STRENGTHENING OF MARTENSITE BY PRIOR DEFORMATION OF
THE AUSTENITE IN A LOW-CARBON IRON-NICKEL ALLOY

By

JAK TARANTO

Submitted in Partial Fulfillment of the
Requirements for the Degree of
DOCTOR OF SCIENCE
at the
Massachusetts Institute of Technology
1963

Signature of the Author
Department of Metallurgy
January 1963

Signature of Professor
in Charge of Research

Signature of Chairman
Department Committee
on Graduate Students
STRENGTHENING OF MARTENSITE BY PRIOR DEFORMATION OF

THE AUSTENITE IN A LOW-CARBON IRON-NICKEL ALLOY

Jak Teranto

Submitted to the Department of Metallurgy on January 7, 1963 in partial fulfillment of the requirements for the degree of Doctor of Science.

ABSTRACT

The effect of prior deformation of austenite by multipass rolling at 300°C on the strength of martensite in a low-carbon iron-31.9 percent nickel alloy has been investigated. The yield strength of such ausformed martensite increases by about 300 pounds per square inch for each percent of prior reduction in thickness of the austenite. Ausformed martensite with 88 percent prior reduction in thickness of the austenite is about 35 percent stronger than regular martensite. Cold-working of martensite is more effective in increasing its strength than is ausforming. However, the ductility of ausformed martensite remains the same as that of regular martensite, whereas the ductilities of cold-worked austenite and of cold-worked martensite decrease with deformation. The observed anisotropy in the tensile properties of the rolled sheet is attributed to the influence of the rolling texture on the resolved shear stress and work-hardening characteristics.

Optical and transmission electron microscopy indicate that the lattice defects present in the deformed austenite are inherited by the subsequently produced martensite, and that the resulting ausformed martensite does not, in general, have fine internal twins like those found in regular martensite. It is concluded that the strength of low-carbon ausformed martensite is increased by the presence of inherited lattice defects and by a reduction in the effective distance between barriers to slip. The large microstrains associated with the ausformed martensite are shown to be a result of, rather than the cause of, the strengthening.

Thesis Supervisor: Morris Cohen

Title: Professor of Metallurgy
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section Number</th>
<th>Page Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abstract</td>
<td>1</td>
</tr>
<tr>
<td>List of Figures</td>
<td>iv</td>
</tr>
<tr>
<td>List of Tables</td>
<td>ix</td>
</tr>
<tr>
<td>Acknowledgments</td>
<td>x</td>
</tr>
<tr>
<td>I Introduction</td>
<td>1</td>
</tr>
<tr>
<td>II Literature Survey</td>
<td>2</td>
</tr>
<tr>
<td>III Selection of Materials and Experimental Procedure</td>
<td>17</td>
</tr>
<tr>
<td>A. Selection of Materials</td>
<td>17</td>
</tr>
<tr>
<td>B. Heat Treatment</td>
<td>20</td>
</tr>
<tr>
<td>C. Deformation of Austenitic and Martensitic Specimens</td>
<td>23</td>
</tr>
<tr>
<td>D. Determination of $M_s$ Temperatures</td>
<td>24</td>
</tr>
<tr>
<td>E. Metallography</td>
<td>26</td>
</tr>
<tr>
<td>F. Mechanical Properties</td>
<td>28</td>
</tr>
<tr>
<td>a. Hardness Measurements</td>
<td>28</td>
</tr>
<tr>
<td>b. Tensile Tests</td>
<td>29</td>
</tr>
<tr>
<td>c. Determination of Amount of Retained Austenite</td>
<td>29</td>
</tr>
<tr>
<td>G. Transmission Electron Microscopy and Preparation of Thin Foils</td>
<td>32</td>
</tr>
<tr>
<td>H. X-ray Diffraction</td>
<td>36</td>
</tr>
<tr>
<td>IV Results</td>
<td>42</td>
</tr>
<tr>
<td>A. Effect of Deformation on $M_s$ Temperature and Amount of Martensite Produced</td>
<td>42</td>
</tr>
<tr>
<td>Section Number</td>
<td>Page Number</td>
</tr>
<tr>
<td>----------------</td>
<td>-------------</td>
</tr>
<tr>
<td>B. Mechanical Properties</td>
<td>45</td>
</tr>
<tr>
<td>C. Metallography</td>
<td>61</td>
</tr>
<tr>
<td>D. X-ray Measurements</td>
<td>74</td>
</tr>
<tr>
<td>E. Electron Microscopy</td>
<td>79</td>
</tr>
<tr>
<td>V Discussion of Results</td>
<td>89</td>
</tr>
<tr>
<td>A. Anisotropy of Tensile Properties</td>
<td>89</td>
</tr>
<tr>
<td>B. Microstructural Aspects</td>
<td>92</td>
</tr>
<tr>
<td>C. Results of X-ray Line-Broadening Measurements</td>
<td>100</td>
</tr>
<tr>
<td>D. General Discussion</td>
<td>103</td>
</tr>
<tr>
<td>Conclusions</td>
<td>110</td>
</tr>
<tr>
<td>Suggestions for Further Work</td>
<td>112</td>
</tr>
<tr>
<td>Biographical Note</td>
<td>114</td>
</tr>
<tr>
<td>Bibliography</td>
<td>115</td>
</tr>
</tbody>
</table>
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure Number</th>
<th>Description</th>
<th>Page Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Alloy number 3 after 24 hours at 1000° C and mechanical polishing. Etchant: 6 percent nital. Magnification: 100X</td>
<td>19</td>
</tr>
<tr>
<td>2</td>
<td>Alloy number 3 after 24 hours at 1000° C. Electrolytic polishing and etching. Magnification: 100X</td>
<td>19</td>
</tr>
<tr>
<td>3</td>
<td>Alloy number 4 in as-received condition. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 100X</td>
<td>21</td>
</tr>
<tr>
<td>4</td>
<td>Alloy number 4 after 24 hours at 1000° C. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 100X</td>
<td>21</td>
</tr>
<tr>
<td>5</td>
<td>Tensile specimen. Dimensions in millimeters</td>
<td>30</td>
</tr>
<tr>
<td>6</td>
<td>Thin foil specimen prepared for first polishing operation</td>
<td>35</td>
</tr>
<tr>
<td>7</td>
<td>Thin foil specimen after second polishing operation</td>
<td>35</td>
</tr>
<tr>
<td>8</td>
<td>Normalized curves showing resistance in arbitrary units versus temperature for 0 percent and 88 percent reduction in thickness of the austenite by rolling</td>
<td>43</td>
</tr>
<tr>
<td>9</td>
<td>Effect of amount of prior reduction of the austenite on the amount of martensite formed by quenching in liquid nitrogen</td>
<td>44</td>
</tr>
<tr>
<td>10</td>
<td>Microhardness of deformed austenite, martensite with prior deformation of the austenite, and deformed martensite as a function of reduction in thickness by rolling.</td>
<td>46</td>
</tr>
<tr>
<td>11</td>
<td>Stress-strain curve for austenite with 0 percent and 88 percent reduction in thickness</td>
<td>47</td>
</tr>
<tr>
<td>12</td>
<td>Stress-strain curves for austenitic and martensitic specimens with no deformation</td>
<td>48</td>
</tr>
<tr>
<td>Figure Number</td>
<td>Description</td>
<td>Page Number</td>
</tr>
<tr>
<td>---------------</td>
<td>-----------------------------------------------------------------------------</td>
<td>-------------</td>
</tr>
<tr>
<td>13</td>
<td>Stress-strain curves for martensite with 0 percent and 88 percent reduction in thickness of the austenite...</td>
<td>49</td>
</tr>
<tr>
<td>14</td>
<td>Effect of volume percent of martensite on ultimate tensile strength and 0.2 percent yield strength (no prior deformation of the austenite)</td>
<td>51</td>
</tr>
<tr>
<td>15</td>
<td>Ultimate tensile strength and 0.2 percent yield strength for longitudinal and transverse specimens of austenite as a function of reduction in thickness by rolling</td>
<td>52</td>
</tr>
<tr>
<td>16</td>
<td>Ultimate tensile strength and 0.2 percent yield strength for longitudinal and transverse martensitic specimens as a function of prior deformation of the austenite by rolling</td>
<td>53</td>
</tr>
<tr>
<td>17</td>
<td>Ultimate tensile strength and 0.2 percent yield strength for longitudinal and transverse martensitic specimens extrapolated linearly to 100 percent martensite as a function of prior deformation of the austenite by rolling</td>
<td>54</td>
</tr>
<tr>
<td>18</td>
<td>Ultimate tensile strength and 0.2 percent yield strength of martensite as a function of deformation by rolling</td>
<td>56</td>
</tr>
<tr>
<td>19</td>
<td>Ductility of austenite as a function of deformation by rolling</td>
<td>57</td>
</tr>
<tr>
<td>20</td>
<td>Ductility of martensite as a function of deformation by rolling</td>
<td>59</td>
</tr>
<tr>
<td>21</td>
<td>Dependence of 0.2 percent yield strength and ultimate tensile strength of austenitic and martensitic (extrapolated to 100 percent martensite) specimens on austenitic grain size</td>
<td>60</td>
</tr>
<tr>
<td>22</td>
<td>Ductility of austenite and martensite as a function of austenitic grain size</td>
<td>62</td>
</tr>
<tr>
<td>23</td>
<td>Transverse section of austenite with 76 percent reduction in thickness. Electrolytic polishing and etching. Magnification: 200X</td>
<td>63</td>
</tr>
<tr>
<td>Figure Number</td>
<td>Description</td>
<td>Magnification</td>
</tr>
<tr>
<td>---------------</td>
<td>-------------</td>
<td>---------------</td>
</tr>
<tr>
<td>24</td>
<td>Rolling-plane section of austenite with 76 percent reduction in thickness. Electrolytic polishing and etching. Magnification: 200X</td>
<td>63</td>
</tr>
<tr>
<td>25</td>
<td>Longitudinal section of austenite with 76 percent reduction in thickness. Electrolytic polishing and etching. Magnification: 200X</td>
<td>64</td>
</tr>
<tr>
<td>30</td>
<td>Longitudinal section of specimen with 76 percent reduction in thickness, quenched to -47° C. Electrolytic polishing and 6 percent nital etch before quench, light etch after quench. Magnification: 500X</td>
<td>67</td>
</tr>
<tr>
<td>31</td>
<td>Longitudinal section of specimen with 76 percent reduction in thickness, quenched to -47° C. Electrolytic polishing and 6 percent nital etch before quench, light etch after quench. Magnification: 500X</td>
<td>68</td>
</tr>
<tr>
<td>32</td>
<td>Longitudinal section of specimen with 34 percent reduction in thickness, quenched to -47° C. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 500X</td>
<td>68</td>
</tr>
<tr>
<td>Figure Number</td>
<td>Description</td>
<td>Page Number</td>
</tr>
<tr>
<td>---------------</td>
<td>-----------------------------------------------------------------------------------------------------------------------------------------------------</td>
<td>-------------</td>
</tr>
<tr>
<td>33</td>
<td>Martensite produced by quenching austenite with no deformation in liquid nitrogen. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.</td>
<td>70</td>
</tr>
<tr>
<td>34</td>
<td>Martensite produced by quenching austenite with no deformation in liquid nitrogen. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.</td>
<td>70</td>
</tr>
<tr>
<td>35</td>
<td>Martensite produced by quenching austenite with no deformation in liquid nitrogen. Annealed for 104 hours at 300° C. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.</td>
<td>72</td>
</tr>
<tr>
<td>36</td>
<td>Martensite produced by quenching austenite with no deformation in liquid nitrogen. Rolled to 32 percent reduction in thickness at room temperature. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.</td>
<td>72</td>
</tr>
<tr>
<td>37</td>
<td>Martensite produced by quenching austenite with no deformation in liquid nitrogen. Rolled to 32 percent reduction in thickness at room temperature. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.</td>
<td>73</td>
</tr>
<tr>
<td>38</td>
<td>Separation of particle size and strain for deformed martensite with 56 percent reduction in thickness. Dotted lines show magnitude of possible experimental scatter.</td>
<td>75</td>
</tr>
<tr>
<td>39</td>
<td>Average microstrains in deformed austenite, martensite with prior deformation of the austenite, and deformed martensite.</td>
<td>78</td>
</tr>
<tr>
<td>40</td>
<td>Effect of annealing at 300° C on the microstrains and hardness of martensite with 0 percent and 88 percent prior reduction of the austenite.</td>
<td>80</td>
</tr>
<tr>
<td>41</td>
<td>Electron transmission micrograph of martensite with no prior deformation of the austenite. Magnification: 80,000X.</td>
<td>81</td>
</tr>
<tr>
<td>Figure Number</td>
<td>Description</td>
<td>Page Number</td>
</tr>
<tr>
<td>---------------</td>
<td>-----------------------------------------------------------------------------------------------------</td>
<td>-------------</td>
</tr>
<tr>
<td>42</td>
<td>Electron transmission micrograph of martensite with no prior deformation of the austenite. Magnification: 30,000X</td>
<td>81</td>
</tr>
<tr>
<td>43</td>
<td>Electron transmission micrograph of austenite with 34 percent reduction in thickness. Magnification: 40,000X</td>
<td>83</td>
</tr>
<tr>
<td>44</td>
<td>Diffraction pattern of Fig. 43</td>
<td>83</td>
</tr>
<tr>
<td>45</td>
<td>Electron transmission micrograph of austenite with 34 percent reduction in thickness. Magnification: 40,000X</td>
<td>84</td>
</tr>
<tr>
<td>46</td>
<td>Diffraction pattern of Fig. 45</td>
<td>84</td>
</tr>
<tr>
<td>47</td>
<td>Electron transmission micrograph of martensite with 34 percent prior reduction in thickness of the austenite. Magnification: 40,000X</td>
<td>85</td>
</tr>
<tr>
<td>48</td>
<td>Diffraction pattern of area enclosed in circle in Fig. 47</td>
<td>85</td>
</tr>
<tr>
<td>49</td>
<td>Electron transmission micrograph of martensite with 34 percent prior reduction in thickness. Magnification: 80,000X</td>
<td>87</td>
</tr>
<tr>
<td>50</td>
<td>Diffraction pattern of Fig. 49</td>
<td>87</td>
</tr>
<tr>
<td>51</td>
<td>Electron transmission micrograph of martensite with 34 percent prior reduction in thickness of the austenite. Magnification: 40,000X</td>
<td>88</td>
</tr>
<tr>
<td>52</td>
<td>Average of longitudinal and transverse 0.2 percent yield strengths of deformed austenite, martensite with prior deformation of the austenite and deformed martensite (extrapolated linearly to 100 percent martensite)</td>
<td>104</td>
</tr>
</tbody>
</table>
## LIST OF TABLES

<table>
<thead>
<tr>
<th>Table Number</th>
<th>Description</th>
<th>Page Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>Nominal Compositions of Low-Carbon Iron-Nickel Alloys</td>
<td>17</td>
</tr>
<tr>
<td>II</td>
<td>Chemical Analysis of Alloys Nos. 4 and 5</td>
<td>18</td>
</tr>
<tr>
<td>III</td>
<td>Composition of the Electropolishing Solution</td>
<td>26</td>
</tr>
<tr>
<td>IV</td>
<td>$\cos\theta\cos\lambda$ for various slip systems in a face-centered cubic crystal with a $(110)\ [\bar{1}12]$ cold-rolling texture. Tensile axes considered parallel to $[\bar{1}12]$ and $[\bar{1}11]$.</td>
<td>91</td>
</tr>
</tbody>
</table>
ACKNOWLEDGMENTS

The author wishes to express his gratitude to Professor Morris Cohen for his support and valuable suggestions during the course of this investigation, to Mrs. Jane Operacz for her assistance in obtaining the data, to Mr. David Hoffman for the numerous exchanges of views and information during the progress of the work, and to the Ford Motor Company for supplying the experimental materials.

