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RESEARCH IN DEVELOPMENT OF IMPROVED
TRIP STEELS

T. J. Koppenaal

Philco-Ford Corporation

Prepared for:

Army Materials and Mechanics Research Center

January 1973

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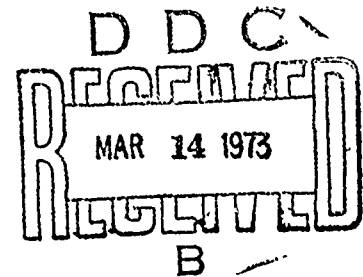
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Philco-Ford Corporation
Aeronutronic Division
Newport Beach, California



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Technical Report by
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Aeronutronic Division
Newport Beach, Calif. 92663

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FOREWORD

This report was prepared by the Advanced Development Operation, Aeronutronic Division of the Philco-Ford Corporation, a subsidiary of the Ford Motor Company. The contract, No. DAAG46-72-G-0047, was administered under the direction of Mr. Arthur Ayvazian of the U.S. Army Materials and Mechanics Research Center, Watertown, Massachusetts.

The author would like to thank Mr. R. P. Sernka for many technical discussions relating to this program. Mr. M. J. Harrigan performed the computer programming analysis in the Discussion, and Professor K. Ono of U.C.L.A. made the acoustic emission measurements of M_s . Mr. J. M. Seay and Mr. W. F. Shirley performed much of the experimental work.

This project has been accomplished as part of the U.S. Army Manufacturing Methods and Technology Program, which has as its objective the timely establishment of manufacturing processes, techniques or equipment to insure the efficient production of current or future defense programs.

ABSTRACT

A program was conducted to establish thermal processing procedures directed at improving the strength of TRIP steels. The general approach was to strain hardened TRIP steels by thermal cycling between martensite and reverted austenite.

A total of 32 alloys that were variations of the "A" type TRIP steels (Fe-9Cr-8Ni-4Mo-2Si-2Mn-0.30C) were evaluated. Of these, thirteen had a M_s in the desired range for thermal processing, $-320^{\circ}\text{F} < M_s < \text{RT}$. However, after cooling at -320°F (forming martensite) and heating to 1500°F (reverting to austenite), the M_s of reverted austenite was above room temperature. With this increase (ΔM_s was about $200-300^{\circ}\text{F}$) the alloys were unsuitable for thermal processing. Several attempts were made to decrease the M_s of reverted austenite to below room temperature. The only successful method was to utilize high reversion temperatures of $1775-1850^{\circ}\text{F}$, but even this approach was not useful due to low hardness values after reversion. A computer program was used to measure the effect of various alloying elements on the M_s of annealed and reverted austenite and A_f , the temperature corresponding to completion of the martensite to austenite transformation. Some unexpected results were obtained; manganese,

for example, lowers the M_s about an order of magnitude more than has previously been noted in more dilute base compositions.

After it became known that the "A" type alloys could not be used for thermal processing, a total of eighteen alloys that were variations of the "B" type TRIP steels (Fe-24Ni-4Mo-.30C) were evaluated. The best mechanical properties were obtained with an Fe-24Ni-4Mo-.20 Si-.20Mn-.30C alloy. After five cyclical reversions the following properties were obtained: 160.0 ksi static yield strength, 193.0 dynamic yield strength, 196.5 static tensile strength, 30% elongation, 40% reduction in area, 27.7 to 28.1 ft.-lbs. Charpy V-notch impact energy at -40°F and 72°F , and a dynamic plane fracture toughness of $89 \text{ ksi} \cdot (\text{in})^{\frac{1}{2}}$ in both the RW (longitudinal) and WT (transverse) orientation.

INTRODUCTION

BACKGROUND

A relatively new class of steels has been developed by Zackay, et al.,⁽¹⁾ that shows an excellent combination of high strength and ductility. Since the high ductility results from the austenite to martensite transformation occurring during plastic deformation, the authors selected the name TRIP steels, which is an acronym of Transformation Induced Plasticity. The exceptionally high yield strength (up to about 220 ksi) of the austenite is produced by a combination of solid solution strengthening, precipitation strengthening, and work-hardening; the first two of these mechanisms is accomplished by alloying and the other by thermomechanical treatment. The work-hardening increment introduced by thermomechanical treatment is probably the most significant.

Despite the excellent strength-ductility relationship in TRIP steels, several limitations and disadvantages are associated with the thermomechanical treatment step in the processing. Since deformations up to about 80% are usually done at temperatures in the 800-1000°F temperature range, rather massive metal forming equipment is needed. This feature is probably the primary reason that TRIP steels have not been further developed.

Associated with this limitation is the result that only comparatively thin sections can be produced. Another major limitation of TRIP steels is that they cannot be welded without destroying the effects of the processing (as with any thermomechanically processed material). Similarly, these steels cannot be used in castings because of the necessary thermomechanical processing. Therefore, only simple shapes representative of normal metal working techniques can be produced without additional machining. Finally, the mechanical properties of TRIP steels are highly anisotropic, and this further limits the usefulness of these alloys. Thus, while the thermomechanical treatment has been a necessary step in the processing of TRIP steels, it places significant limitations on their application.

Koppelaar⁽²⁾ recently demonstrated that certain TRIP steels can be prepared by a thermal processing technique alone, thereby eliminating the undesirable thermomechanical treatment that was previously considered necessary. Briefly, the thermal processing technique makes use of performing the reverse martensite (α') transformation at temperatures below the recrystallization temperature of austenite (γ). In this way, a high defect concentration exists in the austenite following the transformation, and the austenite is essentially work-hardened. By cycling through five $\gamma \rightarrow \alpha' \rightarrow \gamma$ transformations, the hardness of the austenite continuously increased. The original as-quenched austenite was $\sim 0 R_C$ ($80 R_B$), and cooling to liquid nitrogen produced $\sim 70\%$ martensite at a hardness of $31 R_C$. After reverting to austenite the hardness was $28 R_C$; successive cyclical transformations produced austenite with a hardness of $37 R_C$ (2nd cycle), $40\frac{1}{2} R_C$ (3rd cycle), $42 R_C$ (4th cycle), and $42\frac{1}{2} R_C$ (5th cycle). After the fifth reversion cycle, mechanical (tensile) properties were produced that are essentially equivalent to those produced by Zackay, et al.,⁽¹⁾ in the same alloy by a thermomechanical treatment of 80% reduction in area at 930°F . However, the specific alloy that was used in the Aeronutronic program has a yield strength of only 160 to 165 ksi, instead of values of about 220 ksi obtainable in other

TRIP steel alloys. In order for the thermal processing of TRIP steels to become more useable, it would be beneficial to improve upon the strength following the new type of processing.

OBJECTIVE

The objective of the proposed program was to establish thermal processing procedures directed at improving the strength of TRIP steels.

EXPERIMENTAL APPROACH

The experimental approach was to screen a number of alloys that are modifications of the strongest TRIP steel alloy; these alloy modifications were necessary to raise the M_s temperature (the strongest TRIP steel has an $M_s < -452^\circ\text{F}$). For those alloys having an M_s in the desirable range, $-320^\circ\text{F} < M_s < \text{RT}$, the martensite to austenite reversion temperature, A_f , was determined. A final selection was then to be made for those alloys with an M_s and A_f compatible with the thermal processing technique, and various mechanical properties were to be measured as a function of thermal processing.

EXPERIMENTAL PROCEDURE

ALLOY PREPARATION

A total of fifty alloys were used in this investigation. Casting, pouring and solidification were conducted by vacuum induction melting techniques. The analyzed compositions of the alloys are given in Table I. Alloys 1 thru 25 and 44 thru 50 are modifications of the Fe-9Cr-8Ni-4Mo-2Si-2Mn-.30C TRIP steel and alloys 26 thru 43 are modifications of the Fe-24Ni-4Mo-.30C TRIP steel; these two general compositions will be referred to as the "A" and "B" type of alloys, respectively, following the notation originally given by Zackay, et al.⁽¹⁾

The alloys were cast into a mold about 3-1/2 in. x 3-1/2 in. x 7-1/2" long, and were subsequently forged into bars with an 1-1/2 in. x 1-1/2 in. cross-section at temperatures of 2000-2200°F. After forging, a decarburized layer of about .020 in. existed in the bars. Prior to any additional processing, .050 in. was removed from all surfaces by grinding. Following this grinding, all of the bars were homogenized at 2350°F for 10 hours at a vacuum of 1 Torr (back-filled with an inert gas). The bars were rolled at 1800°F to a final thickness of about 1/4 to 1/2 inch, and solution heat treated at 2050°F for 1 hour and water quenched.

