THE NATURE OF AUSTENITE PRODUCED BY
THE REVERSE MARTENSITIC TRANSFORMATION

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ABSTRACT

Stabilization, strengthening, and recovery and recrystallization of austenite produced by the reverse martensitic transformation were investigated in low carbon, vacuum melted 29.4, 30.6, and 32.4 atom percent nickel-iron alloys. Resistance measurements as a function of temperature showed that all alloys were completely stabilized after four or five cycles of slow heating and cooling between -196°C and 500°C. Lattice parameter measurements established that the austenite was stabilized as a result of enrichment in nickel rejected by ferrite which formed under these conditions. When diffusion-controlled reactions were suppressed by rapid heating and cooling, no stabilization occurred in the reversed austenite.

The strain energy introduced into reversed austenite by the reverse martensitic transformation provides sufficient driving force for partial recovery of the reversed austenite by the development of twinned structures and eventual recrystallization by strain-induced boundary migration. The formation of reversal twins during recovery and the high frequency of irregular annealing twins after recrystallization suggest that stacking faults are generated by the reverse martensitic transformation.

Increases in yield strength (0.2 percent offset) of reversed austenite up to 177 percent of the values for annealed austenite were measured after 5 cycles of transformation, the greatest single increase in strength being developed after the first cycle of transformation. The strengthening of the austenite is believed to result from stacking faults produced by the reverse martensitic transformation. Martensitic structures formed from the reversed austenite were also strengthened somewhat due to structural changes inherited from the parent phase.

Recrystallization was studied by quantitative metallography and hardness changes. Characteristic sigmoidal recrystallization kinetics were found and an average growth exponent of 3 was determined. The temperature dependence of recrystallization was characterized by abnormally high activation energies which reflected the segregation of impurity atoms to the migrating boundaries during recrystallization.

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I. INTRODUCTION

There has been relatively little systematic investigation of the reverse martensitic transformation or its effect on the austenite produced by it. The greater part of the pertinent literature is of recent Russian origin. It is the purpose of this thesis to collect and review this information and to investigate with as broad a scope as possible the behavior and structure the austenite produced by one or more cycles of direct and reverse martensitic transformation.

In particular, the stabilization of the reversed austenite with respect to subsequent martensitic transformation, the strengthening of the reversed austenite and the resulting martensite, and the nature and kinetics of the recovery and recrystallization of the reversed austenite will be systematically investigated in low carbon iron-nickel alloys containing 29.5 - 32.5 atomic percent nickel.

It is hoped that the knowledge accumulated will serve as a useful guide for further theoretical investigation of specific aspects of this phenomenon and for possible practical utilization of the controlled introduction of reproducible structural changes within a single phase by means of cyclic phase transformation.
II. LITERATURE REVIEW

The literature was examined to find all the investigations dealing with the effect of the reverse martensitic transformation upon the resulting austenite. To date, the Russian literature has been almost the only source of information on this phenomenon, and the pertinent investigations will be reviewed in detail in this section. Table I presents a brief summary of the principal Russian investigations of the reverse martensitic transformation. The range of ferrous alloys investigated is quite large, but on the whole, alloys with $M_S$ temperatures close to or below room temperature appear to have been selected. Studies on alloys with and without carbon have been included.

One of the most important effects of the reverse martensite transformation is the stabilization of the reversed austenite with respect to subsequent direct martensitic transformation. Some Non-Russian references, not necessarily concerned with the reverse martensitic transformation, will be included in order to supplement the Russian work and to give a more complete picture of the pertinent types of stabilization.

Golovchiner and Tyapkin\(^1\) were among the first investigators to report stabilization of austenite after reversal of the martensitic transformation. They followed the cooling transformation of austenite in a 27 percent nickel alloy that had been reversed just above the $A_F$ temperature (the temperature at which the reverse martensite transformation is just completed,) and found that the $M_S$ was lowered 30°C. However, for a similar alloy containing 1.5 percent titanium the $M_S$ was raised by 90°C after an identical treatment. In both alloys, the $M_S$ gradually returned to its original value as the reversal temperature was increased; the recovery was
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believed to be due in part to recrystallization. Differences in the strength of stabilization were observed after heating in air furnaces and in lead baths, the effect being greater after the slower heating. The greatest effect of heating rate was produced on heating below the $A_f$ temperature. The authors assumed tentatively that the stabilization was due to the reduction in size of the mosaic structure in the austenite, but did not offer any explanation for the increase in $M_s$ for the titanium alloy.

Maksimova and Nikonorova\(^{(2)}\) metallographically studied the stabilization in a 31.7 percent nickel, 5.5 percent cobalt alloy resulting both after a single reversal and after two cycles of transformation. The stabilization accompanying the latter treatment was referred to as "superstabilization." The austenite produced after reversal was found to contain needle-like regions which were quite sensitive to etching. As the reversal temperature was increased above the $A_f$ temperature, this sensitivity gradually diminished, and after recrystallization it had completely disappeared. Significantly smaller amounts of martensite than the 90 percent obtained on cooling annealed austenite to $-60^\circ C$ were observed when the "chemically active" austenite was cooled to the same temperature. A second cycle of stabilization caused complete stabilization at $-60^\circ C$ and the small amount of reversed austenite that had undergone transformation a second time was even more sensitive to etching than before. These authors, as did Golovchiner and Tyapkin\(^{(1)}\), pointed out that stabilization was not due to a change in the chemical composition of the austenite by diffusion processes at the reversal temperature.

Vinogradskaya and Kreslina\(^{(3)}\) found that maximum stabilization (minimum formation of martensite at $-196^\circ C$) of reversed austenite occurred just after
the completion of the reverse martensite transformation at 630°C and that the strength of stabilization was independent of the heating and cooling rates used above room temperature. However, rapid cooling below room temperature prior to reversal increased stabilization on subsequent cooling to liquid nitrogen temperature (above that resulting after slow cooling) and increased the reversal temperature for maximum stabilization to 680°C. The strains produced in austenite during the direct and reverse martensitic transformation were held responsible for the stabilization phenomenon. It was found that relief of these strains (by an unspecified mechanism) during superheating above the $A_t$ temperature restored the original ability of the austenite to transform on subsequent cooling.

Gridnev, Cherepin, and Chernenko\textsuperscript{(4)} used the magnitude of the contraction accompanying the reverse martensitic transformation during heating as an indicator of stabilization in a 21.5 percent nickel, 3 percent manganese, low-carbon alloy. The greatest decrease in the amount of austenite taking part in the reverse transformation came after the first cycle of transformation, and full stabilization was approached but not quite reached after 5 cycles.

Borodin, Malyshev, and Mirtel'stein\textsuperscript{(5)(6)} systemically investigated the effect of carbon on the stabilization of austenite in a 13.7 percent nickel, 9.7 percent chromium, 0.05 percent carbon alloy. They found that stabilization occurred during the early stages of heating austenite (that had not been subjected to martensitic transformation) at 300°C, 400°C, and 500°C, but that destabilization occurred after holding for longer times at these temperatures. No visible separation of a carbide phase was observed after these treatments. After decarburization no evidence of stabilization was found on heating at these same temperatures, and the authors
concluded that an internal redistribution of carbon in austenite was responsible for the stabilization observed.

The relationship of carbon to the stabilization of austenite not subjected to any reversal of the martensitic transformation has been studied by several investigators. All the theories which have been evolved rely on the diffusion of interstitial carbon and/or nitrogen atoms to preferred sites within the parent austenite or at the austenite-martensite interface. Once concentrated at these sites the atoms reduce the ability of martensite to nucleate or to propagate if it has already nucleated. Edmonson\(^{7}\) has reviewed these theories and investigated the relationship of the strength of stabilization to a time-temperature dependent aging above room temperature in a 10 percent nickel, 1 percent carbon steel. He concludes that the stabilization is due to interstitial diffusion and that the material composition determines the actual mechanisms of stabilization.

The importance of carbon and nitrogen in the stabilization of the athermal kinetics of martensitic transformations (in several high-carbon steels and a 30 percent nickel-iron alloy) has been stressed by Crussard and Philibert\(^{8}\). Woodilla, Winchell, and Cohen\(^{9}\) have related the interstitial diffusion of carbon through martensite to stabilization of the isothermal martensitic transformation in a 31 percent nickel-iron alloy.

Borodina et al\(^{5}(6)\) also investigated the effect of the reverse martensitic transformation on the stabilization of a decarburized iron-nickel-chromium alloy. It was found that stabilization did occur. This stabilization, however, was much weaker than that due to carbon and was temporary in nature due to a strong isothermal martensitic reaction
at room temperature which caused the amount of martensite formed to approach
the amount formed in an annealed sample cooled from 1100°C. Stabilization
accompanying reversal was also found in iron-nickel-chromium-manganese
alloys but not in iron-nickel alloys. The authors ascribed this behavior
to difference in the magnitude of local stresses ("stresses of the second
kind") caused by the different compositions of the alloys.

In relation to this type of stabilization, other investigations have
shown that considerable plastic deformation is introduced in the austenite
surrounding martensite plates formed during cooling. Edmonson and Ko\(^{10}\)
in a 34 percent nickel-iron alloy observed slip lines in austenite
adjacent to martensite plates, both during the direct and reverse
martensitic transformation, and Kelley and Nutting\(^{11}\) by means of thin
foil transmission microscopy observed dislocation densities as high as
\(10^{11}\) or \(10^{12}/\text{cm}^2\) in the vicinity of martensitic plates formed in an iron-
nickel-carbon alloy. Not only the austenite was deformed, but deformation
in the martensitic plates was also observed. The result of this severely
distorted austenite lattice is the retardation or stabilization of subsequent
martensitic transformation both during further cooling and after reversal.
Edmonson and Ko demonstrated the effectiveness of this deformed austenite
in impeding transformation when they found that martensite plates formed
after reversal were fragmented and occupied sites different from the ones
they had occupied when formed from annealed austenite. Gaunt and
Christian\(^{12}\) found that the cubic-hexagonal transformation in cobalt-
nickel alloys was completely stabilized from above room temperature to
liquid air temperature after several cycles of transformation due to
hardening of the cubic matrix by stresses arising from the shape-change
accompanying transformation.
Kelley and Nutting\textsuperscript{(11)} have also examined the effect of stacking faults in causing both stimulation and stabilization of the martensitic transformation after small strains have been introduced by plastic deformation. In stainless steel, they have shown that stacking faults can act as nuclei for martensitic plates. Therefore, if stacking faults are produced by plastic deformation and if the matrix surrounding these faults does not exert a large stabilizing influence because of a high dislocation density, then the martensitic transformation will be stimulated. For larger strains, the more severely cold-worked matrix apparently reduced the effectiveness of the stacking faults in stimulating martensite nucleation. Otte\textsuperscript{(13)} has gathered experimental evidence for the presence of stacking faults in various ferrous alloys and there seems to be a good correlation between the faulting tendency of an alloy and the stimulation of the martensitic transformation after small amounts of plastic deformation. Iron-nickel-carbon, iron-chromium-carbon, and iron-nickel-chromium alloys all have a tendency to faulting and all show some stimulation of the martensitic transformation for small strains, while iron-nickel alloys in which little evidence for faulting now exists showed stabilization effects after 5 percent strain\textsuperscript{(31)}. The correlation for iron-nickel alloys, however, is inconclusive since a later investigation\textsuperscript{(28)} showed that the $M_s$ of a 30 percent nickel-iron alloy was significantly raised by cold work.

Another important effect of the reverse martensitic transformation is the marked increase in strength it produces in reversed austenite that has undergone one or more cycles of transformation without appreciable recovery. Gridnev et al\textsuperscript{(4)} in the iron-nickel-manganese alloy they investigated found that the tensile strength and hardness of reversed
austenite formed by rapidly heating (1000°C/sec) a transformed structure containing 60 percent martensite just above the \( A_f \) temperature were only slightly lower than the tensile strength and hardness of the martensitic structure and significantly higher than the same properties in annealed austenite. As the temperature of reversal increased, the tensile strength and hardness gradually decreased, approaching the values for annealed austenite. However, even after heating to 1400°C some strengthening due to the reverse transformation was retained. An investigation of the rate of heating on the strength of reversed austenite showed that hardness after reversal at 600°C increased slightly as the heating rate was increased from 10°C/sec to 10,000°C/sec and that the tensile and yield strengths were relatively insensitive to heating rate. The temperature range over which the reverse martensitic transformation occurred also did not appear to be sensitive to changes in heating rate.

Malyshev et al.\(^{(5)}\) investigated the strengthening of austenite produced after the reverse martensitic transformation as a function of cycles of transformation in a number of alloys (see Table I), and used the term phase hardening to refer to this type of strengthening. The largest increase in strength over the strength of annealed austenite occurred after the first cycle of transformation and further cycles caused much smaller increases in strength of the reversed austenite. The degree of strengthening of the austenite was found to be related to the amount of austenite involved in the direct and reverse martensitic transformation, and the strengthening was attributed to a break-up of the mosaic block structure in the austenite.

All of the measurements of mechanical properties discussed above were made at room temperature after reversal. Blanter and Mashkov\(^{(14)}\), however,
have measured the change in microhardness at temperature through the
temperature range over which the reverse martensitic transformation
occurs in an iron-nickel-manganese-carbon alloy. From room temperature
to about 400°C (120°C below the $A_s$ temperature) there is a continuous
decrease in the hardness of the transformed structure containing 50 percent
martensite. A sharp unexplained increase in hardness was then observed
in the temperature interval from 400°C to the $A_s$. During the reverse
martensitic transformation (520°C to 600°C) the hardness decreased as
expected until 580°C. From this temperature to the $A_r$, the hardness
increased slightly before again decreasing as temperature was raised,
and approached the value for austenite not having undergone any transformation.
The observation that the martensite-austenite mixture formed during the
latter stages of the reverse transformation was softer than just as-reversed
austenite alone was considered an interesting and significant effect of
phase hardening by the authors, but was not explained any further.

One of the aspects of the reverse transformation that has not been
studied in any detail is the process by which reversed austenite can
regain the properties of annealed austenite. Recrystallization as a
process of recovery in properties has received little attention beyond
mere mention in most of the published articles. The most detailed study
was made by Gridnev et al.\(^1\) when they followed the hardness changes in
reversed austenite as a function of time at three temperatures above the
$A_r$. Hardness dropped significantly with time at each temperature and an
activation energy of 35,000 cal/mole was calculated for the softening
process. This value might be doubtful because the curves shown for the
three temperatures were quite different in shape and the final values of
hardness reached at each temperature after identical times at temperature were significantly different. Recrystallization was not mentioned to account for the softening.

Metallographic studies of the microstructure of austenite formed after the reverse martensitic transformation were performed in some of the investigations already discussed. Edmonson and Ko\textsuperscript{(10)} found that a distorted, rippled surface remained in the reversed austenite where martensitic plates had existed prior to reversal, and Maximova and Nikonorova\textsuperscript{(2)} have shown the greatly increased sensitivity of reversed austenite to etching as compared to annealed austenite. It was possible for both of these investigations to be made at room temperature because of the sub-zero $M_s$ temperatures of the iron-nickel alloys investigated. In a more convention steel, direct observation of the relief accompanying the reverse martensitic transformation after heating to 700°C was made by Sokolov and Sadoskii\textsuperscript{(15)}. The shear-type nature of the reversal was confirmed by the relief, its needle-like appearance, and its rapid, simultaneous formation in a number of grains. Relief was not observed in specimens heated for two hours at 600°C where diffusion controlled reactions are expected to occur during tempering.

Diffusion in austenite that has undergone a cycle of martensitic transformation is another aspect of the reverse transformation that has received little attention. One revealing study was made by Gruzin, Kuznetsov, and Kurdumov\textsuperscript{(16)}, when they investigated the self-diffusion of iron in reversed austenite and annealed austenite in two iron-nickel-carbon alloys, and found that diffusion was significantly enhanced in the reversed structure. At 900°C, (about 200°C above the $A_f$ temperature) the diffusion coefficient for the reversed austenite was three times as large
as that in annealed austenite. The difference between the diffusion coefficient of the two structures decreased with increasing temperature until at 1000°C they were almost the same. This behavior was attributed to the substructure formed within the martensitic plates during the direct martensitic transformation and carried over into the austenite after the reverse transformation. Surface diffusion along the substructure boundaries was believed to take place readily, thus leading to the enhanced values of the self-diffusion coefficient of iron in the reversed austenite.
III. EXPERIMENTAL PROCEDURE

A. Alloy Compositions and Preparation

The compositions of the alloys used in this investigation are listed in Table II. Hereafter, the alloys will be identified only by their nickel content in atom percent. This group of alloys was selected for several reasons:

(1) The low $M_s$ temperatures of these alloys enable the austenite to be examined at room temperature both after annealing and after reversal of the martensitic transformation;

(2) The low carbon contents avoid the introduction of additional variables such as tempering reactions and stabilization due to carbon; and

(3) Several experimental and theoretical studies on the direct martensitic transformation in iron-nickel and iron-nickel-carbon alloys have been conducted by previous workers$^{(17)(18)}$ in this laboratory.

