To my parents: ~~Alvaro and Regina~~

To my sisters: Regina, Patricia, Jimena and Elvira

EFFECT OF ALLOYING ELEMENTS IN STEELS ON THE KINETICS
OF THE AUSTENITE TO BAINITE TRANSFORMATION

Contents

Abstract.	v
I. Introduction.	3
II. Experimental Procedures	6
A. Materials	6
1. Choice of Alloy Compositions.	6
2. Preparation	8
3. Chemical Analysis	8
B. Experimental.	11
1. Apparatus Description	11
2. A Typical Run	14
C. Thermodynamic Experiments	15
1. Austenite Transformation Temperature.	15
2. Ms Temperatures	15
3. Isothermal Runs in the Bainite Reaction Range	15
4. Graphical Examples of two Typical Runs.	16
D. Hardness Testing.	16
E. Optical Microscopy.	17
III. Results and Analysis of Data.	18
A. Austenite Transformation Temperatures	18
B. Ms Temperature and Hardness Values	18
1. Martensite Transformation Temperatures.	18
2. Effect of the Quenching Rate on Ms.	21

3. Effect of Austenitizing Temperature on Ms	23
4. Hardness Tests of Ms Structures	24
E. Bainite Transformation Data	23
1. Isothermal Transformation for the Various Alloys. . .	23
2. Diagrams for the Reaction Start of the Austenite to Bainite Transformation	39
3. Effect of the Quenching Rate on the Kinetic and Reproducibility.	39
4. Effect of the Austenitizing Temperature on the Bainite Kinetics	42
5. Hardness Tests of Bainitic Structures	45
F. Optical Microscopy and ASTM Grain Size Characterization .	45
1. ASTM Grain Size of the Treatments Used.	45
2. Effect of Austenitizing Temperature on the Grain Size	45
3. Optical Microscopy of the Martensitic Structures. . .	48
4. Optical Microscopy of the Bainitic Structures	48
V. Results and Discussions	50
A. Results on the Austenite Transformation Temperature and the Effect of Different Alloying Elements.	50
B. Results of the Ms and Discussion of Some Available Empirical Formulae for Ms Determination on the Basis of Alloy Content	51
C. Results on the Hardness Test Readings	57

D. Results of the Bainite Reaction (above the Ms)	58
1. Reaction Start Diagrams Discussion for the Different Alloys (Kinetics)	58
2. Discussion on the Effect of Austenitizing Temperature on the Kinetics	65
3. Determination of Bs on the Basis of Some Existing Empirical Formulae.	66
4. Comments on Optical Microscopy	68
V. Conclusions.	70
Acknowledgements	72
Appendix	73
References	75
Figure Captions.	79
Figures.	82

EFFECT OF ALLOYING ELEMENTS IN STEELS ON THE KINETICS OF THE AUSTENITE TO BAINITE TRANSFORMATION

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ABSTRACT

The purpose of this study was to investigate the effect of different alloying elements on the bainite reaction kinetics. The primary objective was to perform dilatometric studies on four Fe-C-X ternary systems in particular (X = Mo, Ni, Cr, Mn). This technique enables us to obtain the reaction start times for the isothermal decomposition of austenite in the bainite range. The second objective was to extend the investigation to include higher combination systems of these same alloying elements such as Fe-C-Mo-Ni, Fe-C-Ni-Cr, Fe-C-Ni-Cr-Mo in order to determine what their interaction effects are in the bainite transformation.

All the alloying elements studied accelerated the austenite decomposition at isothermal temperature treatments just above the M_s giving for the reaction start curve an S-like curve. Of the alloying elements studied Mn, Ni, Cr (in order of effectiveness) shifted the bainite reaction to longer times (increasing the hardenability). Mo, instead, accelerated the bainite reaction.

All the alloying elements lowered the temperature range of bainite reaction, further additions, even for mixed addition such as Cr-Ni, Mo-Cr Mo-Ni suggested an additive effect in lowering the reaction temperature. However combined additions gave a multiplicative

26

effect on the reaction start times for the isothermal decompositions of austenite, suggesting strong interaction effects. The combined addition Cr-Ni was 6 times more effective than the addition of Ni and Cr alone. Similarly the time for Mo-Ni alloys was five, and for Mo-Cr was four times more effective with respect to bainite hardenability.

1. INTRODUCTION

The most recent work in the field of "steels" for the past years has focused on alloy design. It is well known that the microstructure of alloys has major influence on their mechanical properties. If theories that relate microstructures to mechanical properties are developed and then applied, the empiricism of alloy design can be reduced.^{1,27}

The design of new alloys, or obtaining considerably improvements in the properties of the existing ones, largely involves achieving microstructural control through variations in chemical composition and heat treatment. An understanding of the kinetic mechanisms of formation and reaction product morphologies of the bainite transformation appears to be essential. Some commercial tempered martensitic steels have good pearlitic hardenability without good bainitic hardenability. In these steels significant amounts of ferrite, upper bainite and lower bainite may form during quenching. In small amounts either ferrite or upper bainite are generally considered to have detrimental effects on the fracture toughness. However lower bainite has been considered comparable to tempered martensite^{2, 3, 4} although some conflicting evidence exists.^{5, 6, 7}

The bainite reaction overlaps the proeutectoid ferrite and pearlite reactions at higher temperatures and the martensite reaction at low temperatures. Bainite can be formed either by continuous cooling or isothermal transformation.

The bainite transformation was discovered 40 years ago by Bain.⁸ Even today the bainite transformation is still the subject of debate and controversy.⁹ There are several schools of thought on what metallographic and mechanistic features characterize it.

It has been proposed by Aaronson that a definition in terms of morphology should be accepted.¹⁰ From a microstructural viewpoint, bainite is defined as a nonlamellar aggregate of ferrite and carbide with an acicular morphology. There have been observed since the beginning of bainitic studies two variants of bainite,^{8, 11, 12} 14, 15, 16, 52, 53 upper and lower bainite. In upper bainite the carbides precipitate along the lath boundaries of ferrite laths whereas for lower bainite the carbides precipitate within the ferrite plates at an angle of 55-65° to the major growth direction.

Differentiating these two structures is sometimes difficult and not always possible because the morphology of bainite changes gradually with reaction temperature so no pronounced structural changes are observed over small temperature ranges. (13, 16, 17)

Very little work has been done on the effect of alloying elements on the kinetics of the bainite reaction, as opposed to the work done on the pearlite and preeutectoid ferrite. Recent systematic studies on commercial alloy steel with a magnetometric technique^{17, 18} have shown that the various alloying elements affect the kinetic of the bainite reaction differently. By means of a rapid magnetometric technique a series of the bainite reaction start diagrams were obtained¹⁷ for 4340 with alloying additions of Ni, Cr, Mo, Mn and Si. Previous results were published¹⁹ on different alloy steels.

The commercial steels, and in particular 4340, in which these systematic studies were carried are many component systems (.39 C, .7 Mn, .28 Si, .76 Cr, 1.7 Ni, .2 Mo, .22 Cu). The results of additions of more than one alloying element showed synergistic rather than additive effects on the bainite hardenability kinetics.

Because so many alloying elements were present it is difficult to assert what the actual influence on the kinetics is due to. Many possible interactions are involved.

The primary objective of this investigation was to follow a systematic experimental investigation by means of thermodynamic studies. It is unfortunate that this was not possible in all ternary Fe-C-X ternary systems because some of the reactions take place in fractions of a second. Nonetheless, several ternary systems could be studied (Ni, Cr, Mo, Mn).

The secondary objective, but not less important than the first was to follow the reaction kinetics for the start of the bainite transformation for quaternary and higher combination systems of these same alloying elements, with the purpose of studying possible interaction effects.

A fundamental result was not expected from these studies because of the complexity of the systems but qualitative understanding of their interaction and relative effectiveness on bainite hardenability could be found.

It is hoped that future research on the factors that affect the kinetics, thermodynamic, chemical and morphological aspects of the transformation, and the correlation of microstructure with mechanical properties and heat treatments, could lead to improved progress alloy design.

II. EXPERIMENTAL PROCEDURES

A. Materials

1. Choice of Alloy Compositions

The choice of materials was made on the basis of a need for a simple iron-base system that would permit the study of the bainite transformation and yet improve the separation of the bainite and pearlite reactions, so that their reactions would not interfere.

This was possible in most of the alloys chosen except in the low carbon, low molybdenum ones (1, 2, 3) where the reactions occurred too fast with respect to our recording limits, it was found for the different alloys that this was attained in different degrees.

The elements Mo, Cr, Ni, Mn were chosen. Ni and Mn are austenite stabilizers and Mo, Cr are ferrite formers.^{20, 8, 23} All these elements behave differently with respect to effects on the carbon diffusion in austenite. On the basis of diffusion data by Krishtal²¹ and Blanter²² it can be said that the diffusion of carbon in austenite decreases with increasing content of Cr, Mo, Mn (in order of effectiveness), and increases in the presence of Ni. So it is expected that they could have different effects on the transformation kinetics, especially in the upper bainite range which seems to be a process controlled by nucleation and growth where diffusion plays an important role.

