Effect of Purity on Reliability
Characteristics of High-Strength Steel
First Interim Technical Report

Air Force Materials Laboratory
Contract F33615-75-C-5137

by
H. M. Reichhold, J. G. Bassett,
S. R. Novak, and L. F. Porter

U. S. Steel Corporation
Research Laboratory
Monroeville, Pennsylvania 15146

November 15, 1975

Approved for Public Release;
Distribution Unlimited
### GENERAL DOCUMENT SUMMARY SHEET

<table>
<thead>
<tr>
<th>1. ACCESS NUMBER</th>
<th>2. COMPONENT/PART NAME PER GOVERNMENT SUBJECT THESAURUS</th>
</tr>
</thead>
<tbody>
<tr>
<td>E138-1618</td>
<td>Materials, Steel, Forged, Molded or Cast, Plate</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>3. APPLICATION</th>
<th>4. NOTIFIED</th>
<th>5. DOCUMENT ISSUE (Month/Year)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Engineering</td>
<td></td>
<td>November 1975</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>6. ORIGINATOR'S DOCUMENT TITLE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Effect of Purity on Reliability Characteristics of High-Strength Steel, First Interim Report</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>7. ORIGINATOR'S DOCUMENT NUMBER</th>
<th>8. ORIGINATOR'S PART NAME/IDENTIFICATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>76-H-020 (018/011-1)</td>
<td>N/A</td>
</tr>
</tbody>
</table>

Effect of Purity on Reliability Characteristics of High-Strength Steel, First Interim Report

Vacuum-induction heats of the high-purity-level steels were produced by special multiple-melting procedures and cast into 7-1/2-inch-diameter (19 cm) ingots. The ingots have been shipped to Latrobe Steel Company for vacuum-arc remelting (VAR) to achieve further reductions in oxygen and nitrogen contents and a refined cast structure prior to forging slabs and rolling to plate.

### Key Words for Indexing

- High Purity
- High Strength

### Document Type

- GEN RPT
- NONSTD PART
- SPEC

### Comments

- Describes the melting procedures used to produce the ultra-high-purity and conventional-purity steels, the rolling and heat-treatment procedures, and the properties obtained for the conventional-purity steels.

James Bullmast

USAFA, Aeronautical Systems Div

WPAFB, OH 45433 (G7)

Reproduction or display of this material for sales or publicity purposes is prohibited.
INSTRUCTIONS FOR COMPLETING THE GIDEP GENERAL DOCUMENT SUMMARY SHEET

NOTE: Completion of a Summary Sheet by the participant is not mandatory for document acceptance into GIDEP. A Summary Sheet will be prepared by the GIDEP Operations Center for document submittals received.

PAGE 1 OF Enter the total number of summary sheet pages

BOX

1. Leave blank—entry will be completed by GIDEP Operations Center.
2. Enter standard nomenclature associated with GIDEP Subject Thesaurus selected from Section 12, Policies and Procedures Manual.
3. Indicate application which the device was used (e.g., ground, missile, shipboard, spacecraft, refer to P & P Manual, Section 13).
4. Device manufacturer must be notified of test results. Manufacturer approval of report is not required—include pertinent manufacturer correspondence with document submittal to GIDEP, check NOTIFIED entry. If document is for a nonstandard part or of a general nature and a manufacturer is not identified, check NOT APPLICABLE.
5. Enter month and year of document issue.
6. Enter complete document title exactly as it appears on originator document.
7. Identify document type by inserting letter X by appropriate descriptor.
8. Enter document number exactly as it appears on originator document.
9. Enter part name and identification as assigned by organization/agency originating the report; if not specified, enter N/A (Not Applicable).
10. Delete either SUPERSEDES or SUPPLEMENTS. If document supersedes or supplements an existing GIDEP document, enter GIDEP microfilm access number of appropriate document. If document neither supersedes nor supplements an existing GIDEP document, enter the word NONE.
11. Enter the single symbol coding for environmental exposure as defined in Subject Thesaurus, Section 12, P&P Manual (e.g., C—Salt Spray; V—Vibration; %—Shelf Life); if not specified, enter N/A (Not Applicable).
12. Enter manufacturer abbreviation (if one) and H-4 Code number listed in GIDEP Manufacturer List. If manufacturer is not listed, enter the phrase, SEE BOX 15, enter manufacturer's full name and division (if any) in Box 15. If more than one manufacturer, enter phrase, SEE BOX 15, enter additional manufacturers as appropriate. If manufacturer is not specified, enter N/A (Not Applicable).
13. Enter complete part number. Use open O for alpha letter O, and use 0 for numeric zero. If more than one part number, enter phrase, SEE BOX 15; enter additional part number(s). If a part number is not specified, enter four dashes (-----).
14. Enter standard part number such as the 1N or 2N—diode and transistor designators. For GIDEP purposes, any military assigned number is considered as a government standard part number. If more than one standard number, enter phrase SEE BOX 15, enter additional standard number(s).
15. If subject matter in document can be categorized into more than one major subject category, enter additional subject categories in upper right-hand corner. Briefly summarize test results or material detailed in text of document. Include any pertinent details or comments required for proper interpretation of material presented (e.g., peculiar environmental capabilities, unique electrical characteristics that may be "state-of-the-art," or characteristics that restrict part usage to particular applications or any other details that may aid a prospective user of the part).
16. Enter appropriate words or phrases that enhance information retrieval on subject matter(s) contained in document. As a secondary data retrieval technique within each applicable Major Category (entry 2), the document is referenced in the computer data bank and Report Index according to each key word. Do not use abbreviations or words that are part of the subject category listed in Box 2. Key word phrases are limited to 60 total characters and blank spaces. Separate key words and/or phrases with commas.
17. Enter signature or name of GIDEP Representative.
18. Enter name, city, and state of participant activity or corporation and division submitting the document and GIDEP two-character code (e.g., XI).
In the first part of a study to establish the effect of purity on the properties of high-strength (250 ksi tensile strength) aircraft steels, three steels (AISI 4340, 18Ni maraging, and 10Ni modified) have been successfully vacuum-induction-melted to very-high-purity and normal-purity levels. The processing and heat-treating procedures for the normal-purity steels have essentially been established. When the high-purity steels have been vacuum-arc-remelted at Latrobe Steel Company, fracture-toughness and stress-corrosion testing can be initiated to establish the influence of purity on these properties.


