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STUDY OF THE FORMATION AND EFFECTS OF SIGMA PHASE IN 21-6-9 STAINLESS STEEL

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CONTENTS

Abstract 1

Introduction 1

Investigation of Cracking in Forgings 1

Formation of Sigma Phase 4

Effect of Sigma Phase on Mechanical Properties 12

Tensile Testing 13

Charpy Impact Testing 13

Impact Shear Testing 17

Vendor Contacts 17

Discussion 19

Conclusions 20

Future Work 20

References 21

Appendix (Review of Literature) 22

Description of Sigma 22

Predicting Sigma Formation 22

Formation of Sigma 22

Effect of Sigma on Properties 26

Effect of Delta Ferrite on Properties 28

Effect of Delta Ferrite on Welds 28

Identification and Measurement of Sigma and Delta Ferrite 29

Appendix References 30

TABLES

Table 1 - Chemical Analyses and Delta Ferrite Contents of Samples Used to Determine Nose of Transformation Curve 5

Table 2 - Microprobe Analyses of Phases Present in Sample No. 105457 6

Table 3 - Chemical Analyses of Plates Mechanically Tested 7

Table 4 - Summary of Mechanical Tests Performed 12

Table 5 - Room Temperature Tensile Testing Results 13

Table 6 - Room Temperature Charpy V-Notch Impact Results 16

Table 7 - Room Temperature Impact Shear Results 17

FIGURES

Figure 1 - Crack Observed in Forging in Area of Parting Line 2

Figure 2 - Orientation of Stringers at Parting Line in G. O. Carlson Forging 2

Figure 3 - Orientation of Stringers 90° From Parting Line in G. O. Carlson Forging 3

Figure 4 - Ferrite Distribution at the Parting Line in Simonds Forging 3

Figure 5 - Color Etching of Stringers in Carlson Forging 4

Figure 6 - Sigma Formation in Fringes of Ferrite Stringer in As-Rolled Plate Heat Treated at 1500 °F for 30 Minutes 5

Figure 7 - SEM Photograph of Stringer Found in Sample Shown in Figure 6 5

Figure 8 - Sigma Formation in As-Rolled Plate Sample, Heat Treated at 1400 °F for 30 Minutes 6

Figure 9 - Sigma Formation in As-Rolled Plate Sample, Heat Treated at 1600 °F for 30 Minutes 6

Figure 10 - Ferrite Distribution in As-Rolled Plate 8

Figure 11 - Morphology of Ferrite Phase 9

Figure 12 - Transformation of Delta Ferrite to Sigma in High Ferrite As-Rolled Plate Heat Treated at 1500 °F 10

FIGURES (Cont'd)

Figure 13 - Transformation of Delta Ferrite to Sigma in Low Ferrite As-Rolled Plate Heat Treated at 1500 °F 11

Figure 14 - Longitudinal Tensile Properties Vs. Time at 1500 °F 14

Figure 15 - Transverse Tensile Properties Vs. Time at 1500 °F 15

Figure 16 - Charpy V-Notch Impact Energy Vs. Time at 1500 °F 16

Figure 17 - Impact Shear Values Vs. Time at 1500 °F 18

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STUDY OF THE FORMATION AND EFFECTS OF SIGMA PHASE IN 21-6-9 STAINLESS STEEL

Carol L. Packard, Martin C. Mattaya, and Carl M. Edstrom

ABSTRACT

Work performed to date on the study of the formation and effects of sigma phase in 21-6-9 stainless steel is summarized in this report. Sigma phase was identified in forgings and as-rolled plate by color etching and microprobe analysis. In as-rolled plate sigma was found to start transforming from delta ferrite within 30 minutes at 1500 °F, with almost complete transformation after 24 hours at 1500 °F.

The effect of sigma phase on room temperature mechanical properties was evaluated by tensile testing, Charpy impact testing, and impact shear testing. Sigma phase was found to severely reduce transverse ductility and longtudinal and transverse impact resistance. The greater the amount of sigma present, the greater was its effect on mechanical properties.

Vendor contacts indicated that controlling delta ferrite to a minimum in the as-rolled plate is both possible and practical.

INTRODUCTION

Work performed to date on the study of the formation and effects of sigma phase in 21-6-9 stainless steel is presented in this report. The following subjects are included:

1. Investigation of Cracking in Forgings. Recent investigation of cracking in forgings attributed the cause of cracking to the presence of sigma phase in the forgings. A brief description of the testing of the forgings is given.

2. Formation of Sigma Phase. As-rolled samples of low and high ferrite plate were heat treated to observe the formation of sigma from delta ferrite.

3. Effect of Sigma Phase on Mechanical Properties. The effect of sigma on room temperature tensile properties, Charpy impact energy, and impact shear energy was examined. Longitudinal and transverse specimens were tested.

4. Vendor Contacts. Vendors were contacted to obtain information on 21-6-9 plate quality and control of delta ferrite.

A review of the published literature on sigma phase, with particular emphasis on literature concerning the transformation of delta ferrite to sigma, is included as an Appendix.

INVESTIGATION OF CRACKING IN FORGINGS

Recent investigation of cracking in forgings attributed the cause of cracking to the presence of sigma phase in the forgings. Cracks occurred at the parting line of the forging

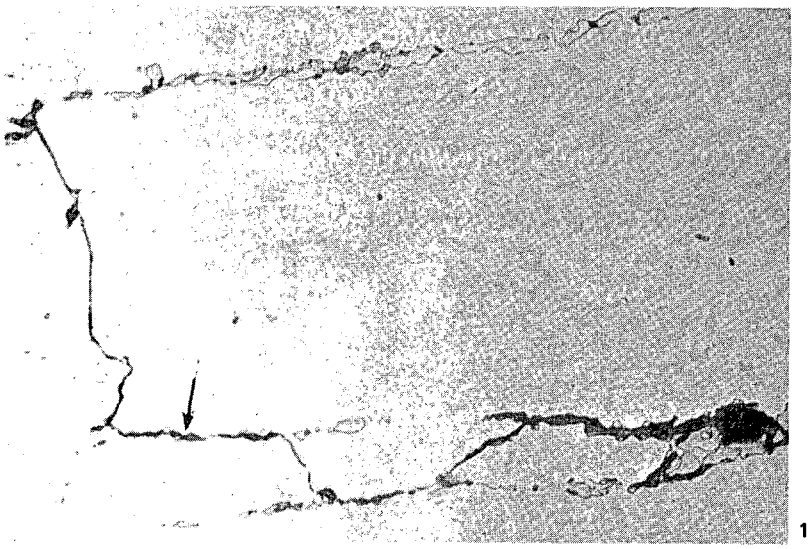

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FIGURE 1. Crack Observed in Forging in Area of Parting Line. Murakami's Etch

100X

FIGURE 2. Orientation of Stringers at Parting Line in G. O. Carlson Forging. Murakami's Etch

through brittle sigma phase stringers which had formed during forging from the delta ferrite phase present in the as-rolled plate stock. Figure 1 shows a crack observed in one of the forgings.

Macroetching showed that the flow lines were nonuniform around the circumference of the part, turning radial (transverse orientation in finished part wall) at the parting line and circumferential approximately 90° from the parting line. The stringers paralleled the flow lines. Figures 2 and 3 show the orientation of the stringers in a forging made from G. O. Carlson plate stock at the parting line and 90° away, respectively.

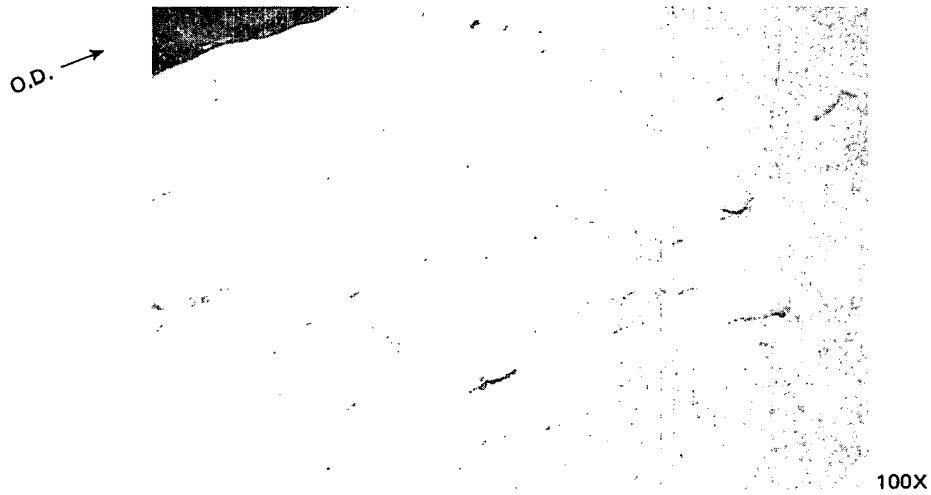

FIGURE 3. Orientation of Stringers 90° From Parting Line in G. O. Carlson Forging. Murakami's Etch

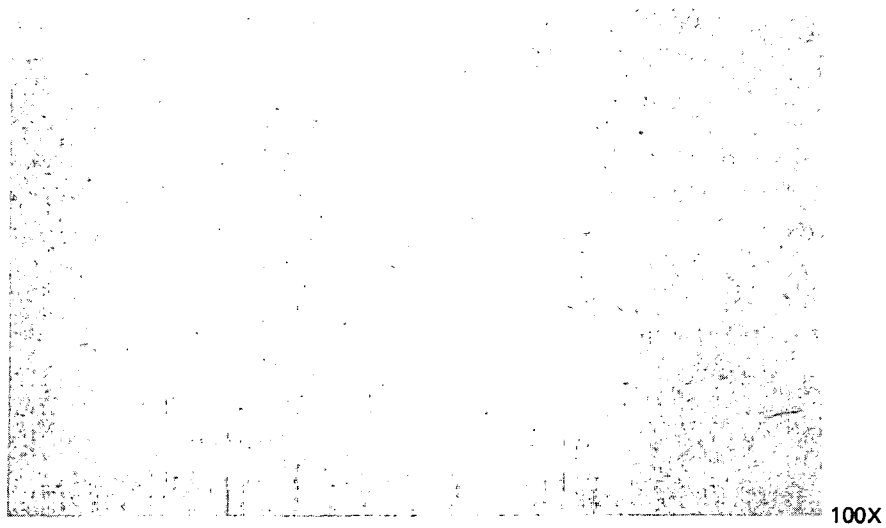

FIGURE 4. Ferrite Distribution at the Parting Line in Simonds Forging. Murakami's Etch

Plate stock and forgings produced from material supplied by two different vendors, G. O. Carlson and Simonds, were examined. A significant difference was noted in the amount of delta ferrite present in the plate stock and in the amount of sigma present in the forgings. The Carlson plate had up to 3.4% delta ferrite, whereas the Simonds plate had <0.6% delta ferrite (see Figures 2-4). Correspondingly, the Carlson forgings had a much greater amount of sigma phase than the Simonds forgings. The Simonds forgings did not exhibit cracking, while cracking was observed in approximately 35 of 129 Carlson forgings.

In work performed by Physical Metallurgy, [1] the composition of the stringers was qualitatively analyzed in the scanning electron microscope using

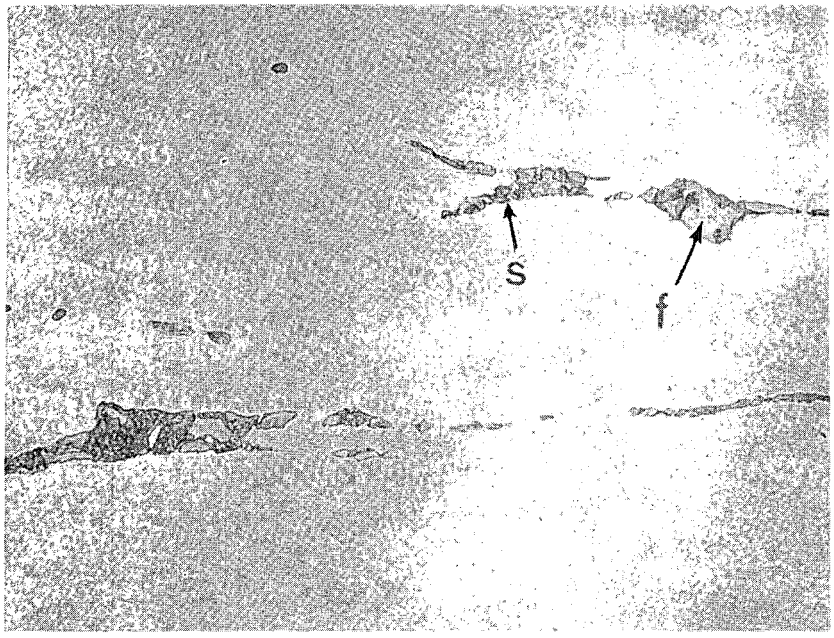

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FIGURE 5. Color Etching of Stringers in Carlson Forging, (f = Ferrite, s = Sigma). Murakami's Etch

X-ray energy dispersive analysis. Quantitative analyses of the stringers and matrix compositions were made with the electron microprobe. Transmission electron microscopy and selected area electron diffraction were used to positively identify sigma by its crystal structure.

