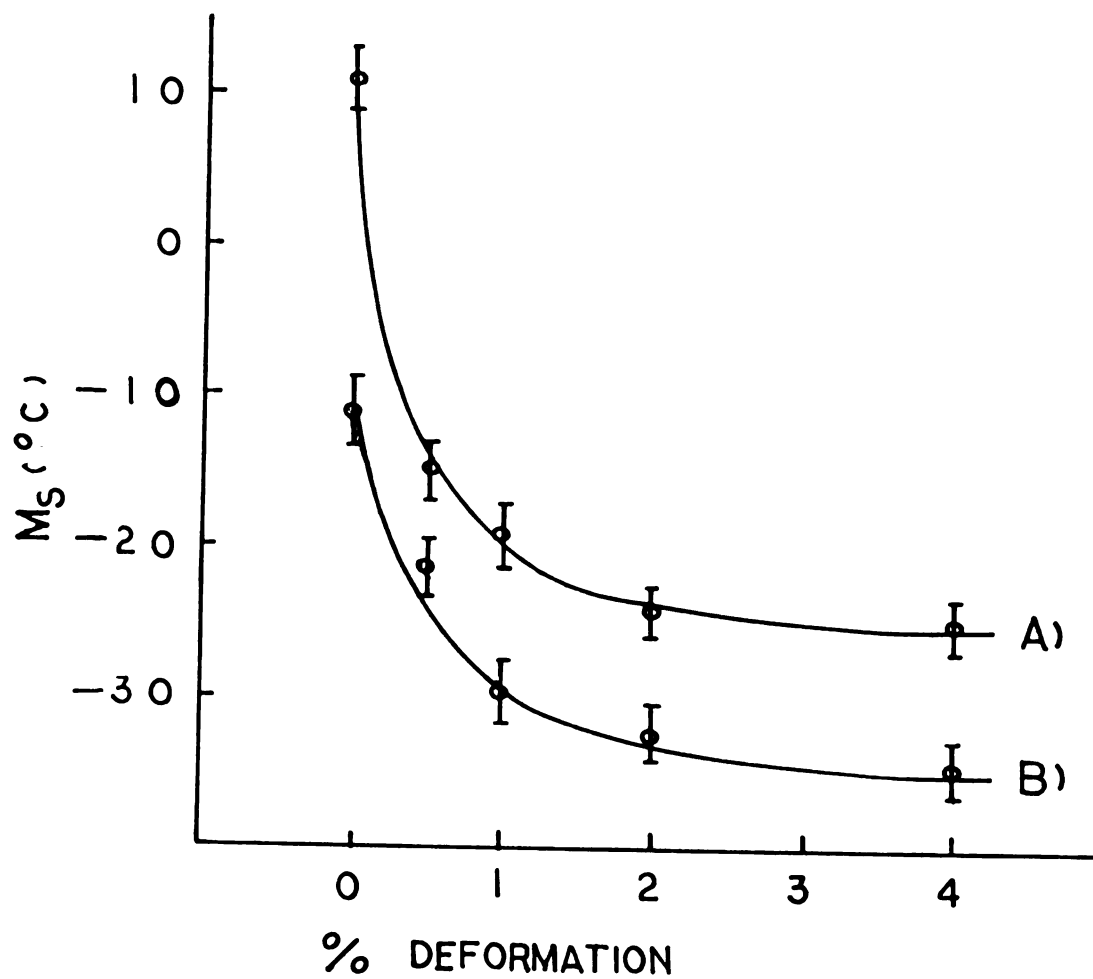


Figure 7. Effect of Prior Deformation in the Austenitic Condition on M_s -Point in a 16Ni-1C Steel
A) Without
B) With Additional Stabilizing Treatment

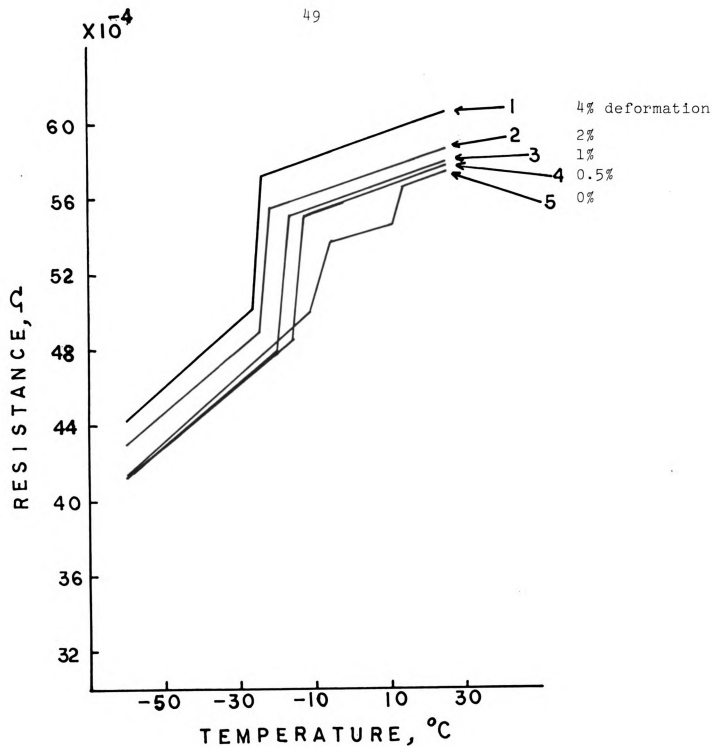


Figure 8. Change in Electrical Resistance as a Function of Temperature in Fe-16Ni-1C alloy Deformed to Variable Extents prior to Transformation

at 39 °C for various lengths of time. The variation in the extent of stabilization have been reported as a function of time in Table 2 and a plot of the data is given in Figure 9. The treatment followed is shown in the schematic diagram (Figure 5-c and 5-d). In the undeformed specimen, the extent of thermal stabilization, θ , varied with time, increasing to a maximum value, and then decreasing at longer aging times. The degree of stabilization for 4 hours is somewhat less than that obtained for 1 hour stabilization. The deformed specimen behaves similarly, but the degree of stabilization is higher in this case. For example, the degree of stabilization for an aging time of 60 minutes is 27 °C for the deformed sample compared with about 20 °C for the undeformed case.