Also on the basis of existing higher component low alloy steels the bainite reaction start diagrams given by Prakash,¹⁷ ASTM,¹⁹ Irving and Pickering²⁴ Kinsman and Aaronson²⁵ Ni, Cr, Mn seemed to retard the bainite reaction as opposed to Mo which in some cases was

Table I. List of alloys prepared and their nominal compositions (given in wt %) before castings.

Specimen No.	Casting No.	Alloy Composition				
		C	Cr	Ni	Mn	Mo
1	7411-27	.2				.3
2	7411-28	.2				.4
3	7411-29	.2				.5
4	7412- 1	.3				.3
5	7412- 2	.3				.4
6	7412- 3	.3				.5
7	7412- 4	.3		2.		
8	7412- 5	.3		1.		
9	7412- 6	.2		2.		
10	7412- 7	.3	1.			
11	7412- 8	.2	1.			
12	7412- 9	.3	1.	1.		
13	7412-10	.3		.1		.5
14	7412-11	.3	1.			.5
15	7412-12	.3	1.	1.		.5
16	7412-13	.2	1.	1.		.5
17	747 - 6	.4			2.	
18	747 - 7	.4			2.2	
19	747 - 8	.4			2.5	
20	747 - 9	.4			2.7	

found to accelerate it. Therefore, it was of most interest to study what these elements would do when in ternary systems Fe-C-x.

The compositions were chosen on the basis of amounts that seemed to affect the transformation for results available on commercial and other alloy steels. It was found that Mn should be effective in the range (2-3) weight %, Ni and Cr in the (1-2) weight % and Mo (.3-.5)%. Mo should not be higher than .4% to avoid undissolved carbides that would require going to higher austenitizing temperatures²⁰ or holding much longer than 15 min at this temperature.

2. Preparation

The 20 lb ingots were produced following the specifications with respect to alloy compositions given in Table I. They were homogenized in vacuum either for 72 hr at 1100°C or for 24 hr at 1400°C. The ingots after homogenization were softened by holding at 600°C for 4 hours. Dilatometer specimens were prepared by cutting from the cold rolled strips (according to specifications in Fig. 1).

3. Chemical Analysis

Chemical analysis were made in all cases by spectroscopic atomic absorption methods. This was done twice with very good consistency except for some of the low carbon values (1, 2, 3, 14) where there was a maximum relative variation of 8%.

A LECO carbon analysis was also done for some specimens which showed complete agreement with the average absorption values. The average chemical compositions from all this analysis are given in Table II.

Emphasis was made on the chemical analysis due to the fact that it was most important to know what the actual alloy content on the different available compositions was for the comparative studies on

Table II. Results of chemical analysis* (given in wt %) of cast alloys.

Specimen N	Casting No.	Composition of Analyzed Elements				
		C	Cr	Ni	Mn	Mo
1	7411-27	.17				.29
2	7411-28	.18				.38
3	7411-29	.17				.48
4	7412- 1	.24				.29
5	7412- 2	.27				.39
6	7412- 3	.3				.49
7	7412- 4	.23		2.04		
8	7412- 5	.29		1.01		
9	7412- 6	.2		2.03		
10	7412- 7	.29	1.			
11	7412- 8	.17	1.			
12	7412- 9	.27	1.01	1.1		
13	7412-10	.25		1.		.48
14	7412-11	.24	.99			.47
15	7412-12	.24	.001**			.3
16	7412-13	.14	.97	1.05		.47
17	747 - 6	.41			1.67	
18	747 - 7	.4			2.13	
19	747 - 8	.41			2.41	
20	747 - 9	.39			2.59	

* 9 came to close to 7 there was not much distinction and 15 showed no Cr or Ni as expected and hence turned out to be close to 4.

** Less than .001.

Table III. Results of chemical analysis of other elements that could be present and affect this investigation. (given in wt %).

Specimen No.	Compositions of the analyzed elements			
	B	O	P	S
1	---	*,001	---	---
2	---	*,001	---	---
3	---	*,001	---	---
7	---	*,001	---	---
5	*,001	*,001	*,005	*,005

*less than.

the kinetics of the reaction for future analysis.

It was known from previous works that the homogenizing treatment changed the chemical composition slightly therefore our results were taken after homogenization.

It was also important to check at random for other alloying elements such as O, B, S, P (which presence even in very low content affect the experiments considerably).

The concentrations of these elements were measured and was found to be less than .001% or .005%, the limits of detection methods available (chemical spectroscopic atomic absorption). Therefore it was concluded that our samples were free from these impurities. The results are listed in Table III.

B. Experimental

1. Apparatus Description

The experimental apparatus is a theta-dilatometer. It consists of a measuring module chamber (see Fig. 3), a vacuum system (Figs. 4, 5) and a recording system, that can record as fast as 20 in/min corresponding to 10^{-3} sec given a reading accuracy of .02 inches.²⁸

The recording, main power, and furnace control units can operate under manual or programmed instruction operating conditions. The transformations were measured as a function of their accompanying length changes as a consequence of their volume change. The specimens were mounted between the gap of two quartz holders with holes inside to let the quenching gas flow (See Fig. 2).

In these studies the interest was not in the absolute volume changes or length changes but rather on where the transformation (volume changes) starts and proceeds with time at a given isothermal hold or temperature gradient. Nevertheless, the equipment was calibrated each time a new set of runs was made, and zeroed for each sample specimen.

The vacuum system consists of a mechanical and a diffusion pump which permitted vacuums of $(10^{-5} - 10^{-6})$ torr range with addition of liquid nitrogen and otherwise $(10^{-5} - .5 \cdot 10^{-4})$ range.

The experimental runs were done in the second range after it was found that there was no decarburization and improving the vacuum did not change our conditions (no oxidation) at the austenitizing temperature treatment of 1080°C for 15 min.

The measuring module chambers and the main parts are specified on Fig. 3. The schematic shown in Fig. 2 slides in the furnace in such a way that the specimen is placed in the center of the induction coil before each treatment.

The equipment is designed in such a way that it is very sensitive to any length change giving an immediate signal in the recording system depending on the sensitivity level.

As in any horizontal dilatometer problems are introduced if the specimen is at any angle rather than aligned to the quartz holders because only the horizontal component of the length change would be recorded. Therefore extra care was taken when mounting each specimen within the chamber.

The thermocouple used was a Pt-(Pt, Rd) thermocouple attached

Table IV. (Experimental conditions of a typical run).

Typical gas pressures (He)	(20-35) LPM Air
Typical vacuums	(.510 ⁻⁴ -10 ⁻⁵) Torr
Typical quenching rates	(50-150)°/sec Martensite runs
	(150-300)°/sec Lower Bainite runs
	(300-450)°/sec Upper Bainite runs
Typical heating rates	6.73°/sec
Typical time resolution	10 ⁻² sec

to the outer surface of the specimen. Of course there is always going to be a temperature gradient with respect to the thickness but since the specimens are so thin (1/16") we will neglect this effect on the basis of heat transfer calculations that showed in the worst case a 4.5°C difference between the inner and outer surface.²⁹

2. A Typical Run

The main features of a typical run consist of heating the specimen under vacuum to the austenitizing temperature 1080°C. The specimen is held for 15 min to allow for carbide dissolution and quenched with He gas flow (before this step one must close the chamber from high vacuum and will remain closed for the rest of the run to avoid damaging the diffusion pump).

For the alloy compositions used the kinetics of the reactions were sufficiently fast that it was desirable to have the quenching rates as fast as possible. The heating was programmed at a speed of 12 in/min; thus gave a heating rate of 6.73°C/sec.

The experimental runs were made in such a way that in most cases the maximum quenching rate was used.

The maximum pressure of the He inlet is 30 LPM air which gave quenching rates depending on the ΔT s involved but the average values are given in Table VI

When isothermal runs were finished the specimen was again quenched to room temperature and finally (after using the ventilation valve to balance to atmospheric pressure) the specimen is removed, and the system is ready for the next run.

C. Thermilatometric Experiments

1. Austenite Transformation Temperature

Each experimental run consisted of heating the sample from room temperature to the austenizing temperature. In the temperature range where the austenite transformation occurred, the volume (therefore length) changes associated with the transformation were recorded to attain the values of austenite start and finish temperatures. Many runs were made on each composition. The values tabulated in Table V are averages of at least 8 runs.

2. Ms Temperatures

In a similar fashion the specimen was quenched to room temperature and the martensite transformation was recorded. This is a very useful information for the bainite studies in order to choose a range above the Ms for the kinetic investigation (Results in Table VI).

3. Isothermal Runs in the Bainite Reaction Range

The specimen is quenched to the desired temperature and held until the reaction is complete or saturated. For some cases however, it was found that after a maximum increase and saturation there was a very slow decrease. This was attributed to a relaxation effect of the quartz holders and was not considered as part of the transformation. In other experiments³⁰ for Nb, Al that had no reactions at these range of temperature there was no reaction but a slight slow decrease with time due to the quartz holders relaxation.

