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Patricio L. Mangonon, Jr.
(Ph.D. Thesis)

August 1968

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STRENGTHENING AND PHASE TRANSFORMATIONS IN 304 STAINLESS STEEL

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ABSTRACT

The $\alpha'$ martensite transformation in 304 stainless steel was induced only by deformation which resulted in very considerable increases in both yield and tensile strengths. Further increase in these properties was achieved by aging the material deformed at -196° at temperatures from 200°C to 400°C. The latter increase in strength depended on the amount of prior deformation. Magneto-, x-ray and transmission electron metallographic techniques revealed that the most probable cause of further strengthening upon aging is the formation of thermally-nucleated martensite.

Detailed transmission electron microscopy revealed rather convincingly that the $\epsilon$ phase is indeed a phase and that the sequence in the transformation is $\gamma \rightarrow \epsilon \rightarrow \alpha'$. Formation of $\alpha'$ is favored at intersections of two $\epsilon$ bands. The orientation relationship between $\gamma$ and $\alpha'$ was found to be predominantly the Nishiyama relationship at the start which changes to the Kurdjumov-Sachs relationship on further deformation. A mechanism based on the most probable atom movements from the $\epsilon$ - hcp (faulted $\gamma$) structure to the bcc structure is proposed which predicts the habit plane, shear direction and amount of shear.
I. INTRODUCTION

Aside from their use in corrosive media, the 18-8 austenitic stainless steels find wide application as structural materials in missile and space vehicles after suitable thermal-mechanical treatment, which is possible in alloys containing metastable austenite. It has been found that the rather low yield and tensile strengths of these alloys in the well-annealed condition can be considerably improved by deformation at room and sub-zero temperatures. During the deformation, the austenite is converted to martensite which produces an increase in strength and work-hardening behavior of the alloys. The amount of martensite produced depends on the amount and temperature of deformation and also on the parameters $M_s$ and $M_d$ of the alloys. By definition the $M_s$ is the temperature at which the martensite transformation starts spontaneously on cooling. On the other hand, $M_d$ is the temperature above which no martensite transformation can be induced by deformation. Both the $M_s$ and $M_d$ are constant parameters for a given austenite composition which by their definitions indicate that the martensite transformation occurs spontaneously below $M_s$ or can be induced by deformation at temperatures between $M_s$ and $M_d$. Since no martensite can be induced to transform above $M_d$, the $M_d$ can therefore be taken as a measure of stability of the austenitic alloy against transformation to martensite. However, the composition-dependent $M_d$ is directly related to $M_s$. Since the $M_s$ is empirically known to be depressed by almost all common alloying elements in steels except Co, Eichelman and Hull proposed the stability factor $S = (\text{Ni}) + 0.68(\text{Cr}) + 0.55(\text{Mn}) + 0.45(\text{Si}) + 27(\text{C+N})$, in which the weight percentages of the elements are inserted in
the respective parentheses. High values of $S$ indicate alloys which are more resistant to the martensitic transformation.

The transformation structures of metastable Fe-Cr-Ni austenites which are obtained by spontaneous transformation or by strain-induced transformation have been widely investigated using both optical $^{12-17}$ and electron microscopy $^{17-23}$ in conjunction with x-ray techniques. In almost all of these investigations, the presence of an $\varepsilon$-(hcp) phase which is closely associated with the $\alpha$-(bcc) phase has been reported. The existence of the $\varepsilon$-phase is also a well-known fact in Fe-Mn alloys $^{24-27,31-33}$.

In line with current concepts $^{28,29}$ the transformation of the $\gamma$-(fcc) phase to the $\varepsilon$- and $\alpha$-phases occurs martensitically since a shape deformation is involved as evidenced by surface-relief effects $^{15,19}$. The $\varepsilon$ appears as sheets on $\{111\}$ while the $\alpha$ is contained within these sheets. Kelly and Nutting $^{22}$ reported that the $\alpha$ formed as needles. Lagneborg $^{17}$ found that the $\alpha$ appeared as needles when it is strain-induced, and as long plates within disc-shaped volumes when produced directly by cooling. Reed $^{14}$ found the $\alpha$ as plates while Kelly $^{23}$ and Reed and Breedis $^{30}$ reported the $\alpha$ as laths.

The orientation relationship between $\gamma$ and $\varepsilon$ is such that close-packed planes and directions are parallel in the two phases while that between $\gamma$ and $\alpha$ was always found to be very close to the Kurdjumov-Sachs (K-S) relationship $^{15,17,18,23}$. Thus, a particular variant of the relationship is

$$
(111)_\gamma \parallel (0001)_\varepsilon \parallel (101)_\alpha
$$

$$
[1\overline{1}0]_\gamma \parallel [\overline{1}2\overline{1}0]_\varepsilon \parallel [1\overline{1}1]_\alpha
$$
It has been found that the long direction of the needles\textsuperscript{17} the plates\textsuperscript{14,17} and the laths\textsuperscript{23} is parallel to the \langle 110 \rangle. Habit planes of the plates and laths were reported as \{225\}\textsubscript{\gamma}, \textsuperscript{14,15,17} \{259\}\textsubscript{\gamma} and \{112\}\textsuperscript{23}.

In spite of the numerous investigations so far, there is still the question of whether the \(\varepsilon\) is a transition phase between \(\gamma\) and \(\alpha\). The consensus of opinion is that \(\varepsilon\) is an intermediate phase.\textsuperscript{12-18} Cina\textsuperscript{12,13} felt that the experimental evidence showing the \(\varepsilon\) forming and later decreasing can only be explained by considering the \(\varepsilon\) as a transition phase. Reed\textsuperscript{14} showed by stain-etching the \(\alpha\) that long \(\alpha\)-plates are induced alongside the \(\varepsilon\)-sheets after repeated cycling from room temperature to sub-zero temperatures. Using transmission electron microscopy, Venables\textsuperscript{18} and Lagneborg\textsuperscript{17} showed that the \(\alpha\) is preferentially nucleated at intersections of two \(\varepsilon\)-bands or an \(\varepsilon\)-band and an active slip plane. On the other hand, Otte and Dash\textsuperscript{19-20} and Breedis\textsuperscript{21} have claimed that the \(\varepsilon\) merely formed as a result of the transformation, \(\gamma \rightarrow \alpha\). The accommodation strain during the latter transformation was supposedly sufficiently high to cause the formation of \(\varepsilon\) because of the low-stacking fault energy in these austenites.\textsuperscript{34} Furthermore, Goldman, et al.\textsuperscript{35} did not obtain a separate \(M_s\) temperature for the transformation, \(\gamma \rightarrow \varepsilon\), which must be above the \(M_s\) of the \(\gamma \rightarrow \alpha\) transformation. On this basis it was concluded that the \(\varepsilon\) is not a transition phase.

Further strengthening of the metastable 18-8 austenitic steels can be achieved upon aging after sub-zero deformation (sometimes called "Zerolling"). Mayne\textsuperscript{5} and Floreen and Mihalisim\textsuperscript{6} found that upon aging at 425°C (800°F) as much as 60,000 psi increase in both yield and tensile strengths was obtained. No explanation was given for this behavior and
it was not established that 425°C was the optimum aging temperature.

Chukhleb and Martynov did a more systematic study by aging their samples from room temperature to 600°C after the sub-zero deformation. The response to the aging treatments was followed by observing the hardness and magnetic susceptibility of each sample. The occurrence of a secondary hardness peak was established in the range from 225°C to 425°C. With the hardness increase, a corresponding increase in magnetic susceptibility was also observed. In fact, a one-to-one correspondence of these two variables was obtained during the aging treatments. Below 225°C, no change was noted. In contrast, aging at 425°C produced a decrease in magnetic susceptibility from that of the as-deformed sample. This decrease in magnetic susceptibility was reconciled with the reversion of α to γ. The secondary hardness increase depended on both the degree of deformation and time at the aging temperature. They established that 400°C (752°F) was the optimum temperature for 18-8 type steels.

It appears that this phenomenon of secondary hardness on aging the deformed 18-8 steel is entirely different from that due to precipitation hardening. Chukhleb and Martynov demonstrated that at the optimum temperature of 400°C, no overaging was observed, i.e., no decrease in hardness was noticed when aging was done from one hour to 120 hours. There was in fact a very slight increase in hardness and amount of magnetic phase when aging was done for 120 hours versus aging for only one hour. The time dependence of the aging response was primarily during the first hour where the significant increase in properties was observed.

Chukhleb and Martynov proposed that the strengthening upon aging was due to the decomposition of the austenite by the precipitation of carbides.
with subsequent formation of α around these precipitated carbides. It was further proposed that additional strength can be obtained also by precipitation of carbides in the α which was previously formed during the sub-zero deformation. Except for the magnetic susceptibility measurements, no other structural evidence was presented to support these proposals.

**Purpose of Research**

Since the secondary strengthening phenomenon differs from the usual precipitation hardening process, the need to study the exact nature of this phenomenon was felt. Knowledge of this mechanism might lead one to design high temperature materials, since it was shown that at the optimum aging temperature, no softening was observed with time. By understanding the nature of this phenomenon, it is hoped that an alloy can be designed with an optimum aging temperature of 600°C-700°C, which would make it an attractive high temperature material.

The purpose of this research was to study the mechanism of this secondary strengthening. In addition to magnetic and x-ray techniques, detailed transmission electron microscopy was employed to examine the structures. It was hoped also that during the course of this study the question of whether the ε-phase is a transition phase or not could be answered.

Commercial 304 stainless steel was used for the investigation because of its freedom from strong carbide formers and its lower carbon content than the 301 type. The object here was to minimize the complications arising from precipitation. The composition of the alloy used was 18.03%Cr, 8.46%Ni, 1.32%Mn, 0.069%C, 0.015%P, 0.006%S, 0.72%Si, 0.08%Co.
and the balance Fe. Analysis of the alloy was given by the supplier.
II. EXPERIMENTAL TECHNIQUES

A. Specimen Size, Preparation and General Method of Investigation

Tensile specimens with dimensions shown in Fig. 1 were obtained from a 0.030-inch thick 304 stainless steel sheet stock. All specimens were annealed at 1100°C for 30 minutes in argon atmosphere and then quenched in water. Different amounts of tensile strains were introduced to the specimens at liquid nitrogen temperature, after which the respective amounts of magnetic phase were determined by measuring the saturation magnetization of the pieces before and after aging at temperatures of 100°C, 200°C, 300°C, 400°C, and 500°C. Aging time was 1-1/2 hours in boiling water for the 100°C aging and in a low-melting salt-bath (~150°C melting temperature) for the other temperatures. After the magnetization measurements, data on mechanical properties at room temperature were obtained by pulling the specimens in the Instron machine.

X-ray, optical and electron metallography were done only on the material within the original 2-inch gage marks of the tensile specimen where the amount of strain is exactly known. The three techniques were used on the same specimen, which makes the one-to-one correspondence of results unquestionable. Thus, x-ray and optical metallography were done first before preparing the specimen for transmission electron metallography.

B. Tensile Deformation and Tensile Data

Tensile deformation was performed with the set-up shown in Fig. 2. Two-inch gage marks were made on the gage sections of the specimens from which the percentage tensile strains (elongation in 2") were measured. The lower end of the specimen was first gripped and then the split styrofoam container lined inside with vacuum grease was placed sitting on the
lower grips. Liquid nitrogen was poured into the container and the level kept such that the 2-inch gage marks were always under liquid nitrogen. The grease acted as a sealant since it solidified at liquid nitrogen temperatures, thus keeping the loss of liquid nitrogen to a minimum. As soon as the specimen reached the bath temperature, the other end was also gripped. This procedure avoided any stresses being set up in the specimen, as would occur if the two ends were gripped first and the specimen then cooled.

The amount of tensile strain, i.e., percent elongation in 2", was obtained by stopping the Instron machine with crosshead speed of 0.5 cm/min. at a certain load level of the load-elongation curve and immediately loosening the grips. The reasons for this was Angel's evidence that the stress level controlled the transformation of austenite to martensite. In this way, reproducibility of strains was obtained within ±0.5% with strains of 4%, 10%, 15% and 20%.

After aging treatments and magnetization measurements were made, new 2-inch gage marks were made on the gage sections before obtaining the load-elongation curve at room temperature, from which the reported data of 0.1%-strain-offset-yield strength, ultimate tensile strength and percent elongation in the new 2-inch gage section were obtained.

C. Magneto-Metallographic Technique

Out of a number of quantities related to magnetic measurements, the saturation magnetization and Curie temperature are structure insensitive. Others, such as permeability, susceptibility and coercive force are highly structure sensitive which implies dependence on internal stresses and strains. Thus, a good measure of the volume percent of magnetic phase in
a material is its saturation magnetization which is independent of internal stresses and strains.

In the presence of an external magnetizing force, $H$ (in oerstads), the total induction in a material at any point is

$$B_T = \Gamma_m H + B_i = \Gamma_m H + 4\pi J$$  \hspace{1cm} (1)

where

- $B_T$ = total induction in gauss
- $\Gamma_m H$ = magnetic field strength in gauss
- $\Gamma_m$ = magnetic constant which is unity in the cgs system
- $B_i$ = intrinsic induction, i.e., increase in induction, above the field strength, which is contributed by the internal fields of the magnetic material when subjected to the external magnetizing force, $H$
- $J$ = pole strength per unit area or magnetic moment per unit volume.

(For a very good review of magnetic testing, see reference 70.) The quantity directly related to the amount of magnetic phase is $B_i$ and can always be calculated by subtracting the field, $H$ (cgs, $\Gamma_m = 1.0$) from the total induction, $B_T$.

Experimentally, the saturation magnetization is obtained by plotting the hysteresis loops at sufficiently high field strengths to ensure saturation. If the total induction $B_T$ is plotted against $H$, the normal hysteresis loop is obtained while an intrinsic loop is obtained when $B_i$ is plotted against $H$. The difference between the two loops is shown in Fig. 3 where it is seen that the normal loop steadily increases with increasing $H$ while in the case of the intrinsic loop, a plateau or constant $B_s$, indicating saturation magnetization, is obtained at high field strengths. Thus, the intrinsic loop is more desirable to calculate the volume percent of magnetic phase.
The experimental set-up that yields such an intrinsic loop is shown in Fig. 4a. Essentially, two search coils are placed side-by-side in an electromagnetic field. The positive terminals of the two coils are electrically connected through a divider (potentiometer) while the negative terminal of coil No. 1 is grounded and the negative terminal of coil No. 2 connects to an SLFS No. 1 (Square Loop Flux Standard), resistor R₁, integrator and finally to the Y post of an X-Y recorder. The X-post of the recorder is connected to the H coil via another SLFS No. 2 and integrator. In the absence of any magnetic material in the coils, the divider is adjusted such that by increasing the field strengths, H (X-coordinate), no signal is obtained in Y, which is the state called complete bucking of the two coils. After complete bucking, insertion of a magnetic material in coil No. 1 causes an emf signal in Y which is directly related to the change in flux, ΔΦ, caused by the magnetic material. The intrinsic flux density or induction, B₁, is obtained by dividing ΔΦ by NA, where N = number of turns in coil No. 1 and A = cross-sectional area of the material. (Compare this method to the differential thermocouple in differential thermal analysis.)

The relation of the emf signal, e_out at Y to ΔΦ is seen in the following. The change in flux dΦ in time dt is

$$e_{in} = \frac{d\Phi}{dt} \quad (2)$$

which goes as input signal to the integrator. The output signal, e_out, of the integrator is

$$e_{out} = \frac{1}{C} \int i \, dt \quad (3)$$

but since,

$$i = \frac{e_{in}}{R}$$
and substituting Eq. (2) into (4),

\[ e_{\text{out}} = \frac{1}{RC} \int e_{\text{in}} \, dt \]

thus,

\[ (\Delta e_{\text{out}}) = \frac{1}{RC} (\Delta \phi) \]  

(5)

The Y-scale was calibrated by introducing a known change in flux, \( \Delta \phi \), from the SLFS and knowing \( NA \),

\[ \Delta B = \frac{\Delta \phi}{NA} \text{, gausses} \]  

(6)

The Y-scale was adjusted to read in gausses per inch, and the sensitivity could be increased to yield an accuracy of \( \pm 10 \) gausses.

