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A DILATOMETRIC STUDY OF THE MARTENSITE TRANSFORMATION AND MARAGING BEHAVIOR OF A Ni - Co - Mo STEEL

BY

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A

THESIS

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Approved by

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ABSTRACT

An experimental investigation of the austenite to martensite transformation and the maraging behavior taking place in a nickel-cobalt-molybdenum steel was conducted for this project.

The austenite to martensite transformation was followed by the dilatometric method, being done on a Leitz Optical Dilatometer. An apparatus was constructed for the dilatometer in order to record dimensional changes at low temperatures, thus broadening the possible applications for the instrument.

The maraging behavior was investigated by means of metallographic analysis and hardness determinations.

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INTRODUCTION

The decomposition of austenite to its various products has been studied by many investigators. The product of the austenite transformation depends on two principal factors: temperature and time. The austenite to martensite transformation in plain carbon and low-alloy steels is commonly the result of rapid cooling rates from the austenite range, but the martensite may form subsequent to air cooling in highly alloyed steels such as the high-nickel "maraging" steels.

The material studied in this work is a highly-alloyed steel containing approximately 19% nickel, 8% cobalt, 5% molybdenum, and 0.02% carbon. This material was developed by the International Nickel Company late in 1959 as one of a series of high-nickel steels. This steel transforms from austenite to martensite on air cooling and is strengthened by a precipitation-hardening mechanism. The steels are termed "mar-aging" steels, because the precipitation reaction which accounts for their ultrahigh strength occurs on aging them when they are in the martensitic condition. These steels can be heat-treated to yield strengths of 250 to 300 ksi with reportedly excellent toughness in the presence of notches or cracks.

The subject of this thesis is to investigate the austenite to martensite transformation and the subsequent maraging behavior by means of dilatometric and metallographic methods. The work includes the development of an apparatus for dilatometric investigations below atmospheric temperatures to supplement those above atmospheric temperatures. This development was done in an effort to broaden the temperature range over which thermal analysis of materials can be performed on the Leitz dilatometer, and to alleviate the necessity of a quenching process.

The work may be divided into two major parts:

1) the martensite transformation
2) the maraging behavior,

each being treated separately, in detail.

CHAPTER II

REVIEW OF THE LITERATURE

PART I -- THE MARTENSITE REACTION

Solid state phase transformations in the various binary and complex alloy systems have been the subject of study by many investigators in the fields of metallurgy, ceramics and physics. One such transformation, a common but very important one, is the martensite transformation.

THE MARTENSITE TRANSFORMATION:-

The word martensite was originally adopted by metallurgists to define the plate-like or acicular structure in quenched steel that is responsible for its outstanding hardness.[1] However, it is now known that the particular type of phase transformation, which produces the hardening of steel, also occurs in many other alloy systems but is not necessarily attended by such hardening. In fact, even in steels, the martensite is not especially hard, if the carbon content is low. Thus high hardness is no longer regarded as an essential characteristic of martensite, although this property first called attention to the far-reaching importance of the constituent in the practical heat treatment of steel. The term martensite has now taken on the broader significance of serving to classify all transformation products that result from a certain kind of solid-state reaction, irrespective of the composition, structure, or properties of the product. This expanded usage of the term does further honor to the pioneering metallurgist, Professor A. Martens, from whose name the word martensite is derived.

In 1949 Jones and Pumphrey[2] made a study of the transformations which occur in iron-rich binary iron-nickel and iron manganese alloys. The progress of the transformations on continuous heating and cooling was followed dilatometrically. The dilatometer trace of an iron-nickel alloy is shown in Figure 1, evidential of the hysteresis effect between the transformations on heating and cooling. They reported that the energy of the lattice strain arising from the transformations appears to be insufficient to explain the observed hysteresis. For the 14.6 atomic percent nickel-iron alloy, dilatometer traces were made using heating and cooling rates of 2°C/minute and 150°C/minute. The traces are essentially the same, the slight differences being due to temperature differences between components during the rapid heating and cooling rates. This similarity of the curves over this wide range of rates of change of temperature indicates that, under these conditions, the transformations are dependent on temperature rather than on time, which is in agreement with the hypothesis that they are diffusionless transformations.

Kaufman and Cohen[3] have also studied the phase equilibria in the iron-nickel system. The martensite-start temperature (M_s) on cooling and the austenite-start temperature (A_s) on heating in the iron-nickel system have been determined for nickel contents up to 33 atomic percent. The M_s-A_s diagram for the iron-nickel system is shown in

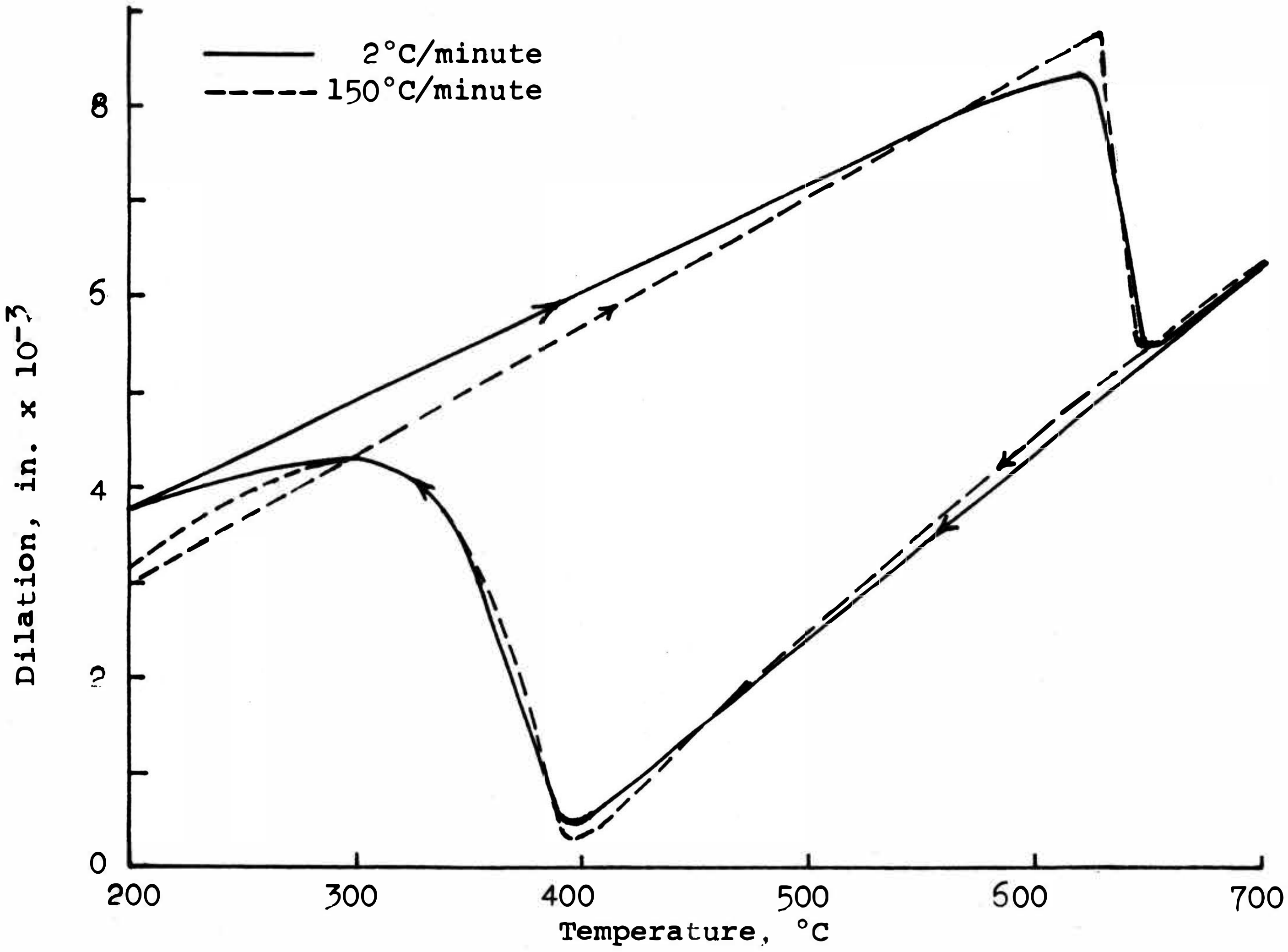


Fig. 1. Dilation curves of 14.6 atomic pct Ni-Fe alloy.

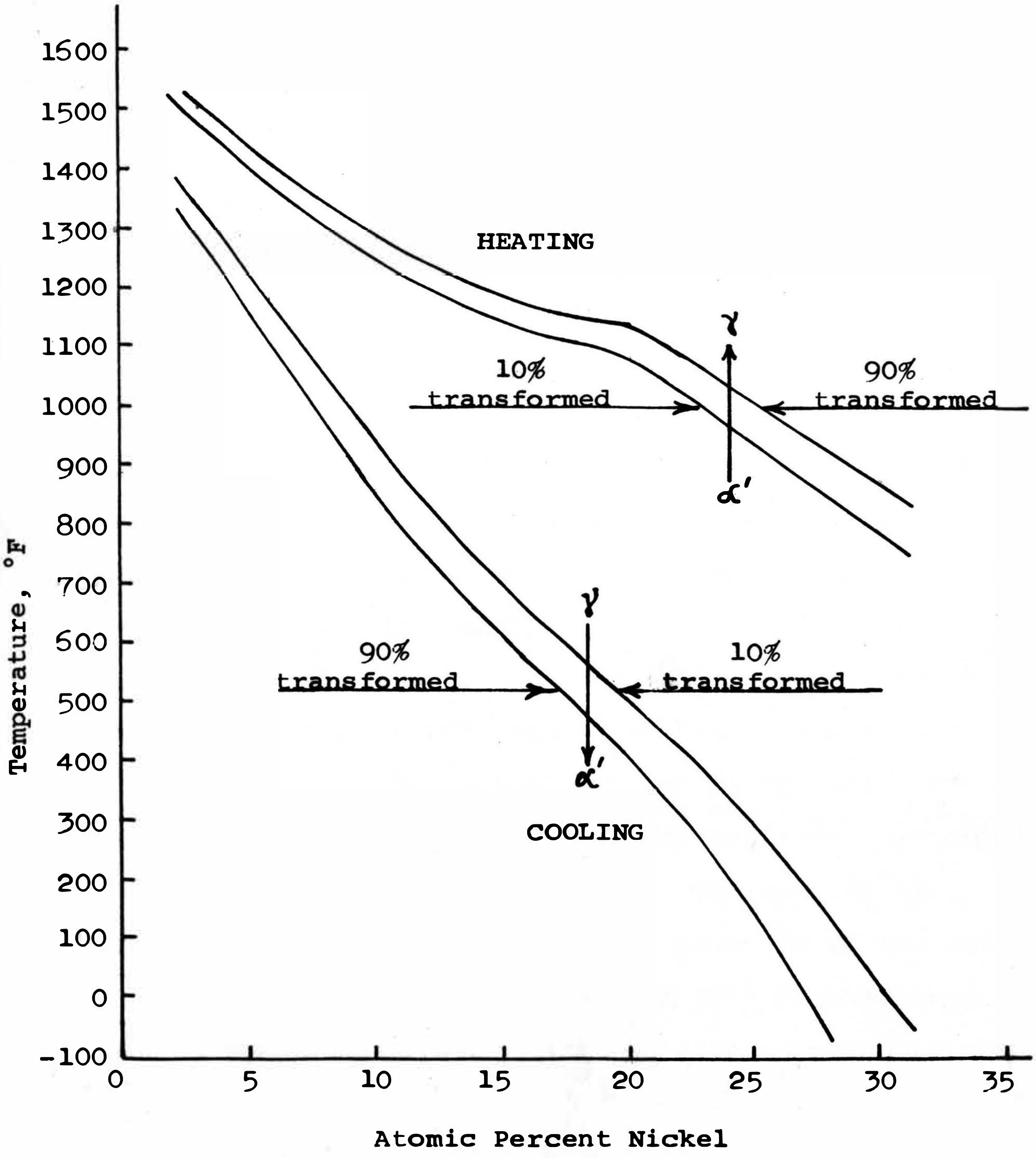


Fig. 2. The Fe-Ni transformation diagram.