4. Graphical Examples of two Typical runs

The typical runs, as described previously, result in chart signals as indicated in Fig. 6 for the martensite transformation and Fig. 7 for a bainite transformation. It is worth pointing out that the temperature and length changes have a lag of (1/16") so this must be considered when As, Af, Ms, Mf are recorded.

The heating rate, quenching rate, and austenitizing treatments are pointed out for the martensitic transformation, in the case of continuous cooling (Fig. 6), and for the bainite transformation in the isothermal run (Fig. 7) the changes in scale have been pointed out to give an idea of the time elapsed in the different regions and holds.

D. Hardness Testing

By means of a Leitz-Wetzlar microhardness testing unit the Vickers microhardness values of heat treated specimens were determined. An applied load of 1000 g was used in measuring the hardness of each specimen from metallographic samples polished and prepared for optical microscopy before etching. Both diagonal indentations were measured and an average value (from five tests made on each specimen) is given as the hardness value (Table IX for the martensitic structures and Table XXVI for some of the bainitic structures). These tests were made to compare their relative hardness values and to see if there was any effect from the alloying elements.

E. Optical Microscopy

The limitations of optical microscopy are recognized for morphological considerations.⁵¹ The best means of distinguishing the detailed structures would have been transmission electron microscopy, however due to the size and shape of the specimens for dilatometric studies it was not possible to prepare thin foils.

Some pictures where martensites and bainites are present are shown in a later section for the purpose of comparison and to see the possible differences due to the differences in alloying elements present.

After each run most of the specimens were mounted in koldmount. Specimens were ground on Si-carbide papers to 600 grit, and polished on a 15 abrasive diamond paste wheel followed by .05µ alumina slurry.

All the specimens for structure observation were etched with 2% nital solution, and where prior austenite grain boundaries were desired a saturated pichral solution with a few drops of HCl was used.

The results of the ASTM austenite grain sizes for the heat treatment at 1080°C for 1' min are tabulated in Table XXVII. There were variations from specimens of different composition and in some cases within the same composition they usually were within ASTM (3-4) or (4-5) range which implies a coarse grain size.

III. RESULTS AND ANALYSIS OF DATA

A. Austenite Transformation Temperatures

The data obtained are given in Table V. The results were obtained for the bcc to fcc austenite transformation on treating the specimen from room temperature to the austenitizing temperature (1080°C). For these data the heating rate was fixed by a programmed heating treatment where the 1080°C was reached in 2.63 minutes (read on chart as 5.25 inches on the 2 in/min scale). This implied a typical heating rate of 6.73°/sec.

The austenite start was detected from the initial slope change and the austenite finish from the final change before the pick-up of the heating rate.

The validity of these results lie within the limits of how much austenite forms before any recordable signal. For example we must understand that As is not .001% transformed but more likely 3% transformed and similarly for Af.

The values given in Table V are average over 3 or more runs (except in the cases of (1, 2, 3, 18) where only 4 heatings were considered).

B. Ms Temperature and Hardness Values

1. Martensite Transformation Temperatures

The martensite transformation temperatures of the different alloys were obtained under the following experimental conditions: Heating

Table V. Austenite transformation temperatures.

Specimen	As (°C)	At (°C)
1	740	940
2	760	940
3	760	940
4	760	930
5	765	920
6	742	830
7	715	819
8	730	840
9	727	830
10	760	850
11	744	905
12	753	857
13	749	875
14	736	848
15	757	937
16	747	853
17	728	770
18	720	765
20	715	750

Table VI. Martensite transformation temperatures.

Specimen	M _s (°C)	M _f (°C)	Quenching Rate (°C/sec)
1	* 497	430	66.6
2	* 528	475	60.7
3	* 502	387	111.2
4	445	307	83.3
5	416	355	53.3
6	396	310	60.7
7	403	270	111.6
8	390	210	148.2
9	400	273	133.3
10	377	280	133.3
11	350	250	102.5
12	380	224	58.9
13	405	276	70.5
14	405	290	66.7
15	418	330	74.0
16	428	310	84.0
17	330	185	133
18	298	130	133
20	300	113	121

* For these compositions with the quenching rates available it was not possible to avoid the nose of the bainite reaction which is too fast due to the low C content and the presence of Mo.

rate $6.73^{\circ}/\text{sec}$ with an average quenching rate of $85^{\circ}/\text{sec}$ *.

The same pressure of the He gas lead to slight variations from the average QR = $85^{\circ}/\text{sec}$. In order to be consistent in comparative studies we checked the effect of the quenching rate at these levels on the Ms values and results indicated no change (as discussed in C.2). So we could place all Ms from different composition and different runs in the same comparative scale despite slight differences in quenching rates.

An Mf determination is very difficult to make. The martensite reaction is often incomplete even at absolute zero temperature.²⁶

The transformation of the last traces of austenite is difficult and there is usually a small amount of retained austenite in the structure of overlapping martensite plates. So one should interpret these Mf as the temperature of which the reaction stops. (Such as indicates in Fig. 6.)

2. Effect of the Quenching Rate on Ms

There was no effect of the quenching rate (within the available ranges) on the Ms transformation temperature for the four alloys studied.

The Ms was not affected by the quenching rate for austenizing temperature of 1080°C or 950°C . Results in Table VII. It showed no change irrespective of the austenitizing treatment.

* This quenching rate is an average over the whole ΔT . Actually the first 600° of quenching have a much faster rate QR = $180^{\circ}/\text{sec}$ which corresponded to a He pressure inlet of 30 LPM air.

Table VII. Effect of Quenching rate on Ms.

Specimen No.	Austenite Temp. (°C)	Quenching Rate (°C/sec)	Ms (°C)
7	1080	111.6	400
7	1080	58.3	400
9	1080	133.3	400
9	1080	83.3	400
11	1080	102.5	350
11	1080	84.12	350
11	1080	100.9	350
18	1090	50.92	300
18	1085	126.19	300
18	1085	133.	300
18	950	52.7	280
18	950	126.2	230

It was found, for example, in composition 18 that the M_s was 300, 290 for 1080°C and 950°C respectively and these did not change for different quenching rates.

3. Effect of Austenitizing Temperature on M_s

The analysis of the data Table VIII showed that there is a distinct rise of the M_s temperature when the austenitizing temperature is increased.

For two of the cases see Fig. 3(a, b).

4. Hardness Tests of M_s Structures

The Vickers microhardness test for 1000 g load was used. Besides the available compositions three other plain carbon alloys were treated in the same way. With the resulting M_s average values (for comparative purposes)

A	.32C	370°C
B	.59C	255°C
C	.88C	200°C

Results are tabulated in Table IX and a plot of microhardness versus carbon content is given in Fig. 9. where the different alloying elements involved are distinguished for each point. A plot of microhardness versus M_s temperature is also plotted in Fig. 10. A linear regression formula for these results is given in Appendix.

E. Bainite Transformation Data

1. Isothermal Transformation for the Various Alloys

The data are presented in a series of Tables from X to XXII where the run number, the corresponding temperature and the times for the initial part of the transformation (recordable reaction start)

Table VIII. Effect of austenitizing temperature on Ms.

Specimen No.	Quenching Rate (°C/sec)	Austenite Temp. (°C)	Ms (°C)
18	133.3	1085	300
18	126.2	1085	300
18	60.9	1090	300
18	126.2	950	280
18	52.7	950	230
9	73.7	1210	410
9	133.3	1080	400
9	80.3	950	390
15	49.1	1200	430
15	63.1	1090	420
15	43.1	1085	418
15	74	955	410

Table IX. Microhardness Vickers test with 1000 g load.

Specimen No.	Ms (°C)	VH	C(wt %)
1		*277	.17
2		*226	.18
3		*308	.17
4	445	396	.24
5	416	440	.27
6	396	533	.3
7	403	453	.23
8	390	528	.29
9	400	445	.20
10	377	422	.29
11	350	398	.17
12	380	490	.27
13	405	464	.25
14	405	470	.24
15	418	385	.25
16	428	399	.14
17	330	575	.41
18	298	655	.4
20	300	716	.39
A	370	520	.32
B	255	716	.59
C	200	936	.88

*1, 2, 3 will not be considered for the graphs because as discussed they were not martensitic structures.

Table X. Isothermal data for alloy 4.

Run No.	Temp. (°C)	t_o (sec)
1	450	.36
2	480	1.26
3	500	2.16
4	520	.48
5	540	2.04
6	600	4.44
7	650	8.76

Table XI. Isothermal data for alloy 5.

Run No.	Temp. (°C)	t_o (sec)
1	435	.281
2	450	3.1
3	460	.563
4	490	.563
5	512	.609
6	555	.703
7	610	1.406
8	650	24.36

Table XII. Isothermal data for alloy 6.

Run No.	Temp. (°C)	t_o (sec)
1	407	1.2
2	423	1.56
3	445	2.52
4	470	1.8
5	487	1.92
6	520	4.92
7	580	5.52
8	600	11.16
9	650	81.36

Table XIII. Isothermal data for alloy 7.

