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1966-12-01

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John S. Dunning
December 1966

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Berkeley, California

AEC Contract No. W-7405-eng-48

# THE MICROSTRUCTURE AND PROPERTIES OF METASTABLE AUSTENITIC HIGH STRENGTH STEELS

John S. Dunning

(M. S. Thesis)

December 1966

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# THE MICROSTRUCTURE AND PROPERTIES OF METASTABLE AUSTENITIC HIGH STRENGTH STEELS

#### John S. Dunning

Inorganic Materials Research Division, Lawrence Radiation Laboratory,
Department of Mineral Technology, College of Engineering,
University of California, Berkeley, California

#### ABSTRACT

A study was made of the effects of composition and thermal mechanical treatment on the structure and properties of metastable austenitic steels. Two topics were studied in detail: (a) the cyclic quenching of austenite where the transformation of austenite is accomplished thermally and (b) the composition dependence of transformation induced plasticity where strain induced transformation occurring during tensile testing favors superior ductilities.

The cyclic quenching of deformed austenite was effective in producing a fine martensite plate size and the fineness of the microstructure resulted in high strengths. Optimum strength-ductility ratios were obtained after two or three cycles of quenching and tempering.

The strain induced austenite-to-martensite transformation was utilized in a simple Fe-Ni-Mo-C alloy to produce enhanced ductilities. The occurrence of the strain induced transformation during tensile testing was related to the percentage of martensite present prior to testing.

#### I. INTRODUCTION

Plastic straining below the recrystallization temperature is the most common method of increasing the strength of metals. However, this increase in strength is usually accomplished at the expense of ductility. The desirability of achieving high strength while retaining high ductility has led to the development of thermal-mechanical treatments which in certain ferrous alloys result in a good balance of strength and ductility.

In the conventional cold working of steels the increase in strength during deformation is achieved by blocking the motion of dislocations. Electron microscopy indicates that dislocations tend to collect in tangled networks in plastically deformed materials. The motion of dislocations generated during further deformation is inhibited by the network and increased stress is required to cause further plastic flow. While cold work can decrease the distance between obstacles to dislocation motion there is a minimum distance attainable and hence a limit to the tensile yield strengths that can be obtained in this way. Further, since the strength is inversely proportional to the distance a dislocation moves, a yield strength increase is almost always attended by a decrease in ductility.

Maraging alloys also possess superior strength and ductility ratios.

In contrast to steels, it is believed that these alloys are strengthened by the presence of finely dispersed precipitates in the form of ordered compounds. Mechanical-thermal processing of these alloys does not improve the strength and ductility as in the case of steels. Improvement in properties has been generally obtained by composition control.

The compromise between strength and ductility associated with conventional cold working is undesirable and is minimized in thermal mechanical processes such as "ausforming". In the ausforming process metastable

austenite is deformed without noticeable recrystallization, prior to transformation to martensite. Ausformed steels are the strongest available commercial steels. Any alloy, system exhibiting a "bay" region in the T-T-T diagram can potentially be deformed in this region and hence is ausformable. Strengthening is thought to be due primarily to the high dislocation density produced; an additional but lesser factor being that of strengthening by the carbide dispersion formed during the deformation of austenite. Deformation in the ausforming process is thought to accelerate diffusion of substitutional elements so that they can form finely dispersed precipitates, usually alloy carbides, at relatively low temperatures.

A thermal-mechanical process developed in this laboratory 4,5 appears to have promise for the production of microstructures that are favorable for high strength and high ductility. Austenite deformed in a manner similar to that in the ausform process is quenched just below the  $\mathbf{M}_{\mathbf{S}}$  temperature and then reheated to temper the martensite formed during the quenching treatment. Quenching is continued to progressively lower temperatures transforming more austenite; intermediate tempering is carried out after each quenching step. Intermediate tempering is employed to induce carbide precipitation on the dislocation network and within newly formed martensite. The process is shown schematically in Fig. 1. Such treatment results in a very fine microstructure. The martensite plate size is believed to be limited initially by the heavy dislocation grid in the deformed austenite; later in the quenching cycle the plate size is further limited by the presence of Previously transformed material. Preliminary studies indicated that the structures produced in this process were characterized by a very high dislocation density and a fine carbide distribution. The strength and ductility of these structures forms part of the study reported herein.

Another interesting and potentially useful phenomenon in obtaining high strength and ductility recently observed in this laboratory is that of "transformation induced plasticity" (designated herein for convenience as "TRIP"). In these studies very high ductilities have been reported over a wide range of strength levels. These unusual combinations of strength and ductility have been attributed, in part, to a strain-enhanced transformation. Martensite, formed by the strain induced transformation of austenite, induces plasticity in a number of ways. The formation of martensite causes an increase in dislocation density and therefore an increase in strain hardening rate. Also, since the martensite is harder than the parent austenite, strain hardening is further increased. The onset of local necking is inhibited by the high strain hardening rate and ductility is enhanced. Plasticity is thus enhanced by the production of dislocations and the prevention of early necking.

Bressanelli and Moskowitz have also reported the beneficial effect on elongation of strain induced transformation occurring during the tensile testing of metastable austenitic stainless steel. The authors demonstrated the importance of strain rate and test temperature in the occurrence of strain induced transformation. They concluded that composition and test temperature directly affect the amount of martensite formed during testing and deformation rate has an indirect affect by influencing the specimen temperature. Only martensite formed in the later stages of tensile testing, during necking, was considered beneficial.

Preliminary studies have shown that high strengths and elongations can be obtained by utilizing the TRIP phenomenon. Yield strengths of over 200,000 psi with elongations approaching 40% have been attained. These results were obtained on complex alloys. The function of the individual alloying elements is not understood at the present time.