The author is also grateful to the Wright Air Development Center for the financial support of this program under Contract No. AF 33(616)-6873.
I. INTRODUCTION

Cold working is one of the most effective and simplest means by which metals can be substantially strengthened. Unfortunately, medium and high-carbon martensitic steels, which constitute the most important class of high-strength materials, cannot in general, be strengthened further by cold working because of their brittleness and their already high inherent flow stress. The working operation can be considerably facilitated if the flow stress of the metal is decreased by raising the temperature. As long as the working temperature is kept below the recrystallization temperature, most of the strain hardening can be retained. However, in carbon steels, the concomitant tempering reactions do not permit the martensite to be worked at a relatively high temperature while retaining high strength at the same time.

In view of the fact that the face-centered cubic austenitic phase of such steels is ductile and can be quite readily worked, it seems natural to wonder whether the martensite produced by quenching a strain-hardened austenite would produce a stronger martensite. The answer is, of course, that a thermomechanical process known as "ausforming" which consists of plastically deforming austenite and then quenching to martensite, produces a material that has not only a considerably higher strength but also good ductility.
The strengthening mechanisms in ausformed steels can be classified under two distinct categories: 1. Microstructural features (strain hardening, dislocations, twins, grain size and shape); 2. solution and precipitation hardening due to the presence of carbon and alloying elements. These two categories of possible strengthening mechanisms are simultaneously present in commercial steels, but because of their basically different natures, it was thought desirable to investigate the two separately. This investigation is confined to strengthening mechanisms of the first category. Since the presence of an appreciable amount of carbon could mask some of the more modest contributions to strengthening, the amount of carbon in the experimental materials was kept low. Another series of experiments designed to provide an understanding of the effect of carbon is presently being carried out by Mr. David Hoffman of this laboratory.

II. LITERATURE SURVEY

The earliest investigation on the effect of deforming austenite on the properties of subsequently formed martensite goes back to 1933 when Wassermann\(^1\) experimented with an iron-30 percent nickel alloy. This alloy was deformed in the austenitic condition by drawing, and then transformed to martensite by quenching in liquid air. When the strength of the martensite obtained by quenching deformed austenite was compared to that of martensite obtained from undeformed austenite,
it was found that prior deformation of the austenite increased the strength of the subsequently formed martensite, and that the strength of the austenite increased, but the strength increment of the martensite over the austenite decreased.

Again in 1933 Pomp and Iwag(2) made similar experiments on four carbon steels with carbon contents of 0.02 to 0.3 percent, and two silicon steels with silicon contents of 2.2 to 3.3 percent. These steels were rolled at various temperatures, and it was observed that rolling at 400 - 600° C increased the strength at room temperature, whereas rolling at higher temperatures caused the strength to decrease again.

These experiments attracted little attention at the time; it was not realized that an essentially new thermomechanical process for strengthening steel had been discovered. It was not until 1951 that this process again began to attract attention, when Harvey(3,4,5) proposed a modified "step-quench" treatment whereby steel is quenched from above its critical temperature into a hot bath where it is held above the $M_s$ temperature for a time which is insufficient for decomposition of the metastable austenite. This is followed by hot working by means of blasting, peening, rolling, swaging, hammering, burnishing, or forging, and then air cooled. Harvey considered that this process was especially important for shot blasting. He observed that high intensities of shot blasting could be applied to an alloy steel with 0.95 percent carbon without any danger of cracking the
material. Shot blasting this steel at a temperature of 270° C
(515° F) increased the surface hardness from 62.4 to 64.7 Rockwell C.
The higher compressive stresses remaining at the surface were
considered to be beneficial.

Similar results were obtained by Bish and O'Neill(6) who deformed
disc-shaped specimens of low alloy steels with 0.93 and 0.51 percent
carbon by drop hammering at 280 and 245° C. This was followed by
water quenching after holding at the temperature of deformation for a
certain time. Their results indicated that maximum hardness was
obtained for an optimum combination of deformation temperature and
time of isothermal holding at the deformation temperature after
deformation.

It was, however, not until 1954 when Lips and Van Zuilen(7)
published the results of their experiments on a 4.5 percent nickel,
1.5 percent chromium, 0.35 percent carbon steel, as well as a 0.9 percent
plain carbon steel, that the potentialities of this new process
attracted widespread attention. These investigators stated that to
obtain outstanding mechanical properties it was necessary to deform
the metastable austenite at a temperature lower than the recrystallization
temperature where the metastable austenite would require a long time to
decompose. They claimed that by using this process the strengths of
these two steels were increased by 30 - 40 percent, with substantial
improvements in elongation and reduction in area. As other investigators
failed to obtain such spectacular results, Steinbrenner(8) speculated
that Lips and Van Zuilen's results were in error due to a mistake in a conversion factor in changing from kilograms per square millimeter to pounds per square inch. This error was acknowledged by Hogendoorn(9) who admitted that the values reported by Lips and Van Zuilen were in error due to a mistake in the conversion factor, but still claimed that strips of steel had been produced in their laboratory with yield strengths equal to those reported by Lips and Van Zuilen (400,000 pounds per square inch) although the elongation was somewhat less.

Gullotti, Eichen and Spretnak(10) who deformed a high nitrogen, low carbon steel prior to the martensitic transformation recorded a 30 percent increase in the elastic limit. Several other investigators carried out similar experiments on various steels without, however, trying to gain a better understanding of the fundamental nature of the process. Nachtman and Moore(11) were able to enhance the strength properties of plain carbon steels by drawing at temperature between 93 and 650° C, while Marshall, Form and Hehemann(12) rolled a 4350 steel at elevated temperatures and obtained an increase in strength after rolling at 535° C (1000° F) and 845° C (1550° F). However, they obtained decreased ductility after deforming at 535° C whereas deforming at 845° C slightly increased the ductility. Ripling(13) used an SAE 1340 steel and obtained an improved toughness and higher yield strength by warm working.
Subsequent investigators began to realize the potentialities of this new process, and, besides attempting to produce steels with improved mechanical properties, also became interested in gaining a better understanding of the mechanisms which caused these improved properties. Thus, Evans and O'Neil\(^{14}\) using a 0.76 percent carbon steel reported some increase in hardness and an improvement in impact resistance after a reduction in area of about 20 percent. When they attempted to deform to more than 20 percent reduction in area, they found that increased deformation enhanced the decomposition of austenite and yielded a softer product. They examined the rolled strips metallographically and found a preferred orientation of the martensitic plates which they thought to be related with the improved properties.

Metallurgists in the Soviet Union seemed to be more interested in improving the toughness properties of steel by this process, and Sokolkov\(^{15}\) was able to retard the development of temper brittleness and substantially increase the energy absorbed for failure by hot rolling constructional alloy steels to 35 percent reduction in area at a temperature below the recrystallization temperature. He suggested that these improved properties were mainly due to a modification of precipitation kinetics.

The first thorough investigations of the effect of the various variables involved in the strengthening of steel by prior deformation of the austenite as well as an examination of the possible mechanisms was done by Schmatz and Zackay\(^{16}\). They used steels with carbon
contents varying from 0.49 to 0.98 percent, 3.96 - 4.50 percent nickel, up to 1.44 percent chromium and up to 1.78 percent silicon and found that the bay area of the TTT diagram was the most convenient for carrying out the deformation of the metastable austenite. The first 25 percent reduction in area was the most highly effective in increasing strength, becoming diminishingly beneficial with greater deformations. They thus obtained ultimate tensile strengths in excess of 400,000 pounds per square inch with good ductility. The hardness and yield strengths also increased. Higher tempering temperatures caused the ultimate tensile strength to decrease rapidly while the yield strength increased slowly, the optimum tempering temperature being 260 - 315° C (500 - 600° F). In their attempt to explain the mechanisms which caused the strengthening, they proposed that a finer martensitic plate size was produced by the prior deformation of the austenite. The presence of stresses in the highly deformed matrix and the precipitation of extremely fine carbides on slip bands of the deformed parent austenite were also proposed as possible mechanisms of strengthening.

In another publication Schmatz, Shyne and Zackay\(^{17}\) introduced the name "Ausforming" for this thermomechanical process for strengthening steel, and indicated that this name was a registered trade mark of the Ford Motor Company. In this same publication they reported some of their results on ausforming two steels with 3 percent chromium, 1.5 percent nickel, 0.75 percent manganese, 0.5 percent
molybdenum, 1.5 percent silicon and, 0.48 and 0.63 percent carbon. These steels had a high hardenability which would prevent the decomposition of the metastable austenite. One group of specimens was oil quenched immediately after deformation while the other group was air cooled to permit some non-martensitic decomposition. It was found that the ultimate tensile strengths and yield strengths of the air cooled specimens were less than the corresponding strengths in the oil quenched specimens. The ductilities were comparable. The strengthening mechanisms which they proposed were the same as those in the reference cited earlier(16). They also found that in non-ausformed as well as in ausformed steels the strength increased and the ductility decreased with increasing carbon content.

In 1959 the literature on ausforming was already so considerable and the process aroused so much interest that a review paper was circulated by the Battelle Memorial Institute(18), summarizing the data of previous publications.

Since then the increasing importance of the strength-to-weight ratio in rocket and missile technology has led to a greater interest in the possibility of strengthening steels by ausforming.

Srawley and Lupton(19) rolled a 12 percent chromium, 0.15 percent carbon steel by multiple passes at 510° C (950° F) with reheat between passes. After cooling to room temperature and tempering at 315° C (600° F), it was found that 75 percent prior reduction in area of the austenite had increased the yield strength by 70 percent while
the elongation had decreased somewhat. Their tensile specimens were taken with the tensile axes in the direction of rolling. The authors thought that if the tensile axes were taken normal to the rolling direction and in the rolling plane the yield strengths would be inferior.

Gullotti\(^{(20)}\) who made tests on steels with widely varying compositions reached the conclusion that in general 70 to 90 percent prior reduction in area of the metastable austenite would increase both ultimate tensile strength and yield strength by about 35 percent. Ductility was found to improve in some steels and to deteriorate in others. Stepwise deformation with reheating in between was proposed if the required deformation was too high to be introduced in a single pass. Multiple-pass rolling was found not to have any different effect as compared to single-pass rolling. The strengthening mechanism proposed by this investigator was a reduction of the martensitic plate size, and the introduction of strains into the lattice.

The effect of ausforming on a commercial steel was investigated by Kula and Dhosi\(^{(21)}\) who used a 4340 steel. This steel has excellent hardenability and a large bay region. After rolling to 72 percent reduction in area at 845° C (1550° F) and tempering at 230° C (450° F), they obtained a 10 percent increase in yield stress; a similar deformation carried out at 540° C (1000° F) increased the yield stress by 19 percent. The rolling was done in a single pass. They reported some recrystallization in the center of specimens rolled to more than
50 percent reduction in area at 845° C (1550° F). These investigators examined in detail several possibilities to explain the strengthening mechanism. They thought that the principal mechanism was that of a refinement of the martensitic plates, and also mentioned, for the first time in a research program concerned with ausforming, that martensite may inherit the defect structure of the parent deformed austenite. This would be well substantiated especially by the dependence of the improvement of the yield strength on the temperature of deformation of the austenite.

In another series of tests performed by Shyne, Zackay and Schmatz(22) on alloy steels with 0.31 to 0.63 percent carbon, a reduction in area of 93 percent by multipass rolling of round stock at a temperature between the $M_s$ and the recrystallization temperature gave an increase in yield strength of 40 - 60,000 pounds per square inch. The authors discussed in detail several possible strengthening mechanisms. These were: 1. An increase in the number of nucleation sites of martensitic plates resulting in a smaller plate size; 2. bending of the austenite lattice may restrict the martensitic shear to shorter plate lengths; 3. heavily worked slip bands may block the progress of advancing plates; and 4. austenitic grains become elongated in shape and their dimensions are reduced in the plane transverse to the rolling direction, thus resulting in a limited size of the martensitic plates. These arguments were based essentially on a detailed mechanism of how the martensitic plate size might be
reduced. The possibility of defects being inherited by martensite from the parent austenitic matrix was not mentioned.

Zackay, in a review of strengthening by martensitic transformation\(^{(23)}\), described the interaction of martensitic plates with grain boundaries, slip bands and twin interfaces of the austenitic matrix. According to the information then available, austenitic grain boundaries as well as decorated slip bands were considered to be impermeable barriers to martensite, whereas twin interfaces and slip bands were considered to be semipermeable to advancing martensitic plates although the morphology might be changed. When passing through semipermeable obstacles, the martensitic plates were seen to be severely distorted or deflected, thereby compartmentalizing the plate to smaller blocks. Thus, Zackay finally came to the conclusion that defect structures in the austenite could be inherited by the subsequently formed martensite.

The effect of prior austenitic grain size was examined by Zackay, Justusson and Schmatz\(^{(24)}\) who rolled an H11 tool steel (0.4 percent carbon, 5 percent chromium, 1.3 percent molybdenum and 0.5 percent vanadium) to 94 percent reduction in area at 480° C (900° F) and 650° C (1200° F). After tempering at 510° C (950° F) the tensile strength of the steel rolled at 480° C (900° F) was found to be somewhat higher than that rolled at 650° C (1200° F). A smaller prior austenitic grain size increased the tensile strength, but the effect was rather small compared to the overall gain obtained
by ausforming. Using another series of alloys with carbon content varying from 0.3 to 0.6 percent, they also showed that the strength increase was proportional to the carbon content, but that the ductility decreased. However, the most important variable in ausforming was considered by these authors to be the amount of prior deformation of the austenite.