Run No.	Temp. (°C)	t_0 (sec)
1	420	.24
2	440	.3
3	450	3.
4	460	1.32
5	480	.6
6	500	.48
7	550	.48
8	600	.96
9	650	33.1

Table XIV. Isothermal data for alloy 8.

Run No.	Temp. (°C)	t ₀ (sec)
1	415	.564
2	425	.36
3	440	.564
4	460	1.5
5	489	1.734
6	512	1.734
7	560	.378
8	610	.936
9	660	3.654

Table XV. Isothermal data for alloy 10.

Run	Temp. (°C)	t_o (sec)
1	385	.12
2	400	.24
3	425	.36
4	440	.48
5	450	1.2
6	460	.36
7	490	.72
8	510	2.4
9	550	.6
10	600	.9
11	650	1.8

Table XVI. Isothermal data for alloy 11.

Run	Temp. (°C)	t_0 (sec)
1	340	.0
2	365	.12
3	400	.0
4	420	.72
5	540	1.56
6	480	.63
7	500	2.4
8	545	2.88
9	600	4.32
10	650	8.28

Table XVII. Isothermal data for alloy 12.

Run	Temp. (°C)	t_o (sec)
1	410	4.8
2	435	6.36
3	460	7.08
4	480	8.88
5	500	4.8
6	540	11.1
7	600	28.08
8	650	26.1

Table XVIII. Isothermal data for alloy 13.

Run	Temp. (°C)	t_0 (sec)
1	420	2.16
2	440	9.72
3	460	4.32
4	480	2.7
5	500	3.96
6	540	13.2
7	600	20.04
8	640	426.9
9	650	366.

Table XIX. Isothermal data for alloy 14.

Run	Temp. (°C)	t_o (sec)
1	415	2.28
2	440	4.08
3	460	5.64
4	475	6.6
5	490	2.4
6	540	15.48
7	600	21.72
8	645	156.

Table XX. Isothermal data for alloy 16.

Run	Temp. (°C)	t_o (sec)
1	420	.12
2	440	.24
3	460	.84
4	480	.36
5	500	.12
6	540	55.56
7	590	181.

Table XXI. Isothermal data for alloy 17.

Run	Temp. (°C)	t_0 (sec)
1	350	13.2
2	400	24
3	455	3.
4	500	4.2
5	560	7.
6	610	37.5
7	650	

Table XXII. Isothermal data for alloy 20.

Run	Temp. ($^{\circ}\text{C}$)	t_0 (sec)
1	328	45.
2	350	43.59
3	372	48.75
4	400	26.06
5	425	15.00
6	455	12.18
7	505	12.65
8	560	38.9
9	600	78.

are given. The corresponding diagrams showing these results for the kinetics of bainite are given in the next section.

2. Diagrams for the Reaction Start of the Austenite to Bainite Transformation

These are given from Figures 11 to 23. They are built on the basis of the dilatometric isothermal data only the starting time of the transformation is given although the specimens were held 15 min isothermally or until reaction was completed or saturation was reached for structure observation which was on the $(10-10^2)$ sec range for most specimens except for the higher temperature range where it could take as long as (10^3-10^4) sec.

3. Effect of the Quenching rate on the Kinetic and Reproducibility

The effect of the quenching rate was studied in two alloys (9, and 15) in order to see if there were any changes in the kinetics and also how the variation of the quenching rate affected the reproducibility.

The data was divided in two parts. For that purpose it will be tabulated in XXIII and XXIV.

From these data, although the evidence is not conclusive it suggests that with slower quenching rates the kinetics of the reaction are faster at the initial part of the transformation but not for further progress of the reaction.

For 9 at 1095°C the reaction should be slower than at 1090°C austenitizing temperature treatment, and it is except for the incubation and initial part of the reaction which seems to be affected by the fact that the quenching rate is ten times slower.

Table XXIII. Effect of quenching rate on the bainite kinetics.

Alloy	QR °C/sec	Aust. Temp. °C	Isoth. T °C	t_0 sec	t_{25} sec	t_{50} sec	t_{75} sec	t_5 sec
9	148.1	1090	450	.34	.54	.86	1.22	3.34
9	12.9	1095	450	.28	1.16	1.6	2.2	8.72
9	118	1090	450	.29	.52	.75	1.05	3.27
15	133	1085	520	.9	1.38	1.62	1.92	2.56
15	148	1085	520	.95	1.34	1.6	1.9	2.5
15	70.1	1085	520	.7	1.1	1.3	1.56	2.3

Table XXIV. Effect on reproducibility.

Alloy	QR °C/sec	Aust. Temp. °C	Isoth. T °C	t ₀ sec	t ₂₅ sec	t ₅₀ sec	t ₇₅ sec	t ₅ sec
9	118	1085	450	.36	.74	.98	1.3	3.34
9	148.1	1090	450	.34	.54	.86	1.22	3.34
9	118	1090	450	.29	.52	.75	1.05	3.27
15	133	1985	520	.9	1.38	1.62	1.92	2.56
15	148	1085	520	.95	1.34	1.6	1.9	2.5
15	69.26	1090	520	.78	1.22	1.41	1.72	2.58
15	70.1	1085	520	.52	.83	1.12	1.37	2.58
15	70.1	1085	520	.7	1.1	1.32	1.56	2.3

In the case of 15 the same thing happens with little effect on the rest of the transformation.

With slight variations in the quenching rate the reproducibility is good. The results would be clearer when interpreted from Fig. 24 and 25 they give an idea of the errors involved by assuming reproducibility. However when the quenching rate is not varied outside the range we work with (130-150°/sec) the change is almost negligible as the data for 15 shows Table XXIV.

4. Effect of the Austenitizing Temperature on the Bainite Kinetics

The effect of the austenitizing temperature was studied on alloys 9 and 15 also, with consistent results for both.

The results showed the lower the austenitizing temperature the faster the reaction started (nucleation) and successively completed or saturated.

The results are in Table XXV. The times for different percentages of saturation level of the reaction are given as well as the corresponding isothermal hold and quenching rate.

Figure 26 and 27 show the effect of austenitizing temperature on the bainite kinetics and the time for progressive stages of the transformation as a function of the austenitizing temperature respectively. For the latter the relation suggested linearity up to 75% of the saturation level (where the reaction stops).

Table XXV. Effect of austenitizing temperature on the bainite kinetics

Alloy	Aust. Temp. °C	Trans. Temp. °C	QR °/sec	t_0 sec	t_{25} sec	t_{50} sec	t_{75} sec	t_s sec
9	1215	450	197.2	.42	.84	1.1	1.37	3.58
9	1090	450	148.1	.27	.52	.75	1.05	3.27
9	950	450	154.3	.1	.4	.56	.74	2.76
15	1200	520	94.4	1.02	1.44	1.78	2.12	4.04
15	1090	520	61.7	.78	1.22	1.41	1.72	2.58
15	955	520	68.4	.26	.62	.98	1.25	1.86

Table XXVI. Hardness of Some Bainitic Structure.

C wt %	Alloy No.		Temperature °C								
			Vickers Hardness Number at 1000 g load								
.27	5	Mo	Ms 416	435	460	490	512	610			
			494.8	308.7	310.6	260.6	243.16	186.56			
.29	8	Ni	Ms 390	415	440	460	489	512	560	610	660
			498.8	309.2	259.8	243.8	227.8	203.6	195.4	162.6	154.6
.29	10	Cr	Ms 277	400	425	450	475	500	550	600	
			422	422	381	326	264	262	211.5	197.5	
.4	17	Mn	Ms 330	350	400	455	500	560	610		
				459	374	300	263	239	220		

5. Hardness Tests of Bainitic Structures

To study the effect of alloying elements on hardness of different bainite structures 4 alloys were studied containing Mo, Ni, Cr, Mn. Vickers hardness values were obtained (average of 5 measurements of each specimen) for some of the bainite structure at different temperatures. Results tabulated (Table XXVI) are plotted in Fig. 28 and 29. Where it was possible alloys with similar carbon content were chosen because the C content is very critical in hardness values. It shows that the behavior is alike with respect to the transformation temperatures although for each alloy the hardness could be lower or higher there was not a wide variation range.

F. Optical Microscopy and ASTM Grain Size Characterization

1. ASTM Grain Size of the Treatments Used

The austenitizing temperatures for all the diagrams was 1080°C. It was held there for 15 min. The corresponding ASTM grain size for all the compositions studied are given in Table XXVII. We can say the majority of them (except those with low C content) lay within (3-4) (4-5) ASTM numbers.

2. Effect of Austenitizing Temperature on the Grain Size

The grain size showed a distinct increase with higher austenitizing temperatures; a change of 950°, 1080°, 1200° gave ASTM corresponding numbers of (6-7), (3-4), (1-2). Which implies an increase in all equivalent spherically shaped grain from diameter $\{ (1-2), (4-5), (8-10) \} \times 10^{-3}$ inches. With respect surface area of 1.57×10^{-6} , 3.18×10^{-5} , 1.27×10^{-4} in².