The analyses for the nickel determined by two different laboratories agreed within ±0.1 percent nickel, and the accuracy of the conductometric carbon analyses of the 29.4, 30.6, and 32.4 atom percent nickel alloys was estimated to be ±0.001 percent carbon.

The 29.7 percent nickel alloy was used only for preliminary experiments to determine the feasibility of the investigation of the reverse martensitic transformation in the iron-nickel system. Detailed study of its behavior after reversal was not performed, however, because of its relatively high carbon and manganese contents. Exact information on the preparation of this alloy is not available, but presumably it was air melted and hot rolled to 1-inch diameter rod.
<table>
<thead>
<tr>
<th>Atomic Percent</th>
<th>Ni</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>29.7</td>
<td>30.78</td>
<td>0.07</td>
<td>0.71</td>
<td>0.17</td>
<td>Balance</td>
</tr>
<tr>
<td>29.4</td>
<td>30.47</td>
<td>0.006</td>
<td>--</td>
<td>--</td>
<td>Balance</td>
</tr>
<tr>
<td>30.6</td>
<td>31.47</td>
<td>0.005</td>
<td>--</td>
<td>--</td>
<td>Balance</td>
</tr>
<tr>
<td>32.4</td>
<td>33.46</td>
<td>0.007</td>
<td>--</td>
<td>--</td>
<td>Balance</td>
</tr>
</tbody>
</table>
The 29.4, 30.6, and 32.4 atom percent nickel alloys were vacuum melted specifically for this investigation. Electrolytic iron and nickel were induction-melted in a stabilized zirconia crucible, held under a vacuum of 1 micron for ten minutes after melt-down, and deoxidized after that time by the addition of an excess of 0.025 percent carbon. The ingots were hot rolled at 1040°C, first to 2-1/4-inch diameter and then to 1/2-inch diameter rod. In order to remove the oxide layer and surface cracks that had formed during rolling, the bar stock was machined from 1/2-inch diameter to 7/16-inch diameter prior to annealing.

B. Heat Treatment

1. Austenitizing

Prior to the fabrication of the test specimens, all of the 7/16-inch diameter stock was homogenized for 24 hours at either 1000°C or 1100°C. This treatment, together with previous hot rolling, was considered sufficient as a starting condition for subsequent testing. Quenching in liquid nitrogen produced uniform distributions of martensite in all samples of the various alloys but one. This exception was in the 32.4 percent nickel alloy tensile bar stock. Large areas of retained austenite were observed on one side of some of the specimen cross sections. This effect might be accounted for by the combination of the very low Ms temperature for this alloy and the large mass of the samples. Lower nickel content alloys and smaller samples of the same alloy did not exhibit similar behavior.

All samples were austenitized in a resistance-wire tube furnace with a 6-inch long heating zone that remained within ±5°C at furnace temperatures between 1000°C and 1100°C. The samples were sealed into evacuated vycor tubing for the heating and were quenched into water without breaking the vycor.

The grain size developed after the homogenizing treatments is given in Table III.
### TABLE III

**Austenitic Grain Size Developed After Austenitizing**

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Austenitizing Temperature</th>
<th>ASTM Grain Size No.</th>
<th>Average Grain Diameter in Microns</th>
</tr>
</thead>
<tbody>
<tr>
<td>32.4</td>
<td>1000°C</td>
<td>2-3</td>
<td>120</td>
</tr>
<tr>
<td>30.6</td>
<td>1100°C</td>
<td>4-5</td>
<td>81</td>
</tr>
<tr>
<td>29.4</td>
<td>1100°C</td>
<td>4-5</td>
<td>100</td>
</tr>
</tbody>
</table>
2. Selection of Reversal Temperatures

A criterion of reversibility proposed by Cohen(19) states that the martensitic transformation should completely reverse itself while still in the potential equilibrium two-phase ferrite plus austenite region. If complete reversal to austenite occurs on heating into the region where ferrite is one of the stable phases, then a displacive shear reaction similar to the process of direct martensitic transformation on cooling is likely to be the mechanism of reversal, and the diffusion-controlled reaction necessary to form stable ferrite has not occurred. The latter reaction must necessarily occur with longer times at temperatures within the two-phase region, but the sluggish substitutional diffusion in iron-nickel alloys as observed by Owen and Liu(20) and analyzed by Kaufman(17) gives the martensite sufficient time to reverse itself completely to austenite.

The portion of the iron-nickel equilibrium diagram determined by Owen and Liu(20) which shows the boundaries of the austenite plus ferrite region is reproduced in Figure 1. The $A_f$ temperatures (temperatures at which the martensite-to-austenite reversal is just completed) were determined by measurement of electrical resistance versus temperature. The standard reversal temperatures were selected to fall above the $A_f$ temperatures, but within the ferrite and austenite region. Table IV gives the $\gamma\rightarrow\alpha$ boundary temperatures, the $A_f$ temperatures, and the selected standard reversal temperatures.

Standard reversal consisted of heating rapidly in a salt bath, holding for a total time of two minutes, and water quenching. The absence of ferrite or martensite after reversal was confirmed by the absence of body-centered cubic lines in X-ray diffraction patterns of the samples and by return
<table>
<thead>
<tr>
<th>Alloy</th>
<th>$A_r (^{\circ}C)$</th>
<th>Boundary ($^{\circ}C$)</th>
<th>Standard Reversal Temp. ($^{\circ}C$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>32.4</td>
<td>402</td>
<td>467</td>
<td>450</td>
</tr>
<tr>
<td>30.6</td>
<td>445</td>
<td>481</td>
<td>475</td>
</tr>
<tr>
<td>29.4</td>
<td>469</td>
<td>490</td>
<td>475</td>
</tr>
</tbody>
</table>
FIG. 1  A PORTION OF THE IRON-NICKEL EQUILIBRIUM PHASE DIAGRAM (AFTER OWEN AND LIU)
of the electrical resistance to values obtained for the same samples in
the well-annealed condition known to consist of 100 percent austenite.

3. Reversal and Recrystallization Treatments

Standard reversals (450-475°C) were performed in salt baths and
recrystallization treatments (600-700°C) in molten lead baths. Temperatures
were measured with a chromel-alumel thermocouple which had been checked
against a calibrated couple. The temperature fluctuation of the pots at
temperature was approximately ±2°C but uncertainty of the exact calibration
of the thermocouple increased the probable error to ±4°C.

The time for the cylindrical 7/16-inch diameter by 3/16-inch long
metallographic samples to reach temperature was determined experimentally
by the immersion of a thermocouple bead through a drilled hole to the
center of a sample. At 700°C, 13.2 seconds were required to bring the
temperature of the center of the sample to within 2°C of 700°C. All
reported times at temperature up to 10 minutes have been corrected for
the time to reach temperature.

4. Thermal Cycling

A cycle of transformation in which the initial structure of the
sample was either annealed austenite or as-reversed austenite consisted
of the following steps:

a. A quench below $M_s$ by immersion in liquid nitrogen,
b. A hold in liquid nitrogen for 30 minutes,
c. Rapid heating in salt bath through $A_s$ and $A_f$ to the standard
   reversal temperature,
d. A hold at the reversal temperature for 2 minutes,
e. Water quench to room temperature.
The final structure then consisted of as-reversed austenite. This procedure for cycling was generally followed throughout this investigation for the resistance and metallographic samples. For tensile specimens, a slight modification was made: the hold at the standard reversal temperature was increased to 5 minutes to allow for the heating of the greater mass of sample.

A schematic diagram for thermal cycling of the 32.4 atom percent nickel alloy is shown in Figure 2.

A cycle of transformation in which the initial and final structure was martensitic would be produced by reversal of the initial martensite to austenite (steps c,d, and e) and the subsequent transformation of this austenite to the final martensitic structure (steps a and b).

C. Experimental Techniques and Specimen Preparation

1. Electrical Resistance Measurements

Two-inch long by 0.0625-inch diameter samples to which nickel-wire current and potential leads were spot welded were used for measurement of electrical resistance as a function of temperature. The current leads were welded to the ends of the samples and the potential leads 0.250 inches from either end to produce a reference length of 1.5 inches.

The samples were prepared from quartered and rounded sections of the 0.4375-inch diameter stock; the sections were alternately swaged and annealed to 0.095 inches in diameter. All anneals were made in evacuated vycor at 1000°C. The samples were then centerless ground to 0.0625±0.0002 inches to remove any surface effects incurred during processing.

A Rubicon portable Kelvin bridge, Catalogue number 1622, was used for the resistance measurements. The readings could be estimated to 0.00001 ohm. The error in measurement varied from ±0.1 percent for austenitic samples to ±0.2 percent for martensitic samples.
FIG. 2

SCHEMATIC DIAGRAM FOR THERMAL CYCLING OF 32.4 ATOMIC PERCENT NICKEL-IRON ALLOY
Measurements of electrical resistance as a continuous function of temperature from 500°C to -196°C were made in a stainless steel chamber, a schematic diagram of which is shown in Figure 3. The chamber was placed in a resistance-wound tube furnace for measurements above room temperature and in an insulated vacuum flask for measurements below room temperature. A regulated flow of liquid nitrogen introduced into the flask lowered the temperature to -196°C at a rate of approximately 3°C per minute; use of a resistance-wound copper core surrounding the chamber in the flask returned the sample to room temperature. The heating rate above room temperature was maintained at 3.5°C per minute. Furnace cooling was used to return to room temperature after the maximum desired temperature had been attained. Furnace cooling gave a cooling rate of approximately 2.5°C per minute from 500°C to 300°C. Below this temperature range, the rate of diffusion in iron-nickel alloys is quite slow(17)(20).

Temperatures were measured with a chromel alumel thermocouple both above and below room temperature. Temperature gradients along the specimen were evaluated by introducing three thermocouples into the chamber, one at either end of the sample and one at the center. The temperature from one end to the other did not vary more than 2°C.

2. **Metallography**

The standard technique for preparation of metallographic specimens consisted of removal of approximately 0.005-inch by grinding and polishing through a series of cloth wheels with fine alumina and diamond abrasives. Etching was done by swabbing with an aqua regia solution consisting of equal parts of nitric acid, hydrochloric acid, and water.
FIG. 3 SCHEMATIC DIAGRAM OF CHAMBER USED FOR ELECTRICAL RESISTANCE MEASUREMENTS
Electropolishing of some of the samples after polishing through a No. 9 diamond paste wheel was also performed to insure removal of any possible surface distortion due to mechanical polishing. A 5 percent perchloric-acetic acid electrolyte was used at a current density of 8 to 10 amperes per square inch.

Standard samples used expressly for hardness and metallography were cut from the 7/16-inch diameter stock and ground to produce parallel faces 3/16-inch apart.

Austenitic grain size was determined by comparison with ASTM standard grain sizes and by counting the number of grain boundary intercepts along a straight-line traverse across the sample. For the latter method, four traverses 0.250 inches long at 45° to one another were made across the specimens. A multiplying factor of 1.65, derived by Lement et al.\(^{(21)}\), was used to convert the average intercept to grain diameter.

Quantitative metallographic measurements were required in this investigation for the determination of retained austenite after transformation at -196°C, and for following recrystallization of reversed austenite by measurement of volume fraction recrystallized.

Volume fraction recrystallized was determined by a lineal analysis in which four 0.250-inch traverses at 45° to one another were made for each sample with the Hurlbut counter technique described by Howard and Cohen\(^{(22)}\). The recrystallized grains had two distinguishing features which made identification possible: 1) curved grain boundaries as opposed to straight boundaries of annealed and reversed austenite, and 2) clean, smooth-etching surfaces as opposed to the presence of surface
markings and irregular etching of the as-reversed grains. The latter distinguishing feature was often the only one present when recrystallization occurred by strain-induced boundary migration, and since careful examination was required to differentiate between the two types of surface within a single grain, the control of speed and focus possible during lineal analysis was necessary.

Point-counting to determine the volume fraction of austenite after transformation at $-196^\circ C$ was accomplished by superimposing a grid with 100 points per square inch over a photomicrograph of the structure taken at 500 magnifications. Hilliard and Cahn$^{(23)}$ have recently made a thorough statistical analysis of the merits of point-counting and lineal analysis and find that the most efficient analysis is point-counting with a two dimensional grid providing that no more than one grid point falls on each particle of the phase to be measured. Higher point densities will increase the labor required for counting, but will not significantly increase accuracy. This criterion was met for the 30.6 percent nickel alloy which contained about 5 percent retained austenite but was not satisfied by the 32.4 percent alloy which contained about 40 percent austenite after transformation.

3. Lattice Parameter Measurements

Debye-Scherrer patterns of annealed and stabilized specimens were obtained in an 11.4 centimeter diameter camera with 15 to 30 minute exposures to unfiltered iron radiation produced by an iron point-focus tube operated at 30 kilovolts and 10 milliamperes. The samples were prepared by etching the 0.0625-inch diameter resistance samples in aqua regia to a diameter between 0.020 and 0.025 inches. Lattice parameters were calculated for all lines with $\theta$ greater than 60$^\circ$ and were plotted against $\cos^2\theta$. The true lattice parameter was then obtained by straight
line extrapolation to $\cos^2 \theta = 0$. The best accuracy which could be obtained was ±0.001A or 0.03 percent of the lattice parameter.

4. Mechanical Properties

The two types of 0.250-inch diameter tensile specimens are shown in Figure 4. The 1-inch gage length sample was used for the 29.4, 30.6, and 32.4 percent nickel alloys to conserve the limited amount of material available.

Samples for the 29.7 percent nickel alloy were transformed and reversed after machining, while samples of the remainder of the alloys were first treated and then machined. The latter procedure was followed to facilitate machining of these relatively soft alloys by having them in a condition other than full annealed, and to eliminate dimensional changes, bending, and surface contamination of the finished samples that might occur during cyclic transformation.

A 60,000-pound hydraulic Baldwin Southwark Universal Testing machine equipped with a microformer extensometer and autographic recorder was used to obtain load-elongation curves. The 0 to 6000-pound scale was sufficient in most cases and the rate of straining was approximately 0.02 inches per inch per minute.

In order to follow the early stages of plastic deformation in the 29.7 percent nickel alloy, two SR-4 strain gages, type 1-12, were mounted in series on opposite sides of each sample. The samples were loaded in 100-pound increments and the residual plastic deformation after release of the load was measured. Temperature fluctuations were compensated for by use of a dummy specimen on which two identical strain gages were mounted. A Baldwin Strain Indicator Type N was used to measure strain to within $1 \times 10^{-6}$. 
FIG. 4  TENSION TEST SPECIMENS
(a) SHOULDERED-END
(b) THREADED-END
5. Magnetic Measurements

Magnetic measurements were made to detect any martensite which might have formed during tensile testing of austenitic samples. Samples 1 cm long and 2 mm in diameter were machined from the elongated gage length and from the undeformed shoulder of the tensile bars. The latter samples were used to calibrate the measurements by determination of intensity of magnetization, \( J \), both in the austenitic condition, \( J_A \), and after transformation at 196°C, \( J_{-196°C} \). The increase in intensity of magnetization over the value for austenite was assumed to be directly proportional to the volume percent martensite introduced. Percent martensite was then obtained from the following relation:

\[
\%M = \frac{J_A + M - J_A}{J_{-196°C} - J_A} \times \%M_{-196°C}
\]

This method at the present time is estimated to be sensitive to amounts of martensite above 1 percent.