The theory that strengthening by ausforming is mainly due to a grain-size effect was investigated by Grange and Mitchell\(^{(25)}\) who also studied the morphology of the martensitic plates in more detail. By choosing a suitable alloy they obtained only about 10 percent martensite upon quenching and were thus able to observe martensitic plates lying with their long axes preferentially aligned in the rolling direction. They also experimented with as L340 steel producing several grain sizes as well as elongated grains by warm working, and found that the warm-worked specimens were (at any corresponding grain size) stronger than the conventionally processed specimens with equiaxed grains. They stated, although inconclusively, that strengthening seemed to be related to the elongation of the austenite grains.

In the meantime Soviet investigators became active in the field of ausforming. Gulyayev and Shigarev\(^{(26)}\) deformed a 0.5 percent carbon alloy steel to 90 percent reduction in area at 900\(^\circ\) C, and after an oil quench obtained higher strength and ductility, while Potak, Ozhrekhovsky, Pevzmer, Roshina and Yermakov\(^{(27)}\) obtained
similar results on alloy steels containing 0.33 to 0.64 percent carbon, but failed to obtain any increase in strength with only 50 percent prior reduction in area. On the other hand, Sokolkov, Petrova and Chuprakova\(^{(28)}\) investigated the effect of deformation temperature on the low temperature \((-195^\circ \text{ C})\) properties of a 0.3 percent alloy steel, and found that the lowest temperature at which they deformed the metastable austenite \((900^\circ \text{ C})\) gave the best combination of strength and ductility by ausforming.

Hosoi and Pinnow\(^{(29)}\) examined the tensile properties of a 410 stainless steel deformed before and after the martensitic transformation. They found that deforming at \(425^\circ \text{ C} \ (800^\circ \text{ F})\) to 57 percent reduction in area improved both ultimate tensile strength and yield strength but reduced ductility. When the steel was first quenched to martensite and then reduced in area by 16 percent at \(425^\circ \text{ C} \ (800^\circ \text{ F})\), a 32 percent increase in ultimate tensile strength, 41 percent increase in yield strength and a 39 percent decrease in elongation were observed. A combined 29 percent reduction in area of metastable austenite plus a 16 percent reduction in area after quenching to martensite at room temperature and at \(425^\circ \text{ C} \ (800^\circ \text{ F})\) did not give better properties in every case than deformation of the metastable austenite alone or deformation of the martensite alone.

At a conference in England, Zackay and Justusson\(^{(30)}\) reviewed the state of knowledge on ausforming. They observed that strength and ductility of quenched and tempered martensitic steels are
inversely related. Above yield strengths of 300,000 pounds per square inch, ductility usually decreases beyond the acceptance level. Strength and ductility were reported to be virtually independent of prior austenitic grain size but very highly dependent on carbon content. These investigators demonstrated that a pronounced martensitic texture was not required for the strengthening of ausformed steel by an experiment in which a cube-shaped specimen of 410 stainless steel was ausformed by sequentially deforming the austenite equally in all three principal axes. The effect of this deformation on the cube was calculated to be equivalent to about 90 percent reduction in area. This specimen showed an increase in strength of about 80 percent while the grain structure appeared to be hardly changed at all.

More recently, transmission electron microscopy of thin foils has been used in studying strengthening mechanisms in ausformed steels. In a research program conducted by the Aeroneutronic Division of the Ford Motor Company\(^{(31)}\), an H11 steel was deformed at various temperatures and to various degrees of reduction in area. The yield strengths were highest for the specimens deformed at the lowest temperatures; at the highest deformation temperatures, a considerable amount of isothermal decomposition products was observed. The strength loss at higher temperatures was attributed to the presence of isothermal decomposition products, whereas recovery was thought to be only of minor importance. It was reported that a fine striated or "twinned" structure was seen throughout the ausformed steel.
The electron diffraction patterns of selected areas showed a great many "satellite reflections" which were attributed to very thin laminated crystals. Excessive broadening of spots was considered to be due to strains, and these broadened spots were obtained mainly in regions where striations were seen. The investigators, therefore, assumed that the striations were regions of high dislocation density, and that the strains around the dislocations caused the broadening. Diffraction patterns from areas with cellular structures gave rings suggesting that several crystallites were contributing to the diffraction patterns.

In a more recent publication, Tamura, Yoshimura, Ibaraki and Tagaya (32) used iron-nickel and iron-nickel-carbon alloys which were austenitized at 900° C, quenched into oil at 200° C, held there for two minutes then ausformed and cooled to room temperature. Some martensite was formed in the iron-nickel alloy during deformation. Hardness measurements showed that the iron-nickel alloy hardened only a little, whereas the effect of ausforming on the iron-nickel-carbon alloy was much greater. Examining thin films of the ausformed material by transmission electron microscopy, the authors detected twins in the iron-nickel-carbon martensite before it was ausformed. However, when this alloy was deformed to 30 percent reduction in area in the austenitic state and subsequently quenched, broad lamellar bands were seen instead of twins. The authors concluded that these broad bands were crystal imperfections in the ausformed martensite.
The effect of cold work on the austenite could be easily seen by transmission electron microscopy.

We may sum up as follows: 1. In all steels strength increases by prior deformation of the metastable austenite. 2. Ausforming does not generally cause any marked deterioration of ductility. 3. Usually, lower temperatures of deformation of the metastable austenite tend to yield the optimum mechanical properties. 4. Decomposition of the metastable austenite into non-martensitic products is deleterious to strength. 5. Most investigators attribute the strengthening mechanism to a refinement of the martensitic plate size or an elongation of the austenite grains, although some investigators have proposed the introduction of strains into the lattice as a strengthening mechanism. 6. Transmission electron microscopy of thin foils of ausformed steels has shown that the fine structure of the martensite (twins) and the possibility of the martensite inheriting lattice defects from the parent deformed austenite may play an important role in the strengthening mechanism on ausforming.
III. SELECTION OF MATERIALS AND EXPERIMENTAL PROCEDURE

A. Selection of Materials

A series of low-carbon iron-nickel alloys were selected with their compositions based on the $M_S$ temperatures as compiled by Winchell (33). Five alloys with the following compositions were prepared for this purpose by the Ford Scientific Laboratory:

<table>
<thead>
<tr>
<th>Alloy number</th>
<th>Nickel (weight percent)</th>
<th>Carbon (weight percent)</th>
<th>Iron</th>
<th>$M_S$ temperature ($^{\circ}$C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>27.84</td>
<td>0.003</td>
<td>balance</td>
<td>+11</td>
</tr>
<tr>
<td>2</td>
<td>28.59</td>
<td>0.008</td>
<td>&quot;</td>
<td>+6</td>
</tr>
<tr>
<td>3</td>
<td>30.33</td>
<td>0.019</td>
<td>&quot;</td>
<td>-8</td>
</tr>
<tr>
<td>4</td>
<td>31.64</td>
<td>0.016</td>
<td>&quot;</td>
<td>-28</td>
</tr>
<tr>
<td>5</td>
<td>33.44</td>
<td>0.010</td>
<td>&quot;</td>
<td>-83</td>
</tr>
</tbody>
</table>

The $M_S$ temperatures were determined by electrical resistivity measurements during slow cooling as described below.

The chemical analysis for alloys number 4 and 5 gave the following results:
TABLE II*

Chemical Analysis of Alloys No. 4 and 5

<table>
<thead>
<tr>
<th>Alloy number</th>
<th>Nickel</th>
<th>Carbon</th>
<th>Manganese</th>
<th>Silicon</th>
<th>Sulfur</th>
<th>Phosphorus</th>
<th>Iron</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>31.9</td>
<td>0.017</td>
<td>&lt;0.003</td>
<td>&lt;0.001</td>
<td>0.005</td>
<td>0.001</td>
<td>balance</td>
</tr>
<tr>
<td>5</td>
<td>33.4</td>
<td>0.011</td>
<td>0.003</td>
<td>0.005</td>
<td>0.005</td>
<td>&lt;0.001</td>
<td>balance</td>
</tr>
</tbody>
</table>

* Numbers express weight percent.

Alloys number 1, 2 and 3 were found not to be suitable for this investigation since, at room temperature, all free surfaces of the austenite were covered with a layer of the body-centered cubic phase which was identified by x-ray diffraction. The drop in electrical resistivity at the $M_s$ temperature indicated that the bulk of the material was austenitic, and that this effect was confined to the surface. A mechanically polished surface of alloy number 3 exhibited patches of the body-centered cubic phase, the rest being face-centered cubic, as shown in Fig. 1. Electropolishing prevented the layer of surface martensite from forming but some "chevrons" of martensite appeared as shown in Fig. 2. The reason for the formation of this surface layer of the body-centered cubic phase is thought to be due to an increase in $M_s$ temperature because of the absence of constraints at the surface. Alloy number 5 did not produce more than 50-60 volume percent martensite even upon quenching in liquid helium. Alloy number 4 was therefore
Fig. 1. Alloy number 3 after 24 hours at 1000° C and mechanical polishing. Etchant: 6 percent nital. Magnification: 100X.

Fig. 2. Alloy number 3 after 24 hours at 1000° C. Electrolytic polishing and etching. Magnification: 100X.
B. Heat Treatment

The iron-31.9 percent nickel alloy was received in the form of slabs 7/8 inches thick, 3 inches wide and about 10 inches long. These slabs were prepared by hot-rolling the as-cast ingots to 1000°C. The material in the as-received condition had an irregular grain size (Fig. 3). In order to obtain a more uniform grain size and to give an austenitizing and homogenizing treatment to the alloy, pieces of the required dimensions were cut from the slabs and annealed at 1000°C for 24 hours. The resulting grain structure is shown in Fig. 4.

The chamber in which the heat treatment was carried out consisted of a stainless steel tube with an internal diameter of 3 inches, closed at both ends and placed in a muffle furnace. Helium of 99.99 percent purity was circulated in the tube during the heat treatment, and a positive pressure of helium above atmospheric pressure was maintained by immersing the gas outlet tube under 3 or 4 inches of water. After completion of the heat treatment the specimens were removed from the furnace and quenched in water as fast as possible in order to prevent any excessive oxidation in air, and any precipitation of the a phase during slow cooling. The formation of a
Fig. 3. Alloy number 4 in as-received condition.
Electrolytic polishing. Etchant:
6 percent nital. Magnification: 100X.

Fig. 4. Alloy number 4 after 24 hours at 1000° C.
Electrolytic polishing. Etchant:
6 percent nital. Magnification: 100X.
superficial oxide layer on the specimens could not be prevented as the specimens were exposed to air while hot during the time elapsed between removal from the furnace and quenching.

The homogeneity of the composition was checked by running a traverse across the thickness direction of the material in the as-received condition with an electron microprobe. The nickel concentration across this traverse was found not to vary by more than \( \pm 1/4 \) percent. The distances over which these variations occurred were about one order of magnitude smaller than the grain size indicating that no segregation was present at the grain boundaries.

Finer grain sizes could be produced from a block of material which had been previously austenitized at 1000\(^\circ\) C for 24 hours by rolling at room temperature to about 70 percent reduction in thickness and then controlling the temperature and time of recrystallization. Temperatures above 800\(^\circ\) C were used to avoid the alpha plus gamma region of the phase diagram. Although the precipitation kinetics in this iron-nickel alloy is known to be very sluggish\(^{(34)}\), it could, nevertheless, be possible for the alpha phase to precipitate on cooling in amounts too small to be detected by metallography or x-ray diffraction. In order to prevent this, the specimens were water quenched after the annealing treatment. In view of the relatively high temperatures which were required for recrystallization, the heat treatment was again carried out in a helium atmosphere.
A third type of heat treatment used in this investigation consisted of strain-relaxation anneals for long periods of time at 300° C, just below the \( A_s \) temperature for this alloy. Because of the low temperature, no oxidation problems were present with this type of heat treatment and the specimens could be safely heat treated in air in a muffle furnace.

All furnaces used in this investigation were calibrated by means of a chromel-alumel thermocouple.

C. Deformation of Austenitic and Martensitic Specimens

The approximate \( M_d \) temperature for the iron-31.9 percent nickel alloy was evaluated by extrapolating the data of Kaufman and Cohen\(^{34}\), who determined the \( A_d \) and \( M_d \) temperatures for various iron-nickel compositions, and was found to be a little below 100° C. This \( M_d \) temperature precluded the possibility of deformation at room temperature since a considerable amount of martensite would form during the plastic straining. On the other hand, the temperature at which the austenite was to be deformed had to be as low as possible in order to preserve the maximum effect of cold-work. Blocks of various thicknesses, about 2.75 inches wide and 3 inches long that had been austenitized as previously described were heated to 300° C in a muffle furnace and reduced by successive passes through a rolling mill. The material was reheated to 300° C for 5 minutes between passes and the same end was inserted between the rolls at each pass. Rolled
sheets with reductions in thickness of $34, 54, 76$ and $88$ percent were prepared. The final product was always of about the same thickness (0.1 inch), the reduction in thickness being controlled by the original thickness of the block. The specimens widened only a few percent during these operations. The elongation, however, was considerable depending on the amount of reduction in thickness desired.

Deformation of martensitic specimens was carried out by preparing blocks similar to those used for deformation of austenitic specimens. After the austenitizing treatment, these blocks were quenched in liquid nitrogen to obtain martensite. The procedure was the same as for austenitic specimens except for the fact that rolling was done at room temperature. With this technique, sheets about 0.1 inch thick were prepared with reductions in thickness of 32, 43, 56, 71 and 87 percent.

D. Determination of $M_S$ Temperatures

The electrical resistivity of martensite in the iron-nickel system being considerably lower than that of austenite, electrical resistivity measurements provide a simple and effective method of following the course of martensitic transformation. If the electrical resistivity of an austenitic specimen is measured while it is being cooled past the $M_S$, a drop in the value of resistivity is observed at the $M_S$ temperature.
The $M_s$ temperatures of annealed as well as deformed austenite were determined by resistivity measurements during slow cooling to subzero temperatures. Strips about 3 inches long, 0.1 inch thick and 0.2 inch wide were machined from annealed austenite and from rolled sheets of austenite. The electrical connections to the specimens were made by soldering nickel wires about 4 inches long to the specimens. The current leads were connected to the extremities of the specimens while the voltage leads were connected about 0.25 inch away from the extremities. The resistance of the specimen was measured by comparing the voltage drop across the voltage leads to the voltage drop across a standard resistance by means of a double Kelvin bridge. An e.m.f. of 6 volts from a storage battery was applied across the current leads.