This investigation was designed a) to study the effectiveness of cyclic quenching in producing alloys having high strength and high ductility and, b) to produce "TRIP" in simpler alloys. A series of four alloys was selected including both relatively simple alloys as well as the more complex ones developed from previous work. Thus, the research reported is concerned with two separate but related topics:

- i. The cyclic quenching of a metastable austenite, wherein the transformation of austenite is accomplished thermally.
- ii. The composition dependence of the TRIP phenomenon, wherein transformation is accomplished by plastic strain.

#### II. EXPERIMENTAL TECHNIQUE

#### A. Alloy Composition

The alloy series used in the cyclic quenching study was selected on the basis of previous work with high strength martensitic steels. 4,5 The series was selected such that the effect of specific elements could uniquely be identified.

The M<sub>S</sub> temperature was designed to be well below room temperature (~ -60°C) so that the austenite was stable at room temperature. A balance was maintained between the carbide formers and carbon in the steel, i.e., sufficient molybdenum was added to combine with all the carbon present in the alloys. The Mo-C ratio was maintained constant in each alloy. Nickel was used to adjust the M<sub>S</sub> of each alloy such that the M<sub>S</sub> was the same for all alloys. The reference alloy was a simple Fe-Ni-Mo-C alloy. The second and third alloys contained 1.5% of Mn and Si, respectively, and the fourth a combination of Mn and Si. The composition of the steels used is shown in Table I.

Table I. Alloying Element (wt.%)

Cod	e	Fe	Ni	Mn .	Si	Мо	C	·
A		bal.	24.4	-	-	4.1	0.25	•
В		bal.	22.0	1.48	<b>-</b> .	4.0	0.23	
C		bal.	• 23.82		1.45	4.03	0.24	
D		bal.	20.97	1.48	1.53	3.57	0.24	: :

The composition of alloy D was similar to alloys referred to earlier which were shown to have an excellent combination of strength and ductility and which demonstrated the TRIP phenomenon. All four alloys were studied

with respect to both the response to cyclic quenching and the occurrence of the "TRIP" phenomenon.

## B. Alloy Processing

The steels were prepared by induction melting in an inert helium atmosphere. The ingots were hot forged to dimensions of 10"x2-1/2"x1/4". The bar stock was cleaned of surface scale prior to subsequent rolling and heat treatment.

Suitable lengths of bar stock were sealed into quartz tubes under vacuum and austenitized for one hour at 1100°C followed by an immediate water quench. Metallographic checks were used to insure that the alloys were completely austenitic.

The bar stock was deformed 80% at a temperature of 500°C. Deformation temperatures of 450, 500 and 550°C were used with the Fe-Ni-Mo-C alloy. A 500°C rolling temperature gave an optimum fine structure (see Fig. 12). Lower temperatures tended to give coarser microstructures while higher temperatures promoted grain boundary precipitation thereby leading to embrittlement. Thus, a 500°C deformation temperature was standardized for the alloy series. Deformation was carried out in steps of 20 mils per pass during the initial stages of deformation and was gradually reduced to 10 mils per pass during the final stages of deformation.

Specimens were heated in a muffle furnace between passes and the rolls of the mill were maintained at a temperature of 400°C during the entire rolling operation. The time to achieve the 80% reduction was less than one hour. After rolling the specimens were water quenched. The final thickness after rolling was approximately 0.060". A series of thermomechanical treatments were evaluated:

- I. Austenitize and deform 80%
- II. Austenitize, deform 80% and quench to liquid nitrogen temperature
- III. Austenitize and cycle quench to liquid nitrogen temperature
  - IV. Austenitize, deform 80% and cycle quench to liquid nitrogen temperature.

Henceforth for convenience these thermomechanical processes will be referred to as Treatment I. Treatment II. etc.

The cyclic quenching part of Treatments III and IV involves quenching the specimen to a temperature slightly below the M<sub>S</sub> followed by a tempering operation prior to again quenching to progressively lower quenching temperatures. There is a tempering step between each quenching step (as shown in Fig. 1). In preliminary studies tempering temperatures of 450°C, 500°C and 550°C were used with fully cycled specimens of all four alloys, but properties appeared insensitive to variation in this tempering range. A cycling tempering temperature of 500°C was standardized upon for the investigation.

Cryogenic baths of ethanol, methyl isopentane, liquid nitrogen and liquid helium were used. The first two medias were cooled in stages with liquid nitrogen to their freezing points.

An iron-constantan thermocouple was used to measure the temperature of the cryogenic baths and temperature variations were minimized by using a large volume of liquid medium with vigorous stirring to reduce temperature gradients. Specimens were held at cryogenic temperatures for five minutes.

The heating medium for tempering was a molten salt bath of nitrates in a resistance-heated container. Specimens were tempered for 10 minutes after each quenching step including the final step. Specimens were removed at various stages of the cycling treatment in order to study in some detail

the changes in microstructure and properties at various stages of transformation.

## C. Mechanical Testing

Tensile specimens were ground from the 0.060" sheet; their dimensions are shown in Fig. 2. Room temperature tensile tests were carried out on an Instron machine using a crossed travel speed of 0.1 cm/minute.

Hardness measurements were made on a Rockwell hardness tester. The Rockwell C hardness scale was used and hardnesses in the range  $R_{\rm c}$  30 to  $R_{\rm c}$  50 were obtained. Hardness measurements were used as a qualitative but rapid means of following transformation. However, since the method was qualitative it was desirable to institute other means of following the phase transformations during processing.

## D. Techniques for Following Structural and Phase Changes

Since TRIP was of special interest it was desirable to correlate the occurrence of the phenomenon with the percentage of martensite present prior to tensile testing. Permeability measurements of partially transformed specimens were compared with standard specimens containing known proportions of FCC austenite and BCC iron.