The H-coil was likewise calibrated and the X-scale adjusted to read so many oersteds per inch. The field strength, \( H \), was increased by increasing the current at the power supply. A current of 25 amps was sufficient to yield the plateau of the intrinsic loop which was obtained by means of a reversing switch in order to get \( \pm H \). The 25 amps corresponded to 5,700 oersteds. The effect of higher field strengths was tested by going to 8,000 oersteds, which gave no change in \( B_i \) from that at 5,700 oersteds. Therefore testing at higher \( H \) than 5,700 was unwarranted.

Magnetization measurements were only obtained from the gage sections of the tensile specimen where the amount of strain was accurately known. This was possible by having the grip sections of the specimens within large iron blocks (shown in Fig. 4b) such that the permeability was very, very low compared to the gage sections. Between the two iron blocks, which were supported by brass spacers, were the two search coils made of lucite spool wound, \( N = 3000 \) turns, with No. 30 copper wire.
The amount of magnetic phase was calculated from $B_s$ by using the reported specific saturation magnetization per unit mass (magnetic moment per unit mass) of 160.4 for 100% martensite of an alloy with almost identical composition.\(^9\) Confidence was gained in this value by plotting the specific saturation magnetization of Fe-Cr alloys\(^{69b}\) where a value of 163 was obtained. Since nickel decreased the specific saturation magnetization,\(^{69b}\) the lower value of 160.4 was used.

It was seen from Eq. (1) that

$$B_i = 4\pi J, \text{ gauss}$$

where $J$ = magnetic moment per unit volume. Thus, to get units of magnetic moment per unit mass, $\sigma_s$

$$\sigma_s = \frac{B_s}{4\pi \rho_\alpha}, \text{ magnetic moment per unit mass}$$

where $B_s$ = intrinsic saturation magnetization

$$\rho_\alpha = \text{density of } \alpha$$

$$\frac{1}{\rho_\alpha} = \left(\frac{1}{\rho_{Fe}}\right)(N_{Fe}) + \left(\frac{1}{\rho_{Cr}}\right)(N_{Cr}) + \left(\frac{1}{\rho_{Ni}}\right)(N_{Ni})$$

where $N_i = \text{fraction of } i^{th} \text{ component}$.

Thus

$$\frac{1}{\rho_\alpha} \approx \left(\frac{1}{7.87}\right)(0.74) + \left(\frac{1}{7.19}\right)(0.18) + \left(\frac{1}{8.90}\right)(0.08)$$

$$\frac{1}{\rho_\alpha} \approx 0.1287$$

$$\rho_\alpha = 7.77 \text{ gm/cm}^3$$

The volume percent $\alpha$ is therefore

$$C_\alpha = \frac{\sigma_s}{160.4} \times 100$$
D. X-ray Metallographic Technique

The x-ray technique to determine relative volume fractions of phases in a multiphase alloy is now well known.\textsuperscript{71,72} It consists of comparing the relative integrated diffracted intensities which are proportional to the volume fractions of the respective phases. The method assumes random orientation, and preferred orientation introduces serious errors.

The Norelco X-ray Diffractometer equipped with diffracted beam (200)-Lif crystal monochromator was used with CuK\textsubscript{\alpha} radiation. This monochromatization technique eliminated a lot of the x-ray fluorescence background, which made possible the use of CuK\textsubscript{\alpha} radiation.

The relative integrated intensities of peaks corresponding to the (10.1)\textsubscript{\epsilon}, (200)\textsubscript{\gamma}, and (200)\textsubscript{\alpha} were compared to calculate volume percents of \epsilon, \gamma, and \alpha. The patterns were obtained from the flat polished surface of the gage sections of the tensile specimens. These peaks were sufficiently separated that interference was highly unlikely. For each peak, the relative integrated intensity, I, is

\[ I = \left( \frac{I_0 e^{-4\mu}}{m^2 c^4} \right) \left( \frac{3 A}{32\pi r} \right) \left( \frac{1}{2} \right) \left[ |F|^2 \times P \times \text{LP} \right] \frac{e^{-2M}}{2\mu} \]  

constant = K

where the first two terms equal a constant, K, independent of kind and amount of diffracting substance, and

\[ v = \text{volume of unit cell} \]
\[ F = \text{structure factor} \]
\[ P = \text{multiplicity factor} \]
\[ \text{LP} = \text{Lorentz polarization factor} = \frac{1 + \cos^2 \theta \cos^2 \theta}{\sin\theta \sin 2\theta} \]
\[ \theta = \text{Bragg angle of each line} \]
\[ \theta_m = \text{Bragg angle for monochromator; for (200)-LiF, } \theta_m = 22^\circ 34', \]

for CuK\(\alpha_1\).

\[ e^{-2M} = \text{temperature factor} \]

\[ 1/2\mu = \text{Absorption factor for flat specimens} \]

Letting,

\[ R = \frac{1}{V^2} \left[ |F|^2 \times P \times LP \right] e^{-2M} \]

then in the three-phase material here,

\[ I_\alpha = \frac{K R_\alpha C_\alpha}{2\mu_m} \quad (10a) \]

\[ I_\gamma = \frac{K R_\gamma C_\gamma}{2\mu_m} \quad (10b) \]

\[ I_\varepsilon = \frac{K R_\varepsilon C_\varepsilon}{2\mu_m} \quad (10c) \]

where \( \mu_m = \text{linear absorption coefficient of mixture} \)

\( C_i = \text{volume fraction of } i^{th} \text{ phase.} \)

Equations (10) together with the fact that

\[ C_\alpha + C_\gamma + C_\varepsilon = 1.0 \quad (11) \]

will yield the \( C_i \)'s. Factors for the calculation of \( R_i \) are shown in Table I while \( C_\alpha \) was obtained from Eq. (8).

E. Optical Microscopy

The gage sections were ground flat on both sides with a series of emery papers before electropolishing in a 1:4 Perchloric-Acetic Acid solution at 12 dc volts and < 10\(^\circ\)C temperature. Etching was done electrolytically in a saturated oxalic acid solution. Observation and photography were done with a Carl Zeiss Optical Microscope. After optical
microscopy, the same specimen was used for the above x-ray work and transmission electron microscopy.

F. Transmission Electron Microscopy

Foils for transmission electron microscopy were prepared by first chemical polishing in the following solution, 50 ml H$_2$O$_2$, 15 ml 37.8% aqueous HCl, 20 ml 48% aqueous HF and 15 ml H$_2$O which was placed in an ice bath to keep the temperature down. Uniform thinning of the specimens was obtained down to 2 mils with no preferential attack of the edges. Initial electropolishing in the above perchloric-acetic acid solution was done until perforations were observed. Very good foils were obtained by doing the final electropolishing in the well-known chromic-acetic acid mixture at 10 dc volts and < 5°C temperature.

Observations of the foils were done at 100 kV in a Siemens Elmiskop IA microscope. High resolution gun-tilt dark field pictures were always obtained in addition to the bright-field picture and electron diffraction. As much as four, sometimes five, patterns appear in one plate and the series of bright and dark field pictures facilitated indexing these patterns and enabled unambiguous interpretation to be made of the features observed in the bright field image.

G. Quenching Experiments

In an effort to determine whether $\alpha$ can be formed on cooling, samples were quenched to liquid nitrogen (78°K) and helium temperatures (4°K). A sample was also held at dry ice temperature (-78°C) for three days to test for isothermal transformation to martensite as reported by Lagneborg. Structures of these specimens were also observed in the electron microscope.
III. EXPERIMENTAL RESULTS

A. Magneto-Metallography

The results of saturation magnetization measurements obtained from the 2-inch gage lengths of the tensile specimens are shown in Figs. 5, 6, 7, and Table II. Each point in the figures is an average of at least three runs with accuracy of ±20 gauss. Figure 5 shows the increase of saturation magnetization as the tensile strain introduced at liquid nitrogen temperature increases. The magnetization was directly related to the amount of α'. This conclusion arises from our knowledge that the austenite and the ε-hcp phase (shown to be present by x-ray techniques and transmission electron microscopy) which can be also considered as a faulted austenite are both paramagnetic. This being the case, the values of $B_s$ can be used directly to calculate volume percent of α' in the 3-phase mixture according to Eqs. (7) and (8). Thus, as expected, the amount of α' increases as the strain increases. Although it appears that a threshold amount of strain (Fig. 5) might be needed to yield some magnetization, a polycrystalline sample which was tensile-strained 0.2% gave a saturation magnetization $B_s = 12$ gauss.

In Fig. 6, one sees the change in saturation magnetization, $ΔB_s$, from that of the as-deformed samples after aging at the temperatures for 1-1/2 hours (see also Table II). It is seen that aging at 100°C and 200°C resulted mostly, except for the 15% tensile strain, in a very slight negative change, $ΔB_s$, while aging at 300°C and 400°C resulted in an increase in $B_s$, i.e., $+ΔB_s$, except for the 15%. It appears from Fig. 6 that aging at temperatures up to 400°C of the 15% strained sample did not yield negative $ΔB_s$ while aging of the 4% strained sample yielded negative $ΔB_s$. 
at all aging temperatures (see also Table II). From Fig. 7 it appears that a certain amount of strain is needed in order to obtain positive $\Delta B_s$ when aging is done at 400°C. It suggests further that a larger amount of strain is needed to get positive $\Delta B_s$ when aging is done at 300°C. The change $\Delta B_s$ at 400°C for 20% tensile deformation when converted to percentage change in $\alpha$ amounts to about 5%.

The occurrence of the peak in $\Delta B_s$ at 400°C (Fig. 6) agrees with the results of Chukhleb and Martynov who measured the magnetic susceptibility of the aged samples. However, Chukhleb and Martynov did not observe by their method the slight drop in magnetization of the samples aged at 100°C and 200°C.

B. Mechanical Properties

The results of tensile properties obtained at room temperature are shown in Figs. 8a, 8b, 8c and in Table III. From Figs. 8a and 8b, it appears that the response to the aging treatment, as far as yield and tensile strengths are concerned, depends on the amount of strain given to the specimens at liquid nitrogen temperature. Both the yield and tensile strengths for the sample strained 4% were consistently lower after aging than the as-deformed properties (R.T. aging). Samples strained 10% showed also consistently lower tensile strengths after aging while the yield strengths varied abnormally - increasing when aged at 100°C and decreasing when aged at 200°C, then coming to the level of the as-deformed samples on aging at 300°C and 400°C.

On the other hand, the yield strengths of samples strained 15% and 20% (Fig. 8a) are generally higher than the as-deformed samples, except for the samples aged at 500°C which have lower properties. For the
samples strained 15%, it appears that aging at 200°C gave the largest increase in yield strength while samples strained 20% yielded largest positive change at 400°C aging. The increase of 40,000 psi in yield strength upon aging a 20% strained sample at 400°C is significant.

The ultimate tensile strengths (UTS), see Fig. 8b, of the samples strained 15% are relatively unchanged for aging temperatures from 100°C to 300°C with only a slight increase on aging at 400°C. For the samples strained 20%, a decrease in UTS is observed on aging at 100°C while an increase is observed on aging from 200°C to 400°C. Again, aging at 500°C decreases the UTS of the samples.

For comparison, the yield strengths and UTS of annealed 304 stainless steel when tested at room temperature and at liquid nitrogen temperature are also shown in Figs. 8a and 8b. It is seen that the yield strength, Fig. 8a, at -196°C did not vary much from that at room temperature which typifies the variation of yield strengths with temperature of fcc metals. From Fig. 8b, the UTS of 304 at -196°C is about 100,000 psi more than that at room temperature. Although the tensile strengths of fcc metals vary considerably with temperature, part of the above increase in UTS must be due to the strain-induced martensite.

The percent total elongation in 2-inch gage length generally decreases as the amount of strain introduced at the LN temperature is increased (Fig. 8c). Again for comparison, data on the annealed sample, tested at room temperature and at LN temperature are shown in Fig. 8c. The data shown for 10% strain were obtained from samples that broke outside the gage marks. These, with 3 similar points for the 20% strained curve,
are shown with asterisks. It appears, however, that for a particular amount of strain, the elongation is relatively unchanged (4% strain) or increases slightly (15% strain) after the samples were aged at all the temperatures. For the 15% strain samples, a very significant result is that an increase in yield strengths and tensile strengths coupled with unchanged or slightly increased ductility is obtained. In light of this, then for the 20% strained samples, which had about 217,000 psi yield strength, the percent total elongation must be a minimum of 10% which is indicated by two data points (see also Table III) taken from samples which broke within the gage marks. Any material with 217,000 psi yield strength and at least 10% elongation makes an attractive structural material.

The effect of further thermo-mechanical treatment after aging is shown in Table IV. Although deformation here is by rolling, the same phenomenon is observed resulting in almost identical results in both yield and tensile strengths of samples given 20% deformation, either as 20% reduction in thickness or 20% total elongation in 2" for the as-deformed and 400°C aging samples. The similarity in properties is also brought out by the almost identical amount of α² present after either type of deformation.

Comparing samples A21 and A22, it is seen that rolling at 400°C results in a larger increase in yield strength than that obtained by merely aging. A larger amount of α was also obtained in the process. With the same amount of deformation, rolling at 400°C also gave higher properties than that obtained by rolling at room temperature and at liquid nitrogen (compare A22 with A31 and A41). Further aging of samples A31 and A41 gave properties which exceeded that of A22. It appears that the aging
at 400°C invariably results in type-III load-elongation curve characterized by no work hardening and sudden drop in load as soon as yielding is obtained.

C. X-ray Metallography

We have seen that $\alpha'$ was induced by the deformation as evidenced by the magneto-metallographic technique. The presence of the $\epsilon$-hcp phase is revealed both by x-ray and transmission electron metallography. The x-ray evidence is shown in Fig. 9a where the presence of the three phases, $\epsilon$, $\alpha'$, and $\gamma$ after 10% tensile deformation is revealed by the diffractometer peaks of each phase. The lattice parameters of the phases are essentially those reported by Reed and Gunther. For each amount of strain, the relative volume percent of the three phases was determined from the integrated intensities of the $(200)_\gamma$, $(10.1)_\epsilon$ and $(200)_\alpha'$ (not shown in Fig. 9) peaks. Figure 10 shows the results of this calculation which indicates that the $\epsilon$-phase reaches a maximum at about 5% tensile strain and then decreases. The results agree with what other investigators found to be evidence that $\epsilon$ must be a transition phase between $\gamma$ and $\alpha'$. Figure 10 suggests also that $\gamma$ decreases linearly as strain increases and conceivably $\gamma$ could be transformed completely to $\gamma$ and $\epsilon$ by extrapolating the line to intersect the abscissa.

It was shown above that the same degree of deformation by rolling in terms of percent reduction in thickness resulted in almost identical amounts of $\alpha'$ as that deformed in tension. The question now is whether the $\epsilon$ also forms during rolling. Such a doubt was raised by Floreen and Mihalism who reported that the $\epsilon$ was not detected. The x-ray diffractometer pattern (Fig. 11) of a sample 10% deformed by rolling at LN temperature reveals in the present investigation also the occurrence of
a-ε during rolling. This result suggests that apparently the same phenomena occur in tension and compression.

The effect of aging on the amount of ε phase is shown in Figs. 9b and 9c. Aging at 400°C (Fig. 9c) resulted in the disappearance of the ε, at least to the extent that it was not detected by x-rays. At 200°C, some ε was still detectable. The result of aging at 100°C, which is not shown here, revealed the amount of phase to be unchanged. This suggests that 200°C is near the reversion temperature of ε, as reported by Reed,¹⁴ and says that the ε will remain stable when chemical polishing with solution temperatures up to 70°C is to be done in the preparation of foils.