Measurements were made by placing the sample in a brass holder between the poles of a 5000-gauss permanent magnet and recording the deflection of a ballistic galvanometer produced as a search coil was quickly passed along the specimen holder from a position not over the sample to a position over the sample. This deflection was caused by a current produced by the motion of the search coil as it cut through the asymmetrical magnetic field caused by the presence of the specimen between the poles of the magnet. This current was directly proportional to the intensity of magnetization. More complete specifications of the apparatus used in this investigation are available from D. Hoffman\(^{(24)}\) of this laboratory and a brief discussion of a similar apparatus is presented by Schaller and Zackay\(^{(25)}\).
D. Transformation Temperatures

The critical transformation temperatures for the direct and reverse martensitic transformations were obtained from the well-defined changes in slope of the electrical resistance vs. temperature curves as martensite formed from austenite or reverted back to it. The results of these measurements for the 29.4, 30.6, and 32.4 percent nickel alloys used in this investigation are tabulated in Table V. Results reported by Kaufman(17) for three alloys of almost the same compositions are also tabulated for comparison. The agreement is fairly good, especially for the $A_s$ temperatures, within the ±10°C deviations reported by Kaufman. Table V shows the influence of nickel on the martensitic transformation in iron-nickel alloys; both $M_s$ and $A_s$ decrease with increasing nickel content.

The $M_s$ values determined in this investigation and by Kaufman are graphically compared in Figure 5. From the results of this investigation it can be seen that the reproducibility of $M_s$ determinations was much better for high $M_s$ temperatures. Another observation made for the 32.4 percent nickel alloy was that the amount of martensite formed during the burst of transformation increased with decreasing $M_s$ observed.

The discrepancy between these two investigations is difficult to resolve especially since the same experimental technique was used and the alloys were of similar purity with low carbon contents. The situation is further complicated by $M_s$ temperatures of -14°C, -40°C, and -105°C reported for 29.4, 30.6, and 32.4 percent nickel alloys respectively by Zackay(26). The experiment technique used for the $M_s$ measurements, however, was not reported by this author. A possible explanation for these discrepancies might be sample compositions differing from the stated nominal values.
TABLE V

Transformation Temperatures of Some Iron-Nickel Alloys

<table>
<thead>
<tr>
<th>Composition (Percent Nickel)</th>
<th>$M_S$ ($^\circ$C)</th>
<th>Martensite at -196$^\circ$C</th>
<th>$A_S$ ($^\circ$C)</th>
<th>$A_f$ ($^\circ$C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>29.4</td>
<td>-21±3</td>
<td>95%</td>
<td>386</td>
<td>469</td>
</tr>
<tr>
<td>29.3*</td>
<td>-30</td>
<td>95%</td>
<td>390</td>
<td>442</td>
</tr>
<tr>
<td>30.6</td>
<td>-52±6</td>
<td>95%</td>
<td>342</td>
<td>445</td>
</tr>
<tr>
<td>30.75*</td>
<td>-72</td>
<td>95%</td>
<td>335</td>
<td>420</td>
</tr>
<tr>
<td>32.4</td>
<td>-124±9</td>
<td>60%</td>
<td>307</td>
<td>402</td>
</tr>
<tr>
<td>32.4*</td>
<td>-146</td>
<td>80%</td>
<td>315</td>
<td>416</td>
</tr>
</tbody>
</table>

* Alloys investigated by Kaufman
Fig. 5 Ms temperature versus nickel content for iron-nickel alloys.
Brook, Entwisle, and Ibrahim\(^{(27)}\) have pointed out that one percent of nickel lowers the \(M_s\) by approximately 15°C while one percent carbon lowers the \(M_s\) by 225°C. Small variations in carbon content, produced either by inhomogeneity or heat treatments where precipitation could take place, would then have a strong effect on the measured \(M_s\).

Grain size has also been found to influence strongly the initiation of transformation in iron-nickel alloys. McReynolds\(^{(31)}\) reported an increase of 30°C in the \(M_s\) of a 29 percent nickel-iron alloy as the grain size was increased from 0.025 mm to 0.25 mm while Macklin and Cohen\(^{(28)}\) observed no change in \(M_s\) but an increase of 40°C in the burst temperature of a 29.5 percent nickel-iron alloy as the grain size increased from ASTM Grain Size Number 9 to an ASTM Grain Size of 4-5. The grain size factor, however, does not seem to resolve the present difference since the grain sizes were approximately the same, 0.1 mm, in both Kaufman's and in this investigation.
IV. PRESENTATION AND DISCUSSION OF RESULTS

A. Stabilization after Slow Reversal

Both the direct and reverse martensitic transformations in the 29.4, 30.6, and 32.4 percent nickel alloys were followed by the measurement of electrical resistance as a function of temperature in an effort to evaluate the stabilizing influence of cyclic reversal on the austenite formed after reversal. The complete hysteresis loops for the heating and cooling transformation were obtained by measurement of the electrical resistance of specimens during heating and cooling at relatively slow rates of 4°C/min.

The results of these measurements are shown in Figures 6, 7, and 8 for the 29.4 percent, 30.6 percent, and 32.4 percent nickel alloys respectively. The behavior of all the alloys with increasing numbers of transformation cycles was similar; accordingly, Figure 7 for 30.6 percent nickel alloys will be used as the basis for a detailed discussion of this behavior. The first cooling run was performed on the sample after it had been austenitized at 1000°C. The electrical resistance decreased linearly with decreasing temperature until at the $M_s$ temperature (~-44°C) a slight deviation from linearity marked the formation of the first martensite. An extremely sharp drop of resistance associated with a "burst" of very rapid athermal transformation occurred somewhat below the $M_s$. Martensite continued to form as the temperature was lowered, but at a decreasing rate, until at ~-196°C about 95 percent martensite had formed. On heating the transformed specimen back to room temperature, a slight curvature of the resistance versus temperature curve was observed. This is believed to be associated with the formation of martensite by isothermal transformation.
RESISTANCE-TEMPERATURE CURVES FOR 29.4 ATOM PERCENT NICKEL-IRON ALLOY

KEY: CIRCLED NUMBERS INDICATE SEQUENCE OF COOLING AND HEATING RUNS

EXPERIMENTAL DETAILS:
SAMPLE LENGTH: 1.5 IN
SAMPLE DIAMETER: 0.0625 IN.
HEATING RATE ABOVE 20°C: 4°C/ MIN
COOLING RATE BELOW 20°C: 3°C/ MIN

FIG. 6 STABILIZATION OF 29.4 ATOM PERCENT NICKEL-IRON ALLOY BY SUCCESSIVE CYCLES OF FORWARD AND REVERSE MARTENSITIC TRANSFORMATION. SLOW HEATING TO AND COOLING FROM 500°C.
FIG. 7  STABILIZATION OF 30.6 ATOM PERCENT NICKEL-IRON ALLOY BY SUCCESSIVE CYCLES OF FORWARD AND REVERSE MARTENSITIC TRANSFORMATION. SLOW HEATING TO AND COOLING FROM 500°C.
FIG. 8  STABILIZATION OF 32.4 ATOM PERCENT NICKEL-IRON ALLOY BY SUCCESSIVE CYCLES OF FORWARD AND REVERSE MARTENSITIC TRANSFORMATION. SLOW HEATING TO AND COOLING FROM 450°C.
in this temperature range. The first heating run above room temperature marked the beginning of the reverse martensitic transformation at about 340°C ($A_s$) and its completion at 445°C ($A_f$). Cooling back to room temperature completed the first cycle of transformation.

As shown in Figure 7, several important differences in the transformation behavior of the austenite produced after the first cycle of transformation were observed during the second cooling run. There was a significant decrease in $M_s$ temperature (from -44°C to -66°C); no sharp burst of martensitic transformation occurred; and the higher resistance of the specimen at -196°C indicated a smaller amount of transformation after completion of cooling at -196°C. All of these changes indicated that the austenite had somehow been stabilized with respect to subsequent direct martensitic transformation as a result of the preceding cycle of transformation. There was one other important difference between the first and second cooling run. This was the decreased resistance of the specimen at room temperature after it had been cooled from above the $A_f$ temperature.

Further cycles of direct and reverse transformation established the behavior observed during the second cooling run. The resistance of the specimen at room temperature was lower after each successive cycle of transformation; the $M_s$ continued to decrease; and smaller and smaller amounts of martensite were formed after cooling to -196°C. The decrease in resistance after the completion of the reversed martensitic transformation and the increased resistance at -196°C due to the stabilization of the direct transformation tended to decrease the width of the transformation hysteresis loop as the cycles of transformation increased. Finally, after 4 cycles of transformation, complete stabilization on heating to -196°C was observed, and the hysteresis loop degenerated into a single line. The same curve of resistance vs. temperature was followed on the fifth cooling from room
temperature to -196°C and on heating back to room temperature. A single curve was similarly found for both heating to above the \( A_f \) and for cooling back down to room temperature.

Figure 9 summarizes the stabilization behavior for the three alloys as a function of cycles of transformation. The parameter selected for a measure of the stabilization was the magnitude of the hysteresis loop at 20°C. This parameter therefore included the "residual" stabilization at room temperature after cooling from above the \( A_f \) temperature as well as the decreased amount of martensite formed during the direct transformation on cooling to -196°C. The percent stabilization was then calculated from the following formula:

\[
\text{Stabilization (percent)} = \frac{\Delta R_{\text{first cycle}} - \Delta R}{\Delta R_{\text{first cycle}}} \times 100
\]

where \( \Delta R_{\text{first cycle}} \) is the electrical resistance of the specimen at room temperature prior to the first direct martensitic transformation minus the resistance at room temperature of the transformed specimen prior to the first reverse martensitic transformation. \( \Delta R \) is the corresponding room temperature difference of electrical resistance for any successive cycle of transformation. The general form of the stabilization curves is similar among the three alloys. The stabilization increases gradually for the first two cycles and then proceeds in larger increments until complete stabilization on cooling to -196°C occurs after four or five cycles of transformation.

The decrease in \( M_s \) temperature to -196°C with increasing cycles of transformation is shown in Figure 10 for the three alloys. The decrease in \( M_s \) is almost directly proportional to the number cycles of transformation.
FIG. 9 STABILIZATION OF IRON-NICKEL ALLOYS AS A FUNCTION OF CYCLES OF TRANSFORMATION. SLOW HEATING AND COOLING DURING TRANSFORMATION
FIG. 10 $M_s$ TEMPERATURE VS CYCLES OF TRANSFORMATION FOR IRON-NICKEL ALLOYS SUBJECTED TO SLOW HEATING AND COOLING RATES DURING TRANSFORMATION
in the 29.4 and 30.6 percent nickel alloys, while the initially low $M_S$
temperature of the 32.4 percent nickel is much less sensitive to the early
cycles of transformation and only drops sharply during the cycle that
causes complete stabilization.

The key to the stabilization accompanying cycles of direct and reverse
martensitic transformation proved to be the marked decrease in resistance
at room temperature of the reverse samples after each complete cycle of
transformation. This decrease became greater with successive cycles of
transformation and was more pronounced at a given level of stabilization
as the nickel content of the alloys decreased. Debye-Scherrer patterns
of reversed samples that had exhibited this resistance-decrease revealed
that diffraction lines of a body-centered cubic phase were present. This
phase could have been either martensite or ferrite, since both are body-
centered cubic in iron-nickel alloys with very low carbon contents.
However, martensite should not have been present because complete
reversibility of the martensitic transformation by heating above $A_T$
had been demonstrated in other experiments of this investigation, and ferrite
would not be expected to be present, even though the heating and cooling
took place through the equilibrium two-phase ferrite plus austenite
region. The basis for the latter statement is the well-known sluggish
formation of ferrite from homogenized austenite in the temperature region
where ferrite should coexist with austenite at equilibrium (see Figure 1).
This behavior has been described in detail by Owen and his coworkers\textsuperscript{(20)(29)}
who, in their efforts to determine the equilibrium iron-nickel diagram,
held specimens at 350$^\circ$C for well over a year and at 400$^\circ$C for 124 days
to develop the equilibrium ferrite plus austenite structures. More
recently, Zakhorova et al\textsuperscript{(30)}, in a study of the mechanism of ferrite
formation from austenite in 30 and 32 weight percent nickel-iron alloys, found that no detectable amounts of ferrite had formed after 200 hours at 400°C. In the present investigation, the electrical resistance versus temperature curves were determined for annealed austenitic specimens both during heating and cooling from 500°C, the latter being the maximum reversal temperature used to study the stabilization phenomenon. No transformation to ferrite was detected and the same resistance curve was followed both during heating and cooling (see curved marked 1 in Figure 6). Kaufman\(^{(17)}\) has shown that the retarded formation of ferrite from austenite can be related to the extremely low rate of nickel diffusion through both face-centered cubic and body-centered cubic lattices in iron-nickel alloys at temperatures below 500°C. Ferrite formation can, however, be greatly accelerated by special treatments. Owen and Liu\(^{(20)}\) cold worked their austenitic samples prior to annealing to accelerate the approach to equilibrium while Zakhorova et al.\(^{(30)}\) found that samples subjected to the direct martensitic transformation and cold worked at room temperature produced the expected equilibrium amount of ferrite when heated for only 6 hours at 400°C.

In view of this evidence, there was the possibility that the body-centered cubic phase formed during the reversal cycle described here was ferrite. If ferrite did form, nickel would have to diffuse from the ferritic regions to the surrounding austenite in order to approach the equilibrium composition requirements shown in the iron-nickel phase diagram. The nickel-enriched austenite would then possess a lower \(M_s\) (see Figure 5) and produce the stabilization of the martensitic transformation in the reversed austenitic structure that was observed.
The lattice parameter of the austenite would be expected to increase with increasing redistribution of nickel atoms from the ferrite to the austenite. In order to detect such changes in lattice parameters that might have occurred during stabilization, the lattice parameters of both well annealed specimens and fully stabilized specimens were carefully determined and compared. The results of these experiments and the lattice parameter data obtained by Owen and Sully\(^{(29)}\) are presented in Figure 11. It can be seen that the lattice parameter of the stabilized austenite is significantly larger than that of annealed austenite for the 29.4 and 30.6 atom percent nickel alloys, the difference increasing as the nickel content of the alloys decreases. These lattice-parameter changes are consistent with the increased amount of ferrite formation, and therefore the extent of redistribution of nickel, required to stabilize the lower nickel alloys. That the amount of ferrite increases in the lower nickel alloys is demonstrated by the larger decrease of resistance at room temperature with decreasing nickel content. The observed change in the lattice parameter of the austenite favors the presence of ferrite rather than martensite, since the formation of martensite involves no composition change and therefore should not cause any change in the lattice parameter of austenite.

Table VI shows the amount of ferrite present in the alloys investigated after complete stabilization. These estimates were based on the assumption that the decrease in resistance due to ferrite formation is directly proportional to the amount of ferrite formed. Also included in Table VI are the amounts of ferrite, calculated from the equilibrium diagram, that would be required to produce austenite with a nickel content high enough to give an \(M_s\) of \(-196^\circ\)C. Extension of the \(M_s\) versus percent nickel curve
FIG. 11 LATTICE PARAMETERS OF AUSTENITE IN IRON-NICKEL ALLOYS
### TABLE VI

**Calculated and Observed Amounts of Ferrite Present After Stabilization**

<table>
<thead>
<tr>
<th>Alloy (atom percent nickel)</th>
<th>Observed After Complete Stabilization</th>
<th>Calculated Amount of Ferrite Coexisting at Equilibrium with 33.2% Ni Austenite</th>
<th>Ratio of Second to Third Column</th>
</tr>
</thead>
<tbody>
<tr>
<td>29.4</td>
<td>32.1%</td>
<td>13.9%</td>
<td>2.3:1</td>
</tr>
<tr>
<td>30.6</td>
<td>22.4%</td>
<td>9.5%</td>
<td>2.4:1</td>
</tr>
<tr>
<td>32.4</td>
<td>6.6%</td>
<td>2.9%</td>
<td>2.3:1</td>
</tr>
</tbody>
</table>
(Figure 5) parallel to the curve determined by Kaufman showed that austenite containing 33.2 atom percent nickel would have an $M_s$ of $-196^\circ C$. The phase diagram shows that austenite of this composition would be formed by isothermal holding at $460^\circ C$ and would coexist with ferrite containing 5.8 percent nickel.

At first glance the large amounts of ferrite formed during stabilization seem to be inconsistent with the much smaller calculated amounts of ferrite required to cause complete stabilization in an equilibrium ferrite-austenite mixture. However, a closer examination of the experimental conditions and probable mechanism of ferrite formation from the reversed austenitic matrix resolves much of this inconsistency.