A search coil containing a specimen of standard size was placed between the poles of an electromagnet. The coil was held in this position by a swinging lever. The coil and specimen could be swung out from the position between the electromagnet pole pieces to a fixed position removed from the poles. A current was induced in the search coil by each of three sequential operations:

i. When the coil was between the pole pieces and the magnetic field was reduced from the maximum value to zero;

- ii. When the coil was removed from the residual magnetic field between the pole pieces to a fixed position away from the poles;
- iii. When the specimen was removed from the coil.

The total current induced in the search coil by this set of operations was measured. The total magnetic induction readings were measured for standard specimens and a calibration curve of induction reading vs ratio of transformed to untransformed material was plotted. Specimens were compared with this calibration curve.

The advantage of this method lay in its simplicity; austenite is paramagnetic, whereas martensite is ferromagnetic and has a high permeability. An accuracy of ±5% in the figure obtained for the amount of transformation was estimated. While the data obtained for higher amounts of transformation were quantitative, the limited accuracy of the method made measurements of small amounts of transformation qualitative.

Metallographic techniques were used to check the magnetic data at points of interest and a good correlation was obtained between the two determinations.

#### III. EXPERIMENTAL RESULTS

Processing factors held constant in the study reported in this paper were austenitizing temperature, deformation temperature and degree of deformation, cycle tempering temperature and time. The variables included were the amount of elevated temperature (500°C) deformation prior to quenching, the quenching technique, the quenching temperature, and the alloy composition.

The purpose of cyclic treatment was to produce a desirable microstructure in terms of resulting mechanical properties. The effect of cycling on alloys both in the austenitic condition and after deformation was studied. A summary of the variables used and their effect on mechanical properties is shown in Table II. Corresponding hardness and phase transformation data are summarized in Table III.

A detailed study was made of the mechanical properties of specimens that were deformed 80% at 500°C and cyclically quenched to temperatures down to liquid helium temperatures. Data were gathered for specimens quenched to various stages of the quenching cycle. Tables IV through VII show tensile data for deformed austenite specimens cyclically quenched to different final quenching temperatures. Five quenching temperatures were used in each case.

Tensile data shown in Tables IV through VII are presented graphically in Figs. 3-6.

Table II. Variation of Tensile Properties with Processing

-					
Process	Alloy	UTS (psi)	YS (psi)	EI%	
Austenitized	A	204,800	155,600	10.2	
Cycle Quench	В	170,100	88,200	11.3	
то-196	C	189,300	133,300	8.7	
(Treatment I)	D	169,800	91,700	10.6	
Austenitized	A	175,850	164,100	40.7	
Def. 80%	В	157,800	152,100	16.7	
(Treatment II)	C	175,500	167,900	7•9	
	D	166,190	159,000	25.3	
Austenitized	A	210,100	179,200	15.0	
Def. 80%	В	174,100	148,100	12.7	
Straight Quench	C	189,300	163,900	7.0	
то-196	<b>D</b>	188,100	170,900	9.8	
(Treatment III)					
Austenitized	A	214,100	194,800	10.8	
Def. 80%	В	178,000	158,700	11.4	
Cycle Quench	C	201,000	190,100	4.9	
то-196	D .	188,200	170,500	. 7.5	
(Treatment IV)					
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Table III. Variation of Hardness and Phase Transformation With Processing

Process	Alloy	Hardness Rockwell C	Wastensit
Austenitiże	A	43.1	91
Cycle Quench	B	36,2	57
TO-196	<b>C</b>	41.5	69
(Treatment I)	D	38 <b>.</b> 9	62
Austenitize	Α	42.8	<b>.</b>
Def. 80%	В	36₊2	5
(Treatment II)	<b>c</b>	40.2	5
	D	38.5	5
Austenitize	A	45.7	60
Def. 80%	$\mathbf{B} = \mathbf{B}$	40.h	32
Straight Quench	c	43.1	42
то-196	D	41.5	30
(Treatment III)			
Austenitize	A	47.1	89
Def. 80%	В	39•3	41
Cycle Quench	C	44.2	64
то-196	D	42.0	48
(Treatment IV)	(1945년 - 1945년 - 1945년) - 국민국민의 전설(1947년 - 1947년)		

Table IV. Variation of Tensile Properties.
With Quenching Temperature
Alloy A: Fe-24Ni-4Mo-0.25C

Quenching Temperature	YS	UTS	EL%	Red in A %
As rolled	161,320 166,900	176,100 175,600	40.3 41.0	53.0
-20°C	160,400	171,200	48.5	53.7
-70°C	156,900	168,700	47.7	51
-120°C	168,700 171,800	193 <b>,</b> 900 193 <b>,</b> 300	12.7 12.7	49.8
-150°C	184,300 183,100	206,200 207,600	11.2 9.7	52•7
-196°C	194,800 191,400	214,100 214,350	9.7 11.9	49.8

Table V. Variation of Tensile Properties with Quenching Temperature

Alloy B: Fe - 22 Ni - 1.5 Mn - 4 Mo - 0.23 C

Quenching Temperature	YS	UTS	EL %	Red in A %
As Rolled	152,100	157,800	16.7%	40.9%
-70	148,400	154,200	27.7	42%
	152,100	157,600	14.2	46.3%
-110	141,000	146,000	46.0	46.5%
	142,300	150,700	15.8	
150	154,800	167,300	13.0	36.1%
<b>-15</b> 0	154,200	164,700	12.3	40.1%
<b>-1</b> 96	160,100	176,100	11.6	39.1%
	157,300	179,600	11.1	38.1%
Liquid	150,200	163,800	11.9	39.4%
Helium	157,500	174,900	10.3	29.3%