The specimen was immersed in an agitated bath of methyl cyclohexane which could be cooled down to -95° C by adding liquid nitrogen. Below -95° C the methyl cyclohexane began to freeze. However, all the $M_s$ temperatures which were measured were well above -95° C and it was not necessary to continue the cooling below this temperature. A resistivity reading was also made on immersing the specimen in liquid nitrogen and one final reading was taken after the specimen had been brought back to room temperature. Since no absolute measurements of resistivity were required, resistivity in arbitrary units was plotted versus temperature and a change in the
slope of the resulting curve indicated the onset of the martensitic
transformation. All measurements were duplicated and the $M_s$
temperatures which were thus determined had a reproducibility of
about $\pm 5^\circ C$. The temperature was measured by means of a copper-
constantan thermocouple whose junction was immersed near the
specimen in the cooling bath. The thermocouple had been previously
calibrated by measuring the temperatures of liquid nitrogen,
frozen carbon dioxide, and ice.

E. Metallography

All specimens for metallographic examination were electropolished
on a "Disa Electropol" electropolishing machine. The electrolyte
used had the following composition:

<table>
<thead>
<tr>
<th>TABLE III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition of the Electropolishing Solution.</td>
</tr>
<tr>
<td>700 milliliters</td>
</tr>
<tr>
<td>120 milliliters</td>
</tr>
<tr>
<td>100 milliliters</td>
</tr>
<tr>
<td>78 milliliters</td>
</tr>
</tbody>
</table>
The electrolyte circulation velocity was kept at a setting between 3 and 5 on the calibrated dial of the machine. A current density of about 10 amperes per square centimeter gave the best polishing conditions for annealed as well as deformed austenitic and martensitic specimens. A new solution had to be prepared when the one being used turned light green, since at that time its effectiveness for polishing was decreased. The surface to be polished was first abraded on wet sand belts followed by silicon carbide abrasive papers, through 600 grit. Electropolishing for 10 to 20 seconds removed all the deformed material and left a mirror-like polished surface. More material could be removed from the surface, when this was necessary, by increasing the duration of polishing but the resulting electropolished surface was not as good as when electropolishing was stopped after 20 seconds at the most. Etching was achieved by immersing in a 6 percent nital solution. Swabbing instead of immersing with the same etching solution gave a better delineation of the boundaries between martensitic plates and the surrounding retained austenite. This was especially useful in the determination of the amount of retained austenite by point counting. Electroetching was tried on the same apparatus as for electropolishing. Very good etching was sometimes obtained, but the etching conditions were found to be extremely sensitive to variations in the current density and electrolyte flow rate; and the results were not reproducible. This technique was
b. Tensile tests: Tensile specimens with their tensile axes parallel and perpendicular to the rolling direction were machined from undeformed and deformed austenite, martensitic specimens produced by quenching both undeformed and deformed austenite in liquid nitrogen, and deformed martensitic specimens obtained by cold-rolling undeformed austenite after quenching in liquid nitrogen. The dimensions of the tensile specimens are shown in Fig. 5. The rolled sheet material was thinned down to the thickness of the tensile specimens by machining off an equal amount from both surfaces. The cross sectional areas in the middle of the gage length were determined from micrometer measurements.

The specimens were tested on a Tinius Olsen Electromatic Universal tensile machine. The cross head speed was calibrated before and after each series of tests. Since the specimens were too small to allow an extensometer or strain gage to be used, the 0.2 percent yield strengths were measured from the load-elongation curves. The ultimate tensile load and total elongation could be read directly from the chart. In calculating the elongation in percent, the total elongation was divided by the gage length of 15 mm.

c. Determination of amount of retained austenite: The saturation magnetization of austenite and martensite being different, a method suggested by Arrott(35,36) for phase composition analysis was considered. If the saturation magnetization of the constituent
Thickness: 2 mm.

FIG. 5 TENSILE SPECIMEN. DIMENSIONS IN MILLIMETERS.
phases are assumed to be structure insensitive, then the total saturation magnetization should be a linear function of the amounts of each phase present. The Curie temperature for the iron-31.9 percent nickel austenitic alloy is above room temperature so that the alloy in the austenitic condition is ferromagnetic although less so than the same alloy in the martensitic conditions. The magnetization behavior of a deformed austenitic specimen was investigated using the 86,000 gauss electromagnet of the National Magnet Laboratory at Massachusetts Institute of Technology. It was found that although annealed austenite saturated at an applied field of a few thousand gauss, a specimen which had been cold worked to about 80 percent reduction in thickness did not come anywhere near saturation even at 86,000 gauss. Hence, measurements of retained austenite could not be made using this method.

Instead, the retained austenite content was evaluated by point counting. The specimens were prepared by cutting the unstretched ends of martensitic tensile specimens after the tensile tests were performed, and point counting was done on the longitudinal sections by making several traverses with an 8 x 8 square grid mounted in the eye piece of a microscope. The statistical accuracy of this method has been analyzed by Hilliard and Cahn\(^{37}\) who have shown that the standard mean deviation is given by \(\sqrt{1/N}\), where \(N\) is the number of points falling on the phase which is being counted. The analysis is based on the assumption that the spacing of the grid is such that no
single feature of the phase being counted is occupied by more than one point. To satisfy this condition, at least approximately, it was found necessary to modify the magnification at which the point counting was done from specimen to specimen. Enough points were counted to keep the mean deviation within 2 percent.

G. Transmission Electron Microscopy and Preparation of Thin Foils

A Siemens Elmiskop I electron microscope operated at 100 kilovolts was used. It was found that a magnification on the fluorescent screen of the microscope of about 20,000 gave the best conditions of resolution for the types of specimen being examined. Higher magnification decreased the visible area with no apparent gain in resolution. Transmission electron micrographs were recorded on a 35 mm film and then enlarged as required. On each area whose micrograph was taken, a selected-area diffraction pattern was also taken. A stereoscopic drive for tilting the specimen was installed in the microscope, but since this tilting mechanism permitted tilting on one axis only, it was found to be of limited usefulness, especially in the examination of twin structures of martensite whose appearance is very sensitive to the orientation.

Two reviews of existing techniques for the preparation of thin foils for transmission electron microscopy\(^{(38,39)}\) indicate that several procedures may be equally satisfactory. Heidenreich\(^{(40)}\) prepared thin foils of aluminum by electrolytic perforation of a sheet 125 microns
thick; Hirsch, Kelly and Menter\textsuperscript{(41)} thinned gold samples by chemical etching, whereas Castaing\textsuperscript{(42)} was able to obtain thin foils of aluminum by ionic bombardment. Other procedures include the Bollmann technique\textsuperscript{(43)}, whereby preferential electropolishing may be obtained at the center of the specimen by using pointed cathodes; the so-called "window technique" used by Nicholson, Thomas and Nutting\textsuperscript{(44)} and Tomlison\textsuperscript{(45)}; the "figure of eight technique" of Brandon and Nutting\textsuperscript{(46)}; and finally the jet machining method of Kelly and Nutting\textsuperscript{(47)} for thinning bulk specimens down to about 100 microns.

The procedure used in this investigation was a modification of the Bollmann technique. The sheet, which was about 0.1 inch thick to start with, was first machined down to about 0.01 inch by removing equal amounts of material from both sides. The sheet was then further thinned down to about 0.005 inch by grinding on a 600 grit silicon carbide abrasive paper. Samuels and Wallwork\textsuperscript{(48)} in an investigation on the depth of the layer deformed by various techniques of surface polishing have shown that the depth of the deformed layer caused by a 600 grit silicon carbide abrasive paper does not exceed 2 microns, or, slightly less than 2 percent of the thickness of 0.005 inch to which the specimens in this investigation were thinned down. Discs 1/8 inch in diameter which fitted the specimen holder of the Elmiskop I electron microscope were punched
from these thin sheets. A disc specimen was then mounted in a platinum foil clip as shown in Fig. 6 and stop-off* lacquer was applied around the periphery on both surfaces. The platinum clip and disc were then immersed in an electrolyte consisting of 7 milliliters, 70 percent perchloric acid and 93 milliliters glacial acetic acid. The specimen was held horizontally and placed between a light source and a low magnification microscope through which the progress of the polishing operation could be observed. An open circuit direct current potential of 60 volts was applied between the specimen holder (anode) and a stainless steel cathode. The bath was agitated by a slowly revolving stirrer, and the temperature was kept at about 20° C by circulating cold water in a coil placed in the electrolyte. The polishing operation was continued until a small hole appeared in the non lacquered region of the specimen.

After the specimen was taken out of the holder, the lacquer was removed and the specimen put back in the holder for further polishing. This operation was continued until several holes appeared in the specimen. When the bridge between two holes was just eaten away, the polishing operation was immediately stopped. The specimen was washed in distilled water, alcohol and ether. In general, regions such as the ones shown within the dotted circles of Fig. 7 had large transparent areas. The whole specimen could be placed in

* Microstop Stop-off Lacquer, manufactured by Michigan Chrome and Chemical Company.
FIG. 6 THIN FOIL SPECIMEN PREPARED FOR FIRST POLISHING OPERATION.

FIG. 7 THIN FOIL SPECIMEN AFTER SECOND POLISHING OPERATION.
the microscope without the necessity of manipulating the thin region. Some annealed austenite specimens were prepared this way and examined in the microscope to determine whether the grinding and punching operations introduced any cold-work. Inasmuch as no unusual structures could be seen, this procedure was adopted. It may be of interest to observe here that in the Bollmann, window, and figure of eight techniques, the thin foil region is cut with a scalpel from the thicker region to which it is attached, thus increasing the changes of distorting it. After the lacquer was removed the specimens could be stored indefinitely, since any oxide that formed would be removed by the second polishing. However, the specimens were examined within a few hours after the second polishing operation as the brightly polished surfaces had a tendency to oxidize.

H. X-ray Diffraction

General Electric XRD-5 Spectrogoniometer was used in performing these experiments. Chromium and cobalt radiations were used and the K\(_2\) components were eliminated with a vanadium filter for the chromium radiation and an iron filter for the cobalt radiation. Since the K\(_2\) radiation of cobalt (1.621 Å) is shorter than the K absorption edge of the iron in the specimen (1.743 Å) considerable background intensity was detected. This was reduced to a manageable level by placing the iron filter in the incident beam instead of in the diffracted beam, thus preventing the cobalt K\(_2\) radiation from
arriving at the specimen. The martensite \{110\}, \{200\} and \{211\} and
the austenite \{200\}, \{111\} and \{220\} reflections could be obtained
with chromium radiation, for higher order reflections it was necessary
to use cobalt radiation.

The sheets of austenite which had been rolled at 300° C were
examined for the presence of martensite by running the whole
spectrum from an angle of 2θ of 50° to 165° at a scanning speed of 2°
per minute using chromium radiation. The recording device was
adjusted for maximum sensitivity, and rolling plane, transverse, and
longitudinal sections were tested from each of the rolled sheets.
Longitudinal and transverse sections were prepared by mounting 3 or
\(\frac{3}{4}\) strips side by side in a bakelite mold, grinding, polishing and
etching. The rolling plane sections were used without any
preparation. It is estimated that the presence of as little as 1 or
2 percent martensite could be detected. No trace of martensite was
detected in any of the rolled sheets. Machined and ground surfaces
were also examined for the presence of martensite. It was found that
when annealed or deformed austenite was ground or machined, the
surface showed no trace of martensite, but the chips were martensitic.

The procedure for determining the retained austenite content in
steels by making measurements of integrated intensities has been
described by Averbach, Castleman and Cohen\(^{49,50,51,52}\). However,
since this method cannot be used when cold work textures are present,
the point counting method described in a previous section was used
instead.
A line-breadth analysis was employed to determine the particle size and microstrain in cold-worked austenite, in martensite after prior deformation of the austenite, and in deformed martensite. When martensite and austenite were present simultaneously, most reflections overlapped and it was only possible to make use of the \{110\} and \{220\} martensite reflections which overlapped only slightly with the \{200\} and \{400\} austenite reflections. A precise Fourier analysis for the separation of microstrain and particle size as described by Warren and Averbach\(^ {53,54}\) was not justified since that type of analysis is especially sensitive to the tails of the diffraction profiles.

In the line-breadth analysis which was adopted, the various contributions to line breadth were assumed to have gaussian profiles and, consequently, the squares of the various contributions could be added as has been shown by Warren and Biscoe\(^ {55}\), i.e.:

\[
B^2 = b_1^2 + b_\epsilon^2 + b_L^2
\]  

where \(B\) is the measured breadth at half-peak intensity, and \(b_1\), \(b_\epsilon\) and \(b_L\) are the half-peak breadths caused by instrumental, strain, and particle-size broadening. In measuring breadths, only the \(K_{\alpha_1}\) peak was considered. The \(K_{\alpha_1}\) and \(K_{\alpha_2}\) peaks were separated by the graphical method proposed by Rachinger\(^ {56}\). For the various martensite lines, \(b_1\) was evaluated by using a well-annealed specimen of pure \(\alpha\)-iron, whereas for the austenite lines a well-annealed
specimen of austenite was employed. The diffraction profiles were recorded on a linear chart at a scanning speed of 0.2° (2θ) per minute with a horizontal divergence of the incident beam of 1° and a detector slit of 0.2°. The time width of the detector slit was thus 60 seconds, and to avoid any distortion of the lines a time constant of 24 seconds was selected. The counting rates were quite low at the background level, and seldom exceeded 1000 counts per second at the peaks. The counting device on this spectrogoniometer consisted of a multichamber geiger tube; the manufacturer's specifications indicated that no counting losses were to be expected at counting rates of less than 10,000 counts per second. The counting rates were thus kept well within the linear range of the counter.