Abstract

Since May 1, 1975, U. S. Steel has been engaged in research under Air Force Materials Laboratory Contract No. F33615-75-C-5137 to develop high-strength steels (with an ultimate tensile strength of 240 to 270 ksi, or 1655 to 1862 MPa) having improved fracture toughness and stress-corrosion resistance to obtain greater reliability in various classes of aerospace structural steels. The critical task in this study is to produce three steels [18Ni (250 grade) maraging steel, AISI 4340 steel, and 10Ni modified steel (AF 1410)] to very high purity levels. After considerable difficulty, vacuum-induction heats of the high-purity-level steels were produced by special multiple-melting procedures and cast into 7-1/2-inch-diameter (19 cm) ingots. The Ingots have been shipped to Latrobe Steel Company for vacuum-arc remelting (VAR) to achieve further reductions in oxygen and nitrogen contents and a refined cast structure prior to forging slabs and rolling to plate.

Concurrently, conventional-purity 500-lb (227 kg) heats of the same three steels were vacuum-induction-melted, cast into 7-by 12- by 24-inch (18 by 30 by 61 cm) slab ingots and cross-rolled to 1-inch-thick (2.5 cm) plate. Preliminary heat treatments were conducted on the 1-inch-thick plates of the conventional-purity steels to establish heat treatments that would provide similar...
strengths in the three steels. When the high-purity VAR steels are received from Latrobe, they will be processed to plate and heat-treated to the same strength level as the conventional-purity steels.

The present report describes in detail the melting procedures used to produce the ultra-high-purity and conventional-purity steels, the rolling and heat-treatment procedures, and the properties obtained for the conventional-purity steels. This represents the work completed during the first six-month period of the contract. In future work, comparisons will be made between the high-purity and the conventional-purity steels with respect to toughness (Charpy V-notch impact properties and $K_{IC}$ fracture toughness), incubation time to initiation of stress-corrosion cracking, stress-corrosion crack-propagation rates, and susceptibility to hydrogen embrittlement. Possible reasons for differences in the behavior of the high-purity and the conventional-purity steels will be studied by employing special experimental and metallographic techniques.
Introduction

Since May 1, 1975, studies have been conducted at the U. S. Steel Research Laboratory to develop high-strength steels (ultimate tensile strength of 240 to 270 ksi, or 1655 to 1862 MPa), having improved fracture toughness and stress-corrosion resistance to obtain greater reliability in structural airframe components. The work is being done under Air Force Materials Laboratory Contract No. F33615-75-C-5137.

In low-strength steels, improvements in steel cleanliness or purity usually result in improvement in both toughness and resistance to stress-corrosion cracking. However, for higher strength steels, there is some evidence that indicates that as the tensile strength exceeds 270 ksi, the improvements in fracture toughness and stress-corrosion-cracking resistance that can be realized by producing ultra-high-purity steels are minimal. If the above relations between strength and toughness and resistance to stress corrosion are found to hold regardless of steel type, composition, or degree of purity achieved, the use of high-strength steels in aircraft applications could be limited by the low resistance to stress-corrosion cracking, even in the useful tensile-strength range of 240 to 270 ksi where improvements in steel melting and processing have been found to produce improvements in fracture toughness.