Table VI

Variation of Tensile Properties with Quenching Temperature

Alloy C: Fe - 24 Ni - 1.5 Si - 4 Mo - 0.25 C

Quenching Temperature	YS	UTS	EI%	Red in A %
As Rolled	167,900	175,500	<b>7.</b> 9	25.5%
-70	168,800	176,000	7•9	20.5%
-110	182,300 174,600	200,850 188,000	6.3 7.0	17.%
-150	195,840 187,960	206,500 198,300	5•2 6•3	17.7% 26.9%
-196	193,100 187,500	204,500 197,900	4.8 5.0	21.4% 20. <i>5</i> %
Liquid Helium	181,800	198,150	4.0	22.3%

Table VII

Variation of Tensile Properties with Quenching Temperature

Alloy D: Fe - 21 Ni - 1.5 Mn - 1.5 Si - 4 Mo - 0.25 C

Quenching `Temperature	YS	UTS	EL %	Red in A %
As Rolled	159,000	166,140	25.3	23.7
-70	157,000	164,900	25.9	25.9
	155,800	162,600	26.9	21.4
-110	156,200 160,400	166,150 166,800	30.1 13.4	24.2
-150	165,200	176,100	16.6	24 <b>.</b> 5
	164,950	177,700	7.9	22 <b>.</b> 8
-196	170,000	185,200	7.9	25.5
	172,000	193,200	7.1	18.4
Liquid	170,000	183,400	11.1	25 <b>.</b> 6
Helium	169,000	183,300	10.3	23 <b>.</b> 5

#### IV. DISCUSSION

A considerable amount of work has been reported in the literature concerning thermomechanical methods of strengthening steels. In recent years Cohen and several coworkers have reported on the effect of thermal cycling of austenitic steels through the martensitic transformation. Cohen's work and the work of several Russian workers however has been concerned with cyclic treatment of a somewhat different nature than that reported here. These workers have studied materials where a reverse martensitic reaction occurs. The alloys are quenched to a given temperature and then reheated to a given tempering temperature. On reheating the martensite reconverts to austenite. In Cohen's earlier work austenitic Fe-Ni alloys were quenched to liquid nitrogen temperatures and then reheated to 450°C. The effects of the reverse martensitic transformation (the conversion of martensite to austenite on reheating) was studied in some detail. Specimens were quenched to a temperature of -196°C and held for 30 minutes prior to reheating to 450°C for 2 minutes; this cycle was repeated and specimens were tested after undergoing from 1 to 5 cycles. Austenite and martensite were both strengthened by cyclic transformation. The strength of both martensite and austenite increased with the number of cycles but the major effect was observed after the initial cycle. The strength of austenite was increased to a greater extent than that of martensite. While the strengths were low due to the low carbon content of the alloy, substantial increases in mechanical properties were observed. Austenitic yield strengths were increased from values of 24,000-32,000 psi to 67,000-74,000 psi after 5 cycles while elongations fell from approximately 70% to 26.0%. Corresponding martensite strengths rose from 60,000-80,000 psi to 86,000-98,000 psi with elongations falling from 30% to 20%. Strengthening was attributed to

stresses and imperfections produced in the Fe-Ni austenites.

Russian workers 9,10 have been concerned mainly with the stabilization of austenite as a result of such cycling treatment. Cohen 11 concluded however that stabilization associated with the reverse martensitic reaction in iron-nickel alloys is due not to cycling but due to compositional changes in the martensite where the heating rate was not sufficiently rapid.

Relatively few workers have investigated the change in mechanical properties such as strength and ductility associated with cycling treatments similar to those used by Cohen. In one of the few recent studies of this nature Gridnev et al. 12 followed the hardness change during cycling but tensile data were not obtained. In the present study tensile data were of prime importance both in following the effects of cyclic treatment and in observing the occurrence of TRIP.

One purpose of the cyclic quenching treatment is to limit the size of martensite plates formed during transformation. During the initial stages of quenching it is assumed that the martensite plate size is limited by the complex dislocation grid formed during deformation of austenite. In the later quenching stages the martensite plate size is presumably further limited by the presence of previously transformed material. Intermediate tempering is employed to induce carbide precipitation on the dislocation network and within newly formed martensite.

All four alloys responded in a similar fashion to processing variables. Cyclic quenching resulted in increasing transformation to martensite. The structure of the martensite thus formed was also improved in that a fine martensite plate size resulted from cyclic treatment. Figures 8 through 11 show the Fe-Ni-Mo-C alloy after various treatments:

Figure 8 Austenitized and cycle quenched to -196°C;
Treatment I.

Figure 9 Austenitized and deformed 80%; Treatment II.

Figure 10 Austenitized, deformed 80% and straight quenched to -196°C; Treatment III.

Figure 11 Austenitized, deformed 80% and cycle quenched to -196°C;

Treatment IV.

The cyclic quenching of non-deformed austenitic specimens of all four alloys (Treatment I) resulted in a relatively high degree of transformation to martensite. The range of transformation in the four alloys was 57% to 91% (Table III), but the martensite plate size was relatively large (Fig. 8).

Deformation of metastable austenite resulted in a fine microstructure.

After an 80% deformation (Treatment II) small quantities of martensite

(<5%) were observed; the microstructure is shown in Fig. 9.

The martensite plate size observed in deformed austenite, straight quenched to liquid nitrogen temperature (Treatment III) was considerably refined (Fig. 10) as compared with cyclically quenched austenite (Treatment I). The amount of martensite in Treatment III was, however, reduced. The range of transformation in the four alloys was 30% to 60% (Table III).

The cyclic quenching of deformed austenite (Treatment IV) was effective in further reducing the martensite plate size (Fig. 11) and at the same time transformation to martensite was favored as compared with straight quenched materials (a range of 41% to 89% martensite was observed in specimens of the four alloys that were cyclically quenched to -196°C).