Figure 2. The $\gamma \rightleftharpoons \alpha'$ transformations on cooling and heating indicated by the M_s and A_s lines occur without change in composition. The γ-phase is the face-centered cubic high temperature phase, the α-phase is the low temperature equilibrium body-centered cubic phase, and the α'-phase is the low temperature metastable body-centered cubic phase (martensite).

If an austenite specimen is cooled below the M_s and held isothermally, stabilization[4] of the remaining austenite occurs and appreciable undercooling below the holding temperature is required to restart the martensitic transformation. The reverse transformation is also stabilized by isothermal holding above A_s. Moreover, no observable isothermal reversal ($\alpha' \rightarrow \gamma$) takes place. Figure 3 shows the stabilization curve obtained for the alloy containing 29.7 atomic percent nickel.[3]

All metallographic evidence indicates that the cooling transformation does not proceed by the growth of existing plates of martensite, but by the sudden formation of new ones which spring full-born into existence as the temperature is lowered. It has been reported by Mehl[5] that the time of formation for a martensite plate is of the order of 10^{-4} seconds and as rapid as 2×10^{-7} seconds in a coarse-grained austenitic steel. A vivid demonstration of the discrete nature of these sudden movements is afforded by the "clicks" that are audible over a suitable accoustical pick-up as the individual shears send elastic impulses through the specimen. The amount of transformation occuring

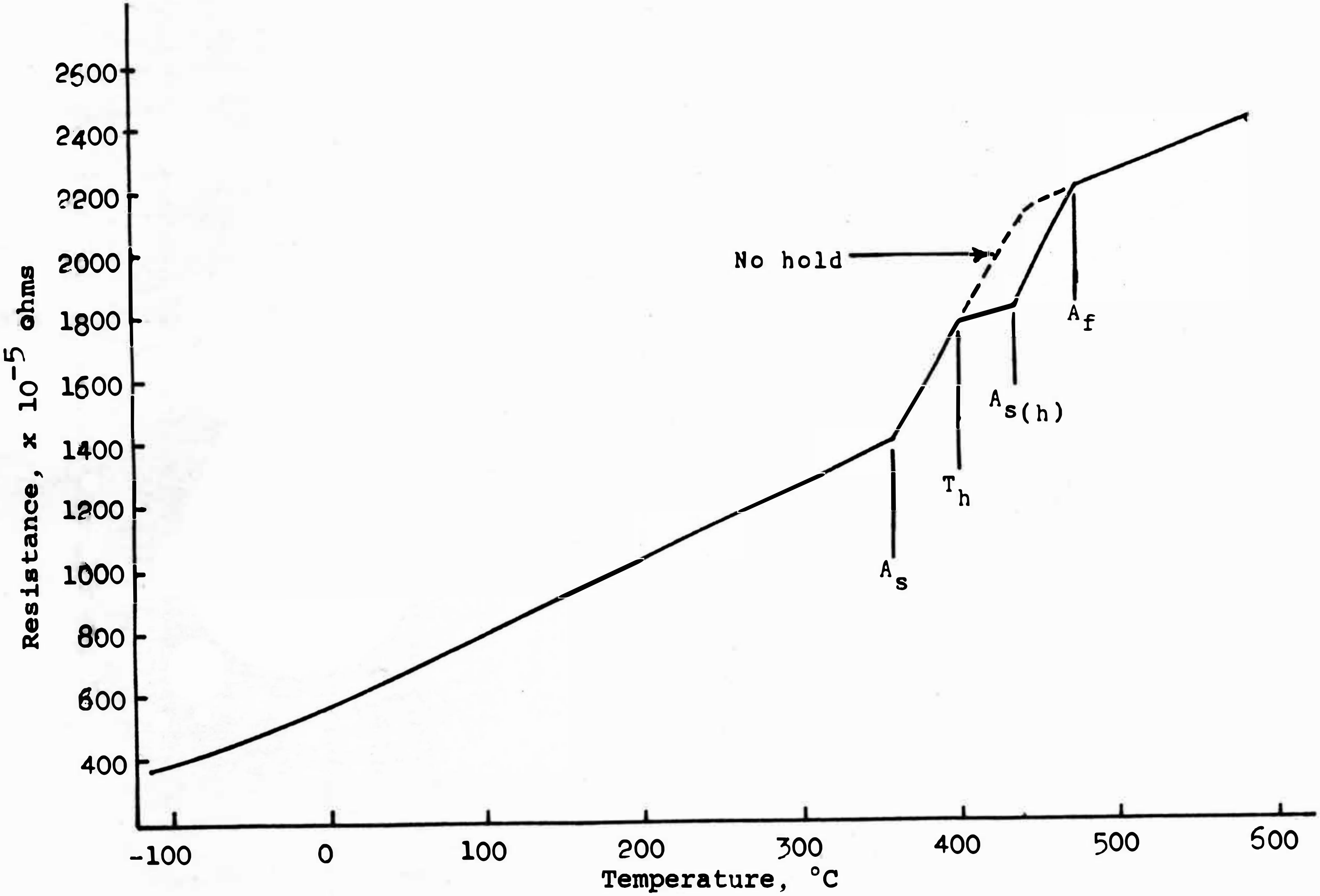


Fig. 3. Diagram for 29.7 atomic % Ni-Fe alloy; Stabilization hold 4 hours at 400°C.

in a single burst is very variable, but amounts up to 10% transformation in a single burst have been observed.[6] In these circumstances, an 80°C temperature rise was measured.

It has been demonstrated[7] that, at least in certain nickel steels, raising the austenitizing temperature above the point of complete carbide solution causes additional lowering of M_s, even though the austenite composition does not change further.

There is some evidence[8] that M_s also depends on the grain size of the parent phase, the tendency being for M_s to rise with increasing grain size. Although the exact relationships between M_s and grain size have not been established, the existing information is significant in demonstrating that large grains favor the formation of martensite by allowing the transformation to set in at a higher temperature during quenching. Unlike nucleation-and-growth reactions, which are generally stimulated by the presence of grain boundaries (fine grain size), these boundaries act as obstacles to shear transformations. Martensite plates never extend across grain boundaries, although they may "trigger" other plates with different orientations in adjacent grains.

CRYSTALLOGRAPHY OF MARTENSITE FORMATION:-

All martensites are solid solutions (except for the pure metals) and may form out of a disordered or ordered parent phase and, in the latter event, inherit the ordered arrangement, as in the Cu-Al alloys. In no case studied thus far (except perhaps for the pure metals) is the martensitic product an equilibrium structure. On isothermal aging it tends to decompose into the stable phase via nucleation and growth.

The lattice parallelisms between martensites and their parent phases are of the same general nature as exhibited by other solid-state transformations.[9] Each martensite plate is a single crystal whose lattice orientation is strictly determined by the lattice orientation of the parent phase. Usually there is a plane and direction in the martensite crystal that lies quite parallel to a somewhat similar plane and direction in the parent crystal. The physical plane of the martensite plate also has a definite orientation with respect to the parent phase. The crystallographic plane in the parent lattice, which lies parallel to the physical plane of the martensite plate, is called the habit plane and is of especial significance, because it defines the plane of the parent crystal along which the principal shear displacement occurs in the martensite reaction.

METHODS AND APPARATUS:-

Methods presently popular which afford the means of conducting studies of solid-state transformations are:

1) x-ray diffraction
2) electrical conductance
3) dilatometry
4) metallography

The dilatometer is an instrument for measuring the expansion or contraction in a material caused by changes in temperature or structure.[13]

The dilatometric method of investigation is particularly adaptable to materials which undergo a large volume change on transformation, such as the austenite to martensite transformation in steels.

In 1944 Fletcher and Cohen[10] successfully introduced low temperature dilatometry when a long range investigation of the dimensional stability of steel, particularly the subatmospheric transformation of retained austenite, was started at the Massachusetts Institute of Technology at the request of the Sheffield Foundation of Dayton, Ohio. The main direction of their work was toward the high carbon steels. The dilatometer used was of the dial gage type and accomodated a 4-inch long by 3/8-inch diameter specimen. The device was in an upright position and a cryostat was made and adapted to it, also in the vertical position, for the purpose of containing the liquid oxygen or nitrogen coolant.

PART II -- AGE HARDENING

Precipitation from supersaturated solid solution is one of the most important reactions in the solid state of metals. It is responsible for age-hardening, a commercially applied process for increasing strength and hardness or permanent magnetism of a large variety of alloys. Although the process originated in an aluminum-copper alloy, it has since led to the development of important alloys of all the common base metals from silver to zinc.

THEORIES OF AGE HARDENING:-

Many investigators have proposed various theories to explain the precipitation and age-hardening phenomena. "It is quite clear, however, that the precipitate does not have the normal structure when it first forms, that the atomic movements required to form the equilibrium structures may not be simple, and that the concept of a 'critical dispersion' for maximum hardness cannot be reconciled with the complexities of the process in view of the several contributing reactions that influence hardness."[11] The important distinction between the pre-precipitate concepts and the more recent knowledge of the precipitation reaction is founded in the structure of the "particle" when it first becomes distinct, chemically or structurally, from the matrix. The older concepts of "knots" by Merica,[13] and of "ordering" by Desch,[14] and more recently the concepts of "clusters" by Guinier,[15] of "order hardening" by Harker,[16] and "vacant lattice site migration" by Rohner[17] all proposed segregation of the solute in the matrix with no change in the structure but only slight changes in interplanar spacing induced by the segregation; thus the particle would be different only chemically. The only reliable data supporting any of these contentions up to 1951 appear to be those of Guinier and Preston,[18] but these have been reconciled to precipitation of a new phase where the first detectable particles must have the structure of the transition lattice.

Observations have been that a coherent precipitate is a greater hardening agent than the incoherent precipitate and that the number and size of particles alone are not the controlling factors of age-hardening. The difference in hardening capacity of a coherent precipitate and an incoherent precipitate probably originates in the distribution of strain in the matrix. With a coherent precipitate the strains extend into an appreciable volume of the surrounding matrix. On the other hand, the strains about an incoherent particle must be isolated in a very thin zone, since sharp diffraction patterns are obtained for both phases in a two-phase alloy in equilibrium. The strained matrix about a coherent precipitate particle would be expected to hinder slip both from the standpoint of the irregularities in atomic planes and of a possible increased atomic cohesion.[11] Thus the effective size of the volume that hinders slip would appear to be several times the size of the precipitate particle. In this way a coherent precipitate of the same dispersion as an incoherent precipitate would exhibit a greater probability of resisting slip. On the other hand, an incoherent dispersion of particles of a size comparable to the effective size of the coherent precipitate would require gross depletion of the solid solution which in itself is a softening process. For hardening by a coherent precipitate fewer particles and less depletion are required. A still smaller quantity of precipitate distributed along grain boundaries would be expected to provide hardening also.

THE WIDMANSTATTEN STRUCTURE:-

In recognition of the importance of crystallographic relationship between phases in solid-state reactions, Mehl and Barrett[19] published in 1931 the first of a series of papers on the Widmanstatten structure. A note from the first paper of the series describing this structure is of historical interest here:

> "First discovered in 1808 by Alois de Widmanstatten, Director of the Imperial Porcelain Works in Vienna. Widmanstatten polished and etched a surface upon a meteorite known as the Agram Iron and observed the structure since known in his honor as the Widmanstatten figure or structure. Widmanstatten attempted, apparently, no explanation of this structure, and in fact did not publish an account of his discovery, though it shortly became known amoung crystallographers and mineralogists."