The results tabulated for two alloys studied in Table XXVIII also plotted in Fig. 30 where d means the diameter of the

Table XXVII. ASTM Grain Size for the alloys at 1080°C (hold 15 min).

Spec. No.	ASTM	Spec. No.	ASTM
4	3-4	12	1-2
5	3-4	13	2-3
6	3	14	3
7	4-5	15	3-4
8	3	16	3-4
9	4	17	5
10	5	18	4
11	1	20	5

**Table XXVIII. ASTM grain size for different austenitizing temperature.
(15 min).**

Spec. No.	AT (°C)	ASTM (No.)
9	950	6
9	1080	4
9	1210	2
15	955	6-7
15	1085	3-4
15	1200	(1-2)

corresponding spherically shaped grains. All the results are derived from holding at the austenitizing range for 15 min.

3. Optical Microscopy of the Martensitic Structures

Some optical microscopy studies were made. In no way is this an attempt of a morphological study, but it was done to get an overview of the different alloy's martensitic and bainitic structures. As was discussed previously the limitations of optical microscopy for these purpose is recognized. The structures were taken, except where specified at 1000X magnification. Figure 31 through 49 show the martensitic structures of 19 of the casted alloys. ^{*}(1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 20).

4. Optical Microscopy of the Bainitic Structures

These are shown only for four of the alloys studied 5, 8, 10, 17 which correspond to Fe-C-Mo, Fe-C-Ni, Fe-C-Cr, Fe-C-Mn system. Different bainitic transformation temperatures and their resulting structures are shown for each alloy.

Figures 50, 51, 52, 53, and 54 correspond to alloy 8 (Fe-C-Ni) at isothermal holds 660°C, 512°C, 489°C, 450°C, 400°C. Figures 55, 56 and 57 correspond to alloy 5 (Fe-C-Mo) at 610°C, 512°C, 435°C. Figures 58, 59, 60 and 61 correspond to alloy 10 (Fe-C-Cr) at 600°C, 555°C, 490°C and 460°C. Finally Figs. 62, 63, 64 and 65 correspond to alloy 17 (Fe-C-Mn) at 610°C, 560°C, 400°C and 350°C.

* Note as was pointed out previously 31, 32, 33 are not martensitic and 34 is not fully martensitic due to interference with fast pearlite and bainite kinetics.

It was attempted to have both types of bainite showing upper and lower bainite although in some cases they are mixed and a distinction is not easy.

V. RESULTS AND DISCUSSIONS

A. Results on the Austenite Transformation Temperature and the Effect of Different Alloying Elements

In any hardening process on heat treatment the heating of the steel is one of the most important steps. Careful considerations should be taken because the final properties are profoundly altered by the degree of carbide dissolution, also affected is the degree of homogenization of the alloying elements and carbon.

For many steels, specifically commercial steels, the optimum austenitizing temperature have been established by a long process of experience. However even here the actual period of time during which diffusion may occur is not stressed. (Bain, Paxton)²⁶

"No useful diffusion occurs until austenite is established (at a certain minimum temperature) and, thereafter it occurs at a rate that increases very rapidly with increasing temperature of heating.

Furthermore, the different elements diffuse at such different velocities that, in principle, at least each composition should be so heated as to assume the required homogeneity of austenite".

The ternary equilibrium systems for some elements have been studied and the results of those for Mo, Mn, Cr²⁶ will be compared with the results.

Table V shows the data for which the Ae_1 (As) eutectoid and Ae_3 (Af) temperatures will be compared.

Comparative results showed that Cr and Mo restricted the austenite stable region. With Mo being most effective and that Mn expanded the austenite stable range slightly while Ni exerted little influence which was in agreement with published results.

It can be said from their results that the choice of 1080°C as austenitizing temperature for C contents below .4 weight % was a good choice, that enables the temperature of maximum dissolved carbon in austenite for our alloys.

For steels that have two or more alloying elements systematic studies have been done in very few instances so no comparison was made. It is suggested that if one alloy raises and the other lowers the transformation temperature the effect is not additive or average.

Also note for instance the fact that Fe-C-Cr(10), and Fe-C-Mo(6), and Fe-C-Cr-Mo(14). Show that their interaction effect of Cr-Mo is less effective in raising A_{e_1} than their independent effect.

B. Results of the Ms and Discussion of Some Available Empirical Formulae for Ms Determination on the basis of Alloy Content

It is known that the Ms temperature is markedly affected by the composition of the austenite (being primarily determined by the carbon content and to a lesser extent by the alloy content).

The results of the Ms measurements showed that the quenching rate within our variation range had no effect on the Ms temperature. This result is consistent with a recent publication by Donachie and Ansell³¹ where a study over a wide variation of quenching rates and its effect on Ms was made.

According to their study, the M_s as a function of quenching rate indicated a nominal plateau for slow quenches (which is our case). Also it indicated that the first changes in the M_s occur at a quench rate of $8,300^\circ\text{C}/\text{sec}$ (lower critical rate) and that no further changes in M_s occurred at rates above $18,300^\circ\text{C}/\text{sec}$ (upper critical quench rate) and there was a maximum increase of 73°C for high C alloy and 40° in low C alloy.

Figure 66, shows where our quench rate lies (within a very low quenching rate of their study).

The results of the M_s also showed that the austenitizing temperature affected the M_s values. It was observed that the higher the austenitizing temperature the higher the M_s . (For 18 from 950°C to 1085°C there was a corresponding 20°C increase, for 9 from 950 to 1210°C there was a corresponding 20°C increase, for 15 from 955 to 1200 there was a corresponding 20°C).

These results are consistent with prior studies by Prakash Babu¹⁷ and Ansell and Breinan³² and Sastri and West³³ who observed an increase of 25°C in the M_s from 800°C to 1200°C change in austenitizing temperature.

The increase in the M_s is believed to be due to the increase in grain size at higher austenitizing temperatures which could result in a decreased flow strength of the prior austenite. The decreased resistance of the austenite matrix to the deformation accompanying the formation of martensite caused this increase.

There have been several formulae suggested for the determination of M_s which presuppose that all the carbides have been dissolved and are usually a result of statistical empirical averages on

different alloy steels.

These formulae in some cases have been discussed by their authors as being limited to a linear dependence approximation which in many cases is far from the actual case.

An equal determination of the temperature at which the reaction ends M_f is an extremely difficult task especially because even experimental determination is not consistent as it was pointed out earlier. There are indications as early as 1946 (Grange and Stewart)³⁴ that carbon depresses the M_f temperatures even more markedly than it does the M_s temperatures.

The formulae studied here for comparative purposes with experimental values are the following:

1. Additive types

Payson and Savage³⁵

$$M_s \text{ } ^\circ\text{F} = 930 - 570C - 60Mn - 50Cr - 30Ni - 20Si - 20Mo - 20W \quad (1)$$

Grange and Stewart³⁴

$$M_s \text{ } ^\circ\text{F} = 1000 - 650C - 70Mn - 35Ni - 70Cr - 50Mo \quad (2)$$

Nehrenberg³⁶

$$M_s \text{ } ^\circ\text{F} = 930 - 540C - 60Mn - 40Cr - 30Ni - 20Si - 20Mo \quad (3)$$

Rowland and Lyle³⁷

$$M_s \text{ } ^\circ\text{F} = 930 - 600C - 60Mn - 50Cr - 30Ni - 20Mo - 20Si - 20W \quad (4)$$

Stevens and Haynes³⁸

$$M_s \text{ } ^\circ\text{F} = 1042 - 853C - 60Mn - 30Cr - 30Ni - 38Mo \quad (5)$$

Andrews³⁹

$$M_s \text{ } ^\circ\text{C} = 539 - 423C - 3014Mn - 17.7Ni - 12.1Co - 7.5Mo \quad (6)$$

11. Product types

There have been some attempts to take into account second order and higher order terms using product formulae of the alloy and C content.

Carapellas⁴⁰

$$M_s \text{ } ^\circ F = 925(1-.62C)(1-.092Mn)(1-.033Si)(1-.045Ni)(1-.070CrO) \\ (1+.120Co)(1-.029Mo)(1-.018W). \quad (7)$$

Andrews³⁹

$$^* M_s \text{ } ^\circ C = 512-453C-16.9Ni+15Cr-9.5Mo+217(C)^2-71.5(C)(Mn) \\ -67.6(C)(Cr) \quad (8)$$

* tried to account for interaction effects and variation from linearity.

The results from these calculations are shown in Table XXIX and the variations involved with respect to the present experimental results are given in Table XXX.

Results from these calculations showed that Nehremberg's linear formula gave the best fit followed by Payson and Savage and Andrews product formula considering interaction effects.

The major discrepancies for Nehremberg's values coincided with very low C content with respect to the rest of the alloys (.14 for instance) or the ones that included Cr because it is known that Cr interaction effects are strong and not linear.³⁹ Attempts to consider the second order interaction effects in the present cases does not give improved values.