Experimentally, ferrite was formed in these transformed and reversed alloys during both continuous heating to and cooling from $500^\circ C$, not during an idealized isothermal holding at $460^\circ C$. If the maximum effective temperature of appreciable substitutional diffusion is taken at $300^\circ C$, then the specimens with the heating and cooling rates used, spent a total time of 150 minutes in the $300^\circ C$ to $500^\circ C$ temperature range per cycle. During this time, solution as well as formation of the ferrite could have taken place because of the changing requirements of equilibrium with changing temperature. Evidence for this solution can be seen in Figure 6 where the amount of ferrite present at $500^\circ C$ is smaller at the end of the sixth and seventh heating runs than at the end of the fourth and fifth heating runs. On return to room temperature, however, this difference has been largely eliminated by additional formation of ferrite during cooling. At temperatures below $460^\circ C$, appreciably more ferrite would be stable than the idealized amount tabulated in Table VI.
If the mechanism of ferrite formation is considered, it can be seen that concentration gradients must exist both within and around the ferrite nuclei as nickel is rejected. Therefore it is likely that only a portion of the austenite surrounding the ferrite nuclei is sufficiently enriched in nickel to be stabilized at -196°C and that the nickel content of the ferrite is higher than the approximately 6 percent nickel expected under equilibrium conditions. In order for stabilization to be generalized throughout the whole of the specimen under these conditions, a much larger percentage of ferrite, about 2.3 times as much, seems to be required for stabilization than under well-established equilibrium conditions. The change in lattice parameter verifies this argument.

If the composition of the stabilized austenite were uniformly 33.2 percent nickel or above, than the lattice parameters of the stabilized specimens should correspond to the lattice parameter of austenite containing this amount of nickel. The parameters observed, however, were much smaller than this value, despite the fact that they were significantly higher than those determined in annealed specimens.

The relative ease with which ferrite forms during the reversal of the martensitic transformation as compared to its formation from annealed austenite can be explained by the interaction of several factors. The fact that a body-centered cubic lattice already exists during reversal on heating means that the volume expansion accompanying nucleation of a body-centered cubic phase from a close packed face-centered cubic structure has already been accommodated. The importance of this effect has been demonstrated by Zakhorova et al.(30) who followed the nucleation of ferrite from austenite in single crystals of a 32 percent nickel-iron alloy at 400°C by means of metallographic and x-ray diffraction techniques. The

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1 Recent experiments have shown that ferrite also does not form from reversed austenite during heating to and cooling from above the Ast.
first ferrite nuclei formed only after 200 hours, and were shown to align themselves in layers along the (111) planes of the austenite. As the nuclei formed, stresses developed which eventually exceeded the elastic limit of the austenite at 400°C and caused a breakdown in coherency of the nuclei with the austenitic matrix. Zones of deformation were visible around the particles and eventually polygonization or recrystallization occurred in their vicinity. After the breakdown of coherency, the further growth of the ferrite was believed to be diffusion-controlled as opposed to the cooperative movement of atoms that the authors ascribed to the formation of the layer structure. Very little agglomeration of the ferrite occurred after holding for 800 hours at 400°C, and even after 3560 hours the ferrite particles were quite small. In view of this evidence it is not likely that ferrite forms from the reversed austenite during the relatively short times available for diffusion in this experiment.

Diffusion is another factor that is quite important in explaining the increased rate of ferrite formation from a martensitic structure. Nucleation most probably at first occurs by a subtle change of composition within the last portions of martensite to reverse. Here again the presence of a body-centered cubic phase is advantageous. The diffusion coefficient of nickel in ferrite, although quite small between 300 and 500°C is on the order of a factor of 10^4 greater than that in face-centered cubic iron-nickel austenite, and so nickel atoms would be transported more readily through the body-centered cubic martensite to the austenitic matrix as the high nickel martensite is converted to the low nickel ferrite.

In addition to more favorable conditions for diffusion if martensite is already present, atom mobility in the austenite itself is also enhanced by the reverse martensitic transformation. Cuzin et al. (16), as discussed
in Part II, page 12, have shown that the self-diffusion coefficient of iron in iron-nickel alloys is significantly greater in reversed austenite as a result of substructural imperfections than in well annealed austenite at the same temperatures. Their investigation was performed at temperatures well above the $A_f$. At temperatures below the $A_f$, as in the present investigation where recovery has not been as effective in restoring order to the lattice disrupted by the direct and reverse transformation, the enhancement would be expected to be even greater. In addition, the dislocations observed to be formed in the austenite adjacent to martensitic plates could act as sources and sinks for vacancies to aid in the movement of nickel atoms away from the nuclei and iron atoms toward the nuclei by substitutional diffusion.

As the number of cycles of transformation increase, both the increased amount of ferrite present and the continued disruption of the austenite lattice would tend to accelerate both ferrite formation and the partition of nickel between the ferrite and austenite, thereby accelerating the rate of stabilization. This type of behavior accounts for the shape of the curves in Figure 9. The resolution of some of the ferrite close to the $A_f$ during the latter cycles of reversal would explain the sigmoidal shape of the curve for the 29.4 percent nickel alloy.

The ferrite formed during reversal was identified by its etching characteristics. Normally the austenitic and transformed specimens had to be etched with aqua regia to develop structures in a reasonable length of time. The stabilized ferritic specimens, however, were attacked severely after etching for short times with both 3 percent and 6 percent nital. The microstructures of the stabilized specimens showed that small globular particles of ferrite were concentrated in the regions formerly
occupied by the martensitic plates as would be expected from the above discussion. Figure 12 shows this microstructure for the 32.4 atom percent nickel alloy. In the 29.4 and 30.6 atom percent nickel alloys the ferrite formation was much more general. The ferrite formation, then, can be seen to stabilize those regions which because of the presence of structural difference or strain embryos were most sensitive to martensitic transformation on cooling.¹

In summary, complete stabilization at -196°C of the structure produced by cycles of direct and reverse martensitic transformation was observed in 29.4, 30.6, and 32.4 percent nickel-iron alloys after four or five complete cycles of transformation. This stabilization was accounted for by the enrichment of austenite adjacent to ferrite particles and the consequent lowering of the Ms. The ferrite particles were believed to be nucleated from martensite during heating just prior to complete reversal because favorable conditions for diffusion existed and no volume change had to be accommodated. Once nucleated, the ferrite could grow both during heating to and cooling from above the Af at relatively slow rates.

B. Stabilization after Rapid Reversal

The influence of rapid reversal of the martensitic transformation on subsequent direct transformation in 29.4, 30.6, and 32.4 atom percent nickel-iron alloys was determined by measurement of electrical resistance of reversed specimens during cooling below room temperature. In the preceding sections, cycles of direct and reverse transformation have been shown to cause complete stabilization of austenite at -196°C. This stabilization was attributed to the diffusion-dependent enrichment of

¹ The partitioning of these preferred sites by ferrite nucleation and growth would account for reduction in the initial burst of martensitic transformation just below Ms.
1000x  6 percent nital

Figure 12. Rapidly etching particles of ferrite in a 32.4 atom percent nickel alloy completely stabilized after 5 cycles of transformation.
austenite in nickel content as ferrite formed during slow heating and
cooling through the temperature range where the reverse martensitic
transformation occurred. In order to eliminate any such diffusion-
controlled reactions, samples tested in the experiments to be described
here were rapidly heated by immersion in salt baths to standard reversal
temperatures just above the $A_p$, held for two minutes, and water quenched
to room temperature. This procedure allowed a clearer evaluation of the
influence of structural changes produced in austenite by the reverse
martensitic transformation on subsequent direct transformation. A
minimum of recovery from the effects of the reverse transformation were
expected to occur during the two-minute hold at the standard reversal
temperature. These temperatures, $450^\circ C$ for the 32.4 atom percent nickel
alloy, and $475^\circ C$ for the 29.4 and 30.6 atom percent nickel alloys, were
selected for the reasons discussed in Part III.

The resistance versus temperature curves obtained by cooling specimens
of the 29.4, 30.6, and 32.4 atom percent nickel-iron alloys are presented
in Figures 13, 14, and 15 respectively. Each figure shows the curve
determined for the specimen in the annealed condition and after each of
four successive cycles of direct and reverse martensitic transformation.
The curves under these conditions of rapid reversal are markedly different
from the ones obtained for the slow reversal in that the $M_s$ is either
unchanged or increased and little stabilization, if any, occurs at $-196^\circ C$.
Table VII summarizes the effect of rapid reversal on each of the three
alloys. Two opposing trends seem to be established as the nickel content
of the alloys increases: an increased enhancement of the initiation of
the direct martensitic transformation is observed while stabilization of
the final stages of transformation increases.
FIG. 15 COOLING TRANSFORMATION IN 29.4 ATOM PERCENT NICKEL-IRON ALLOY AFTER SUCCESSIVE CYCLES OF RAPID HEATING TO AND COOLING FROM 475°C.
RESISTANCE–TEMPERATURE CURVES FOR 30.6 ATOM PERCENT NICKEL–IRON ALLOY

- First Cooling – After 1000°C Anneal
- Second Cooling – After First 475°C Reversal
- Third Cooling – After Second 475°C Reversal
- Fourth Cooling – After Third 475°C Reversal
- Fifth Cooling – After Fourth 475°C Reversal

FIG. 14 COOLING TRANSFORMATION IN 30.6 ATOM PERCENT NICKEL–IRON ALLOY AFTER SUCCESSIVE CYCLES OF RAPID HEATING TO AND COOLING FROM 475°C.
RESISTANCE - TEMPERATURE CURVES FOR 32.4 ATOM PERCENT NICKEL - IRON ALLOY

- FIRST COOLING - AFTER 1000°C ANNEAL
- SECOND COOLING - AFTER FIRST 450°C REVERSAL
- THIRD COOLING - AFTER SECOND 450°C REVERSAL
- FOURTH COOLING - AFTER THIRD 450°C REVERSAL
- FIFTH COOLING - AFTER FOURTH 450°C REVERSAL

FIG. 15 COOLING TRANSFORMATION IN 32.4 ATOM PERCENT NICKEL - IRON ALLOY AFTER SUCCESSIVE CYCLES OF RAPID HEATING TO AND COOLING FROM 450°C.
# TABLE VII

**Effects of Rapid Reversal on Cooling Transformation**

<table>
<thead>
<tr>
<th>Alloy atom percent nickel</th>
<th>Change in $M_S$ from $M_S$ of Annealed Specimen</th>
<th>Stabilization at -196°C After Reversal</th>
</tr>
</thead>
<tbody>
<tr>
<td>29.4</td>
<td>slight decrease: -2°C after 4 cycles</td>
<td>none</td>
</tr>
<tr>
<td>30.6</td>
<td>increase: +11°C after 4 cycles</td>
<td>very slight</td>
</tr>
<tr>
<td>32.4</td>
<td>increase: +35°C after 2 and 3 cycles +22°C after 4 cycles</td>
<td>about 6.3 percent less martensite</td>
</tr>
</tbody>
</table>
Malyshev, Borodina and Mirtel'stein\(^{(5)(6)}\) have reported similar results in that they observed no stabilization in a 27.8 percent nickel-iron alloy after subjecting specimens to cycles of direct and reverse transformation. They also, however, observed stabilization after reversal in iron-chromium-nickel and iron-manganese-chromium-nickel alloys, and so from both their investigations and the present one it would seem that stabilization due to structural changes occurring in austenite following the reverse transformation are sensitive to the composition of the austenite.

One of the effects of the direct and reverse martensitic transformation discussed in Part II was the plastic deformation of austenite surrounding martensite plates. Although this deformation is quite local and non-homogenous in nature, it may be quite general throughout a specimen if the martensitic transformation approaches completion. For this reason it is instructive to compare the results of the experiments now under consideration with investigations of the effect of cold work on the initiation of the martensite transformation. Several investigations have been conducted on iron-nickel alloys with compositions close to those of the alloys used in this investigation. McReynolds\(^{(31)}\) has found that a strain of 5 percent produced by uniform elongation lowers the \(M_s\) of a 29 percent nickel alloy by 20°C. This result is contrary to that found by Machlin and Cohen\(^{(28)}\) who found an increase in \(M_s\) and burst temperature of both single crystals of a 31 percent nickel-iron alloy deformed by compression and polycrystalline specimens of a 29.5 percent nickel-iron alloy strained by wire drawing. The single crystals were deformed from 10 to 50 percent and an increase in the burst temperature from 8 to 17°C was observed. In the polycrystalline samples
the $M_s$ increased progressively as the deformation was increased to 40 percent while the burst temperature at first increased slightly and then decreased.

Two types of structural defects in face-centered cubic austenite have been considered as aids to the nucleation of martensite. Embryos consisting of a body-centered cubic lattice separated from the austenitic matrix by a regular array of dislocations have been proposed by Cohen (32) to exist well above the $M_s$ temperature. As temperature is lowered through the $M_s$, embryos of the critical size already present can grow spontaneously into martensite plates with a decrease in free energy. Experimental evidence for the martensitic embryos has been reported by Richman et al (33) in 28.0, 29.3, and 30.7 atom percent nickel-iron alloys. The embryos were observed by direct examination of thin films of the alloys by transmission electron microscopy. The observations of Machlin and Cohen were consistent with the embryo theory of martensite nucleation, the enhancement resulting from the formation or enlargement of embryos by the introduction of dislocation networks during the plastic deformation, and stabilization associated with a decreased burst temperature after larger amounts of cold work resulting from structural barriers to the effective growth of the smaller embryos.

Stacking faults are the other type of defect which have been proposed (11) as nuclei for martensite needles. The correlation of stacking fault tendencies of various alloys and enhancement of the martensitic transformation with small amounts of deformation are considered to confirm this proposal. It appears, however, that this analysis is based on incomplete evidence for the effect of cold work on iron-nickel alloys since the depression
of $M_S$ with cold work reported by McReynolds\(^{(31)}\) for a 29 percent nickel-iron alloy, in conjunction with the observed low stacking fault tendency of iron-nickel alloys, is used as an example which shows that stimulation of the martensitic transformation is not produced in alloys that do not fault readily on working. Machlin and Cohen\(^{(26)}\) in a later investigation, however, did find a significant stimulation of the initial stages of the martensitic transformation of alloys for an iron-nickel alloy of similar composition after cold working. In other ferrous alloys it appears that the stacking fault tendencies and stimulation of the martensitic transformation correlate fairly well within the limits of the small amount of data available for these phenomena.

As a result of the above discussion it can be seen that enhancement of the $M_S$ after reversal could be explained either by the introduction of strain embryos or stacking faults into the austenitic matrix by the reverse transformation. The embryo theory would also explain the stabilization in the 32.4 atom percent nickel-iron alloy if the reverse transformation together with the initial martensite plates formed on cooling are assumed to partition the austenite into regions whose small size would make ineffective the smaller embryos that should become critical at the lower temperatures where stabilization is observed to take place. Metallographic evidence for the presence of stacking faults after reversal is presented in the next section.

The dependence of the $M_S$ variation with nickel content observed in this investigation, and the difference in stabilization behavior of alloys of other compositions reported elsewhere, make it tempting to try to relate the effect of nickel and other elements on the stacking fault
tendency in their respective iron alloys to the observed changes. This direction, however, is not fruitful because of the lack of data in this area. At the present time, only the work of Otte\textsuperscript{13} is pertinent. His results for several iron-nickel alloys are inconclusive in that no diffraction lines of the hexagonal closed packed structure expected in faulted austenite were observed, while some lines appeared that could not be identified as either FCC or HCP. From a comparison of the relatively low frequency of annealing twins in iron-nickel alloys to their profuse formation in iron-chromium-nickel alloys, he inferred that the stacking fault energy in iron-nickel alloys is comparatively high.

The reduced driving force for martensitic transformation in higher nickel alloys\textsuperscript{(34)} might actually explain the observation that the most significant changes in transformation behavior occurred in the 32.4 percent nickel alloy. The inherently more stable austenite of this alloy might be expected to benefit more from the effects of reversal, be it the formation of stacking faults or strain embryos, than the lower nickel alloys where the driving force for transformation is already high.

C. The Reverse Martensitic Transformation

The microstructural nature of the reverse martensitic transformation was followed by the examination of initially transformed specimens of the 32.4 atom percent nickel alloy that had been reversed by 2 minute holds in molten salt at 20°C intervals through the temperature range of the reverse transformation. Room temperature hardness measurements were also made on the samples. The resulting hardness versus temperature curve is shown in Figure 16 and a series of photomicrographs of the microstructure produced during and after the reverse martensitic transformation are shown in Figures 17 to 22.
FIG. 16  HARDNESS VS TEMPERATURE FOR 32.4 ATOM PERCENT NICKEL-IRON ALLOY. EACH SAMPLE TWO MINUTES AT TEMPERATURE.
Figure 17. 32.4 percent nickel alloy. Martensitic structure after transformation at -196°C.