Figure 9b and 9c tell us also that most of the ε reverts back to the γ-phase as shown by the (111)γ and (200)γ peaks. There is also a slight increase in α' as shown in Fig. 12 for a sample deformed 20% and aged at 400°C which suggests that part of ε is also used up to form new γ. The increase in area (integrated intensity) of the (200)α'-peak after aging was 5% over that of the as-deformed state. This agrees well with the magnetic measurements which showed an increase of 5% in the saturation magnetization upon aging the 20% tensile deformed sample.

D. Optical Microscopy

An optical micrograph of the annealed sample is shown in Fig. 13. The average grain size of the samples is about 3 mm. During deformation, the grains are deformed heterogeneously as shown by Fig. 14 for a 2% tensile deformed sample because of orientation effects. Figures 15a and 15b are the optical micrographs of a sample tensile deformed 20% at LN temperature before and after aging at 400°C, respectively. The pits seen in Figs. 13-15a are from electropolishing. The effect of aging is seen to be general darkening of the structure indicative of tampering process and precipitation of carbides.
E. Transmission Electron Microscopy

1. Identification of the phases.

A typical transmission electron micrograph obtained from partially transformed metastable 18-8 austenitic stainless steels is shown in Fig. 16. The dark band is the ε-hcp phase with the α' (btd) denoted by M being contained in the dark band at the intersection of 2 bands. The austenite is the area between bands denoted by A.

Identification of these phases was done by taking high resolution gun-tilt dark-field images in addition to the diffraction pattern and bright-field image. Indexing of any diffraction pattern which contains several different orientations or structures is greatly facilitated with the aid of the dark-field images. Figure 17a is the diffraction pattern of the central area of the bright-field picture, Fig. 17b. A schematic sketch of the diffraction pattern is shown in Fig. 17c with the spots A, B, C, D, E, and F being tilted to the optic axis to get the corresponding dark-field images shown in Figs. 18a, 18b, 18c, 18d, 18e, and 18f, respectively. (A 10μ size objective aperture was used to separate the spots.) The circular dark-field images were taken with the intermediate aperture (20μ) left in the optic axis to outline the central area in Fig. 17b. It is seen that the images (Figs. 18b and 18e) from spots B and E (Fig. 17c) lighted the same areas which says that spots B and E belong to one structure. The diffraction pattern is very nearly symmetrical and the net of reciprocal lattice points constructed with spots B and E was indexed and found to be that of an fcc [110] zone. The spots B and E turned out to be [111] and [002], respectively. Similarly, it is seen
that Figs. 18a, 18c, and 18d lighted essentially the same features and again spots A, C, and D must belong to one structure. Knowing the camera constant from the indexed fcc matrix pattern, the interplanar spacings corresponding to these 3 spots were found to be essentially those from the \((01\bar{1}0)_\epsilon\), \((01\bar{1}1)_\epsilon\) and \((0\bar{1}\bar{1}1)_\epsilon\) hcp planes respectively, with the zone axis being the \([2\bar{1}\bar{1}0]_\epsilon\). The presence of the hcp phase was further confirmed by the occurrence of the double diffraction spot (0001), Figs. 17a and 17c. In the presence of 3 phases, double diffraction can be very complicated but the spot indicated in Fig. 18c indexed uniquely as 0001 hcp. Although double diffraction spots do not appear in patterns of the cubic crystals, bcc and fcc, if they occur independently, the situation is greatly altered when the 2 phases are present together.

The last dark-field image, Fig. 18f, due to spot F (Fig. 17c), lighted the white area in the central portion of Fig. 17b together with the matrix. From the previously indexed patterns, fcc and hcp, it was seen that the \((0002)\) hcp and \((111)\) fcc were coincident with F. Since the feature lighted up in Fig. 18f was neither fcc nor hcp, the logical conclusion is that it is the \(\alpha'\) bcc phase. The calculated interplanar spacing was very close to the \((110)_{\alpha'}\) and together with spot G the bcc phase was found to be close to the \((100)_{\alpha'}\) orientation.

The positions of the matrix fcc spots and the hcp spots in Fig. 17a correspond to those predicted ideally (\(c/a = 1.63\)). The occurrence of both the fcc and hcp spots simultaneously at their normal positions indicates that there is no shifting of the fcc spots. The interpretation of Fig. 17a is simply the superposition of three diffrac-
tion patterns with each phase forming its own diffraction net. The [2\(\overline{1}\)0] hcp pattern was thus superposed on the [110] fcc matrix pattern and was not treated as a shifted fcc pattern. The absence of shifting can mean either, 1) that the shifting of fcc spots due to random faulting as applied to x-rays \(^{41}\) does not apply, or 2) that we have regular faulting on every other (111). The second possibility is the more probable since in stress-induced martensitic transformation of this sort, a maximum shape change is observed when only one out of three possible \(a/6(112)\) shears operates on a given set of (111) planes. \(^{77}\) Operation of the three \(a/6(112)\) shears alternatively results in a net shape change of zero.

The \(\epsilon\)-hcp is seen to be coherent with \(\gamma\) and forms as very thin sheets on (111)\(\gamma\) as evidenced by the streaking and its (111) direction in the diffraction pattern. In Fig. 17b, these sheets are edge-on, i.e., perpendicular to the plane of the foil, and are seen projected as lines running bottom left to top right corner. On the other hand, the \(\alpha^\prime\) appears to have no regular shape which differs from the lenticular martensites observed in other iron alloys. Within the \(\alpha^\prime\) are noted some contrast effects (indicated in Fig. 17b by the small circles) which are seen to be inherited by the \(\alpha^\prime\) from the \(\gamma\). The sizes of these defects are too large to be ascribable to interstitials but could be either precipitates in the \(\gamma\) or surface artifacts introduced by the polishing operation. No independent precipitate spots were noticed in the diffraction pattern.
2. The $\epsilon$ as a transition phase; nucleation and growth of $\alpha'$

Figure 17b suggests that $\alpha'$ is nucleating and growing at slip band intersections so if one considers $\epsilon$ as a phase it must be an intermediate phase. In no case was the $\alpha'$ observed to form in the undeformed regions of $\gamma$. Areas such as A in Fig. 17b indicate that intersections of two $\epsilon$-bands (two faults) are preferential $\alpha'$-nucleation sites. The bigger-tipped arrows point to small $\alpha'$ crystals at the intersections of two faults. For clarity, an enlargement of this area is shown in Fig. 19. As a result of the intersection process, a noticeable displacement of the intersected fault is observed such as that shown in B, Fig. 17b. Further discussion of this point will be presented later.

The manner in which the $\alpha'$ grows can be seen with the aid of the dark-field image, Fig. 18d, which was seen to result from the tilting of the (0111) hcp spot (spot D in Fig. 17c) into the optic axis. The dark area D in Fig. 17d is the $\alpha'$ bcc which lighted up with the [110] bcc spot, Fig. 18f. Within D, it is seen that a small fleck of $\epsilon$ lighted up with the (0111) hcp spot. This fleck is indicated in Fig. 17b by the small arrow. A similar area in C points also to the fact that the $\epsilon$ phase can be within the $\alpha'$. Thus it appears that the $\alpha'$ grows in a manner analogous, if one might recall, to the classical recrystallization process by engulfing $\epsilon$(or faulted $\gamma$). In the recrystallization of deformed crystals, the new equiaxed crystals grow at the expense of the strained crystals.

Further evidence of the nucleation and growth of $\alpha'$ is provided by Figs. 20 and 21. Figure 20b is the bright-field picture of the diffraction pattern, Fig. 20a, with the sketch of the pattern shown in Fig. 20c. The spots A, B, and C (Fig. 20c) when tilted to the optic axis gave the
dark-field pictures, Figs. 20d, 20e, and 20f, respectively. It is seen (Fig. 20c) that spot A is from the bcc phase which has a high dislocation density (Fig. 20b). In Fig. 20d, the dislocations are seen to reverse contrast. The spots B and C both belong to the faulted \( \gamma \) and Figs. 20e and 20f reveal again the presence of flecks of faulted \( \gamma \) within the bcc phase.

Areas marked N in Fig. 21 suggest also that \( \alpha' \) nucleation occurs near twin and grain boundaries. Area G in Fig. 21 suggests further that the nuclei at N grow sideways and lengthwise and show how the faulted \( \gamma \) can be within the \( \alpha' \) crystals. Area D shows how the dislocations in the \( \alpha' \) are inherited from the debris of the faulted regions. What appears to be some internal twinning of \( \alpha' \) in \( R \) (Fig. 21) is believed to be still the faulted (\( \varepsilon \)) structure, evidenced by similarity to D. At the boundaries of two \( \alpha' \) crystals such as T, striations are observed which look very similar to the very fine twinning observed in Fe-Ni-C alloys. However, in the absence of \( \alpha' \) twinned diffraction spots, it is difficult to be absolutely sure that this is in fact twinning.

Figure 22 shows the formation of \( \alpha' \) near the grain boundaries. It is especially to be noted that \( \alpha' \) was apparently nucleated on the side of the grain boundary where considerable faulting has occurred. Although intersections of \( \varepsilon \)-bundles, twin and grain boundaries are preferred sites of nucleation of \( \alpha' \), it is shown in Fig. 22 that \( \alpha' \) can form alongside of the fault. This fact is better seen in Fig. 23 where the long direction of \( \alpha' \) runs parallel to the length of the fault. This phenomenon was also observed by Reed upon cyclically quenching a similar metastable austenitic alloy from room temperature to sub-zero temperature. Such an
observation is again indicative of the $\epsilon$ being a transition phase.

All the evidence presented thus far points to the $\epsilon$ or faulted $\gamma$ as an intermediate stage in the transformation $\gamma \rightarrow \alpha'$. A further confirmation of this belief is provided by the pictures in Fig. 24. Figure 24a is the diffraction pattern of the bright-field picture (Fig. 24b) while Figs. 24c and 24d are the dark-field pictures from the matrix and hcp spots, respectively. It is seen in Figs. 24b and 24d that the $\epsilon$ occurs independently and continuously in one grain (twin) with no $\alpha'$ crystals associated with it. This independent occurrence of $\epsilon$ is enough to refute the belief that $\alpha'$ must nucleate first which, upon formation, will fault the austenite.\textsuperscript{19-21} The strong implication of the latter belief is that the $\epsilon$ cannot independently occur and expect the $\alpha'$ to be always contained within the $\epsilon$-band. The evidence provided by Figs. 24b and 24d is contrary to this alternative mechanism.

It is also observed in Fig. 24b that the adjoining grains are considerably faulted. The nucleation of $\epsilon$ appears to occur both at the twin boundaries and within the matrix, as evidenced by the small flecks of $\epsilon$ between main bands in Figs. 24b and 24d.

3. Structures of a 13% tensile strained sample at R.T.

Having seen the $\epsilon$ to be apparently an intermediate phase for samples strained at LN temperature, the structure of a sample 13% tensile-strained at room temperature was examined. This amount of deformation at room temperature was not sufficient to induce the $\alpha'$ formation as evidenced by its being non-magnetic. At room temperature, the steel turned magnetic only at the necked region of the tensile specimen after about 55-60% total elongation was obtained. X-ray diffractometer traces of the 13%
tensile strained sample revealed also no α' but at the same time failed to reveal the ε-phase. However, transmission electron microscopy established the formation of ε in light of the evidence in Fig. 25. The ε appear as flecks (Figs. 25b and 25c) similar to those observed in Figs. 24b and 24d. With the foil orientation close to [110]γ, the ε appears as thin sheets on [111]γ evidenced by the streaking in the diffraction pattern, Fig. 25a, and its long direction runs parallel to the (211)γ. The streak in the diffraction pattern, Fig. 25a, is maximized about one-sixth the distance between spots along the (111)γ and is interpreted as hcp spot rather than a twin spot. Since this sample was non-magnetic, this result supports the idea that the ε is in fact an intermediate phase.

It is observed in Figs. 25b and 25c that in spite of the greater amount of strain imparted to this sample at room temperature, the ε appears to be discontinuous in comparison to the structure of a sample strained 4% at -196°C, Figs. 24b and 24d. This seems to imply that faulting at room temperature is less favorable than at -196°C which means that the stacking fault energy is somehow higher at room temperature than at -196°C. Evidence of the variation in stacking fault energy with temperature will be presented later. This apparent increase in stacking fault energy accounts for the dislocation substructures shown in Figs. 26a and 26b which were formed also in the sample strained 13% at room temperature. Rather than treat the difference between the structures in Figs. 25 and 26 as due to segregation, it is better explained by the heterogeneous deformation of grains as shown in Fig. 14. It appears that the grains represented by those in Fig. 26 received greater deformation than that in Fig. 25. The dislocation structures shown in Fig. 26 conform with
those found by Breedis. 78

4. The $M_s$ temperature and structures of as-quenched samples.

Quenching the annealed 304 stainless steel to liquid nitrogen (78°K) and helium (4°K) temperatures did not make the material magnetic. This means that the $M_s$ of this alloy is <4°K which supports what other investigators found. The structures of the samples quenched to 78°K and 4°K are similar.

No evidence of $\epsilon$ nor $\alpha'$ formation was found although at 4°K some micro-twinning was observed as shown in Fig. 27b. The twin spot, Fig. 27a, is one-third the distance between spots along the $(111)_\gamma$ direction as compared to one-sixth for the hcp $\epsilon$. Most of the areas however show only dislocations as in Fig. 28. The orientation of the foil (Fig. 28) is very close to the $[110]_\gamma$ and the dislocations tend to lie on a $(111)_\gamma$.

It is interesting to note however that at temperatures below 400°C, the stable phase of this alloy is the $\alpha$ as illustrated in Fig. 29. 79 The fact that no martensite was formed even when quenched to 4°K attests to the sluggishness of the reaction $\gamma \rightarrow \alpha$ which allows these alloys to remain austenitic when quenched from 1100°C. Thus, it is conceivable that if one quenches and holds the alloy at a temperature for some time, the reaction might proceed. Lagneborg 17 found some isothermal transformation in 304 stainless steel and the start of the transformation was found to be similar to the C-curves of the isothermal-transformation diagrams of low alloy steels.