TABLE I

CHEMICAL COMPOSITIONS OF ALLOY STEELS

nominal weight percent alloying element
analyzed weight percent alloying element

ALLOY NUMBER	HEAT NUMBER*	Cr	Ni	Mo	Mn	Si	W	Co	C
1	3855	$\frac{9.0}{8.95}$	$\frac{8.0}{8.08}$	$\frac{4.0}{4.01}$	$\frac{.50}{.50}$	$\frac{2.0}{1.85}$	—	—	$\frac{.30}{.33}$
2	3820	$\frac{9.0}{9.07}$	$\frac{8.0}{8.08}$	$\frac{4.0}{4.08}$	$\frac{.50}{.51}$	$\frac{2.0}{2.01}$	—	—	$\frac{.25}{.27}$
3	3821	$\frac{9.0}{9.03}$	$\frac{8.0}{8.02}$	$\frac{2.0}{2.16}$	$\frac{.50}{.49}$	$\frac{2.0}{2.01}$	—	—	$\frac{.30}{.31}$
4	3822	$\frac{9.0}{9.01}$	$\frac{8.0}{7.99}$	$\frac{2.0}{2.16}$	$\frac{.50}{.52}$	$\frac{2.0}{2.01}$	—	—	$\frac{.25}{.24}$
5	3823	$\frac{9.0}{9.01}$	$\frac{8.0}{7.99}$	—	$\frac{.50}{.55}$	$\frac{2.0}{2.03}$	—	—	$\frac{.30}{.30}$
6	3824	$\frac{9.0}{9.16}$	$\frac{8.0}{7.90}$	—	$\frac{.50}{.54}$	$\frac{2.0}{2.03}$	—	—	$\frac{.25}{.26}$
7	3856	$\frac{9.0}{9.21}$	$\frac{8.0}{7.99}$	$\frac{4.0}{4.14}$	$\frac{.50}{.49}$	$\frac{.50}{.49}$	—	—	$\frac{.30}{.31}$
8	3825	$\frac{9.0}{9.08}$	$\frac{8.0}{7.88}$	$\frac{4.0}{4.07}$	$\frac{.50}{.49}$	$\frac{.50}{.41}$	—	—	$\frac{.25}{.26}$
9	3826	$\frac{9.0}{9.10}$	$\frac{8.0}{7.63}$	$\frac{2.0}{2.16}$	$\frac{.50}{.49}$	$\frac{.50}{.54}$	—	—	$\frac{.25}{.25}$
10	3857	$\frac{9.0}{9.06}$	$\frac{8.0}{8.11}$	—	$\frac{.50}{.48}$	$\frac{2.0}{1.96}$	$\frac{4.0}{3.86}$	—	$\frac{.30}{.31}$
11	3827	$\frac{9.0}{9.16}$	$\frac{8.0}{7.88}$	—	$\frac{.50}{.48}$	$\frac{2.0}{1.78}$	$\frac{2.0}{2.02}$	—	$\frac{.25}{.26}$

TABLE I - Continued

ALLOY NUMBER	HEAT NUMBER*	Cr	Ni	Mo	Mn	Si	W	Co	C
12	3828	$\frac{9.0}{9.05}$	$\frac{8.0}{7.91}$	—	$\frac{.50}{.49}$	$\frac{.50}{.51}$	$\frac{4.0}{3.81}$	—	$\frac{.25}{.27}$
13	3829	$\frac{9.0}{9.15}$	$\frac{6.0}{5.90}$	$\frac{4.0}{4.21}$	$\frac{.50}{.50}$	$\frac{2.0}{1.94}$	—	—	$\frac{.25}{.26}$
14	3859	$\frac{9.0}{9.11}$	$\frac{6.0}{6.14}$	$\frac{4.0}{4.13}$	$\frac{.50}{.51}$	$\frac{2.0}{1.88}$	—	$\frac{2.0}{1.93}$	$\frac{.30}{.31}$
15	3830	$\frac{9.0}{9.36}$	$\frac{6.0}{5.90}$	$\frac{4.0}{4.06}$	$\frac{.50}{.49}$	$\frac{.50}{.50}$	—	—	$\frac{.25}{.26}$
16	3831	$\frac{9.0}{9.34}$	$\frac{4.0}{3.95}$	$\frac{4.0}{4.16}$	$\frac{.50}{.49}$	$\frac{2.0}{1.86}$	—	—	$\frac{.25}{.26}$
17	3832	$\frac{9.0}{9.45}$	$\frac{4.0}{3.90}$	$\frac{4.0}{4.13}$	$\frac{.50}{.45}$	$\frac{.50}{.44}$	—	—	$\frac{.25}{.26}$
18	3833	$\frac{9.0}{8.52}$	$\frac{8.0}{7.56}$	$\frac{4.0}{3.88}$	$\frac{.50}{.45}$	$\frac{2.0}{1.81}$	—	$\frac{1.5}{1.00}$	$\frac{.25}{.26}$
19	3858	$\frac{9.0}{9.01}$	$\frac{8.0}{8.25}$	$\frac{4.0}{4.03}$	$\frac{.50}{.51}$	$\frac{2.0}{1.88}$	—	$\frac{3.0}{2.98}$	$\frac{.30}{.30}$
20	3834	$\frac{9.0}{9.14}$	$\frac{8.0}{8.20}$	$\frac{4.0}{3.98}$	$\frac{.50}{.51}$	$\frac{.50}{.50}$	—	$\frac{3.0}{2.93}$	$\frac{.25}{.27}$
21	3835	$\frac{9.0}{9.14}$	$\frac{8.0}{8.13}$	—	$\frac{.50}{.50}$	$\frac{.50}{.52}$	$\frac{4.0}{3.74}$	$\frac{3.0}{2.89}$	$\frac{.25}{.27}$
22	3836	$\frac{9.0}{9.17}$	$\frac{8.0}{8.20}$	$\frac{4.0}{4.11}$	$\frac{.50}{.50}$	$\frac{.50}{.42}$	—	$\frac{5.0}{5.15}$	$\frac{.30}{.31}$
23	3837	$\frac{9.0}{9.05}$	$\frac{6.0}{6.12}$	$\frac{4.0}{4.10}$	$\frac{.50}{.48}$	$\frac{2.00}{2.02}$	—	$\frac{2.0}{1.75}$	$\frac{.25}{.27}$
24	3838	$\frac{9.0}{9.33}$	$\frac{6.0}{5.97}$	$\frac{4.0}{4.08}$	$\frac{.50}{.49}$	$\frac{.50}{.49}$	—	$\frac{2.0}{1.70}$	$\frac{.25}{.27}$
25	3839	$\frac{9.0}{9.30}$	$\frac{4.0}{4.02}$	$\frac{4.0}{4.14}$	$\frac{.50}{.50}$	$\frac{.50}{.45}$	—	$\frac{4.0}{3.96}$	$\frac{.25}{.25}$
26	3860	—	$\frac{24.0}{23.60}$	$\frac{4.0}{3.85}$	—	—	—	—	$\frac{.36}{.36}$