The extent of the presence of sigma phase in five forgings was determined using color etching to differentially stain the ferrite and sigma phases. Two different color etches were used to distinguish the sigma phase from delta ferrite in the forgings and in work subsequently described in this report. Boiling Murakami's reagent [10 g $K_3Fe(CN)_6$, 10 g KOH, 100 ml H_2O] in 3 minutes stains the ferrite yellow, sigma blue, and carbides dark. Figure 5 shows stringers in a Carlson forging which were etched with Murakami's reagent. A second etch, modified Murakami's [30 g $K_3Fe(CN)_6$, 30 g KOH, 150 ml H_2O–90 to 95 °C for ~15 sec] stains the matrix austenite a light tan, the ferrite a medium tan, and the sigma phase a reddish brown color. Photographs of samples etched with modified Murakami's reagent are included later in this report. Microprobe analysis was used to establish a positive relationship between the phases and colors. Black and white photographs are presented in this report and the various phases appear as different shades.

FORMATION OF SIGMA PHASE

To observe the formation of sigma, as-rolled samples of plate with varying delta ferrite contents were examined before and after heat treating to transform the delta ferrite to sigma. The transformation of delta ferrite to sigma was determined using a combination of color etching techniques, microprobe and SEM analysis, and magnetic measurements obtained using the Severn indicator.

To determine the approximate temperature of the nose of the ferrite to sigma transformation curve (that temperature at which the transformation from ferrite to sigma is most rapid), samples of high delta ferrite (Carlson) plate (see Table 1) were heat treated at temperatures ranging from 1200 to 1900 °F for 30 minutes and then water quenched. The maximum amount of sigma phase was observed to form in the samples heat treated at 1500 °F.

TABLE 1. Chemical Analyses and Delta Ferrite Contents of Samples Used to Determine Nose of Transformation Curve

Heat No.	Material Control No.	C	S	N	O (ppm)	Cr	Ni	Mn	Si	P	Al	% Delta Ferrite
96773	105457	0.030	0.0015	0.28	<10	20.51	6.36	8.72	0.32	0.011	0.024	<0.6 - <3.0
96773	105459	0.029	0.0028	0.28	<10	21.31	6.69	8.72	0.33	0.007	0.023	>1.5 - <3.0
96773	105461	0.028	0.0028	0.28	<10	19.37	6.36	8.72	0.34	0.008	0.009	>1.5 - <3.0
96773	105472	0.030	0.0013	0.29	<10	20.59	6.64	8.72	0.30	0.010	0.007	>0.6 - <2.5

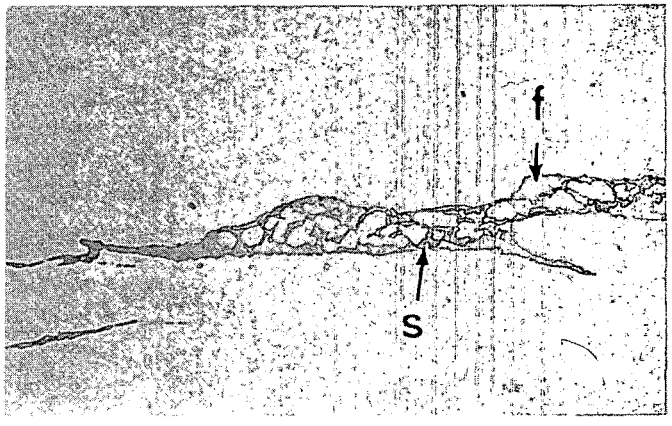

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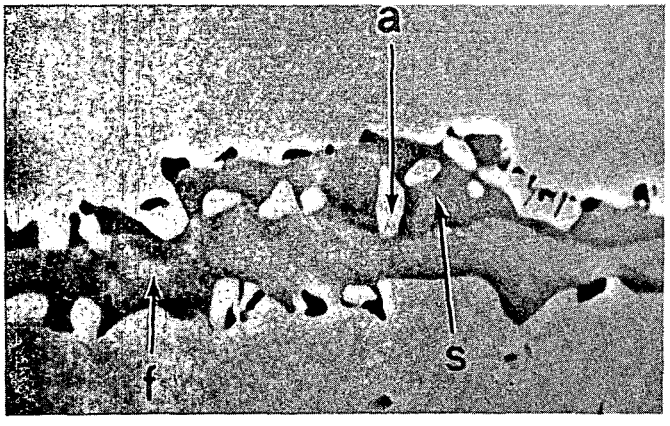
FIGURE 6. Sigma Formation in Fringes of Ferrite Stringer in As-Rolled Plate Heat Treated at 1500 °F for 30 minutes, (f = Ferrite, s = Sigma). Murakami's Etch



4000X

FIGURE 7. SEM Photograph of Stringer Found in Sample Shown in Figure 6, (a = Austenite, f = Ferrite, s = Sigma)

Lesser amounts of sigma phase were observed in samples heat treated at 1400 to 1600 °F for 30 minutes. This data indicates that the nose of the curve lies on the 1500 °F line.

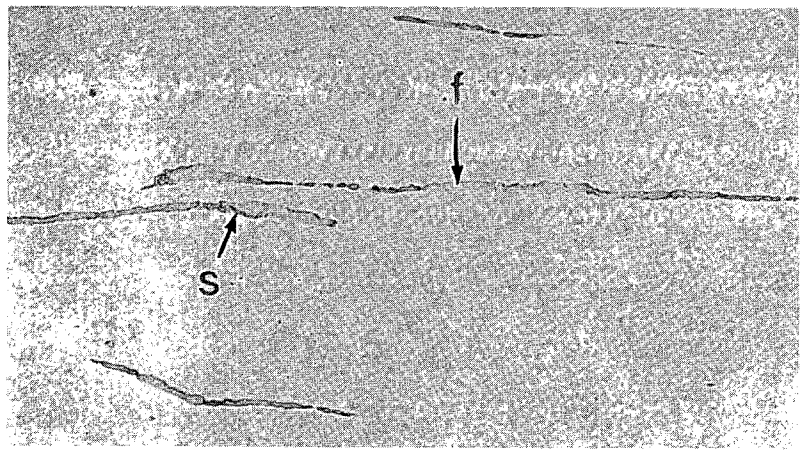
Figure 6 shows the formation of sigma phase in the fringes of the ferrite stringers in the plate heat treated at 1500 °F for 30 minutes. The sample was etched with Murakami's reagent which colored the ferrite yellow and the sigma blue. In the black and white photograph the sigma appears darker than the ferrite. Figure 7 is an SEM photograph of one of the stringers present in this sample. Three phases were found in the stringer-delta ferrite, sigma, and austenite particles of a composition different than the matrix. In the photograph the sigma is the dark phase at the top edge of the stringer. The black holes present indicate that a fourth phase may have been etched out, possibly carbides. The compositions of the sigma, delta ferrite, and austenite phases in the heat-treated sample and the composition of ferrite in an adjacent as-rolled sample were determined by microprobe analysis. The average compositions of the different phases are given in Table 2. The Cr/Fe ratios of the matrix, delta ferrite, and sigma phase

TABLE 2. Microprobe Analyses of Phases Present in Sample No. 105457

Sample Condition	Phase	Composition (%) Fe	Cr	Ni	Mn	Si
As Rolled	Delta Ferrite	64.1	25.6	3.1	6.8	0.39
Heat Treated 1500 °F 30 min.	Matrix	63.1	20.0	7.0	9.5	0.38
1500 °F 30 min.	Delta Ferrite	63.7	24.8	3.0	7.0	0.49
1500 °F 30 min.	Sigma	59.8	30.0	2.3	7.2	0.63
1500 °F 30 min.	Austenite in Sigma	67.4	20.6	4.0	7.4	0.57

were 0.32, 0.39, and 0.50 respectively, indicating the chromium enrichment of both the ferrite and sigma phases. Both the ferrite and sigma phases were lower in Ni than the matrix.

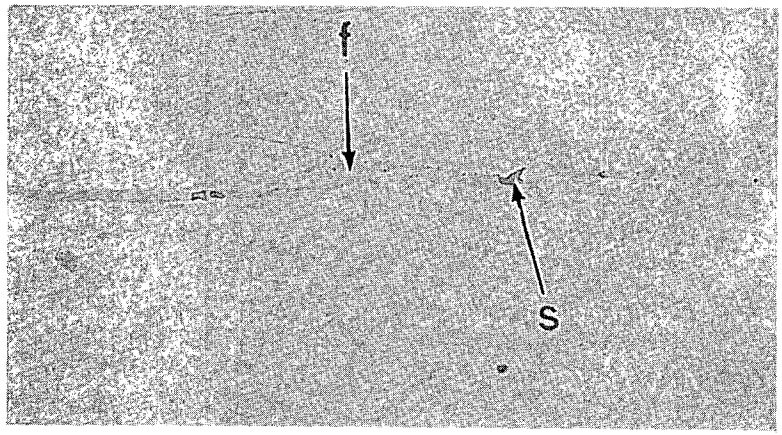
Figures 8 and 9 show the sigma phase which formed in the samples heat treated at 1400 and 1600 °F for 30 minutes. Compared with the 1500 °F treatment, a smaller amount of sigma was formed. In the black and white photographs the sigma phase appears darker than the ferrite and is seen to form within the ferrite stringers.



500X

FIGURE 8. Sigma Formation in As-Rolled Plate Sample, Heat Treated at 1400 °F for 30 minutes, (f = Ferrite, s = Sigma). Murakami's Etch

FIGURE 9. Sigma Formation in As-Rolled Plate Sample, Heat Treated at 1600 °F for 30 minutes, (f = Ferrite, s = Sigma). Murakami's Etch



500X

To further determine the kinetics of the ferrite to sigma transformation and the morphology of the ferrite and sigma phases, as-rolled plate samples with delta ferrite contents ranging from <0.6% to <3.8% were heat treated at 1500 °F for times ranging from 0-24 hours. Samples from three different heats with three different ferrite contents were examined in three different orientations. The chemical analyses of the samples are given in Table 3. Figure 10 illustrates the differences in ferrite content. Although Heat 7724 and Heat 38025 were both found to contain <0.6% ferrite by the Severn gauge, metallographically Heat 7724 was found to contain only a small amount of ferrite while Heat 38025 was found to contain 0.23-0.5% ferrite as measured by the ten-line counting technique. For the ten-line counting, ten parallel and equally spaced lines were drawn on a photograph and the percent ferrite was determined by dividing the total length of the ferrite intercepts by the total length of the lines.

To determine the three-dimensional orientation and morphology of the ferrite and sigma phases in the low and high ferrite material, microspecimens of the as-rolled and heat-treated plate were prepared. The ferrite and sigma phases were found to be platelets which lie parallel to the plate surface. When the two surfaces perpendicular to the rolled surface were polished, the ferrite and sigma appeared as stringers. Figure 11 shows the morphology of the ferrite. The sigma phase had a similar morphology.