Figure 18. 32.4 percent nickel alloy. Initial stages of reversal of martensite plates after 2 minutes at 342°C.
Figure 19. 32.4 percent nickel alloy. Partially reversed martensite plates after 2 minutes at 360°C.

Figure 20. 32.4 percent nickel alloy. Final stages of reversal after 2 minutes at 400°C. Note traces in austenite marking positions of former martensite plates.
Figure 21. 32.4 percent nickel alloy. Complete reversal after 2 minutes at 412°C.

Figure 22. 32.4 percent nickel alloy. Typical structure of reversed austenite after a standard reversal at 450°C.
The hardness versus temperature curve shows that hardness increased very slightly to a maximum at 200°C and then decreased gradually as the reversal temperature was increased to the $A_f$. A similar increase in hardness prior to the beginning of the reverse transformation has been found by Blanter and Mashkov\textsuperscript{(14)}. Although the latter authors offered no explanation, it is quite likely that the peak is associated with carbide precipitation in the martensitic matrix. Sakamoto and Sugeno\textsuperscript{(35)} have observed an internal friction peak in an austenitic low carbon 30 percent nickel-iron alloy due to carbon and nitrogen at 200°C. Possibly the enhanced movement of carbon through the 40 percent retained austenite of the transformed structure aids in creating the slight hardness rise at this temperature. The uniform hardness drop during the reverse transformation between the $A_s$ and $A_f$ corresponds to the decreasing amount of martensite present after reversals at progressively higher temperatures. Above the $A_f$ temperature, (402°C), the hardness of the completely reversed specimens showed very little sensitivity to increasing reversal temperature and in fact remained constant for all two minute reversals up to 650°C. It should also be noted that the total change in hardness on going from the as-transformed structure containing about 60 percent martensite to the as-reversed austenite is considerably higher than the hardness of the annealed austenite (about 20 points $R_B$).

The photomicrographs show the microstructures resulting from the reversal of martensite and its replacement by austenite. Comparison of the microstructure of a specimen reversed at a temperature just above the $A_s$ (Figure 18) with the microstructure of an as-transformed specimen
(Figure 17), shows that the first martensite to reverse has come largely from the periphery of the martensite plates, the balance of which remain intact. After reversals at higher temperatures, however, the plates themselves begin to break up, as shown in Figure 19, in a manner similar to the "piecewise" reversal that has been observed in a 31.7 atom percent nickel alloy by Kaufman. After reversals at still higher temperatures but still below the $A_f$, diffuse markings in the austenite surrounding the small residual amounts of martensite that have yet to reverse become apparent. Figure 20 shows those markings which seem to delineate the trace of the original martensitic plates that had originally contained this residual martensite. The microstructure of a specimen that has been completely reversed, as shown in Figure 21, consists entirely of these diffuse etching areas in the austenite. These areas have no definite boundaries and seem to assume the morphology of "ghost" colonies of martensite.

The microstructures of specimens reversed at a temperature approximately $50^\circ C$ above the $A_f$ are significantly different from the as-reversed structures just described. A typical example is shown in Figure 22. It can be seen that a clearly developed structure has replaced the diffuse markings of the austenite reversed just above the $A_f$. These islands of structure have well defined boundaries and seem to align themselves in a manner similar to the residual pieces of martensite within a reversing martensitic plate.

There is strong metallographic evidence that this structure is in a twinned orientation to the austenitic matrix. Several instances of direct merging of this structure with the large annealing twins formed during high temperature annealing have been observed. Figure 23 shows such an example. Two large annealing twins, one approximately three times as wide as the other, are seen to slant diagonally across the photomicrograph.
the lower right portion and also in the upper left hand part of these
twins the structure produced by the martensitic transformation and its
reversal within the twins can be seen to merge directly with the thin
band of twin oriented matrix separating the annealing twins. It can also
be seen in the remainder of the grain outside of the twins that the
structure produced by reversal etches very similarly to the annealing
twins. A similar example is shown in Figure 24.

Other evidence for the twinned orientation of the structure produced
by reversal to the matrix is provided by the interaction of slip bands
with the islands of structure. Slip in FCC lattices occurs on the
close packed (111) planes, and therefore the identification of these
planes could be made by their intersections with the surface of a
polished and etched sample following deformation severe enough to produce
slip. Figure 25 and 26 show that the (111) planes in the reversal
structure and matrix are mirror images of each other across the interface
of the islands and the matrix. The slip bands identifying the (111)
planes were produced by compression of a polished metallographic specimen
in a vice.

The formation of the reversal twins described above appears to have
resulted from a recovery process made possible by the increased thermal
energy at approximately 50°C above the $A_t$. Just above the $A_f$ the
austenite that had undergone reversal of the martensitic transformation
was severely strained and it is quite likely that the atoms in these
regions were displaced appreciably from their equilibrium positions.
The formation of faults in a structure such as this is a reasonable
assumption, since the strain energy of the reversed structure could
effectively be reduced as the atoms settle into equilibrium positions.
Figure 23. Merging of reversal structure with annealing twins in 29.7 atom percent nickel-iron alloy reversed at 600°C for 15 minutes.

Figure 24. Merging of reversal structure with annealing twins in 30.6 atom percent nickel-iron alloy reversed at 600°C for 10 minutes.
Figure 25. Intersection of slip bands with reversal structure in 32.4 atom percent nickel-iron alloy reversed at 450°C after 3 cycles of transformation.

Figure 26. Portion of the same field as above at a higher magnification.
within the faults while still maintaining their nearest-neighbor requirements with the matrix across the fault interface.

If the FCC lattice is considered as a stacking of close packed (111) planes with the atoms of each plane in a regularly repeating sequence of the possible positions, ABC, then two types of stacking faults could occur on heating as the atoms settle into equilibrium positions A, B, and C. A deformation fault would form if the normal stacking sequence ABCABC was changed to ABCACABC where the change in the positions (from B to C) of the atoms of only one plane has occurred and resulted in the creation of HCP structure CACA within the FCC matrix. A growth or twin fault would be represented by a reversal of the normal stacking sequence from ABC to CBA above the last plane belonging to the normal sequence of stacking. A clearer picture of the different types of fault is shown schematically in Figure 27 which was taken from a discussion by Warren of the theoretical basis for obtaining experimental evidence for faulting by x-ray diffraction techniques.

The large size of the reversal twins, many about 0.04 mm long, makes it seem that very large numbers of atoms have cooperatively shifted into fault positions by means of local thermal fluctuations. This is reasonable since the coordinated movements of groups of atoms would be more likely to initiate a stable faulted structure than random thermal fluctuations of individual atoms which could be readily displaced from twinned positions under the influence of their untwinned neighboring atoms. Nevertheless, the formation of these large twins by the regular extension of growth or twin faults does not seem compatible with the distorted, irregular structure of the reversed austenitic matrix from which they form. Deformation faults or multiple deformation faults as shown in (c) of Figure 27 would seem to be
Figure 27. Faulting on the (111) planes of a FCC metal.
(a) normal FCC sequence; (b) single deformation fault;
(c) double deformation fault; (d) growth or twin fault.
more likely as the underlying structure of the reversal twins. Many of these deformations faults could form simultaneously in clusters and eventually unite to form the large twins observed. Support for this proposal comes from the striated appearance of the reversal twins occasionally observed and the replacement of many of the large reversal twins by a much finer structure after a number of cycles of transformation. The slip bands within the islands to which the faults are parallel suggest how these faults are oriented with respect to the reversal twins observed (see Figures 25 and 26).

Both the diffuse marking of as-reversed austenite and the characteristic alignment of the reversal twins formed at higher temperatures suggest a definite relationship to the prior martensitic structure. The crystallography of martensitic transformations has recently been the subject of several extensive reviews(37)(38)(39). Theory requires the martensitic lattice to be formed by a pure strain and rotation of the parent austenite lattice to give a total lattice deformation that accounts for the correct orientation relationship between the parent and product. However, if the condition of invariant-plane strain is to be met, there must also be a lattice-invariant shape deformation such as twinning or slip on a fine scale within the martensitic plate. Direct evidence for the presence of such twins within martensite plates parallel to the (112)_M planes has been obtained by Kelley and Nutting(11) in an iron-nickel-carbon steel and by Mishiyama et al(40) in a 30 percent nickel-iron alloy. Other evidence for the presence of twins and stacking faults by x-ray diffraction line broadening studies of martensite have been reviewed by Winchell(18).
The internal deformation of martensitic plates by twinning might account for the piecewise reversal of the martensite plates as opposed to their almost instantaneous growth to full size in iron-nickel alloys on cooling. However, the reason that some large blocks of twinned martensite reverse, while adjacent blocks remain martensitic cannot be explained. Other factors which should be considered but which also cannot be adequately assessed at the present time because of insufficient data and limited theoretical development are the effect of the dislocation interface between the austenite matrix and martensite on reversal and the effect of thermal fluctuations during heating. The total interaction of all these factors results in the production of the strained and distorted structure inherited by the austenite just above the $A_f$ which then lowers its energy by the recovery that results in the reversal twins already discussed and the eventual recrystallization of the matrix which will be discussed in a later section.
D. Strengthening by the Reverse Martensitic Transformation

The mechanical properties of both austenitic and transformed specimens of the 29.4, 30.6, and 32.4 atom percent nickel-iron alloys were determined as a function of cycles of martensitic transformation. Figures 28 through 33 show the variation of the tensile strength, yield strength (0.2 percent offset), elongation (percent in 1-inch gage length), and hardness with increasing cycles of transformation for the austenitic (Figures 28, 30, and 32) and the transformed (Figures 29, 31, and 33) specimens of the three alloys. Figure 34 compares the fracture stress, reduction of area, and elongation of the martensitic and austenitic structures produced by varying cycles of transformation in the 29.4 atom percent nickel alloy. Table VIII summarizes the changes in tensile and yield strengths with cycles of transformation by listing the percentage increase of these properties as compared to specimens that had not been subjected to any cyclic transformation. Values that are doubtful because of observed abnormal amounts of retained austenite have not been tabulated but are plotted in the figures.

All tensile and yield stresses were obtained from the engineering stress-strain curves. In order to determine if the mechanical properties of the austenitic specimens were influenced by transformation to martensite during tensile testing, small samples were machined from the necked ends of the broken tensile bars of the 30.6 and 32.4 atom percent nickel alloys and subjected to both magnetic and x-ray diffraction inspection. Within the limits of experimental error, no evidence of the presence of martensite was observed. In addition, none of the stress strain curves for any of the alloys exhibited any sharp, discontinuous increases in strength that might have resulted from transformation during testing.
TABLE VIII

Increase of Tensile and Yield Strengths of Austenitic
and Transformed Specimens after Cyclic Transformation

<table>
<thead>
<tr>
<th>Alloy (Atom percent Nickel)</th>
<th>Condition</th>
<th>Yield Strength Prior to Cycling (psi)</th>
<th>Increase in Yield Strength after 1 cycle (percent)</th>
<th>Increase in Yield Strength after 3 cycles (percent)</th>
<th>Increase in Yield Strength after 5 cycles (percent)</th>
</tr>
</thead>
<tbody>
<tr>
<td>29.4</td>
<td>Austenitic</td>
<td>24,200</td>
<td>155</td>
<td>172</td>
<td>177</td>
</tr>
<tr>
<td></td>
<td>Martensitic</td>
<td>82,600</td>
<td>13.2</td>
<td>10.8</td>
<td>18.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>30.6</td>
<td>Austenitic</td>
<td>29,900</td>
<td>106</td>
<td>135</td>
<td>136</td>
</tr>
<tr>
<td></td>
<td>Martensitic</td>
<td>84,700</td>
<td>9.7</td>
<td>11.6</td>
<td>--</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>32.4</td>
<td>Austenitic</td>
<td>32,200</td>
<td>85</td>
<td>--</td>
<td>130</td>
</tr>
<tr>
<td></td>
<td>Martensitic</td>
<td>59,500</td>
<td>--</td>
<td>--</td>
<td>44.5</td>
</tr>
<tr>
<td></td>
<td></td>
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</tbody>
</table>
MECHANICAL PROPERTIES OF AUSTENITE VS CYCLES OF TRANSFORMATION FOR 29.4 ATOMIC PERCENT NICKEL-IRON ALLOY. REVERSALS AT 475°C.
FIG. 29 MECHANICAL PROPERTIES OF MARTENSITIC STRUCTURE VS CYCLES OF TRANSFORMATION FOR 29.4 ATOM PERCENT NICKEL-IRON ALLOY. REVERSALS AT 475°C
FIG. 30 MECHANICAL PROPERTIES OF AUSTENITE VS CYCLES OF TRANSFORMATION FOR 30.6 ATOM PERCENT NICKEL-IRON ALLOY. REVERSALS AT 475°C
FIG. 31 MECHANICAL PROPERTIES OF MARTENSITIC STRUCTURE VS CYCLES OF TRANSFORMATION FOR 30.6 ATOM PERCENT NICKEL-IRON ALLOY. REVERSALS AT 475°C
FIG. 32 MECHANICAL PROPERTIES OF AUSTENITE VS CYCLES OF TRANSFORMATION FOR 32.4 ATOM PERCENT NICKEL-IRON ALLOY REVERSALS AT 450°C
FIG. 33 MECHANICAL PROPERTIES OF MARTENSITE VS CYCLES OF TRANSFORMATION FOR 32.4 ATOM PERCENT NICKEL-IRON ALLOY. REVERSALS AT 450°C
FIG. 34  COMPARISON OF SOME MECHANICAL PROPERTIES OF AUSTENITIC AND MARTENSITIC STRUCTURES IN 29.4 ATOM PERCENT NICKEL-IRON ALLOY
Inspection of Table VIII and the pertinent figures shows that the strength increases due to cycling in austenitic specimens of all three alloys were much greater than the increases in martensitic specimens. The major increase in strength for both structures was observed after the first cycle, and subsequent cycles of transformation caused proportionately smaller increases. Malyshev et al\(^{(5)}\) have reported a similar behavior for the iron base alloys they investigated. The yield strength, hardness, and elongation were much more sensitive to the effects of cycling than was the tensile strength. In a 29.4 atom percent nickel specimen, for example, the yield strength was increased by 177 percent after five cycles of transformation compared to the 45 percent increase in tensile strength. The fracture stress of the austenite was somewhat higher than that of the martensite up to 3 cycles of transformation in the 29.4 atom percent nickel alloy as shown in Figure 34, primarily because of the greater reduction of area at fracture. After five cycles of transformation, a total time of 30 minutes at the reversal temperature of 475°C may have allowed a segregation, which will be discussed later, to occur at the austenite grain boundaries, and so caused the decrease in reduction of area observed. The fracture stress of the austenite based on this doubtful reduction of area has not been plotted.

In an attempt to explain the marked strength increases following cycles of direct and reverse martensitic transformation, the observed changes in structure following cycling will be described. The austenitic structures, in which the greatest increases in strength occur, will be discussed first. Two different processes by which structural changes can be introduced into a reversed austenitic matrix can be distinguished. The one is the plastic deformation or the introduction of dislocation networks that occurs in austenite adjacent to martensitic plates both during the direct and reverse
transformation, and the other is the internal structural rearrangement within the plates and its consequent influence on the austenite produced from the plates after reversal. It is predominately the latter process which causes the strengthening in the 29.4 and 30.6 atom percent nickel alloys, since the transformation is virtually complete and no stabilization is observed with cycling after cooling to -196°C. However, since the deformation of the austenite by the first plates to form is incorporated into martensitic plates that form at lower temperatures, there is no clear separation of the influence of the two processes in these alloys. In the 32.4 atom percent nickel alloy, the martensitic transformation is only 60 percent complete after cooling to -196°C, and so the structural changes contributed by the transformed areas would be proportionately reduced as the deformed areas around the martensite areas assumed a more independent role in the total strengthening.

The microstructure of austenite after one full cycle of direct and reverse transformation has been shown in Figure 22 and the likelihood of the formation of the reversal twins formed from stacking faults produced during the initial recovery of the as-reversed austenite has been discussed in the preceding section. Figure 35 shows the microstructure of an austenitic specimen of the 32.4 percent nickel alloy that has been subjected to five complete cycles of transformation. The relatively large reversal twins have been replaced by a much finer and more uniform distribution of twins throughout the microstructure. Inspection of the other areas of this sample showed that some of the larger twins persisted just as colonies of much finer twins existed in some areas of the specimen that had been subjected to only one cycle of transformation. Nevertheless,
Figure 35. Microstructure of reversed austenite in 32.4 atom percent nickel-iron alloy after five cycles of martensitic transformation.