5-4 Effect of Deformation prior to Formation of Martensite on Stabilization

Austenitized and quenched specimens were deformed to various extents ranging from 0.5% to 4.0% by tensile elongation prior to martensite formation of two different amounts. Results are observed for 35% and 50% martensitic transformation. The treatment followed is shown in the schematic diagram (Figure 5-d). The alteration in the extends of stabilization following stabilizing treatment at 39 °C for 1 hour appears to have different trends. This is particularly so when the

Table 2. Degree of Stabilization as a Function of Time at the Aging Temperature.
(a) undeformed
(b) deformed 2% prior to transformation.

	Austenitizing Temperature	Quenching	M _s	% Martensite	Arrest Temp. T _A	Stabilizing Time	M' _s	θ=M _s -T _A
a) undeformed	1100 °C for 1hr	Water	10.15	33.1	-31.5	5 min.	-45	13.5
	"	"	9.25	32.5	-36.5	20 min.	-54	17.5
	"	"	-3.5	32.4	-29.5	1 hr.	-49.7	20.2
	"	"	2.5	32.9	-29	4 hrs.	-45.5	16.5
b) deformed	"	"	-35.5	35.6	-41	3 min.	-54	13
	"	"	-31.5	34.4	-43	10 min.	-60.5	17.5
	"	"	-34	36.5	-41	30 min.	-63	22
	"	"	-37	38.2	-38	60 min.	-65	27

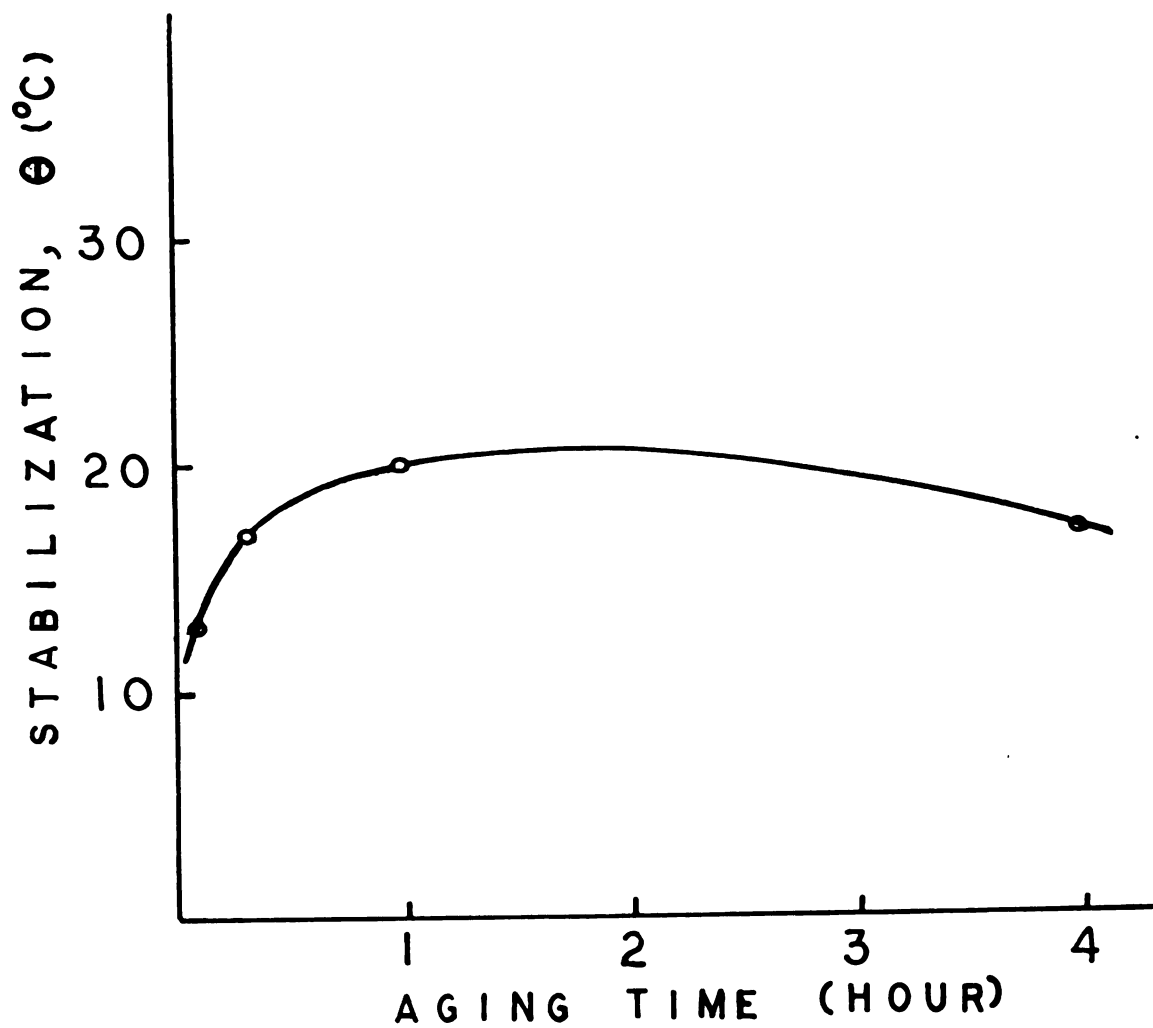


Figure 9-a Degree of Stabilization as a Function of Stabilizing Time at 39°C Containing about 30% prior formed Martensite without prior Deformation