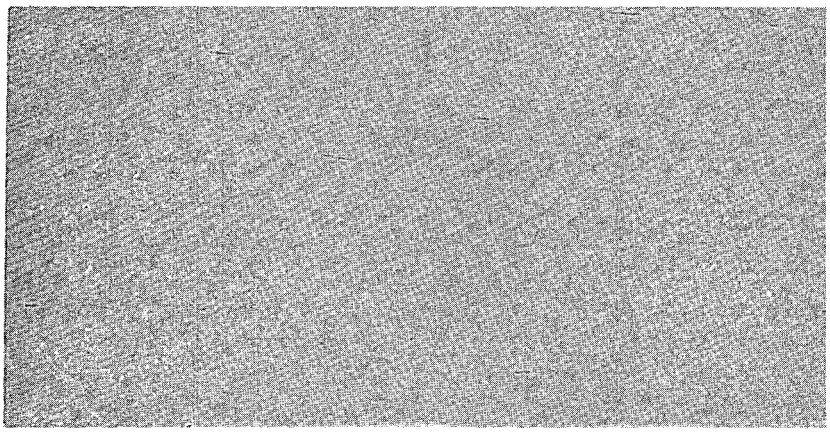
Figure 12 shows the increasing amount of delta ferrite to sigma transformation in the high ferrite plate with increasing time at 1500 °F. These samples were etched with modified Murakami's reagent, which stains the ferrite phase medium tan and the sigma reddish brown (instead of blue). After 30 minutes sigma is seen to start forming at the fringes of the ferrite. After 24 hours transformation to sigma is almost complete. Figure 12 also shows what appears to be carbide precipitation at the ferrite/austenite interphase boundary. Published transformation curves for 316L stainless steel show that the nose of the $M_{23}C_6$ precipitation curve lies to the left of the sigma transformation curve, indicating that carbide precipitation precedes sigma formation in that alloy.[2] The 21-6-9 alloy may behave similarly. Austenite-

TABLE 3. Chemical Analyses of Plates Mechanically Tested*

Heat No.	Material Control No.	C	S	N	O (ppm)	Cr	Ni	Mn	Si	P	Al	Vendor
7724	105488	0.019	<10	0.27	<10	19.2	6.5	9.7	0.36	0.016	0.005	Simonds
38025	106471	0.037	0.003	0.26	<10	19.1	6.4	8.2	0.50	0.013	0.0002	Simonds
38025	106484	0.034	0.002	0.25	<10	19.6	6.5	8.6	0.52	0.014	0.0002	
38025	106492	0.039	0.003	0.26	<10	19.4	6.3	8.2	0.42	0.014	0.0002	
96773	105458	0.029	0.0027	0.28	<10	20.24	6.90	8.72	0.37	0.019	0.027	Carlson
96773	105465	0.028	0.0024	0.28	<10	21.30	6.93	8.72	0.33	0.003	0.004	
96773	105466	0.028	0.0017	0.29	<10	20.84	6.54	8.72	0.31	0.004	0.007	
96773	105473	0.031	0.0009	0.28	<10	20.87	6.89	8.72	0.31	0.005	0.005	
96773	106741**	---	---	---	---	---	---	---	---	---	---	

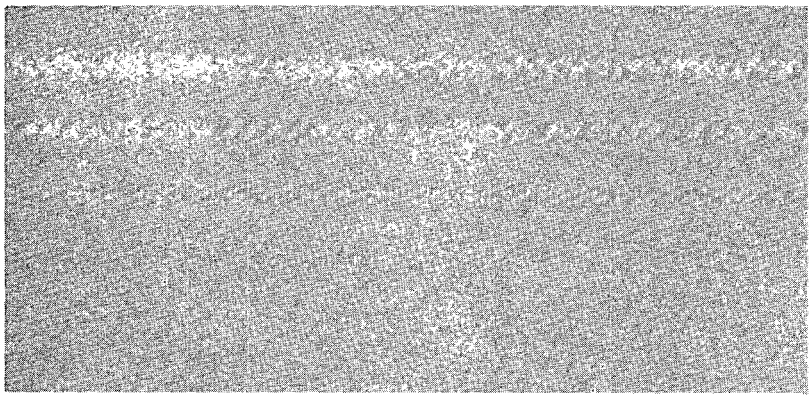
*Analyses in percent unless otherwise noted.
**No chemical analysis run at Rocky Flats.

FIGURE 10. Ferrite Distribution in As-Rolled Plate. Murakami's Etch



100X

(a) Sample MC# 105488 Heat 7724



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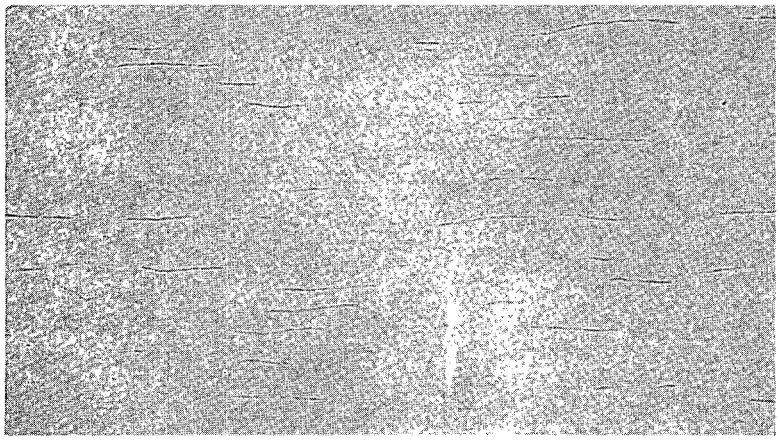
(b) Sample MC# 106492 Heat 38025



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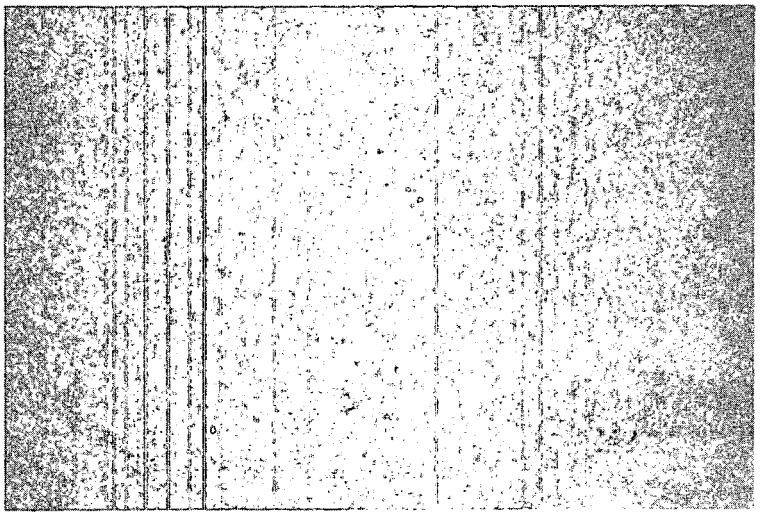
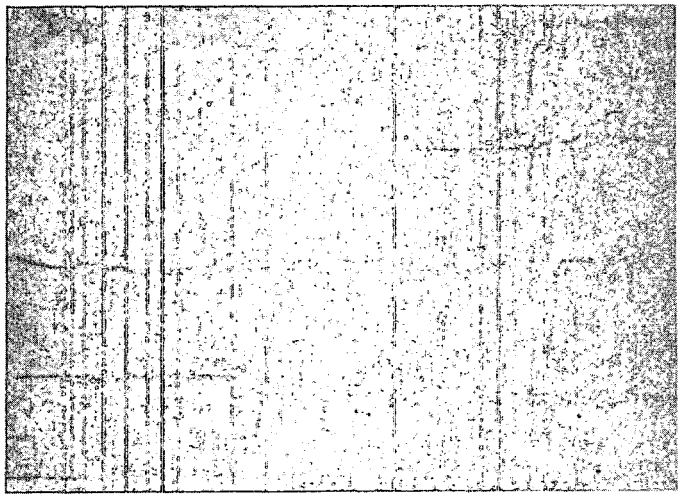
(c) Sample MC# 106741 Heat 96773

FIGURE 11. Morphology of Ferrite Phase. Modified Murakami's Etch



500X

(a) Parallel to Plate Surface



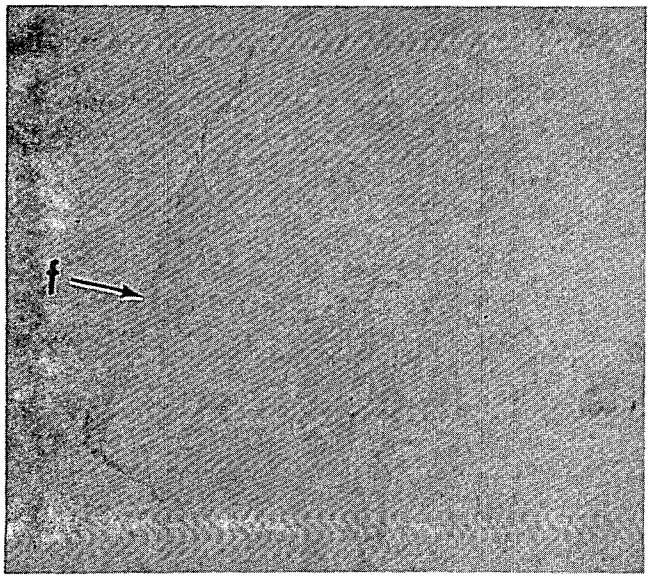
500X

(b) Perpendicular to Plate Surface

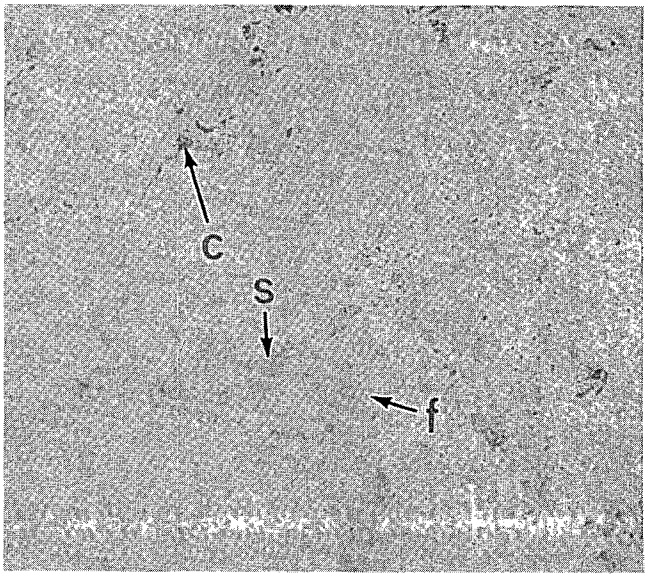
500X

(c) At 90° to Figure 11 (b)

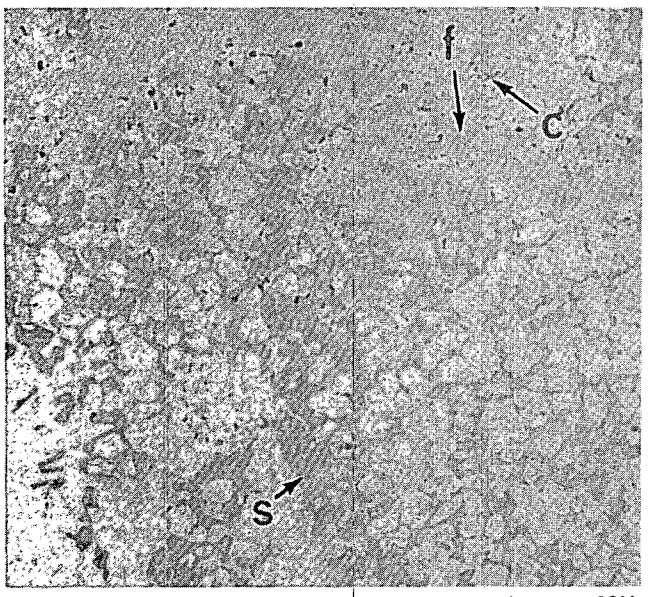
FIGURE 12. Transformation of Delta Ferrite to Sigma in High Ferrite As-Rolled Plate Heat Treated at 1500 °F. Modified Murakami's Etch