One specimen was therefore quenched to -78°C (dry ice in acetone) and held for three days. After three days at -78°C, no detectable amount of martensite was formed. The structures reveal however the
5. **Orientation relationships between \( \gamma, \varepsilon \) and \( \alpha' \)**

The Kurdjumov-Sachs \(^{44}\) (K-S) and the Nishiyama \(^{45}\) (N) relationships are the two prominent ones reported for the orientation between \( \gamma \) and \( \alpha' \) during the martensitic transformation \( \gamma \rightarrow \alpha' \) in iron alloys. Both these relationships have the \((111)_\gamma\) converting to the \((110)_\alpha'\), the difference being in the parallelism of directions in the \((111)_\gamma\), to those in the \((110)\) as illustrated in Fig. 31. In the K-S, \([\overline{1}01]_\gamma\) is parallel to the \([\overline{1}1\overline{1}]_\alpha\) while the Nishiyama has the \([\overline{2}11]_\gamma\) parallel to the \([1\overline{1}0]_\alpha\). The relation between the two orientations is a rotation of \(5°16'\) of the bcc lattice about the \([110]_\alpha\) axis. The complete orientation relationships are best shown in stereograms (pp. 145-146 of Ref. 40) and for the particular variants shown there, these relationships are

1) **K-S**

<table>
<thead>
<tr>
<th>fcc</th>
<th>bcc</th>
</tr>
</thead>
<tbody>
<tr>
<td>((111)_\gamma)</td>
<td></td>
</tr>
<tr>
<td>((10\overline{1})_\gamma)</td>
<td></td>
</tr>
<tr>
<td>((\overline{1}2\overline{1})_\gamma)</td>
<td></td>
</tr>
</tbody>
</table>

2) **Nishiyama**

<table>
<thead>
<tr>
<th>fcc</th>
<th>bcc</th>
</tr>
</thead>
<tbody>
<tr>
<td>((0\overline{1}1)_\gamma)</td>
<td></td>
</tr>
<tr>
<td>((\overline{1}1\overline{1})_\gamma)</td>
<td></td>
</tr>
<tr>
<td>((21\overline{1})_\gamma)</td>
<td></td>
</tr>
</tbody>
</table>
The intermediate ε-phase complicates matters only in that α' must now form from it. The orientation relationship between γ and ε is simple in that close-packed planes and directions coincide, i.e.,

\[(\text{111})_\gamma || (0001)_\varepsilon \text{ and } [\text{10\overline{1}}]_\gamma || [\text{11\overline{2}0}]_\varepsilon.\]

The final orientation between the γ and α' via the ε-phase was observed by others to be the K-S relationship.\textsuperscript{15,17,18,23}

In Figs. 17c and 20c, the orientation between the γ and α' via the ε is predominantly the Nishiyama relationship. This has been observed in a number of cases and the manner in which the K-S relationship arises can be explained by Fig. 32. Figures 32 d,e,f, and g are the dark-field pictures of spots A, B, C, and D (three spots) indicated in Fig. 32c. The indexing of the diffraction pattern, Fig. 32b, with the aid of the dark-field pictures revealed, as shown in Fig. 32c, that two bcc patterns are present. One bcc pattern has the zone axis \((100)_{\alpha 1}\) indicating the N relationship while the other has the zone axis \((111)_{\alpha 2}\) corresponding to the K-S relationship. Figure 32c shows that a \((110)\) direction of the bcc crystal in the \((111)_{\alpha 2}\) orientation is rotated about 5° from another \((110)\) direction of the other bcc crystal in the \((100)_{\alpha 1}\) orientation. This agrees remarkably well with that shown in Fig. 31.

In the presence of considerable faulting, the indexing of the matrix pattern was done employing the technique of Andres, et al.\textsuperscript{40} The matrix was found to be close to the \((110)_{\gamma}\) orientation. The presence of the hcp phase was again confirmed by the double diffraction spots indicated in Fig. 32c.

Figure 32a shows two intersecting faults, I and II. As a result of the intersection, the intersected fault, I, is noticeably displaced in
the \(\langle 211\rangle \gamma\) direction (compare Figs. 32a and 32c) and at the regions of intersection, \(\alpha'\) crystals are formed. The relative amounts of \(\alpha'\) crystals in the \(\langle 111\rangle_{\alpha_2}\) (K-S) orientation and in the \(\langle 100\rangle_{\alpha_1}\) (N) orientation can be observed by comparing Figs. 32d and 32f. Figure 32d is the dark-field picture of spot A in the \(\langle 111\rangle_{\alpha_2}\) orientations while Fig. 32f is the dark-field image of spot C in the \(\langle 100\rangle_{\alpha_1}\) orientation. It is not hard to see that at the regions of intersection, a very small amount of \(\alpha_2'\) in the K-S (Fig. 32d) orientation is present compared to \(\alpha_1'\) in the N orientation (Fig. 32f). Since it is also seen from Figs. 17b and 20b that the \(\alpha'\) crystals are predominantly in the N orientation, the results of Fig. 32 imply that eventually the N orientation will change to the K-S. At the region of the intersection (indicated by the arrow in Fig. 32a), the process appears to be that fault II sheared fault I to the right, indicating dislocation motion from left to right, forming \(\alpha_1'\) in the N orientation (compare Fig. 32f). On further deformation, dislocations on fault II pile up on the \(\alpha_1'\) crystal which build up stresses able to rotate part of \(\alpha_1'\) to the K-S orientation, \(\alpha_2'\). The manner in which this rotation is accomplished will be discussed and a mechanism will be proposed.

6. The decrease of \(\varepsilon\) after reaching a maximum amount.

Another interesting point provided by Fig. 32f is that further deformation, i.e., pile-up of dislocations on \(\alpha_1'\), apparently enables the \(\alpha_1'\) crystal to grow into the region of non-faulted \(\gamma\) or into the part of fault II ahead of \(\alpha_1'\). This is suggested by the \(\alpha_1'\) crystals pointed by arrows in Fig. 32f. This observation conceivably explains the behavior of \(\varepsilon\), in that it reaches a maximum volume percent in the structure at
fairly low strains (-5%) and then decreases thereafter (see Fig. 10). The growth of $\alpha'$ into non-faulted $\gamma$ is illustrated also in Fig. 33 where it is also seen how faults are formed around bcc crystals and possibly by splitting of dislocations originating from the existing faults ($\epsilon$) as suggested by the area pointed by the arrow. It thus appears that initial deformation forms the $\epsilon$ (faulted $\gamma$) rather easily at low strains. Enough slip systems are nucleated to provide intersecting regions for the formation of $\alpha'$ crystals. After a certain amount of deformation, the $\alpha'$ is able to grow on the faults or non-faulted $\gamma$ which naturally decreases the percent of faults while the percent $\alpha'$ steadily increases with increasing deformation.
7. Other Observations on As-Deformed Samples at LN Temperature.

Except for the structures of as-quenched materials (shown in Figs. 27, 28 and 30) and the 13% tensile strained sample at R.T. (shown in Figs. 25 and 26), the rest of the electron micrographs presented thus far are those which were tensile strained 4% (Figs. 16, 19, 23, 24, and 32) and 15% (Figs. 17, 18, 20-22, and 33) at LN temperature. Other than the observations as regards the \( \epsilon \) being an intermediate phase and the nucleation and growth of \( \alpha' \), it is especially to be noted that dislocations during deformation tend to be confined to the faulted \{111\}. The perfect \( \gamma \), such as A in Fig. 16, has relatively few dislocations compared to the dark-band of \( \epsilon \) containing the \( \alpha' \), M, crystal. The dark band suggests that this is the result of overlapping of several faults on the same \{111\}, which were produced by the deformation.

With the 4% strained samples, most of the \( \alpha' \) produced is in the immediate vicinity of the region of intersection of two faults. On the other hand, with the 15% strained sample it is seen (Figs. 17, 20 and 21) that the faulted \( \gamma \) (\( \epsilon \)) between two \( \alpha' \) crystals was consumed as the \( \alpha' \) crystals grow. This accounts for the higher \( \epsilon \) in the sample with about 4% strain than the areas with larger strains.

In the as-deformed condition, the \( \alpha' \) crystals are relatively higher in contrast without having signs of tempering. In general, the \( \alpha' \) tend to have their long-direction along a single direction trace with no criss-crossing of two \( \alpha' \) crystals (Fig. 33). These observations will now be compared with the structures of aged samples.
8. Strained and Aged Samples at 200°C and 400°C.

a. ½% Tensile Deformation. It was shown from the x-ray results that the ε-phase starts to disappear at about 200°C (see Fig. 9b). The manner in which the ε reverts back to the austenite is simply the shrinking of stacking faults which is shown in Fig. 34. In this figure, the α' crystals which appear here like needles at the intersection of two faults are dark in contrast which is accounted for by tempering. The areas indicated by C point to the shrinking of faults and for edge-on faults such as that seen in Fig. 17, the dislocations are seen as dots in D. It appears also that the dislocations cross-slip into regions of non-faulted γ which, together with the fact that the faults shrunk, indicate that the stacking fault energy at 200°C must be higher than at room temperature. Further qualitative evidence of this variation in stacking fault energy is shown in Figs. 35a and 35b which came from foils of the same orientation. Figure 35a is from a sample aged at 200°C while Fig. 35b is from a sample aged at 400°C where it is easily noted that faults shrunk more upon aging at 400°C than at 200°C. A much clearer picture of the variation of stacking fault energy is provided by extended dislocations at various temperatures illustrated in Fig. 36. Gallagher showed that, of the measurable parameters for the calculation of the stacking fault energy, the radius of the inscribed circle in the extended node is the best. Thus, without going too much into details, it is seen in Fig. 36a that the inscribed radius of the sample quenched at -196°C is noticeably greater than the node at 200°C (Fig. 36b) and 400°C (Fig. 36c) which means that the stacking fault energy at -196°C is much lower than at 200°C and 400°C. It is also to be noted that the
inscribed radius at 200°C is larger than that at 400°C.

From a number of pictures, it appears that upon aging the 4% tensile deformed sample at 200°C and 400°C, the α' crystals also revert to γ. This is illustrated in Fig. 37 for a sample aged at 200°C and Fig. 38 for a sample aged at 400°C. The dissolution of α appears to occur by the generation of dislocations within α which is indicated by areas such as A in Fig. 37. In Fig. 38a, the dark α' crystals reversed contrast in the dark field picture, Fig. 38b, which seems to indicate that the α (marked I) is disappearing.

b. 15% Tensile Deformed. The structures of samples strained 15% and then aged are similar to those which were deformed 4% only in regions where the amount of α is small. This similarity is demonstrated in Figs. 39a and 39b which correspond to structures of samples aged at 200°C and 400°C respectively. Again, it is seen that the faults shrink and dislocations cross-slip into the nonfaulted γ region upon aging. However, in regions where a lot of α was present before aging, the structures of samples strained 15% differ from those which were strained 4%. A typical area where large α crystals are found is shown in Fig. 40a where S is the area where small α crystals were formed while L is the region where large α crystals A were formed. It is seen that in S the faults have shrunk while at L they appear to be unaffected, as seen better in the dark-field picture, Fig. 40b, due to the matrix spot. In the big α crystals A, some precipitation of presumably carbides is indicated by the circles. These precipitates light up with the dark-field picture (Fig. 40c) due to the bcc spot which is reasonable since carbides have d-spacings very close to bcc iron. Judging from the wavy and platelet appearance of
these carbides with the long direction parallel to (100), it can be only surmised in the absence of other separate precipitate spots in the diffraction pattern that these are \(\varepsilon\)-carbides. These characteristics are typical of \(\varepsilon\)-carbides. The \(\alpha\) crystals marked N in Fig. 40a do not light up with the crystals A but lighted up by a separate spot illustrated in Fig. 40d. This means that crystals A and N are of different orientations. As seen from Figs. 40a, 40c, and 40d, most of the \(\alpha\) crystals are in orientation A. From the results indicated in the as-deformed samples that the \(\alpha\) crystals tend to have the same orientation, crystals A must have formed during the deformation. This implies that crystals N must have formed during the aging in a different variant which was not observed in the as-deformed structures. The criss-crossing of \(\alpha\) crystals in aged samples is better illustrated by Fig. 41a with the dark-field picture of \(\alpha\) crystals in two different orientations (Fig. 41b and 41c), and dark-field picture due to the matrix spot (Fig. 41d). These two crystals (1 and 2 in Fig. 41a) were found not to be twin-related. The appearance of 1 and 2 suggests that crystal 1 must have formed first, then 2 due to the general darkening of its structure, indicating some tempering involved. The newly formed crystals such as 2 (Fig. 40b) resemble more the lenticular plates found in other systems.

It appears then that new \(\alpha\) crystals are forming at the expense of the faulted \(\gamma\) upon the aging at 200°C. In regions where this happens, aging at 400°C will consume the faulted \(\gamma\) as illustrated in Fig. 42a. The areas marked D are the \(\alpha\) crystals formed during the deformation and are seen to be heavily dislocated which can be due to precipitation of carbides. The areas D are identified as bcc by taking a diffraction
pattern from only this part. The crystals N (Figs. 42a and 42c) must have again formed during the aging because of its relatively lighter contrast than D. In Fig. 42a, the arrows point to areas where small amounts of faulted γ are seen.

9. Reversion of α to γ at 500°C.

From the magnetization measurements, aging at 500°C resulted in a large drop in the amount of α. It is reasonable to reconcile this fact with the reversion of α to γ which is supported by the pseudo-binary diagram of Fig. 29.

The reversion of α to γ was studied by looking at the structures of a 10% tensile strained sample, aged at 500°C for one hour. Figure 43a shows the bright field picture of an area where partial reversion of α to γ occurred. The α crystals are shown in the dark-field picture, Fig. 43b, while Fig. 43c is the dark-field picture of the matrix. It is seen in Fig. 43c that some fcc phases are contained within the α crystals, A. In the diffraction pattern, streaking of the α spots was observed in the (211)α direction which is parallel to the thin direction of the fcc phase within the α crystals A. This streaking suggests that the fcc material is forming on the [211]α plane.

Figure 44a shows another α crystal A which underwent partial transformation to the fcc phase, F, within A and clearly shown in Fig. 44b. The small dark flecks within A in Fig. 44a appear to be precipitated carbides. The arrows point to larger carbide particles and it appears that the nuclei of fcc phase in the α to γ transformation originated from these particles.
10. Summary of Experimental Results.

The experimental results of this investigation can be summarized as follows:

a) Magnetization measurements revealed that martensite formed in the commercial 304 stainless steel only when strain-induced. The amount of martensite increased with increasing amounts of deformation at -196°C.

b) Upon aging a sample deformed at -196°C to induce martensite, additional amounts of \( \alpha \) were formed upon aging from about 200°C to 400°C. The maximum amount of additional \( \alpha \) was formed at 400°C. This increase in \( \alpha \) was shown by magneto-, x-ray, and transmission electron metallographic techniques.

c) The results of the mechanical properties measurements indicate that the response to aging depends on the amount of deformation at -196°C which was given the material. It appears that at least 15% deformation is needed to increase further both the yield and tensile strengths after aging.

d) It was also found that an increase in percent elongation ductility was observed with the increase in strengths.

e) It was conclusively shown that the \( \epsilon \) or faulted \( \gamma \) is indeed an intermediate phase in the transformation \( \gamma \rightarrow \alpha' \). The \( \epsilon \) was shown to occur independently in an entire grain in the as-deformed samples at -196°C. Straining at R.T. and holding 304 stainless steel at -78°C also revealed the existence of \( \epsilon \) without \( \alpha \).

f) The \( \alpha' \) was preferentially nucleated at intersections of two faulted bands, grain and twin boundaries, although evidence of \( \alpha \) formation alongside the \( \epsilon \) was shown.
g) The nuclei of $\alpha'$ appear to be needle-like which grow into laths. Growth of the nuclei occurs by consumption of the faulted $\gamma$ which explains why the $\epsilon$ decreases after reaching a maximum amount at relatively low strains.

h) The orientation between $\gamma$ and $\alpha'$ via the $\epsilon$ was found to be predominantly the Nishiyama relationship which changes to the Kurdjumov-Sachs relationships on further deformation.

i) The reversion of $\epsilon$ to $\gamma$ starts about 200°C which was shown to be due apparently to the collapse of the faults. This result suggests a rise in the stacking fault energy with temperature which was demonstrated in the decrease in radius of the inscribed circle within the extended dislocation node.

j) The increase in the amount of $\alpha$ does not result from the growth of the $\alpha'$ but rather due to formation of new $\alpha'$ crystals. These newly formed $\alpha'$ crystals have lenticular appearance resembling those found in low-alloy steels.

k) At 500°C, part of the $\alpha$ reverted back to $\gamma$ which appeared to start by dissolution of carbides.
IV. DISCUSSION OF RESULTS

A. The \( \varepsilon \)-hcp Phase

The existence of the hcp phase has been shown here by electron and x-ray diffraction techniques without doubt. In electron diffraction, this was shown by the presence of the \([0\overline{1}1]_\varepsilon\) spots and the presence of the double diffraction spot (0001), while the peaks corresponding to (10\overline{1}0), (10\overline{1}1) and (10\overline{1}2) were observed by x-ray diffraction.