Deformed and cyclically quenched specimens (Treatment IV) thus resulted in the finest microstructure. These specimens also resulted in the highest

tensile and yield strengths. Photomicrographs of the other three alloys after deformation and cyclic treatment are shown in Figs. 12 through 14.

As may be seen in these photomicrographs the amount of transformation in deformed and cyclically quenched specimens (Treatment IV) was not high, being 89%, 41%, 64%, and 48% in the case of alloys A, B, C, and D, respectively. The mechanical stabilization of austenite during deformation was probably a contributing factor since corresponding transformations in cyclically quenched specimens of undeformed austenite were 91%, 57%, 69%, and 62%, respectively.

In the fully cycled specimens (Treatment IV) the martensite present was tempered to varying degrees as shown by its varied response to etching (Figs. 11-14). Martensite formed early in the quenching cycle was tempered to a higher degree than martensite formed later in the cycle which was subjected to fewer tempering steps.

Specimens cyclically quenched to liquid nitrogen temperatures, regardless of prior treatment, did not exhibit superior ductility in terms of percent elongation. The elongation of both deformed and undeformed austenite was drastically reduced by transformation to martensite at the lowest temperature (-196°C), regardless of quenching technique. In all cases elongations after quenching to -196°C were in the range 5-1%. However, in the <u>initial</u> stages of quenching in Treatment IV (for alloys A, B, and D) ductilities appeared to be enhanced by cycling, as shown in Figs. 5 through 6. The low elongations obtained with alloy C remained largely unexplained. The alloys were all designed to have an Mg temperature in the region of -60°C but mechanical stabilization of austenite during deformation presumably caused a depression of the Mg temperature. Generally, the first quench of deformed austenite specimens of all

(Treatment IV) alloys resulted in no transformation. On subsequent quenching martensite was produced, but the ductility remained high if the percentage of martensite produced prior to tensile testing was low. (Figures 3-6.) However, in the later stages of quenching when a significant proportion of tempered and untempered martensite (> 30%) was present in the microstructure conventional strength and ductility data were obtained. Thus, in the latter stages of quenching an increase in yield strength due to increased transformation was accompanied by a decline in ductility. It was apparent that superior ductilities were obtained only during the early stages of quenching where little thermally induced transformation was occurring. Apparently a full cyclic treatment was not conducive to high strength and high ductility.

Other work with highly deformed austenitic steels in this laboratory indicates that superior strengths and ductility can be obtained without cyclic treatment by utilizing the TRIP phenomenon. Table VIII shows results obtained with a complex alloy (Fe-9%Cr-8%Ni-2%Si-1%Mn-4%Mo-0.25%C). This alloy was deformed 80% at 500°C in the austenitic condition. Specimens tested in three conditions (as-deformed, deformed and quenched to -196°C and deformed, quenched and tempered) all gave high yield and ultimate strengths with elongations in the neighborhood of 30%. These combinations of high strengths and high ductilities were not observed in the cyclic treatments discussed earlier. However, in the case of the Fe-Ni-Mo-C alloy (Treatment IV), similar elongations were observed for specimens cycled once or twice.

In the present investigation the TRIP phenomenon was observed with alloys A and D with Treatment II and, as reported above, in the early stages of cycle quenching (Treatment IV). Elongations up to 48% were

Table VIII. Tensile Data for an Fe-Cr-Ni-Si-Mn-Mo-C Alloy After an 80% Deformation in the Austenitic Condition

Heat Treatment	Properties					
After Deformation	Yield Strength	Ultimate Strength	Tensile	Hardness (R <sub>c</sub> )	Elonga- tion	
Air cooled-no tempering	207,900 psi	282,300	psi	50	27%	
Air cooled- quenched in LN-no tempering	204,000 psi	281,000	psi	50	30%	
Air cooled and quenched in IN tempered at 45°C	215,400 psi	291,000	psi	. 51	27%	

obtained with alloy A utilizing Treatment IV. However, TRIP was only obtained in any case where less than 1% martensite, tempered or untempered, was present prior to testing. When quenching, for example, was continued to lower temperatures serrated stress strain curves were not obtained and a sharp decline in ductility resulted. The fact that serrated stress strain curves were not obtained was indicative of the fact that the TRIP phenomenon was not operative. A typical serrated stress strain curve is shown in Fig. 7. It was apparent that the stability of the austenite after processing had to be such that the strain induced transformation occurred with the onset of incipient necking in the specimen. Where significant amounts of martensite were present in the microstructure prior to tensile testing, necking and then fracture occurred.

While it is not apparent why TRIP was observed in only two of the four alloys, the enhanced elongations obtained with the Fe-Ni-Mo-C alloy suggests that complex alloys are not necessary for the occurrence of the TRIP phenomenon.

It was concluded that the TRIP phenomenon can be obtained with relatively simple alloys such as alloy A. However, it cannot be assumed that other elements (such as Si and Mn in alloy D) are not necessary in obtaining the unusual combination of ultra high strength and high ductility observed in Fahr's studies.

#### V. CONCLUSIONS

- I. The cyclic quenching of undeformed austenitic steels (Treatment I) resulted in a relatively high degree of transformation. However, the martensite plate size was relatively large. After five cycles of quenching and tempering the percent martensite resulting in the four alloy systems ranged from 57 to 91. The mechanical properties of the fully cycled steels were not appreciably different from those imparted by conventional heat treatments.
- 2. Deformation of austenite followed by cooling to room temperature (Treatment II) resulted in an essentially austenitic structure (less than 5% martensite). Good combinations of strength and ductility were obtained. Ductility was enhanced by the TRIP phenomenon in the case of alloys A and D.
- 3. Martensite formed at liquid nitrogen temperatures from deformed austenite (Treatment III) was finer than that formed from undeformed and cyclically quenched austenite (Treatment I). The amount of martensite formed, however, was less than that of Treatment I. The range of transformation in the four alloys was 30% to 60%. Presumably mechanical stabilization of the austenite retarded the transformation. The fine martensitic structure resulted in high strength, but the TRIP process was not operative and low ductilities were obtained.
- 4. The cyclic quenching of deformed austenite (Treatment IV) was effective in further reducing the martensite plate size. The transformation was also enhanced by this treatment as the range of martensite content was 41% to 89% in the four alloys. The extreme fineness of the microstructure was reflected in the high strength of these alloys.
- 5. Prior austenite deformation tended to depress the austenite-tomartensite transformation for all the alloys studied.