Thus, a program was planned to determine the extent to which purity can affect the stress-corrosion characteristics
of steels in the 240- to 270-ksi tensile-strength range in which three classes of high-strength steels were to be produced at a normal-purity level and at the highest purity level achievable in useful quantities under modern laboratory melting practices. The classes of steels that were to be examined were (1) a maraging steel—18Ni (250 grade); (2) a conventional quenched and tempered steel—AISI 4340 steel; and (3) a recently developed low-carbon exceptionally high-toughness quenched and tempered steel—10Ni modified steel (also known as modified HY-180 or AF 1410). For each purity level, each of the three steels was to be treated to the same strength level and the following material characteristics determined: (1) tensile and Charpy V-notch impact properties; (2) fracture toughness \( K_{IC} \); (3) incubation time for the initiation of stress-corrosion cracking (using both smooth and precracked specimens); (4) threshold for stress-corrosion cracking \( K_{Isc} \); (5) stress-corrosion crack-propagation rate \( \frac{da}{dt} \) vs \( K_{II} \); and (6) susceptibility to hydrogen embrittlement (using precracked specimens). Possible reasons for differences in behavior of the high-purity and conventional-purity steels will be studied by employing special experimental and metallographic techniques.

The present report summarizes the work completed during the first six-month contract period, and also outlines the work in progress.
Discussion

The area of major difficulty in the proposed program was expected to be the attainment of the ultra-high-purity levels in the selected steels. As implied previously, achievement of the desired purity levels is essential to the success of the program.

Melting Stock

Selected melting stock was used to melt both the normal-purity and the ultra-high-purity steels. The stock was checked for the presence of tramp elements by analyzing one of the first normal-purity heats produced (4340 steel). The results of the chemical analyses for arsenic (<0.002%), antimony (<0.0004%), tin (<0.002%), and copper (0.006%) determined for this heat (Table I-A) are believed to be representative of the amounts of these elements in the melting stock, and therefore, in all the heats produced. The tramp elements investigated are known to increase the susceptibility of alloy steels to temper embrittlement. However, the levels detected are below the levels that normally increase the susceptibility of steels to temper embrittlement.

Chemical Analyses

Because of the very restricted ranges and low levels of certain of the elements in these steels, special analytical techniques were used, and sometimes more than one method was used for a particular element.
The techniques used to determine the compositions of the steels were as follows: carbon and sulfur - combustion analysis plus infrared detection; manganese, silicon, nickel, chromium, molybdenum, and cobalt - average of optical spectrographic and X-ray spectrometric or only X-ray spectrometric analysis; copper and titanium - optical spectrographic or X-ray spectrometric analysis; phosphorus and arsenic - colorimetric analysis (molybdenum-blue complex); aluminum - optical emission analysis, solution technique; oxygen - neutron-activation analysis; nitrogen - inert-gas fusion with a thermal-conductivity measurement; antimony - colorimetric analysis (rhodamine B); tin - optical spectrographic analysis or hydrogen-sulfide separation plus polarographic analysis. The X-ray spectrometric analysis and more sophisticated techniques were used for the higher alloy steels (18Ni and 10Ni) as explained below.

The analysis of the 18Ni (250 grade) maraging steel and the 10Ni modified steel for very low levels of manganese—Mn concentration of 10 parts per million (ppm)—presented special analytical problems. In addition to other elements, these steels contain 8 and 14 percent cobalt, and residual and 2 percent chromium, respectively. Because both cobalt and chromium interfere in the usual chemical methods for determining low concentrations of manganese, another method had to be developed.

In this new method, the bulk of the interfering elements is chemically separated from the manganese, and the manganese level
is then determined photometrically as permanganate in the presence of only small concentrations of cobalt and chromium. The color of the manganese species is then bleached by chemical reduction, and the background color due to any chromium and cobalt is measured. Manganese is determined by difference. This procedure has a detection limit of 5 ppm manganese. Results on synthetic standards at the 10-ppm level are accurate to within 2 ppm manganese.

**Melting Procedures**

For this study, each of these steels was vacuum-induction-melted to two purity levels—a normal-purity level and a high-purity level, Tables I-B and I-C. To produce the steels with purity levels similar to that attained by current production procedures (normal-purity steels), low-sulfur electrolytic iron was used as the base melt stock. The iron, carbon, nickel, molybdenum, and, when required, chromium and/or cobalt were charged into the crucible and melted in an atmosphere of 50 torr (6665 Pa) of argon. The argon was required to control the carbon boil during melting. At melt, the chamber was pumped to less than 1 torr (133 Pa), and a pin sample was obtained from the bath to determine the carbon level. The chamber was then repressurized to 200 torr (26.7 kPa) with argon. As soon as the carbon content of the bath was established, the silicon, sulfur, phosphorus, and necessary carbon additions were made. To obtain the desired nitrogen level, the pressure in the chamber was increased with nitrogen gas to establish the required partial pressure of nitrogen over the bath,
and then the nitrogen-bearing manganese, electrolytic manganese, aluminum, and titanium were added as required. Additional argon was added to the chamber, the temperature was adjusted to 2860 F (1571 C), and the steel was poured into a cast-iron mold to produce a 7-inch-thick by 12-inch-wide by 24-inch-high (18 by 30 by 61 cm) ingot weighing about 500 lb (227 kg), including the hot top.