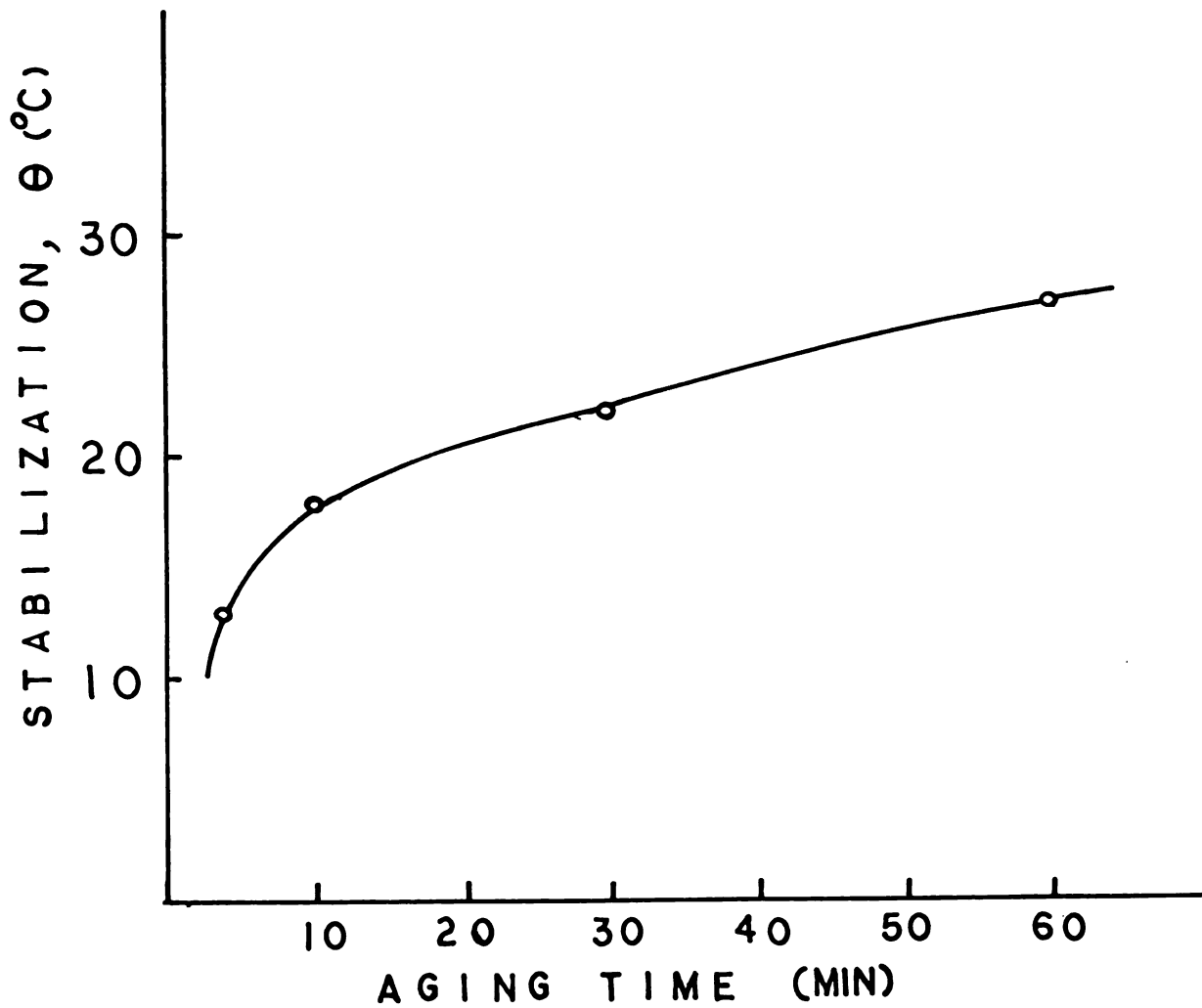


Figure 9-b Degree of Stabilization as a Function of Stabilizing Time at 39°C Containing about 30% prior formed Martensite with 2% prior Deformation.

extent of deformation was rather small such as 0.5%. In case of specimens containing 35% martensite the degree of stabilization increases with 0.5% deformation and with further increase in deformation consistently decreases up to 4% deformation. On the other hand in case of specimens containing 50% martensite the degree of stabilization decreases with increase in deformation up to 2% and then slightly increases when extent of deformation was 4%. The trend of alteration in the degree of stabilization having variation in the extent of deformation followed by different amount of martensite formation has been given in Table 3 and Figure 10.

5-5 Effect of Deformation of Partially Transformed Samples on Stabilization

Specimens containing 30% martensite, formed by sub-cooling, were deformed at room temperature by tensile elongation of 0.5 and 1% and subsequently stabilized at 39 °C for 1 hour. The extent of deformation could not be increased as the specimens failed in brittle manner beyond about 1% strain. When compared as a function of increasing strain, the degree of stabilization increases linearly with deformation (strain) as shown in Figure 11. The treatment followed is shown in the schematic diagram (Figure 5-e).

Table 3. Variation in the Degree of Stabilization as a Function of the Extent of Deformation prior to Formation of Martensite (a) 35% Martensite, (b) 50% Martensite

	Austenitizing Temperature	Quenching	% Deformation	% Martensite	M _S	T _A	M' _S	Θ=M _S -T _A
a) 35% Martensite	1100 °C for 1hr.	Water	0	39.2	-9.5	-53	-75.2	22.2
			.5	38.6	-31.5	-34	-64.5	30.5
			1	32.7	-33.5	-36	-61	25
			2	38.2	-37	-38	-65	27
			4	32.4	-37	-43.5	-64	20.5
b) 50% Martensite	1100 °C for 1hr	Water	0	50.7	11.25	-58	-92	34
			.5	49.7	-14.5	-53	-79.5	26.5
			1	51.6	-18.7	-56	-79.7	23.7
			2	51.4	-23.7	-61	-82.5	21.5
			4	52.8	-25	-60.5	-84.5	24