Dash and Otte and Breedis proposed that during the transformation of \( \gamma \) to \( \alpha' \) in these alloys the \( \varepsilon \) was formed only as a result of the formation of the \( \alpha' \). Presumably, the accommodation strain is sufficiently high to fault the \( \gamma \) adjacent to the nucleated \( \alpha' \). Two rather strong implications of this belief are that 1) with the sequence \( \gamma \rightarrow \alpha' \rightarrow \varepsilon \), one does not expect the \( \varepsilon \) to be contained within the \( \alpha' \) crystal and 2) the \( \varepsilon \) cannot exist independently of \( \alpha' \), i.e., wherever \( \varepsilon \) is formed, \( \alpha' \) must be present. Figures 17, 18, 20 and 21 show rather conclusively that the \( \varepsilon \) is contained within the \( \alpha' \) crystals while Fig. 24 shows that \( \varepsilon \) exists independently of \( \alpha' \). Although a number of investigations have been conducted on martensitic transformations in austenitic stainless steels, the results provided by Figs. 17, 18, 20, 21, and 24 have not been obtained. Thus, although the probability of faulting \( \gamma \) as soon as \( \alpha \) forms is undeniable, the sequence of events at the start of the transformation must be \( \gamma \rightarrow \varepsilon \rightarrow \alpha \). The latter sequence explains also the fact that \( \varepsilon \) will reach a maximum amount and then decrease as the strain increases.

With the sequence, \( \gamma \rightarrow \varepsilon \rightarrow \alpha \), there is one further question to be answered. If this sequence were true, one expects, as in Fe-Mn alloys.
to observe a separate Ms temperature for the transformation \( \gamma \rightarrow \epsilon \) which must be above the Ms of \( \gamma \rightarrow \alpha \). With this philosophy, Goldman et al.\(^{35} \) did not find a separate Ms employing the differential thermal analysis technique. Kaufmann\(^{82} \) calculated, however, that the free energy change during the martensitic fcc \( = \) hcp transformation is only about 50 cal/mole. Since \( \Delta F = \Delta H - T \Delta S \) and most thermal techniques measure \( \Delta H \), it is conceivable that thermal techniques are not sensitive enough to detect this small amount of heat.

The disappearance of the \( \epsilon \) upon aging in the 304 stainless steel appears to be similar to what was observed in Fe-Mn alloys.\(^{25} \) Gordon-Parr\(^{25} \) found that tempering quenched Fe-Mn powders up to 450° brought about an initial increase in \( \epsilon \), at the expense of both the bcc and fcc phases. Upon continued tempering however, the \( \epsilon \) was destroyed and the stable bcc structure was produced.

B. A Mechanism For The Transformation \( \epsilon \rightarrow \alpha' \)

From the results of transmission electron microscopy, the following were shown:

(a) The \( \epsilon \) was shown to be an intermediate phase in the transformation i.e., the sequence of events is \( \gamma \rightarrow \epsilon \rightarrow \alpha' \).

(b) Intersection of two \( \epsilon \) bundles, grain and twin boundaries are preferred sites for \( \alpha' \)-nucleation.

(c) The orientation relationship between \( \gamma \) and \( \alpha' \) changes from the Nishiyama (N) to that of Kurdjumov-Sachs (K-S).

In light of these results, a mechanism will now be proposed to show a most probable sequence of atom movements.
1. Lattice Correspondence Between \( \varepsilon \) (Faulted \( \gamma \)) and \( \alpha' \).

Since martensitic transformations are diffusionless, atom movements in going from one structure to another must be minimal. This requires the initial and final structures to have some lattice correspondence. Lattice correspondence means that 3 mutually perpendicular axes in the parent phase correspond to 3 mutually perpendicular in the product phase.

In the transformation from the fcc lattice to bcc lattice in iron alloys, the Bain lattice correspondence is always assumed.

A possible correspondence between the hcp and bcc structures is shown in Fig. 45. The normal atom positions in the hcp are indicated by the filled and open circles, being in the \((0001)_{\varepsilon}\) and \((0002)_{\varepsilon}\), respectively. When the atoms in the open-circled positions are moved the distance

\[
\begin{vmatrix}
\vec{a}_{\varepsilon} /
\sqrt{3} (1010) \\
\vec{a}_{\gamma} / 12 (511)
\end{vmatrix}
\]

to the center of the prismatic planes (positions indicated by crossed-open circles), an essentially bcc structure is obtained except for some changes in dimensions and angles. A would be bcc unit cell is outlined by the heavy-dotted lines. When transformation proceeds in the manner illustrated in Fig. 45, the Nishiyama relationship results with the correspondence of axes in the three structures being indicated.

The relative dimensions of the hcp and bcc structures are shown in Fig. 46 and 47. The lattice parameters \( \varepsilon \) of the \( \varepsilon \) are \( |\vec{c}| = 4.14\,\text{Å} \) and \( |\vec{a}| = 2.54\,\text{Å} \) with \( (c/a)_{\varepsilon} = 1.63 \). The lattice parameter of \( \alpha' \) is \( 2.86\,\text{Å} \).
and Fig. 47 shows the bcc lattice in the way it is related to the hcp as indicated in Fig. 45. Figure 46b shows the (0001)\(_\alpha\) plane which becomes the (110)\(_\alpha '\) (shown by dotted lines) when the 4.40\(\text{Å}\)-distance between atoms in the \(\langle 10\bar{1}0\rangle\)\(_\epsilon\) reduces to 4.04\(\text{Å}\) (interactomic distance in the \(\langle 110\rangle\)\(_\alpha '\)) with a simultaneous change in angles between \(\langle 11\bar{2}0\rangle\)\(_\epsilon\) (equivalent to \(\langle 110\rangle\)\(_\gamma\)) form 60° to 70°32'.

To complete the dimensional change there is a slight contraction in the c-direction from 4.14\(\text{Å}\) to 4.04\(\text{Å}\) (compare Fig. 46a and 47) while the open-circle atom (Fig. 46a) moves 0.733\(\text{Å}\) along the \(\langle 10\bar{1}0\rangle\)\(_\epsilon\) to the center of the prismatic plane. The transformation involves a volume expansion of about 1.5%.


Since the Nishiyama orientation relationship between \(\gamma\) and \(\alpha'\) was found to predominate in the early stages of transformation, the most probable atom movements are those indicated by Figs. 45 and 46a. To have the atoms move in this way, a biaxial compressive stress system, indicated in Fig. 46a, must act along the [111]\(_\gamma\) and [\(\bar{2}\)11]\(_\gamma\). (Since the \(\epsilon\) is a faulted \(\gamma\), the equivalent indices in terms of \(\gamma\) will be used hereon.) The resulting distortion imparted to the hcp is a homogeneous strain with the principal strains being -0.0819, +0.126, and -0.0242 in the three invariant mutually perpendicular directions [\(\bar{2}\)11]\(_\gamma\), [01\(\bar{1}\)]\(_\gamma\) and [111]\(_\gamma\), respectively.

The distortion of the (111)\(_\gamma\) is pure shear with the angles between the (110)\(_\gamma\) direction which become the (111)\(_\alpha\) directions changing from
Simultaneous with the homogeneous strain distortion, it is proposed that the atoms in the open circled-position moved to the cross-circled position (Fig. 46a). This can be understood if one considers that the \((c/a)_c\) is 1.63 which implies that bonding between the atoms is non-directional. One can assume then that the atoms are packed ideally as hard spheres and say that the "central-force approximation" in bonding between atoms applies where the interaction of nearest-neighbor atoms as dictated by their separation predominates. Thus, as the dimensional and angular distortions are occurring, the atoms in the \([0002]_c\) will tend to have minimum interaction energy with its nearest-neighbors by being situated symmetrically at the center of the prismatic planes. One can think of a mechanical analog by imagining the atoms to be interconnected with springs (Fig. 48). As the biaxial compressive stress is imposed, springs 4-1 and 4-5 push on atom 4 while springs 4-3, 4-7 and 4-6 are pulling atom 4. One readily sees that the new equilibrium position of atom 4 will be at 4' since beyond this position, repulsive forces due to atoms 8 and 9 will again be felt.

It is seen therefore that the homogeneous distortion conceived here converts the faulted \(\gamma(c)\) structure to the bcc structure. Assuming isotropic behaviour the stress \(\sigma_{[\overline{2}11]_\gamma}\) must be very much bigger than the \(\sigma_{[111]}\) since the strain along [\(\overline{2}11\)] is more than three times that along the [111]. Conceivably, \(\sigma_{[111]}\) might not at all be required since the contraction along the [111] can possibly be obtained through the central force approximation of bonding. This can be seen easily through the mechanical analog model (Fig. 48). Essentially, we are then concerned
mostly with the $\sigma_{[\overline{2}11]}$, although the presence of a biaxial stress system can be present at regions of intersections of two faults ($2 \in \text{bundles}$). This point will be taken up further later.

Thus the main distortion involved with this mechanism is the pure shear in the $\langle 111 \rangle_\gamma$. The ease with which the pure shear can be initiated is seen by the fact that the $\langle 211 \rangle_\gamma$ are the directions of easy faulting in $\gamma$ and the motions of Shockley partial dislocations are also in these directions. Having outlined the would-be $(110)_{\alpha'}$ in the $(111)_{\gamma'}$ (see Fig. 45), one sees that the $[\overline{2}11]_\gamma$ is also essentially the $(1\overline{1}0)_{\alpha'}$, which says that the shear system $[\overline{2}11]_{\gamma}(111)_{\gamma}$ is essentially the same as the system $(1\overline{1}0)_{\alpha'}(110)_{\alpha'}$. Since we have considered the atoms being packed as hard spheres, Zener stated that this situation offers no shear resistance along $(1\overline{1}0)_{\alpha'}(110)_{\alpha'}$.

In comparing the model presented here with prior published work, Venables conceived of a simple shear mechanism along the $(11\overline{2}0)_\epsilon(0001)_\epsilon$ (equivalent to $\langle 110 \rangle_{\gamma}(111)_{\gamma}$) to convert the hcp structure to the bcc structure. Before going further, it is necessary to distinguish the pure shear process from that of simple shear. In simple shear, the $(111)_{\gamma}$ shear plane in undistorted and unrotated with the atoms being displaced from their initial positions along the shear direction in amounts proportional to their perpendicular distance from the shear plane. It is one case of invariant plane strain which results in the misalignment of atoms in the $(111)$ unless the shear is exactly $\frac{1}{2} [\overline{2}11]_{\gamma}$.

On the other hand, the pure shear suggested here distorts the $(111)_{\gamma}$ and leaves the vertical alignment of atoms unchanged. The distortion is
homogenous whereby the three mutually perpendicular directions \([\{211\}_\gamma\), \([\{010\}_\gamma\) and \([\{111\}_\gamma\) remain mutually perpendicular after the distortion. It is seen that the simple shear mechanism by Venables cannot yield the bcc structure. Furthermore, shear along the \([\{110\}_\varepsilon\) is a much more difficult path for the atoms to move along since they are touching along this direction. Atoms tend to move easier in the \((10\overline{1}0)_\varepsilon\) which is supported by experiments of Okamoto\(^{51}\) showing that it is much easier to generate the partial dislocation in the direction \((10\overline{1}0)\) in an Al-Ag alloy by simple thermal activation with the electron beam in the electron microscope. This evidence confirms the \((\overline{2}11)_\gamma\) as the easy shear direction. Furthermore, in Venables’ model, the interatomic distance along the \([\{110\}_\varepsilon\) changed from 2.54Å to 2.48Å to conform to the \((111)_\alpha\) interatomic spacing. The process is therefore not a simple shear since points in the shear plane must remain equidistant before and after the process.

On the other hand, Zener\(^{47}\) proposed a half-twin shear in the \((\overline{2}11)_\gamma\) direction to obtain the bcc lattice from the fcc lattice. He postulated that if the bcc is mechanically stable, the fcc lattice will automatically acquire the bcc lattice. The problem here is that again in simple shear the three mutually perpendicular directions mentioned above cannot be maintained invariant. To avoid this difficult Lagneborg\(^{17}\) envisaged the splitting of a pure edge Shockley partial into half-Shockleys, e.g.,

\[
\frac{1}{6}\langle \overline{2}11\rangle = \frac{1}{12}\langle \overline{2}11\rangle + \frac{1}{12}\langle \overline{2}11\rangle.
\]

Both these mechanisms suffer however in that no mention is made about the angular change. Using a tetrahedral model of \((111)_\gamma\) planes made of "ping-pong balls" (hard spheres), Bogers and Burgers\(^{53}\) showed how the fcc lattice can be converted to the bcc
lattice. It consisted of two successive shears on two different \( \{111\}_\gamma \) planes, the shear system being of the type \( \langle \overline{2}11 \rangle \{111\}_\gamma \). By shearing the tetrahedral model of hard spheres on a \( \{111\}_\gamma \) in the amount of \( \begin{pmatrix} a \\ \overline{13} \\ \langle \overline{2}11 \rangle \end{pmatrix} \), the angles between the \( \langle 110 \rangle_\gamma \) changed from 60° to 70°32' in two of the four octahedral planes which are called "70°" planes along another \( \langle \overline{2}11 \rangle_\gamma \) will complete the transformation. The second displacement amounts to \( \begin{pmatrix} a_{bcc} \\ 2 \\ \langle 110 \rangle \end{pmatrix} \) which corresponds to one of the partial dislocations due to the splitting of the perfect dislocation \( \frac{a_{bcc}}{2} \langle 111 \rangle \) in the \( \langle 110 \rangle \) bcc planes. The two shears proposed by Rogers and Burgers involve however a displacement of the atoms perpendicular to the shear plane which arises because of the actual size of the hard spheres. Although they claim that this mechanism can predict the orientation relationship of \( \gamma \) and \( \alpha' \) by choosing the proper shear systems, no mention of these systems are made in regards to the Nishiyama and Kurdjumov-Sach relationship but only stated that the chance of obtaining these relationships is high. Furthermore, the state of the atoms being displaced perpendicular to the shear planes appears to imply that they are thermodynamically unstable. In fact, the shear \( \begin{pmatrix} a \\ \overline{13} \\ \langle \overline{2}11 \rangle \end{pmatrix} \) brings the atoms right on top of the hump between the primary and subsidiary minima in the plot of microelastic strain energy vs. free energy of the real crystal. A somewhat vague mechanism was proposed by Otte and Dash in claiming that \( \alpha' \) nucleated first before the \( \varepsilon \) phase. It was proposed that the nucleus of martensite can be formed by the motion of Shockley partials. We have seen so far that in order to get the bcc lattice, the Shockley partials must invariably split but no mention of this splitting was made.
On the basis of the above discussion, it is felt that the mechanism proposed here is very plausible. It might be mentioned here that the mechanism assumes the faulted $\gamma(\varepsilon)$ as the starting point while some of those discussed above start from the perfect $\gamma$. This point will be taken up further. What is even more encouraging is the fact that it predicts the habit plane, orientation relationship, shape deformation and possibly the internal fine structure of martensite. Before this is taken up, it is first necessary to justify the presence of the stresses proposed here and why intersection of $\varepsilon$ bundles, grain and twin boundaries are preferred sites of nucleation.

3. Compressive Stresses at Intersections of $\varepsilon$-Bundles, Grain and Twin Boundaries.

The primary stress which we are concerned with here is the $\sigma_{[\overline{2}11]}$ (Fig. 46a) since it was argued that the contraction along the $[\overline{2}11]$ is more than three times larger than that along $[111]$. It will now be shown that compressive stresses along $[\overline{2}11]_\gamma$ are indeed present at intersections of two faults which are seen to be preferred sites of $\alpha'$-formation. Figure 49 shows two intersecting $(111)_\gamma$ planes and it is assumed that faults are present in each of them. Consider the fault in the $(111)_\gamma$ plane with the leading Shockley partial, $\overrightarrow{E}_1 = \frac{\overrightarrow{a}}{6} [\overline{1}21]$. As fault B approaches A, very high local stresses are set up, either attractive or repulsive.