Some difficulty was encountered in meeting the nitrogen specification for the 18Ni maraging steel. The composition of 18Ni maraging steel was such that nitrogen as an alloy (nitrogen-bearing manganese or nitrogen-bearing chromium) could not be used. Therefore, the nitrogen level was obtained by absorption from a partial atmosphere of nitrogen gas maintained in the vacuum chamber. The heats were held under the partial pressure for 1 hour to obtain the desired nitrogen content in the steel. The partial pressure was based on a calculated solubility of nitrogen in molten steel at 1 atmosphere (101 kPa) pressure.* That is,

\[ N = K \sqrt{p} \]

where \( K \) = equilibrium constant for nitrogen solubility in liquid steel at 1 atmosphere of nitrogen and 2912 F (1600 C); 0.028 for the 18Ni maraging steel.

\( p \) = pressure, in atmospheres.

However, the nitrogen content of the heat made by using the above procedure was low, an indication that the constant may be only about one-half of that indicated above or that equilibrium

---

was not obtained. By using a value of 0.014 for K, but otherwise the same procedure as outlined above, the desired level of nitrogen in the steel was subsequently obtained.

The second group of heats—those made to the high-purity level (Table I-C)—required a more complicated melting technique. To meet the low phosphorus level (<0.001%), different batches from the same lot of electrolytic iron were sampled and analyzed, and then the iron from the batch with the lowest phosphorus level was used. To meet the low sulfur level (<0.001%), a lime-crucible melting practice that had been used in past studies was tried. However, this method of desulfurization was only successful when the oxygen content of the steel had been reduced to a low value (approximately 20 ppm) by using a relatively high level of aluminum. Because aluminum and other strong deoxidizers could not be used in producing the steels in this program, preliminary trials were made by using the carbon in the 4340 steel and the titanium in the maraging steel as deoxidizers to attain the low oxygen known to be required for effective desulfurization of the liquid bath. However, these practices did not produce sulfur levels as low as desired. Thus, it was necessary to adopt a double-melting practice utilizing a lime crucible.

Since high-purity lime crucibles are not commercially available, a special fused lime of very low sulfur content (<0.001%) was obtained from the Muscle Shoals Electrochemical Company, Tascumbia, Alabama. The lime crucibles were fabricated
inside a 500-lb prefired magnesia crucible installed in the induction furnace in the normal manner. A 1-inch (25.4 mm) layer of the special lime was rammed on the bottom of the magnesia crucible. Then a fabricated 1/8-inch-thick (3.2 mm) sheet-steel liner with a diameter 1 inch less than that of the magnesia crucible was centered inside the prefired magnesia crucible, and the special lime was rammed in the 1/2-inch (12.7 mm) space between the steel liner and the walls of the magnesia crucible. Electrolytic iron was then melted in the crucible to sinter the lime as well as melt out the steel liner. The lime crucible obtained by this technique was used to melt the high-purity steels. Although magnesia crucibles can be used to melt 20 heats, the lime crucible produced by this technique lasted for only 5 to 8 heats. Also, because a relatively high silicon content (0.020 to 0.035%) was observed in the first heat melted in a newly rammed and sintered lime crucible, essentially two heats had to be melted and discarded before attempting to melt the high-purity steels. The first heat melted out the steel liner and sintered the lime, and the second heat further conditioned the lime lining to prevent the pickup of silicon.

As indicated earlier, the melting of the high-purity steels was a double-melt practice. Because of the potential contamination by manganese, chromium, or cobalt from a previous heat, the full double-melting procedure for each grade of steel had to be completed before the melting of another grade of steel could be initiated.
The first melt in the double-melting procedure was a desulfurizing melt and consisted of charging sufficient electrolytic iron, electrolytic nickel, electrolytic chromium, pure molybdenum, and pure cobalt to meet the final specification for the steel. When the charge was melted, the bath was vacuum-carbon-deoxidized to decrease the oxygen content. This deoxidation was designed to yield a carbon content in the bath which would not exceed 0.02 percent for the 18Ni maraging steel and 0.05 percent for the other two grades. After the vacuum-carbon deoxidation was completed, the bath was further deoxidized with 0.05 percent aluminum, and then 0.30 percent (579 grams) of a misch-metal alloy (rare-earth metals) was added to the bath. A preliminary sample of the metal was then obtained and submitted to the chemical laboratory to determine the sulfur content of the bath. If the sulfur content at this time was greater than 5 ppm, an additional 200 grams of the misch-metal was added to the 193-kg bath and a sample was again submitted for sulfur content. This practice was continued until a sulfur content of 5 ppm or less was achieved. Control of the bath temperature during the time required for sulfur analyses was necessary, and the temperature was not permitted to exceed 2850 F (1566 C). This temperature control was important because at high temperatures—that is, greater than 2900 F (1593 C)—loss of the deoxidizers or desulfurizers would be experienced.
When a sufficiently low sulfur level was achieved, the bath temperature was adjusted to 2860°F (1571°C) and the metal was poured into a 9-inch-diameter (23 cm) mold. A sample of the metal was sent to the chemical laboratory for complete chemical analyses, and the ingot surface was completely machined to eliminate any slag that might contain sulfur. Before proceeding with the next stage in the melting procedure, a wash heat was melted in the lime crucible because experience had shown that the sulfur removed from the metal during desulfurizing would be retained on the surface of the lime-crucible lining and would contaminate the following heat unless the crucible was desulfurized by melting a wash heat.