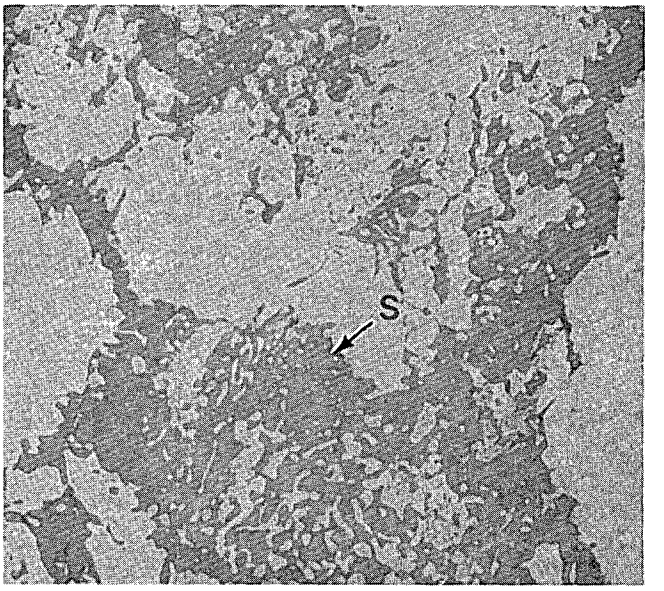
(a) As-Rolled (f = Ferrite) 500X



(b) 1500 °F, ½ hr (f = Ferrite, s = Sigma, c = Carbides) 500X



(c) 1500 °F, 5 hr (f = Ferrite, s = Sigma, c = Carbides) 500X

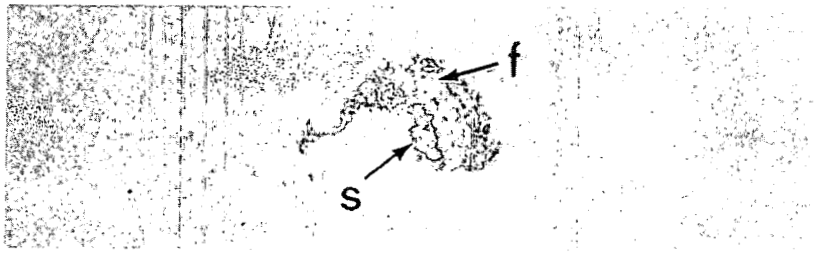


(d) 1500 °F, 24 hr (s = Sigma) 500X

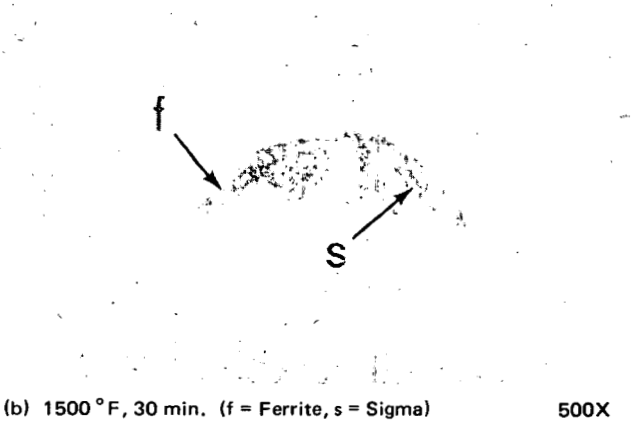
delta ferrite boundaries are a likely place for $M_{23}C_6$ nucleation since there is a concentration of chromium in the delta ferrite and high carbon in the austenite. The interphase boundary provides a preferred nucleation site while the adjoining matrices provide the Cr and C for the carbide while it grows.[3]

Figure 13 shows examples of transformation of delta ferrite to sigma in the low ferrite as-rolled plate from Heat 38025 which was heat treated at 1500 °F for 30-90 minutes. The sigma phase, stained blue in these photomicrographs, is seen to nucleate at the edges of the ferrite stringers. Although no samples were heat treated for times

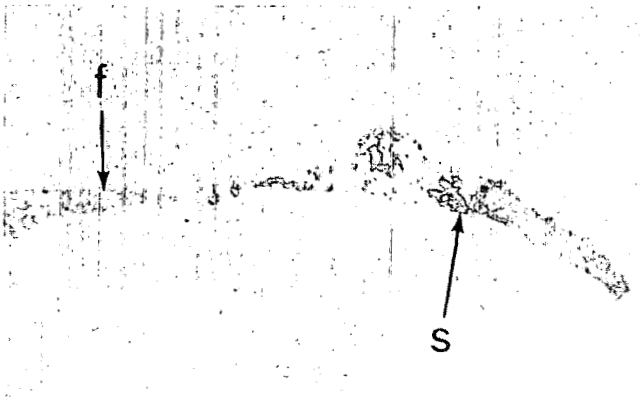
FIGURE 13. Transformation of Delta Ferrite to Sigma in Low Ferrite As-Rolled Plate Heat Treated at 1500 °F. Murakami's Etch



(a) 1500 °F, 30 min. (f = Ferrite, s = Sigma) 500X



(b) 1500 °F, 30 min. (f = Ferrite, s = Sigma) 500X



(c) 1500 °F, 90 min. (f = Ferrite, s = Sigma) 500X

longer than 90 minutes, it is expected that increasing amounts of sigma would form with increasingly longer heat treating times, as in the high ferrite plate shown in Figure 12.

EFFECT OF SIGMA PHASE ON MECHANICAL PROPERTIES

The effect of sigma on mechanical properties was evaluated by testing room temperature tensiles, Charpy V-notch impacts, and impact shear tests. As-rolled plate samples with delta ferrite contents ranging from <0.6% to <3.8% and heat treated at 1500 °F for 0-24 hours were mechanically tested (see Table 4). In Table 4 and the subsequent discussion, the term longitudinal refers to test specimens oriented parallel to the plate surface and transverse refers to test specimens oriented perpendicular to the plate surface. The percent ferrite was measured before and after heat treating using the Severn Indicator.

TABLE 4. Summary of Mechanical Tests Performed*

Heat No.	Material Control No.	Heat Treatment	Mechanical Tests Performed*
7724	105488	As-Rolled	L&T Tensiles L&T Charpy V-Notch Impacts L&T Impact Shear Tests
		1500 °F, 30 min.	L&T Tensiles L&T Charpy V-Notch Impacts L&T Impact Shear Tests
		1500 °F, 90 min.	L&T Tensiles L&T Charpy V-Notch Impacts L&T Impact Shear Tests
38025	106492	As-Rolled	L&T Charpy V-Notch Impacts
38025	106471	1500 °F, 30 min.	L&T Tensiles L&T Charpy V-Notch Impacts
38025	106484	1500 °F, 90 min.	L&T Tensiles L&T Charpy V-Notch Impacts
96773	106741	As-Rolled	L&T Tensiles L&T Charpy V-Notch Impacts
96773	105458	As-Rolled	L&T Impact Shear Tests
96773	106741	1500 °F, 30 min.	L&T Tensiles L&T Charpy V-Notch Impacts
96773	105466	1500 °F, 30 min.	L&T Impact Shear Tests
96773	106741	1500 °F, 90 min.	L&T Tensiles L&T Charpy V-Notch Impacts
96773	105465	1500 °F, 5 hr	L&T Impact Shear Tests
96773	105473	1500 °F, 24 hr	L&T Impact Shear Tests

*L = Longitudinal
T = Transverse

Tensile Testing

Room temperature tensile testing results are shown in Table 5. Figures 14 and 15 illustrate the variation in tensile properties with heat treating time at 1500 °F for the samples tested from Heats 7724 (MC# 105488) and 96773 (MC# 106741).

The major effect of heat treating and the formation of sigma on tensile properties was to reduce the ductility, as measured by percent elongation and percent reduction in area, in the transverse direction. For the low ferrite plate from Heat 7724 (MC# 105488), the transverse elongation decreased from 57 to 49% after heat treating at 1500 °F for 90 minutes. For the high ferrite plate from Heat 96773 (MC# 106741), the corresponding decrease was from 44 to 12%, indicating the more adverse effect of larger amounts of sigma. The reduction in area values show a similar trend. Another effect of the presence of sigma was a reduction in transverse tensile strength of the high ferrite plate.

Charpy Impact Testing

Results obtained from room temperature Charpy V-notch impacts are shown in Table 6. Figure 16

TABLE 5. Room Temperature Tensile Testing Results

Heat No.	Sample No.	Heat Treatment	Percent Delta Ferrite (Severn Indicator) Before Heat Treat	After Heat Treat	YS (ksi)	TS (ksi)	Elong* (%)	RA (%)
7724	105488	As-Rolled	<0.6	---	L 67.3	113.0	59	80.3
		1500 °F, 30 min.	<0.6	<0.6	L 71.5	112.8	59	80.0
		1500 °F, 90 min.	<0.6	<0.6	L 69.8	110.8	60	80.0
		As-Rolled	<0.6	---	T 67.0	112.4	57	74.3
		1500 °F, 30 min.	<0.6	<0.6	T 71.4	112.4	57	72.5
		1500 °F, 90 min.	<0.6	<0.6	T 67.5	109.7	49	50.7
38025	106471	1500 °F, 30 min.	<0.6	<0.6	L 70.4	114.6	64	78.5
38025	106484	1500 °F, 90 min.	<0.6	<0.6	L 71.0	113.4	59	78.0
38025	106471	1500 °F, 30 min.	<0.6	<0.6	T 70.2	112.2	54	66.1
38025	106484	1500 °F, 90 min.	<0.6	<0.6	T 70.3	111.8	54	62.2
96773	106741	As-Rolled	>0.6 - <1.4	---	L 76.9	114.6	60	81.4
		1500 °F, 30 min.	>0.6 - <1.4	<0.6 - <1.0	L ---	---	--	---
		1500 °F, 90 min.	>0.6 - <1.4	<0.6	L 80.0	114.4	57	75.5
		As-Rolled	>0.6 - <1.4	---	T 77.2	115.6	44	61.4
		1500 °F, 30 min.	>0.6 - <1.4	<0.6 - <1.0	T 77.2	103.6	22	35.9
		1500 °F, 90 min.	>0.6 - <1.4	<0.6	T 78.0	90.2	12	17.8

*0.3-in. gage length (measured on broken specimens)