Attractive stresses are set up when the two leading partial dislocations attract each other to lower their total line energy. This situation arises if $\overrightarrow{b}_2$ is the leading partial in B and the following reaction ensues.

$$\frac{\overrightarrow{a}}{6} [\overline{2}11] + \frac{\overrightarrow{a}}{6} [211] = \frac{\overrightarrow{a}}{3} [011]$$
The resultant dislocation is stair-rod which joins the two faults A and B together. Being a sessile dislocation, the configuration blocks further movement of mobile dislocations in exactly the same role as grain or twin boundaries. Since further deformation will pile up mobile dislocations at these regions, and the glissile partial dislocations move in the (211) directions, the compressive stress $\sigma_{(211)}$ necessary to initiate the pure shear is obtained.

On the other hand, if repulsive stresses are set as B approaches A, the tendency is to maintain an equilibrium separation distance between the two. In the presence of applied stress (e.g., deformation), B can be made to approach A with very high repulsive stresses being set up. A situation is thus created whereby the leading partial is being repulsed by fault A while at the same time pushed to approach A by the applied stress - a compression region. Furthermore, when the stress is sufficient, fault B might be able to intersect fault A creating a step in fault A. Ashbee proposed that, at these regions of intersections, positive or negative dilatations can be produced with the formation of vacancies or interstitials, respectively. He stated that of the three partials in the intersecting fault (e.g., B), only one will produce the positive dilation while the other two produce negative dilation upon completion of the intersecting process. We know now that if two dislocations exist, each of the dislocations is displaced by an amount equal to the $|\mathbf{b}|$ of the other dislocation. Thus, if we consider now $\mathbf{b}_j = \frac{8}{6} [\overline{1}21]$ as the leading partial in B, its reaction with $\frac{8}{6} [\overline{2}11]$ (fault A) is

$$\frac{8}{6} [\overline{2}11] + \frac{8}{6} [\overline{1}21] = \frac{8}{6} [\overline{3}12]$$
which is seen to be energetically unfavorable. It might be mentioned in passing that line energies of dislocations are being considered in so far as trying to understand the qualitative nature of the stresses at intersections of faults and that these energies might not have too much significance in the phase transformation itself. Now the tendency for \( \mathbf{b}_3 \) is to intersect A and if \( |\mathbf{b}_3| \) is resolved along the three perpendicular directions \([111]_\gamma\), \([\bar{2}11]_\gamma\) and \([0\bar{1}1]_\gamma\), the qualitative nature of the stresses can be seen. The resolved components of \( |\mathbf{b}_3| \) in these directions are shown in Fig. 49 and it is seen that \( \mathbf{b}_{32}^\gamma \) opposes the direction of motion of the leading partial in A (i.e., against \([\bar{2}11]_\gamma\) ) and that \( \mathbf{b}_{31}^\gamma \) acts downwards. Thus simply from the geometry the qualitative evidence of the biaxial compressive stress system proposed in Fig. 46a is shown here.

4. Change of Orientation Relationship from Nishiyama to Kurdjumov-Sachs

The growth of the \( \alpha' \) nuclei can be quite easy because of the volume expansion on transformation. The change in volume necessarily compresses the adjacent faulted regions and thus induces them to transform or produces new faults in the surrounding austenite which provide new sites for nucleation. At the intersection of two faults which are joined by a stair-rod dislocation, the growth of \( \alpha' \) from one fault into another is easily understood.

The evidence shown by the series of pictures in Fig. 32 suggests that the Nishiyama (N) relationship between \( \gamma \) and \( \alpha' \) predominates at the start of the transformation. It is seen also that a small amount of \( \alpha' \) is in the Kurdjumov-Sachs (K-S) and since we know from Fig. 31 that the K-S differs from the N relationship by a rotation of \( 5^\circ16' \), this part of
$\alpha'$ must have been rotated $5^\circ 16'$ from the rest of the $\alpha'$. This is supported by the angular difference between $(110)_{\alpha'}$ directions in the two diffraction patterns corresponding to the N and K-S orientation (see Fig. 32c). It is thus proposed that when $\alpha'$ stopped growing, further pile-up of dislocations into it produced stresses which were sufficient to cause the necessary $5^\circ 16'$ rotation to change the orientation relationship to that of the K-S.

In the mechanism proposed here, the pure shear distortion imparted to the $(111)_\gamma$ plane to get the N relationship must therefore be rotated $5^\circ 16'$ about $[111]_\gamma$ axis to obtain the K-S relationship. Now, it is proven mathematically by Love that a combination of pure shear distortion and simple rigid body rotation is equivalent to a simple shear process. The manner in which the simple shear system is obtained is shown in Fig. 50 (compare to Fig. 1 of Ref. 48). The open circles indicate the normal positions of atoms in the $(111)_\gamma$ plane, (thus the plane of the paper is $(111)_\gamma$) with the perpendicular directions $[0\overline{1}1]_\gamma$ and $[\overline{2}11]_\gamma$ as the axes. As proposed a lateral compressive force in the $[\overline{2}11]_\gamma$ produces the pure shear distortion with contraction along this direction and expansion along the $[0\overline{1}1]_\gamma$. The positions of the atoms after this pure shear is indicated by the dotted circles and dotted parallelogram. Application of the clockwise $5^\circ 16'$ rigid body rotation about the $[111]_\gamma$ axis to the dotted parallelogram brings the atoms to their positions indicated by the triangles and results in the K-S relationship. Love proved that this two-step-movement of the atoms is equivalent to a simple shear process in the direction from the open circled positions to the triangulated positions indicated by the arrows with the shear plane containing this direction and the axis perpendicular to $[0\overline{1}1]_\gamma$ and $[\overline{2}11]_\gamma$, which is $[111]_\gamma$. 
The shear direction which is parallel to \( X \) is obtained by a counter-clockwise rotation of the orthogonal axes from their initial positions through one-half the compliment of \( 5^\circ 16' \), i.e., \( 1/2(90^\circ - 5^\circ 16') \). The simple shear system is the \( X \)-direction and the plane containing \( X \) and \([111]\). The angles between \( X \) and the \([0\overline{1}1]\) and \([\overline{2}11]\) axes are \( 47.64^\circ \) and \( 42.36^\circ \), respectively. Letting the indices of the shear direction be \([u,v,w]\) and knowing that it lies on the \((111)_\gamma\) plane, the direction is obtained by solving the following equations:

\[
\begin{align*}
-\frac{v + w}{\sqrt{u^2 + v^2 + w^2}} &= \sqrt{2} \cos 47.64^\circ \\
-\frac{2u + v + w}{\sqrt{u^2 + v^2 + w^2}} &= \sqrt{6} \cos 42.36^\circ \\
u + v + w &= 0
\end{align*}
\]

The shear direction turns out to be \([6.90, 2, 8.90]\) (\([-729]\)) with the shear plane being \((10.9, 15.80, 4.90)\) which is close to \((11, 16, 5)\). It must be noted here that both shear direction and shear plane are highly irrational. Furthermore, it is interesting to note that the shear plane is only about \( 5^\circ \) from the \((\overline{5}, 9, 2)_\gamma\), \( 12^\circ \) from the \((121)_\gamma\) and \( 13^\circ \) from the \((2, 5, \overline{2})_\gamma\). The significant point here is that the \((259)^{17}_{\gamma}\), \((225)^{15,17}_{\gamma}\) and the \((112)^{23}_{\gamma}\) are all reported as possible habit planes of the \( \alpha' \). If one appreciates the scatter of data in regards to the determination of the habit-plane, it is very tempting to call \((11, 16, 5)\) the habit plane of \( \alpha' \). However, one cannot talk of habit-planes in \( \gamma \) of \( \alpha \) when the latter is strain induced. Nevertheless, it is interesting to compare the proposed mechanism with current crystallographic theories.
of martensite crystallography which refer to formation of $\alpha$ on quenching and noting the similarities.

5. **Comparison of Proposed Mechanism with Current Crystallographic Theories of Martensitic Transformation**

The current crystallographic theories of martensite transformation are primarily based on the following experimental findings by Greninger and Troiano:

1) The habit plane of martensite is a very high index plane which is probably irrational.
2) The habit plane changes from a $(225)_{\gamma}$ for alloys with $<1.4\%C$ to a $(259)_{\gamma}$ for alloys $>1.4\%C$.
3) The average measure shear angle was $10.75^\circ \pm 2^\circ$ with accuracy in shear directions of $\pm 10^\circ$.
4) When the austenite lattice was deformed stereographically with the average experimentally determined direction and angle of shear, a triclinic lattice was obtained and in order to arrive at a bcc lattice a second macroscopically inhomogeneous shear was proposed. The second shear, though macroscopically inhomogeneous, is homogeneous within the intermediate triclinic lattice.
5) The second shear plane was most likely the $(112)_{\alpha}$, while the first shear plane (and direction) which was identified with the habit plane was an irrational plane in the austenite lattice. The high index habit plane proved untenable, the shear mechanisms proposed by Nishiyama and Kurdumov and Sachs which resulted in the $(111)_{\gamma}$ habit plane. The total shape distortion came essentially from the first simple shear with the second shear contributing none. Careful examination after partial transformation to martensite of fiducial scratches on the austenite before transformation revealed that the habit plane is unrotated and essentially undistorted. It was then...
concluded that the shape deformation is an invariant plane strain with simple shear being an example.

At this point, it is worthwhile pointing out the similarities of the above findings with the present mechanism. Both rely on the simple shear for the shape deformation with the mechanism predicting the angle of shear to be \(2 \times 5.27^\circ = 10.54^\circ\) and the habit plane close to the \((259)_\gamma\) \([3, 10, 15]_\gamma\) and \((9,22,33)_\gamma\) habit planes were also reported by Greninger and Troiano with the shear direction varying from \([243]_\gamma\) to \([2, 13, 8]_\gamma\) in an Fe-22Ni-0.8C alloy. The \((9,22,33)\) is very close to the calculated \([4.90, 10.9, 15.80]_\gamma\) irrational plane. Machlin and Cohen \(^{61}\) found also the \((9,22,33)\) habit plane in an Fe-30Ni alloy with the shear direction of \((156)_\gamma \pm 2^\circ\) lying on the \((111)_\gamma\). The latter result compares very well with the calculated \((729)_\gamma\) direction which lies also on the \((111)_\gamma\). The amount of shear of \(0.20\) found by Machlin and Cohen also compares with the amount of shear \(\gamma = 2 \times \tan 5^\circ 16^\prime\) \(^{48}\) calculated from the present mechanism.

The orientation relationship of \(\gamma\) and \(\alpha\) in the alloys investigated by Greninger and Troiano and Machlin and Cohen is very close to the K-S. Current crystallographic theories \(^{49,62,63}\) generally accept the two-shear process conceived by Greninger and Troiano. In order to treat it mathematically using matrix algebra, it was necessary to factor the first simple shear strain into a pure strain \(B\) and a rigid body rotation \(R\). Since the pure strain is always assumed to be the Bain strain, an invariant plane strain was not obtained so that an inhomogeneous shear \(S\) was introduced to insure the invariant plane strain, F. Bowles and MacKenzie \(^{49,62,63}\) made an allowance for a small uniform distortion of the habit plane by introducing the dilation parameter, \(\lambda\), while the
W-L-R theory regards the habit plane as undistorted and unrotated. Except for this minor difference, both theories are essentially the same. The input data to these theories are the knowledge of initial and final structures with their respective lattice parameters. The invariant plane strain $F$ is

$$F = \lambda RB$$

with the shape deformation being determined by RB.

It is not necessary to inquire more deeply into the mathematical treatment of these theories to see the nature of the habit plane. In the manner illustrated by Bilby and Christian this can be seen by the deformation of a unit sphere (Fig. 51). It is assumed that the sphere has three mutually perpendicular lines $X_1$, $X_2$, and $X_3$, which are unrotated during a pure strain deformation. The principal strains along these lines are $e_1$, $e_2$, and $e_3$. To insure an undistorted plane one of these must be zero and the other two must be of opposite sign or at least one must be zero. In Fig. 51 it is assumed that $e_1 = 0$ with axis $X_1$ perpendicular to the drawing and $e_2 < 0$ while $e_3 > 0$. The $X_2$-$X_3$ plane suffered a pure shear deformation with the sphere turning into an ellipsoid. In the sphere, there are two unstretched lines along $A'B'$ and $C'D'$ as well as the unstretched $X_1$ axis; these two lines represent in fact two undistorted planes. In order to have an undistorted, unrotated plane (condition for habit plane), it is necessary to impose a rigid body rotation to bring one of the planes represented by $A'B'$ or $C'D'$ to its initial position $AB$ or $CD$. A clockwise rotation will result in $AB$ being the habit plane.

The deformation of the above sphere is exactly the same as that
being proposed in the present mechanism. If \( X_2 \) is placed along \([\overline{2}11]_\gamma\) and \( X_3 \) along \([0\overline{1}1]_\gamma\), the pure shear and rotation results in a simple shear along the A-B direction with the shear plane being that containing AB and \( X_1 \) which is along the \([111]_\gamma\) (compare with Fig. 30). Since this plane was seen to be both unrotated and undistorted, it is a "habit plane."

The invariant plane strain in the present mechanism is a simple shear with the shear direction lying in the shear plane. A more general case of invariant plane strain has the shear direction not in the invariant plane. This could be a reason why the predicted "habit plane" deviated much from the \([225]_\gamma\). Nevertheless the mechanism predicts very well the \([259]_\gamma\) and the amount of shear. It might also be added that even the current theories cannot as yet satisfactorily account for the \([225]_\gamma\) habit plane. Furthermore, the small dilatation of the habit plane assumed by Bowles and MacKenzie is seen here to be the contraction along the \([111]\) by about 2.4\% which is within the realm of their proposal. There is also a question about the current assumption that the \([225]_\gamma\) is the habit plane for the exact Kurdjumov-Sachs relationship. This assumption was supposedly started by the work of Jaswon and Wheeler where the \([225]_\gamma\) was found to be one of three planes which do not undergo change in direction during transformation. It was argued then that if this plane underwent a large change in direction, the surrounding austenite must be highly distorted to accommodate this movement. The fact is however that with \([225]_\gamma\) habit planes (steels with less than 1.4\%C) the austenite grains were highly distorted to give very ill-defined back-reflection Laue spots (if any) to render the method unsuitable for orientation determination. According to Jaswon and Wheeler, this experimental evidence implies that the \([225]_\gamma\) must have undergone a large change in direction during the transformation.
and thus cannot be a habit plane according to their assumptions.

6. The Rotation and Fine Structure of Martensite

Although it was suspected for some time that internal twinning existed in martensite, Kelly and Nutting were the first to show this fine structure by using transmission electron microscopy. It is proposed that this fine structure in martensite can be accounted for by the present theory and that this must be related to the 5°16' rotation of the bcc lattice to yield the K-S relationship. Jaswon and Wheeler showed that the strain tensor calculated from the martensitic transformation has components that are all finite. It was concluded that this tensor cannot be decomposed into a symmetric tensor and a rigid-body rotation and that any decomposition is purely formal, which has no physical significance. Thus, the 5°16' rotation cannot be rigid rotation. However, the bcc lattice rotation can be achieved by spontaneous deformation of the martensite or austenite in the presence of the constraints imposed by the matrix. Spontaneous deformation can occur as slip in both martensite and austenite and also twinning in the martensite.

The deformation of the martensite can be understood with the aid of Fig. 50. After the pure shear, it is seen that the (111) plane is converted to the (110) plane. In the presence of the compressive stress along the [211]γ which is parallel to the [110]α, shear stresses are present along the [111]α and [111]α, which are two directions for slip or twinning in the bcc lattice. Slip or twinning is most favorable along the [111]α in the (112) plane which is perpendicular to the plane of drawing. With the constraints imposed by the surrounding matrix
and the shear stress along the $[111]_\alpha'$, the rotation of the lattice can be achieved.