The machined low-sulfur ingot, which weighed about 350 pounds (159 kg), was cut into four sections. These sections, together with additional electrolytic iron and alloys to produce the desired composition, were charged into the desulfurized crucible and remelted.

The electrolytic iron and the alloys added were assumed to contain sufficient oxygen to oxidize any excess aluminum and misch-metal remaining in the desulfurized steel during the remelting operation. The final composition indicates that this assumption was correct (Table I-C).

When the low-sulfur charge was remelted, a sample of the metal was sent to the chemical laboratory to be certain that the
sulfur content was not greater than 10 ppm. When it was confirmed that the sulfur level was satisfactory, the melt was finished, as described below, to meet the aim specification.

The range, aim, and final check chemical composition of the three high-purity heats are shown in Table I-C. In particular, it should be noted that the sulfur levels for the 4340, 10Ni modified, and 18Ni maraging steels were 9, 7, and 5 ppm, respectively.

The finishing practice for each of the three heats varied, depending on their composition. Finishing of the 4340 grade involved the addition of 0.05 percent carbon to the steel at a chamber pressure of 100 torr (13.3 kPa) of argon, and then evacuating the chamber until a chamber pressure of 200 microns was achieved. The chamber was then pressurized to 200 torr (26.7 kPa) with argon. The carbon content of the bath was determined, a carbon addition amounting to 0.36 percent was made, and again the pressure in the chamber was lowered by pumping. When the chamber pressure reached 5 torr (667 Pa), buildup of a steel skull from steel splashing on the top of the furnace became serious and further pumping was stopped. Samples were submitted for analysis of carbon content by combustion and for analysis of chromium, nickel, and molybdenum contents by X-ray spectrometric analysis. A final adjustment of carbon and chromium was made, and then all the manganese was added. The chamber was pressurized to 550 torr (73.3 kPa) with argon, the
temperature adjusted to 2835 °F (1557 °C), and the metal poured into a 7-1/2-inch-diameter (19 cm) mold. This 4340 ingot bled or rimmed for at least 10 minutes before the bleeding of metal out of the ingot center ceased. This ingot is expected to be quite porous and may present difficulties in vacuum-arc remelting.

The 18Ni maraging steel was less of a problem to finish because it required only the addition of titanium to the molten bath. However, since the oxygen level of the bath was unknown and the possibility of titanium oxidation existed, an 0.51 percent titanium addition was made to the liquid bath, and a sample was submitted to determine the composition of the bath before tapping the heat. The sample indicated that the steel contained 0.46 percent titanium and that the nickel, molybdenum, and cobalt contents were within the specified range. An additional 0.04 percent titanium addition was made, the bath was heated to 2860 °F (1571 °C), and the heat was tapped into the 7-1/2-inch-diameter mold.

Finishing the 10Ni modified steel heat required only the addition of 0.16 percent carbon at a chamber pressure of 200 torr (26.7 kPa). After sampling to confirm that the carbon and sulfur concentration in the steel was satisfactory, the bath temperature was adjusted and the heat was tapped into the 7-1/2-inch-diameter mold. This ingot rimmed in the mold, but not as seriously as that observed for the 4340 heat.
After the chemical composition of each ingot had been checked, the three ingots were shipped to Latrobe Steel Company for vacuum-arc remelting.

Because the production of the high-purity steels required as many as eight different elements to be controlled to very low levels, difficulty was experienced in achieving the desired level for all the elements in the same heat. Thus, many heats had to be rejected because one or more of these eight elements was higher than desired. To produce the three high-purity steels with the chemical compositions shown in Table I-C, five attempts were made to produce the 4340 steel, two attempts were made to produce the 10Ni modified steel, and four attempts were made to produce the 18Ni maraging steel. Because each attempt represented three melting steps, a total of 33 melts was required to produce these high-purity steels.