- 6. Alloys cooled to liquid nitrogen temperature, regardless of prior treatment, exhibited poor ductility. The presence of more than about 15% untempered martensite prior to tensile testing appeared to be quite detrimental.
- 7. The initial cycles of quenching and tempering for alloys processed by Treatment IV generally gave structures characterized by an excellent combination of strength and ductility. From the appearance of an autographic record of the stress-strain curve it can be concluded that the TRIP phenomenon was operative in these alloys, viz., incipient necking was delayed by the increased rate of strain hardening occasioned by the strain-induced austenite-to-martensite transformation.
- 8. It can be concluded that for these alloys and for this particular form of cyclic processing one or two cycles of alternate cryogenic treatment followed by tempering is beneficial, but more than this number is deleterious. This result may be tentatively rationalized on the basis of the following factors: one, the TRIP process is most effective when there is a large amount of austenite available that is stable with respect to thermal energy, but unstable with respect to strain energy; and, two, the presence of more than a critical amount of relatively untempered martensite (tempered for one cycle only) is embrittling.
- 9. The effectiveness of the TRIP phenomenon in preventing early necking and thereby significantly increasing the elongation at high strength levels in the Fe-Ni-Mo-C alloy suggests that further studies of this effect be confined to simpler alloys than those studied earlier.

#### VI. FUTURE WORK

Further work on the simplication of alloy composition and factors influencing the strain induced transformation of austenite is required. Specific factors requiring additional attention are:

- 1. The chemical and mechanical stabilization of austenite caused by deformation;
- 2. The effect of the amount and temperature of austenite deformation on the resulting mechanical properties;
- 3. The effect of the tensile strain temperature on the occurrence of TRIP and its effect on mechanical properties; and
- 4. The effect of the tensile strain rate on the occurrence of TRIP

  and its effect on mechanical properties.

In addition to the above studies the determination of more complex engineering properties such as the fracture toughness and the fatique resistance of such steels would be of interest.

#### ACKNOWLEDGMENTS

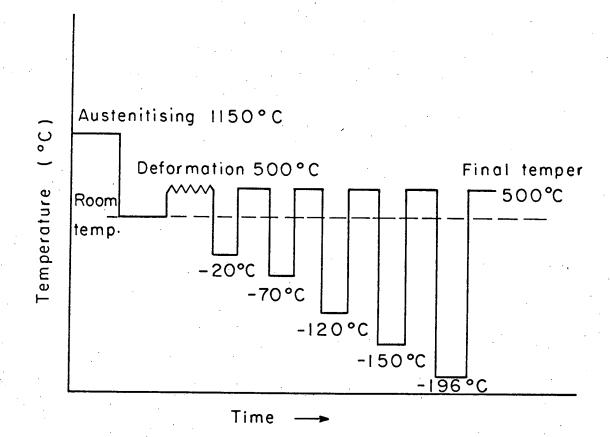
The author wishes to express his grateful appreciation to Professor Victor F. Zackay, Department of Mineral Technology, University of California, for his guidance, encouragement and support throughout the course of this investigation.

This work was performed under the auspices of the United States

Atomic Energy Commission through the Inorganic Materials Research Division
of the Lawrence Radiation Laboratory.

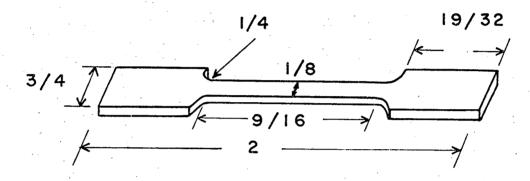
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MUB-13714

Fig. 1 Schematic representation of cyclic quenching treatment employed



Measurements in inches (not to scale)

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Fig. 2 Tensile specimen used in the determination of mechanical properties. The thickness of the specimen was approximately 0.060 inches and the gage length was 0.50±0.001 inches.

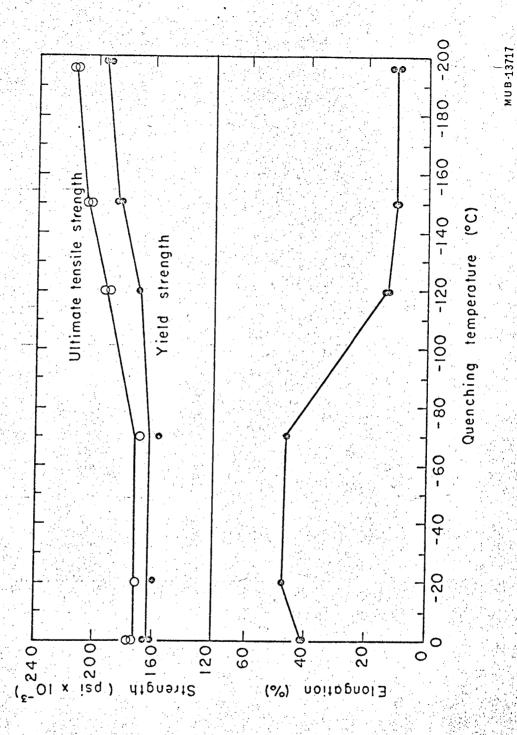


Fig. 3 Effect of quenching temperature on the tensile properties of Alloy A, Fe-23Ni-4Mo-0.25C. The alloy was austenitized at 1100°C for 45 minutes and deformed 80% prior to cyclic quenching. A tempering step (10 minutes at 500°C) was carried out after each quench.