FIGURE 14. Longitudinal Tensile Properties Vs. Time at 1500 °F

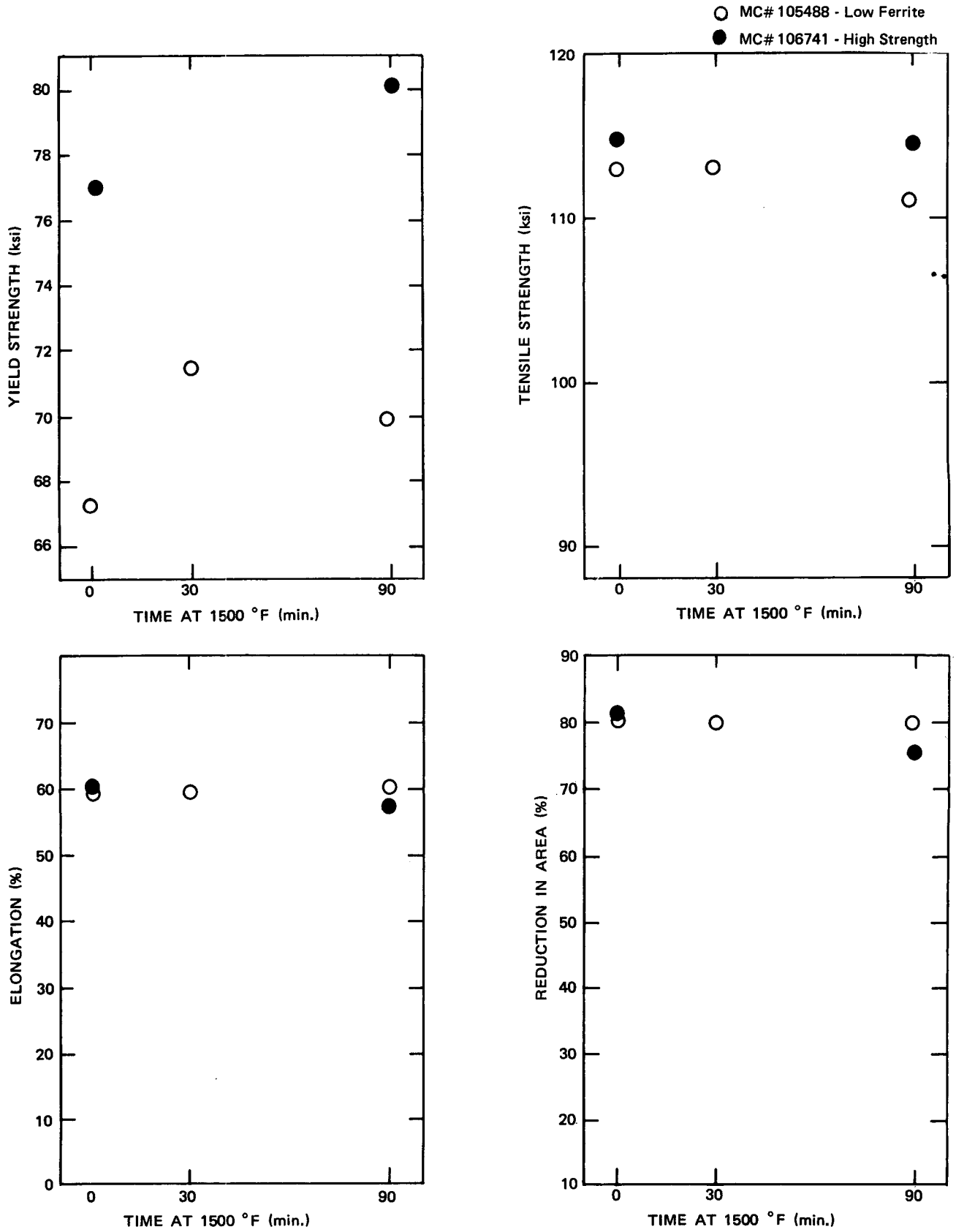


FIGURE 15. Transverse Tensile Properties Vs. Time at 1500 °F

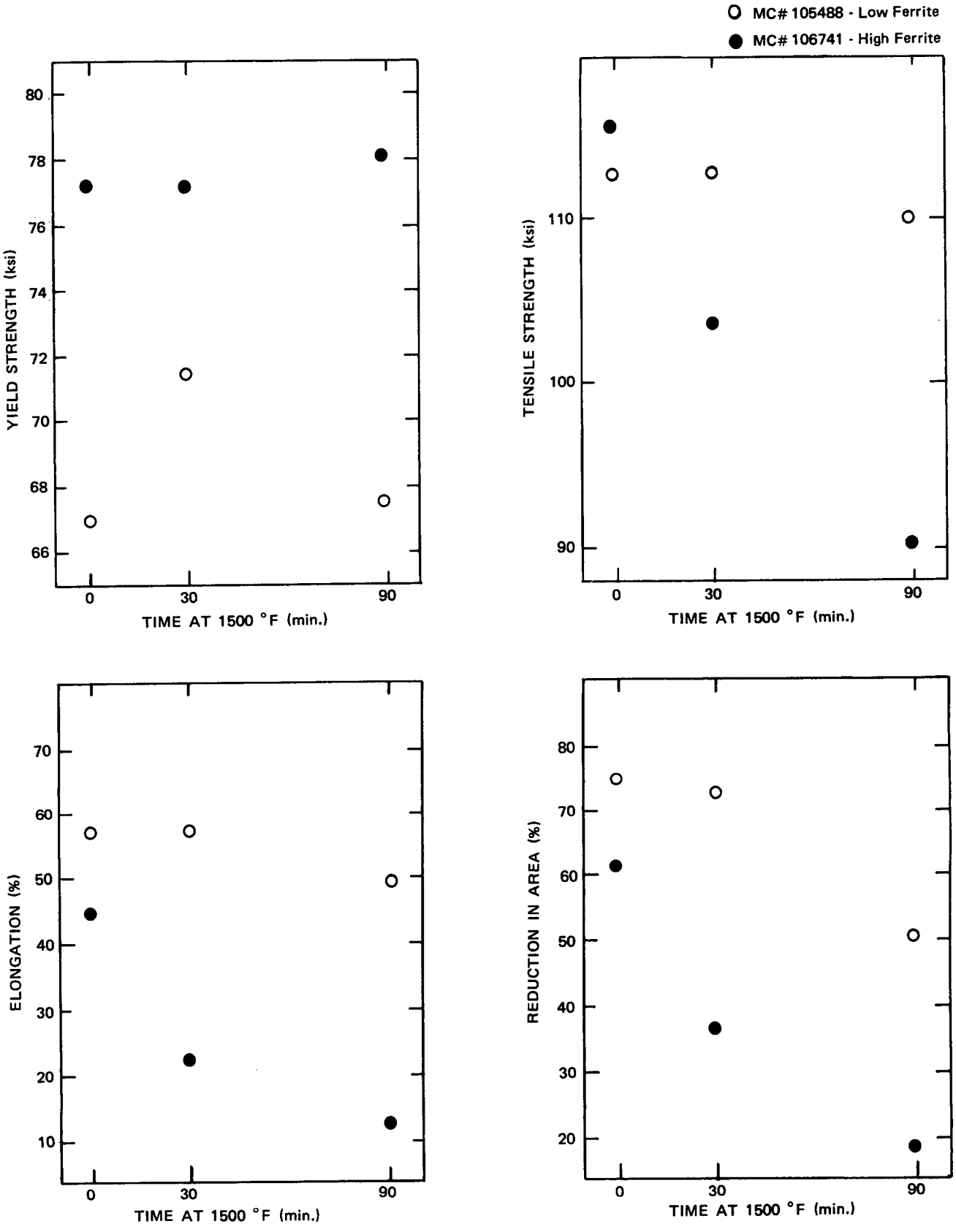
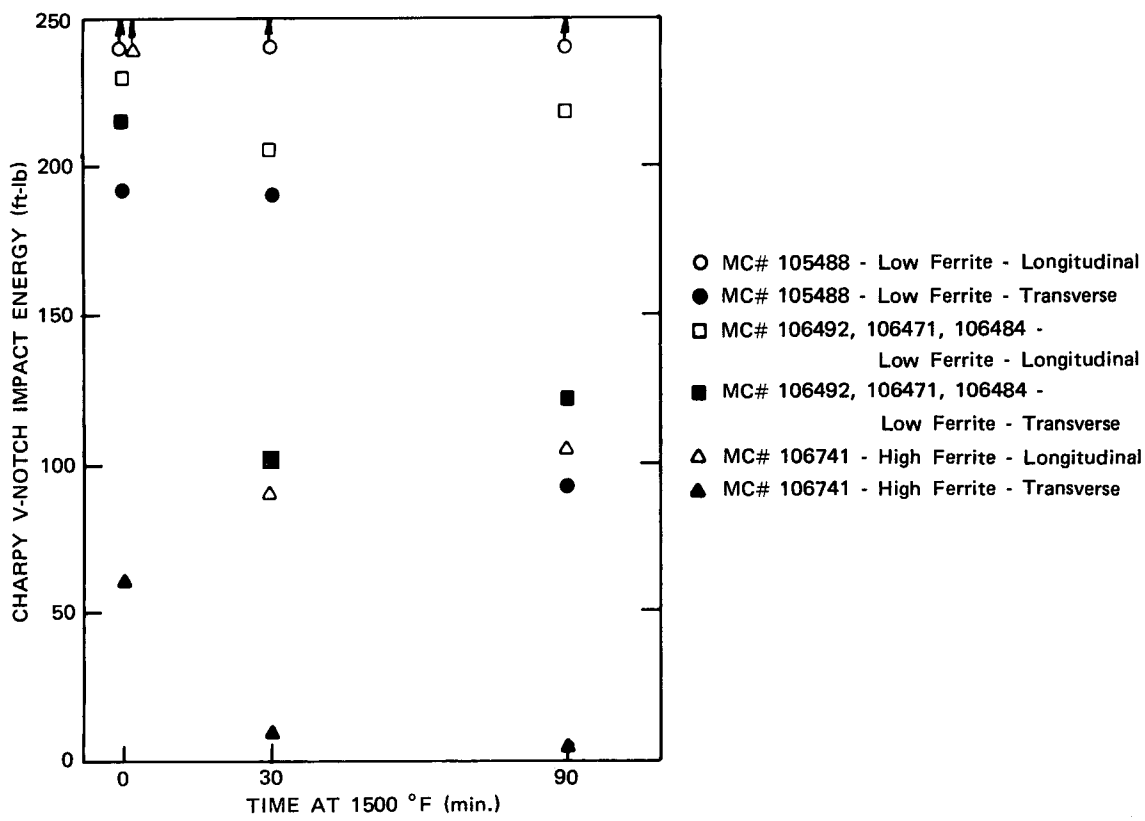


TABLE 6. Room Temperature Charpy V-Notch Impact Results

Sample No.	Heat Treatment	Percent Delta Ferrite (Severn Indicator) Before Heat Treat	After Heat Treat	Impact Energy (ft-lb) (Average of 2 Tests)	
105488	As-Rolled	<0.6	---	L >240	T 192
	1500 °F, 30 min.	<0.6	<0.6	L >240	T 191
	1500 °F, 90 min.	<0.6	<0.6	L >240	T 92
106492	As-Rolled	<0.6	---	L 230	T 216
106471	1500 °F, 30 min.	<0.6	<0.6	L 206	T 102
106484	1500 °F, 90 min.	<0.6	<0.6	L 217	T 122
106741	As-Rolled	<0.6 - <1.4	---	L >240	T 62
	1500 °F, 30 min.	<0.6 - <1.4	<0.6 - <1.0	L 88	T 8
	1500 °F, 90 min.	<0.6 - <1.4	<0.6	L 104	T 6

FIGURE 16. Charpy V-Notch Impact Energy Vs. Time at 1500 °F



shows the variation in impact energy with heat treating time at 1500 °F. The notch on the longitudinal impact specimens was oriented so that the "V" pointed downward from the surface of the plate. The notch of the transverse impact specimens was oriented so that the "V" pointed toward the midthickness of the plate.

For the low ferrite plate, the longitudinal impact energies were not significantly affected by heat treating. The transverse impact energies decreased after heat treating, indicating the adverse effect of sigma formation.

For the high ferrite plate, both the longitudinal and transverse impact energies decreased after heat treating to form sigma. After heat treating at 1500 °F for 30-90 minutes, the transverse impact energy was only 6-8 ft-lb. In addition, the low as-rolled impact energy of 62 ft-lb in the transverse direction indicates that a large amount of delta ferrite alone is detrimental to toughness.

Impact Shear Testing

The impact shear test specimen is a pin 0.400-in. long and 0.130-in. diameter and is broken in the same manner as an Izod impact test. The test is conducted by placing the specimen in an upright position in a holding die so that one-half of the specimen is unsupported. A flat anvil impacts the specimen parallel with the protruding length and the absorbed energy is recorded.[4]

Results obtained from room temperature impact shear tests are shown in Table 7. Figure 17 shows the variation in impact shear value with heat treating time at 1500 °F.

The major effect of the presence of sigma on the impact shear values was a reduction in transverse impact strength in the high ferrite plate. Note the much lower transverse impact strengths of the high ferrite plate after heat treating at 1500 °F for 5 and 24 hours.

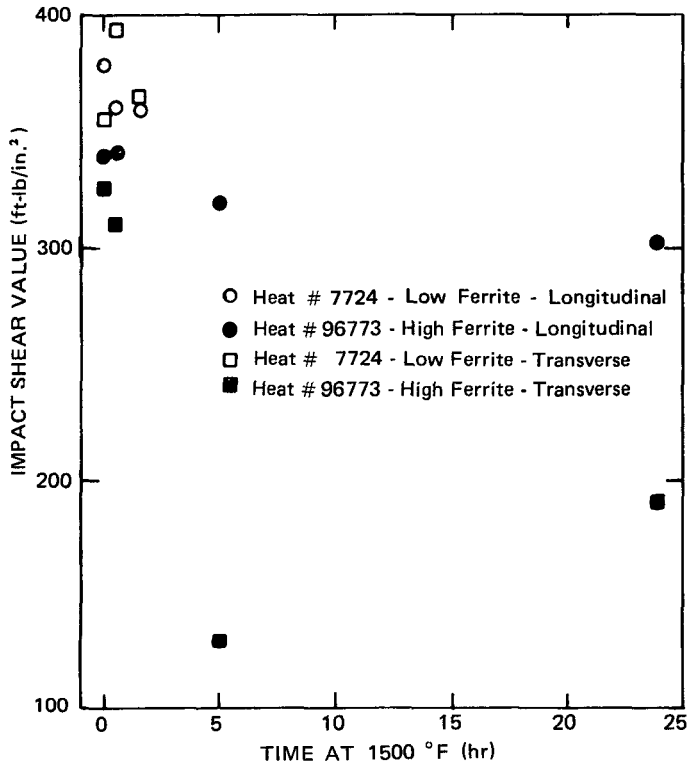
VENDOR CONTACTS

One approach to controlling the amount of sigma formation is to control the amount of delta ferrite present in the plate stock. A review of vendor/ Rocky Flats correspondence was performed and

TABLE 7. Room Temperature Impact Shear Results

Sample No.	Heat Treatment	Percent Delta Ferrite (Severn Indicator) Before Heat Treat	After Heat Treat	Impact Shear Value (ft-lb/in.²)	
105488	As-Rolled	<0.6	---	L 379.4	T 355.6
	1500 °F, 30 min.	<0.6	<0.6	L 359.4	T 393.5
	1500 °F, 30 min.	<0.6	<0.6	L 357.7	T 365.2
105458	As-Rolled	>1.4 - <3.8	---	L 338.1	T 326.2
105466	1500 °F, 30 min.	>3.0 - <4.0	>1.4 - <3.1	L 341.4	T 310.1
105465	1500 °F, 5 hr	>3.0 - <4.0	<0.6 - <1.4	L 317.6	T 129.4
105473	1500 °F, 24 hr	>3.0 - <4.0	<0.6	L 301.0	T 188.2

FIGURE 17. Impact Shear Values Vs. Time at 1500 °F



Armco Steel and Guterl Steel (formerly Simonds) were contacted to obtain information on 21-6-9 plate quality. Comments pertinent to the forging cracking problem and the formation of sigma phase from delta ferrite are summarized below:

1. The G. O. Carlson plate used for the forgings which experienced cracks was received in February 1977. It was found to contain more than 3% ferrite but was accepted by the Material Review Board.