Little did he know the important part it would play in modern metallurgy. The structure is produced when a new phase precipitates from solid solution. It also forms in the eutectoid reaction and when a metal undergoes an allotropic transformation.[11]

In the first[19] of a series of studies on some precipitation alloys by Mehl, the subject was the well-defined pattern of precipitate plates in Al-Ag alloys. When the number of different directions of the plate traces in several grains on a polished surface were counted, a maximum of four was found for each matrix grain. Mehl thought that since there are four (111) planes in the face-centered cubic matrix, the plates must be parallel to (111) matrix planes. On comparing the atomic array on (111) matrix planes with the (001) plane in hexagonal precipitate, a close similarity was noted both in position and in spacings, and thus was postulated the crystallographic relationship which was then confirmed by x-ray diffraction analysis.

In their first paper on the Widmanstatten structure, Mehl and Barrett advance a tentative hypothesis:

> "When a phase forming by precipitation from a solid solution takes the form of plates, the lattice plane on which the new phase forms is that upon which the atoms are nearly identical in position and interatomic distance to some plane in the precipitate lattice."[19]

This is now recogonized as one of the fundamental concepts of inter-phase relations of heterogeneous mixtures. Its more recent corollary concerns the formation of the coherent transition structure, for when the matching is nearly identical, the new phase grows continuous with the matrix to particles of detectable thickness. Geisler says that this implies that the coherent precipitate exhibits a lattice of the same type as that of the equilibrium phase but merely strained in dimensions so as to fit on the Widmanstatten plane of the matrix.

Cottrell[20] says that, in general, the condition for coherence requires the precipitate to adopt a metastable lattice or to strain its equilibrium lattice to fit that of the matrix and that in either case, the free energy of the precipitate is increased by a certain amount per atom which is essentially independent of the number of atoms in the precipitated particle. The size effect, so important in incoherent precipitation, has a minor significance in coherent precipitation and to a first approximation can be ignored. Cottrell goes on to state that the free energy relation between the parent solid solution and the coherent metastable precipitate is significantly greater than the free energy between the parent solid solution and the equilibrium precipitate, such that the solubility limit of the coherent precipitate in the parent solid solution is higher than that of the equilibrium precipitate.

The crystallographic features of the precipitate are specified by two pieces of information:

1) A statement of the orientation of the principal crystallographic axes in the precipitate relative to those of the matrix.

2) A statement of the habit of the precipitate, that is, the shape of the particle, and also, in the case of plate-shaped or needle-shaped particles, of the crystallographic plane or direction in the matrix which is parallel to the plane of the plate or the axis of the needle. In many, but not all, precipitates the particles are plate-shaped and the regular pattern of such plates on certain crystallographic planes of the matrix crystal gives rise to forms of the well-known Widmanstatten structure.

Well developed Widmanstatten patterns are observed when both the matrix and precipitate are cubic with similar lattice parameters. The plates generally are parallel to (100) planes of the matrix and thus a maximum of three trace directions is exhibited on the polished surface.[11] Such structures are observed in aged alloys of Cu-Ag,[21] Cu-Ni-Co,[22] and Cu-Ni-Fe,[23] in which the phases are face-centered cubic, and aged Fe-Ni-Al[23] alloys, in which the phases are both body-centered cubic. In all these alloys the precipitate lattice has the same orientation as the matrix crystal when coherency is lost, but presumably the various particles have one of the three orientations of the transitional tetragonal cell at the coherent transition state.

THE 18% NICKEL MARAGING STEELS:-

The findings[24] of the Research Laboratory of The International Nickel Company, Incorporated, for the 18% nickel maraging steels regarding the annealing behavior, martensitic transformation, the maraging behavior, and the maraging mechanism are now summarized and listed below:

1) As the annealing temperature is raised, the amount of austenite on cooling to room temperature decreases until only the body-centered cubic phase is present. The minimum annealing temperature required to eliminate all of the austenite is approximately 1350°F (732°C). Only with high annealing temperatures was the Widmanstatten morphology evident in the microstructures of the body-centered cubic phase.

2) Annealing at 1800°F produced an M_s of approximately 310°F with the M_f around 210°F.

3) The maraging behavior was followed by hardness determinations at increments of time of aging at temperature for different annealing temperatures. When annealed at 1500°F and air cooled, the material had a hardness of Rc 28, and when maraged at 900°F

for 3 hours, the hardness increased to a value of Rc 52. There were no significant dimensional changes during maraging.

4) X-ray and electron diffraction measurements showed a slight decrease in the lattice parameter of the body-centered cubic structure after maraging. Electron diffraction determinations indicated that ordering was taking place during maraging. It appeared that this ordering reaction yielded a bcc superlattice based on Fe_2MCo (where M could be Ni, Mo, or Co). Because of the similarity of the scattering factors of iron, nickel, and cobalt, however, the resolution of the superlattice structure was poor, and the findings were only tentative. Light microscopy, replica electron microscopy, and transmission electron microscopy failed to establish the presence of a precipitate.

CHAPTER III

PREPARATION OF THE STEEL

The steel was prepared by the Research Laboratory of the International Nickel Company, Incorporated, Bayonne, New Jersey, and sent from their Stock Number 04210.

<u>CHEMICAL ANALYSIS:-</u>

The chemical analysis of the steel as received from the supplier is as follows:

Carbon-------------0.026%

Manganese----------0.010%

Phosphorus---------0.008%

Sulfur-------------0.022%

Silicon------------0.064%

Aluminum-----------0.060%

Zirconium----------0.010%

Calcium------------0.010%

Boron--------------0.0039%

Molybdenum---------4.59%

Cobalt-------------7.87%

Nickel-------------18.72%

Iron---------------Balance

The steel was shipped in two pieces:

Bar: ½-inch diameter by 6 feet long

Wire: 1/8-inch diameter by 6 feet long

HOMOGENIZATION OF THE STEEL:-

"Homogenizing" is a process of heat treatment, at high temperatures (and usually for a long time), intended to eliminate or decrease chemical segregation by diffusion.

The homogenization should be carried out at temperatures well above the upper transformation temperature in order to effectively promote a complete diffusion of the alloying elements and to break up the structure which may exist in the steel as a result of hot forming.

All samples were homogenized in a furnace at 900°C (1652°F) for one hour. The homogenization was carried out in air, since the carbon content of the steel is so low so as not to pose a decarburization problem.

CHAPTER IV

THE MARTENSITE FORMATION RANGE

SAMPLE PREPARATION:-

The samples for the Leitz dilatometer were prepared from the 1/8-inch wire. The six-foot length of wire was cut into approximately 15-inch lengths and were then annealed in a furnace at 900°C for 30 minutes. Upon removal from the furnace, they were immediately swaged to 0.115 inch in order to straighten the pieces, making about three passes through the swaging dies. The pieces were then re-annealed at 900°C for one hour to remove the mechanical stresses induced by the swaging operation. Subsequent to this, small sections of approximately 50 mm in length were cut from the 15-inch lengths. The ends of the samples were then ground flat and the whole sample surface was rough polished on fine emery cloth to provide good contacting and frictionless surfaces. The sample is shown in Figure 4.

THE DILATOMETER:-

The dilatometric analysis employed the use of the Leitz Wetzlar dilatometer, model HTV. This is an autographic optical lever thermal-expansion apparatus. The dilatometer stand, the photographic camera, and the electric furnace are all mounted on one base. The dilatometer stand carried the so-called "dilatometer head" and the light source, a low voltage bulb (6 volts, 5 amps).

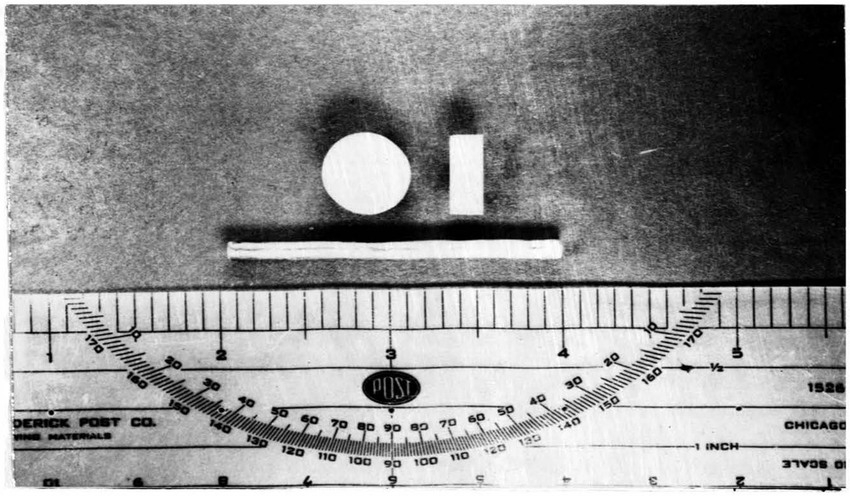


Fig. 4.

Specimens of the Ni - Co - Mo steel prepared for the dilatometric analysis, metallographic analysis, and hardness determinations.

The dilatometer head contains the mechanism for guiding the prism and the light-point, the device for mounting the specimens, and two adjusting screws for moving the light point on the projection screen in any way desired. The test rod of the metal to be examined and a standard body of known expansion (e.g., chronin, electrolytic copper, or pure aluminum) are inserted into the quartz tubes, the front ends of which are bowl-shaped and which are fastened to the adjusting head of the dilatometer. By means of special intermediate quartz rods the expansion of the specimens is transmitted to the recording system through which a prism supported by three points - the so-called measuring bridge - is actuated. The light beam from the lamp is led over this guiding prism, so that the light point, in accordance with the expansion or contraction of the specimen, is moved along the projection surface of the camera or records curves photographically.

The dilatometer is equipped with a slide-wire rheostate which is manually operated to control the temperature and the heating rate, an electric resistance furnace, book-type photographic paper holders, and a timing mechanism which replaces the book-type holder on the end of the camera box. The timing mechanism contains a rotating drum which holds the photographic paper and is actuated by a simple, hand-wind clock. The device permits three drum speeds of 50, 100, and 150 mm per hour on the drum surface, by means of interchangeable sets of driving cogs which slide on axles on the outside of the clock and are fastened by set screws.

Some advantages of the dilatometer are:

1) Since the measuring bridge is in constant operation, no illusionary effects will appear, and all phenomena occurring during the investigation may always be reproduced in exactly the same manner as they occur, provided that all requirements for a duplication of results prevail.

2) The specimen and the standard metal body are placed in the furnace horizontally and next to each other, to insure uniform heating and cooling of the two rods.

3) The registration occurs in a rectangular coordinate system, so that the curves are easily computed. The expansion of the material to be tested is reproduced directly as a function of the temperature (absolute curve).

CALIBRATION OF COORDINATE AXES:-

Since the dilatometer records changes in length as a function of temperature, the axes of the expansion curve must be calibrated. For calibration of the expansion (ordinate), a special optical micrometer calibrating device is substituted for the test specimen holding device. The motion of a micrometer screw is transmitted to the guiding bolt of the dilatometer, and it is read off from a graduated and illuminated glass scale in its absolute amount by means of a special microscope. By turning the micrometer spindle, it is thus possible to move the reflecting prism of the dilatometer head and the light point on the projection surface by predetermined intervals. At each interval the light is turned on to expose the sensitized paper, being sure to mark the origin. This method of calibration is reliable and simple.

For temperature calibration, the 50 mm standard electrolytic copper rod designed for the apparatus was inserted into the rear quartz tube which activates horizontal movements. In place of the quartz tube which holds the test specimen, an adjusting bolt is inserted for arresting the second moveable supporting point. An iron-constantan thermocouple is inserted into the cavity of the standard body and connected to a potentiometer, which is adjusted to compensate for room temperature. Then the standard body is heated slowly and the temperatures recorded from the balanced potentiometer readings. When a specified temperature is reached, it is kept constant for a short period, and an exposure is made by switching on the light. In this manner a number of point marks are obtained on the photographic paper for the computation of the curves. Exposures were made at 50°C intervals.