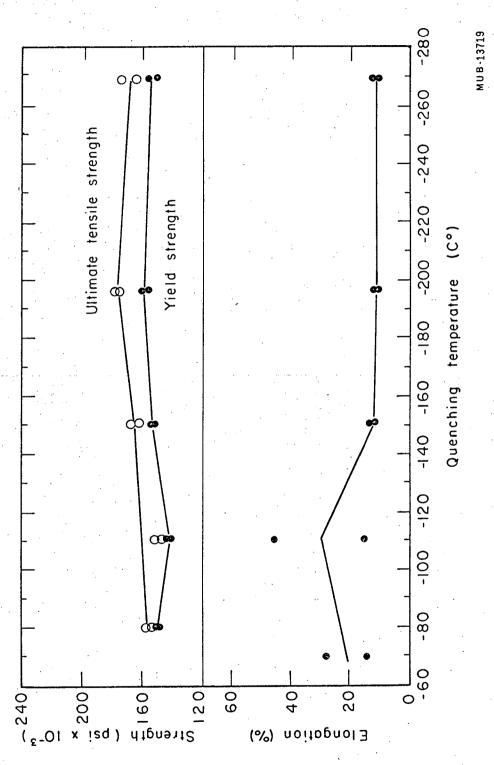


Fig. 4 Effect of quenching temperature on the tensile properties of Alloy B, Fe-22Ni-1.5Mn-4Mo-0.23C.

The alloy was austenitized at 1100°C for 45 minutes and deformed 80% prior to cyclic quenching. A tempering step (10 minutes at 500°C) was carried out after each quench.

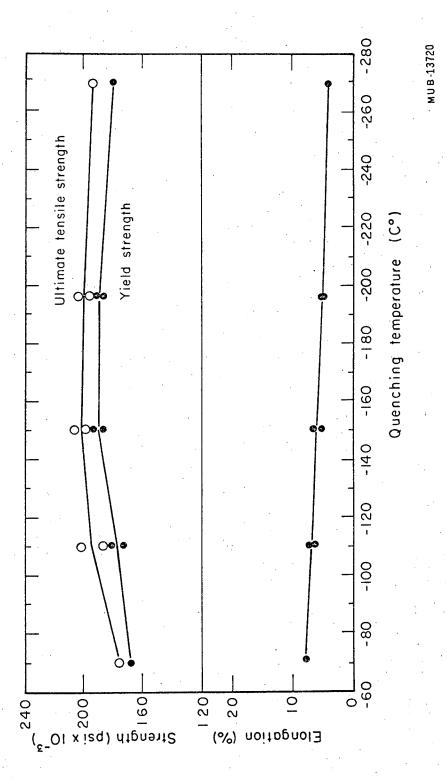


Fig. 5 Effect of quenching temperature on the tensile properties of Alloy C, Fe-24Ni-1.5Si-4Mo-0.25C. The alloy was austenitized at 1100°C for 45 minutes and deformed 80% prior to cyclic quenching. A tempering step (10 minutes at 500°C) was carried out after each quench.

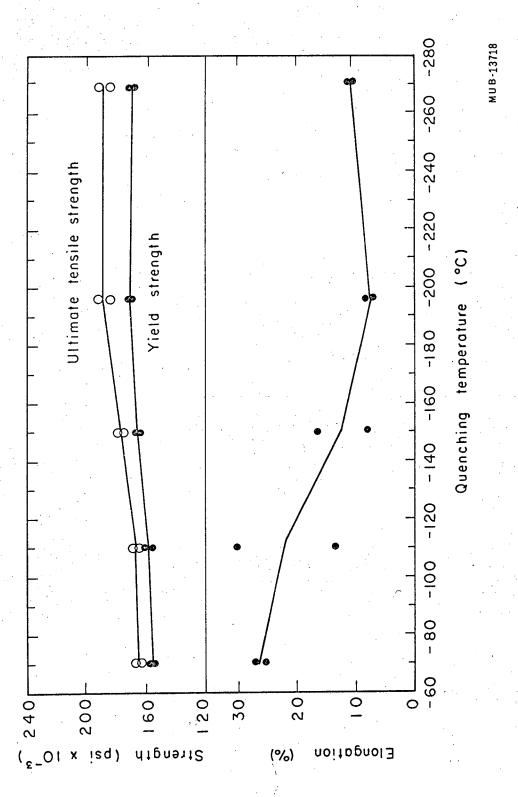
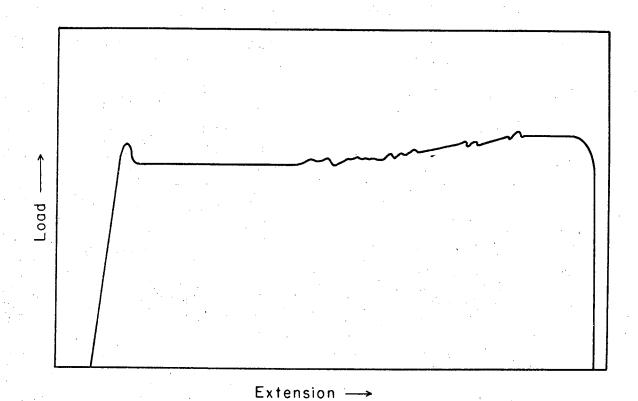


Fig. 6 Effect of quenching temperature on the tensile properties of Alloy D, Fe-21Ni-1.5Mm-1.5Si-4Mo-0.25C. The alloy was austenitized at 1100°C for 45 minutes and deformed 80% prior to cyclic quenching. A tempering step (10 minutes at 500°C) was carried out after each quench.