To separate the particle size and microstrain, it was necessary to have two different orders of the same diffraction line. For the angular dependence of the particle size, the relationship given by Jones(57) was used, whereas the microstrain was assumed to vary with the angle according to the relationship given by Stokes and Wilson(58) and Megaw and Stokes(59). These relationships are:

\[ b_L = \frac{\lambda}{L \cos \theta} \]  

(2)

and \[ b_\epsilon = \frac{4\epsilon}{\cot \theta} \]  

(3)
From equation (1) we can write

\[ b^2 - b_1^2 = b^2 = b_\epsilon^2 + b_L^2 \]  \hspace{2cm} (4)

Combining equations (2), (3) and (4) as was done by Werner, Averbach and Cohen\(^{(60)}\), the following relationship is obtained:

\[ \left( \frac{b \cos \theta}{\lambda} \right)^2 = \left( \frac{1}{L} \right)^2 + (4\epsilon)^2 \left( \frac{\sin \theta}{\lambda} \right)^2 \]  \hspace{2cm} (5)

By plotting \((b \cos \theta/\lambda)^2\) versus \((\sin \theta/\lambda)^2\) \(L\) can be calculated from the intercept and \(\epsilon\) from the slope of the line drawn through the points corresponding to two or more orders of a given diffraction line.

The stacking-fault probability in the deformed face-centered cubic austenite could be calculated by measuring the peak shift of the \(\{111\}\) and \(\{200\}\) reflections. The stacking-fault density \((\alpha)\) has been shown by Patterson\(^{(61)}\) to be given by the following relationship:

\[ \Delta(2\theta_{\{200\}} - 2\theta_{\{111\}}) = -\frac{45/3}{\pi^2} \alpha (\tan \theta_{\{200\}} + \frac{1}{2} \tan \theta_{\{111\}}) \]  \hspace{2cm} (6)

According to the theory, the presence of stacking faults shifts the \(\{200\}\) and \(\{111\}\) reflections in opposite directions so that the two lines provide an internal standard. The angular separation of the two peaks was measured from the centers at half peak intensities of the \(K_{\alpha_1}\) components. The doublet was again resolved by Rachinger's\(^{(56)}\) graphical method. The two peaks were recorded in a single run and the
accuracy of these measurements was estimated to be about 0.03° (28).

Twin and deformation faults in the body-centered cubic martensite affect the measured particle size. According to the theory worked out by Guentert and Warren(62) and Hirsch and Otte(63):

\[
\frac{1}{L} = \frac{1}{D_p} + \frac{1}{D_f}
\]  

(7)

where \(D_p = \frac{k}{1.5 \alpha + \beta}\) apparent particle size due to twins and deformation faults

\[
\alpha = \text{probability of finding a deformation fault in a \{211\} plane}
\]

\[
\beta = \text{probability of finding a twin fault on a \{211\} plane}
\]

\(D_p = \text{true particle size}
\]

\[
k_{\{110\}} = \sqrt{\frac{3}{2}}; \quad k_{\{200\}} = \frac{3a}{4}; \quad k_{\{211\}} = \sqrt{\frac{3}{6}}
\]  

(9)

\(a\) being the lattice parameter. To separate \(D_p\) and \(D_f\) from the observed value of \(L\), it is necessary to obtain \(L\) from two different reflections and their higher orders. Since it was possible to get only one martensite reflection with its second order in this investigation, \(D_p\) and \(D_f\) could not be separated.
IV. RESULTS

A. Effect of Deformation on $M_s$ Temperature and Amount of Martensite Produced

The $M_s$ temperature as determined by electrical resistivity measurements during slow cooling was not changed to any measurable extent by rolling the austenite at 300° C. Two typical resistivity curves are shown in Fig. 8. The change in slope at the $M_s$ temperature of the curve for the austenite with no deformation is abrupt whereas the curve for the austenite with 88 percent reduction in thickness bends down more gradually. It can thus be inferred that deforming the austenite reduced the amount of martensite formed in a given temperature interval while the temperature at which the first plates appeared was not appreciably changed. Part of the higher resistivity below the $M_s$ temperature of the curve for the austenite with 88 percent reduction in thickness as compared to the curve with no reduction was probably due to the effect of cold work.

The amount of martensite formed upon quenching in liquid nitrogen as a function of the amount of prior reduction in thickness of the austenite is shown in Fig. 9, as determined by point counting on specimens cut from the ends of tensile specimens. The first 35 percent reduction in thickness caused a substantial decrease in the amount of martensite formed whereas further reduction caused a lesser decrease. It was found that when larger sections were quenched the amount of
FIG. 8 NORMALIZED CURVES SHOWING RESISTANCE IN ARBITRARY UNITS VERSUS TEMPERATURE FOR 0% AND 88% REDUCTION IN THICKNESS OF THE AUSTENITE BY ROLLING.
FIG. 9 EFFECT OF AMOUNT OF PRIOR REDUCTION OF THE AUSTENITE ON THE AMOUNT OF MARTENSITE FORMED BY QUENCHING IN LIQUID NITROGEN.
martensite formed was less. This was attributed to a slower cooling rate during quenching which may have allowed some stabilization of the austenite to take place.

B. **Mechanical Properties**

Austenite had a high rate of work hardening (Fig. 10). The increase in hardness was rapid at small reductions in thickness and levelled off with increasing reductions, whereas the hardness of martensite with prior deformation of the austenite and of deformed martensite increased more slowly.

Microhardness measurements on individual martensitic plates in ausformed martensite as well as in cold-worked martensite revealed a steady increase with the amount of reduction, the hardness of the deformed martensite plates being generally higher than that of ausformed martensite for any given reduction (Fig. 10). These hardness measurements were made by indenting the martensitic plates on the rolling plane section of the rolled sheets. Indents could be made only on the larger plates which were visible.

Typical stress-elongation curves for austenite, austenite and martensite with no prior deformation of the austenite, and with prior deformation of the austenite are shown in Figs. 11, 12 and 13, respectively. Deformation increased the strength of austenite while drastically reducing the elongation to rupture (Fig. 11). A comparison of the stress-elongation curves in Figs. 11 and 12 indicates
FIG. 10  MICROHARDNESS OF DEFORMED AUSTENITE, MARTENSITE WITH PRIOR DEFORMATION OF THE AUSTENITE, AND DEFORMED MARTENSITE AS A FUNCTION OF REDUCTION IN THICKNESS BY ROLLING.
FIG. 11 STRESS-STRAIN CURVES FOR AUSTENITE WITH 0% AND 88% REDUCTION IN THICKNESS.
Fig. 12 Stress-strain curves for austenitic and martensitic specimens with no deformation.
FIG. 13 STRESS-STRAIN CURVES FOR MARTENSITE WITH 0% AND 88% PRIOR REDUCTION IN THICKNESS OF THE AUSTENITE.
that the strengthening due to the formation of martensite was
similar to the effect of cold working the austenite. On the other
hand, prior deformation of the austenite had the effect of
increasing the strength of the subsequently formed martensite while
leaving the elongation unaffected (Fig. 13).

The variation of the 0.2 percent yield strength and ultimate
tensile strength with the volume fraction of martensite (Fig. 14)
provided the justification for linear extrapolations of ultimate
tensile strengths and 0.2 percent yield strengths to 100 percent
martensite. A series of austenitic specimens with no deformation
were quenched to various temperatures between room temperature and
liquid nitrogen in order to produce varying amounts of martensite.
The quenching baths consisted of methyl cyclohexane cooled to the
required temperature by means of liquid nitrogen. The ultimate
tensile strength and 0.2 percent yield strength when plotted versus
the amount of martensite varied almost linearly (Fig. 14). The
small deviations from a straight line were within the experimental
scatter of the tensile data.

The strength in the transverse direction of rolled austenite was,
in general, a few percent higher than in the longitudinal direction
(Figs. 15 and 17). In Fig. 16 are shown the corresponding values for
martensitic specimens. This curve was plotted from the actual values
calculated from the stress-elongation curves, without correcting for
the retained austenite. As the amount of prior reduction in
FIG. 14  EFFECT OF VOLUME PERCENT OF MARTENSITE ON ULTIMATE TENSILE STRENGTH AND 0.2 PERCENT YIELD STRENGTH (NO PRIOR DEFORMATION OF THE AUSTENITE).
FIG. 15  ULTIMATE TENSILE STRENGTH AND 0.2 PERCENT YIELD STRENGTH FOR LONGITUDINAL AND TRANSVERSE SPECIMENS OF AUSTENITE AS A FUNCTION OF REDUCTION IN THICKNESS BY ROLLING.
FIG. 16 ULTIMATE TENSILE STRENGTH AND 0.2 PERCENT YIELD STRENGTH FOR LONGITUDINAL AND TRANSVERSE MARTENSITIC SPECIMENS AS A FUNCTION OF PRIOR DEFORMATION OF THE AUSTENITE BY ROLLING.
FIG. 17 ULTIMATE TENSILE STRENGTH AND 0.2 PERCENT YIELD STRENGTH FOR LONGITUDINAL AND TRANSVERSE MARTENSITIC SPECIMENS EXTRAPOLATED LINEARLY TO 100 PERCENT MARTENSITE AS A FUNCTION OF PRIOR DEFORMATION OF THE AUSTENITE BY ROLLING.
thickness of the austenite increased, the amount of martensite formed during refrigeration decreased. At the same time, however, the difference between the strength of deformed austenite and the corresponding martensite decreased so that the corrections by extrapolating to 100 percent martensite turned out to be rather small (Fig. 17). The values of the ultimate tensile strengths and 0.2 percent yield strengths shown in Fig. 17 are only a few percent higher than the corresponding values of Fig. 16 throughout the whole range of reduction in thickness. As in the case of the deformed austenitic specimens, the strength of the martensitic specimens in the transverse direction was higher after prior deformation of the austenite than in the longitudinal direction.

The ultimate tensile strength and 0.2 percent yield strength for deformed martensitic specimens as a function of the amount of reduction in thickness are shown in Fig. 18. Strengthening with increasing reduction in thickness was rapid at low reduction in thickness, levelled off at intermediate reductions and increased again at higher reductions. There seemed to be no consistent difference between the transverse and longitudinal values.

The ductility of the austenite decreased very substantially with a relatively small amount of reduction in thickness, but the transverse and longitudinal ductilities did not exhibit any consistent differences (Fig. 19). The ductility of martensite with previous deformation of the austenite remained practically constant
FIG. 18 ULTIMATE TENSILE STRENGTH AND 0.2 PERCENT YIELD STRENGTH OF MARTENSITE AS A FUNCTION OF DEFORMATION BY ROLLING.
FIG. 19 DUCTILITY OF AUSTENITE AS A FUNCTION OF DEFORMATION BY ROLLING.
throughout the whole range of reduction in thickness with no
significant difference between the transverse and longitudinal
directions (Fig. 20), while the ductility of deformed martensite
decreased slowly but steadily with increasing amount of reduction
in thickness, the elongation in the longitudinal direction being
slightly higher than in the transverse direction.

The dependence of 0.2 percent yield and ultimate tensile
strengths of austenitic and martensitic specimens on austenitic
grain size was determined by cold-rolling strips of austenite to
about 70 percent reduction in thickness, and annealing this rolled
stock at 800°, 900° and 1000° C for 30 minutes in a helium
atmosphere. This produced grains whose average linear intercepts
on a polished surface were 0.0227 mm, 0.0248 mm and 0.0332 mm,
respectively, whereas the austenite which had been annealed at
1000° C for 24 hours had an average linear intercept of 0.0905 mm.
These values were converted to average grain diameters by
multiplying by 1.65 (64). The 0.2 percent yield and ultimate tensile
strengths are plotted as a function of the average grain diameter to
the minus one half power in Fig. 21. The strength of austenite
increased very little by decreasing the grain size by a factor of
about 4, whereas the strength of the martensitic structure produced
by quenching the same austenitic structures increased by about 20
percent over the same range of grain sizes. The strengths of the
martensitic specimens were converted to 100 percent martensite by
FIG. 20 DUCTILITY OF MARTENSITE AS A FUNCTION OF DEFORMATION BY ROLLING.
FIG. 21 DEPENDENCE OF 0.2% YIELD STRENGTH AND ULTIMATE TENSILE STRENGTH OF AUSTENITIC AND MARTENSITIC (EXTRAPOLATED TO 100% MARTENSITE) SPECIMENS ON AUSTENITE GRAIN SIZE.
linear extrapolation. The volume fraction of martensite in these specimens was determined by point counting. The ductility of austenite and martensite showed no significant changes over the same range of grain sizes (Fig. 22).

C. Metallography

The equiaxed grains such as present in the well-annealed structure of Fig. 4, upon rolling at 300° C, were flattened in the transverse section (Fig. 23), elongated in the rolling plane section (Fig. 24), and elongated and narrowed in the longitudinal section (Fig. 25). The change in shape of individual grains was approximately the same as the overall change in shape of the rolled sheet, with the restriction, of course, that the continuity of the volume had to be preserved. Intersecting slip could be seen clearly on the longitudinal section, at an angle of approximately 45° to the rolling direction, while on the rolling plane section slip bands could be faintly distinguished only in the transverse direction. No slip bands could be seen in the transverse sections. The etching effects associated with the appearance of the slip bands were probably caused by the segregation of interstitial impurities (e.g. carbon) to regions of high strain, such as dislocation arrays on slip planes, during the rolling operations at 300° C. Some segregation may have also occurred at room temperature, but it was found that an austenitic specimen deformed at room temperature did not exhibit such pronounced etching effects.
FIG. 22  DUCTILITY OF AUSTENITE AND MARTENSITE AS A FUNCTION OF AUSTENITIC GRAIN SIZE.
Fig. 23. Transverse section of austenite with 76 percent reduction in thickness. Electrolytic polishing and etching. Magnification: 200X.

Fig. 24. Rolling-plane section of austenite with 76 percent reduction in thickness. Electrolytic polishing and etching. Magnification: 200X.
Fig. 25. Longitudinal section of austenite with 76 percent reduction in thickness. Electrolytic polishing and etching. Magnification: 200X.

Fig. 26. Martensite produced by quenching well-annealed austenite in liquid nitrogen. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 200X.
Martensite produced by quenching a well-annealed specimen of austenite in liquid nitrogen is shown in Fig. 26. This is to be compared with Figs. 27, 28 and 29 which are the martensitic specimens corresponding to Figs. 23, 24 and 25, respectively, after quenching in liquid nitrogen. At first glance, the martensitic structure appears to have been refined a great deal by the prior deformation. However, by quenching to an intermediate temperature where the austenite was only partially transformed and modifying the etching procedure to improve the contrast between austenite and martensite, it was found that the refinement of the martensitic structure was only apparent. Indeed, the size of some martensitic plates was considerably increased (Fig. 30). Two configurations of martensitic plates were seen in specimens with prior deformation of the austenite, namely long plates parallel to the rolling direction (Fig. 30) and a "chevron" configuration (Fig. 31) with the martensitic plates more or less perpendicular to the slip bands of the deformed austenite. Figure 31 also reveals that the slip bands did not always constitute impermeable barriers to martensitic plates since all the plates crossed several slip bands. However, most martensitic plates stopped before impinging upon a grain boundary which indicated that some slip bands were effective in stopping the growth of martensitic plates while others were not. When martensitic plates crossed slip bands a disturbance was left in the plates by the slip bands (Fig. 32) indicating that the lattice defects of the deformed austenite might
Fig. 27. Transverse section of martensite produced by quenching austenite with 76 percent reduction in thickness in liquid nitrogen. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 200X.