TABLE I - Continued

ALLOY NUMBER	HEAT NUMBER*	Cr	Ni	Mo	Mn	Si	W	Co	C
27	3861	—	$\frac{24.0}{23.91}$	$\frac{6.0}{6.03}$	—	—	—	—	$\frac{.30}{.32}$
28	3862	$\frac{2.0}{1.94}$	$\frac{22.0}{21.84}$	$\frac{4.0}{3.85}$	—	—	—	—	$\frac{.30}{.32}$
29	3863	$\frac{4.0}{4.12}$	$\frac{20.0}{20.15}$	$\frac{4.0}{3.95}$	—	—	—	—	$\frac{.30}{.32}$
30	3864	$\frac{6.0}{6.06}$	$\frac{18.0}{17.85}$	$\frac{4.0}{3.98}$	—	—	—	—	$\frac{.30}{.31}$
31	3865	$\frac{2.0}{1.90}$	$\frac{20.0}{19.80}$	$\frac{3.0}{2.97}$	$\frac{1.0}{1.02}$	—	—	—	$\frac{.30}{.31}$
32	3866	$\frac{4.0}{4.15}$	$\frac{18.0}{17.72}$	$\frac{3.0}{3.04}$	$\frac{1.0}{1.03}$	—	—	—	$\frac{.30}{.32}$
33	3867	$\frac{6.0}{6.06}$	$\frac{16.0}{15.89}$	$\frac{3.0}{3.07}$	$\frac{1.0}{1.01}$	—	—	—	$\frac{.30}{.31}$
34	3868	$\frac{2.0}{1.94}$	$\frac{22.0}{21.83}$	$\frac{4.0}{3.86}$	$\frac{1.0}{1.04}$	—	—	$\frac{1.0}{.97}$	$\frac{.30}{.31}$
35	3869	$\frac{4.0}{4.11}$	$\frac{20.0}{19.95}$	$\frac{4.0}{3.92}$	$\frac{1.0}{1.01}$	—	—	$\frac{1.0}{.97}$	$\frac{.30}{.31}$
36	3870	$\frac{6.0}{6.02}$	$\frac{18.0}{17.91}$	$\frac{4.0}{3.97}$	$\frac{1.0}{1.01}$	—	—	$\frac{1.0}{.95}$	$\frac{.30}{.31}$
37	VE-307**	—	$\frac{24.0}{24.2}$	$\frac{4.0}{3.97}$	$\frac{.20}{.25}$	$\frac{.20}{.24}$	—	—	$\frac{.30}{.30}$
38	3889	$\frac{1.0}{.96}$	$\frac{22.0}{21.79}$	$\frac{4.0}{3.79}$	—	—	—	—	$\frac{.30}{.28}$
39	3884	$\frac{2.0}{1.78}$	$\frac{20.0}{20.15}$	$\frac{4.0}{3.86}$	—	—	—	—	$\frac{.30}{.32}$
40	3885	$\frac{1.0}{.94}$	$\frac{24.0}{23.99}$	$\frac{3.0}{2.91}$	—	—	—	—	$\frac{.30}{.30}$
41	3886	$\frac{2.0}{1.81}$	$\frac{24.0}{23.63}$	$\frac{2.0}{2.01}$	—	—	—	—	$\frac{.30}{.30}$

TABLE I - Continued

ALLOY NUMBER	HEAT NUMBER*	Cr	Ni	Mo	Mn	Si	W	Co	C
42	3887	$\frac{1.0}{.96}$	$\frac{22.0}{21.89}$	$\frac{3.0}{2.96}$	-	-	-	-	$\frac{.30}{.30}$
43	3888	$\frac{2.0}{1.85}$	$\frac{20.0}{19.91}$	$\frac{2.0}{2.03}$	-	-	-	-	$\frac{.30}{.30}$
44	3890	$\frac{9.0}{9.16}$	$\frac{8.0}{8.27}$	$\frac{4.0}{4.12}$	$\frac{1.0}{.97}$	$\frac{2.0}{1.86}$	-	-	$\frac{.30}{.31}$
45	3891	$\frac{9.0}{9.01}$	$\frac{8.0}{8.28}$	$\frac{4.0}{4.11}$	$\frac{1.5}{1.48}$	$\frac{2.0}{1.90}$	-	-	$\frac{.30}{.31}$
46	3892	$\frac{9.0}{8.84}$	$\frac{8.0}{7.98}$	-	$\frac{1.0}{.95}$	$\frac{2.0}{1.87}$	$\frac{4.0}{3.86}$	-	$\frac{.30}{.32}$
47	3893	$\frac{9.0}{8.89}$	$\frac{8.0}{8.20}$	-	$\frac{1.5}{1.49}$	$\frac{2.0}{1.96}$	$\frac{4.0}{3.92}$	-	$\frac{.30}{.31}$
48	3894	$\frac{9.0}{9.05}$	$\frac{10.0}{10.24}$	$\frac{4.0}{4.06}$	$\frac{0.5}{.49}$	$\frac{2.0}{1.87}$	-	-	$\frac{.30}{.31}$
49	3895	$\frac{9.0}{8.89}$	$\frac{12.0}{12.18}$	$\frac{4.0}{4.03}$	$\frac{0.5}{.49}$	$\frac{2.0}{1.83}$	-	-	$\frac{.30}{.32}$
50	3896	$\frac{11.0}{11.18}$	$\frac{8.0}{8.31}$	$\frac{4.0}{4.09}$	$\frac{0.5}{.48}$	$\frac{2.0}{1.81}$	-	-	$\frac{.30}{.30}$

S and P are < .010 for all alloys

* Crucible Materials Research Center heat number

** Cannon-Muskegon heat number

MEASUREMENT OF M_s , A_f and \pm' .

The martensite start temperature, M_s , was measured by either of two techniques. For those cases where $-320^\circ\text{F} < M_s < \text{RT}$, acoustic emission was used to determine the M_s . Cubic samples with an edge dimension of about 1/4 inch were slowly cooled from room temperature, and the total integrated noise count was recorded as a function of temperature. For those cases where $M_s > \text{RT}$, dilatometry techniques were used to determine the M_s . A sample about two inches long was heated in a tube furnace at 1500°F under an inert gas, held for 20 minutes, and the furnace removed from the sample. During cooling, the length was recorded on a photographic film as a function of testing temperature.

Dilatometry measurements were also used to determine the temperature, A_f , corresponding to completion of the martensite to austenite transformation. A pre-heated tube furnace was positioned around the sample, and length changes were again recorded on photographic film as a function of temperature.

The amount of martensite formed by cooling to -320°F (liquid nitrogen) was determined by X-ray diffraction measurements using the method outlined by Lindgren.⁽³⁾ The integrated intensities of the (200) and (211) martensite reflections and the (200) and (220) austenite reflections were determined using CrK_α radiation. For a randomly oriented grain structure, the accuracy of this method is about $\pm 1\%$. As a preferred orientation develops, however, large errors can exist.

MECHANICAL PROPERTIES

Tensile testing was done with an Instron testing machine using a strain rate of 2×10^{-2} minute⁻¹. Samples had a reduced gauge length of one inch and were 1/4 in. diameter (unless otherwise stated). All quoted tensile results are an average of two determinations.

Pre-cracked Charpy samples were used to measure K_{Ic} . Standard Charpy V-notch samples (B = W = .394 in.) were pre-cracked to a total crack depth (including the V-notch) of about .12 in. \pm .01 in. using tension-zero fatigue loading. Generally 30,000 to 60,000 cycles were necessary to obtain the desired crack length. Testing of both the pre-cracked samples and Charpy V-notch samples was done on a 240 ft.-lb. Riehle Impact Machine that was equipped with a Model 500 Dynatup System. This instrumentation allows velocity, load, and integrated energy to be measured during the test. Duplicate samples were used for the impact tests. The Riehle machine used for these tests had passed the AMRC performance requirements (specified in ASTM Standard E-23).

RESULTS

Since different types of experimental problems and limitations were encountered with the "A" and "B" alloys, the results obtained with each alloy type will be presented separately.

THE "A" ALLOYS

Following forging and solution heat treatment (i without homogenization) the alloys were characterized by chemical inhomogeneities in the form of a banded microstructure. An example of this is shown in Figure 1, which is of alloy 15. The banding is such that some areas have an M_s above room temperature while most of the volume has an M_s below room temperature. Following homogenization and solution heat treatment, the microstructure of alloy 15 appeared as shown in Figure 2. Although some amount of banding is still present, the homogeneity was significantly improved by the homogenization treatment.