7. Nucleation and Embryos of Martensite

It is seen that the present theory of martensite crystallography explains very well the characteristics of habit plane, orientation relationship, shape deformation and fine structure of the martensite reaction. The predictions of this theory compare very well with the experimental findings of Greninger and Troiano$^{59,60}$ and Machlin and Cohen$^{61}$ and the predictions of current phenomenological theories of martensite crystallography.$^{49,62-64}$

Some distinct differences with the current theories must be pointed out however. First, while previous theories started with the perfect fcc lattice to transform to bcc lattice, the present theory assumed the faulted fcc lattice. Secondly, current theories involved always a two-shear process while the present theory involves only an equivalent simple shear. The second difference stems essentially from the first difference, since no single shear process can convert a perfect fcc lattice to the bcc lattice. In the phenomenological theories, the Bain strain creates much more distortion than the present theory that a second shear mode is necessarily proposed in order that an invariant plane strain will be obtained.

Since the present theory is being proposed on the basis of experimental results in 304 stainless steel, the agreement of its predictions with experimental results in other alloy systems$^{59-61}$ is believed not to be fortuitous. It is proposed that nucleation and embryos of martensite occur at stacking faults in the other iron alloys.
and that transformation proceeds in the manner conceived here. This conviction stems from the fact that a unit dislocation in an fcc lattice was shown by Seeger\textsuperscript{67} to be actually split and the degree of splitting depends on the stacking fault energy of the alloy. Thus, faults exist in the fcc structure and compressive stresses are set up on quenching because of the excess vacancy concentration over the equilibrium value at the $M_s$ temperature. The evidence of the compressive stresses is shown by the fact that the resistivity of quenched materials decreases, which is ascribed to annealing of vacancies. From dislocation theory, decrease of vacancy concentration is achieved by compressive forces.

The whole idea looks even more encouraging in light of the recent study of Dash and Brown\textsuperscript{68} on the nucleation and growth of martensite in Fe-32.3% Ni alloy. Although no evidence of embryos was found, the martensite was found to nucleate preferentially at twin or grain boundaries and the evidence indicated that the fcc $\rightarrow$ bcc transformation was associated with a shear of the type \{111\}_{\gamma} \langle112\rangle_{\gamma}. At the early stages of the transformation, the shear took the form of a fault which was thought to be a precursor to the transformation or an emissary effect.

C. Correlation Between Mechanical Properties and Structures

Figures 8a and 8b demonstrate that increasing amounts of deformation at -196°C resulted in considerable increase in both yield and tensile strengths at room temperature. Thus, for 20% elongation at -196°C, the yield strength at room temperature increased by about 135,000 psi while the U.T.S. was higher by about 100,000 psi than the annealed sample. Now, increase in strength can be explained either by increase in dislocation density in the matrix which is usually referred
to as work-hardening, or by precipitation or formation of new phases which, in this case, are $\alpha$ and $\epsilon$. It was observed however that the deformation at $-196^\circ C$ is inhomogeneous in that the deformation is confined within certain bands to form $\epsilon$ and $\alpha$. Thus, dislocation density over an area in the as-deformed sample has no meaning in that one region is very faulted while the adjacent area is unfaulted. One can only conclude then that the increase in strength must be due mostly to the $\epsilon$-phase and the $\alpha$-phase formation. In the sense that strengthening is thought of as increasing resistance to dislocation motion, both the $\epsilon$ and $\alpha$ phases act as obstacles. It is seen, however, in Fig. 10 that at 20% elongation the amount of the $\epsilon$ is practically nil and therefore the dominant strengthening phase is the $\alpha$ which increased proportionately with the increase in yield and tensile strength.

The response to aging of the samples strained at $-196^\circ C$ depends on the amount of strain. It appears that in order to get further strengthening a minimum of 1% strain is needed. Otherwise, the strengths after aging were found to be generally lower than the as-deformed condition as demonstrated by the 4% and 10% strain curves of Figs. 8a and 8b.

Upon aging the strained samples, the results reveal that the following can happen, 1) conversion of most of the $\epsilon$ phase to $\gamma$ and some to $\alpha$ and 2) tempering of the strain-induced $\alpha$. The magnetization measurements after aging do not give the exact change in $\alpha$ as was done in the as-deformed sample because the carbides are themselves magnetic. However, the saturation magnetization of carbides is about two-thirds that of iron. Thus, one expects a decrease in saturation magnetization of the system if no process other than the precipitation of carbides occurs.
Such a technique was used in following the tempering process of martensites. In the event that \( \alpha \) is also formed, the \( \Delta B_s \) data in Figs. 6 and 7 include the effect of precipitation of carbides and the thermally-activated formation of \( \alpha \). The positive \( \Delta B_s \) indicates more \( \alpha \) are actually formed than that formed by using \( \Delta B_s \) directly in Eqs. (7) and (8). A negative \( \Delta B_s \) implies that the dominant process is the tempering of \( \alpha \).

In general tempering below 200°C revealed that the \( B_s \) of the sample was unchanged or slightly decreased (see Fig. 6 and Table II). It appears then that the dominant process is precipitation of carbides at 300°C and 400°C, \( \Delta B_s \) is positive except for the 4% tensile strained sample, indicating that some \( \alpha \) has formed. A negative \( \Delta B_s \) does not however necessarily preclude \( \alpha \) formation. It says only that if \( \alpha \) forms, the increase in \( \alpha \) is not sufficient to make \( \Delta B_s \) positive or zero.

The structures of 4% strained samples did not reveal any new \( \alpha \) formation after aging at 200°C and 400°C. The \( \alpha \) which generally appeared to be needle-like was darker, indicating precipitation, and the \( \epsilon \) or faults shrunk. At the same time it is seen that both yield and tensile strengths were lower than the as-formed sample (Figs. 8a and 8b). In the tempering process, \( \epsilon \) carbides are generally formed below 200°C which are coherent with the matrix. Above 200°C, the stable Fe\(_2\)C forms which is non-coherent. The coherent \( \epsilon \) results in slight increase in hardness of martensite. Although no aging at 100°C was done for the 4% strained sample, the slight increase in Y.S. at 100°C for the 10% strain sample can be attributed to this. The decrease in yield strength at 200°C indicates the amount of strengthening that the \( \epsilon \) or faults are contributing. At this temperature
(200°C), the carbides in $\alpha$ have not sufficiently overaged to decrease the strength as is known in the tempering process. It was seen however that at 200°C, the faults started collapsing.

The upward trend of both the yield and tensile strengths for the 4\% and 10\% deformation curves from 200°C to 400°C indicate some $\alpha$ formation. In general, continued tempering decreases the hardness of fresh martensite and since it is also found that the apparent stacking fault energy is higher at 400°C, the upward trend on tempering above 200°C can only be attributed to $\alpha$-formation. Although this cannot be definitely stated for the 4\% curve, the fact that $\Delta E_s$ became positive for the 10\% strain sample after aging at 300°C and 400°C (see Fig. 6) tells us that new $\alpha$ has formed.

Having discussed the behavior of the 4\% and 10\% strained samples, the increase in strengths of samples strained 15\% and above can be attributed to $\alpha$-formation. The structures reveal that at these deformations the strain-induced $\alpha$ are fairly large crystals. Upon aging, the faulted $\gamma$ between two large $\alpha$ crystals appears to be unaffected as compared to areas containing needle-like $\alpha$ (see Fig. 40). Instead it was observed that new crystals in a different orientation from the "old" crystals are formed in these faults, until most of these faults are consumed (see Fig. 42).

The yield strength for the 15\% strain sample was found to be highest after aging at 200°C. Thereafter, the yield strength drops but still is higher than the as-deformed sample until aging at 500°C. Although $\alpha$ continues to form at 300°C and 400°C (see Fig. 7), the slight decrease can
be attributed to "overaging of carbides in the old martensite crystals." The formation of ε carbides and new α crystals presumably contribute to the strength at 200°C while the over-tempering softens the "old crystals" at 300°C and 400°C.

In contrast to the amount of strain-induced α (~35%) after 15% strain at -196°C, the volume percent α after 20% strain is ~60%. At this stage, one might then consider the α as the matrix rather than the γ. Presumably the strain-induced α must be large and more α can form which probably offsets the overaging due to tempering. Aging at 500°C and above results in lower strengths and this was shown to be the reversion of α to γ.

Thus, it is seen that although precipitation of carbides in the strain-induced α can contribute to the strengthening, it appears that the formation of thermally-activated α contributes the most. At 400°C or below, Fig. 29 shows that (α + carbides) are the thermodynamically stable phase. In contrast, at 500°C a three phase field is shown which accounts for the reversion. The strain-induced α has been shown to occur martensitically because of relief-effects. It is hard to say whether the thermally-activated α formation is martensitic or not in the absence of surface-relief effects. The appearance of these new α crystals are more lenticular, however, than the strain-induced α which might lead us to suspect they are martensitic. Moreover, it is seen that these new crystals are fairly light in contrast (see Figs. 41 and 42) which suggests that no tempering has occurred, which means that they are supersaturated with carbon. In contrast, a bainitic transformation involves
precipitation of carbides similar to tempered martensite. Thus, it is very likely that these new \( \alpha \) crystals also formed martensitically. In line with current concepts, this is not impossible since it is now known that martensites can be nucleated either thermally or athermally.\(^{29}\) The rate-controlling step is the nucleating stage.

Although the ordinary reaction from \( \gamma \) to \((\alpha + C)\) at the aging temperatures used here is very sluggish,\(^{79}\) the presence of internal stress fields due to the strain-induced \( \alpha \) must have catalyzed the thermal nucleation. It is of course true that internal stress fields are relieved at higher temperatures than room temperature, but these are not relieved instantaneously. Thus it is possible to nucleate thermally new \( \alpha \) crystals very close to large strain-induced \( \alpha \) crystals, creating a criss-cross pattern to accommodate better the strains involved in the transformation.
V. SUMMARY AND CONCLUSIONS

It has been shown that $\alpha'$ formation in 304 stainless steel is rather difficult by just quenching. The martensite formation was induced by tensile deformation at -196°C where the amount of $\alpha$ increased proportionately with increase in both yield and tensile strengths at room temperature. Although the $\epsilon$-phase contributed a little, the strain induced $\alpha$-phase formation is the main cause for strengthening. It has also been shown that the thermally-nucleated $\alpha$ is the dominant factor in the further strengthening upon aging the strained samples. The $\gamma$, $\epsilon$, and $\alpha'$ phases were unambiguously identified in a partially transformed sample. The results reveal that the sequence of events in the transformation is $\gamma \rightarrow \epsilon \rightarrow \alpha'$, clearly establishing $\epsilon$ as an intermediate phase. The orientation relationship between the $\gamma$ and $\alpha'$ was found to be the Nishiyama relationship at the start which changes to the Kurdjumov-Sachs relationship. In light of this evidence, a model for the transformation of the faulted $\gamma(\epsilon)$ to $\alpha'$ was proposed which yielded characteristics similar to martensite crystallography, as regards habit plane, shear direction, amount of shear and fine structure of martensites observed.
Acknowledgements

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REFERENCES


42. A. Seeger, Z. Metallkunde 47, 653 (1956); 44, 247 (1953).


46. G. Wassermann, Archiv fuer das Eisenhuttenwesen, 16, 647 (1933).


Table I. Values of factors in calculation of $R$.

$\text{CuK}_\alpha \lambda = 1.54\AA$

Monochromator (200) LiF bent crystal

$\theta_M$ with CuK$_\alpha = 22^\circ 34' = 22.57^\circ$

<table>
<thead>
<tr>
<th>Factor</th>
<th>(101)$_e$</th>
<th>(200)$_\gamma$</th>
<th>(200)$_{\alpha'}$</th>
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<tr>
<td>$2\theta_B$</td>
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<td>50.8$^\circ$</td>
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<td>25.4$^\circ$</td>
<td>32.5$^\circ$</td>
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<tr>
<td>$\sin\theta_B$</td>
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<td>0.429</td>
<td>0.537</td>
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<tr>
<td>$\sin\theta_B/\lambda$</td>
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<td>0.278</td>
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<td>17.3</td>
<td>15.2</td>
</tr>
<tr>
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<td>15.8</td>
<td>13.90</td>
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<td>$f_{Ni}$</td>
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<tr>
<td>$f^*_{alloy}$</td>
<td>17.6</td>
<td>16.8</td>
<td>15.0</td>
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<tr>
<td>$</td>
<td>F</td>
<td>^2$</td>
<td>3X(17.6)$^2$</td>
</tr>
<tr>
<td>$P$</td>
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<td>6</td>
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<td>$LP$</td>
<td>4.27</td>
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$f^*_{alloy} = 0.18 f_{Cr} + 0.08 f_{Ni} + 0.74 f_{Fe}$

$^+$ See Reference 71.
Table II. Tabulated results of magnetic measurements.

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<th>% Tensile Strain at -196°C</th>
<th>$B_s$, saturation magnetization</th>
<th>Aging Temperature °C</th>
<th>$\Delta B_s$ gauss After Aging</th>
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<tr>
<td></td>
<td>(gauss before aging) after aging</td>
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<tr>
<td>3.5%</td>
<td>580</td>
<td>R.T.</td>
<td>-13</td>
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<tr>
<td>3.5%</td>
<td>580</td>
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<td>-13</td>
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<tr>
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<td>627</td>
<td>300°C</td>
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<tr>
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<td>200°C</td>
<td>+30</td>
</tr>
<tr>
<td>15%</td>
<td>6,980</td>
<td>300°C</td>
<td>+130</td>
</tr>
<tr>
<td>15%</td>
<td>6,810</td>
<td>400°C</td>
<td>+270</td>
</tr>
<tr>
<td>15%</td>
<td>5,010</td>
<td>500°C</td>
<td>-1660</td>
</tr>
<tr>
<td>19.75%</td>
<td>8,490</td>
<td>R.T.</td>
<td>-13</td>
</tr>
<tr>
<td>19.5%</td>
<td>7,800</td>
<td>100°C</td>
<td>-30</td>
</tr>
<tr>
<td>20%</td>
<td>8,340</td>
<td>200°C</td>
<td>0</td>
</tr>
<tr>
<td>19.5%</td>
<td>8,390</td>
<td>300°C</td>
<td>+170</td>
</tr>
<tr>
<td>20.25%</td>
<td>8,820</td>
<td>400°C</td>
<td>+460</td>
</tr>
<tr>
<td>20%</td>
<td>8,740</td>
<td>500°C</td>
<td>-2340</td>
</tr>
<tr>
<td>25.25%</td>
<td>10,300</td>
<td>R.T.</td>
<td>-13</td>
</tr>
<tr>
<td>24%</td>
<td>10,250</td>
<td>400°C</td>
<td>+430</td>
</tr>
</tbody>
</table>
Table III. Tabulated room temperature mechanical properties.