Fig. 28. Rolling plane section of martensite produced by quenching austenite with 76 percent reduction in thickness in liquid nitrogen. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 200X.
Fig. 29. Longitudinal section of martensite produced by quenching austenite with 76 percent reduction in thickness in liquid nitrogen. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 200X.

Fig. 30. Longitudinal section of specimen with 76 percent reduction in thickness, quenched to -47°C. Electrolytic polishing and 6 percent nital etch before quench, light etch after quench. Magnification: 500X.
Fig. 31. Longitudinal section of specimen with 76 percent reduction in thickness, quenched to -47°C. Electrolytic polishing and 6 percent nital etch before quench, light etch after quench. Magnification: 500X.

Fig. 32. Longitudinal section of specimen with 34 percent reduction in thickness, quenched to -47°C. Electrolytic polishing. Etchant: 6 percent nital. Magnification: 500X.
be inherited by the martensite.

Otte and Read\(^{(65)}\) have reported that twin structures could not be seen by optical microscopy in a 2.8 percent chromium, 1.5 percent carbon martensite with an \(M_s\) of about \(-35^\circ\) C, but that striations could be seen at a magnification of 10,000X by replication electron microscopy. In this investigation the fine structure could be seen in the martensitic plates when the specimens were etched in a more concentrated nital solution (12 percent) for 2 or 3 times the regular etching period and examined at 1500X magnification with an oil immersion lens (Fig. 33). Not all martensitic plates showed twins. This was not taken to signify the absence of twins but rather a dependence of the etching effect on the orientation of the twinning plane with respect to the surface under observation. Another effect observed in the martensitic structure with no prior deformation of the austenite is shown in Fig. 34. The pairs of parallel lines seen in the larger martensitic plate were probably regions of either twinning or slip to accommodate the small martensitic plates impinging upon the larger one.

The examination of martensitic structures after prior deformation of the austenite did not reveal any internal structures in the martensitic plates, except for the disturbances left by the slip bands of the parent austenite. Failure to observe any fine structure could not, however, be taken to signify that there was no such fine structure since the metallographic surfaces of the deformed specimens.
Fig. 33. Martensite produced by quenching austenite with no deformation in liquid nitrogen. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.

Fig. 34. Martensite produced by quenching austenite with no deformation in liquid nitrogen. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.
were too uneven to reveal any finer structures. Instead, these specimens had to be examined by transmission electron microscopy of thin foils.

Figure 35 shows the appearance of martensitic plates produced from well-annealed austenite after the quenched material had been subjected to a stress-relief anneal below the $A_s$ temperature, at 300° C, for 104 hours. Large numbers of etch pits could be distinguished in the martensitic plates and the inner appearance of the martensitic plates was much more uneven than the corresponding structure before this heat treatment. Apparently, the stress-relief anneal caused the yield point of the martensite to decrease and plastic flow took place to accommodate the highest stresses which were present.

The longitudinal sections of a martensitic specimen rolled at room temperature to a reduction in thickness of 32 percent exhibited severely distorted plates (Fig. 36) and rows of etch pits along slip bands (Fig. 37). All the deformation seemed to have taken place by slip and distortion of the plates. The difference in the appearance of these two micrographs and the stress-relieved martensite (Fig. 35) is due to the fact that in Figs. 36 and 37 the martensitic plates had to comply with the macroscopic change in shape of the rolled sheet, whereas in Fig. 35, deformation took place only where stress concentrations existed.
Fig. 35. Martensite produced by quenching austenite with no deformation in liquid nitrogen. Annealed for 104 hours at 300° C. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.

Fig. 36. Martensite produced by quenching austenite with no deformation in liquid nitrogen. Rolled to 32 percent reduction in thickness at room temperature. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.
Fig. 37. Martensite produced by quenching austenite with no deformation in liquid nitrogen. Rolled to 32 percent reduction in thickness at room temperature. Electrolytic polishing. Etchant: 12 percent nital. Magnification: 1500X.
D. X-ray Measurements

The effects of local strain and particle size were separated by measuring the breadths of the \(\{200\}\) and \(\{400\}\) reflections for austenite and the \(\{110\}\) and \(\{220\}\) reflections for the martensite. The profiles of the \(\{200\}\) austenite and \(\{110\}\) martensitic reflections were determined with chromium radiation, and those of the \(\{400\}\) austenite and \(\{220\}\) martensite with cobalt. Particle size and strain were separated by substituting the corresponding breadths in equation (5). A sample calculation is shown graphically in Fig. 38.

The solid line passes through the experimental points while the dotted lines show the upper and lower limits of slope, assuming an experimental scatter of \(\pm 5\) percent in determining the value of \(b\).

It is seen that the value of \(e\) which is calculated from the slope can have an error of about \(\pm 10\) percent, while the value of the particle size \(L\) which is calculated from the intercept on the ordinate (equal to \(1/L^2\)) can give rise to relatively large errors. One of the dotted lines in Fig. 38 has a negative intercept, giving an imaginary value of \(L\) which is meaningless. In several cases negative values were calculated for \(1/L^2\) and in other cases the computed values of \(L\) varied by such large amounts that they were considered to be unreliable and have, therefore, not been included in the data presented here. The values of the microstrains, however, have an estimated maximum error of \(\pm 10\) percent. The measured breadths could
FIG. 38 SEPARATION OF PARTICLE SIZE AND STRAIN FOR DEFORMED MARTENSITE WITH 56 PERCENT REDUCTION IN THICKNESS. DOTTED LINES SHOW MAGNITUDE OF POSSIBLE EXPERIMENTAL SCATTER.
not be exactly reproduced and a scatter of about \( \pm 5 \) percent could not be circumvented. There are several reasons which could explain this experimental scatter:

1. The material may have been inhomogeneously deformed. The breadth of the reflections would thus vary with the spot of the specimen being illuminated by the incident beam.

2. A large grain size combined with the fact that only very few grains are suitably oriented to diffract at a given Bragg angle may limit diffraction to very few grains, thus causing the measured breadth not to be truly representative of the whole structure.

3. With a given collimator the area of the specimen which is illuminated by the incident beam varies inversely with \( \sin \theta \). The area which is illuminated when the Bragg angle is set for a low-angle reflection is, therefore, larger than for a large angle. Different grains are illuminated in each case, and if the material is not homogeneously deformed a difference may result in the two orders of the same reflection.

4. The depth of penetration of the beam in the specimen is also a function of the angle and the same argument applies here.

5. The graphical separation of the \( K_{\alpha_1} \) and \( K_{\alpha_2} \) peaks may have also introduced an error since it is assumed for this separation that the intensity of the \( K_{\alpha_1} \) is twice that of the \( K_{\alpha_2} \).
6. Finally, the angular dependences of the effects of particle size and strain on line breadth were derived by assuming a model which is obviously not perfect. Also the analysis of line breadth assumes a Gaussian profile of all the different contributions to the intensity and this is only true with a certain degree of approximation.

The microstrains for deformed austenite, martensite with prior reduction of the austenite, and deformed martensite as a function of reduction in thickness are shown in Fig. 39. The increases in the microstrains which were observed were much larger than the estimated possible scatter of \( \pm 10 \) percent.

The particle size could be measured with some accuracy only in deformed austenitic specimens. After 76 percent reduction in thickness the measured particle size was about 1000 Å, decreasing to about 800 Å at 88 percent reductions. At reductions lower than 76 percent the particle size was too large to produce any appreciable line broadening.

An attempt was made to measure the probability of finding a stacking fault in deformed austenite. No line shift larger than the experimental accuracy of determining such a shift could be detected. This accuracy was about 0.03° (28), and hence a probability of finding one stacking fault in about 300 \{111\} planes or more could not be detected by this method. Thus, it was ascertained that if stacking faults were present in deformed austenite they had a very low density.
FIG. 39 AVERAGE MICROSTRAINS IN DEFORMED AUSTENITE, MARTENSITE WITH PRIOR DEFORMATION OF THE AUSTENITE, AND DEFORMED MARTENSITE.
The microstrains in martensite with no prior deformation and with 88 percent prior reduction in thickness of the austenite were also determined after isothermal anneals at 300° C. After about 10 hours at this temperature the microstrains dropped to approximately 70 percent of their original values, while longer anneals caused only minor changes (Fig. 40). The microstrain of martensite with 88 percent prior reduction in thickness of the austenite decreased somewhat more rapidly than that of martensite with no prior deformation of the austenite.

E. Electron Microscopy

Martensite with no prior deformation of the austenite, austenite with 34 percent reduction in thickness, and martensite with 34 percent prior reduction of the austenite were examined by transmission electron microscopy of thin foils. Specimens with more reduction gave poor resolution and, consequently, the observations reported here are confined to specimens with 34 percent reduction in thickness. In martensitic specimens with no prior deformation of the austenite, a fine twin structure similar to that reported by Nishiyama and Shimizu(66,67), Kelly and Nutting(68), Warlimont(69) and Cohen(70) was observed (Fig. 41). In some cases the twin spacing was found to be denser at the center of the martensitic plate along the "mid-rib" (Fig. 42), as previously reported by Warlimont(69) for similar alloys.
**Fig. 40** Effect of annealing at 300°C on the microstrains and hardness of martensite with 0% and 88% prior reduction of the austenite.

- X 0% Reduction in thickness
- O 88% Reduction in thickness
- △ Hardness (0% Reduction)
- Δ Hardness (88% Reduction)
Fig. 41. Electron transmission micrograph of martensite with no prior deformation of the austenite. Magnification: 80,000X.

Fig. 42. Electron transmission micrograph of martensite with no prior deformation of the austenite. Magnification: 30,000X.
However, in general, only a small portion of a large martensitic plate could be seen (Fig. 41) and it was not possible to determine whether this configuration was present in all plates.

Examination of deformed austenite revealed a cellular structure consisting of areas of high dislocation density completely enclosing areas of lesser dislocation density. The areas of high dislocation density appeared as tangles (Figs. 43 and 45). The selected area diffraction patterns (Figs. 44 and 46) indicated that large regions containing several such tangles were single crystals with very slight misorientation between adjacent regions. It is, therefore, likely that the dislocation tangles could be regarded as subgrain boundaries separating the subgrains which themselves contained relatively low dislocation densities.

Comparing the measured particle size of deformed austenite (about 1000 Å) with Figs. 43 and 45 revealed that the size of the coherently reflecting domains corresponded to the size of the cells seen in the areas of lesser dislocation density, while the size of the subgrains was about one order of magnitude larger.

Most of the diffraction patterns indicated that a \{110\} of the austenite plane was perpendicular to the beam, i.e. in the rolling plane (Figs. 44 and 46).

When the austenite has been previously deformed, most of the subsequent martensite appeared as large plates with only some vestiges of striations (Fig. 47) or with no indication at all of any striations
Fig. 43. Electron transmission micrograph of austenite with 34 percent reduction in thickness. Magnification: 40,000X.

Fig. 44. Diffraction pattern of Fig. 43.
Fig. 45. Electron transmission micrograph of austenite with 34 percent reduction in thickness. Magnification: 40,000X.

Fig. 46. Diffraction pattern of Fig. 45.
Fig. 47. Electron transmission micrograph of martensite with 34 percent prior reduction in thickness of the austenite. Magnification: 40,000X.

Fig. 48. Diffraction pattern of area enclosed in circle in Fig. 47.
(Fig. 49). Selected area diffraction from such martensitic regions of ausformed specimens produced patterns which were those corresponding to single crystals of the body-centered cubic martensite (Figs. 48 and 50). Some areas of the ausformed specimens had the appearance of Fig. 51 where the striations suggested the presence of twins. However, the selected area diffraction patterns from such areas could not be interpreted because of the large number of spots and double reflections which were present. The structures shown in Figs. 47 and 49 were encountered much more often than that of Fig. 51. Figure 49 shows a similar area within a martensitic plate at higher magnification. The selected-area diffraction pattern (Fig. 50) was again that of a single crystal. Some areas, however, had the appearance of Fig. 51, where parallel lines suggested the presence of twins. However, the structures of Figs. 47 and 49 were more commonly encountered. The diffraction patterns corresponding to areas showing features like those of Fig. 51 could not be interpreted because of the large number of spots and double reflections which were present.
Fig. 49. Electron transmission micrograph of martensite with 34 percent prior reduction in thickness. Magnification: 80,000X.

Fig. 50. Diffraction pattern of Fig. 49.
Fig. 51. Electron transmission micrograph of martensite with 34 percent prior reduction in thickness of the austenite.
Magnification: 40,000X.
V. DISCUSSION OF RESULTS

A. Anisotropy of Tensile Properties

Both rolled austenite and ausformed martensite exhibited anisotropy in their tensile properties, the yield and ultimate tensile strengths being generally higher in the transverse than in the longitudinal direction (Figs. 15, 16 and 17). The data for deformed martensite (Fig. 18) showed a considerable amount of scatter, and no such consistent trend was visible.

This kind of anisotropic behavior in cold-rolled face-centered cubic metals has been observed in iron-nickel alloys (71), silver (72), aluminum (73, 74) and bronze (73). A similar behavior has also been reported for cold-rolled low-carbon steel (75).

A simple explanation for this anisotropic behavior of the cold-rolled austenite may be provided by considering the principal cold-rolling texture which is known to be of the type (110) [112] for most face-centered cubic metals, including the 31.9 percent iron-nickel alloy used in this investigation (76, 77).