The hardness and structure following homogenization, solution heat treatment, and water quenching to room temperature is given in Table II. Of the first twenty-five alloys evaluated in this program, eleven had an M_s above room temperature, which eliminates these alloys for thermal processing. One of the remaining alloys, number 19, had an M_s below -320°F and was also

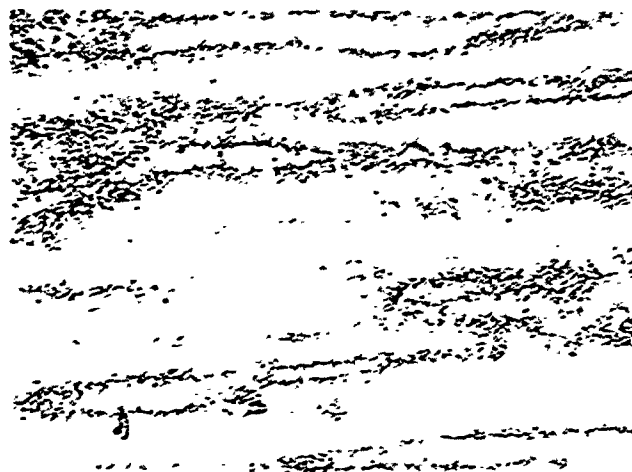


FIGURE 1. MICROSTRUCTURE OF ALLOY 15 FOLLOWING FORGING AND SOLUTION HEAT TREATMENT (BUT WITHOUT ANY HOMOGENIZATION). 100X



FIGURE 2. MICROSTRUCTURE OF ALLOY 15 FOLLOWING FORGING, HOMOGENIZATION AND SOLUTION HEAT TREATMENT. 100X

TABLE II

PHYSICAL PROPERTIES OF "A" TYPE ALLOYS

ALLOY NUMBER	AS- QUENCHED HARDNESS, R _c	AS- QUENCHED STRUCTURE	M _s , °F	HARDNESS AFTER COOLING TO -320°F, R _c	AMOUNT OF α' AFTER COOLING to -320°F, %	A _f , °F
1	14	γ	-81	42	27	1401
2	10	γ	-94	41	78	1398
3	6	γ	-38	42	39	1455
4	10	γ	-80	46	63	1423
5	11	γ	25	49	83	1360
6	36	α'	>RT	49	100	1401
7	9	γ	-94	41	54	1418
8	7	γ	-54	46	87	1409
9	40	α'	>RT	47	99	1335
10	7	γ	-103	39	43	1430
11	9	γ	-80	44	77	1407
12	4	γ	-89	48	68	1412
13	28	α'	>RT	53	83	1468
14	27	α'	>RT	50	86	1432
15	43	α'	>RT	53	83	1456
16	49	α'	>RT	55	93	1499
17	47	α'	>RT	50	100	1509
18	29	α'	>RT	48	100	1421
19	15	γ	<-320	17	0	-
20	4	γ	-78	43	81	1394
21	1	γ	-70	45	82	1387

TABLE II, (Cont'd)

PHYSICAL PROPERTIES OF "A" TYPE ALLOYS

ALLOY NUMBER	AS- QUENCHED HARDNESS, R _c	AS- QUENCHED STRUCTURE	M _s , °F	HARDNESS AFTER COOLING TO -320°F, R _c	AMOUNT OF α' AFTER COOLING to -320°F, %	A _f , °F
22	4	γ	-49	37	71	1412
23	30	α'	>RT	50	84	1459
24	46	α'	>RT	49	100	1447
25	50	α'	>RT	50	98	1483
44	16	γ	<-320	-	0	-
45	16	γ	<-320	-	0	-
46	8	γ	<-320	-	0	-
47	12	γ	<-320	-	0	-
48	15	γ	<-320	-	0	-
49	14	γ	<-320	-	0	-
50	20	γ	<-320	-	0	-

eliminated. The remaining thirteen alloys had M_s values between 25°F and -103°F, which is compatible with the thermal processing technique. The M_s values were measured by acoustic emission techniques as described in the previous section. For most of the alloys, the M_s could not simply be taken as the observed temperature (on cooling) corresponding to the initial noise detection (martensite formation), because chemical inhomogeneities of the type shown in Figure 2 produced a small amount of martensite formation at temperatures higher than the bulk M_s . As a result, some extrapolation was necessary to obtain the reported M_s values, and due to this extrapolation many of the reported M_s values are probably no more accurate than $\pm 20^\circ\text{F}$. After cooling to -320°F to form martensite, the hardness of these thirteen alloys was in the range of 37-49 R_c , which is appreciably harder than can be obtained with the "B" alloys after the first martensite formation (30-33 R_c). The amount of martensite formed by the cooling to -320°F is also shown in Table II; this data was determined by X-ray diffraction, as discussed earlier. For some alloys such as 1, 3, 7 and 10, the amount of martensite observed metallographically did not correlate with the amount determined by X-ray diffraction. It was assumed that the presence of textures led to errors in the X-ray diffraction determinations of these values.

The dilatometry measurements of the temperature, A_f , corresponding to the completion of the martensite to austenite transformation indicated that $1360^\circ\text{F} < A_f < 1455^\circ\text{F}$ for the thirteen alloys of interest, as shown in Table II. This again appeared to be compatible with the thermal processing technique. However, on cooling to room temperature after heating to 1500°F (which is above A_f), each of the thirteen alloys was now martensitic indicating that the M_s had increased to above room temperature. Table III shows values of the martensite start temperature, M'_s , measured in reverted austenite. The increase, ΔM_s , in the martensite start temperature was approximately 200 to 300°F for these alloys.

TABLE III

 M_s Data of Selected "A" Type Alloys

<u>ALLOY NUMBER</u>	<u>M_s, °F</u>	<u>M'_s, °F</u>	<u>ΔM_s, °F</u>
1	-81	154	235
2	-94	176	270
3	-38	165	203
4	-80	158	238
5	25	223	198
7	-94	165	259
8	-54	161	215
10	-103	172	275
11	-80	215	295
12	-89	188	277
20	-78	158	236
21	-70	161	231
22	-49	134	183

A few additional measurements were made to confirm this increase in M_s . Samples of alloys 4 and 7 were solution heat treated, cooled to -320°F to form martensite and returned to room temperature. X-ray diffraction measurements of the diffraction angle, (2θ) , of the (110), (200) and (211) reflections of the martensite were determined using CrK_α radiation. The samples were then reverted at 1500°F , cooled to room temperature and the same measurements re-determined. The results are shown in Table IV and the diffraction angles of the martensite that forms from reverted austenite is seen to be essentially identical to the martensite formed from annealed austenite. Thus, the crystallography of the two martensites are concluded to be equivalent.

Metallographic observations were also used to evaluate the martensites. Figure 3 shows alloy 7 after the initial cooling to -320°F , and the structure is seen to contain plate martensite and austenite. After reversion at 1500°F and cooling to room temperature, the structure appeared as shown in Figure 4, new martensite and virgin austenite can be clearly seen as well as dark areas that are either reverted austenite or new martensite formed from reverted austenite. The metallographic appearance is quite similar to that previously observed by Koppelaar⁽²⁾ in the "B" type TRIP steels. With the metallographic and X-ray diffraction results, it was concluded that the increase in M_s was a confirmed observation.

Since the increase in M_s to above room temperature would eliminate all of the remaining thirteen alloys from the thermal processing technique, a number of experiments were performed in an attempt to lower M'_s below room temperature. The first attempt consisted of cycling the thirteen alloys between -320°F (martensite) and 1500°F (reverted austenite) for five complete reversion cycles; it was hoped that additional cycling would stabilize the austenite. After the fifth reversion at 1500°F , however, all of the alloys were still martensitic on cooling to room temperature.