**Rolling of Normal-Purity Steels**

The three normal-purity 500-lb induction-furnace-melted ingots were hot-charged into a 2150 F (1177 C) furnace and straightaway-rolled to 3-inch-thick (7.6 cm) slabs. The slabs were cut into four equal pieces, reheated at 2150 F, and cross-rolled to 1-inch-thick plate. Four pieces, about 1 by 13 by 30 inches (2.5 by 33 by 76 cm) were obtained from each heat. The 10Ni modified and 18Ni steels were water-quenched after each rolling; the 4340 steel was air-cooled after each rolling. The
rolling ratio (ingot axis to final rolling direction) for the plates was about 1 to 1.25.

Heat Treatment and Mechanical-Property Tests of Normal-Purity Steels

Coupons from the three normal-purity heats were obtained to determine the tempering or aging treatment that would result in a tensile strength of about 250 ksi (1724 MPa). The 4340 steel was double-austenitized at 1650 and 1525 F (899 and 829 C), and was oil-quenched from each temperature. Sections of the as-quenched coupon were tempered at 400 F (204 C) for 5 hours, at 450 F (232 C) for 2 and 8 hours, and at 500 F (260 C) for 1 hour. The 10Ni modified steel was double-austenitized at 1650 and 1500 F (899 and 816 C), and was water-quenched from each temperature. Sections of the as-quenched coupon were tempered at 950 F (510 C) for 5, 6, and 7 hours. The 18Ni steel was double-austenitized at 1650 and 1525 F, and was water-quenched from each temperature. Sections of the as-quenched coupon of this steel were aged at 900 F (482 C) for 4, 5, and 6 hours. The samples of the 10Ni modified and 18Ni steel were water-quenched after tempering, whereas the samples of the 4340 steel were air-cooled after tempering because the tempering temperatures were below the temperature range where embrittlement could occur on slow cooling.

For each steel in each heat-treated condition, three longitudinal 0.252-inch-diameter (6.4 mm) tension-test specimens and nine Charpy V-notch impact-test specimens were machined.
The tension tests were conducted at room temperature, and the impact tests were conducted at +75, 0, and -80 °F (26, -18, and -62 °C).

The results of the mechanical-property tests are shown in Table II. With respect to the desired 250-ksi tensile strength, these data indicate that the 10Ni modified steel when tempered at 950 °F for 6 to 7 hours can attain a tensile strength of 252 to 247 ksi (1737 to 1703 MPa), and that the 18Ni steel when aged at 900 °F for about 5 hours can attain a tensile strength of 252 ksi. For the 4340 steel, the most promising tempering temperature explored thus far was 450 °F for 8 hours for which a tensile strength of 266 ksi (1834 MPa) was attained. For the above treatments, the yield-to-tensile ratios for the 4340, 10Ni, and 18Ni steels were about 0.85, 0.93, and 0.95, respectively. A tempering treatment of 475 °F (246 °C) for 8 hours will be explored for the 4340 steel to attempt to lower the tensile strength and raise the yield-to-tensile ratio.

As can be seen in Table II, the 10Ni modified steel exhibited a higher tensile ductility and a lower yield strength for a given tensile strength then the 18Ni steel. As would be expected, the tensile ductility of the 0.40 percent carbon 4340 steel was lower than that of either the 10Ni or the 18Ni steel. The level of notch toughness of these steels was generally low and similar, although the higher nickel steels exhibited somewhat higher impact values.
Summary

During the first six months of work on Air Force Materials Laboratory Contract No. F33615-75-C-5137 ("Effect of Purity on Reliability Characteristics of High-Strength Steels"), AISI 4340 steel, 18Ni (250 grade) maraging steel, and 10Ni modified steel have been vacuum-induction-melted to the desired normal- and high-purity compositions. The normal-purity steels have been processed to plate and subjected to trial heat treatments to establish the heat-treating procedures that will provide equivalent tensile properties in each of the steels. The results of the work to date can be summarized as follows:

1. Selected low-sulfur melting stock was shown to contain satisfactorily low levels of the tramp elements antimony, arsenic, tin, and copper, and the melting stock was deemed to be suitable for the intended studies.

2. Normal-purity steels were produced with little difficulty except for achieving the desired nitrogen level in the maraging steel.

3. Vacuum-induction-melted heats of the high-purity steels were produced only after considerable experimental difficulty and the development of special, multiple-melting techniques.

4. Special chemical-analysis methods were used to determine the composition of the high-purity steels, and a special technique was developed to obtain accurate analyses for manganese.
5. The vacuum-induction-melted high-purity steels are now being vacuum-arc-remelted (VAR) and forged to slabs by Latrobe Steel Company.

**Future Work**

When the forged VAR slabs of the high-purity steels are received from Latrobe Steel, they will be cross-rolled to 1-inch-thick (25.4 mm) plate in a manner similar to that used for the normal-purity heats. The final chemical analyses will be conducted on the plates of these steels.

Heat-treating studies will be conducted on the high-purity plates to establish the tempering and aging times needed to obtain tensile strengths for these steels similar to those of the normal-purity steels.