This same procedure was repeated for the subatmospheric temperature calibration, except that the calibration was made during both cooling and heating, and that the leads to the potentiometer were reversed in polarity.

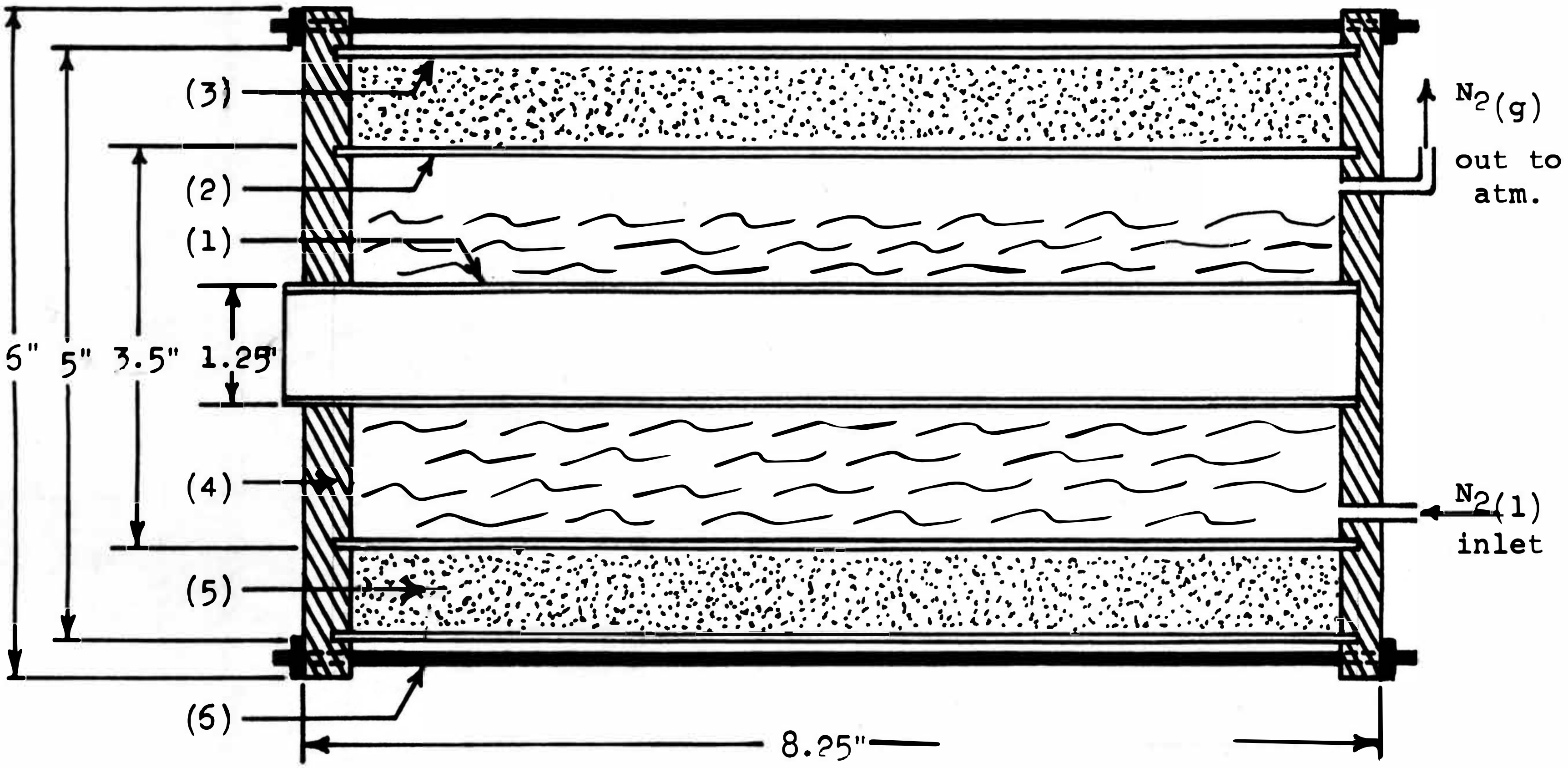
When both the ordinate and the abscissa were calibrated, the coordinate net was drawn according to the calibration points obtained.

DESIGN AND CONSTRUCTION OF APPARATUS FOR ANALYSIS AT SUBATMOSPHERIC TEMPERATURES:-

The Leitz dilatometer had no provisions for the thermal analysis of materials at subatmospheric temperatures. Since the dilatometer is on a horizontal base, analysis of materials at low temperatures is difficult because the specimens cannot be cooled to low temperatures without immersing them in a bath, which involves quenching them from ambient temperatures. The quenching process has two major disadvantages and they are that the specimen and the quartz tubes are subjected to thermal shock, which, if great enough will result in the fracture of the quartz tubes thereby making the apparatus non-functional.

It is because of the above mentioned disadvantages and the need for an apparatus to allow analysis at a lower rate of temperature change that a device adaptable specifically to the Leitz dilatometer was constructed. The device alleviates the quenching process and its inherent disadvantages, and permits thermal analysis at substmospheric temperatures, allowing the desired gradual rate of temperature change.

A cross-sectional view of the adapting apparatus is shown in Figure 5. The apparatus is of a simple design and construction, consisting essentially of three concentric cylinders with the end plates secured by tie rods.



Code:

(1) Inner cylinder wall
(2) Center cylinder wall
(3) Outer cylinder wall
(4) End plate
(5) Insulation
(6) Tie-rods

Cross section of "thermal can" adapted for Leitz dilatometer

Figure 5

The volume between the outer cylinder and central cylinder is loosely filled with Hyflo Super-Cel insulation (a diatomaceous silica product), and the volume between the inner and central cylinders being provided to contain the liquid coolant. The aluminum end plates were grooved to accomodate "O" rings and the ends of the cylinders, except for the center of one end plate which was machined to allow the inner cylinder to be open on that end. Holes were then drilled around the peripheral edge of the end plates, and steel tie-rods threaded on each end were inserted and drawn down tight to prevent any leakage of coolant. The open end of the inner cylinder was press-fitted into the end plate to be certain of leak prevention at that point. The inner cylinder is also of chosen diameter as to allow very close tolerances between its inner wall and the vacuum tube surrounding the specimens; this is to insure better thermal conductivity from the tube to the coolant. This "thermal can" is also designed for easy disassembly, if necessary.

The coolant is stored in a 25-liter Purox liquified gas container which is equipped with a pressure withdrawal tube. A ¼-inch copper tube is soldered to the withdrawal tube and enters the bottom of the coolant chamber of the "thermal can." The fitting of the copper tube at the can is loose enough to allow hand-fitting and easy disassembly, the fitting being sealed during operation. The coolant is "pushed" out of the Purox container into the can by means of a cylinder of compressed gas, regulating the pressure manually. The pressure line from the gas cylinder was accomodated with a 3-way valve to allow depressurization of the Purox container to the atmosphere. By means of this regulation of the pressure in the container, the rate of flow of the coolant and consequently its volume in the "thermal can" can be controled. Since liquid nitrogen was used as the coolant, the gas chosen to "push" it out was helium because it has a much lower boiling point than nitrogen, thus preventing the freezing-shut of the opening.

The assembly of the apparatus for the dilatometric analysis at subatmospheric temperatures is shown in Figure 6, and the assembly for analysis above atmospheric temperatures is shown in Figure 7.

PROCEDURE OF THE ANALYSIS AT HIGH AND LOW TEMPERATURES:-

Subsequent to the calibration of the coordinate axes, the previously prepared steel specimen was placed in the quartz tube making sure of good contact of surfaces by gently tapping the tube, and the thermocouple inserted in the standard copper rod. The ceramic vacuum tube was placed over the specimens, fitted into the vacuum head, and the "thermal can" slid over the entire tube. Water was circulated through the "cooling chamber" to prevent any deleterious effects of temperature changes in the delicate mechanism in the dilatometer head. Liquid nitrogen was then introduced into the can until the lowest

Fig. 6.

View of the apparatus used for the dilatometric analysis of the material at subatmospheric temperatures.

Fig. 7.

View of the Leitz dilatometer for the thermal analysis of materials at and above atmospheric temperatures.

temperature attainable (-150°C) was reached and held constant. The light was then switched on and the can was allowed to warm up to room temperature by vaporization of the liquid nitrogen. At room temperature the "thermal can" was removed and replaced by the electric furnace, the vacuum pump turned on, and the specimens were slowly heated at a rate of approximately 10°C per minute to a temperature of 900°C. The temperature was held at 900°C for ten minutes to allow the specimens to come to "equilibrium," and then the furnace was turned off and removed from the dilatometer to allow the specimens to air cool to room temperature. The "thermal can" and liquid nitrogen was again applied for temperatures down to -150°C to detect any further martensite transformation at the subatmospheric temperatures.

Another run was made of the heating cycle, but the specimens were furnace-cooled from 900°C to room temperature to detect the effect, if any, of the cooling rate on the M_s temperature.

A dilatometer trace was also made during a maraging heat treatment at 475°C (890°F) over a period of three hours to detect any dimensional changes taking place during the aging treatment. This isothermal analysis was accomplished by use of the timing device, previously described, supplemented by the 2X accessory optical magnifying system which is fitted into the front of the camera.

METALLOGRAPHIC SUPPLEMENT:-

Specimens of the steel for metallographic analysis were placed in the furnace on the dilatometer and given heat treatments similar to those during the dilatometric analysis. The specimens were then prepared by usual metallographic methods using the Linde B alumina abrasive for the final polish. The polished specimens were chemically etched with a solution of 50 ml HCl, 25 ml HNO_3, 1 gm $CuCl_2$, and 150 ml H_2O.

Figure 10 is a photomicrograph of a specimen annealed at 900°C for one hour and furnace cooled, showing the equilibrium room temperature structure.

Figure 11 is a photomicrograph of a specimen annealed at 850°C and air cooled, showing a massive indistinct structure of the metastable martensite.

Figure 12 shows the structure of a specimen annealed at 850°C and water quenched, yielding a more revealing structure of the metastable martensite than the as-annealed specimen; the photomicrograph shows the areas of surface upheaval due to the shearing martensite reaction.

Figure 13 is a photomicrograph of the quenched specimen after it was re-annealed at 650°C (1200°F) and air cooled. The structure is somewhat lamellar appearing and is approximately 50 percent austenite (light etching areas) and 50 percent martensite (dark etching areas). Apparently at this annealing temperature the austenite formed during annealing became partially stabilized and did not retransform to the body-centered cubic structure on cooling to room temperature.

RESULTS:-

The results of the athermal dilatometric analysis are shown in the trace in Figure 8. As noted on the curve the A_s and A_f temperatures are approximately 580°C and 775°C, respectively. The M_s and M_f temperatures are approximately 148°C (300°F) and 60°C (140°F), respectively.

The coefficient of thermal expansion of the body-centered cubic (martensite) phase over the temperature range from -150°C (-240°F) to 580°C (1075°F) is calculated from the dilation curve:

Expansion 7.75 x 10^{-3} in/in.

Temperature range 1315°F (730°C)

Linear coefficient of thermal expansion

$$\frac{7.75 \text{ in/in}}{1315^{\circ}\text{F}} \qquad 5.9 \times 10^{-6} \text{ in/in/}^{\circ}\text{F}$$

$$10.6 \times 10^{-6} \text{ in/in/}^{\circ}\text{C}$$

This compares well with the published values of 5.6 x 10^{-6} for 18% Ni and 6.2 x 10^{-6} for the 20% and 25% Ni steels, since the steel tested in this work has 18.7% Nickel.

The isothermal dilatometric analysis of the maraging behavior is shown in Figure 9. This curve shows the change in length as a function of the time at the maraging temperature. The curve in Figure 9 is not an absolute curve, but is only representative of the relative dimensional changes taking place during maraging, since the trace could not be read with great accuracy.