One thing is evident from these type of studies, that is: we are in need of formulae to determine transformation temperatures but on more theoretical grounds; where the factors involved in these

Table XXIX. Ms as determined by some calculations from existing formula and comparison with observed experimental value in °C

Spec. No.	Linear (Additives)							Product	
	Ms Exp.	Payson and Savage	Granger and Stewart	Nehrenberg	Rowland and Lyle	Stevens and Haynes	Andrews	Carapella	Andrews
4	445	420	450	423	416	441	435	416	413
5	416	409	437	414	405	425	421	406	402
6	395	398	424	403	393	409	408	387	391
7	403	392	421	395	388	417	407	382	385
8	390	390	422	395	385	406	398	385	382
9	400	402	431	405	398	432	419	391	396
10	377	379	402	390	374	407	404	454	394
11	350	417	442	426	414	463	455	497	445
12	380	367	387	377	363	391	393	441	384
13	405	398	422	402	394	415	412	391	391
14	405	390	406	400	386	421	422	466	406
15	418	415	445	419	411	435	430	413	409
16	428	405	419	413	403	450	446	499	429
17	330	314	336	320	307	312	315	307	314
18	298	301	322	308	295	301	286	281	304
20	300	289	307	296	283	291	295	279	296

Table XXX. ΔM_s as determined from the formulae in °C.

Spec No.	Ms. Exp.	ΔM_s (°C)							
		Payson and Savage	Granger and Stewart	Nehrenberg	Rowland and Lyle	Stevens and Haynes	Andrews	Carapella	Andrews
4	445	-25	5	-22	-29	- 4	-10	-29	-32
5	416	- 7	21	- 2	-11	9	+ 5	-10	-14
6	395	+ 3	29	8	- 2	14	13	- 8	- 4
7	403	-11	18	- 8	-15	14	3	-21	-18
8	390	0	32	5	- 5	16	- 2	- 5	- 8
9	400	2	31	5	- 2	32	19	- 9	- 4
10	377	2	25	+13	- 3	30	27	77	17
11	350	67	92	76	64	113	105	147	95
12	380	-13	7	- 3	-17	11	13	61	4
13	405	- 7	17	- 3	-11	10	7	-14	-14
14	405	-15	1	- 5	-19	16	17	61	1
15	418	- 3	27	1	+ 7	17	12	5	- 9
16	428	-13	- 9	-15	-25	22	18	+71	1
17	330	-16	6	-10	-23	-18	-15	-23	-16
18	298	3	24	10	- 3	3	-12	-17	6
20	300	-11	7	- 4	-17	- 9	- 5	-21	- 4
$\Sigma \Delta M $		198	351	190	253	338	283	579	247
$\overline{\Delta M}$		12.38	21.94	11.88	15.81	21.13	17.88	36.19	15.44

determinations of transformation temperatures from the chemical composition should be explained from basic principles.

C. Results on the Hardness Test Readings

It has long been known that the effect of C is fundamental in determining the properties of martensite (i.e. hardness).

Small additions at low concentrations are more effective than equal increments at higher concentrations.

A secondary factor is the composition of austenite in terms of alloys other than C where the C content is relatively low and the quench is to develop maximum hardness.

In the present case of low C contents (except for the plain C steels A, B, C for comparison) the alloy composition made some differences. Results indicated that Mn and Ni seem to be higher than plain C steel but Mo and Cr varied.

A comparative curve for plain C steels for 15 kG load on Vickers test is given in Fig. 9

A linear function seems to fit approximately the Microhardness versus M_s transformation temperature results. The lower the M_s the higher the hardness. Since M_s is a function of C and the alloying elements there is a relation between hardness and alloying elements. (Fig. 10).

The approximate quantitative function gave the following results. See Appendix for detailed formulas.

$$VHN(1000 \text{ gr}) = -2.11 \frac{[VHN]}{^{\circ}\text{C}} \times M_s(^{\circ}\text{C}) + 1306.4 [VHN] \quad (9)$$

with a coefficient of determination $r^2 = .93$

Hardness values for the different bainite structures decrease with increasing temperature of transformation the results did not give a linear function but results seemed to be consistent with prior results on tensile strength versus different transformation temperatures² for low carbon steels. These results were consistent for the different alloys present Ni, Cr, Mo, Mn (Fig. 28-29).

D. Results of the Bainite Reaction (above the Ms)

1. Reaction Start Diagrams Discussion for the Different Alloys (Kinetics)

In the present studies there was an acceleration of the austenite decomposition at isothermal temperature treatments just above the Ms giving as result for the reaction start curves, S-shaped rather than a C-shaped curves.

This decrease in the incubation period for the nucleation of lower bainite was observed consistently in all of the 13 cases studied, in some cases more pronounced.

S-shape curves for the bainite reaction have also been reported by other investigators.^{17, 19, 41, 42, 43}

although their studies were mainly on commercial steels (where many alloys have been also present). For the present ternary and quaternary systems studied this acceleration is most alloys was also the case.

It has to be pointed out that this curves are extrapolation

from the experimental data shown in Figs. 11 through 23. So the information of what happens in between could be different from this curves although big changes in nucleation times through small temperature ranges should not be expected. This was found in some instances (Fig. 5, 7, 12, 16 and 17) which from a thermodynamic point of view could be questionable.

In some cases separate C curves for lower and upper bainites start to show as for 8 (.29C, 1.01 Ni) and 11 (.17C, 1. Cr) and in one case where C was very low 16 (.14 C, .97 Cr, 1.05 Ni, .47 Mo) the S-shape was also observed.

The relative dominance of upper and lower bainite reaction is not only determined by the carbon diffusion rates but also by the nucleation rates of carbides and alloys present. The alloy composition plays a major role in determining these factors. Heheman⁹ claims that the rate at which precipitation of carbide occurs from the supersaturated ferrite is responsible for the difference in morphology between upper and lower bainite.

The present results showed that all of the alloying elements studied (Ni, Cr, Mn) except Mo shifted the bainite curves to longer times, increasing the incubation period and lowering the kinetic bainite start. Different alloying elements affected this differently.

Mo instead accelerated the bainite reaction and raised the Ms temperature. As it was shown previously the fact that the reactions were too fast did not permit the study of alloys 1, 2, 3 as planned because of experimental equipment limitations.

1. If one concentrates in observations on the upper bainite nose for the different alloy; then
 - a. No addition lowered the temperature (of shorter incubation time bainite nose) and accelerated the bainite reaction.
 - b. Ni addition decreased the temperature of the nose also, such that 2.04% was more effective than 1.01% in retarding the bainite reaction (despite the fact that the C content was higher for the latter)
 - c. Cr and Mn behaved similarly but in order of effectiveness 1.67% Mn was more effective than 1.2% Ni which was more effective than 1.0% Cr in lowering the Bs; although equally effective on the hardenability for equal C content (Refs to Ni, Cr, above).
 - d. Combined additions 1. Cr + 1. Ni was much more effective than 1 Cr and/or 1 Ni implying that the interaction is very strong in retarding the bainite reaction, which turned out not to be additive but a multiplicative effect. (at least 6 times more effective than the addition of Cr and Ni alone).

With respect to lowering the Bs results showed the effect is not as marked and rather seems to be additive.

- e. For combined additions of .48 Mo + 1. Ni and .99 Cr + .47 Mo, Mo-Ni was more effective than Mo-Cr and again the retardation of bainite was not an additive effect. However the Bs decrease suggested additive interactions.
 - f. For the C-Cr-Ni-Mo combined effect, unfortunately the C content resulting from the initial cast was very low. Nonetheless, for such low C content the presence of Cr-Ni-Mo and their interaction was strong enough to bring it into the present ranges of detectable

times for nucleation, which would not be the case if only F-C-Mn were present.

The shifting of the bainite reaction to lower temperatures restricts greatly the upper bainite range and promotes formation of lower bainite giving finer bainitic structures and hence improving the properties in the steels, because it is the upper bainite what is detrimental.

Hence these type of studies are necessary when maximizing design considerations.

2. If one concentrates in observations on the upper-lower bainite bay for the different alloys, then

a. Table XXXI shows the same tendency for the different alloys and the temperature of the maximum incubation times for the bays. Again the most effective results on the hardenability seem to come from the combinations Ni-Mo, Ni-Cr, Cr-Mo (in decreasing order of effectiveness).

Although Mn seemed the most effective for hardenability by itself. If only one alloy was considered it seemed additive with increase of alloy content otherwise this was not the case as for example Mn and Ni.

b. A plot of the position of the bay with respect to the C content is done in Fig. 67 where it was compared with a previous study by Pickering.

It shows the tendency is maintained although as it is expected is below in temperature scale because the location of the bay does not indicate the start of the lower bainite transformation but an

overlap of both upper and lower bainite kinetics.

Comparison with previous studies on commercial steels showed consistency with these results.

The closest systematic study done in the field is that followed by Prakash Babu with his magnetic permeability technique on 4340 steels with alloying elements added.¹⁷ It was found in the present study as well that the lowering of the Bs temperature range seemed to be additive, whereas the rate of bainite formation was not.

It was retarded by increasing amounts of alloying elements (with the exception of Mo) when added alone.