Fracture toughness ($K_{IC}$) and stress-corrosion-cracking ($K_{Iscc}$) tests will be initiated as soon as the heat-treating cycles have been established and the plates have been given the final heat treatments.
### Table I

**Chemical Composition of Steels Prepared—Percent**

#### A. Impurity Level of Melting Stock*

<table>
<thead>
<tr>
<th></th>
<th>As</th>
<th>Sb</th>
<th>Sn</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>&lt;0.002</td>
<td>&lt;0.0004</td>
<td>&lt;0.002</td>
<td>0.006</td>
</tr>
</tbody>
</table>

#### B. Normal-Purity-Level Steels**

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Co</th>
<th>O</th>
<th>Al***</th>
<th>N</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Range</td>
<td>4340</td>
<td>0.38</td>
<td>0.65</td>
<td>0.008</td>
<td>0.008</td>
<td>0.20</td>
<td>1.75</td>
<td>0.75</td>
<td>0.22</td>
<td>-</td>
<td>0.003</td>
<td>0.015</td>
<td>0.008</td>
</tr>
<tr>
<td></td>
<td>0.42</td>
<td>0.75</td>
<td>0.012</td>
<td>0.012</td>
<td>0.30</td>
<td>1.85</td>
<td>0.85</td>
<td>0.28</td>
<td>-</td>
<td>0.005</td>
<td>0.035</td>
<td>0.012</td>
<td></td>
</tr>
<tr>
<td>Aim</td>
<td>&quot;</td>
<td>0.40</td>
<td>0.70</td>
<td>0.010</td>
<td>0.010</td>
<td>0.25</td>
<td>1.80</td>
<td>0.80</td>
<td>0.25</td>
<td>-</td>
<td>0.004</td>
<td>0.025</td>
<td>0.010</td>
</tr>
<tr>
<td>Check</td>
<td>&quot;</td>
<td>0.40</td>
<td>0.71</td>
<td>0.010</td>
<td>0.011</td>
<td>0.27</td>
<td>1.80</td>
<td>0.82</td>
<td>0.25</td>
<td>-</td>
<td>0.0016</td>
<td>0.034</td>
<td>0.008</td>
</tr>
<tr>
<td>Analysis</td>
<td>&quot;</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Range</td>
<td>10Ni</td>
<td>0.15</td>
<td>0.10</td>
<td>0.008</td>
<td>0.008</td>
<td>0.08</td>
<td>9.50</td>
<td>1.90</td>
<td>0.90</td>
<td>13.50</td>
<td>0.003</td>
<td>NA(^1)</td>
<td>0.003</td>
</tr>
<tr>
<td></td>
<td>0.17</td>
<td>0.20</td>
<td>0.012</td>
<td>0.012</td>
<td>0.12</td>
<td>10.50</td>
<td>2.10</td>
<td>1.10</td>
<td>14.50</td>
<td>0.005</td>
<td></td>
<td>0.007</td>
<td></td>
</tr>
<tr>
<td>Aim</td>
<td>&quot;</td>
<td>0.16</td>
<td>0.15</td>
<td>0.010</td>
<td>0.010</td>
<td>0.10</td>
<td>10.00</td>
<td>2.00</td>
<td>1.00</td>
<td>14.00</td>
<td>0.004</td>
<td>LAP(^2)</td>
<td>0.005</td>
</tr>
<tr>
<td>Check</td>
<td>&quot;</td>
<td>0.18</td>
<td>0.14</td>
<td>0.010</td>
<td>0.011</td>
<td>0.10</td>
<td>10.15</td>
<td>2.01</td>
<td>1.00</td>
<td>14.00</td>
<td>0.0022</td>
<td>&lt;0.002</td>
<td>0.006</td>
</tr>
<tr>
<td>Analysis</td>
<td>&quot;</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Range</td>
<td>18Ni</td>
<td>0.01</td>
<td>0.08</td>
<td>0.008</td>
<td>0.008</td>
<td>0.08</td>
<td>17.50</td>
<td>-</td>
<td>4.60</td>
<td>7.50</td>
<td>0.003</td>
<td>0.04</td>
<td>0.008</td>
</tr>
<tr>
<td></td>
<td>0.03</td>
<td>0.12</td>
<td>0.012</td>
<td>0.012</td>
<td>0.12</td>
<td>18.50</td>
<td>-</td>
<td>5.10</td>
<td>8.00</td>
<td>0.005</td>
<td>0.08</td>
<td>0.012</td>
<td>0.50</td>
</tr>
<tr>
<td>Aim</td>
<td>&quot;</td>
<td>0.02</td>
<td>0.10</td>
<td>0.010</td>
<td>0.010</td>
<td>0.10</td>
<td>18.00</td>
<td>-</td>
<td>4.85</td>
<td>7.75</td>
<td>0.004</td>
<td>0.06</td>
<td>0.010</td>
</tr>
<tr>
<td>Check</td>
<td>&quot;</td>
<td>0.032</td>
<td>0.11</td>
<td>0.011</td>
<td>0.009</td>
<td>0.10</td>
<td>18.00</td>
<td>&lt;0.05</td>
<td>4.82</td>
<td>7.71</td>
<td>0.0017</td>
<td>0.019</td>
<td>0.008</td>
</tr>
<tr>
<td>Analysis</td>
<td>&quot;</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(Continued)
Table I (Continued)