   G. O. Carlson's solutions to the excess ferrite[5] were:

   a. Raise the nickel limits from between 5.5-7.0% to between 6.0-7.5%.

   b. Reduce the rolling temperature from 2300 to 2150 °F.

   c. Add a billet soak before rolling.

   None of the above changes have been tried because G. O. Carlson has not supplied any plate since 1977.

2. Data obtained from Armco Steel[6] shows that ferrite in 21-6-9 can be controlled by thermal treatments. Armco performed an experiment with 21-6-9 of the following composition: 20.9% Cr-7.06% Ni-0.27% N_2-8.9% Mn-0.01% C-0.52% Si-0.32% Mo. In this experiment small ingots were cast and the material was subjected to thermal treatment to determine the effect on ferrite concentration. Some of the ingots were rolled prior to the thermal treatments and others remained in the as-cast form. The wrought plate was produced by casting a 3-in. X 3-in. X 3-in. billet and rolling to 0.8-in. X 3-in. X 11-in. plate. Prior to rolling, the ingots received the following thermal processing: 2 hours soak at 2000 °F, slowly heated to 2200 °F, and held at this temperature for 2 hours prior to rolling. The ferrite content after the thermal processing was:

Product: As-Cast 1-in. X 3-in. X 9-in. Ingot
Wrought 0.8-in. X 3-in. X 11-in. Plate

Thermal Treatment	As Cast	Wrought
None	5.4% Ferrite	0% Ferrite
3 hr 2000 °F WQ	3.7% Ferrite	0% Ferrite
3 hr 2200 °F WQ	2.2% Ferrite	0% Ferrite
3 hr 2300 °F WQ	3.9% Ferrite	0.4% Ferrite
3 hr 2350 °F WQ	15.0% Ferrite	3.2% Ferrite
3 hr 2400 °F WQ	9.6% Ferrite	4.9% Ferrite
3 hr 2450 °F WQ	10.5% Ferrite	13.5% Ferrite

3. Routinely supplying plate stock with <0.6% delta ferrite is no problem according to Guterl Steel (formerly Simonds).[7] Lowering of the maximum limit on delta ferrite in the specification would be acceptable. Guterl Steel also indicated that faster cooling rates are more conducive to delta ferrite formation.

Controlling the delta ferrite content by chemistry adjustments must be approached with caution. Reducing the ferrite in the as-received plate by changing the chemistry may interfere with the ability to form delta ferrite during welding. Ferrite in the welds helps prevent cracking in the deep penetration EB welds required in this application.[8]

DISCUSSION

In 21-6-9 stainless steel the formation of sigma phase from delta ferrite occurs quickly. From data collected for this report, it appears that the most rapid formation of sigma occurs at approximately 1500 °F. In as-rolled plate at 1500 °F transformation of sigma from ferrite begins within 30 minutes and is almost complete after 24 hours. In future work complete transformation curves will be determined and the effect of cold work on the transformation rate will be examined.

The forging problem is related to a combination of high delta ferrite content (>0.6%), a thermal forging history which allows the delta ferrite to transform to the brittle sigma phase, and a poor flow line geometry (transverse to the wall of the forging).

Sigma forms within 30 minutes at 1500 °F which is a temperature seen in forging. The sigma probably forms during heating for forging. Retained work in the forging from previous operations accelerates the transformation on subsequent heating cycles into the 1400 to 1600 °F temperature range. Raising the forging temperature to above 1600 °F combined with rapid heating past the nose of the delta ferrite to sigma transformation curve (1400 to 1600 °F) should reduce the amount of sigma in the forgings. However, since the transformation is so rapid at 1500 °F and appears to be significantly increased in the presence of cold work, some transformation is probable even if the forging temperature was raised. Raising the forging temperature would also result in yield strengths below specification in the forgings.

As summarized in Figures 14 through 17, the effect of sigma on mechanical properties is greatly dependent on specimen orientation. The results obtained from tensile testing illustrate the drastic loss in transverse ductility (as measured by percent elongation and percent reduction in area) with increasing amounts of sigma formation. The longitudinal ductility was not affected by the presence of sigma. The impact shear test results indicate a severe reduction in transverse impact strength with sigma formation in high ferrite plate heat treated at 1500 °F for 5 and 24 hours. Sigma formation had an adverse effect on Charpy V-notch impact energy in the transverse direction in both low and high ferrite plate. In the high ferrite plate, the longitudinal Charpy energies were also severely affected by sigma formation. Charpy V-notch impact testing appears to be a more sensitive method than impact shear testing for evaluating the effect of sigma on impact resistance.

With respect to the transverse flow lines in the area of the parting plane of the forgings, it should be emphasized that both burst test and forging problems are associated with the combination of flow line geometry and the presence of sigma phase. Transverse flow lines are undesirable in critical applications and tensile ductility is generally poorer in the transverse orientation. For optimum impact resistance, limiting the amount of delta ferrite appears to be necessary even if only longitudinal flow lines are present.

As the result of burst testing, it is known that 3.0% delta ferrite is unacceptable and that less than 0.6% appears to cause no problems. However, there is not enough data available at intermediate percentages to define the maximum acceptable level of delta ferrite. Future work will be aimed at defining this limit and incorporating it into the material specification. This work is outlined later in this report.

It is recommended that (1) tensile properties be determined as a function of grain flow and delta ferrite/sigma content in forgings, (2) production forgings be examined metallographically in a transverse orientation at the mouth of the forgings, and (3) in the future, forging processes be developed which preclude transverse grain flow.

There appear to be a number of means to reduce the amount of delta ferrite in the stock we buy from the mills. They are as follows:

1. Homogenization (approximately 2200 °F, 6 hr) to dissolve remnant delta ferrite in the casting.

2. Control of breakdown temperature to enhance solution, instead of precipitation, of the delta ferrite.

3. Modification of the chemistry to reduce the alloy's "Cr equivalent," thereby reducing the tendency to form delta ferrite and sigma.

4. Revision of the mill specification to lower the maximum limit for delta ferrite.

CONCLUSIONS

1. Sigma phase can be identified in 21-6-9 via color etching. Complementary application of a second identification technique is helpful in relating specific colors to phases.

2. Delta ferrite transforms to sigma within 30 minutes at 1500 °F.

3. Sigma phase severely degrades tensile ductility and impact resistance in the transverse orientation. Longitudinal properties, on the other hand, are generally only slightly affected. However, Charpy V-notch impact testing indicates that both the transverse and longitudinal impact resistance of high ferrite plate is adversely affected by the presence of sigma phase.

4. When tensile bars are only taken from areas exhibiting longitudinal grain flow, the results will be insensitive to the presence of sigma and therefore do not represent the true quality of the forgings. Tensiles should be taken at the parting plane where the transverse flow is located.

5. The best way to monitor the production forgings for micro cracks along delta ferrite-sigma stringers is to review the forgings metallographically in a transverse orientation at the mouth of the forgings.

6. There are a number of ways the mill can reduce delta ferrite in the cast product. They are:

   a. Homogenization

   b. Control of ingot breakdown

   c. Chemistry adjustment

7. It should be possible to reduce, but not eliminate, the amount of transformed sigma in the forgings by raising the forging temperatures above 1600 °F and by rapidly heating through the 1400 to 1600 °F temperature range. However, raising the forging temperature will also result in a concomitant loss in forging strength.

8. Additional work must be done to determine the maximum allowable delta ferrite (and sigma) we can tolerate in this particular application. Mechanical testing should be performed to help establish this limit.

9. Additional work must also be done to better characterize the ferrite distribution in the as-received plate.

FUTURE WORK

Work is in progress, or future work is planned, to achieve the following objectives:

1. Determine sigma transformation curves for the annealed plate and plate cold worked 20, 40, and 60%.

2. Determine the thermal treatment required to solutionize delta ferrite.

3. Obtain additional data on the effect of sigma on mechanical properties to establish the maximum acceptable amounts of delta ferrite and sigma for this application.

4. Better characterize the ferrite distribution within the as-received plates. Preliminary work on eddy current testing indicates that it is a promising prospect for nondestructively determining the differences in ferrite content within a plate.

5. Continue vendor contacts.

6. Propose modifications to the present specification for 21-6-9 plate.

7. Establish new standards for measuring delta ferrite in 21-6-9 stainless steel.

8. Possibly expand the study to examine sigma formation in other alloys such as 22-13-5.

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APPENDIX
(Review of Literature)

A literature survey was performed to review the published literature on sigma phase, with particular emphasis on literature concerning the transformation of delta ferrite to sigma. Literature on the transformation kinetics of delta ferrite and sigma, the factors affecting the formation of delta ferrite and sigma, the effects of delta ferrite and sigma on the alloy properties, and methods to identify and measure delta ferrite and sigma are summarized below.

Description of Sigma

Sigma phase exhibits a tetragonal space lattice with 30 atoms/unit cell and a c/a ratio of ~0.52.[1] Sigma phase is very hard and brittle at room temperature, with Fe-Cr sigma phase having a hardness of ~68 R_C.[2] At room temperature sigma phase is nonmagnetic and has essentially zero ductility.[3]

Predicting Sigma Formation

Hull[4] evaluated fifteen elements with respect to sigma and chi embrittlement. Four hundred and fifty alloys were prepared based on a 16 Cr - 20 Ni - 10 Mn - 2.25 Mo class of stainless steels. Alloy dependency of embrittlement was determined using multiple regression analysis and expressed as "equivalent chromium." The expression is:

$$\text{"Equivalent Cr"} = \text{Cr} + 0.31\ \text{wt \% Mn} + 1.76\ \text{Mo} + 0.97\ \text{W} + 2.02\ \text{V} + 1.58\ \text{Si} + 2.44\ \text{Ti} + 1.70\ \text{Cb} + 1.22\ \text{Ta} - 0.226\ \text{Ni} - 0.177\ \text{Co}. \quad (1)$$

It was found that the severity of embrittlement is proportional to the "equivalent Cr" content over approximately 18%; below that, no embrittlement occurs.

In the analysis carbon and nitrogen were held to 0.002-0.005 wt % and 0.0015 wt %, respectively. Their effect is important, however, and in greater amounts may deplete the matrix of Cr and other carbide forming elements. The "equivalent Cr" is reduced, thus reducing the tendency for sigma and chi formation and for the resulting embrittlement. If one ignores the high nitrogen content in 21-6-9 alloy, approximately 0.28%, the "equivalent Cr" is approximately 22.5 wt %. Assuming that nitrogen will combine with Cr and other carbide formers in an M_X (N,C), a metal nitro-carbide, the "equivalent Cr" content should be below 22.5. Another shortcoming of the analysis in trying to apply the results to sigma formation in 21-6-9 is that the microstructure of the alloys in Hull's work did not appear to have delta ferrite, as does the 21-6-9 alloy in this investigation. As stated earlier, delta ferrite has a significant effect on sigma formation kinetics. The "equivalent Cr" equation, however, can be a useful tool in analyzing a stainless steel for sigma forming tendencies.