This retardation effect was not linear. It was found that the effect of 2.04 Ni and 1.01 Ni, $Ni/Ni = 1.85$ had a ratio on the nucleation time effect of 1.27 is less than if the effect were linear (in the present investigation).

From Mn 2.55, 1.67 with Mn/Mn 1.53 had a ratio on the nucleation times of 4 which is more than twice the additive effect.

For Ni-Cr/Ni = 2.1 the ratio on the nucleation time was 12.7 which is more than six times the additive effect.

For Ni-Cr/Cr = 2.1. It was the same as the later.

For Mo-Ni/Ni = 1.48 the ratio was 7.03 which corresponds to five times the additive effect.

For Mo-Cr/Cr = 1.46 the ratio is 6.5 is more than four times the additive effect.

If we try to correlate these numbers we can say only that Mn and Cr when added alone lead to multiplicative effects Ni gave more of an additive effect when added alone.

If combined; interaction effects played a major role where in order of effectiveness, Ni+Cr followed by Mo+Ni and Mo+Cr in retarding the bainite reaction.

The observation that Ni is more effective in the presence of Cr is in agreement with that of Prakash Babu¹⁷ and that of Brophy and Miller's result.⁴⁴

It is recognized that these are qualitative inferences from our data. But the effects of combination of two or more alloying elements on the bainite transformation were too complex to infer as to what extent the effects of one element are weakened or strengthened by the presence of another element.

It was not expected that the results would be similar to those done on 4340 alloy which contain .39 C, .7 Mn, .28 Si, .76 Cr, 1.7 Ni, .2 Mo, and .22 Cu. Because other interaction effects were believed to be present here. Nonetheless the results were surprisingly similar as if for Fe-C-X systems.

What these seem to indicate is that the other alloying elements present such as Cu, Al might play a minor role in this reaction or that their individual interaction effects cancel each other and such alloys as Cr, Ni, Mo and Mn affect significantly the bainite kinetics.

Attempts were made to correlate the present results with C content, alloy content, lattice parameter mismatch, C diffusion and how it is affected by different alloys present for carbon diffusion in austenite and or ferrite with no success. These results were difficult to rationalize and are just presented as empirical

Table XXXI. Temperature at which the upper-lower bainite bay is located.

Spec. No	C	Mo	Cr	Ni	Mn	Bainite Reaction Start Diagrams
4	.24	.29				500
5	.27	.39				450
6	.3	.49				445
7	.23			2.04		450
8	.29			1.01		470
10	.29		1.			450
11	.17		1.			450
12	.27		1.02	1.1		480
13	.25	.48	1.			440
14	.24	.47	.99			475
16	.14	.47	.97	1.05		460
17	.41				1.67	400
20	.39				2.55	380

facts within the limits of our experimental technique.

2. Discussion on the Effect of Austenitizing Temperature on the Kinetics

The effects of austenitizing temperature showed that the lower the austenitizing temperature the faster the reaction on one case the lower bainite effect on specimen number 9 was studied in the other the upper bainite effect on 15 was studied.

From our results it shows that the austenitizing temperature has more effect in the incubation time for upper bainite formation than for lower bainite (although more data should be required to assure this). There has been conflicting data in the literature as to what the effect of austenitizing temperature is on the bainite reaction. Those that found that increasing the grain size (higher austenitizing temperature) the reaction rate was not affected: Davenport, et al.;⁴⁶ the reaction rate was increased: Cottrell and Ko⁴⁷ and Graham and Axon⁴⁹ and those who found the reaction is slower. Barford and Owen⁴⁸ and Prakash Babu.¹⁷

It is difficult to separate the effect of the increased austenite grain size and the increased chemical homogeneity within the austenite grains.

These results from Fig. 26 also shows that the non-linearity of % transformed versus time is maintained for any austenitizing temperature and that the 1250°C treatment renders this effect more pronounced. Other investigators have found that also

At 950 it is possible that for 15 min the specimen will not be fully austenitic. So some sites for preferred inhomogeneous nucleation might be present at this temperature, (inhomogeneities).

Also the grain size is smaller hence there are more grain boundary surfaces contributing to the nucleation of bainite therefore, it should be a faster reaction. At 1200°C for instance the grain size is larger so the available grain boundary surface for nucleation is reduced which is consistent with a slower reaction.

Results suggested are in agreement with the fact that upper bainite nucleates at the grain boundaries but lower bainite can also nucleate within the grain therefore the effect should be less for the latter.

3. Determination of Bs on the Basis of Some Existing Empirical Formulae

From observation on results: combined additions of alloying elements to Fe-C-X system gave an additive effect in lowering the bainite temperature ranges. It showed that the C content played also a major role.

Two equations were studied (based on additive effects) to determine the Bs

Steven and Haynes¹⁷

$$Bs \text{ } ^\circ C = 830 - 270 - 90Mn - 37Ni - 70Cr - 83Mo \quad (10)$$

Prakash Babu¹⁷

$$Bs \text{ } ^\circ C = 839 - 300C - 60Mn - 33Ni - 63Cr - 130Mo - 45Si \quad (11)$$

Results are given in Table XXXII an average of these two will be made and marked as a calculated (not an empirical) Bs on the reaction start diagrams Fig. 11-23.

At temperatures near the Bs the austenite does not completely transform and part of the retained austenite⁹ transforms to martensite during cooling to lower temperature that

Table XXXII. B_s on the basis of empirical formulae.

Alloy	Steven and Haynes B_s ($^{\circ}\text{C}$)	Prakash Babu B_s ($^{\circ}\text{C}$)
4	729	741
5	707	724
6	685	708
7	702	692
8	718	714
10	659	681
11	725	714
12	660	645
13	701	653
14	643	656
16	640	646
17	615	569
20	569	495

was the case for instance with 17 at isothermal hold of 650°C and 5 at 600°C.

4. Comments on Optical Microscopy

- i. In the case of Fe-C-Ni Fig. 50 shows a structure that tries to be pearlite and it is not, it is a very degenerate form of pearlite at 660°. Followed by Fig. 51 a more degenerate structure that tries to be bainite at 512°C just below at 489°C we can see a definite upper bainite structure. Fig. 52, Fig. 53 (450°C) there is a mixture of upper and lower bainite structure followed by Fig. 54 where lower bainite predominates.
- ii. In the case of Fe-C-Mo at 610 (Fig. 55) again a very degenerate type of structure that tries to be pearlite forms at lower temperatures 512°C Fig 56. Upper bainite forms and at 435°C Fig. 57. A mixture of upper and lower bainite is formed in the corresponding microstructure.
- iii. In the case of Fe-C-Cr at 600°C proeutectoid ferrite and the corresponding expected eutectic matrix shows Fig. 58, at 555°C again a degenerate type of pearlite appears characteristic of some Cr-steels. At 490°C and 440°C one can see upper bainite structures Fig. 60 and 61. Below it was observed a mixture of upper and lower bainite structures.
- iv. In the case of Fe-C-Mn at 610°C proeutectoid ferrite and the corresponding expected eutectic matrix shows Fig. 52 at 560°C partially transformed upper bainite appears (Fig. 63). At 400°C a mixture of upper and lower bainite with upper bainite predominating Fig. 64 and vs occurs at 350°C in Fig. 65. One can see that although the temperature ranges of the bainites are not the same for the different ternary systems. The

optical features of the bainites are similar. To be able to see the differences in morphology carbide distribution, alloy segregation (if any) one should go to higher resolutions as discussed before.

It should be pointed out though that at lower magnifications it was observed that upper bainite nucleates at the grain boundaries but lower bainite does it also within the grains which is consistent with very many other investigations.

There was no clear cut temperature of transition for upper and lower bainite optical microscopy showed a range at which lower and upper bainite temperature were both present.

V. CONCLUSIONS

Based on the various results of this investigation, the following conclusions are drawn:

1. Comparative results showed that Cr and Mo restricted the austenite stable region (Mo being most effective) and that Mn expanded the austenite stable range slightly while Ni exerted little influence. Their interaction effect was not additive or average on the transformation temperatures Ae_1 , Ae_3 .

2. Results of the M_s transformation temperatures showed, i. that the quenching rate differences within the available range had no effect on the M_s . ii. That the higher the austenitizing temperature the higher the M_s from 950°C-1200°C there was a corresponding increase of 20°C on the M_s . iii. Comparison of experimental M_s values and existing empirical formulae indicated that for these low C low alloy steels. Nehremberg is linear, Eq. (8), gave the best fit.

$$M_s \text{ } ^\circ\text{F} = 1000 - 650C - 70Mn - 35Ni - 70Cr - 50Mo$$

iv. A linear function approximation seemed to fit the microhardness versus M_s transformation temperature results.

$$VHN (1000 \text{ gr}) = 2.11 \left[\frac{VHN}{^\circ\text{C}} \right] \times M_s (^\circ\text{C}) + 1306.4 [VHN]$$

3. Hardness values for the different bainite structures decreased with increasing temperature of transformation, results indicated non-linearity for the four alloys studied.