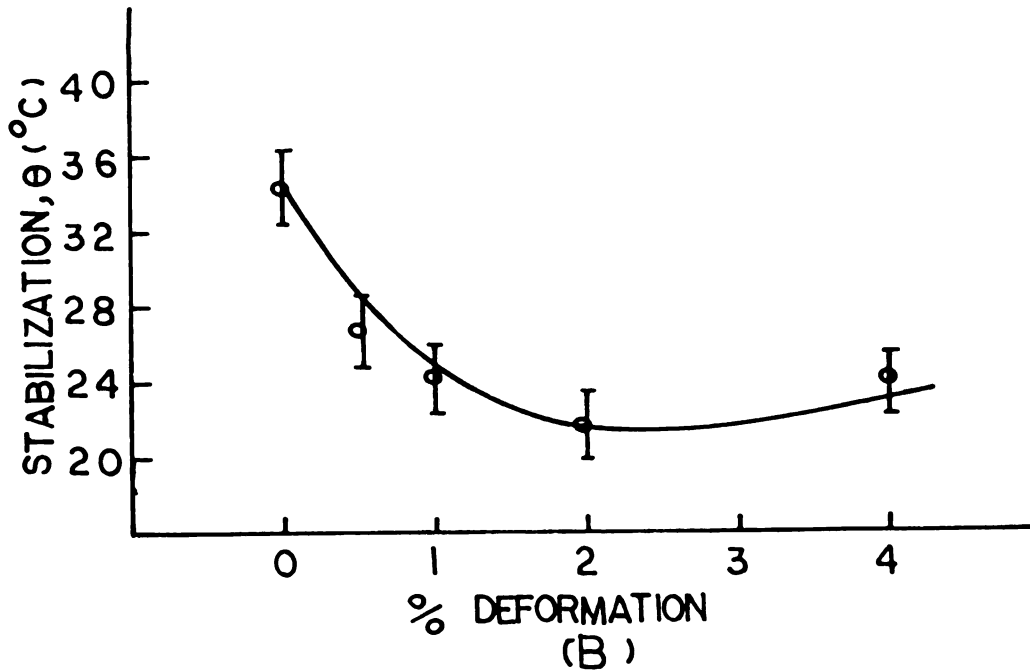
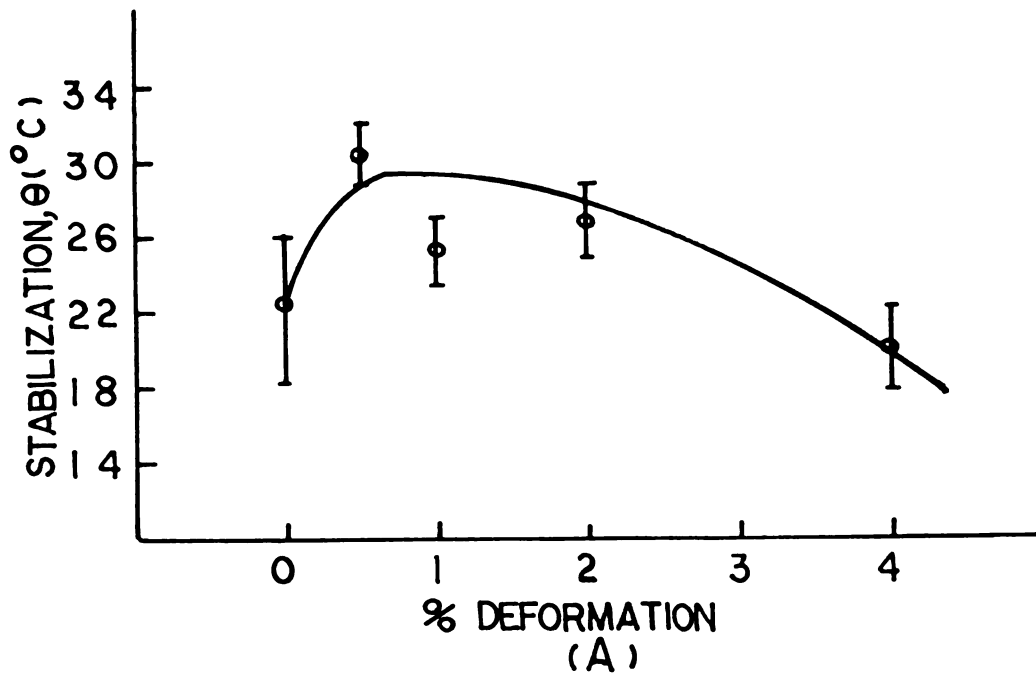


Figure 10. Variation of Extent of Thermal Stabilization with Varying Amount of Deformation for 1 Hour Aging Treatment at 39°C . Specimens are First Deformed, then Cooled to Produce A) 35% and B) 50% Martensite and up Quenched to the Aging Temperature of 39°C .