Formation of Sigma

In alloys balanced in composition so that they contain no delta ferrite, the precipitation of sigma is quite sluggish and is encountered only on prolonged exposure at elevated temperature.[5] However, in cold worked 316 TiM stainless steel which apparently contains no delta ferrite, sigma can form in about 10 minutes at 800 °C.[6] In alloys which are

predominantly austenitic, though containing some ferrite, sigma may form without the aid of retained work during ordinary commercial heat treatments.[5] In alloys containing some ferrite dispersed in the austenite matrix, the preferential formation of sigma from ferrite has been explained by the following four factors.[7]

1. The crystallographic structure of ferrite resembles that of sigma more closely than does the structure of austenite.

2. The atomic mobility is greater in ferrite than in austenite.

3. The preferential precipitation of carbides in the ferrite sites concentrates the alloying elements locally or distorts the ferrite lattice and thereby facilitates the formation of sigma.

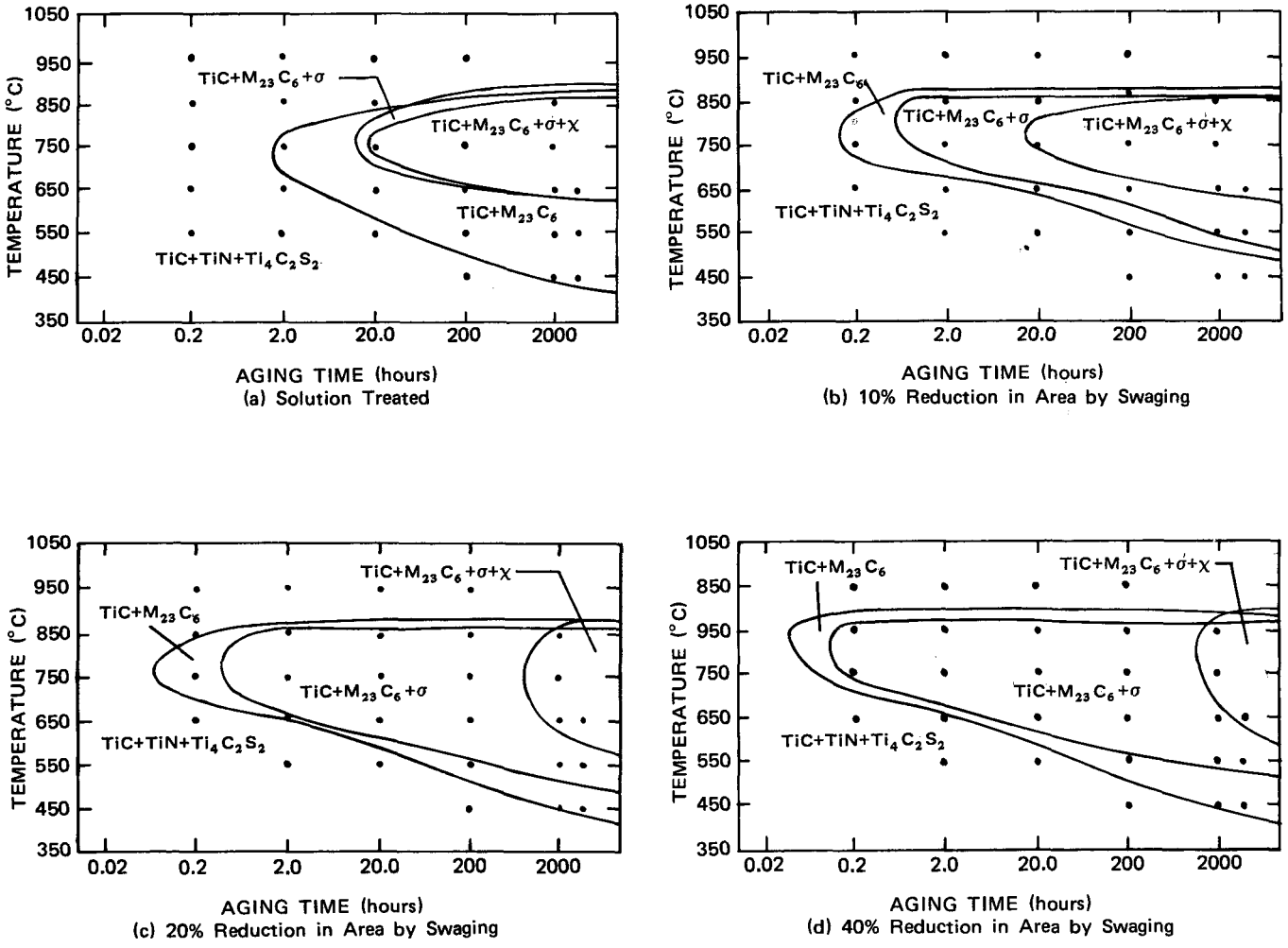
4. The segregation of Cr in ferrite in preference to austenite tends to produce ferrite areas which are considerably higher in Cr than the normal alloy composition. Since sigma is high in Cr it would naturally form from high Cr, ferrite areas.

Duplex compositions exposed for lengthy periods at precipitation temperatures may also show highly dispersed sigma particles in the austenite matrix.

The rate of formation of sigma form delta ferrite seems to be a function of the initial concentration of alloying elements in the ferrite. More highly alloyed ferrite tends to transform to sigma more rapidly than ferrite having a low initial concentration of alloying elements.[8] Aging results in disproportionation of the alloying elements and transformation to sigma.[9]

In Fe-Cr-Ni alloys sigma phase preserves, after transformation, the form and distribution of the old delta ferrite phase.[10] Sigma phase develops in the form of platelets oriented on specific planes of the delta ferrite crystallographic lattice.[9] Either a needle or a plate form of sigma can be seen according to the orientation of the delta ferrite as related to the polished plane of the specimen.[11] In partially ferrite compositions sigma formation starts in the fringes of the ferrite areas.[12] Sigma nucleates at high energy boundaries such as triple grain junctions, grain and twin boundaries and interphase boundaries.[6] Coupled with the high Cr content of the ferrite it is reasonable to expect that the sigma first nucleates and grows at the ferrite-austenite interface boundary. In a composition highly susceptible to sigma formation, all the ferrite may disappear and, by two-way diffusion, cause formation of sigma and austenite if the alloy is held at suitably high temperatures for sufficiently long periods.[12]

The ferrite-to-sigma transformation is remarkably accelerated by prior cold work[10] which may enhance the formation of sigma, even more than does the presence of ferrite, in alloys exposed between 1050 and 1700 °F.[8] The time-temperature-precipitation diagrams shown in Figure A-1 illustrate this effect in a Type 316 TiM steel. There are a number of different mechanisms by which cold work can enhance sigma formation. Weiss and Stickler[13] showed that sigma can nucleate at deformation fault intersections. It has been suggested that sigma formation is enhanced by the increased diffusion rates in highly cold worked, dislocated structures.[14] And lastly, cold work can cause recrystallization during aging in the sigma forming range. Apparently the boundaries between recrystallized and unrecrystallized grains are preferential sites for nucleation and growth.[15] Grot and Spruiell[16] have suggested that



* Arnold S. Grot and Joseph E. Spruiell, "Microstructural Stability of Titanium-Modified Type 316 and Type 321 Stainless Steel," in *Metallurgical Transactions A*, Vol. 6A, p. 2025, (1975). Copyright American Society for Metals and The Metallurgical Society of AIME 1975.

FIGURE A-1. Time-Temperature-Precipation Diagrams for Type 316 TiM Steel*

these sites were made more favorable because of partial resolution of carbides or Laves phase which resulted in increased amounts of sigma forming elements. This effectively increases the local "equivalent Cr" content.

Cold work is also effective in promoting a finer distribution and less acicular type of sigma.[8] The rate of sigma formation is also increased when stresses are applied to alloys exposed to temperatures between 1050 and 1700 °F.[17] Nitronic 40 (21-6-9) is reported to be resistant to sigma formation up to 1100 °F, above which long exposure under stressed conditions is not recommended.[18]

Sigma transformation follows the familiar C-curve behavior characteristic of nucleation and growth processes;[19] the rate of transformation being dependent on both time and temperature.[20] The fastest generation of sigma occurs not at the top of the temperature range but about midway between the maximum and minimum temperatures at which it can be formed.[21] Sigma phase in susceptible stainless steels is precipitated between 1000 and 1700 °F, the exact range varying somewhat in different compositions.[22] The temperature above which sigma becomes unstable and tends to transform into austenite or ferrite depends on the composition and structure of the alloy.[17] Generally, in steels at which maximum amounts of sigma form at or below 1500 °F, sigma phase becomes unstable above 1700 °F.[17] Stainless steels with compositions susceptible to rapid sigma precipitation may have to be water quenched from temperatures above 1900 to 2000 °F to prevent the precipitation of sigma phase.[8]

Sigma precipitation is usually preceded by $M_{23}C_6$ and accompanied by chi precipitation. Like sigma, $M_{23}C_6$ carbide formation is accelerated by cold work.[15] In alloys containing ferrite the carbide preferentially nucleates at the austenite-ferrite interface boundary.[23] Besides being at a high energy location, the forming nucleii are immediately adjacent to the high Cr in the ferrite and the high carbon of the austenite. The $M_{23}C_6$ carbides have been reported to have a positive,[24] negative,[25] and neutral effect[14] on sigma nucleation. At the extremes it appears that dissolving carbides would enhance sigma formation because of the new availability of the sigma forming elements (i.e., Cr) and precipitating or stable carbides might exert a negative effect because of local depletion of sigma formers. After a sufficient amount of diffusion to eliminate these chemical gradients, the carbide-matrix boundary could provide a nucleation site for sigma.[25] It has also been suggested that carbides can retard sigma formation by retarding recrystallization.[15]

Alloy conditions which favor sigma also favor chi formation. Chi has a B.C.C. α-manganese crystal structure which, like sigma, is stabilized by Cr, Mo, Mn, and others. Sigma and chi precipitate in the same temperature range as $M_{23}C_6$ (650-900 °C) and nucleate at similar sites. Chi precipitation is also enhanced by cold work.[26] An exception occurs in 316 Ti modified stainless steel.[27] Optically, sigma and chi are generally indistinguishable.[28] Unlike sigma, chi can dissolve appreciable amounts of carbon.[29]

Unfortunately, there appears to be little information on the effect of chi on mechanical properties. However, Hull[30] suggests that both sigma and chi have embrittling tendencies. Since chi can form as an intermetallic compound or a type $M_{18}C$ carbide,[24] it does appear that chi could also have an embrittling effect.

Most 300 series steels containing >17 wt % Cr are capable of forming sigma.[31] Fully austenitic varieties are just as capable of sigma formation as duplex alloys, although in

fully austenitic alloys sigma forms directly from austenite along the grain boundaries.[31] Elements commonly added to stainless steels to stabilize the carbide, to enhance creep and stress-rupture properties, or for deoxidation in the steel mill seem to widen the temperature range for sigma precipitation.[22] Most of the alloying elements which support the formation of ferrite also considerably enhance the susceptibility of the alloy to the formation of sigma.[32] The effect of carbon is to decrease sigma formation in Fe-Cr alloys owing to the removal of Cr from solid solution by the formation of chromium carbides.[33] Nickel present in amounts >4% makes an alloy less susceptible to sigma formation.[33] Manganese promotes austenite formation and thus exerts a negative effect on sigma formation.[33] Small amounts of silicon markedly accelerate the rate of formation of sigma.[33] The effect of some other elements can be seen in Equation (1), listed earlier in this Appendix.

The ferrite content of an alloy can be controlled by increasing the austenite forming elements to the upper limits and the ferrite forming elements to the lower limits of the specification.