<table>
<thead>
<tr>
<th>% Tensile Strain at -196°C</th>
<th>Aging Temp. °C</th>
<th>Yield Strength (0.1% offset), psi</th>
<th>Tensile Strength psi</th>
<th>% Elongation in 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.5%</td>
<td>R.T.</td>
<td>75,800</td>
<td>131,000</td>
<td>42.0</td>
</tr>
<tr>
<td>3.5%</td>
<td>200°C</td>
<td>63,400</td>
<td>116,000</td>
<td>43.0</td>
</tr>
<tr>
<td>3.5%</td>
<td>300°C</td>
<td>67,900</td>
<td>116,700</td>
<td>43.5</td>
</tr>
<tr>
<td>4.5%</td>
<td>400°C</td>
<td>71,500</td>
<td>119,800</td>
<td>39.0</td>
</tr>
<tr>
<td>4.5%</td>
<td>500°C</td>
<td>66,200</td>
<td>115,500</td>
<td>42.0</td>
</tr>
<tr>
<td>10%</td>
<td>R.T.</td>
<td>95,000</td>
<td>156,500</td>
<td>7.5†</td>
</tr>
<tr>
<td>10%</td>
<td>100°C</td>
<td>103,500</td>
<td>153,500</td>
<td>9.0†</td>
</tr>
<tr>
<td>9.5%</td>
<td>200°C</td>
<td>82,600</td>
<td>130,000</td>
<td>5.0†</td>
</tr>
<tr>
<td>8.5%</td>
<td>300°C</td>
<td>96,800</td>
<td>136,700</td>
<td>10.5†</td>
</tr>
<tr>
<td>9.5%</td>
<td>400°C</td>
<td>98,200</td>
<td>136,700</td>
<td>5.0†</td>
</tr>
<tr>
<td>10%</td>
<td>500°C</td>
<td>93,200</td>
<td>134,000</td>
<td>14.5†</td>
</tr>
<tr>
<td>16%</td>
<td>R.T.</td>
<td>141,000</td>
<td>184,000</td>
<td>15.5</td>
</tr>
<tr>
<td>15%</td>
<td>100°C</td>
<td>147,200</td>
<td>186,000</td>
<td>15.2</td>
</tr>
<tr>
<td>15%</td>
<td>200°C</td>
<td>165,000</td>
<td>183,500</td>
<td>17.5</td>
</tr>
<tr>
<td>15%</td>
<td>300°C</td>
<td>161,000</td>
<td>182,000</td>
<td>17.0</td>
</tr>
<tr>
<td>15%</td>
<td>400°C</td>
<td>154,000</td>
<td>188,500</td>
<td>18.0</td>
</tr>
<tr>
<td>15%</td>
<td>500°C</td>
<td>128,000</td>
<td>154,500</td>
<td>25.5</td>
</tr>
<tr>
<td>19.75%</td>
<td>R.T.</td>
<td>177,000</td>
<td>195,000</td>
<td>4.5†</td>
</tr>
<tr>
<td>19.5%</td>
<td>100°C</td>
<td>176,000</td>
<td>182,000</td>
<td>2.0†</td>
</tr>
<tr>
<td>20.0%</td>
<td>200°C</td>
<td>197,000</td>
<td>198,000</td>
<td>11.8</td>
</tr>
<tr>
<td>19.5%</td>
<td>300°C</td>
<td>199,000</td>
<td>201,000</td>
<td>11.0</td>
</tr>
<tr>
<td>20.25%</td>
<td>400°C</td>
<td>217,000</td>
<td>219,000</td>
<td>2.0†</td>
</tr>
<tr>
<td>20%</td>
<td>500°C</td>
<td>155,000</td>
<td>169,500</td>
<td>18.5†</td>
</tr>
<tr>
<td>25.25%</td>
<td>R.T.</td>
<td>215,000</td>
<td>*</td>
<td>0†</td>
</tr>
<tr>
<td>24.0%</td>
<td>400°C</td>
<td>244,000</td>
<td>*</td>
<td>0.5%†</td>
</tr>
</tbody>
</table>

† Specimen broke outside of gage marks.
*Catastrophic failure.
Table IV. Effect of further thermomechanical treatment.

(Deformation by rolling)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Thermomechanical Treatment</th>
<th>$B_s$, (gauss)</th>
<th>Yield Strength (psi)</th>
<th>Tensile Strength (psi)</th>
<th>%Elongation in 2&quot;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Saturation Magnetization ($% a'$)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A1</td>
<td>Rolled, 20% reduction in thickness at LN</td>
<td>9.920 (63%)</td>
<td>187,000</td>
<td>199,000</td>
<td>9%</td>
</tr>
<tr>
<td>A21</td>
<td>Same as A1, then aged at 400°C for 2 hours.</td>
<td>10,550 (67%)</td>
<td>216,000</td>
<td>*</td>
<td>5%</td>
</tr>
<tr>
<td>A22</td>
<td>Same as A1, then rolled at 400°C, 9% reduction in thickness</td>
<td>10,790 (68.5%)</td>
<td>231,000</td>
<td>234,000</td>
<td>1%</td>
</tr>
<tr>
<td>A31</td>
<td>Same as A21, then rolled at R.T. 9% reduction in thickness</td>
<td>11,090 (70.5%)</td>
<td>219,000</td>
<td>225,000</td>
<td>5%</td>
</tr>
<tr>
<td>A32</td>
<td>Same as A31, then aged at 400°C for 2 hours.</td>
<td>11,100 (70.6%)</td>
<td>242,000</td>
<td>*</td>
<td>1%</td>
</tr>
<tr>
<td>A41</td>
<td>Same as A21 rolled at LN, 9% reduction in thickness</td>
<td>10,940 (69.7%)</td>
<td>216,000</td>
<td>224,000</td>
<td>5%</td>
</tr>
<tr>
<td>A42</td>
<td>Same as A41, then aged at 400°C for 2 hours.</td>
<td>11,600 (73.7%)</td>
<td>255,000</td>
<td>*</td>
<td>1%</td>
</tr>
</tbody>
</table>

* Type III(75) load-elongation curve - catastrophic failure.
FIGURE CAPTIONS

Fig. 1  Size of tensile sheet specimen.
Fig. 2  Schematic drawing of set-up for tensile deformation at -196°C.
Fig. 3  Schematic drawing of normal and intrinsic magnetic induction hysteresis loops, which should be symmetrical about the origin.
Fig. 4  Schematic drawing of set-up for obtaining intrinsic induction hysteresis loop.
Fig. 5  Saturation magnetization at R.T. of tensile deformed samples as a function of tensile strain at -196°C.
Fig. 6  Change in saturation magnetization, $\Delta B_s$ at R.T., after aging tensile-deformed (at -196°C) samples, versus aging temperature.
Fig. 7  Change in saturation magnetization, $\Delta B_s$ at R.T. after aging at 400°C and 300°C versus amount of strain at -196°C.
Fig. 8  Mechanical properties at R.T. versus aging temperature for different tensile strains at -196°C.
   a) yield strength (0.1% offset)
   b) ultimate tensile strength (U.T.S.)
   c) percent elongation in 2" gage
Fig. 9  X-ray diffractometer traces at R.T. of a 10% tensile deformed sample at -196°C.
   a) before aging
   b) after aging at 200°C for 1-1/2 hours
   c) after aging at 400°C for 1-1/2 hours
Fig. 10 Volume fractions of $\gamma$, $\epsilon$, and $\alpha'$ phases of as-deformed samples as a function of tensile strain at -196°C.
Fig. 11 X-ray diffractometer trace at R.T. of a sample rolled at -196°C with reduction in thickness of 10% showing presence of a phase.

Fig. 12 X-ray diffractometer trace of (200)\textsubscript{\alpha}' peak of a 20% tensile deformed sample at -196°C before and after aging at 400°C showing increase of \alpha' after aging.

Fig. 13 Optical micrograph of annealed 304 stainless steel. Average grain size is about 3 mm. Electropolished and then electro-etched with saturated oxalic acid.

Fig. 14 Optical micrograph of a 2% tensile strained sample showing heterogenous deformation of grains.

Fig. 15 Optical micrographs of a 20% tensile strained sample.
   a) before aging
   b) after aging at 400°C for 1-1/2 hours

Fig. 16 Typical microstructure of partially transformed metastable 18-8 austenitic steels, showing the bcc phase (M) within the dark band of \epsilon and untransformed austenite (A). (Sample was 4% tensile strained at -196°C.)

Fig. 17 a) Electron diffraction pattern
   b) bright-field image of area used for identification of phases
   c) sketch of diffraction pattern.
   Sample was 15% tensile deformed at -196°C.

Fig. 18 a), b), c), d), e), f), are dark field pictures due to spots. A, B, C, D, E, and F, respectively, indicated in Fig. 17c.
Fig. 19  Enlargement of area indicated by bigger arrows in Fig. 17b showing nucleation of \( \alpha' \) at intersections of two faults.

Fig. 20  a) Electron diffraction pattern and b) bright field image of area showing nucleation and growth of \( \alpha' \), c) sketch of diffraction pattern (a); d), e), and f) are dark field pictures due to spots A, B, and C, respectively indicated in c).

Sample was 15\% tensile strained at -196\°C.

Fig. 21  Composite electron micrograph showing nucleation of \( \alpha' \) near twin boundaries (indicated by \( N \)), growth of \( \alpha' \) at \( G \). Sample 15\% strained at -196\°C (see text for fuller description.)

Fig. 22  Electron micrograph showing formation of \( \alpha' \) at grain boundaries 15\% strained at -196\°C.

Fig. 23  Formation of \( \alpha' \) alongside the faulted \( \gamma(\varepsilon) \), 4\% strained at -196\°C.

Fig. 24  a) Electron diffraction pattern

b) bright field image of area showing the \( \varepsilon \) to exist independently of \( \alpha' \) across entire grain.

c) and d) dark field images due to \( \gamma \) and \( \varepsilon \) spots respectively. 4\% strained at -196\°C.

Fig. 25  a) Electron diffraction pattern

b) bright field image of area from sample strained 13\% at R.T. showing presence of \( \varepsilon \).

c) and d) are dark field images due to \( \varepsilon \) and \( \gamma \) spots, respectively.

Fig. 26  Other dislocation substructures of sample strained 13\% at R.T. showing a) alignment of dislocations along [211]_\( \gamma \) and b) formation of cell structure.
Fig. 27  Micro-twinning in sample quenched to 4°K.
Fig. 28  Typical microstructures of as-quenched samples to 78°K and 4°K.
Fig. 29  Pseudo-binary equilibrium diagram of 18-8 Cr-Ni alloy with carbon (after Kinsel and Franks 79).
Fig. 30  Electron micrograph showing formation of ε in isothermally held sample for 3 days at -78°C (dry ice temperature).
Fig. 31  Relationship between the Kurdjumov-Sachs and Nishiyama orientation relationships between γ and α' during martensitic transformations in iron alloys.
Fig. 32  a) Bright field image
          b) electron diffraction pattern of region indicated by arrow at the intersection of faults I and II.
          c) sketch of diffraction pattern (a)
          d, e, f, g) dark field images of spots due to A, B, C, and D shown in (c) (see text for full description), 4% strained at -196°C.
Fig. 33  Electron micrograph showing growth of α' into non-faulted γ and alignment of long-direction of α' laths along one direction. 15% strained sample at -196°C.
Fig. 34  Electron micrograph showing shrinking of faults, 4% strained at -196°C and aged at 200°C for 1-1/2 hours.
Fig. 35  Structures of 4% strained sample at -196°C after aging at a) 200°C and b) 400°C showing qualitatively the increase of stacking fault energy with temperature.
Fig. 36  Extended dislocation nodes found from samples
a) quenched to -196°C
b) aged at 200°C
c) aged at 400°C

Fig. 37 Structure of 4% strained sample at -196°C and aged at 200°C showing apparent dissolution of α' at A.

Fig. 38 Structure of 4% strained sample at -196°C and aged at 400°C showing dissolution of α' indicated by I in both (a) bright field and (b) dark field image.

Fig. 39 Structure of 15% strained sample at -196°C and aged at (a) 200°C and (b) 400°C showing collapse of faults in areas with small amounts of α'.

Fig. 40 Structure of 15% strained sample and aged at 200°C showing collapse of faults in S and formation of new α' crystals N in areas like L containing large α' crystals A. Circles indicate carbides.

a) bright field image
b) dark field image of matrix spot
c) dark field image of strain-induced α'
d) dark field image with thermally nucleated α', N, being lighted.

Fig. 41 Structure of 15% strained sample and aged at 200°C showing criss-crossing of α' crystals in (a) bright field image. The thermally nucleated α' is lighted in dark field image (b) while the strain-induced α' is lighted up in (c). (d) is the dark field of matrix spot.
Fig. 42 Structure of 15% strained sample aged at 400°C showing loss of faulted γ through thermal nucleation of α crystals N.
   a) bright field image
   b) dark field image of strain-induced α'
   c) dark field image of thermally nucleated α'
   d) dark field image of matrix

Fig. 43 Structure of 10% strained sample at -196°C and aged at 500°C for 1-1/2 hours showing partial reversion of α' crystals A.
   a) bright field picture
   b) dark field picture of α' crystals A
   c) dark field picture of matrix

Fig. 44 Structure of 10% strained sample at -196°C and aged at 500°C for 1-1/2 hours with partial reversion of α' crystal A into γ indicated by F apparently originated at carbide particles indicated by arrows.

Fig. 45 Lattice correspondence of HCP structure (faulted γ) and BCC structure.

Fig. 46 Dimensions in the HCP structure.

Fig. 47 Dimensions in the BCC structure.

Fig. 48 Mechanical analog of proposed mechanism.

Fig. 49 Intersection of two faults.

Fig. 50 Combination of pure shear and rigid rotation is simple shear.

Fig. 51 Traces of the undistorted planes in their initial positions AB, CD, and their final positions A'B', C'D'/ (After Bilby and Christian.57)
0.030" THICK SHEET

Fig. 1
Fig. 2

LIQUID NITROGEN

STYROFOAM LINED WITH VACUUM GREASE

TENSILE SPECIMEN

TENSILE GRIPS

2" GAGE 0.030"

XBL 687-1280
Fig. 3
Fig. 5

Bs, SATURATION MAGNETIZATION, GAUSS

% TENSILE STRAIN
(STRAINED AT LIQUID NITROGEN TEMPERATURE)
Fig. 6

$\Delta B_s$, CHANGE IN SATURATION MAGNETIZATION, GAUSS

AGING TEMPERATURE, °C
(AGING TIME, 1 1/2 HOURS)
ΔB_s, CHANGE IN SATURATION MAGNETIZATION AFTER AGING, IN GAUSS

% TENSILE STRENGTH

T_A = 400°C

T_A = 300°C

Fig. 7
Fig. 8a
Fig. 8b
Cu Kαλ = 1.54 Å
(200) LiF DIFFRACTED BEAM MONOCHROMATOR

a. AS DEFORMED
b. AFTER 200°C AGING
c. AFTER 400°C AGING

(200)γ  (10.1)ε  (110)α(111)γ  (100)ε

Fig. 9
Fig. 10
Fig. 11
Fig. 13

Fig. 14

XBB 687-4084
Fig. 15
Fig. 16
Fig. 17
Fig. 18
Fig. 20
Fig. 25
Fig. 30

(a)  

(b)  XBB 687-4097
Fig. 31
Fig. 32
Fig. 32 (continued)
Fig. 34
(a) XBB 687-4091

(b) 0.5 µ

Fig. 35
Fig. 40
Fig. 41
Fig. 42
WOULD BE BCC
● ATOMS IN (0001)
○ ATOMS IN (0002)
--- OUTLINES OF THE (110)α'

\[ \frac{c}{a} = 1.63 \]

Fig. 45
Fig. 46

(a)

VOLUME/2 ATOMS = 23.10 Å³

0.723 Å

C = 4.14 Å

(b)

θ = 75.4 Å

= 6.410 Å

= 4.211 Å

= 1.211 Å

= 0.680 Å
VOLUME / 2 ATOMS = 23.45 Å³
\[ \vec{b}_1 = \frac{\vec{a}}{6} [\overline{2} 1 1] \]
\[ \vec{b}_2 = \frac{\vec{a}}{6} [\overline{2} 1 1] \]
\[ \vec{b}_3 = \frac{\vec{a}}{6} [\overline{2} 1] \]

\[
\begin{align*}
|\vec{b}_{31}| &= 0.692 \ \text{Å} \\
|\vec{b}_{32}| &= 0.245 \ \text{Å} \\
|\vec{b}_{33}| &= 1.270 \ \text{Å}
\end{align*}
\]

Fig. 49

XBL 687-1294
Fig. 50
Fig. 51
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