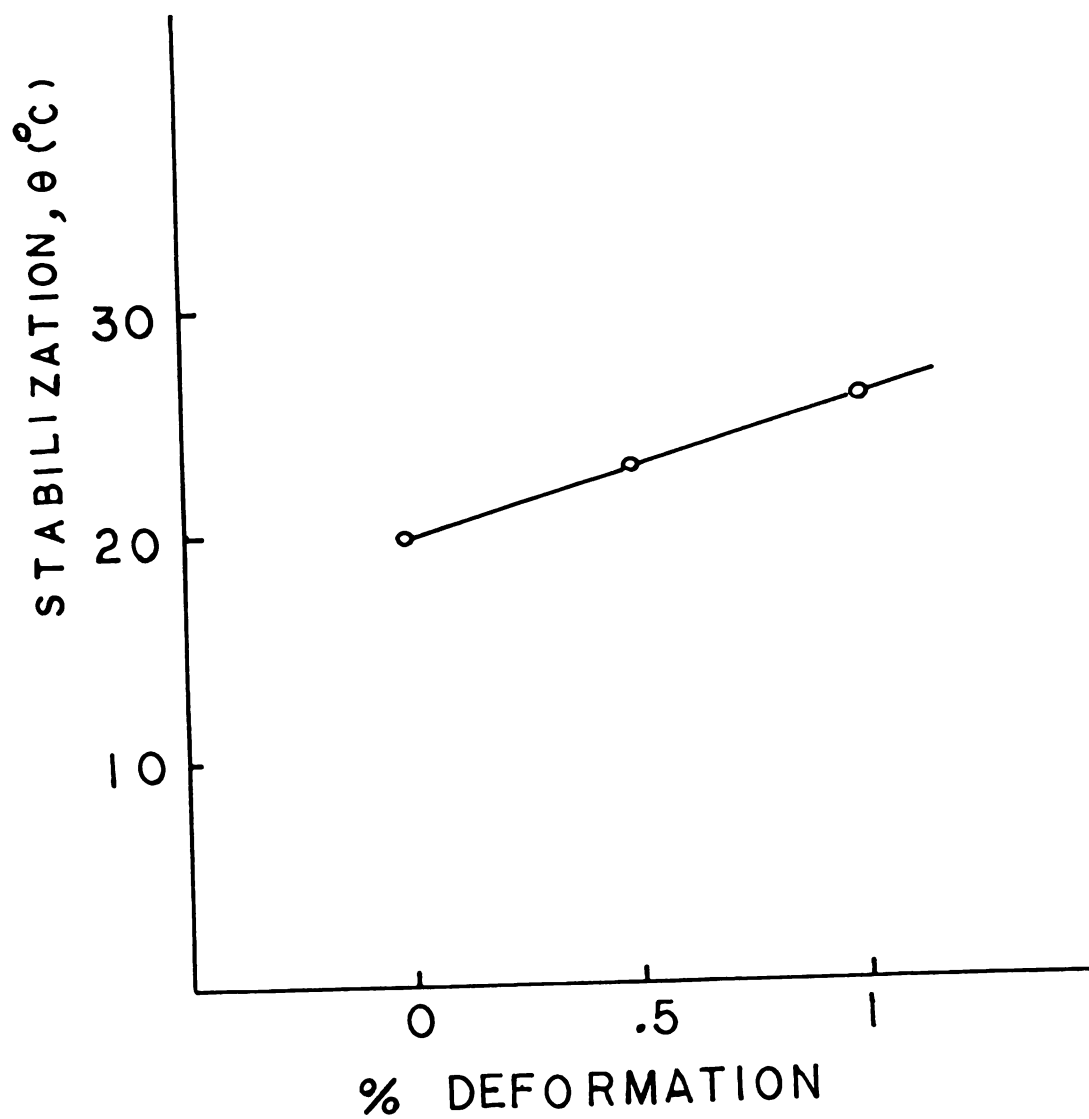


Figure 11. Variation of the Extent of Stabilization in Specimens Deformed with 30% prior Martensite.

5-6 Effect of Deformation on Microstructure

Optical photomicrographs of specimens transformed to ~30% with 0.5% strain and without prior strain have been presented in Figure 12. Prestrained specimen show an effect of bunching or clustering of martensite plates. The plates are thinner and shorter in the strained situation. It may be referred that earlier Durlu and Christian⁴⁶ showed that a decrease of the semi-thickness-to-radius (c/r) ratio of martensite plates resulted in prior plastically strained Fe 26.4 Ni 0.24C alloy. Work of Datta and Raghavan⁴⁷ further indicated that martensite plates become progressively thinner with increasing prestrain. When optical micrographs of specimens deformed after formation of martensite (~30%) and then retransformed after stabilization at 39°C is compared with the specimens, similarly treated but undeformed, some noticeable change in this microstructure may also be confirmed. The deformed specimen retransformed with formation of extremely fine plates within the residual austenite space (Figure 13). This effect on the shape and size of the martensite formed after stabilizing treatment is clearly the result of deformation. Martensite formed in strained lattice is much smaller and thinner.

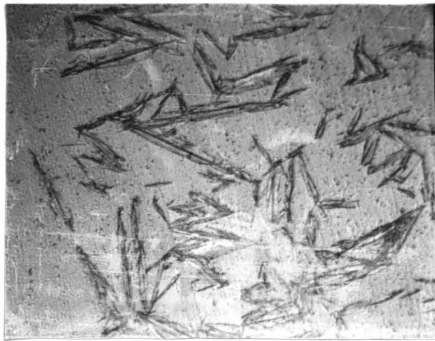


Photo 12-a Microstructure of Specimens
Containing 30% Martensite (X400)
Etchant - 4% Nital

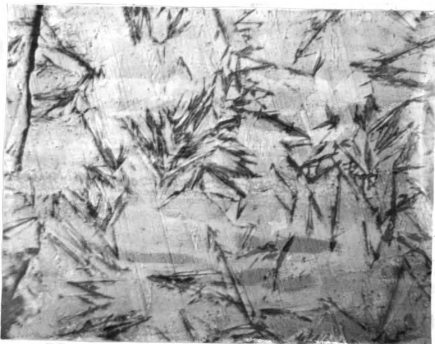


Photo 12-b Microstructure of Specimens
Containing 30% Martensite with
0.5% prior Deformation (X400)
Etchant - 4% Nital



Photo 13-a Microstructure of Stabilized
and Retransformed Specimen Showing
Effect of 0.5% Deformation following
30% Martensite Formation (Total
Martensite 79%) (X400)
Etchant - 4% Nital



Photo 13-b Microstructure of Stabilized
and Retransformed Specimen following
30% Martensite Formation (Total
Martensite - 56%) (X400)
Etchant - 4% Nital

DISCUSSION

Plastic deformation prior to martensite formation causes substantial depression of M_S -point in the alloy ($Fe - 16N_1 - 1C$) under study and the extent of depression consistently increases with increasing deformation within the present experimental range of 0.5% to 4% strain. Martensite transformation in deformed specimens is initiated by a large burst. The size of a burst is observed to increase with increasing deformation. Depression of M_S -point is also found sensitive to rate of straining. A slower strain rate, for a constant amount of deformation, produces a greater depression of the M_S -point. Such a depression of M_S -point, in plastically deformed specimens, is not usually experienced. Normally small plastic deformation of the austenite is expected to raise the M_S temperature. However, we believe that this observed depression of M_S is related to high carbon content of the alloy as will be discussed.