TABLE IV

DIFFRACTION ANGLE (2θ) OF VARIOUS MARTENSITE REFLECTIONS

<u>ALLOY</u>	<u>THERMAL TREATMENT</u>	<u>$(110)_{\alpha'}$, 2θ, DEGREES</u>	<u>$(200)_{\alpha'}$</u>	<u>$(211)_{\alpha'}$</u>
4	-320°F	68.7	106.3	155.0
4	-320°F plus reverted	68.9	106.5	155.6
7	-320°F	68.4	105.8	153.8
7	-320°F plus reverted	68.4	105.9	154.5

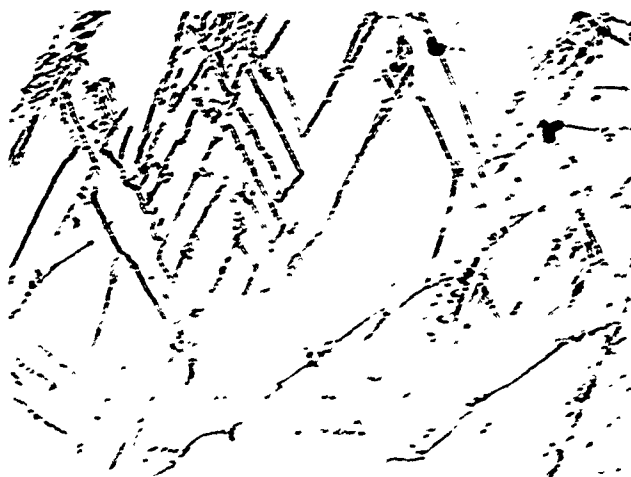


FIGURE 3. MICROSTRUCTURE OF ALLOY 7
AFTER INITIAL COOLING TO -320°F SHOWING PLATE MARTENSITE
AND AUSTENITE. 1000X



FIGURE 4. MICROSTRUCTURE OF ALLOY 7 AFTER
COOLING TO -320°F , REVERSION AT 1500°F AND COOLING TO
ROOM TEMPERATURE.
1000X

The only method that was observed to keep the M'_S below room temperature was by the use of higher reversion temperatures. Samples of the thirteen alloys were reverted at progressively higher temperatures (using a different sample for each reversion) until the M'_S was below room temperature. The results are summarized in Table V, which shows the hardness and structure for the various reversion temperatures. Table V shows that eleven of the alloys could be reverted at temperatures between 1775°F and 1850°F and remain austenitic on cooling to room temperature (the M'_S of the other two alloys, 5 and 21, remained above room temperature for reversion temperatures as high as 1900°F). These eleven alloys were cycled through five cycles between -320°F and the reversion temperature established by this method. The hardness and structure after the fifth complete reversion cycle is shown in Table VI, and only three of the alloys (7, 10 and 22) were austenitic at room temperature after the five cycles. However, the hardness of these three alloys was too low (24-31 Rc) to be of interest.

A number of processing variables were evaluated in an attempt to decrease M'_S to below room temperature, but none of these were successful. A short summary of each of these attempts follows:

1. The solution heat treating temperature was varied from 1800°F to 2150°F.
2. The cooling rate from the solution heat treating temperature was varied by water quenching, air cooling and furnace cooling.
3. A martensite formation temperature of -100°F was used to vary the amount of martensite produced during the first $\gamma \rightarrow \alpha'$ transformation.

TABLE V

HARDNESS AND STRUCTURE OF SELECTED "A" ALLOYS AFTER
REVERSION AT VARIOUS TEMPERATURES

ALLOY NUMBER	HARDNESS, Rc, AND STRUCTURE AFTER REVERSION AT INDICATED TEMPERATURE					
	1500°F	1750°F	1775°F	1800°F	1850°F	1900°F
1	45 α'	28 α'	31 v			
2	43 α'	29 α'		31 α'	20 v	
3	43 α'	28 α'	26 α'	16 v		
4	43 α'	24 α'	21 v			
5	44 α'	22 α'		26 α'	17 α'	17 α'
7	43 α'	27 α'	27 v			
8	42 α'	22 α'		29 α'	13 v	
10	43 α'	22 α'	30 v			
11	45 α'	22 α'		15 α'	12 v	
12	45 α'	20 α'		19 α'	19 v	
20	42 α'	19 α'	20 v			
21	43 α'	11 α'		4 α'	17 α'	5 α'
22	39 α'	18 α'	15 v			

TABLE VI

HARDNESS AND STRUCTURE OF SELECTED "A" ALLOYS AFTER
FIVE REVERSION CYCLES

ALLOY NUMBER	REVERSION TEMPERATURE, °F	HARDNESS AFTER FIVE REVERSION CYCLES, R _C	STRUCTURE AFTER FIVE REVERSION CYCLES
1	1775	33	α'
2	1850	26	α'
3	1800	24	α'
4	1775	19	α'
7	1775	24	γ
8	1850	7	α'
10	1775	31	γ
11	1850	11	α'
12	1850	2	α'
20	1775	20	α'
22	1775	24	γ

4. Thermal treatments between the -320°F martensite formation and the reversion treatment were also varied. In some tests samples were withdrawn from the liquid nitrogen container and placed directly into the salt pot at the reversion temperature (1500°F). In other cases, the martensitic samples were given a tempering at 1100°F for one hour, air cooled and reverted.
5. Heating rate to the reversion temperature was varied between direct immersion into a liquid salt and placement of the samples in an air furnace.
6. The reversion time was varied between 30 seconds and 60 minutes.
7. Post-reversion treatments consisting of direct cooling to temperatures of either 800, 600 or 400°F for 1 to 16 hours were evaluated.
8. The cooling rate after reversion was varied by water quenching, air cooling and furnace cooling.

In summary, the only method established for keeping M'_S below room temperature was the use of high reversion temperatures (1775 - 1850°F), and this method had the disadvantage of producing low hardness responses (probably because of recovery occurring at the high reversion temperatures).

Near the end of this program, seven additional "A" type alloys (numbers 44-50) became available for evaluation. The objective with these alloys was to decrease M_S (and hopefully M'_S) of alloys similar to types 1 and 10 by about 200°F . In this way, both M_S and M'_S would be in the desired range (below room temperature and above -320°F). However, alloys 44-50 all had

an $M_s < -320^\circ\text{F}$. (See Table II). This observation was obviously unexpected. Comparing alloy 44 with alloy 1, the only difference is 0.97% Mn in alloy 44 as compared to .50% in alloy 1. This increase of 1/2% Mn lowered the M_s from -81°F in alloy 1 to $< -320^\circ\text{F}$ in alloy 44. Therefore, the M_s is decreased by at least 473°F for one weight percent manganese in this particular base composition; by comparison, Andrews⁽⁴⁾ showed that in other base compositions the change in M_s is about -54°F for one weight percent manganese. This will be discussed in more detail in the Discussion section.

THE "B" ALLOYS

After it was established that none of the original twenty-five "A" type of alloys evaluated on this program would be suitable for thermal processing, it was decided to evaluate alloy modifications of the "B" type. Alloys 26-37 were the next group to be evaluated. Table VII shows various physical properties of these alloys. All the alloys had an $M_s < \text{RT}$, but ten of the twelve also had an $M_s < -452^\circ\text{F}$. Thus, only two of the twelve alloys had a suitable M_s for thermal processing. These two alloys, 26 and 37, were cycled between martensite and reverted austenite using various temperatures, and the austenite hardness for both alloys at various stages of the cycling is given in Table VIII. The data in this table indicates that neither varying the martensite formation temperature between -320°F and -452°F (liquid helium) nor varying the reversion temperature between 1250°F and 1300°F significantly affects the hardness of reverted austenite. It was also observed that alloy 37 did not completely revert to austenite at 1250°F . Based upon these results, a martensite formation temperature of -320°F and a reversion temperature of 1300°F were selected as the standard cycling temperatures for future processing. Following five cyclical reversions, the M'_s of alloys 26 and 37 were measured and the results are given in Table IX. The increase in M_s due to thermal cycling in these "B" alloys is seen to be about $30-50^\circ\text{F}$, which is