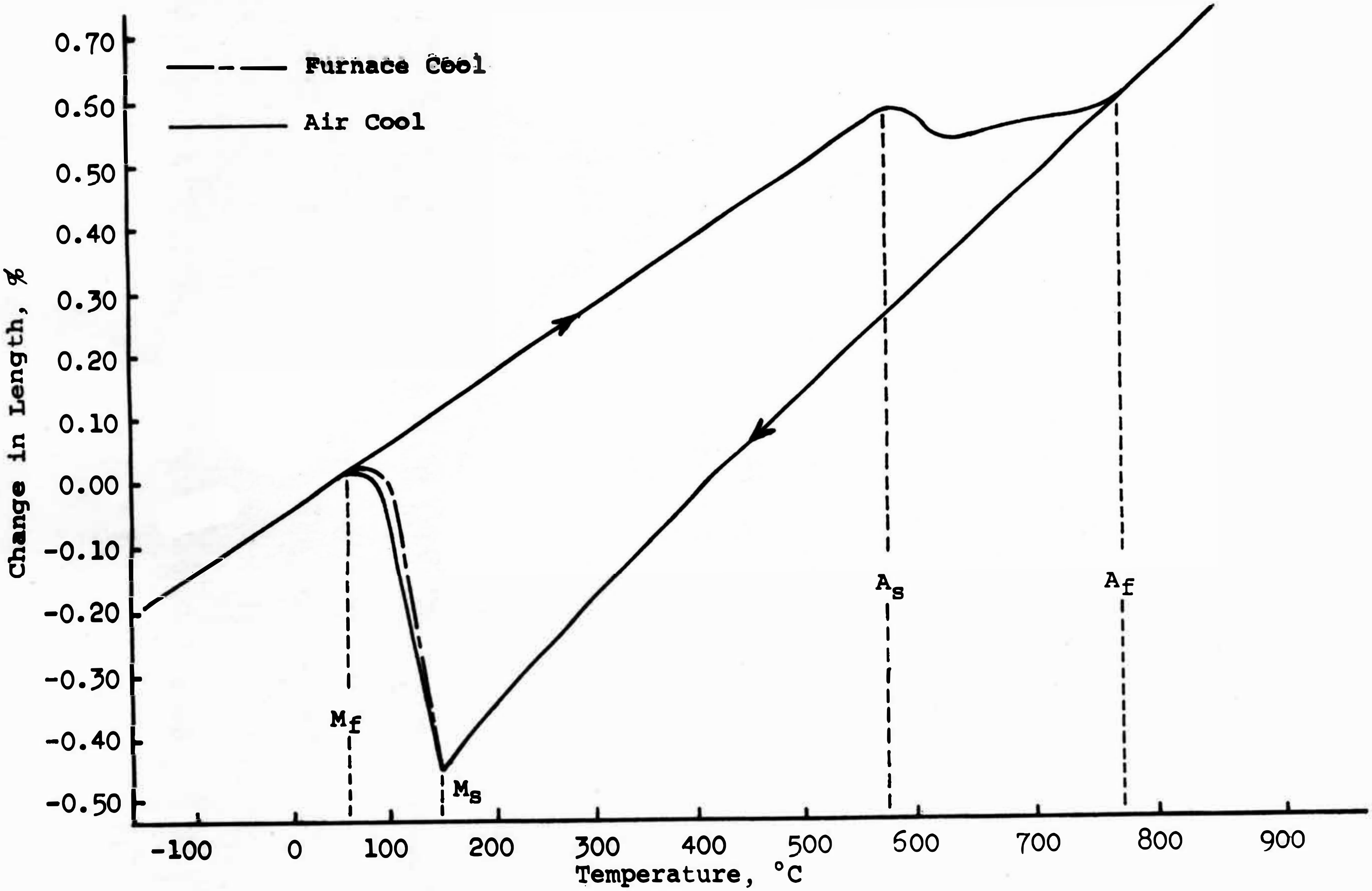


Fig. 8. Leitz dilatometer trace of change in length with temperature of an alloy of 18.7 Ni, 7.9 Co, 4.6 Mo, bal Fe.

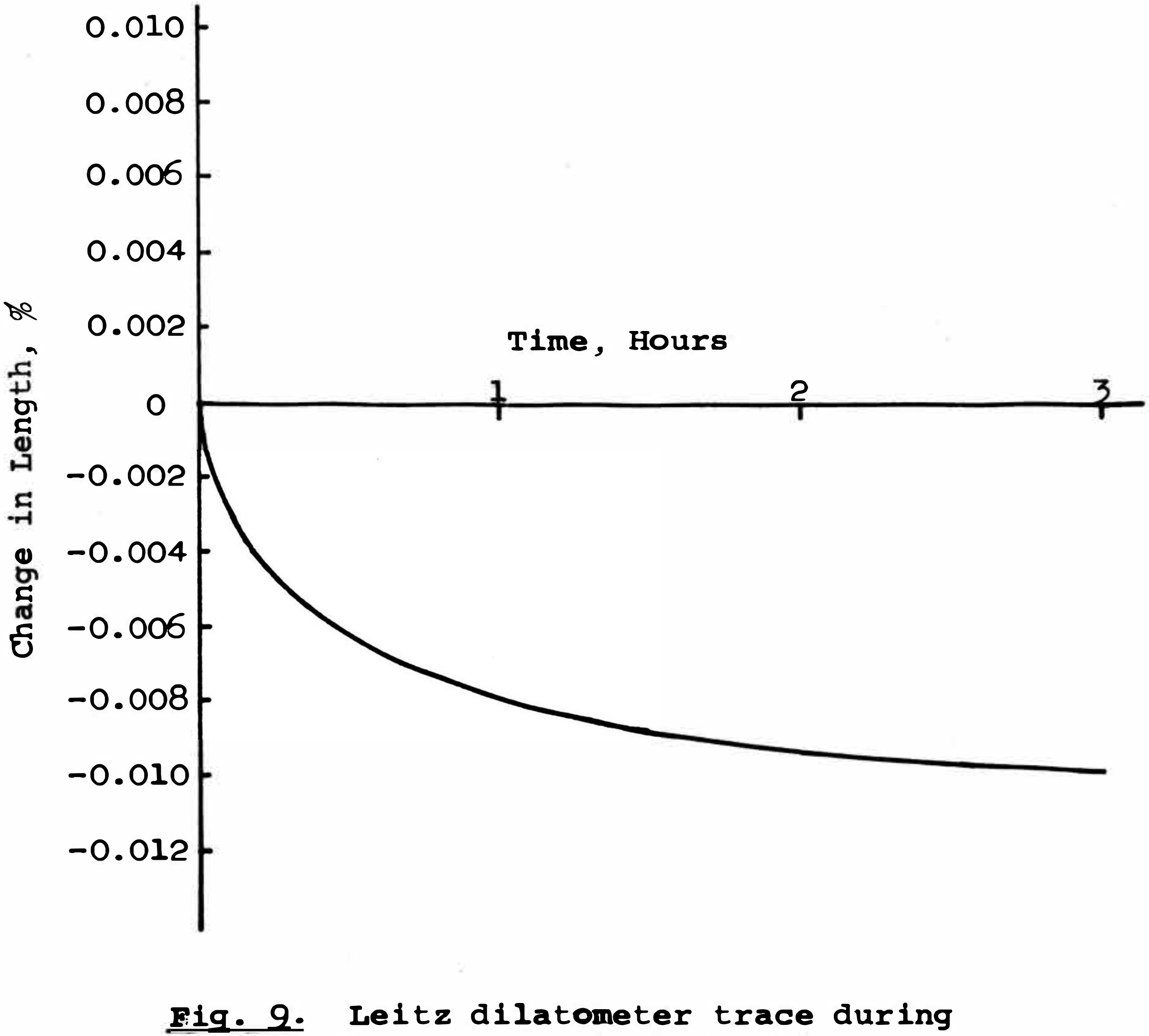


Fig. 9. Leitz dilatometer trace during isothermal maraging at 475°C.

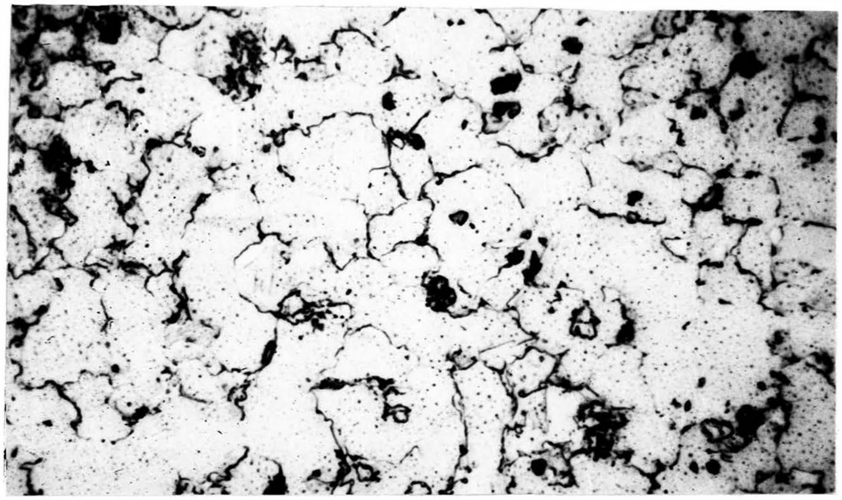


Fig. 10.

Annealed one hour at 900°C, furnace cooled. Equilibrium room temperature structure; 250X; R_c 20. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

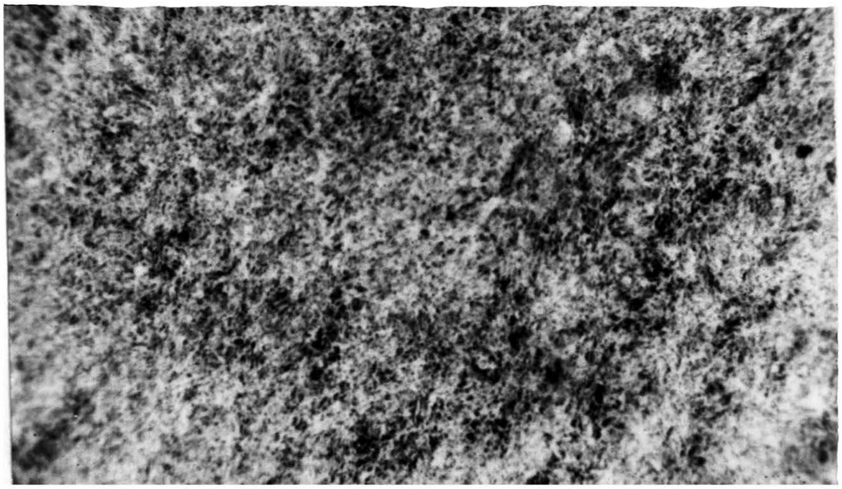


Fig. 11.

Annealed one hour at 850°C, air cooled. Massive structure of metastable martensite. 250X; R_c 21. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

Fig. 12.

Annealed for one hour at 850°C, water quenched. Metastable martensite revealing surface upheaval due to the shearing formation of martensite. 250X; R_c 22. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

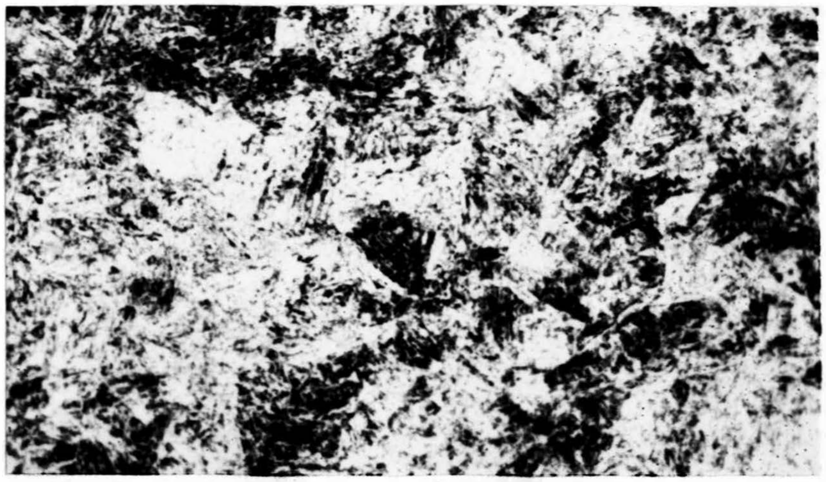


Fig. 13.