When austenite is plastically deformed above the M_d temperature, dislocations are introduced and thereby regions of high internal stress are created. This situation would cause segregation of C-atoms to the region of dislocation arrays comprising the potential embryos which would have otherwise raised the M_S if the interstitial content (C, N_2 etc.) was zero or very low. Besides segregation of C-atoms to the dislocations, introduced by deformation, in a high carbon alloy, carbon segregation

can also occur at preexisting embryonic sites. The latter will imply a depression of M_S in the absence of plastic deformation. Both of these mechanisms are operative in a plastically deformed high-carbon alloy. The decoration of dislocations increase the matrix strength and thereby introduces a kinetic barrier to the martensite interface motion. The potency of martensite embryos on the other hand is decreased by carbon diffusion during aging above M_S . These factors increase the chemical driving force necessary for transformation and thus M_S is depressed. Relatively speaking, if the strain aging due to deformation (i.e. strengthening of matrix) has a greater effect on the M_S , then strain rate will have a noticeable effect. In fact the strain-rate sensitive nature of the depression of M_S -point, for a fixed amount of prior deformation (2%), leads to such a conclusion in the present case. Slower rate of straining produces larger depression of M_S -point.

The burst size during martensite formation is a function of grain size and the chemical driving force at M_b ³⁴. The grain size remaining constant, larger burst with increased depression of M_S -point is quite in order and our experimental result are, therefore, consistent.

The degree of stabilization, θ , in partially transformed specimens, with prior plastic strain of varying amounts, seems to have somewhat different trends when the amount of transform-

ation is varied. Two different amounts of martensite have been studied, viz (1) ~ 50% and (2) ~ 35%. For stabilization treatment conducted after ~ 50% martensite formation, the degree of stabilization, θ , has been found to decrease monotonically with the amount of prior strain upto the uppermost strain employed in the present study (~ 4%). However, if the stabilization treatment is given after forming ~ 35% martensite formation, θ is found to increase first (upto ~ 0.5% strain) then decrease monotonically (beyond 0.5% strain upto 4% strain).

It has been suggested that internal stress due to plastic deformation or partial transformation can aid martensite formation by a process of stimulation of the existing embryos.^{48,49}

On the other hand, plastic deformation results in strain hardening of austenite. Dislocation accumulation become insurmountable obstacles for the propagation of the martensite plates. The strain-hardening of austenite is expected to depress the M_s -temperature. A decrease in M_s -temperature is expected to take place with increasing yield strength of austenite at M_s -temperature. A comparison of the energy necessary to deform surrounding austenite (in order to accommodate transformational volume and shear strains) with chemical driving force at M_s in a series of Fe-Ni-Cr-C alloy demonstrated that the resistance to plastic deformation represents a substantial portion of

non-chemical free energy for this alloy series.⁵⁰ Earlier work⁴⁶ on Fe 26.4Ni 0.24C and Fe 24Ni 0.45C has shown that M_b temperature first rises with deformation and then falls with higher deformation. If interstitials such as carbon etc. are present then an additional effect of hardening due to strain aging must be considered as has been pointed out earlier.

Strain-aging effect appears to be dominating in this alloy even when the amount of strain is rather low (0.5%). This is evident in the nature of plot showing the effect of depression of M_s -point as a function of increasing strain (Fig. 7).

It is now necessary to inquire why the stabilization increases when the amount of prior deformation is small ($\sim 0.5\%$) and the stabilization is conducted after a small volume % transformation (e.g. $\sim 35\%$). The chemical driving force, Δg_c , for martensitic transformation can be defined as

$$\Delta g_c = \alpha (T_0 - M_s) \dots\dots\dots (20)$$

where T_0 is the thermodynamic equilibrium temperature and

α is a proportionality constant. If plastic deformation depresses the M_s temperature to a new temperature M'_s , then according to Fig. 14, the driving force at M'_s is larger ($\Delta g'_c > \Delta g_c$). First, the above model will suggest that the size of the burst is larger as the % strain increases, since in our present alloy M_s decreases (and hence $\Delta g_c^{M_s}$ increases) as %

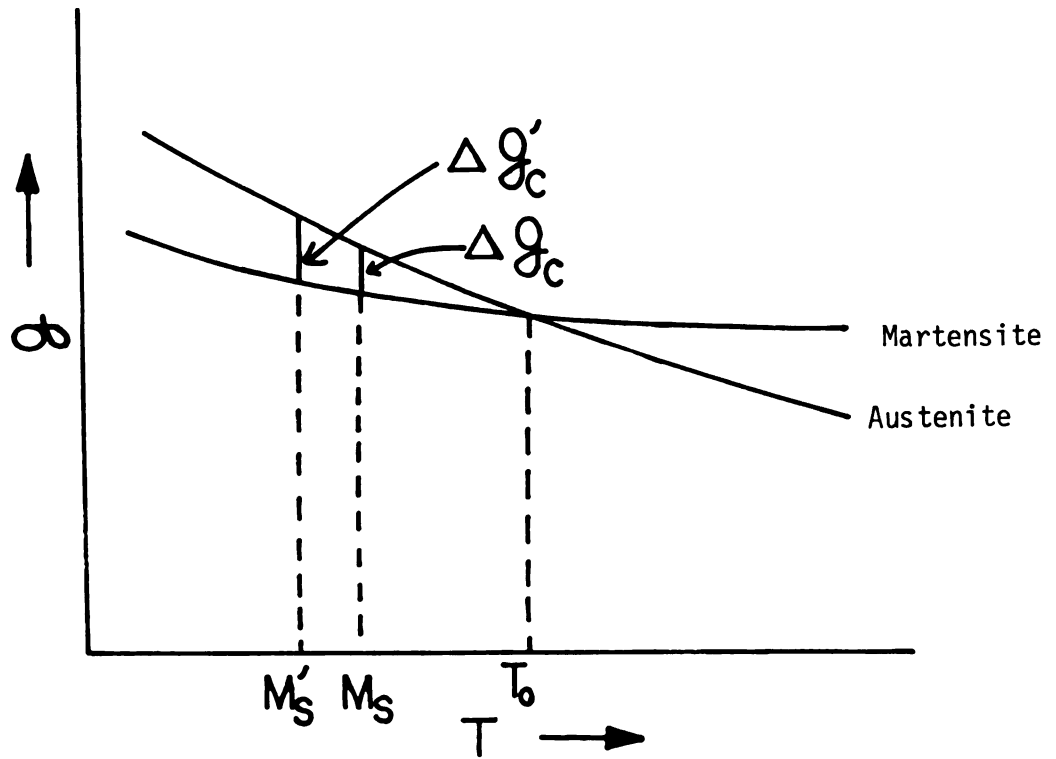


Figure 14. Schematic Presentation of g vs. Temperature

strain increases. Experimentally this is varified. Secondly, the supercooling below T_0 , necessary to produce 35% martensite, is less than that for 50% martensite. Thus:

$$\left. \begin{array}{l} (T)_{35\%M} > (T)_{50\%M} \\ \text{and } (\Delta g_c)_{35\%M} < (\Delta g_c)_{50\%M} \end{array} \right\}$$

where $(T)_{35\%M}$ and $(T)_{50\%M}$ are temperatures at which 35% and 50% martensite respectively form. Thus for small deformation and small % of martensite the driving force is relatively small due to two factors:

- 1) Suppression of M_S is small.
- and 2) $(T_0 - M'_S)$ is small.

Therefore we may conclude that the small increment of driving force

$$\Delta(\Delta g_c) = \alpha (M'_S - M_S)$$

is not sufficient to overcome the strain-aging induced kinetic barriers as discussed earlier. If, however, the % transformation, after which stabilization is conducted, is large, then the sample must be cooled down to a lower temperature (e.g. $T_{50\%M}$) and hence the driving force is larger. If this driving force is large enough to overcome kinetic barriers due to strain aging, then stabilization will decrease. As the % strain increases, $(T_0 - M'_S)$ increases and thus the stabilization decreases again. This tentative model seems to explain the observed variation of stabilization with % transformation as

well as % plastic strain.

Our last result concerns the effect of deformation after partial transformation. When a partially transformed sample (~30% martensite) is deformed, the degree of stabilization increases linearly with increasing strain up to 1% (Fig. 11). Unfortunately, such an experiment could not be conducted at larger strains because of the brittle manner of samples after giving 1% strain. Since martensite was formed prior to deformation, the effect of deformation is highly localized in the untransformed austenite volume. Under this circumstance the result obtained can be easily explained by assuming, strain aging and strengthening of the remaining austenite during stabilization aging at 39°C. This can be viewed as if the M_s temperature of the remaining austenite has been depressed by strain aging and further transformation can only occur at a lower temperature than the original arrest temperature.

It has been shown in section 5-6 that the martensite formed, in the sample deformed after 30% martensite, is smaller and deformed after 30% martensite, is smaller and thinner than the previous martensite plates. This result can now be explained in terms of kinetic barrier for interphase motion as discussed above.

One further evidence that C diffusion is responsible for the various stabilization effects, is the dependence of

stabilization on the aging time. In deformed and partially transformed specimens, the extent of stabilization varies with time of aging at 39 °C increasing to a maximum value and then decreasing at longer aging times. This result confirms earlier work^{28,29} on plain C-steels and Fe-Ni alloys without any deformation. The degree of stabilization at relatively longer time of aging has been considered analogous to overaging in solid state precipitation.

7. CONCLUSIONS

(1) Plastic deformation prior to transformation of austenite causes depression of M_s -point in a Fe 16Ni 1C steel.

(2) Depression of M_s -point caused by prior plastic deformation increases with the extent of deformation. The increase in the depression of M_s -point, however, is comparatively small with higher deformation in the range of deformation studied.

(3) Depression of M_s -point is found to be sensitive to rate of straining. Slower strain rate for a constant deformation produces more depression of M_s .

(4) Martensite formation is found to start always with burst of a reasonable size. The size of burst following prior deformation is larger with higher extent of deformation and consequently lower temperature of martensite formation.

(5) Number of bursts in undeformed specimens is observed to be more than those for deformed specimens. For the latter a single large burst is the most common mode of transformation.

(6) In deformed specimens, after forming 50% prior martensite, the degree of stabilization, θ , decreases consistently with the amount of prestrain up to about 2% and then slightly increases with further strain up to the upper limit of strain employed in the present study ($\sim 4\%$).

(7) In deformed specimens, after forming about 35% martensite, increases for small prestrain (0.5%) but decreases monotonically (beyond 0.5% strain) up to 4% strain.

(8) When a partially transformed sample (~30% martensite) is deformed the degree of stabilization increases linearly with increasing strain up to 1%. Unfortunately such an experiment could not be conducted at larger strains because of the brittle failure of samples beyond 1% strain.

(9) In deformed and partially transformed specimens, the extent of stabilization varies with time of isothermal aging at 39°C, increasing to a maximum value and then decreasing at longer aging time.

APPENDIX

Procedure for obtaining a specific amount of martensite during resistance measurement.

1) Initially resistance data from RT to liquid N_2 temperature is obtained starting from austenitic condition. After maximum transformation (cooled to liquid N_2) change of resistance on heating is also to be noted.

On the basis of above the following are calculated:

Average resistance drop per $1^\circ C$ in austenite = R_γ

Average resistance drop per $1^\circ C$ in martensite = R_α

Resistance drop for 1% martensite formation = R_M

Maximum amount of martensitic is found (based on X-ray diffraction) as 80%. Then, $R_M = \frac{R_{M_S} - R_{195} - R_\alpha (M_S - 195)}{80}$

where R_{M_S} is the resistance at M_S and R_{195} at $-195^\circ C$. For avoiding effect of size difference in specimens all the values are divided by R_{M_S} and the ratio R_γ/R_{M_S} , R_α/R_{M_S} and R_M/R_{M_S} obtained. It is expected that these ratios would not be altered much from specimen to specimen.

2) For stabilization experiments the steps followed are:

(A) Resistance is measured up to the transformation point and average value of R_γ is determined.