Figure 36. Transformed microstructure of the 32.4 atom percent nickel alloy after five cycles of transformation. Fine reversal twins are inherited by both retained austenitic and martensitic plates.
Figures 22 and 35 are representative of the most significant differences that develop as the cycles of transformation are increased.

In addition to the microscopically visible structure, it is quite likely that submicroscopic distributions of faults are present in addition to the visible twins, and these may possibly account for the marked strengthening that occurs in austenite after reversal. In the theory of work hardening of FCC metals as discussed by Cotrell\(^{41}\) and Seeger\(^{42}\) the interaction of stacking faults and the dislocations that bound them are considered to present very effective barriers to dislocation movement. It has been proposed that the unit dislocations on (111) planes dissociate into half-dislocations to lower their energy by the following reaction:

\[
\frac{1}{2} \mathbf{a}[101] + \frac{1}{6} \mathbf{a}[211] + \frac{1}{6} \mathbf{a}[112]
\]

The half-dislocations repel each other and as they move apart a stacking fault corresponding to HCP stacking of (111) planes is formed between them. They continue to move apart until the energy of the fault just balances the repulsive force between the two half-dislocations. The resulting structure is referred to as an extended dislocation and the motion of this dislocation under applied stress results in plastic deformation. The interaction of extended dislocations on intersecting slip planes produces a sessile dislocation bounded by ribbons of stacking fault and their respective partial dislocations on each of the intersecting planes. This configuration, referred to as a Lomer-Cottrell dislocation, effectively blocks subsequent slip on these planes until the applied stress has piled up sufficient dislocations to cause cross slip by the recombination of the partial dislocations.

In addition to the type of block formed during deformation, obstacles to dislocation glide already present prior to deformation, as in the case now under consideration, would also serve to increase flow stress. Basinski\(^{63}\) has discussed such an increase in stress accompanying constriction and jog formation during the intersection of one extended dislocation by another in FCC metals.

A number of investigations have related the high work hardening capacity of certain FCC cubic alloys to high concentration of stacking faults. One of the earliest
was by Barrett\(^{43}\), who detected stacking faults by x-ray diffraction both after quenching and after cold work of copper-silicon alloys that were known to work harden rapidly. The faulting was quite sensitive to composition, occurring only when the silicon content reached four percent, and the faulting produced during quenching was attributed to plastic strain produced on cooling. The stacking fault susceptibility of chromium-nickel-iron alloys reported by Otte\(^{13}\) correlates well with the known high strain-hardening capacity of stainless steels. Nishiyama and Shimizu\(^{44}\) more recently have established that high concentrations of stacking faults exist in Hadfield high manganese steel, well known for the controversy over the cause for its high work hardening capacity, by examination of cold-worked foil by transmission electron microscopy.

In view of the above discussion, the marked increase in the strength of the austenitic structures produced in this investigation by cyclic martensitic transformation is satisfactorily explained by the introduction of stacking faults after reversal. The continued increase in strength with increasing cycles of transformation can be explained by the increased number and more uniform distribution of smaller faults produced by additional cycles of reversal. These faults, however, seem to be unique in that they are produced only by reversal of the martensitic transformation, since little or no evidence exists for the presence of faults in annealed iron-nickel alloys or the production of faults during cold working. Examination of the stress-strain curves in Figure 65 of the as-reversed (2 minutes at 650°C) and the fully recrystallized (30 minutes at 650°C) specimens shows that the imperfections introduced in the former during reversal are as effective in producing work hardening as those introduced into the recrystallized (annealed) specimen by plastic deformation. Calculation of the true stresses shows that the yield strength (0.2 percent offset) of the as-reversed specimen is equivalent to the strength produced in the recrystallized specimen after 20 percent uniform elongation in tension.
Faulting as a consequence of transformation as opposed to formation by cold work has also been known to follow the martensitic FCC to HCP transformation in cobalt which occurs on cooling below about 390°C. Houska and Averbach (45) in a recent x-ray line broadening study have reported relatively high probability for the existence of both growth and deformation faults in the HCP phase as a result of transformation. In the case of the HCP phase, a fault consisted of a sequence of FCC stacking of (111) planes. This example of faulting produced by transformation is, however, not entirely analogous to the faulting produced by the reverse martensitic transformation in iron-nickel alloys. Since two close-packed structures can evidently coexist easily with one another in the cobalt, after partial transformation for example, stacking fault energies would be expected to be quite low. Likewise, the appearance of a HCP phase accompanies high stacking fault tendencies in the cold-worked copper-silicon alloys, stainless steel, and Hadfield manganese steel discussed previously. This is not the case for iron-nickel alloys where no evidence of a stable HCP structure exists over a wide range of compositions about 30 percent nickel.

The strengthening of martensitic structures after cyclic transformation is significant but not nearly as great as the strengthening of the austenitic structures. Again, the most marked increase (about 10 percent) in both yield and tensile strengths occurs after the first cycle of transformation. These initial increases are followed by more gradual increases after further cycles of transformation. The interplay of a number of factors are associated with the strengthening of the transformed martensitic structures. These factors can be divided into two groups: morphological changes in the martensite and work hardening of retained austenite in the transformed structures.
In the 29.4 and 30.6 atom percent nickel alloys where the direct martensitic transformation is 95% complete at -196°C and where little or no stabilization is observed with increasing cycles of transformation, only the morphological changes in the martensite seem to be related to the strengthening, while in the 32.4 atom percent nickel alloy, in which transformation is only 60 percent complete, the work hardening of the retained austenite in addition to morphological changes contribute to strengthening. Two major morphological changes seem to occur. The first is the noticeable decrease in width of the martensitic plates after cycling and the tighter packing of the plates into clusters between the retained austenite. Figures 36 through 38 show this change in the transformed microstructure after cyclic transformation when compared to Figure 17 in which the microstructure of martensite formed from annealed austenite is shown. It can be seen that the martensitic plates in the latter structure are comparatively broad and free of any internal structure. The decreased width of the plates after cycling would account for some strengthening because of the reduced path for dislocation movement within the plates.

The appearance of internal structure within the martensitic plates after one or more cycles of transformation is the second major morphological change observed in the transformed structures. This fine structure appears to consist of small pieces of austenite that does not transform on cooling with the remainder of the plate and seems to assume configuration within the martensitic plate similar to the distribution of the reversal twins in the austenite. It is quite likely that these twinned regions in the reversed austenite are not suitably oriented for the martensitic shears that transform the balance of the austenite surrounding them. Configurations corresponding to the island-like reversal twins are seen within the martensitic plates of Figures 37 to 39 and distributions of the finer twins formed after
Figure 37. Transformed microstructure of the 32.4 atom percent nickel alloy after five cycles of transformation.

Figure 38. Same field as above at higher magnification. Note islands of retained austenite present within the martensitic plates.
Figure 39. Transformed microstructure of 32.4 atom percent nickel alloy after five cycles of transformation showing clusters of narrow martensitic plates with internal retained austenite.

Figure 40. Transformed microstructure of 30.6 atom percent nickel alloy after five cycles of transformation. Fine twins of austenite are inherited by martensitic structure.
several cycles of transformation are shown in Figures 36 and 40. In the latter two microstructures, the fine twins of austenite are retained in both the austenitic and martensitic phases after transformation. Since these fine twins are a result of the retention of twins formed in austenitic areas that were formerly occupied by martensitic plates, this behavior would indicate that some of the martensitic plates had formed at different sites after reversal or fewer plates had formed. In view of the marked strengthening the twinned and faulted structure caused in austenite, the martensitic plates that inherit this structure should also be significantly strengthened by the more frequent obstacles to dislocation movement.

The work hardening of the retained austenite undoubtedly contributes to the strengthening of the transformed structures of 32.4 atom percent nickel alloy. Throughout most of the microstructure of this alloy the austenite retained after transformation at -196°C is quite uniform in etching characteristics, with no evidence of a twinned structure (Figures 37, 38, and 39). These areas are therefore strengthened only by the plastic deformation caused in them by the displacements accompanying the previous transformation. However, in a few areas the appearance of twins (Figure 36) in the retained austenite indicates that some transformation occurred during previous cycles in these regions, but for some reason did not occur during the final transformation which produced the observed structure. These areas would then be hardened both by the presence of faulting due to martensitic transformation and its reversal as well as by the plastic deformation already mentioned.

The total interaction of these structural changes introduced into the martensitic plates and the retained austenite after transformation of reversed
austenitic structures produce the observed strengthening of the transformed specimens. The relative contribution of each is difficult to assess but it would be expected that the inheritance of the faulted structure that so effectively strengthened the reversed austenite would also be an important cause of the hardening in transformed structures.

The discussion up to this point has been concerned only with the strengthening of the vacuum-melted iron-nickel alloys containing 0.005 to 0.007 percent carbon. The 29.7 atom percent nickel alloy containing 0.07 weight percent carbon, 0.71 weight percent manganese and other small amounts of impurities was not tested as extensively as the lower carbon alloys, but the tests made indicate that the strengthening of the austenitic structure produced by the reverse martensitic transformation in this alloy is of the same order of magnitude as in the 29.4 atom percent alloy. Increases of 143 and 173 percent in yield strength were observed after one and two cycles of reversal at 500°C respectively. The stress-strain curves from which these values were determined are shown in Figure 61. It appears that the small additional amount of carbon in this alloy did not produce any appreciable increments in strengthening, but it is quite likely that amounts of carbon greater than this would make additional contributions to strengthening by interaction with the imperfections of austenite subjected to reverse martensitic transformation.

E. Recovery and Recrystallization of Reversed Austenite

Recovery and recrystallization in the conventional sense are differentiated by several important changes occurring within a strained metal during the annealing process. Recovery is usually assumed to occur without an incubation period and the isothermal kinetics have been found to follow first-order equations of the form
\[
\frac{dx}{dt} = -kx
\]  \hspace{1cm} (E-1)

where \( x \) is the fraction of the property change due to straining that has not yet recovered. On the other hand, an incubation period is usually associated with recrystallization, and the isothermal recrystallization kinetics follow an equation of the form

\[
x = 1 - \exp\left(-bt^n\right)
\]  \hspace{1cm} (E-2)

where \( x \) is the fraction of the strained structure recrystallized at any time \( t \), \( n \) is an exponent which depends on the geometry of recrystallization, and \( b \) takes into account the nucleation and growth rates of recrystallization. Equation E-2 represents the characteristic sigmoidal recrystallization curve obtained experimentally if \( x \) is plotted against time at a constant recrystallization temperature.

During recovery, properties (such as electrical conductivity) that are most affected by localized displacement of atoms or the introduction of imperfections on a fine scale recover by local rearrangement of the atoms without the movement of large-angle grain boundaries. Usually, mechanical properties are only slightly changed during this rearrangement. The recrystallization process differs in that strain-free grains nucleate and grow by the movement of large-angle boundaries. On completion of this process the mechanical properties have decreased approximately to their values in the unstrained state.

The driving force for both recrystallization and recovery is the volume strain energy introduced during the straining process. In almost all investigations of recrystallization, this strain energy is introduced by cold working. This investigation differs in that the strain introduced by the reversal of the martensitic transformation is the source of the
driving force for recovery and recrystallization. In the experiments of Aust and Rutter\(^{(46)(47)}\), a source other than cold work for the introduction of strain energy has also been utilized. In the latter case the striation structure formed on solidification of melt-grown single crystals of high purity tin and dilute lead-tin alloys provided a stable driving force for the study of grain-boundary migration.

In the present investigation the kinetics of recovery and recrystallization of reversed austenite formed after a cycle of direct and reverse transformation were followed initially by hardness changes of specimens that had been held for various times at temperatures between 600°C and 700°C. The results of the hardness measurements are shown in Figures 41, 42, and 43 for the 29.4, 30.6, and 32.4 atom percent nickel alloys respectively. Each point on the curves represents the average hardness of an individual sample held at temperature for the time shown. One side of the sample was used for the hardness measurements, the other for metallographic examination. The hardness changes of a single sample were not followed since hardness indentations were observed to cause local recrystallization on subsequent reheating; it was also desirable to minimize the error in establishing the true time at temperature which successive heating and cooling of a single sample during its recrystallization would have introduced. Specimens of the 32.4 atom percent nickel alloys were given a standard reversal treatment prior to isothermal holdings. Samples of the other alloys were reversed on heating directly to the holding temperature.

Inspection of the hardness versus time curves shows that the hardness changes took place in two definite stages: the first was a slight decrease in hardness sometimes followed by a plateau, and the second, a sharp decrease in hardness after longer times at temperature. The first stage was associated
FIG. 4.1 HARDNESS VS TIME AT REVERSAL TEMPERATURE FOR 29.4 ATOM PERCENT NICKEL-IRON ALLOY
Fig. 42 Hardness vs Time at Several Reversal Temperatures for 30.6 Atom Percent Nickel-Iron Alloy.
with the recovery of the reversed austenite. This recovery has already been discussed in a previous section where the formation of reversal twins from the diffuse-etching strained, austenitic structure resulting from the reverse transformation just above the \( A_T \) was described. Examination of the austenitic microstructures produced during the recovery stage showed that not all of the diffuse etching structure was converted into these reversal twins. Rather, some of the diffuse etching structure persisted with the reversal twins for substantial periods of time at temperatures, although it did appear to diminish in intensity after longer times at temperature. This recovery behavior can be seen in the unrecrystallized portions of the microstructures shown in Figures 46, 47, and 48 which show the microstructure of samples of the 32.4 atom percent nickel alloy just after reversal at 450°C and after longer times at 600°C.

Examination of the microstructures accompanying the rapid hardness change showed that this stage was associated with the recrystallization of the reversed austenite. The sequence of microstructural changes occurring in the 32.4 atom percent nickel alloy on going from annealed austenite to reversed and fully recrystallized austenite is shown in Figures 44 to 49 inclusive. Figure 44 illustrates the microstructure of annealed austenite, Figure 45 the transformed structure after cooling to -196°C, and Figure 46 the structure after a standard reversal at 450°C. It can be seen that the cycle of direct and reverse martensitic transformation has caused the formation of a twinned and distorted structure within the austenitic grains but has not changed the configuration of straight-sided grain boundaries and annealing twins characteristic of annealed austenite. Figure 47 indicates the early development of grain boundary curvature and
Figure 44. Microstructure of 32.4 percent nickel alloy annealed 24 hours at 1000°C.

Figure 45. Microstructure of 32.4 percent nickel alloy transformed at -196°C for 30 minutes. Large martensitic plates surrounded by retained austenite.
Figure 46. Microstructure of 32.4 percent nickel alloy in as-reversed condition after heating at 450°C for 2 minutes. No recrystallization is apparent.

Figure 47. Microstructure of 32.4 percent nickel alloy recrystallized 23 percent after 240 minutes at 600°C. Note development of grain boundary curvature and clean etching surfaces of recrystallized grains.
Figure 48. Microstructure of 32.4 percent nickel alloy recrystallized 79 percent after 360 minutes at 600°C. Markings of austenite not yet recrystallized are difficult to distinguish.

Figure 49. Microstructure of 32.4 percent nickel alloy recrystallized completely after 1000 minutes at 600°C. All boundaries are curved and irregular twins are present within grains.
the appearance of smooth etching areas characteristic of recrystallized austenite. A much later stage of recrystallization is shown in Figure 48 and its completion in Figure 49. All grain boundaries of the as-recrystallized structure exhibit definite curvature and the grains contain irregular twins, many with curved instead of straight boundaries. The high incidence of twins and their irregular boundaries, indicates that faulting produced in the reversed austenite by the reverse martensitic transformation has affected the stacking of atomic planes during the growth of the recrystallized austenite.

The grain size change of specimens of the 32.4 atom percent alloy as a function of time at 625°C and 650°C is shown in Figure 50. The number of grain boundary intercepts per inch were determined for each sample both in the annealed condition and after heating for various times at either 625°C or 650°C. The difference between the two measurements is plotted. At both temperatures the number of boundary intercepts per inch increased as recrystallization was initiated and new strain-free grains appeared. This relatively sharp increase was somewhat misleading since the severely curved boundaries of a single newly recrystallized area were often intersected more than twice during a straight line traverse. The change in grain size also seemed large in that relatively few nucleation centers for recrystallization were observed.