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Fig. 7 Diagrammatic representation of a typical serrated stress strain curve.



Fig. 8 Alloy composition Fe-23Ni-4Mo-0.25C. Austenitized at 1100°C and cycle quenched to -196°C. The cycle tempering temperature was 500°C. Magnification 400x

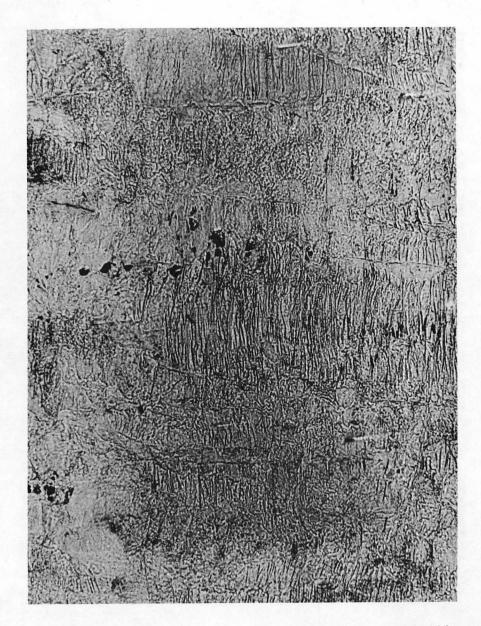


Fig. 9 Alloy composition Fe-23Ni-4Mo-0.25C. Austenitized at 1100°C and deformed 80% at 500°C. Magnification 250x

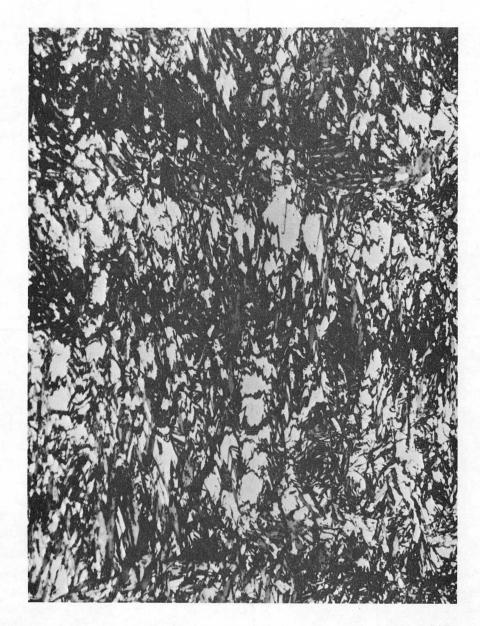


Fig. 10 Alloy composition Fe-23Ni-4Mo-0.25C. Austenitized at 1100°C, deformed 80% at 500°C and straight quenched to -196°C. A final tempering at 500°C was used. Magnification 400x

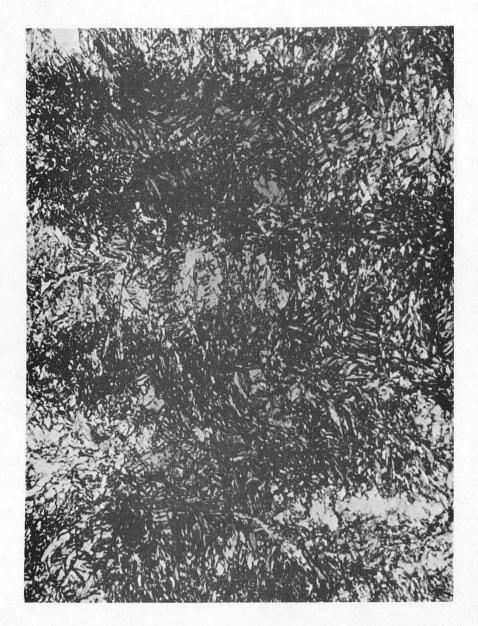


Fig. 11 Alloy composition Fe-23Ni-4Mo-0.25C. Austenitized at 1100°C, deformed 80% at 500°C and cycle quenched to -196°C. The cycle tempering temperature was 500°C. Magnification 400x



Fig. 12 Alloy composition Fe-22Ni-1.5Mn-4Mo-0.25C. Austenitized at 1100°C, deformed 80% at 500°C and cycle quenched to -196°C. The cycle tempering temperature was 500°C. Magnification 400x

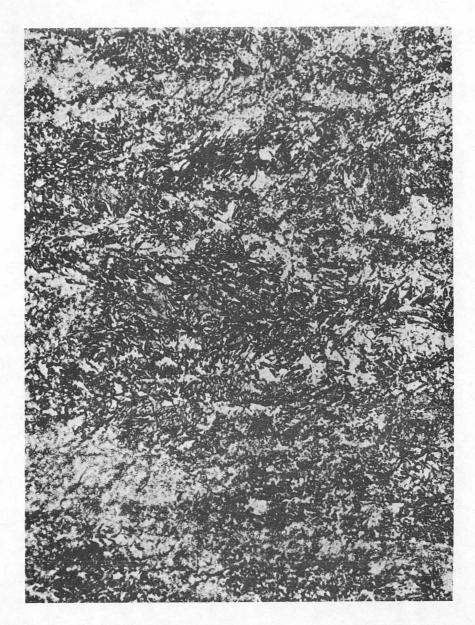


Fig. 13 Alloy composition Fe-24Ni-1.5Si-4Mo 0.25C. Austenitized at 1100°C, deformed 80% at 500°C and cycle quenched to -196°C. The cycle tempering temperature was 500°C. Magnification 400x



Fig. 14 Alloy composition Fe-21Ni-1.5Si-4Mo-0.25C. Austenitized at 1100°C, deformed 80% at 500°C, and cycle quenched to -196°C. The cycle tempering temperature was 500°C. Magnification 400×

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