The resolved shear stress acting in a slip direction is given by

\[ \tau = \sigma \cos \theta \cos \lambda \]  \hspace{1cm} (10)

where \( \sigma \) is the applied uniaxial stress, \( \lambda \) the angle between the direction of the tensile axis and the slip direction, and \( \theta \) the angle between the tensile axis and the normal to the slip plane (78).
If several possible slip systems are present, slip will take place only on those for which \( \cos \theta \cos \lambda \) is a maximum. Assuming that the rolled sheet has a perfect \([\bar{1}12]\) \([\bar{1}12]\) rolling texture, it is noted from Table IV that if a tensile stress is applied along the rolling direction \([\bar{1}12]\), \( \cos \theta \cos \lambda \) has the maximum value of 0.408 for only two slip systems, namely the \([\bar{1}01]\) \((\bar{1}11)\) and \([01\bar{1}]\) \((\bar{1}11)\), whereas if a tensile stress is applied in the transverse direction \([\bar{1}1\bar{1}]\) there are six slip systems for which \( \cos \theta \cos \lambda \) has the maximum value of 0.271, namely the \([\bar{1}10]\) \((\bar{1}11)\), \([\bar{1}10]\) \((\bar{1}1\bar{1})\), \([01\bar{1}]\) \((\bar{1}11)\), \([01\bar{1}]\) \((\bar{1}1\bar{1})\), \([\bar{1}0\bar{1}]\) \((\bar{1}1\bar{1})\) and \([\bar{1}0\bar{1}]\) \((\bar{1}1\bar{1})\) systems. Hence, only two slip systems can operate if the stress is applied in the longitudinal direction, whereas six systems operate if it is applied in the transverse direction.

The higher resolved shear stress causing slip when the tensile stress is applied in the \([\bar{1}12]\) longitudinal direction can account for the lower yield strength observed in that direction. On the other hand, the presence of six slip systems when the tensile axis is the \([\bar{1}1\bar{1}]\) against only two when it is the \([\bar{1}12]\) results in quite different work-hardening characteristics between the two directions. The presence of several intersecting slip systems provides more obstacles to slip and may raise the yield strength as well as the ultimate tensile strength.

Another possible explanation of the observed anisotropy may lie in a difference of the effective grain size along various slip
TABLE IV

\[ \cos \theta \cos \varphi \] for various slip systems in a face-centered cubic crystal

with a (110) [\{112\}] cold-rolling texture. Tensile axes considered parallel to [\{112\}] and [\{111\}].

<table>
<thead>
<tr>
<th>Slip Direction</th>
<th>{110}</th>
<th>{110}</th>
<th>{101}</th>
<th>{01}</th>
<th>{011}</th>
<th>{101}</th>
</tr>
</thead>
<tbody>
<tr>
<td>\theta {112}</td>
<td>54.74°</td>
<td>90°</td>
<td>30°</td>
<td>73.22°</td>
<td>30°</td>
<td>73.22°</td>
</tr>
<tr>
<td>{111}</td>
<td>35.26°</td>
<td>90°</td>
<td>90°</td>
<td>35.26°</td>
<td>90°</td>
<td>35.26°</td>
</tr>
<tr>
<td>Slip Plane</td>
<td>{111} {111} {111} {111} {111} {111} {111} {111}</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>{112} tensile axis ( \lambda )</td>
<td>61.87° 61.87° 19.47° 90° 61.87° 90° 61.87° 19.47°</td>
<td>90° 61.87° 61.87° 19.47°</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \cos \theta \cos \varphi )</td>
<td>0.271 0.271 0 0 0.408 0 0.136 0.273</td>
<td>0 0.408 0.136 0.273</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>{111} tensile axis ( \lambda )</td>
<td>70.53° 70.53° 70.53° 70.53° 70.53° 70.53° 70.53° 70.53°</td>
<td>70.53° 70.53° 70.53° 70.53°</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>( \cos \theta \cos \varphi )</td>
<td>0.271 0.271 0 0 0 0.271 0.271 0 0 0.271 0.271</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
directions in the deformed material. With increasing deformation, the grains become more anisotropic in their dimensions with the result that the anisotropy of the tensile properties should become more pronounced. However, the difference between transverse and longitudinal strengths was only a few percent and had no tendency to increase with reduction (Figs. 15, 16 and 17). For this reason the explanation based on the difference in the resolved shear stresses required to cause slip and on the work-hardening characteristics in the transverse and longitudinal directions is more likely to be the correct one.

It should be noted that most specimens of deformed austenite which were examined by transmission electron microscopy revealed that the rolling plane was indeed a [110] plane, as can be seen, for example, in the electron diffraction patterns of Figs. 44 and 46.

The deformed martensite also exhibited anisotropy, but relatively little is known about the slip and work-hardening mechanisms in body-centered cubic martensite, so that a simple analysis cannot be provided at the present time.

B. Microstructural Aspects

Both light microscopy and transmission electron microscopy revealed several important differences between ordinary martensite and ausformed martensite. The martensitic plates produced by quenching annealed austenite were randomly distributed (Fig. 26) whereas those produced by
quenching rolled austenite had their long axes preferentially oriented either along the rolling direction (Fig. 30) or in a chevron pattern at an angle to the rolling direction (Figs. 31 and 32). The plates which were aligned with their long axes along the rolling direction of the austenite (Fig. 30) were, in general, much larger than the plates which were seen when the austenite had not been previously deformed (Fig. 26). At high magnification and after heavy etching, the martensitic plates produced from annealed austenite contained fine striations (Fig. 33). Transmission electron microscopy of such specimens revealed that these striations were fine twins with a spacing of roughly 100 Å (Figs. 41 and 42). Ausformed martensitic plates, on the other hand, did not exhibit such striations (Figs. 47 and 49). These martensitic plates were seen to cross the slip bands of the deformed austenite, and wherever this occurred a disturbed area was left in the martensitic plate. Some etch pits could be observed in the martensitic plates where the slip bands intersected them (Fig. 32). It would be natural to conclude from these observations that lattice defects were introduced into the martensitic plates by working the parent austenite matrix.

Transmission electron microscopy and selected-area electron diffraction further revealed that most of the ausformed martensitic plates had no fine twins (Figs. 47 and 49). The diffraction patterns from such areas (Figs. 48 and 50) indicated that these martensitic plates were single crystals. It has been pointed out by Richman (79):
that if the electron beam is normal to a \{115\} or \{221\} plane of the thin foil the presence of \{112\} twins in the martensite produces diffraction patterns consisting of a superposition of matrix and twin patterns which look exactly like the diffraction patterns corresponding to single crystal orientations with the electron beam normal to a \{111\} and \{100\} plane, respectively. Richman, however, also indicated that when the beam is normal to a \{115\} or \{221\} plane the twins are so oriented as to give a good contrast consisting of alternating dark and light striations. The selected-area diffraction pattern of Fig. 48 was interpreted as that of a single crystal of martensite in view of the fact that no such striations were visible in the corresponding electron transmission micrograph (Fig. 47). Within the martensitic plates a structure resembling the dislocation structure of the deformed austenite was seen. However, these structures could not be resolved as well as the dislocation structure of the deformed austenite. The reason is thought to be due to the fact that the interface of an advancing martensitic plate consists of a dislocation network. When these dislocations intersect the dislocations present in the deformed austenite, a large number of jogs are formed with the result that the dislocations left behind are so kinked that they give a fuzzy contrast. The alternative possibility, i.e. that the advancing martensitic plate sweeps away the dislocation structure of the austenite and leaves a perfect lattice structure behind is quite unlikely on account of the evidence presented here; in any case, it is
difficult to imagine how this could happen. It should be further noted that the presence of these contrasts within the martensitic plates is an additional indication that fine twins are not present, as discussed by Kelly and Nutting (68), since the contrast caused by twins would be sufficient to effectively mask the contrast due to other microstructural features.

From the observations made by transmission electron microscopy on martensite with and without prior deformation of the austenite, it was concluded that the martensitic plates produced by quenching annealed austenite were, with almost no exceptions, internally twinned, whereas the martensitic plates produced by quenching deformed austenite were, in general, single crystals containing imperfections. Occasionally, structures resembling fine internal twins of martensite were seen in the ausformed martensitic specimens (Fig. 51). However, these were the exception rather than the rule. In addition, these structures resembling fine twins extended only over narrow areas. The selected-area diffraction patterns from these regions included a large number of spots from surrounding areas as well as from double reflections. This resulted in such complicated patterns that no identification of the crystallographic structure could be made. However, one can eliminate, here, the fine internal twins of the martensite as an important factor contributing to the strengthening of ausformed martensite although some twinning may still be present in ausformed martensite.
The deformed martensitic material, on the other hand, presented quite a different situation. Figures 36 and 37 show examples of the deformation structures in martensite. These micrographs indicate that in addition to closely spaced slip markings (Fig. 36), the martensitic plates were also severely distorted as a whole (Fig. 37). The resulting structure has a higher strength than the ausformed martensite. This is purely a work-hardening phenomenon. It is unfortunate that so little is known about work hardening in body-centered cubic metals that the interpretation given in this subsection cannot be carried any further at this time. It should be noted that the dislocations which are present in the deformed austenite have Burgers' vectors which are characteristic of the face-centered cubic lattice. When a martensitic plate sweeps through an austenitic area containing dislocations, the lattice defects which are inherited by the martensitic plate need not have the same characteristics as the dislocations which are introduced into a body-centered cubic lattice by deformation. They could, for example, have Burgers' vectors which are not possible for dislocations introduced into a body-centered cubic lattice by cold work. It is, therefore, not astonishing, that martensitic plates with inherited lattice defects should have properties quite different from those of martensitic plates with lattice defects introduced by cold work.
In a martensitic structure produced from annealed austenite, the growth of martensitic plates was generally stopped by impingement upon grain boundaries or other martensitic plates. In the deformed austenitic matrix, on the other hand, in addition to grain boundaries and other martensitic plates, some slip bands were also found to act as barriers to advancing martensitic plates, i.e. some martensitic plates stopped at slip bands after having crossed a number of them. If the austenite-martensite interface is assumed to be a dislocation network, the growth of a martensitic plate proceeds by this network of dislocations advancing through the austenite. In the case of a deformed austenitic matrix, the dislocation interface has to intersect a large number of dislocations, thus acquiring jogs. Since a dislocation containing jogs leaves a trail of interstitials or vacancies behind, more driving force is required for the continuation of the interface movement. It is likely that after having crossed a certain number of dislocations, the interface dislocations acquire so many jogs that the available driving force is no longer sufficient to drive the interface, and the growth of the martensitic plate comes to a stop.

Most of the investigators cited in the Literature Survey (Chapter II) who studied the strengthening of martensite by prior deformation of austenite as a function of deformation temperature reached the conclusion that lower deformation temperatures produced a stronger martensite. This behavior tends to confirm the hypothesis
that at least part of the strengthening of the martensite is caused by lattice defects being inherited by the martensite from the parent deformed austenite. The work hardening of a face-centered cubic lattice is indeed very strongly dependent on temperature as has been shown by Malyshev et al\(^{(80)}\). These investigators observed that when an austenitic steel is plastically deformed the deformation is less and less localized in the vicinity of grain boundaries as the temperature of deformation is decreased, and that, at the same time, the subgrain size decreases and the dislocation density and the number of slip bands increase. In other words, deformation at lower temperatures takes place by the introduction into the lattice of a larger number of defects than at the higher temperatures. If the strengthening of ausformed martensite is affected by inherited defects it would, therefore, be quite natural to expect that lower temperatures of deformation of the metastable austenite would introduce more defects into the subsequent martensite and would cause more strengthening. However, the results of the strengthening of ausformed martensite as a function of working temperature in steels reported by many investigators is obscured by the fact that in general no attempts were made to determine the extent of decomposition of the metastable austenite into martensite, bainite or pearlite before quenching to produce martensite. The deformation-temperature dependence of strengthening by ausforming is, therefore, not presented here as decisive evidence, but simply because it is
consistent with one of the proposed mechanisms of strengthening.

The second mechanism which may contribute to the strengthening of martensite by ausforming is the decrease in the distance between barriers to slip along the slip directions. The martensitic interfaces may be just as effective as austenitic grain boundaries in strengthening by this mechanism. Figure 21 shows the considerable strengthening takes place by reducing the martensitic plate size since the strengthening of the martensitic specimens was much more sensitive to reduced austenitic grain size than the austenitic specimens. A quantitative discussion of this strengthening mechanism is not possible at this time since relatively little is known about the slip systems in the body-centered cubic martensite, not to mention the fact that a measurement of the average linear intercept of microstructural features like those shown in Figs. 27 or 29 would be impossible. To circumvent this difficulty and to provide a more quantitative basis for the separation of the two contributions to strengthening (i.e. inherited defects and decreased effective grain size) an experiment has been devised whereby cold work will be introduced into the austenite by explosive loading. Since no appreciable change in shape of the grains will result from this method of cold-working, any observed increase in strength should be caused by inherited lattice defects. At the time of this writing, however, the results were not available. On the other hand, it is interesting to note that the ductility of martensitic specimens remains
practically constant while the austenitic grain size, and hence the martensitic plate size, is changed by a factor of about 4 (Fig. 22). This behavior of the ductility as a function of martensitic plate size is very similar to the behavior of the ductility of ausformed martensite where it is also independent of the amount of prior reduction in thickness of the austenite. The introduction of defects into martensite by strain-hardening reduces the ductility considerably (Fig. 22) and hence, the ductility behavior observed for ausformed martensite suggests that a reduction of the effective distance between slip barriers must be at least a contributory factor in the strengthening process.

C. Results of X-ray Line-Broadening Measurements

The accuracy of the x-ray measurements and the fact that the contribution to line breadth of small particle size was effectively masked by the much larger contribution of microstrains were described in Chapter III. In order to make accurate particle-size measurements, the strains have to be almost completely relieved and this was found not to be possible even after very long anneals below the \( A_s \) temperature. One way of making particle-size measurements would be the method of Arbusov et al.\(^{(31)}\) whereby martensitic plates are electrolytically extracted from their surrounding matrix, thus completely relieving the strains. Accurate x-ray measurements of the particle size can then be made. Attempts at reproducing Arbusov's
technique of electrolytic extraction were not successful, and more experimentation is required.

The microstrains in a crystal lattice may be divided into three types:

1. Strains which are due to dislocations and other lattice defects and extend only over a few interatomic distances after which they decrease very rapidly.

2. Strains which are appreciable over dimensions comparable to the grain size of the material. For example, the situation of a martensitic plate impinging upon another martensitic plate creates variable strains which extend over regions of both plates.

3. Dilatational strains: The martensitic plates because of their concomitant volume expansion are subjected to a compressive hydrostatic stress which causes a uniform compressive strain.

Most of the line broadening in martensite is thought to be caused by strains of type 2 above, whereas strains of type 1 are expected to contribute only a very small amount of broadening. Strains of type 3 cause a shift of the x-ray diffraction lines, but in view of the extremely broad diffraction profiles caused by the strains small shifts of the lines could not be measured.