In order to resolve the above peculiarities and to follow the recrystallization process more unambiguously than possible with hardness measurements which were sensitive to the overlapping of recovery and recrystallization, the volume fraction of recrystallized austenite, x, as a function of time at recrystallization temperature was measured by lineal analysis. Quantitative metallography was possible since the
recrystallized austenite could be differentiated from reversed, strained austenite because of its clean etching characteristics and the curved boundaries which always surround it. The results of the lineal analysis are shown in Figures 51 and 52 for the 30.6 and 32.4 atom percent nickel alloys respectively. The characteristic sigmoidal fraction recrystallized versus time curves were obtained for recrystallization of the 32.4 atom percent nickel alloy at all temperatures investigated, while the recrystallization kinetics of the 30.6 atom percent alloy differed somewhat in that, at most of the recrystallization temperatures, once recrystallization was initiated it proceeded quite rapidly to completion without the characteristic retardation in the last stages. One possible explanation of the latter behavior might be the almost complete recovery of reversed austenite that had not yet recrystallized. This recovery would tend to remove the distinguishing features of the reversed austenite and make its identification difficult. Some support for this explanation comes from hardness values of samples that appear to be fully recrystallized which are higher than the values of annealed austenite.

The formation of strain-free, recrystallized regions was observed to occur predominantly by the gradual extension into adjacent grains of certain grain boundaries of a relatively few reversed grains. As the boundary migrated it left behind an undistorted area which could be distinguished from the unrecrystallized portion of the grain only by its smooth etching characteristics and not by the formation of any well-defined boundary. Examples of this nucleation behavior are shown in Figures 53 and 54. In Figure 53 one of the original grains of the reversed austenitic matrix has almost doubled in size by the extension of one of its boundaries, while corrugations with small radii of curvature are just beginning to
GRAIN SIZE CHANGE DURING RECRYSTALLIZATION OF 32.4 ATOM PERCENT NICKEL-IRON ALLOY

GRAIN SIZE CHANGE (INCHES)

TIME (MINUTES)

625°C

650°C

INCH INTERCEPTS

FIG. 50
FIG. 51  
FRACTION RECRYSTALLIZED VS TIME AT REVERSAL TEMPERATURE FOR 30.6 ATOM PERCENT NICKEL-IRON ALLOY
FIG. 52 FRACTION RECRYSTALLIZED VS TIME AT REVERSAL TEMPERATURE FOR 32.4 ATOM PERCENT NICKEL-IRON ALLOY
Figure 53. Strain-induced boundary migration of a grain in a reversed specimen of the 32.4 atom percent nickel held at 600°C for 240 minutes.

Figure 54. Early stage of strain-induced boundary migration in a reversed specimen of the 32.4 atom percent nickel alloy held at 600°C for 120 minutes. Note presence of heavy grain boundary attack along migrating boundary and its absence along twin and other boundaries.
develop along two other portions of its original boundary. Figure 54 shows an earlier stage of the migration process. The curved boundaries and their tendency to etch deeply are characteristic of the migrating boundaries in iron-nickel alloys investigated. Nucleation of strain-free grains at preferred sites within the grains and along the grain boundaries was also observed, but not nearly so frequently as the initiation of recrystallization by grain boundary extension.

Beck (48)(49)(50) and his coworkers have extensively studied the type of grain boundary extension process described above in high purity aluminum and refer to it as strain-induced boundary migration. Most of the strain-free grains formed after annealing aluminum cold worked up to 40 percent were observed to form by this process. With greater amounts of cold work, the recrystallization occurred by the conventional nucleation and growth of strain-free grains. The driving force for the initiation of strain-induced boundary migration is the strain energy stored in a matrix that has been cold worked moderately, as found by Beck, or as in the case of this investigation, the strain energy introduced into the austenitic matrix by the reversal of the martensitic transformation. The release of strain energy by boundary migration causes the boundaries to move away from their centers of curvature as opposed to movement of boundaries toward their center of curvature during regular grain growth where the driving force is the interfacial surface energy associated with the grain boundaries.

Other characteristics of the strain-induced boundary type of migration type of recrystallization observed by Beck were the immediate appearance of migration without an apparent incubation period (48) and the
marked orientation dependence\(^{(49)}\) of the migrating boundaries. Large-angle boundaries separating adjacent lattices which were orientated relative to one another by rotation of 30 to 45° about a \([111]\) normal to the boundary were found to be the most mobile boundaries, while the boundaries between grains with either the same orientation or a twinned orientation corresponding to \([111]\) rotations of 0° and 60° respectively were almost always found to be stationary. This difference of boundary mobility with orientation is believed to be closely associated with grain boundary diffusion and its dependence on orientation. Large-angle boundaries with a high degree of disorder would be expected to maintain a higher rate of diffusion than low-angle boundaries with less misfit and tighter packing of atoms. This orientation dependence for diffusion along grain boundaries has been found. In nickel\(^{(51)}\), for example, the activation energy for grain boundary self-diffusion is 26 kilocalories per mole for high-angle boundaries and 66 kilocalories per mole (the value for lattice self-diffusion) for boundaries of better fit.

Although Beck reports that strain-induced boundary migration was observed almost immediately on annealing highly worked high purity aluminum, he has proposed a model\(^{(52)}\) to account for sigmoidal isothermal recrystallization kinetics when strain-induced boundary migration occurs. In the initial stage of boundary migration, he assumes that the driving force is the difference in energy between the strained matrix of the grain being penetrated by the migrating boundary and the interfacial energy necessary to extend the boundary. The energy of the grain being invaded may be quite small, possibly only the energy of the substructure formed by recovery of the lightly deformed matrix, but still greater than the strain energy of the grain which is the source of the migrating
boundary. The net effect of the small energy differences is to cause a gradual first stage of recrystallization or incubation period. At a later time, however, in the unrecrystallized grains adjacent to the recrystallizing grain, the migrating grain boundary moves toward its center of curvature in these grains as in grain growth, and so the two processes of recrystallization and grain growth might be considered to occur simultaneously. An accelerated rate of recrystallization is thus explained.

The temperature dependence of the recrystallization of the strained austenite produced by the reverse martensitic transformation was analysed by assuming a rate equation of the following form was obeyed:

\[
\frac{1}{t} = A \exp \frac{-Q}{RT} \quad \text{(E-3)}
\]

where

\( t \) = time at which a given amount of recrystallization was attained at a temperature, \( T \).

\( Q \) = activation energy for recrystallization.

\( A \) = constant.

Plots of \( \log \frac{1}{t} \) versus \( \frac{1}{T} \) for the 29.4, 30.6 and 32.4 atom percent nickel alloys are shown in Figures 55, 56, and 57 respectively. The rates for the beginning and middle of the recrystallization process were taken from both the hardness versus time and the fraction recrystallized versus time plots. The straight-line plots are based on least-squares calculations of the slopes and intercepts for the straight lines from the experimental data. The activation energies were also calculated by least-squares analysis and are tabulated in Table IX. It can be seen that there is reasonably good agreement between the activation energies for recrystallization calculated for the beginning and middle of recrystallization
FIG. 55  \( \frac{1}{t} \) VS \( \frac{1}{T} \) FOR RECRYSTALLIZATION OF 29.4 ATOM PERCENT NICKEL-IRON ALLOY

- 50% RECRYSTALLIZATION; FROM HARDNESS VS TIME
- BEGINNING OF RECRYSTALLIZATION; FROM HARDNESS VS TIME
Fig. 56  \( \frac{1}{T} \) vs \( \frac{1}{T} \) for recrystallization of 30.6 atom percent nickel-iron alloy
FIG. 57 \( \frac{1}{t} \) VS \( \frac{1}{T} \) FOR RECRYSTALLIZATION OF 32.4 ATOM PERCENT NICKEL - IRON ALLOY
### TABLE IX

**Activation Energies of the Recrystallization of Austenite**

Produced by the Reverse Martensitic Transformation in Iron-Nickel Alloys

<table>
<thead>
<tr>
<th>Alloy (atom percent nickel)</th>
<th>Activation Energy (cal/mol)</th>
<th>For Beginning of Recrystallization</th>
<th>For Middle of Recrystallization</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>From</td>
<td>From</td>
<td>From</td>
</tr>
<tr>
<td></td>
<td>hardness vs time</td>
<td>fraction recrystallized vs.time (x=0.05)</td>
<td>fraction recrystallization vs time (x=0.5)</td>
</tr>
<tr>
<td>29.4</td>
<td>82,000 ($R_B=80$)*</td>
<td>----</td>
<td>81,000 ($R_B=68$)*</td>
</tr>
<tr>
<td>30.6</td>
<td>76,500 ($R_B=83$)*</td>
<td>79,500</td>
<td>74,000 ($R_B=73$)*</td>
</tr>
<tr>
<td>32.4</td>
<td>89,500 ($R_B=78$)*</td>
<td>95,000</td>
<td>95,000 ($R_B=71$)*</td>
</tr>
</tbody>
</table>

* Hardness values at which rates for beginning and middle recrystallization were taken.
and between those calculated from the hardness versus time and fraction recrystallized versus time curves. A further check was made by modifying equation E-2 to the form:

\[ x = 1 - \exp \left[ - \frac{1}{(Kt)^n} \right] \]  \hspace{1cm} (E-4)

so that \( K \) becomes a constant with dimensions \( \text{time}^{-1} \) which may be considered to describe the combined temperature dependence of the nucleation and growth rates for recrystallization:

\[ K = B \exp \left[ - \frac{Q}{RT} \right] \]  \hspace{1cm} (E-5)

Taking the logarithm of equation E-4 twice and rearranging terms yields

\[ \log \log \frac{1}{1-x} = \log \frac{k^n}{2.3} + n \log t \]  \hspace{1cm} (E-6)

The plot of \( \log \log \frac{1}{1-x} \) versus \( \log t \) shown in Figure 58 for the recrystallization of the 32.4 atom percent nickel alloy for the four temperature investigated then enabled the value of \( K \) to be obtained. The activation energy of recrystallization calculated from the temperature dependence of \( K \) was 93.0 kcal/mol, in good agreement with the values recorded in Table IX for this alloy. The growth exponent \( n \) was also calculated and found to vary irregularly with temperature from 2.8 to 3.4 with an average value of 3.1 over the temperature range investigated. This value of \( n \) indicates recrystallization proceeds three dimensionally throughout the volume of the strained austenite.

The attempt to relate the measured activation energies describing the temperature dependence of the recrystallization to known activation energies for the self-diffusion of nickel or iron in iron-nickel alloys was unsuccessful. Since recrystallization took place primarily by the strain-induced migration of grain boundaries, the transfer of single iron
FIG. 58  LOG LOG $\frac{1}{1-X}$ VS LOG 1 FOR RECRYSTALLIZATION OF 32.4 ATOM PERCENT NICKEL-IRON ALLOY.
and nickel atoms from the strained material across these boundaries to the elementary process involved and therefore the rate of grain-boundary self-diffusion would be expected to be the rate determining factor. This has recently been found to be the case by Aust and Rutter\cite{47} for grain boundary migration of certain characteristically orientated boundaries in very dilute alloys of tin in lead.

The activation energy for the lattice diffusion of nickel in a face-centered cubic 30 percent nickel-iron alloy is approximately 65 kcal/mol\cite{54} and the activation energy for the lattice self-diffusion of iron in a 25 percent nickel-iron alloy has been found to be 79 kcal/mol\cite{55}. The most recent activation energy reported for the grain-boundary self-diffusion of iron in FCC iron is 41 kcal/mol\cite{62}. At first glance the measured activation energies for the 29.4 and 30.6 atcm percent nickel alloys seem to be almost the same as for that of the lattice self-diffusion of iron. This agreement, however, may be fortuitous in view of the grain-boundary segregation effect that was observed and which will be discussed in detail below. Even without this segregation, the measured values are higher than values for grain-boundary self-diffusion. At this point it should be mentioned that the measured activation energies may not really be representative of a single thermally activated process and may only be characteristic of the slope of the log $\frac{1}{t}$ versus $\frac{1}{T}$ plots which represent the net result of several interacting processes. Therefore the measured activation energies are not true activation energies but are "apparent" in the sense of Läcke and Detert\cite{56}.

Segregation at the migrating grain boundaries was first suspected by the severe attack of the etchant on these boundaries. The same etching technique did not attack most of the stationary boundaries and was never
observed at twin boundaries within the grains. Dark boundaries in annealed austenite were due to steps between grains of different levels after heavy etching. Good examples of the heavily etching boundaries can be seen in Figure 54 where strain-induced boundary migration is just beginning and in Figure 48 where recrystallization is well advanced and considerable grain boundary migration has occurred. The selective nature of the grain boundary attack can also be noticed in these figures. Further evidence that a segregation effect is the cause of this heavy grain-boundary attack is that the latter seemed to be associated with almost every migrating grain at any stage of its migration. If a precipitation reaction were occurring, the migrating boundaries would at some point break away and leave the precipitate behind. This behavior was occasionally observed but so infrequently that it did not seem to be a characteristic effect of the process.

Further evidence for the presence of a segregated grain-boundary film came from examination of an electro-polished and unetched specimen of the 30.6 atom percent nickel alloy which had been fully recrystallized after reversal at 650°C for 30 minutes. Figure 59 shows the resultant microstructure. It can be seen that the grain-boundary effect is manifested by two fine parallel lines, resolvable at 1500x, which follow the recrystallized grain boundaries. A phase contrast microscope brought out this effect even more clearly. Perryman (57) has found a very similar metallographic effect in tin-copper alloys which he attributed to equilibrium segregation. He found that the two lines, one usually slightly darker than the other, were due to a ridge and trough etching effect at the boundaries which appeared after electropolishing without
1500x

Electropolished in 5% Perchloric-Acetic Acid Electrolyte

Figure 59. Grain boundary film in reversed specimen of 30.6 atom percent nickel completely recrystallized after 30 minutes at 650°C.
etching. A taper section of such a boundary showed one side of the boundary was raised above the level of the adjacent grains and the other side was below the level of the grains. Perryman reasoned that the effect was not due to an insoluble impurity because it was not left behind when the boundaries migrated during recrystallization. The effect also seemed to be a function of the orientation of adjacent grains since it was not present at twin boundaries and occurred discontinuously where twins met the grain boundaries. Other metallographic evidence for the presence of equilibrium segregation is reviewed by McLean(58). Not all cases exhibit the ridge and trough effect. The segregation of carbon and nitrogen in iron and low carbon steels produces only ridges while iron at the grain boundaries of aluminum produces broad grooves on etching.

The segregation of carbon at the grain boundaries which migrate during the recrystallization of the reversed iron-nickel alloys used in this investigation is most likely responsible for the grain-boundary etching effect. Hücke and Detert(56) have developed a quantitative theory to account for the influence of impurity atoms on recrystallization. The theory is based on the premise that solute atoms are preferentially attracted to grain boundaries where their elastic interaction with the solvent matrix is a minimum. The tendency to segregate is assumed to be the larger the difference in atom radius between the foreign and base metal atoms. The energy gained by the migration of a foreign atom to a dislocation boundary can be roughly estimated from the formula developed by Cotrell:

\[ V = \frac{4}{3} \pi r^3 \sigma \left( \frac{1 + \eta}{1 - \eta} \right) \eta \]  \hspace{1cm} (E-7)

where

\[ \eta = \frac{r - r_F}{r} \]  \hspace{1cm} (E-8)
and

\[ r = \text{radius of base atom} \]
\[ r_F = \text{radius of foreign atom} \]
\[ G = \text{shear modulus} \]
\[ \sigma = \text{Poisson's ratio} \]

On the basis of the above formula, there should be little tendency for either iron or nickel to segregate at the migrating grain boundaries since their atomic radii are almost the same (about 1.27 angstroms). Carbon, on the other hand, has an atom radius of about 0.8 angstroms in iron\(^{(59)}\), and so should exhibit a strong tendency to segregate at the grain boundaries. When appropriate values are substituted into equation E-7 for the 30.6 atom percent nickel alloy \( (J = 5.95 \times 10^{-11}\text{dynes/cm}^2, \sigma = 0.26, \text{estimated}) \), and the constraints of the interstitial sites in a FCC cell are taken into account, a value of \( V \) is significantly higher than the interaction energy of 0.5 ev calculated by Cotrell for carbon in BCC iron and is obtained. In a FCC structure the interaction energy should be even lower than in BCC iron because of the greater solubility of carbon and the larger interstitial positions. Strictly, E-7 should be applied only when a solute atom substantially replaces a solvent atom on its lattice site. Nevertheless, if 0.5 ev is taken as a very rough estimate of the energy decrease when a carbon atom segregates to the boundary in the recrystallizing austenite, the grain boundary concentration, \( C_B \), at thermal equilibrium can be estimated from the formula

\[ C_B = C e^{\frac{V}{kT}} \quad (E-9) \]

where \( C \) is the average concentration of carbon atoms (0.005 weight percent in the 30.6 atom percent nickel alloy). At 650\(^{\circ}\)C, then, the equilibrium
concentration at the grain boundaries would be approximately 2.6 percent and is indicative of the strong tendency of carbon to segregate. At higher temperatures the tendency would be much less and in fact, examination of specimens annealed at 1000°C revealed no grain-boundary effect. When these annealed specimens were heated to 650°C without transformation, the effect did develop.