4. In the present studies there was an acceleration of the austenite decomposition at isothermal temperature treatments just above the M_s giving as result for the reaction start curves which have S-shape

rather than a C-shaped curve.

5. The present results showed that all of the alloying elements studied Mn, Ni, Cr (in order of effectiveness) except Mo shifted the bainite curves to longer times increasing the incubation period and lowering the kinetic bainite start. Different alloying elements affected this differently.

5. Mo instead accelerated the bainite reaction.

6. All the alloying elements lowered (the temperature of shortest incubation time in the nose) which implies a lowering of B_s . Further addition suggested an additive effect even for mixed additions as Cr+Ni, Mo+Cr, Mo+Ni.

7. Combined additions however gave no additive but multiplicative effects on the reaction start times for the isothermal decomposition of austenite.

i. Combined additions of Cr-Ni was at least 6 times more effective than the additions of Ni and Cr alone implying a strong interaction for the hardenability of bainite.

ii. Mo-Ni and Mo-Cr combinations gave similar results showing that despite the action of Mo when added alone it interacts with Ni, Cr increasing the hardenability of bainite.

iii. Increasing the Mn content when Mn alone is present also gave multiplicative results on the bainite hardenability.

8. The effects of austenitizing temperature on the kinetics of the transformation showed that the lower the austenitizing temperature the faster the bainite reaction (this effect being slightly stronger for upper bainite).

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APPENDIX

Hardness versus Ms transformation temperature equation.

In order to derive the linear equation given in Section IV-C

$$\text{VHN (1000 gr load)} = \frac{2.11}{^{\circ}\text{C}} \text{Ms } ^{\circ}\text{C} + 1306.4$$

A linear regression program (using the least square method of the hardness (y) versus Ms (x) temperature data) was used

$$\text{VHN} = \alpha \text{Ms} + \beta$$

$$y = \alpha x + \beta$$

$$\alpha = \frac{\sum y \sum x^2 - \sum x \sum y}{n \sum x^2 - (\sum x)^2}$$

$$\beta = \frac{n \sum y x - \sum x \sum y}{n \sum x^2 - (\sum x)^2}$$

$$\text{With coefficient of determination } r^2 = \frac{n \sum xy - \sum x \sum y}{n(n-1) S_x S_y}$$

where $S_x S_y$ are the standard deviation of x and y given by

$$S_x = \sqrt{\frac{\sum x^2 - (\sum x)^2/n}{n-1}}$$

$$S_y = \sqrt{\frac{\sum y^2 - (\sum y)^2/n}{n-1}}$$

For data given in Table IX

$$\alpha = 2.117/^{\circ}\text{C}$$

$$\beta = 1306.38$$

$$r^2 = .93$$

Where a perfect fit would imply $r^2 = 1$.

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FIGURE CAPTIONS

Fig. 1. Dimension specifications of the dilatometer specimens.

Fig. 2. Specimen mounting conditions.

Fig. 3. Dilatometer III-R measuring module chamber.

- A. Micrometer adjusting barrel.
- B. Location of calibration shim placement.
- C. Locking screw measuring module travel.
- E. Set screws holding glass rod which centers measuring rods and determines the specimen gap between the rods.
- F. Induction coil furnace where the specimen is placed for heating treatments.
- G. Thermocouple attached to specimen (inside the furnace).
- H. Hose that connects the quenching valve to the specimen through which gaseous helium flows for fast quenches.

Fig. 4. Dilatometer III-R. Vacuum System.

Fig. 5. Dilatometer III-R. Pumping Station.

- A. On-off switch, mechanical pump.
- B. On-off switch, diffusion pump.
- C. Switch controlling soft vacuum Gage E
(To left - vacuum at fore pump
to right - vacuum in specimen chamber).
- D. Switch controlling ion Gage F.
- E. Soft vacuum gage.
- F. Hand Vacuum ion gage.
- G. Flow valve and meter for quenching gas.

- Fig. 6. Typical run for Ms determination . (fast quench).
- Fig. 7. Typical run for the bainite transformation (isothermal run)
- Fig. 8. Effect of austenitizing temperature on Ms
A. Alloy 9, B. alloy 15.
- Fig. 9. Microhardness versus carbon content in martensitic structures.
Comparison with Bain and Paxton's plain carbon steels and 15 kg load.
- Fig. 10. Microhardness versus martensite transformation temperature.
- Fig. 11-23. Diagrams for starting time (curve) of the austenite to bainite transformation for the various alloys studied.
(4, 5, 6, 7, 8, 10, 11, 12, 13, 14, 16, 17, 20)
- Fig. 24. Effect of quenching rate on the bainite kinetics.
- Fig. 25. Effect of slight variations in quenching rate on reproducibility.
- Fig. 26. Effect of austenitizing temperature on the bainite kinetics for alloys 9 and 15.
- Fig. 27. Reaction times for progressive stages of the transformation versus the austenitizing temperature.
- Fig. 28-29. Microhardness versus bainite transformation temperature for 4 alloys (5, 8, 19, 17).
- Fig. 30. Effect of austenitizing temperature on grain size. d-diameter of an equivalent spherically shaped grain.
- Fig. 31-49. Show the martensitic structures of 19 of the alloys they are respectively 1, 2, 3* (discussed as not really martensitic) 4, 5, 6, 7, 8, 10, 11, 12, 13, 14, 16, 17 and 20.

- Fig. 50. Alloy 8 at 660°C isothermal hold. Degenerate pearlite.
- Fig. 51. Alloy 8 at 512°C. Degenerate structure.
- Fig. 52. Alloy 8 at 489°C. Upper bainite.
- Fig. 53. Alloy 8 at 460°C. Upper-lower bainite.
- Fig. 54. Alloy 8 at 400°C. Lower bainite.
- Fig. 55. Alloy 5 at 610°C. Degenerate pearlite.
- Fig. 56. Alloy 5 at 512°C. Upper bainite.
- Fig. 57. Alloy 5 at 435°C. Upper-lower bainite.
- Fig. 58. Alloy 10 at 600°C. Preutectoid ferrite and eutectic matrix.
- Fig. 59. Alloy 10 at 555°C. Degenerate pearlite.
- Fig. 60. Alloy 10 at 460°C. Upper-lower bainite.
- Fig. 61. Alloy 10 at 460°C. Upper-lower bainite.
- Fig. 62. Alloy 17 at 610°C. Preutectoid ferrite and eutectic matrix.
- Fig. 63. Alloy 17 at 560°C. Partly transformed upper bainite.
- Fig. 64. Alloy 17 at 400°C. Upper-lower bainite.
- Fig. 65. Alloy 17 at 350°C. Lower-upper bainite.
- Fig. 66. Effect of quenching rate on M_s after Ansell.³¹ Showing the quenching rate working range.
- Fig. 67. Upper-lower bainite range versus carbon content. Pickering.⁴⁵

XBL 75 12-9273

Fig. 1.

NOT TO SCALE

XBL 7512-9274

Fig. 2.

XBB 7512-2569R

Fig. 3.

XBL7512-9275

Fig. 4.

Y11. 53-2368A

Fig. 5.

Fig. 6

Fig. 7

XBL 7512-9278

Fig. 8.

Fig. 9.

XBL 7512-9288

Fig. 10.

XBL 7512-9289

Fig. 11.

Fig. 12.

XBL 7512-9291

Fig. 13.

XBL 7512-9292

Fig. 14.

XBL 7512-9293

Fig. 15.

XBL 7512-9294

Fig. 16.

XBL 7512-9295

Fig. 17.

XBL 7512-9296

Fig. 18.

XBL 7512-9297

Fig. 19.

XBL 7512-9298

Fig. 20.

XBL 7512-9299

Fig. 21.

XBL 7512-10,001

Fig. 22.

XBL 7512-10,002

Fig. 23.

Fig. 24.

XBL7512-9282

FIG. 25.

XBL 7512-9283

Fig. 27.

XBL 7512-9280

Fig. 28.

Fig. 29.

XBL 7512-9279

Fig. 30.

Fig. 31.

Fig. 32.

Fig. 33.

ZBB 7712-9086

Fig. 34.

20μ The same magnification scale
for all the microstructures shown.

Fig. 35.

Fig. 36.

Fig. 37.

XBB 7512 9085

Fig. 38.

Fig. 39.

Fig. 40.

Fig. 41.

XBB 7537-9064

Fig. 42.

Fig. 43.

Fig. 44.

Fig. 45.

XBB 7512-9083

Fig. 46.

Fig. 47.

Fig. 48.

XBB 7512-9087

Fig. 49.

Fig. 50.

Fig. 51.

Fig. 52.

XBB 7512-9080

Fig. 53.

Fig. 54.

Fig. 55.

Fig. 56.

XBB 7512-9079

Fig. 57.

Fig. 58.

Fig. 59.

Fig. 60.

XBB 7512-9082

Fig. 61.

Fig. 62.

Fig. 63.

Fig. 64.

XBB 7512-9081

Fig. 65.

- 121 -

XBL 7512-9285

Fig. 60.

XBL7512-9286

Fig. 11.