Quenched from 850°C, re-annealed at 650°C, air cooled. Approximately 50% martensite and 50% retained austenite. 250X; R_c 17.5. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

As can be seen, very little dimensional change has taken place; the trace is greatly expanded to show more clearly the approximate rate of change. The trace shows a contraction of the order of 0.010% after maraging for 3 hours at 475°C.

The discussion of these results will follow in a later section.

PRECAUTIONS AND SAFETY CONSIDERATIONS:-

Precautions to be taken for the dilatometer itself to insure good results are:

1) Do not exceed the maximum current indicated by red marks on the ammeters on the light transformer and the slide-wire rheostat, in order to prevent the premature failure of the electrical components.
2) Insure good contact between the specimens in the quartz tubes by tapping them gently to prevent erratic results on the photographic trace.
3) Always mark the origin and the axes for each run during the investigation.

When using liquified gases, some general safety precautions which should be observed to prevent injury to personnel and damage to equipment are listed as follows:

1) Never admit liquid nitrogen to a container of any kind unless it is properly vented or has adequate safety relief devices. "Bottled-up" liquid nitrogen will build pressure as it vaporizes, and may

burst the equipment used to contain it.

2) Wear goggles for eye protection when liquid nitrogen is being handled in open containers.
3) Do not discharge waste nitrogen vapor into unventilated spaces. Dilution of the oxygen in air required for breathing must be avoided.
4) Liquid nitrogen is extremely cold (-320°F). Do not spill on hands or clothing. Wear gloves when handling liquid nitrogen equipment or parts that have been cooled in liquid nitrogen. Gloves should be of a relatively impervious material, such as leather. They should be loose fitting so that if liquid nitrogen does get into a glove, it can be thrown off the hand quickly. Liquid nitrogen on the skin produces an effect similar to a severe burn.
5) When cooling parts in liquid nitrogen, immerse cautiously to avoid splashing.

Two important general principles involved in pipe line design for liquid nitrogen may also be mentioned:

1) Use a safety pressure relief valve in every length of line between two shut-off valves.
2) Slope all lines up from the tank. Avoid gas traps.

CHAPTER V

MARAGING BEHAVIOR

SAMPLE PREPARATION:-

All specimens used to study the maraging behavior were taken from the ½-inch diameter bar. Specimens were cut to a width of 3/16-inch on a cut-off wheel and were taken from a section approximately 12 inches from one end of the bar. Specimens were prepared in six groups, each group having six specimens except for one group which had 15 specimens for a close check of the effect of maraging time on the hardness.

HEAT TREATMENT AND HARDNESS DETERMINATIONS:-

In the study of the maraging behavior, only three factors were varied: annealing temperature, maraging temperature, and time.

Since the maraging treatment incorporates a heat treatment at elevated temperatures, several different heat treatments were given to groups of specimens in order to note the effect on hardness, thereby giving an insight to the peculiarities or characteristics of the hardening process. A separate group of specimens was used for each heat treatment.

The method used to study the effects of the variables mentioned may be summarized and shown in the following table:

Group No.	Annealing Temperature, °C	Maraging Temperature, °C	Maraging Time, Hours
1	800	475	0 to 4
2	900	375	0 to 4
3	900	425	0 to 4
4	900	475	0 to 4
5	900	525	0 to 4
6	1000	475	0 to 4

Hardness determinations were made to study the effects of the variables because hardness is a convenient measure of the tensile strength of the material, since for ductile materials, increases in strength are generally accompanied by parallel increases in hardness. The hardness determinations were made on a Rockwell hardness tester using the "C" scale (150 kg load), and the hardness values reported are averages of three readings on each specimen. The specimen surfaces on which the hardness determinations were made were finished on silicon carbide paper (600 grit) just previous to the test. The Rockwell hardness machine was checked and calibrated with standard hardness blocks in the R_c hardness range.

METALLOGRAPHIC ANALYSIS:-

The hardness determinations following the maraging behavior of the steel was supplemented by a metallographic analysis to note any changes in microstructure, which might accompany the hardening mechanism. The metallographic analysis was made only on one group of specimens following the maraging behavior, because it is assumed to be representative of any changes taking place in the other specimens of the other groups.

The method of analysis was to investigate the microstructures of specimens which were all given the same annealing treatment (900°C) and were all maraged at the same temperature (475°C), but at different times during the maraging treatment. Each microstructure presented here is representative of the entire specimen, and those presented (though not complete) are representative of the group; the presentation of the complete series would only make this thesis cumbersome, since there is no additional information to be derived from the unpresented microstructures.

RESULTS:-

The body-centered cubic martensite has the Widmanstatten morphology which is consistent throughout the group of maraged specimens.

Aside from the fact that the microstructures exemplify the Widmanstatten structure, which signifies that a precipitation reaction has taken place, there is no apparent or significant visual change in the microstructures during the maraging treatment.

The microstructure of the specimen in the annealed condition is shown in Figure 11, and the results of the metallographic analysis of the maraged specimens are on the following pages.

The effects of annealing temperature, maraging time, and maraging temperature on hardness are manifested in the curves in Figures 21 and 22.

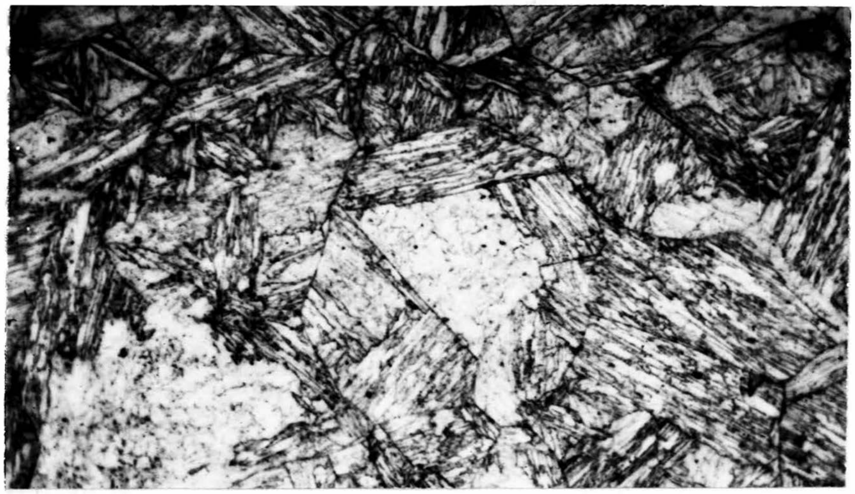


Fig. 14.

Annealed 900°C, air cooled, maraged 475°C for 15 minutes, air cooled. Bcc martensite, Widmanstatten structure; 500X; R_c 35. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

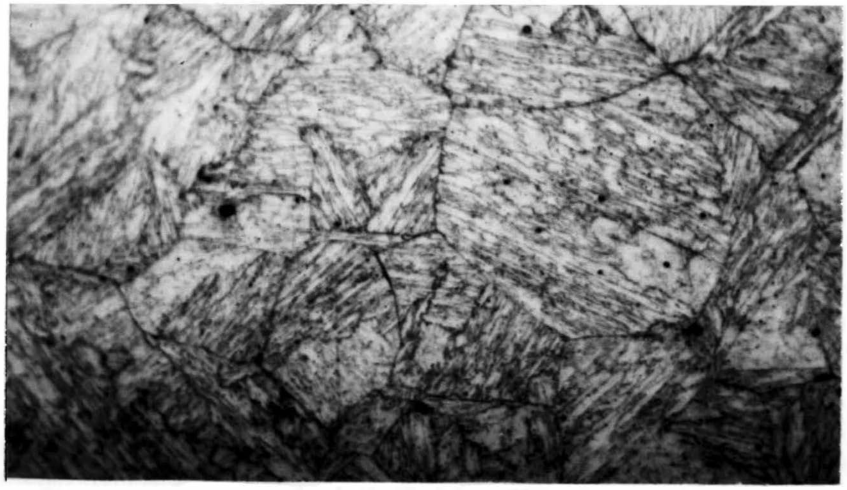


Fig. 15.

Annealed 900°C, air cooled, maraged at 475°C for 30 minutes, air cooled. Bcc martensite, Widmanstatten structure. 500X; R_c 37.5. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

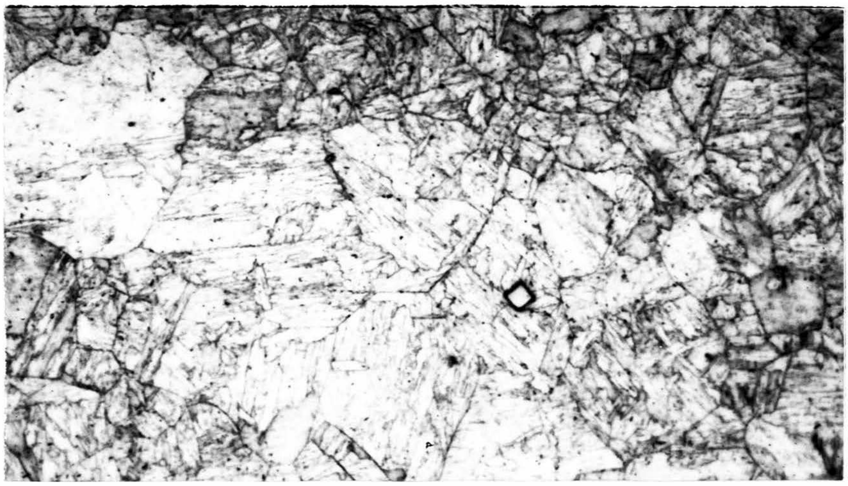


Fig. 16.

Annealed 900°C, air cooled, maraged at 475°C for 1 hour, air cooled. Bcc martensite, Widmanstatten structure; 250X; R_c 40.2. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

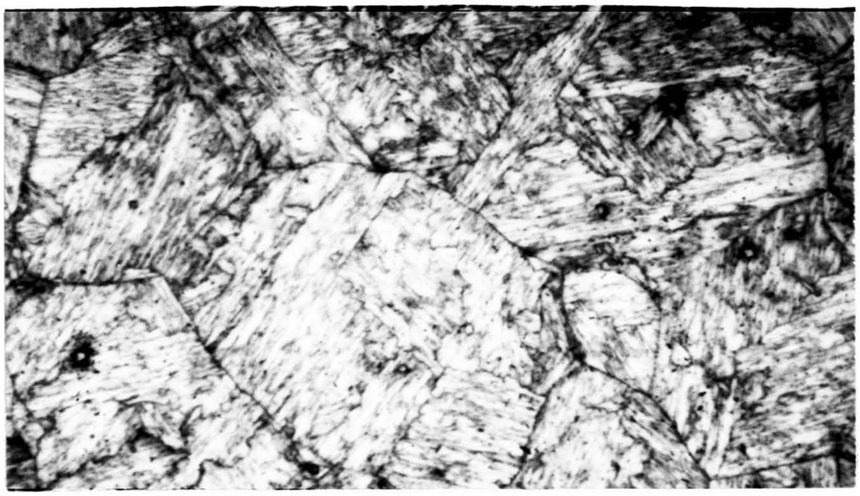


Fig. 17.