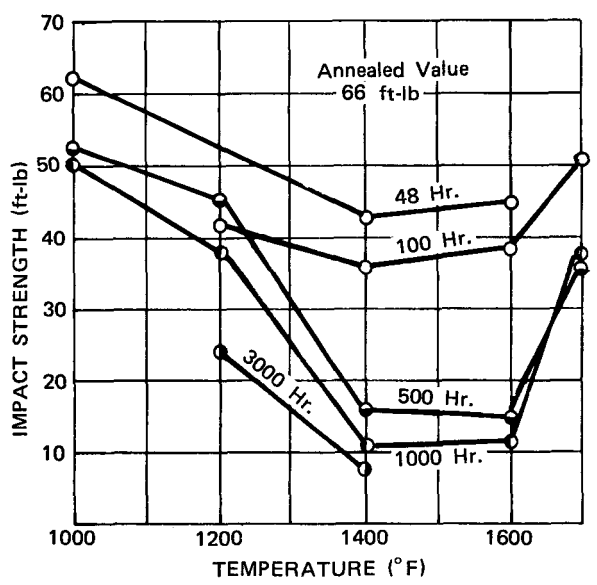
Effect of Sigma on Properties

The precipitation and growth of sigma particles cause considerable changes in the properties of an alloy. The transformation of relatively ductile delta ferrite to the brittle sigma phase is of importance in commercial applications because it usually adversely affects the mechanical properties.[34] The magnitude of the effect of sigma on the mechanical and corrosion properties of stainless steel is greatly dependent not only upon the amount of sigma present but also on the particle size and distribution.[35] The embrittling effect of sigma is more pronounced at room temperature than at elevated temperatures.[36]

The most sensitive test for the presence of sigma phase is the room temperature impact strength.[37] The effect of aging on the room temperature impact strength of Type 310 is shown in Figure A-2. The lowered impact strength was attributed primarily to sigma

FIGURE A-2. Effect of Aging on Room-Temperature Impact Strength of Type 310*

*A. J. Lena, "Effect of Sigma Phase on Properties of Alloys," in *Metal Progress*, p. 95, August 1954. Copyright American Society for Metals 1954.



formation.[38] In Fe-Ni-Cr alloys in the range 20-35% Ni and 15-30% Cr, embrittlement as measured by room temperature impact properties increases rapidly with the first few percent of sigma formed, regardless of base composition.[39] Figure A-3 shows the correlation between room temperature impact energy and percent sigma for Fe-Ni-Cr alloys exposed 3000 hours at the given temperatures. In partially ferritic chromium-nickel stainless steels even the initial formation of sigma in the fringes of ferrite patches severely lowers the bending and impact properties.[12] Hall and Algie[31] reported that sigma containing steels have on occasion shattered like glass when dropped to the floor. High temperature impact tests indicate that the presence of sigma, which has a discontinuous morphology, does not embrittle an alloy at service temperatures as much as at room temperature.[40] However, severe embrittlement can occur at high strain rates; i.e., shock loading.[41]

The presence of sigma phase in delta ferrite appears to play a significant role in fracture formation and propagation. Fractures often follow the original ferrite-austenite matrix interface which has become embrittled by sigma.[42] The extensive platelike morphology of sigma is an excellent source for crack initiation and propagation and has a grave effect on elevated temperature rupture strength.[43] Larger particles of sigma in a segregated pattern are more effective in lowering the ductility than small, finely distributed particles.[12] Since sigma precipitation interferes with hot-working operations, it sould be brought into solution by a suitable heat treatment prior to hot working.[12] Permanent damage results if microcracks were formed during the presence of sigma.[3]

The hardness and tensile strength are usually not significantly affected by the presence of sigma phase[44] and are poor tests for detecting sigma.[38] However, in some steels considerable increases in strength can be produced. We have shown in this report that sigma can degrade the ultimate tensile strength of 21-6-9. Figure A-4 shows the changes in room temperature properties owing to the formation of sigma in a steel with 0.10 C, 1.42 Si, 0.59 Mn, 4.29 Ni, 25.5 Cr and 1.63 Mo. Generally, the influence of a relatively non-continuous sigma phase on the mechanical properties is relatively small.

FIGURE A-3. Correlation of the Effect of Sigma on the Impact Resistance of Commercial Quality Iron-Nickel-Chromium Alloys*

*A. M. Talbot and D. E. Furman, "Sigma Formation and Its Effect on the Impact Properties of Iron-Nickel-Chromium Alloys," in *Transactions of the A.S.M.*, Vol. 45, p. 439, (1953). Copyright American Society for Metals 1953.

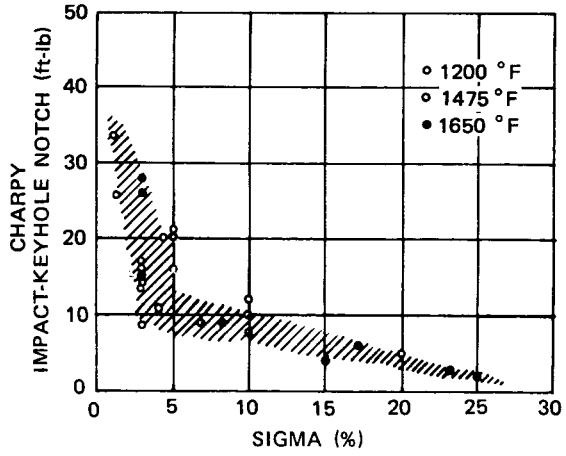
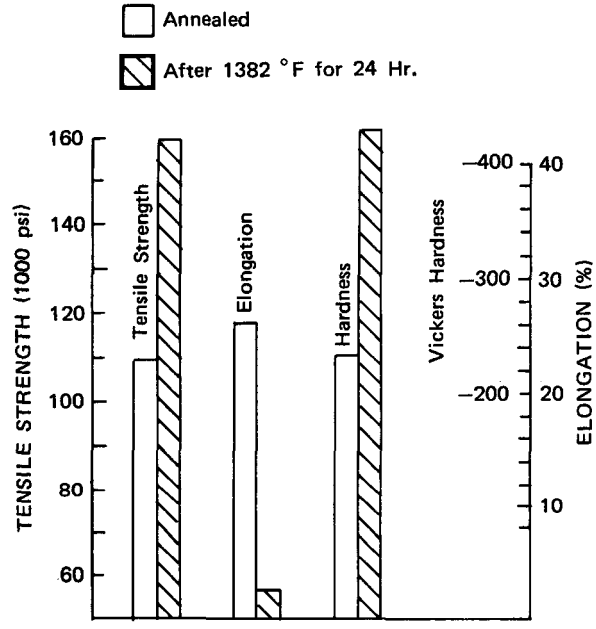


FIGURE A-4. Changes in Room-Temperature Properties Due to Formation of Sigma in a Steel of 0.10 C, 1.42 Si, 0.59 Mn, 4.29 Ni, 25.5 Cr, 1.63 Mo*

*A. J. Lena, "Effect of Sigma Phase on Properties of Alloys," in *Metal Progress*, p. 95, August 1954. Copyright American Society for Metals 1954.



The presence of sigma phase reduces the corrosion resistance of stainless steels in strong, oxidizing acids. The greater the amount of sigma precipitation the greater the chromium depletion of the matrix. In certain highly corrosive solutions the lower chromium content of the matrix as well as possible local electrolytic-cell action may considerably increase corrosion rates. A fine intergranular precipitate of sigma reduces corrosion resistance more effectively than large coalesced particles.[45]

Effect of Delta Ferrite on Properties

For all grades of stainless steel, the optimum hot workability is obtained when the structure of the steel at the hot working temperature consists essentially of a single phase. When ferrite is present, the difference in plasticity between the soft ferrite and tough austenite may cause rupture. Small amounts of ferrite in the structure of austenitic stainless steels can be tolerated, but must be kept within proper limits either by adjustment of the chemical composition of the steel or by adjustment of hot working temperatures.[46] Conversely, Kane[47] has recently reported that delta ferrite improves ingot hot workability in 310 stainless steel. Apparently the ferrite provides additional recrystallization sites and thus increases the annealing rate during deformation.

Effect of Delta Ferrite on Welds

The presence of a small amount of delta ferrite (~3-4%) has been reported to be helpful in reducing the hot cracking of stainless steel welds. The steel must be of a composition which allows the formation of delta ferrite during welding. Use of a Schaeffler diagram[48] permits

prediction of the ferrite content of a weld. This diagram is constructed by grouping the alloying elements into ferrite formers and austenite formers and multiplying by experimentally derived factors to get the corresponding nickel and chromium equivalents for a given alloy. Using the nickel and chromium equivalents for an alloy, the percent ferrite can be read from the diagram.

The delta ferrite has been reported to serve several functions. The most common theory for the effect of delta ferrite in reducing cracking in welds is based on the assumption that ferrite has a greater solubility than austenite for certain harmful elements and impurities. The function of the ferrite is to decrease the concentration of the injurious impurities at the austenite grain boundaries and thus to reduce their damaging effect on hot cracking.[49] Another theory is that the beneficial effect of delta ferrite results from the lower interfacial energy of the austenite-ferrite boundary than the austenite-austenite boundary. If the delta ferrite particles are sufficiently numerous and well distributed at the austenite grain boundaries, they can prevent hot cracking by supporting the small but increasing stresses imposed by thermal contraction of the alloy as it freezes under restraint until all the liquid has solidified and the austenite-austenite boundaries can bear their share of the load. If most of the ferrite forms as plates within the austenite grains, it is less effective.[50] Ferrite-containing weld metal also combats the harmful effects of precipitated carbides on the corrosion resistance. Carbides normally precipitate in and around the ferrite pools, instead of forming a continuous intergranular network.[51]

Identification and Measurement of Sigma and Delta Ferrite

One optical metallographic technique useful for identification of delta ferrite and sigma phase is the use of chemical staining etchants. The ferrite and sigma phases are stained different colors but definition of the phases is not positive and complementary application of another identification technique is helpful in relating specific colors to phases. After making a positive relationship between the phases and colors, the color staining technique can be used to identify delta ferrite, austenite, and sigma phases.[52]

Magnetic etching has also been used to identify delta ferrite and sigma. A thin film of iron colloid (sub-micron size iron oxide particles suspended in a fluid) is placed on a suitably prepared specimen. The attraction of the colloid particles to the delta ferrite phase is very pronounced and easily observed by optical metallography by switching an externally applied magnetic field on and off.[53]

Sigma phase and delta ferrite can also be identified by quantitative chemical analyses performed on a microprobe. Also, using a bulk extraction method the various phases can be extracted from the matrix. The residue obtained can be analyzed by X-ray powder diffraction techniques and the absolute weight fractions of various phases computed.[54]

The amount of delta ferrite present in a steel can be measured by several techniques. Some methods and instruments used for measuring delta ferrite are metallographic examination, Severn Gage, Magne Gage, eddy current testing, and the Ferrite Meter.

Metallographic determination of percent ferrite is hampered by the counting technique used and the aspherical shape of the ferrite particles. The procedure is time consuming and is not applicable for nondestructive testing.[55]

The Severn Gage uses a balance beam, and compares the attraction of a permanent magnet on one end to the sample, and to different standards. The ferrite content of a sample is bracketed between two standards.[55]

In the Magne Gage the force of attraction between a permanent magnet and the sample is measured against that applied to a lever arm by a spring. Values are obtained relative to the standards used to calibrate the spring force.[55]

Using a phase-sensitive eddy current system point-to-point variations in ferrite content in a sample can be detected with good reproducibility. Ferrite levels of 1% or more are easily detected. Eddy current testing requires a calibration standard having the same electrical conductivity as the sample.[55]

The Ferrite Meter operates on a similar principle to eddy current testing. An alternating current passed through the excitation coil induces a voltage in the two parts of the receiver coil windings, which are connected in opposition. When the probe is kept away from the metal sample surface, the output voltage is zero. When the probe is brought into contact with an austenite-ferrite mixture, the magnetic field is disturbed owing to the presence of ferrite, and the ferrite meter probe develops a voltage which depends on the amount of ferrite present.[55]

Since no dependable technique for ferrite measurement has been found, the Welding Research Council has adopted a procedure which uses the Magne Gage and standards made with various thicknesses of a nonmagnetic coating over a magnetic substrate. Ferrite levels are reported as ferrite numbers (FN). The FN is essentially equivalent to ferrite content up to about 8%. The standards bear no relation to the physical situation of ferrite in an austenite matrix.[55]

A common weakness of ferrite measuring techniques lies in calibration. The orientation of the ferrite particles affects the magnetic response. The chemical composition of the ferrite in a standard will also affect the calibration of ferrite measuring instruments. The Severn Gage and Magne Gage measure the average ferrite content within a large area, whereas the Ferrite Meter is able to detect smaller zones of lower or higher ferrite content.[55]

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