Since it is quite likely on the basis of the above discussion that carbon is the segregating element, the fortuitous relationship between the measured activation energy for the self-diffusion of iron in iron-nickel alloys becomes more obvious. Gruzin and Kuznetsov\(^{(55)}\) showed that the activation energy for the self-diffusion of iron in a 25 percent nickel-iron alloy decreased continuously from 79 kcal/mol to 58 kcal/mol when the carbon content of the alloy was increased from 0.02 percent to 0.90 percent. Structural changes produced during reversal have also been found\(^{(16)}\) to enhance diffusion rates. Therefore, increased carbon at the grain boundaries and reversal would be expected to yield a lower activation energy than observed if only the diffusion of iron atoms across the boundaries separating grains of annealed austenite were rate controlling.

Ilöke and Detert have offered an explanation for the apparent high activation energies due to impurity atom segregation at grain boundaries during recrystallization. They assume that the impurity atoms will migrate with the boundary if it moves slowly enough and the maximum velocity of the grain boundary with the attached foreign atoms will be determined by the mobility of the foreign atoms. For this situation, the temperature dependence should be described by the activation energy of diffusion of the impurity atoms. At a higher temperature fewer foreign atoms will be attached to the boundary, the total attractive force between
the atoms and the boundary will be decreased, and the boundary may be able
to break away and migrate more rapidly. The increased boundary velocity
will then be reflected in the \( \frac{1}{t} \) versus \( \frac{1}{T} \) plots as an increase in slope which
yields a higher apparent activation energy.

Turnbull\(^{(60)}\) has also offered an explanation for high observed
activation energies of recrystallization. He proposes that the
changing dispersion of inclusions with temperature superimposes the
process of precipitation or solution on the fundamental process of atom
transfer across the migrating boundaries and so increases the temperature
dependence of the rate of recrystallization and the experimentally
observed activation energy.

The theoretical explanation for high activation energies by Lücke and
Detert is the more appropriate one for the recrystallization behavior of
the reversed austenite in this investigation since very few carbide
particles were observed to be left behind the migrating boundaries. The
large concentration of carbon atoms that must move with the migrating
boundaries no doubt slows the rate of migration at the lower temperatures.
At the higher temperatures, according to equation E-9, the atmosphere of
carbon atoms that the boundary must drag along becomes exponentially more
diffuse, and the rate of boundary migration increases and approaches that
of the basic process of the transfer of nickel and iron atoms across the
boundaries. If the high temperature rates are assumed to be closest to
the true recrystallization rates in the absence of impurities, the effect
of the slower rates due to segregation at lower temperatures is to
increase the slope of the \( \log \frac{1}{t} \) versus \( \frac{1}{T} \) lines and therefore to increase
the observed activation energy for recrystallization. Because the two
simultaneous processes of segregation and atom transfer across the
the boundaries at the lower temperatures merge into a single process at higher temperatures, this activation energy obtained from over the entire temperature range investigated cannot be related to any one thermally activated process.

The above explanation does not take into account any boundary-orientation effects. It is known from Beck's\(^{48}\) work that strain-induced boundary migration in aluminum is quite sensitive to the orientation of the grains adjacent to the migrating boundary, the boundary mobility being higher, the greater the degree of misorientation. Ratheneau and Custers\(^{61}\) similarly have found in iron-nickel alloys that the most rapidly moving boundaries during secondary recrystallization are those which would be produced by a misorientation between adjacent grains corresponding to a 30° rotation about a \([11]\) axis normal to the boundary. Therefore it is likely that the boundaries which migrate during the presently considered recrystallization of reversed austenite are relatively high angle, disordered boundaries which in addition to promoting more favorable conditions for diffusion can also accommodate larger concentrations of carbon atoms than the lower angle, more stationary boundaries. This would account for the observations that all the migrating, curved boundaries exhibit the etching effect due to segregation, while at the same time the straight grain boundaries and twin boundaries produced during the prior annealing treatment which remain stationary during recrystallization are not at all sensitive to segregation etching effects. The importance of boundary orientation was also demonstrated by Aust and Rutter\(^{47}\) when they showed recently that the temperature dependence of the rate of grain-boundary migration in dilute alloys of tin in lead is sensitive to both the concentration
of tin and the orientation of the boundary. Activation energies for some high-angle grain boundaries increased to abnormally high values of tin, much higher than the activation energy for the lattice diffusion, with increased amounts of tin. For other "special" high-angle boundaries the activation energy was insensitive to the tin content and was equal to the activation energy for grain boundary self-diffusion of lead. The latter boundaries were found to separate grains which had relatively good matching in that a number of sites in both grains coincided. The atoms in these sites would not have to move at all as the boundary migrated past them and there would be a lower tendency for segregation to occur because of the closer packing at the boundary.

F. Recovery of Mechanical Properties

The change in the mechanical properties of reversed austenite during its recovery and recrystallization was followed in two series of experiments. In the first, tensile tests of transformed and reversed specimens of the 29.7 atom percent nickel alloy (containing 0.07 percent carbon and 0.71 percent manganese) were performed on specimens that had been heated at various temperatures above the \( A_r \) for a constant time of fifteen minutes. In the other, reversed specimens of the vacuum melted 30.6 atom percent nickel alloy were tensile tested after being held at 650°C for various times. It should be noted that the behavior of 29.7 atom percent nickel alloy has not been described in detail up to this point, and that the temperature dependence of its recovery and recrystallization differs somewhat from that of the other alloys.

The results for the 29.7 atom percent nickel specimens are shown in Figures 60, 61, and 62. Figure 60 shows the stress-strain curves for
the early stages of deformation which were obtained by loading the specimens in increments and measuring the residual plastic strain after each successive load was released. Figure 61 shows the entire engineering stress strain curve for each sample and the change in tensile strength, yield strength (0.2 percent offset), reduction of area (percent at fracture), and total elongation (percent in 2 inch gage length) as the reversal temperature is raised is shown in Figure 62. Inspection of these figures shows that the properties at first change gradually as the temperature of reversal is raised from 500°C to 700°C and then change markedly after the 800°C reversal.

The gradual decrease in strength and increase in ductility are related to the recovery processes taking place in the strained austenite produced by the reverse martensitic transformation. The microstructure of the sample reversed at 500°C is shown in Figure 63. It can be seen that the austenite etches quite unevenly and that the diffuse surface markings seem to align themselves in the zig-zag fashion associated with the autocatalytic formation of martensitic plates on cooling. After reversal at 600°C, the prevalence of the surface markings has been reduced and a fine twinned structure with definite boundaries has appeared. Figure 64 shows this microstructure. The twinned islands can be seen to align themselves in parallel groups. The microstructure within the austenitic grains after reversal at 700°C is quite similar, the only difference being a slight broadening of the twinned islands. Up to 700°C, no change in the position or shape of the straight grain boundaries characteristic of annealed austenite was observed, and even at 700°C only occasional boundaries of the austenitic grains were beginning to show the development of the corrugated curvature which preceded recrystallization by strain-induced boundary migration.
FIG. 60

INITIAL DEFORMATION BEHAVIOR OF AUSTENITE FORMED DURING 15 MINUTE REVERSALS AT TEMPERATURES SHOWN. NICKEL-IRON ALLOY CONTAINING 30.78 PERCENT NICKEL, 0.07 PERCENT CARBON AND 0.71 PERCENT MANGANESE.
FIG. 61  STRESS-STRAIN CURVES OF AUSTENITE FORMED DURING 15 MINUTE REVERSALS AT TEMPERATURES SHOWN. NICKEL-IRON ALLOY CONTAINING 30.78 PERCENT NICKEL, 0.07 PERCENT CARBON, AND 0.71 PERCENT MANGANESE.
FIG. 62  MECHANICAL PROPERTIES OF 30.78 PERCENT NICKEL, 0.07 PERCENT CARBON, 0.71 PERCENT MANGANESE ALLOY AS A FUNCTION OF REVERSAL TEMPERATURE.  
15 MINUTES AT TEMPERATURES TO 800°C  
24 HOURS AT 1100°C
Figure 63. Microstructure of reversed 29.7 atom percent (30.8 weight percent) nickel alloy after holding at 500°C for 15 minutes.

Figure 64. Microstructure of 29.7 atom percent (30.8 weight percent) nickel alloy after holding at 600°C for 15 minutes.
Therefore the strength changes after reversals up to 700°C were primarily due to the recovery processes taking place within the austenitic grains at sites that had experienced reversal of the martensitic transformation.

The stress-strain curves for the specimen reversed at 800°C showed a marked drop in strength and a corresponding increase in ductility after reversal. Examination of the microstructure of this sample showed that complete recrystallization had occurred and the mechanical properties were almost the same as for the annealed specimen. No evidence of fine structure within the grains was present and the recrystallized grain boundaries were characteristically curved. Many irregularly shaped twins were present in the recrystallized grains indicating that the tendency to fault had been stimulated by the movement of the migrating boundaries through the strained and faulted matrix of the reversed austenite.

The recovery of the mechanical properties of the reversed 50.6 atom percent nickel alloy held at 650°C was generally similar to that above, but seemed to be much less sensitive to initial changes in structure. For example, the specimen held for eight minutes at 650°C was estimated to be 15 percent recrystallized but the strength properties were only slightly lower than for the sample held 5 minutes at 650°C and which was observed to have not undergone any appreciable recrystallization. The deformation behavior during the recrystallization is summarized in the engineering stress-strain curves presented in Figure 65 and the changes in properties with time at 650°C are shown graphically in Figure 66.

Examination of Figure 66 shows that the tensile and yield strengths decreased and total elongation increased as expected with longer times at temperature as recrystallization advanced. However, the reduction of area decreased unexpectedly from 86 percent in the as-reversed condition to 50 percent in the fully recrystallized condition.
FIG. 65  ENGINEERING STRESS-STRAIN CURVES FOR 30.6 ATOM PERCENT NICKEL-IRON ALLOY AFTER HOLDING FOR TIMES SHOWN AT 650°C
FIG. 66  CHANGE IN MECHANICAL PROPERTIES OF 30.6 ATOM PERCENT NICKEL-IRON ALLOY DURING RECRYSTALLIZATION AT 650°C
Closer inspection of the stress-strain curves shows that the nonuniform elongation (elongation after maximum stress) of specimens decreased with increasing times at temperature in agreement with the decrease in reduction of area. Inspection of the microstructure showed that the development of the grain boundary segregation discussed in the preceding section closely paralleled the ability of the specimens to deform after necking, and that intergranular cracking due to this segregation appeared throughout most of the gage length of the fully recrystallized specimen after fracture.

The development of a similar grain-boundary effect and brittleness was not observed in the 29.7 atom percent nickel alloy containing 0.07 percent carbon. Inspection of the microstructures of this alloy shown in Figures 23, 63, and 64 in comparison with the microstructures of the other alloys of higher purity reveals that fine particles are dispersed throughout the austenitic matrix. It is quite possible that the carbon has been effectively removed from solution by precipitation with another impurity element (perhaps manganese) in this alloy and so is not free to segregate at the grain boundaries as recrystallization occurs.
V. CONCLUSIONS

The present investigation of the behavior and structure of the austenite produced by the reverse martensitic transformation has established the following points:

1) Complete stabilization of reversed austenite to -196°C was produced in 29.4, 30.6, and 32.4 atom percent nickel-iron alloys after four or five cycles of direct and reverse transformation during slow heating and cooling. This stabilization is attributed to the diffusion-controlled formation of ferrite and the accompanying enrichment of the surrounding austenite in nickel made possible by heating and cooling during the reversal at rates of approximately 3°C per minute.

2) However, no stabilization of the reversed austenite results if the successive reversal cycles are performed by rapid heating and cooling.

3) The $M_s$ of reversed austenite in the 32.4 atom percent nickel alloy is raised up to 35°C after cycles of rapid reversal. This stimulation of the initial stages of martensitic transformation is consistent with either the embryo or stacking fault theories of the martensitic transformation.

4) The strain energy introduced into austenite by the reverse martensitic transformation provides sufficient driving force for recovery and eventual recrystallization on heating above the reversal temperature.

5) Recovery occurs by the formation of microscopically observable twins and the disappearance of the diffuse-etching regions characteristic of austenite immediately after reversal.

6) Stacking faults in iron-nickel austenite appear to be formed as a result of the reverse martensitic transformation on the basis of microscopically visible twinned structures that develop after cycles of
reversal and the irregularly shaped twins that appear with high frequency after recrystallization.

7) The strength properties of reversed austenite in iron-nickel alloys are increased markedly (up to 177 percent) over their values in annealed austenite as a result of cyclic martensitic transformation, with the greatest increase occurring after the first cycle. Strengthening is discussed in relation to the presence of stacking faults.

8) The martensitic structures produced from reversed austenite are strengthened significantly but much less so than the austenite. Observable morphological changes account, at least in part, for the strengthening of the martensite.

9) The mechanical properties of reversed austenite change somewhat during recovery, but return rapidly to the values of annealed austenite once recrystallization has been initiated.

10) Recrystallization of the reversed austenite occurs primarily by strain-induced boundary migration characteristic of slightly deformed metals, even though the mechanical strengthening due to reversal corresponds to a large amount of plastic deformation.

11) Sigmoidal kinetics are observed for the recrystallization of reversed austenite and the average growth exponent was calculated to be about 3, indicating three dimensional growth of the recrystallized areas.

12) The temperature dependence of recrystallization of the reversed austenite is not consistent with any single thermally activated process. Segregation of carbon to the migrating grain boundaries is believed to account for the observed high apparent activation energies as well as the intergranular brittleness of recrystallized specimens and the unusual grain-boundary etching effects of the austenite.
VI. SUGGESTIONS FOR FUTURE WORK

Many questions of a fundamental nature are raised by the behavior of austenite produced by the reverse martensitic transformation observed in this investigation. One of the foremost is the faulting tendency of iron-nickel austenite in the annealed and cold-worked condition as opposed to the reversed condition. At the present time, the stacking-fault energy in the former condition is considered to be high, but this investigation faults suggests that stacking faults do form as a result of the reverse martensitic transformation. An investigation to study faulting and other imperfections directly by transmission electron microscopy would therefore be of great value in resolving the actual faulting situation in iron-nickel alloys.

The stimulation of the initiation of the martensitic transformation after reversal leads to the theoretical question of martensite nucleation. It appears that appropriate heat treatment, i.e., annealing versus reversal, could produce structural changes in a single alloy that would allow experimental testing of both embryo and stacking fault theories of martensitic nucleation.

An investigation to determine the mechanism of the formation of the reversal twins observed in partially recovered austenite would also be worthwhile. Determination of the structure and crystallography of these twins and their crystallographic relation to the structure of martensite would contribute significantly to a fuller understanding of the martensitic transformation.

Recrystallization by strain-induced boundary migration of reversed iron-nickel austenite is one of the few examples of this process reported. Critical experiments could be designed to test existing theories of grain boundary migration during recrystallization. Comparison with recrystallization
studies of iron-nickel alloys after conventional cold working would also be fruitful.

The nature of the carbon segregation at the migrating boundaries, could be studied effectively with the electron microscope and the contribution of carbon to recrystallization kinetics could be assessed by complete decarburization of the iron-nickel alloys.

Increased amounts of carbon in the reversed austenite would be expected to contribute even further to strengthening, and therefore an evaluation of the effect of carbon and other elements on strengthening and other aspects of the reverse transformation would be worthwhile.
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BIOGRAPHICAL NOTE

This author was born in Philadelphia, Pennsylvania on May 14, 1933. He was graduated from Cheltenham Senior High School, Elkins Park, Pennsylvania in June 1951, and received the degree of B. Met. E., Cum Laude from Lehigh University in June 1955. He was employed in the metallurgical development department of the Superior Tube Company, Norristown, Pennsylvania from June 1955 to September 1956.

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