In this investigation, the microstrains in the ausformed martensite increased substantially with the amount of prior deformation of the austenite (Fig. 39) while prolonged anneals at 300° C, just
below the $A_s$ temperature, relieved part of the microstrains without affecting the hardness (Fig. 40).

According to Kurdjumov\(^{82,83}\), the broadening of x-ray lines due to strains is caused by non-uniform elastic deformation of the martensitic plates, i.e. by strains of type 2. Upon removing the constraints around the martensitic plates by electrolytically extracting them Kurdjumov was able to show that the strain broadening completely disappeared. The same result may be achieved by annealing the specimens, although in this case the strains are not completely relieved. The mechanism of strain relief in the martensitic plates appears to be one of plastic flow. At room temperature, the stresses on the martensitic plates cause a certain amount of strain and an equilibrium is established. When the temperature is raised the flow stress is lowered and the martensite is not able to withstand the same maximum stress. Some plastic flow occurs in the martensite in order to accommodate the highest stresses. When the specimen is again brought to room temperature, the strain is lower than before.

Figure 35 which shows the appearance of martensitic plates after annealing at 300°C for 104 hours indicates that such plastic flow does indeed occur. It can be concluded, therefore, that the level of microstrains present in martensite does not contribute to the strength of the martensite but on the contrary is an indication of the maximum possible elastic deformation of the martensite. Thus, the observed lattice strain is a measure rather than a cause of the elastic limit.
and yield strength of the martensite.

The inherited lattice defects of the martensite may cause part of the increase in the yield strength. This is confirmed by microhardness measurements on individual martensitic plates (Fig. 4). On the other hand, it is also quite possible that a reduced distance between barriers to slip may require higher stress concentrations to produce slip. Therefore, it is likely that the increased microstrains in ausformed martensite are caused by both of these mechanisms simultaneously.

D. General Discussion

Figure 52 shows the average of longitudinal and transverse yield strengths of deformed austenite, martensite with prior reduction of the austenite, and deformed martensite. The data for martensite with prior reduction of the austenite were corrected for retained austenite by linear extrapolation to 100 percent martensite. The results for deformed martensite were assumed to represent the values for 100 percent martensite since the amount of retained austenite originally present was only a few percent and most of it transformed to martensite during the deformation process. The point corresponding to 0 percent reduction is, of course, identical for both kinds of martensite. These data were taken from Figs. 15, 17 and 18 and plotted on the same graph in the manner described in order to summarize how the strength varied with deformation in the three different situations.
FIG. 52 AVERAGE OF LONGITUDINAL AND TRANSVERSE 0.2% YIELD STRENGTHS OF DEFORMED AUSTENITE, MARTENSITE WITH PRIOR DEFORMATION OF THE AUSTENITE AND DEFORMED MARTENSITE (EXTRAPOLATED LINEARLY TO 100% MARTENSITE).
The increase in the yield strength of martensite by prior deformation of the austenite was relatively modest in magnitude (about 15,000 pounds per square inch after 88 percent prior reduction in thickness of the austenite), but was quite appreciable as a fraction of the strength of the regular martensite (about 35 percent). On the other hand, when martensite was deformed over about the same range of reduction, the increase in yield strength was approximately 100 percent. The yield strength of the austenite increased by about 170 percent after 88 percent reduction in thickness. At 0 percent reduction, the strength increment on transforming from austenite to martensite was comparable to the original strength of the austenite, whereas at higher reductions this strength increment was only a small fraction of the strength of austenite.

The increase of about 35 percent in the strength of ausformed martensite over that of regular martensite found in the low-carbon iron-nickel alloy is comparable to the increase in strength obtained with commercial steels containing carbon and alloying elements. However, the absolute strength is very low when only a small amount of carbon is present. On the other hand, when the amount of strengthening is expressed in terms of pounds per square inch per percent of prior reduction in thickness of the austenite a 35 percent increase for the low-carbon alloy corresponds to about 300 pounds per square inch for each percent reduction, as compared to 700 and 1300 pounds per square inch for each percent reduction for a 4340 and 8611 steel, respectively.\(^{(84)}\)
The microhardness of deformed austenite, ausformed martensite and deformed martensite (Fig. 10) exhibited a somewhat different situation. The hardness increment on going from austenite to martensite was large and persisted with increasing deformation. The microindents caused severe local deformation and the microhardness was, consequently, more representative of the ultimate tensile strength than of the yield strength. Martensite work-hardens much more rapidly than austenite (Fig. 12). Hence, the stress required to cause severe deformation may be considerably higher for martensite than for austenite while such large differences need not appear in the yield strengths (Fig. 52).

The greater ductility of ausformed martensite already reported by several researchers and confirmed in this investigation is perhaps as important as the increase in strength. There was no decrease in the ductility of ausformed martensite even after 88 percent prior reduction in thickness of the austenite, in contrast with a substantial lowering of the ductility of deformed martensite (Fig. 20) and deformed austenite (Fig. 19). An improved ductility combined with high strength promises a wide field of applications for ausforming since in conventionally heat treated high strength materials the ductility can be improved only at the cost of a considerable loss of strength. The fundamental mechanism which controls the ductility and elongation to rupture is not well understood at present but the similarity between the ductility of ausformed martensite as a function
of the amount of prior deformation of the austenite (Fig. 20) and the ductility of martensite as a function of prior austenitic grain size (Fig. 22) is quite striking.

The anisotropy of rolled austenite and of the subsequently produced martensite was found to be of minor importance. However, in view of the fact that the increase in strength by ausforming was not very different in the rolling and transverse directions, anisotropy can be eliminated as a fundamental consideration in ausforming.

A complete elucidation of the strengthening mechanisms in ausforming is important not only because it sheds some light on the results reported here but especially because some of the conclusions may apply to other materials as well. With the fine internal twins and microstrains of martensite eliminated as important factors contributing to higher strength, the two mechanisms that remain possible are the inherited defects of martensite and a reduced distance between barriers to slip. The increased microhardness of the martensitic plates with prior reduction of the austenite (Fig. 10) definitely shows that the strength of the martensite is increased regardless of martensitic plate size, while the variation of yield strength with austenitic grain size and, hence, with martensitic plate size (Fig. 21) demonstrates that a decrease in the distance between barriers to slip may be just as effective as inherited defects in increasing the strength. Because of the small amount of carbon (0.017 percent) present in the experimental material in this investigation, it can be concluded that these
strengthening mechanisms operated quite independently of carbon. However, the tremendous increment in strength caused by the presence of carbon in any martensite is well known, and in particular Zrackay and Justusson have reported that the difference in yield strength between conventionally produced martensite and ausformed martensite with 91 percent prior reduction of the austenite was approximately constant in the range of 0.3 to 0.6 percent carbon, although the absolute strength increased steadily with carbon. The strength increments they reported were roughly 30 percent of the strength of the corresponding conventional martensites.

Some uncertainty remains regarding the most appropriate way of expressing the amount of strengthening by ausforming. If the strength increment caused by ausforming is expressed as a fraction of the strength of regular martensite the results of the present work show that the strength of ausformed martensite increases about 35 percent with a carbon content of 0.017 percent. On the other hand, the data of Zrackay and Justusson indicate that the strength increment in steels with 0.3 to 0.6 carbon is comparable to the 35 percent increase observed with low carbon. However, if the strength increments are expressed in terms of pounds per square inch for each percent of prior deformation of the austenite then there is a strong dependence on the carbon content. As an illustration, if a low carbon martensite has, for example, a yield strength of 70,000 pounds per square inch while a medium carbon martensite has a yield strength of 200,000 pounds
per square inch a 35 percent increase of the yield strengths of both martensites by ausforming with 90 percent prior reduction will result in the two materials having yield strengths of 94,500 and 270,000 pounds per square inch, respectively, while the corresponding increases of the yield strength expressed in pounds per square inch for each percent of prior reduction will be about 270 and 780. This apparent difference is due to the magnitude of the strength of the regular martensite which is strongly dependent on carbon. To avoid confusion, the strength of regular martensite should be included in any data on the strength of ausformed materials.

No matter how the strength increment of martensite by ausforming is expressed it is quite clear that some of the strengthening is independent of carbon since even an alloy with only 0.017 percent carbon shows an appreciable amount of strengthening. It appears that the effect of the presence of carbon can be considered as a superimposition of a large increment of strength on the more modest strength increments already caused by the microstructural changes in the martensite (i.e. inherited lattice defects and a reduced average distance between barriers to slip). The presence of inherited lattice defects in ausformed martensite becomes a question of special importance if carbon is present as they may provide nucleation sites for very dense and finely distributed dispersions of carbides.
CONCLUSIONS

1. The strength of martensite in low-carbon iron-nickel alloys increases with prior deformation of the austenite, but this strengthening is smaller than the prior work-hardening of the austenite. Moreover, this method of increasing the strength of martensite is not as effective as deformation of the martensite after transformation.

2. The ductility of ausformed martensite remains almost constant up to 88 percent prior reduction in thickness of the austenite. On the other hand, the ductility of deformed austenite and of cold-worked martensite undergo an appreciable decrease over the same range of reduction.

3. Rolled austenite and ausformed martensite are slightly anisotropic in strength, with higher strength being found in the transverse direction than in the longitudinal direction. This behavior is explained by the nature of the rolling texture, at least in the case of austenite. The ductility, however, is substantially the same in both directions.

4. X-ray line broadening measurements show that the microstrains in martensite increase with prior deformation of the austenite. Strain relieving anneals decrease the microstrains considerably while the hardness remains unchanged, thus indicating that the microstrains are a measure of the yield strength of the martensite rather than a cause of the strength.
5. No accurate particle-size measurements of the martensite can be made since the particle-size broadening is effectively masked by the much larger microstrain broadening.

6. Light and transmission electron microscopy provide evidence indicating that the lattice defects of the deformed austenite matrix are inherited by the martensite, and may thus cause at least part of the strengthening.

7. The increase of the microhardness of martensitic plates with prior reduction of the austenite confirms that inherited lattice defects strengthen the martensite.

8. A reduction in the effective distance between barriers to slip can also cause a substantial strengthening. This was demonstrated by producing smaller martensitic plate sizes from austenite with finer grains.

9. Two mechanisms are proposed for the strengthening of low-carbon ausformed martensite:

   a. Martensite inherits the lattice defects of the rolled austenite.

   b. A reduced distance between interfacial barriers to slip strengthens the material.
SUGGESTIONS FOR FURTHER WORK

1. Experiments whereby austenite is strain-hardened by explosive shock loading without distortion of the grains are required to separate quantitatively the two strengthening mechanisms which have been proposed for low-carbon martensite.

2. A better understanding of the work-hardening characteristics of body-centered cubic crystals is required for an analysis of the anisotropy exhibited by ausformed martensite.

3. Further transmission electron microscopy is necessary on ausformed martensite with lower prior reductions of the austenite for elucidating the nature of the lattice defects introduced into the martensite by ausforming.

4. No attempt was made in this investigation to explain the improved ductility of ausformed martensite. Improved ductility is as important as increased strength for certain applications. A better understanding of this behavior would be desirable.

5. Solution and precipitation hardening of ausformed martensite by the addition of carbon and alloying elements should be investigated, inasmuch as much higher strength levels are possible with commercial materials containing these elements.
6. The interaction of carbon with lattice defects in martensite should be investigated as this is thought to be a very important factor in the strengthening of steel.

7. Accurate x-ray measurements of strains and particle sizes in martensite would be made possible by electrolytically extracting martensitic plates from their surrounding matrix. Further experimentation is required for developing this technique.

8. The reason why the ausformed martensite has no fine internal twins and whether this is true for other alloys as well should be investigated within the general framework of the morphology of martensite.

9. Since martensite can be much more effectively strengthened by cold working than by ausforming, the possibility of strain-hardening martensite without extensive deformation (e.g. by explosive shock loading) should be investigated as a practical strengthening process.
BIOGRAPHICAL NOTE

The author was born in Istanbul, Turkey in 1931. He graduated from Robert Academy in Istanbul in 1946, attended Robert College in Istanbul and subsequently received the Matriculation Certificate of the University of London. He then served in the Turkish Army and was discharged in 1953 with the rank of lieutenant. In 1957, he came to the United States and enrolled as a graduate student at Rice Institute in Houston, Texas, where he received the Master of Science Degree in 1959. This thesis was entitled "Stacking Faults in Platinum."

In 1959, he entered the Graduate School at Massachusetts Institute of Technology as a candidate for the Doctor of Science Degree and became a Research Assistant in the Department of Metallurgy.
BIBLIOGRAPHY

1. G. Wassermann, Archiv für Eisenhüttenwesen, 6 (1932-33) 347.


5. R. F. Harvey, Metal Treating, 6, No. 3 (1955) 6.


15. E. N. Sokolkov, Physics of Metals and Metallography, 7, No. 3 (1959) 60.


24. V. F. Zackay, W. M. Justusson and D. J. Schmatz, Metal Progress, 80, No. 3 (1961) 68.


27. Y. M. Potak, Y. F. Ozrhekhovsky, L. M. Pevzmer, I. N. Roshina
    and N. N. Yermakov, Metallovedeniye i Termischeksaya Obrabotka
    Metallov, No. 5 (May 1961) 2.

28. Y. N. Sokolkov, S. N. Petrova and N. F. Chuprakova,
    Metallovedeniye i Termischeksaya Obrabotka Metallov, No. 5
    (May 1961) 12.


30. V. F. Zackay and W. M. Justusson, British Iron and Steel Inst.,
    Harrogate Conference (May 23 and 24, 1962).

31. Aeroneutronic Division of Ford Motor Company, Research on

32. I. Tamura, H. Yoshimura, M. Ibaraki and M. Tagaya, Memoirs of the
    Institute of Scientific and Industrial Research, Osaka University,

33. P. Winchell, Ph.D. Thesis, Massachusetts Institute of Technology,


36. A. Arrott, Ferromagnetic Analysis in Metallurgy, Scientific
    Laboratory, Ford Motor Co., Dearborn, Mich.


42. R. Castaing, Revue de Metallurgie, 52 (1955) 669.


No. 2342 (February 1948).

52. B. L. Averbach and M. Cohen, Trans. AIME, 176 (1948) 401.


60. F. E. Werner, B. L. Averbach and M. Cohen, Trans. ASM, 49 (1957) 823.


64. B. S. Lement, B. L. Averbach and M. Cohen, Trans. ASM, 46 (1954) 851.


72. E. Schmid and G. Wassermann, Metallwirtshaft, 10 (1931) 409.

73. H. Unckel, Z. Metallkunde, 31 (1939) 104.