Annealed 900°C, air cooled, maraged at 475°C for 1 hour 15 minutes, air cooled. Bcc martensite, Widmanstatten structure. 500X; R_c 41.2. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

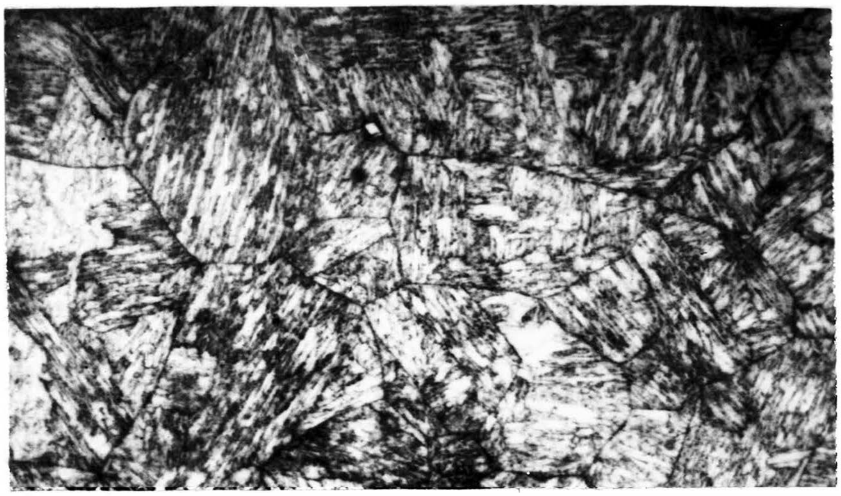


Fig. 18.

Annealed 900°C, air cooled, maraged 475°C for 1 hour 30 minutes, air cooled. Bcc martensite, Widmanstatten structure; 500X; R_c 42.0. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

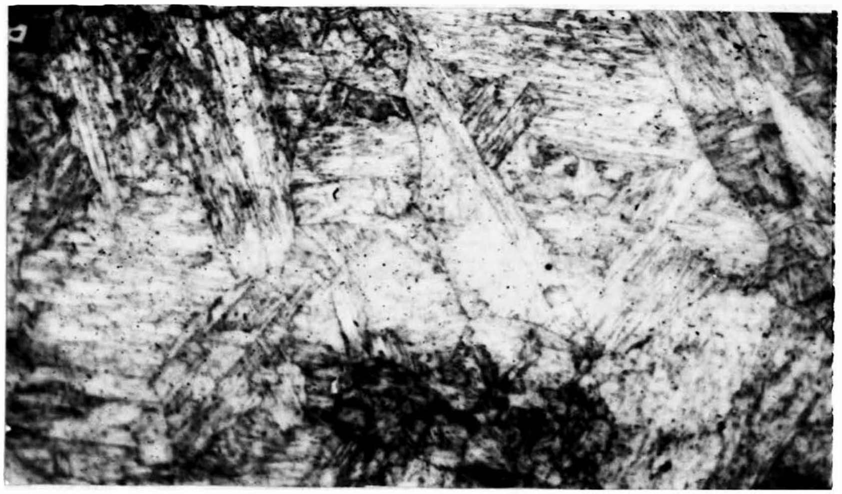


Fig. 19.

Annealed 900°C, air cooled, maraged 475°C for 3 hours, air cooled. Bcc martensite, Widmanstatten structure; 500X; R_c 44.5. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

Fig. 20.

Annealed 900°C, air cooled, maraged at 475°C for 3 hours 30 minutes, air cooled. Bcc martensite, Widmanstatten structure; 500X; R_c 45.0. Etchant: HCl, HNO_3 and $CuCl_2$ solution.

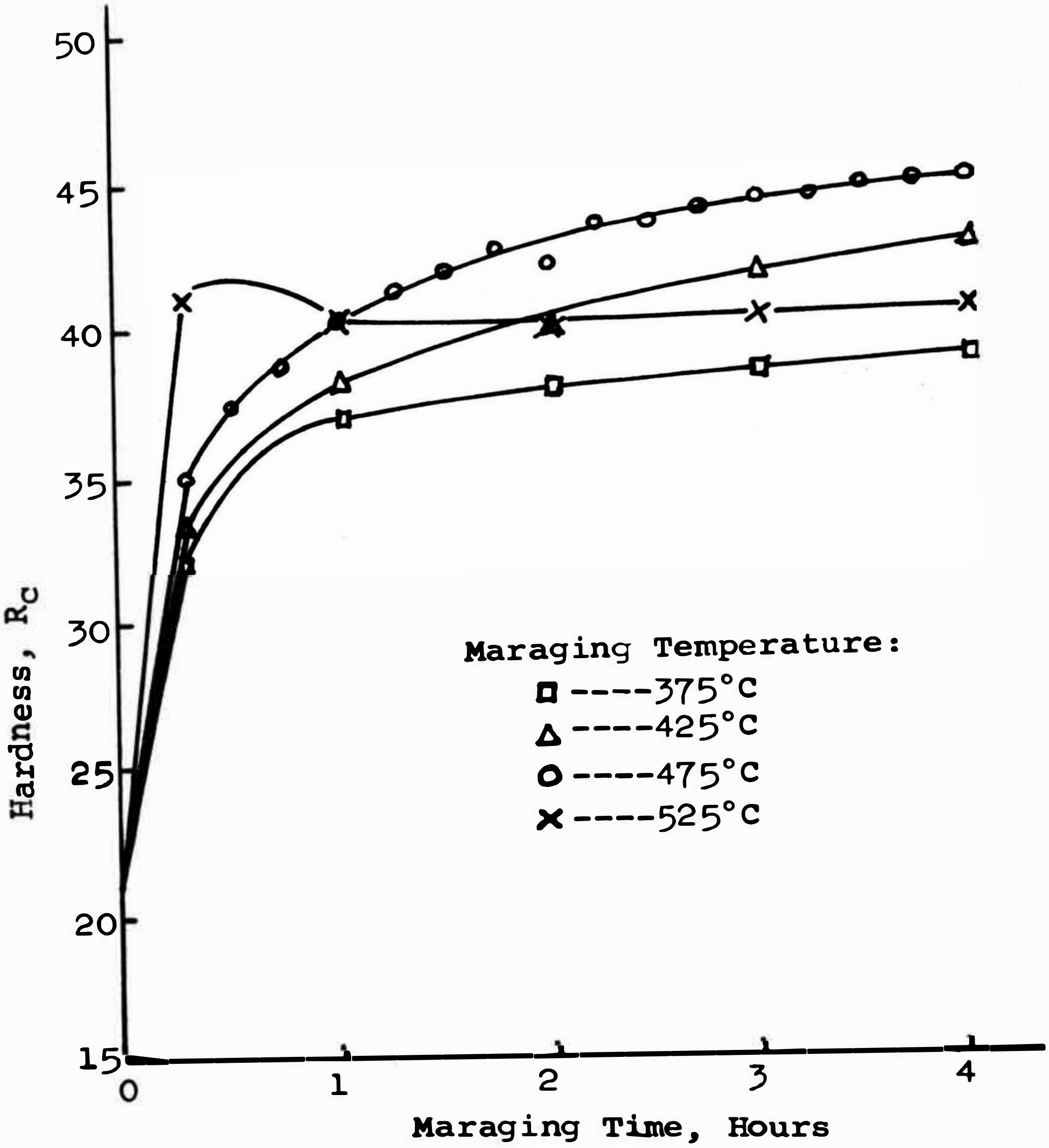


Fig. 21. Effects of Maraging Temperature and Time on Hardness. Annealed 1 hour at 900°C.

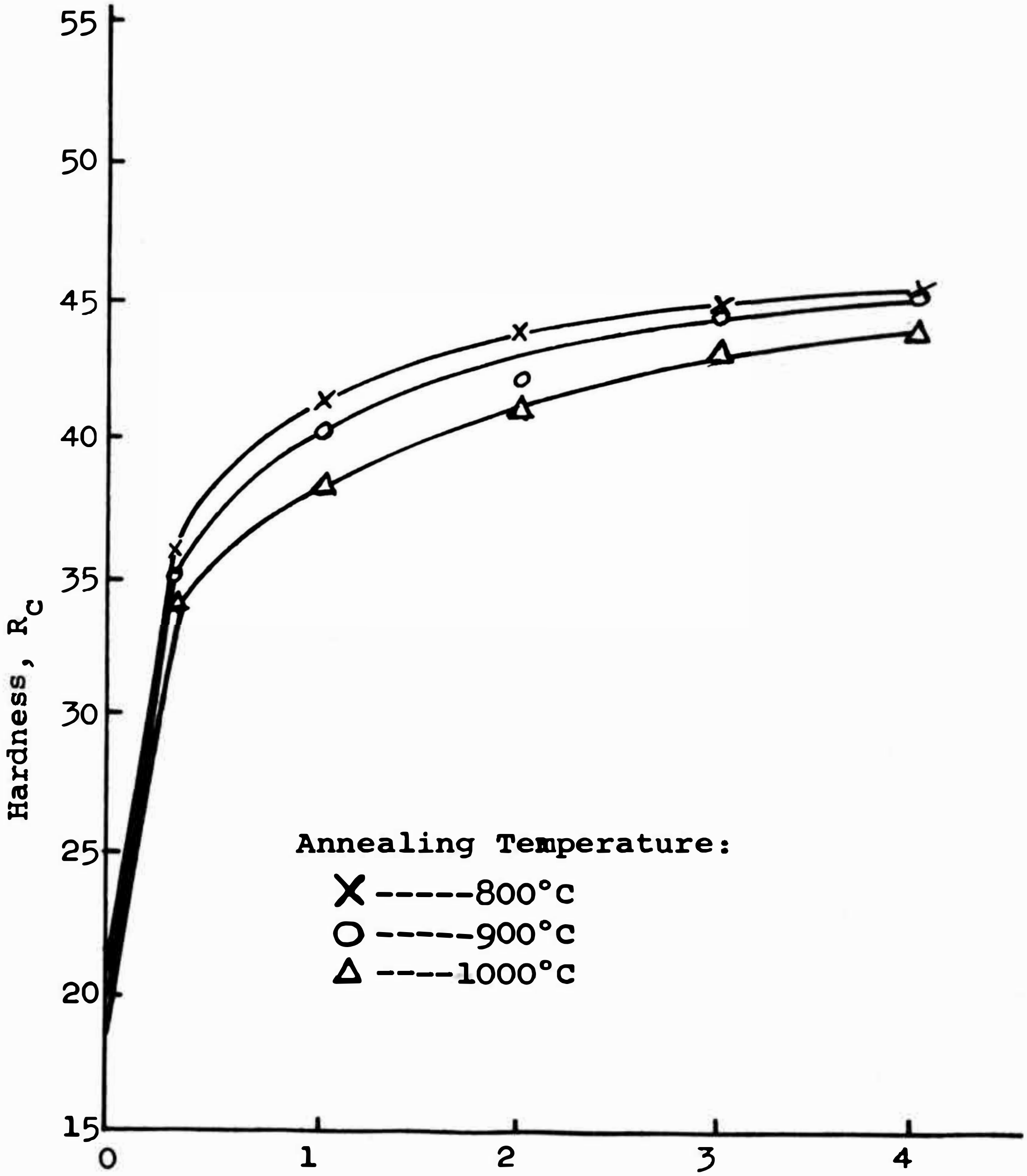


Fig. 22. Effects of Annealing Temperature and Maraging Time on Hardness. Maraged at 475°C.

CHAPTER VI

DISCUSSION AND INTERPRETATION OF RESULTS

THE MARTENSITE TRANSFORMATION:-

The dilatometer trace presented in the results of Chapter IV (Figure 8) serves to provide the temperature range over which martensite formation occurs athermally, the M_s and M_f being 148°C and 60°C, respectively. As noted in the figure, the significance of the wide difference in cooling rates is only slight. Apparently a slower rate of cooling is more conducive to the transformation by allowing more time at temperatures for the progress of the transformation than does the faster cooling rate. The manifestation of this difference is that for a particular temperature in the martensite formation range, a larger percentage of martensite will be formed in the course of cooling at the slower rate. That the M_s temperature is virtually unaffected, while the rate of formation is affected, by the rate of cooling may be consequential of one or both of the following phenomena that could be taking place:

1) partial or short-time stabilization of some of the austenite during the faster rate of cooling, thus impairing or reducing the rate of marentsite formation
2) partial isothermal transformation of martensite occurring concurrently with the athermal transformation during the slower rate of cooling.