TABLE VII

PHYSICAL PROPERTIES OF "B" TYPE ALLOYS

ALLOY NUMBER	AS-QUENCHED HARDNESS, R_B	AS-QUENCHED STRUCTURE	M_s , °F	HARDNESS AFTER COOLING TO -320°F, R_C
26	73	Y	-90	32
27	80	Y	<-452°F	-
28	74	Y	<-452°F	-
29	76	Y	<-452°F	-
30	79	Y	<-452°F	-
31	70	Y	<-452°F	-
32	73	Y	<-452°F	-
33	78	Y	<-452°F	-
34	71	Y	<-452°F	-
35	72	Y	<-452°F	-
36	76	Y	<-452°F	-
37	65	Y	-104	33
38	69	Y	-320°F < M_s < RT	26
39	72	Y	-320°F < M_s < RT	15
40	67	Y	-320°F < M_s < RT	20
41	63	Y	-320°F < M_s < RT	79 (R_B)
42	68	Y	-320°F < M_s < RT	34
43	68	Y	-320°F < M_s < RT	37

TABLE VIII

AUSTENITE HARDNESS OF ALLOYS 26 and 37

AFTER VARIOUS REVERSION TREATMENTS

AUSTENITE HARDNESS, R_c

<u>PROCESSING STEP</u>	<u>ALLOY 26 NOTE 1</u>	<u>ALLOY 26 NOTE 2</u>	<u>ALLOY 26 NOTE 3</u>	<u>ALLOY 37 NOTE 1</u>
Annealed	(73R _B)	(73R _B)	(73R _B)	(65R _B)
1st Reversion	29-1/2	29	29-1/2	28
2nd Reversion	36-1/2	35-1/2	37	35
3rd Reversion	40	39	40	38
4th Reversion	41-1/2	41	41	40
5th Reversion	42	42	42	41
6th Reversion	41-1/2	42	40	
7th Reversion	41			

Note 1: Cycled between -320°F and 1300°F

Note 2: Cycled between -320°F and 1250°F

Note 3: Cycled between -452°F and 1250°F

TABLE IX

 M_s VALUES OF ALLOYS 26 AND 37

ALLOY	$M_s,$ °F	$M'_s,$ °F	$\Delta M_s,$ °F
26	-90	-36	54
37	-104	-71	33

appreciably less than that observed in the "A" alloys.

The microstructures of alloys 26 and 37 following five cyclical reversion are shown in Figures 5 and 6, respectively. The amount and appearance of the reverted austenite is similar to that previously noted.⁽²⁾ While alloy 37 was homogeneous with respect to the microstructure shown in Figure 6, the microstructure of alloy 26 also showed areas of virgin austenite as shown in Figure 7. At high magnification, these areas of virgin austenite were observed to contain numerous small precipitates as shown in Figure 8.

In the first series of tensile tests with alloys 26 and 37, tensile samples with a 1/8 inch diameter reduced section were machined prior to cycling. The results obtained with these samples are shown in Table X, and a number of features are noted. While the yield and tensile strength of the two alloys after five reversion are nearly the same, the elongation values differ greatly. This could be due to either the higher carbon content of alloy 26 (which would increase the stability of the austenite with the result that less martensite forms during testing) or the non-homogeneous structure shown in Figure 7. The strength levels obtained with alloy 37 are similar to those previously observed⁽²⁾ in .060 inch thick samples but lower than that observed in ~1/4 inch thick samples. Another series of tensile samples of alloy 37 were cyclically reverted using samples with a 1/4 inch diameter reduced section and the results are shown in Table XI. Both the yield and tensile strength are seen to increase in the thicker samples. This thickness effect has been previously established⁽²⁾, and an explanation for the effect still is not known. All later tensile tests were made with samples with a 1/4 inch diameter reduced section. Table XI also shows that processing through seven reversion cycles increases the tensile strength but decreases both the yield strength and the elongation.



FIGURE 5, MICROSTRUCTURE OF ALLOY 26 AFTER FIVE CYCLICAL REVERSIONS SHOWING ALMOST ALL REVERTED AUSTENITE. 100X

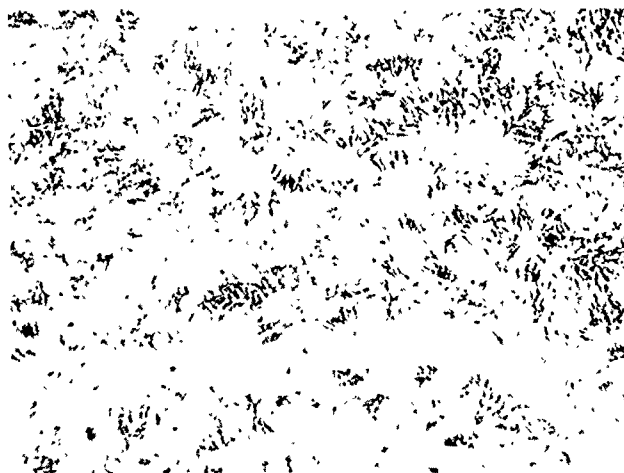


FIGURE 6. MICROSTRUCTURE OF ALLOY 37 AFTER FIVE CYCLICAL REVERSIONS SHOWING NEARLY ALL REVERTED AUSTENITE, ALTHOUGH SMALL PATCHES OF VIRGIN AUSTENITE ARE PRESENT. 100X

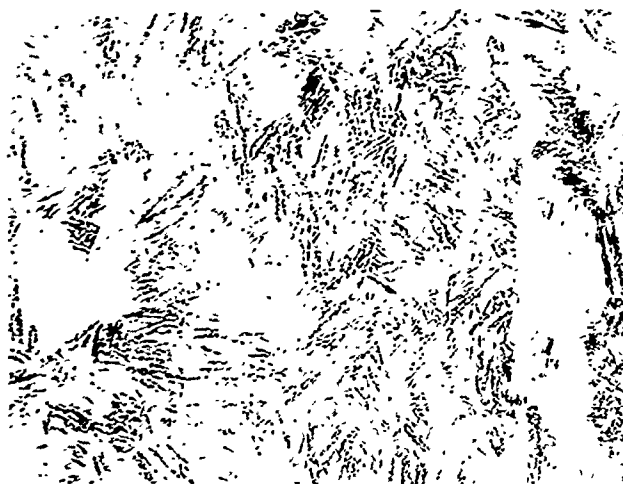


FIGURE 7. MICROSTRUCTURE OF ALLOY 26 AFTER FIVE CYCLICAL REVERSIONS SHOWING LARGE ISOLATED PATCHES OF VIRGIN AUSTENITE WITHIN A MATRIX OF REVERTED AUSTENITE. 50X



FIGURE 8. MICROSTRUCTURE OF ALLOY 26 AFTER FIVE CYCLICAL REVERSIONS SHOWING PRECIPITATES IN THE VIRGIN AUSTENITE. 2500X

Table A

TENSILE PROPERTIES OF ALLOYS 26 and 37
FOLLOWING THERMAL CYCLING

ALLOY	THERMAL TREATMENT	YIELD STRENGTH, ksi	TENSILE STRENGTH, ksi	ELONGATION %	REDUCTION IN AREA, %
26	Solution Heat Treated	27.3	81.3	44	83
26	1 Reversion Cycle	82.5	133.2	21	61
26	5 Reversion Cycles	152.4	174.0	12	20
37	Solution Heat Treated	32.0	89.4	37	77
37	1 Reversion Cycle	80.2	128.5	22	70
37	5 Reversion Cycles	147.3	178.0	30	59

Note: All data in this Table obtained with tensile samples with a 1/8 inch diameter in reduced section.