(B) Taking the resistances before and after transformation as

R_B and R_{A_1} and corresponding temperature as T_B and T_{A_1} .

Resistance at M_S , R_{M_S} may be calculated as $R_{M_S} = R_B - R_\gamma(T_B - M_S)$

(C) % martensite at T_{A_1} is then calculated as follows:

$$\% \text{ martensite} = \frac{1 - R_{A_1}/R_{M_S} - R_\gamma/R_{M_S} (M_S - T_{A_1})}{R_M/R_{M_S}} = X_1$$

(D) Amount of transformation on following lower temperature

(T_{A_2}) is then calculated as

% martensite at $T_{A_2} = X_2 =$

$$\frac{R_{A_1}/R_{M_S} - R_{A_2}/R_{M_S} - \left[R_\gamma/R_{M_S} \left(\frac{X_1}{100} \right) + R_\alpha/R_{M_S} \left(1 - \frac{X_1}{100} \right) \right] (T_{A_2} - T_{A_1})}{R_M/R_{M_S}} + X_1$$

(E) Amount of transformation on successive lowering of temperature is calculated accordingly.

REFERENCES

1. R.F. Bunshah and R.F. Mehl, Trans. AIME, 1251 (1953).
2. K. Mukherjee, Trans, AIME, 242, 1495 (1968).
3. M. Cohen, Trans. AIME, 212, 171 (1958).
4. L. Kaufman and M. Cohen, Prog. Metals, Phys., 7, 165 (1958).
5. M. Cohen, E.S. Machlin and V.J. Paranjpe, Thermodynamics in Physical Metallurgy, Amer. Sco. of Metals, 242 (1949)
6. F.C. Frank, Acta Met., 1, 15 (1952).
7. H. Knapp and U. Dehlinger, Acta Met., 4, 289 (1955).
8. C.B. Olson and M. Cohen, Metall. Trans. 7A, 1897 (1976); 1905 (1976); 1915 (1976).
9. C. L. Magee, Phase Transofmrations, ASM 115, (1970).
10. K.E. Easterling and A.R. Thölen, Acta Metall. 24, 333 (1976)/
11. M. Suezawa and H.E. Cook, Acta. Metal. 28, 423 (1980).
12. B.L. Averbach, M. Cohen, and S.G. Fletcher, Trans, ASM 40, 728 (1948)
13. B.L. Averbach and M. Cohen, Trans. ASM 41, 1024 (1949)
14. G.V. Kurdjumov and O.P. Maksimova, Doklady Akad, Nauk, SSSR 61, 83 (1948).
15. G.V. Kurdjumov and O.P. Maksimova, Doklady Akad. Nauk, SSSR 73, 95 (1950)
16. S.C. Das Gupta and B.S. Lement, Trans. AIME, 727 (1951).
17. E. S. Machlin and Morris Cohen, Trans. AIME, 489 (1952).
18. S.A. Kulin and G.R. Speich, Trans. AIME, 258 (1952).
19. R.E. Cech and J.H. Hollomon, Trans. AIME, 685 (1953).

20. C.H. Shih, B.A. Averbach, and M. Cohen, Trans. AIME, 1265 (1955).
21. K. Wakasa and C.M. Wayman, Metallurgy 14, 37 (1981).
22. W.J. Harris and M. Cohen, Trans. AIME 180, 447 (1949).
23. E.S. Machlin and M. Cohen, Trans. AIME 191, 746 (1951).
24. S.C. Das Gupta and B.S. Lement, Trans. AIME 530 (1953).
25. E.R. Morgan and T. Ko, Acta Metall. Vol. 1, 36 (1953).
26. B. Edmondson, Acta Metall. Vol. 5, 208 (1957).
27. J. Woodilla, P.G. Winchell and M. Cohen, Trans. AIME 215, 849 (1959).
28. K.R. Kinsman and J.C. Shyne, Acta Metall. Vol. 14, 1063 (1966).
29. K.R. Kinsman and J.C. Shyne, Acta Metall. Vol. 15, 1527 (1967).
30. S. Jin and D. Huang, Metall. Trans. A, Vol. 7A, 745 (1976).
31. S.G. Glover and T.B. Smith, Institute of Metals Monograph No. 18, 265 (1955).
32. O.P. Maksimova et al., Dokl Acad. Nauk SSSR 134, 871 (1960).
33. G.V. Kurdjumov, J. Met., 449 (1959).
34. A.R. Entwisle and J.A. Feeney, Inst. of Metals, Spec. Rep. No. 33, 156 (1969).
35. H. Suzuki and T. Honma, J. Jpn Inst. Met. 21, 263 (1957).
36. E.S. Machlin and M. Cohen, Trans. AIME, 191, 746 (1951).
37. R.E. Cech and D. Turnbull, Trans. AIME 206, 124 (1956).
38. J. Philibert and C. Crussard, Rev. Metall. 53, 973 (1956).
39. J.R. Patel and M. Cohen, Acta. Met., Vol. 1, 531 (1953).
40. G.V. Kurdjumov, J. Iron Steel Inst., 195 (1960).

41. E. M. Breinan and G.S. Ansell, Met. Trans., Vol. 1, 1513 (1970).
42. A. Ankara, J. Iron Steel Inst., Vol. 208, 819 (1970).
43. L.E. Pope, Metall. Trans., Vol. 3, 2151 (1972).
44. J.R.C. Guimaraes and J.C. Shyne, Scripta Metall., Vol. 4, 1019 (1970).
45. P.C. Maxwell, A. Goldberg and J.C. Shyne, Metall. Trans., Vol. 5, 1305 (1974).
46. T.N. Durlu and J.N. Christian, Proc. of Conference on Mart. Transformation, 343 (1979).
47. R. Datta and V. Raghavan, Scripta Met. 615 (1983).
48. G.V. Kurdjumov, JISI, 28 (1960).
49. M. Tokizane, Scripta Met. 10, 459 (1976).
50. R. Pradhan and G.S. Ansell, Met. Trans. A, Vol. 9, 793 (1978).

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