The occurrence of the latter phenomenon would be more probable than that of the former, since the M_f temperatures of the two rates are approximately the same, and the presence of stabilization would result in a lower M_f temperature than that indicated by the curve.

That these two phenomena do take place, providing certain conditions prevail, is exemplified and substantiated by the studies of martensite stabilization in plain carbon and low-alloy steels by Harris and Cohen[7] and by Klier and Troiano,[27] and the evidence of isothermal martensite formation (even in absence of changing chemistry) in cases reported by Averbach, Cohen, and Fletcher[28] and Averbach and Cohen.[29]

Stabilization is a remarkable process in that it reportedly occurs without any detectable change in the parent phase and is unrelated to possible side reactions going on in the martensite product. According to Geisler,[11] stabilization has been observed during continuous cooling at slow rates, manifesting itself either in a reduction of the average amount of martensite formed per degree of dropping temperature or in a lowering of the M_s temperature. It is most significant that the martensite transformation, which is not suppressible by rapid quenching, can be inhibited to a considerable degree by retarded cooling because of the advent of the stabilizing process.

There does not appear to be any distinct morphology associated with the martensite in the microstructures of the specimens which were supplemental to the dilatometric analysis of the martensite transformation for the annealing temperatures and cooling rates given.

THE MARTENSITE REVERSION:-

From an analysis of the martensite to austenite reversion in the Fe-Ni alloy exemplified in the dilatometer trace in Figure 1, it is obvious that the reversion is uninhibited and occurs over a small range of temperature. A sharp reversion of the martensite in the alloy under study is noticeably not prevalent in the dilatometer trace in Figure 8. This very significant extension of the temperature range of austenite reversion could be the result of one or both of the following phenomena taking place:

1) partial stabilization of the martensite during the heating process, as demonstrated in Figure 3 from the work of Kaufman and Cohen[3]
2) reversal of martensite to the parent phase on heating at temperatures where the latter is still unstable (with respect to nucleation-and-growth products)[11]

The occurrence of the partial stabilization may be apparent from the short drop in the heating curve after the start of the austenite formation. A considerable amount of martensite may have been stabilized in a short time, requiring a considerable increase in temperature to complete the reversion to austenite. In addition, it appears that the reversion of the martensite was not done in large volumes, consequently the trace shows a small amount of dimensional change over the temperature range. This could be explained by a proposal that the contraction associated with the austenite formation is approximately balanced by two opposing factors: the expansion of the stabilized martensite and the previously formed austenite with the increase in temperature. The predominance of the austenite expansion with the increase in temperature results in the gradual increase in length.

With regard to the second phenomenon, the reversal of the martensite transformation has been found in Cu-Zn and Fe-Ni[2] alloys, as shown in Figure 1 by Jones and Pumphrey. The fact that, while the reverse transformation may occur with considerable hysteresis, it takes place at temperatures (below T_e) where the parent phase is still unstable, serves to explain in part the large temperature range over which the reversion takes place.

It is proposed by the author that each of the phenomena mentioned plays an important and significant part in the mechanism taking place during the reversion of the martensite to austenite.

THE MARAGING BEHAVIOR:-

The maraging behavior of the steel is typical of the aging behavior of alloys which involve precipitation reactions in their hardening mechanisms, as is shown by the general shapes of the curves in Figures 21 and 22. As is evident from the curves, hardness not only increases with the maraging time, but with maraging temperature, reaching an optimum level at a temperature of 475°C (900°F).

Maraging at a temperature above this value results in a decrease in the hardness level. Hardness also increases with decreasing annealing temperature, for those temperatures used, reaching an optimum level corresponding to an annealing temperature of 800°C. In the early stages of the hardening process, the rate of attainment of hardness increases with increasing maraging temperature and decreasing annealing temperature. After the initial hardening, the rate of increasing hardness is approximately the same for the different annealing and maraging temperatures.

The hardness values reported by the International Nickel Company of a somewhat similar steel, after various heat treatments which were similar to those given to specimens in this work, were approximately five units higher (on R_c scale) than those obtained for the specimens in this work. There was a slight difference in compositions of the steels, the major difference being the addition of 0.4% titanium to their steel while the chemical analysis of the steel used for this work showed no content of titanium. Since titanium is an interstitial scavenging element and a significant hardening element, it is felt that the comparison of values show good agreement and the lower hardnesses obtained are justifiable in light of the absence of titanium from the chemistry of the steel.

A striking feature in the maraging behavior is that retrogression, or "over-aging," does not appear to occur in the titanium-containing steel or the steel used for this thesis. This is evidenced by the constant and prolonged hardness level at long aging times. Again making reference to Figure 21, it is noticeable and quite significant that at the high maraging temperature of 525°C (1000°F) the drop in hardness from the value during early maraging is quite small and the hardness persists into long maraging times.

Another feature of the maraging behavior of the alloy is the "precipitate" - - conspicuous in its absence using light microscopy. Two convincing effects of the aging treatment suggesting the presence of a precipitate (coherent or incoherent) are: the increase in hardness with increasing aging time and the morphology of the structure, i.e., the obvious presence of the Widmanstatten figure. The Widmanstatten figure suggests precipitation of a new phase from solid solution.

THE MECHANISM OF MARAGING:-

In view of the very limited information offered in this work, only a few generalizations regarding the maraging mechanism may be made and the effects observed in the behavior can at most only be indicative.

Since there is no microscopic evidence of a precipitate particle, it must not and cannot be concluded that no precipitate is formed. In fact, the very absence of a visible particle should suggest that a precipitate coherent with the matrix could be present, thereby suggesting (in a reverse line of thought) that the precipitate would have approximately the same lattice parameter as the matrix. In view of the major elements (Fe, Ni, Co, Mo) in the chemistry of the alloy, a coherent precipitate is more than just feasible, but presents itself as a strong possibility.

A coherent precipitate could strengthen the matrix, if it were much harder than the matrix. This would require a precipitate of significantly different chemical composition than that of the matrix, but analysis of the hardness data shows that initial hardening proceeds very rapidly, and it seems unlikely that sufficient long range diffusion could occur in the short time to allow the formation of such precipitates.

Consideration of the large increase in hardness resulting from the maraging treatment (e.g., annealed 900°C, maraged at 475°C for 3 hours) should be indicative that a very high degree of coherency is not probable, while at the same time a low degree of coherency is not probable either, because the International Nickel Company reported excellent ductility and toughness for the alloy after hardening, since slip by the movement of dislocations would be greatly impaired by a very low degree of coherency.

The absence of over-aging in the alloy together with the observation that the lower annealing temperatures yield higher hardnesses during the aging treatment throws light on the possibility that ordering could be responsible for increasing hardness. The kinetics of the hardening, i.e., the rapid initial increase, also is quite suggestive of an ordering reaction (probably short range order), and studies[30] have indicated that the degree of order changes much more rapidly when an alloy is initially ordered than when it is initially disordered. As mentioned in the literature review, previous electron diffraction studies of the maraged alloy gave some evidence of ordering based on a body-centered cubic structure of the composition Fe_2MCo.

From the scarce information available, it is felt that either an ordering reaction or a coherent precipitate or combination of both is responsible for the hardening behavior in this alloy, but there is little basis to justify a choice at this time. The discussion and interpretation presented above regarding the hardening mechanism is provided only for consideration of some possibilities, and in no way is conclusive. Further work is necessary to clarify the situation.

METALLOGRAPHIC ANALYSIS:-

The presence of the Widmanstatten figure in the microstructures is significant, and on close examination, one can see three principal directions within one grain; this is shown especially well in the microstructure of Figure 17. According to Mehl,[17] this count of three directions signifies the formation of precipitate plates parallel to (100) planes in the body-centered cubic matrix. Verification of this would require a stereographic projection of the pole figures of the crystal lattice.

CHAPTER VII

CONCLUSIONS

The conclusions which may be drawn from the results of the work are listed as follows:

1) The martensite formation range for the material investigated is from 148°C (M_s) to 60°C (M_f).
2) Cooling rate has little effect on the transformation temperatures.
3) No further martensite transformation occurs on cooling to subatmospheric temperatures.
4) Martensite transforms to austenite on heating with a high degree of hysteresis.
5) The linear coefficient of thermal expansion in the temperature range from -150°C to 580°C is 10.6×10^{-6} in/in/°C.
6) Hardness increases with time at the maraging temperature after an appropriate solution anneal.
7) Hardness increases with maraging temperature for a particular maraging time after an appropriate solution anneal.
8) Hardness and the initial rate of attainment of hardness increases with decreasing annealing temperature.
9) The mechanism of hardening is presently unknown.

SUGGESTIONS FOR FURTHER STUDY

Both the maraging steel and the method of dilatometric analysis provide interesting and fruitful topics for further study.

The construction of the dilatometric adaptation apparatus offers a useful tool for investigations of transformations and solid-state reactions which are accompanied by dimensional changes at subatmospheric temperatures.

A more detailed study of the maraging behavior and its mechanism should be made by electrical resistivity measurements, incorporating an analysis of the effects of varying certain factors, such as the austenitic grain size, and of the changes in physical properties taking place during the maraging treatment.

Strictly speaking, more revealing and conclusive information would result from a neutron diffraction study of the changes taking place during maraging, probably detecting the "precipitate" responsible for the hardening. Neutron diffraction, in contrast to x-ray diffraction, is suggested as the method of analysis because, for neutron diffraction, the scattering factors of iron, nickel, and cobalt are sufficiently different as to allow distinguishment of one of the elements from the others.

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APPENDICES

APPENDIX I

MARAGING BEHAVIOR AT 375°C (ANNEALED 900°C)

Specimen Number	Maraging Time Hours	Hardness R_c
1	0	21.0
2	0.25	32.0
3	1.0	37.0
4	2.0	38.0
5	3.0	38.5
6	4.0	39.0

APPENDIX II

MARAGING BEHAVIOR AT 425°C (ANNEALED 900°C)

Specimen Number	Maraging Time Hours	Hardness R_c
1	0	21.0
2	0.25	33.5
3	1.0	38.3
4	2.0	40.0
5	3.0	42.0
6	4.0	43.0

APPENDIX III

MARAGING BEHAVIOR AT 475°C (ANNEALED 900°C)

Specimen Number	Maraging Time Hours	Hardness R_c
1	0	21.0
2	0.25	35.0
3	0.50	37.5
4	0.75	38.7
5	1.00	40.2
6	1.25	41.2
7	1.50	42.0
8	1.75	42.5
9	2.00	42.2
10	2.25	43.5
11	2.50	43.5
12	2.75	43.8
13	3.00	44.5
14	3.25	44.8
15	3.50	45.0
16	3.75	45.1
17	4.00	45.2

APPENDIX IV

MARAGING BEHAVIOR AT 525°C (ANNEALED 900°C)

Specimen Number	Maraging Time Hours	Hardness R_c
1	0	21.0
2	0.25	31.0
3	1.00	40.2
4	2.00	40.0
5	3.00	39.8
6	4.00	40.0

APPENDIX V

MARAGING BEHAVIOR AT 475°C (ANNEALED 800°C)

Specimen Number	Maraging Time Hours	Hardness R_c
1	0	20.0
2	0.25	36.0
3	1.0	41.5
4	2.0	44.0
5	3.0	45.0
6	4.0	45.5

APPENDIX VI

MARAGING BEHAVIOR AT 475°C (ANNEALED 1000°C)

Specimen Number	Maraging Time Hours	Hardness R_c
1	0	19.0
2	0.25	34.0
3	1.0	38.3
4	2.0	41.0
5	3.0	43.0
6	4.0	44.0

VITA

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