TABLE XI

TENSILE PROPERTIES OF ALLOY 37

<u>THERMAL TREATMENT</u>	<u>YIELD STRENGTH, ksi</u>	<u>TENSILE STRENGTH, ksi</u>	<u>ELONGA- TION, %</u>	<u>REDUCTION IN AREA, %</u>
Solution heat treated	33.6	92.7	50	79
One Reversion Cycle	84.8	130.9	40	66
Three Reversion Cycles	136.9	176.4	40	50
Five Reversion Cycles	160.0	196.5	30	40
Seven Reversion Cycles	147.2	208.1	25	42

Tensile samples of alloy 37 processed through five reversion cycles were tested at various testing temperatures with the objective of determining the M_D (defined as the highest temperature at which the austenite to martensite transformation can be strain induced). The results are shown in Table XII, and both the elongation on magnetic response indicates that $150^\circ\text{F} < M_D < 175^\circ\text{F}$.

A number of pre-cracked Charpy samples of alloys 26 and 37 were evaluated in various conditions. Table XIII shows values of the dynamic plane strain fracture toughness, K_{Id} , and the total absorbed impact energy per unit area, $\frac{W}{A}$, obtained with the pre-cracked samples. After five reversion cycles, the K_{Id} of alloy 37 is seen to be greater than that of alloy 26. This correlates with the better strength-elongation values obtained in alloy 37 shown in Table X. Another important feature seen in Table XIII is that the value of K_{Id} is the same for samples in the longitudinal (RW) and transverse (WT) orientations. This demonstrates one of the principle advantages of the thermal processing technique as compared to thermomechanical processing.

Charpy V-notch samples of alloy 37 processed through five reversion cycles were tested at 72 and -40°F and the results are shown in Table XIV. The impact energy is seen to be essentially temperature independent, unlike quench and tempered steels at these strength values. Table XIV also shows that the dynamic yield strength is 193 ksi at room temperature (the instrumented Charpy test can be used to determine the dynamic yield strength under proper conditions). This represents an increase of 33 ksi over the static yield strength (the loading rate was about 1000 inches/minute for the dynamic tests and .02 inches/minute for the static-tests).

The effect of post-reversion heat treatments on the tensile properties was investigated using samples of alloy 37 processed through five

TABLE XII

TENSILE PROPERTIES OF ALLOY 37 AFTER
PROCESSING THROUGH FIVE REVERSION CYCLES

TESTING TEMPERATURE °F	YIELD STRENGTH, ksi	TENSILE STRENGTH, ksi	ELONGA- TION %	REDUCTION IN AREA %	MAGNETIC RESPONSE AFTER TEST
72	160.0	196.5	30	40	Magnetic
150	147.4	161.8	30	60	Magnetic
175	150.0	172.8	15	53	Non-Magnetic
200	154.9	170.7	14	55	Non-Magnetic
300	150.7	168.5	14	52	Non-Magnetic

TABLE XIII

IMPACT RESULTS WITH PRE-CRACKED CHARPY SAMPLES
OF ALLOYS 26 and 37

ALLOY	NUMBER REVERSION CYCLES	SAMPLE ORIENTATION	K_{Id} , ksi-(in) ^{1/2}	$\frac{W}{A}$, in-lb/in ²
26	1	RT	140	6458
26	2	RT	121	3362
26	5	RT	73	1322
37	1	RT	129	5108
37	3	RT	83	2003
37	5	RT	89	1467
37	7	RT	89	1513
37	5	WT	89	1440

TABLE XIV

CHARPY V-NOTCH TEST RESULTS WITH ALLOY 37

SAMPLES PROCESSED THROUGH FIVE REVERSIONS

TEST TEMPERATURE, °F	IMPACT ENERGY, ft.-lbs	DYNAMIC YIELD STRENGTH, ksi	STATIC YIELD STRENGTH, *
72	28.1	193.0	160.0
-40	27.7	195.3	

* From Table XI

reversion cycles. Following the fifth reversion at 1300°F, samples were heat treated at 1350°F for either 5 minutes or 60 minutes. The results are shown in Table XV. The five minute treatment produced a very small decrease in yield and tensile strength and a small increase in elongation. Following the 60 minute treatment, both the yield strength and elongation decrease as compared to the as-reverted condition. Data from Table XI for 3 and 7 reversion cycles is included in Table XV for comparison. The post-reversion heat treatments evaluated here do not appear to improve upon the strength-elongation relationship that can be achieved by reversion alone.

As a summary, a number of mechanical and physical properties of alloy 37 are shown in Table XVI.

Near the end of this program, six additional "B" type alloys, number 38-43, became available for evaluation. Some physical properties of these alloys are shown in Table VII, and all of the alloys had an H_S in the desired range, $-320^\circ\text{F} < H_S < \text{RT}$. However, only three of these, (38, 42 and 43) had hardness levels of interest ($>26\text{R}_C$) after cooling to -320°F . Samples of these three alloys were cyclically processed between -320°F and 1300°F . The 1300°F reversion temperature was selected from the results obtained with alloys 26 and 37. Table XVII shows the hardness of alloys 38, 42 and 43 for various reversions, and it is seen that maximum values of about 41R_C were obtained after 5 to 7 reversions. This is about the same level as previously obtained with alloys 26 and 37. These results were obtained during the last week of the program and additional evaluations (such as H_S , H'_S and A_f measurements and tensile and impact tests) could not be made.

TABLE XV

THE EFFECT OF POST-REVERSION HEAT TREATMENTS
ON THE TENSILE PROPERTIES OF ALLOY 37 PROCESSED
THROUGH FIVE REVERSION CYCLES

NUMBER OF REVERSION CYCLES	POST-REVER- SION HEAT TREATMENT	YIELD STRENGTH, ksi	TENSILE STRENGTH, ksi	ELONGATION %	REDUCTION IN AREA, %
5	None	160.0	196.5	30	40
5	1350°F, 5 min.	156.0	192.1	32-1/2	38
5	1350°F, 60 min.	136.9	196.3	27	41
3	None	136.9	176.4	40	50
7	None	147.2	208.1	25	42

TABLE XVI

MECHANICAL AND PHYSICAL PROPERTIES OF

ALLOY 37

Yield Strength	160.0 ksi (static), 193.0 ksi (dynamic)
Tensile Strength	195.5 ksi
Elongation	30%
Reduction in Area	40%
K_{Id}	89 ksi-(in) ^{3/2} in both RT and WT orientations
$\frac{W}{A}$	1467 in-lb/in ² RT orientation, 1440 in-lb/in ² WT orientation
C_v Impact Strength	28.1 ft-lbs at 72°F, 27.7 ft-lbs at -40°F
M_s (annealed austenite)	-104°F
M_s (reverted austenite)	-71°F
M_D	150°F < M_D < 175°F
A_f	1250°F < A_f < 1300°F

Note: Except as noted, all data obtained following five cyclical reversions between -320°F and 1300°F.

TABLE XVII

AUSTENITE HARDNESS OF ALLOYS 38, 42 and 43
AFTER VARIOUS REVERSION TREATMENTS

PROCESSING STEP	AUSTENITE HARDNESS, R _c		
	ALLOY 38	ALLOY 42	ALLOY 43
1st Reversion	23	28	27
2nd Reversion	33	36	34
3rd Reversion	37	39	37
4th Reversion	38	40	39
5th Reversion	38	41	39
6th Reversion	40	41	39
7th Reversion	39	41	39

SURFACE HARDENING TREATMENTS

Since several potential applications for TRIP steel would require a high surface hardness, both nitriding and hard chrome plating surface hardening treatments were evaluated. Nitriding case-hardens steel directly through the formation of nitrides in a surface layer of metal. Nitrogen is introduced at moderate temperatures (generally 950°F to 1050°F), and the processing requires no subsequent heat treatment. Thus, it is compatible for use with thermally processed TRIP steels. Samples of alloy 37 processed through five cyclical reversions were subjected to three different commercial nitriding processes: (1) standard gas nitriding, (2) Malcomizing* and (3) Tuf-triding*. Upon evaluation, however, it was found that none of these treatments proved effective. The gas nitriding produced only a thin nitrogen rich case that was severely cracked and irregular. Both the Malcomizing and the Tuf-triding produced cases which appeared sound; however, the nitrogen rich layers were not harder than the base alloy (41R_C). It is felt that the absence of good nitride forming alloying elements was responsible for the lack of hardening in the case. In order for nitriding to be effective it is known that steels must contain aluminum, chromium, molybdenum or vanadium additions to form nitrides. It was hoped that alloy 37 (Fe-24Ni-4Mo-0.30C) contained enough molybdenum to produce a hard case. Apparently the molybdenum was not free to form nitrides in this alloy. The 9Cr-8Ni-4Mo-2Mn-2Si-.30C series TRIP steels should respond to nitriding because of their relatively high chromium content.