C. High-Purity Steels

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Co</th>
<th>O</th>
<th>Al**</th>
<th>N</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Range</td>
<td>4340</td>
<td>0.38</td>
<td>0.65</td>
<td>0.001</td>
<td>0.001</td>
<td>0.005</td>
<td>1.75</td>
<td>0.75</td>
<td>0.22</td>
<td>-</td>
<td>0.001</td>
<td>0.01</td>
</tr>
<tr>
<td>Aim</td>
<td>&quot;</td>
<td>0.40</td>
<td>0.70</td>
<td>LAP</td>
<td>LAP</td>
<td>LAP</td>
<td>1.80</td>
<td>0.80</td>
<td>0.25</td>
<td>-</td>
<td>LAP</td>
<td>LAP</td>
</tr>
<tr>
<td>Check</td>
<td>&quot;</td>
<td>0.37</td>
<td>0.57</td>
<td>0.0014</td>
<td>0.0009</td>
<td>0.003</td>
<td>1.81</td>
<td>0.77</td>
<td>0.27</td>
<td>0.022</td>
<td>&lt;0.002</td>
<td>0.0027</td>
</tr>
</tbody>
</table>

Range | 10Ni | 0.15 | 0.001| 0.001| 0.001| 0.005| 9.50 | 1.90 | 0.90 | 13.50| 0.001| 0.01| 0.001| - |
| Aim   | "    | 0.16 | LAP  | LAP  | LAP  | LAP | 10.00 | 2.00 | 1.00 | 14.00| LAP  | LAP | LAP | - |
| Check | "    | 0.17 | 0.001| 0.0010| 0.0007| 0.007| 10.00 | 1.95 | 1.03 | 13.90| <0.002| 0.0019| - |

Range | 18Ni | 0.003| 0.001| 0.001| 0.001| 0.005| 17.50 | -   | 4.60 | 7.50 | 0.001| 0.01| 0.001| 0.40|
| Aim   | "    | LAP  | LAP  | LAP  | LAP  | LAP | 18.50 | 5.10 | 8.00 | max  | max  | max  | max  | 0.50 |
| Check | "    | <0.005| 0.001| 0.0006| 0.0005| 0.009| 18.30 | <0.05| 4.83 | 7.80 | 0.003| 0.0017| 0.43| - |

* Determined on AISI 4340 heat.
** Analyses determined on slab product.
*** Total aluminum content.
1) NA - None Added.
2) LAP = Low As Possible.
3) Analyses determined from spoon sample obtained during the pouring of the electrode ingots [Prepared for vacuum-arc-remelting (VAR)].
4) Oxygen \((O_{2})\) level to be determined after VAR processing.
Table I (Continued)

NOTE: The heat numbers of the six steels (with a 7518 prefix number) are as follows:

<table>
<thead>
<tr>
<th>Steel</th>
<th>Normal-Purity Level</th>
<th>High-Purity Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>4340</td>
<td>8042-1X</td>
<td>8045-10</td>
</tr>
<tr>
<td>10Ni</td>
<td>8044-1X</td>
<td>8047-4</td>
</tr>
<tr>
<td>18Ni</td>
<td>8043-3X</td>
<td>8046-8</td>
</tr>
<tr>
<td>Steel</td>
<td>Tempering Temp, F</td>
<td>Time, hr</td>
</tr>
<tr>
<td>-------</td>
<td>------------------</td>
<td>---------</td>
</tr>
<tr>
<td>4340</td>
<td>400</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>450</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>450</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>1</td>
</tr>
<tr>
<td>10Ni</td>
<td>950</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>950</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>950</td>
<td>7</td>
</tr>
<tr>
<td>18Ni</td>
<td>900</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>900</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>900</td>
<td>6</td>
</tr>
</tbody>
</table>

*Room Temperature

NOTE: The 4340 and 18Ni steels were double-quenched from 1650 and 1525 F; the 4340 steel was oil-quenched, whereas the 18Ni steel was water-quenched. The 10Ni modified steel was double-water-quenched from 1650 and 1500 F. The tensile data represent average values for triplicate 0.252-inch-diameter specimens, whereas the impact data represent the average of either duplicate or triplicate specimens.

**Conversion Factors**

\[
C = \frac{5}{9} (F - 32)
\]

1 ksi = 6.895 MPa

1 inch = 25.4 mm

1 ft-lb = 1.36 J

76-H-020 (018/011-1)