* Malcomizing, Tuf-triding and Electrolyzing are commercial trade names. The companies supplying these services consider them to be propriety processes and details of the processing are not available.

Finally, hard chrome plating was evaluated as an alternate type of case hardening. The method used was a commercial process called Electrolyzing,* which consists of electrolytically depositing chromium at temperatures of about 200°F. This process produced a hardened surface at about 65-70 R_c on reverted samples of alloy 37. However, when a plated sample 1/4 inch thick was bent through an angle of 135°, the plating did not adhere to the base metal. Additional work may help solve this bonding problem.

* See note on page 41

DISCUSSION

The primary objective of this program was to develop higher strength TRIP steels using the thermal processing technique. This goal was not achieved, primarily because of the large increase in the M_s of reverted austenite as compared to annealed austenite and to a less extent to the large compositional dependency of the M_s on some of the alloying elements.

The increase in M_s (about 200 to 300°F) that was observed with the "A" alloys was totally unexpected since no shifts larger than about 50°F have been reported in the literature.⁽⁵⁾ Changes in M_s due to prior plastic straining have been studied in detail in Fe-Ni-C alloys and maximum changes of about 50°F (decreases as well as increases) have been reported.^(5,6,7) The Fe-Ni-Mo-C "B" alloys evaluated in this program also showed a relatively small change; the M_s of alloy 26 increased about 54°F and the M_s of alloy 37 increased about 33°F following reversion. The cause of the large increase in M_s due to reversion (strain hardening) in the "A" alloys is not known. A further complicating observation is that following the normal thermo-mechanical processing (80% reduction at 800-1000°F) in the standard "A"

alloy (Fe-9Cr-8Ni-4Mo-2Si-2Mn-.30C) the $M_s < -452^\circ\text{F}$. Thus, the increase in M_s due to strain hardening introduced by reverse shear transformations may not occur when strain hardening is accomplished by mechanical working. Without this large shift in M_s observed in the "A" alloys, it is thought that higher strength TRIP steels would have been developed on this program. This is based upon the higher hardness of the "A" alloys in both the solution heat treated and martensitic condition (-320°F treatment) as compared to the "R" alloys.

The second unexpected feature that was observed in this program was the large effect of various alloying elements on the M_s . As briefly commented upon in the Results Section, manganese lowered the M_s in the "A" alloys approximately an order of magnitude larger than has been reported previously. One of the secondary goals of this program was to establish the effects of alloying elements in the "A" type alloys on the M_s and A_f . Empirical equations such as those given by Andrews⁽⁴⁾ were developed based upon data obtained with alloys 1-25. The equations were assumed to be of the form,

$$Y = A_0 + A_1X_1 + A_2X_2 + \dots + A_nX_n,$$

where Y is the temperature of interest (M_s , M'_s or A_f), $X_i = X_1$ through X_n is the percent by weight of an alloying element and $A_i = A_0$ through A_n is the coefficient giving the effect of each alloying element. A computer program was utilized to perform multiple linear regression analyses based upon a least squares method to determine the A_i coefficients. The composition of each alloy and the experimental values for M_s , M'_s and A_f used in this analysis are those given in Tables I, II and III. For the determination of the M_s coefficients, only the thirteen alloys having an M_s between room temperature and -320°F were used, and the M'_s and A_f coefficients were determined with the twenty-four alloys having an $M_s > -320^\circ\text{F}$ (all but alloy 19).

A few difficulties arose during this analysis. Some of the alloying elements did not vary significantly enough in content for a reasonable computer solution. For example, the chromium content varied only between 8.95% and 9.21% in the thirteen alloys used for determining the M_s coefficients. When this situation arose, a value was assigned for the coefficient of the alloying element in question. The value for the coefficient was chosen based upon both those coefficients reported by Andrews⁽⁴⁾ and the coefficients of other alloying elements developed by the computer analysis. Following assignment of these coefficients, the computer program was used to determine the coefficients for the remaining alloying elements. This procedure of assigning values for some of the coefficients introduced a small change (less than 5°F per weight percent of alloying element) in the computer determined coefficients.

Another difficulty was that for certain alloys the predicted values of Y using the coefficients that were determined were significantly different than the measured values. It is felt that this was probably a result of chemical segregation, either in the form of banding as previously noted or in the form of precipitation (which effectively changes the chemistry of the matrix). In order to improve the accuracy of the computer predicted coefficients, the 3 or 4 alloys (depending upon which value of Y is being considered) that exhibited the greatest disparity between the predicted and experimental temperatures were excluded from the data and the coefficients were re-determined with the computer program. This again resulted in only minor changes in the value of the coefficients. The coefficients that resulted from the above procedures are listed in Table XVIII together with the standard deviation obtained for the predicted versus measured temperatures. Using the coefficients listed in the table, the average difference between the calculated and experimental values for M_s , M'_s and A_f is $\pm 15^\circ\text{F}$, $\pm 12^\circ\text{F}$ and $\pm 14^\circ\text{F}$, respectively. The table also shows the values of the coefficients developed by Andrews⁽⁴⁾ for M_s and A_{c_3} (which might be

TABLE XVIII

COMPUTER GENERATED COEFFICIENTS FOR THE EFFECT
OF ALLOYING ELEMENTS ON M_s , M'_s and A_f

ALLOY ELEMENT	COEFFICIENTS, °F per wt. % alloying element				
	M_s	M'_s	A_f	M_s , Andrews (4)	A_{c3} , Andrews (4)
Cr	-125.0A	-54.5	+9.8	-21.7	-19.8
Ni	-117.5	-66.2	-18.9	-31.8	-27.8
Mo	-21.5	-11.4	+10.4	-13.5	+56.7
Mn	-660.0	-316.9	-54.0	-54.7	-54.0
Si	+5.4	+5.4	+15.6	0	+80.4
W	-24.6	-7.6	+11.8	-9	+23.6
Co	+8.2	-1.0	-0.2	+27	-
C	-300.0A	-100.0A	-200.0A	-761.4	-365.4/C
A_o , °F	2497	1412	1494		
Standard Deviation, σ	<u>+18.97</u>	+15.32	<u>+20.90</u>		

A = assumed value

expected to be somewhat analogous with A_F). Some alloying elements are seen to have about the same coefficient while other elements do not (the effect of manganese on the M_s for example). It is obvious that the effect of the various alloying elements in the "A" TRIP steel matrix is significantly different than in the alloys used by Andrews⁽⁴⁾. Although this was responsible, in part, for not meeting the goals of the program, the data is now available for future work in the "A" type alloys.

RECOMMENDATIONS

Using the analysis of the effect of alloying elements on the M_s and M'_s given in the previous section, it seems likely that additional alloying development work with the "A" type alloys could produce compositions that are compatible with the thermal processing technique. The approach that appears most likely to succeed is to totally eliminate Mn as an alloying element and use Cr, Ni or Mo to adjust the M_s and M'_s to the desired ranges. Not only would it be difficult to keep the Mn at the required composition (probably something like $0.80 \pm .05\%$), but Mn also appears to influence the increase in M_s to M'_s . By eliminating Mn it should then be easier to both control the M_s and lower the difference between M_s and M'_s . Once the M_s and M'_s are adjusted to the desired ranges, it appears that thermal processing of the "A" alloys should produce strength levels superior to the "B" alloys. This conclusion is based upon the hardness of martensite after cooling to -320°F in alloys with an $M_s > -320^\circ\text{F}$. The "B" alloys have hardness values of 30-35 R_c after the -320°F treatment, but the hardness of the "A" alloys is in the 40-49 R_c range. Based upon these factors, it is recommended that an alloy development program be undertaken using the general approach described above.

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