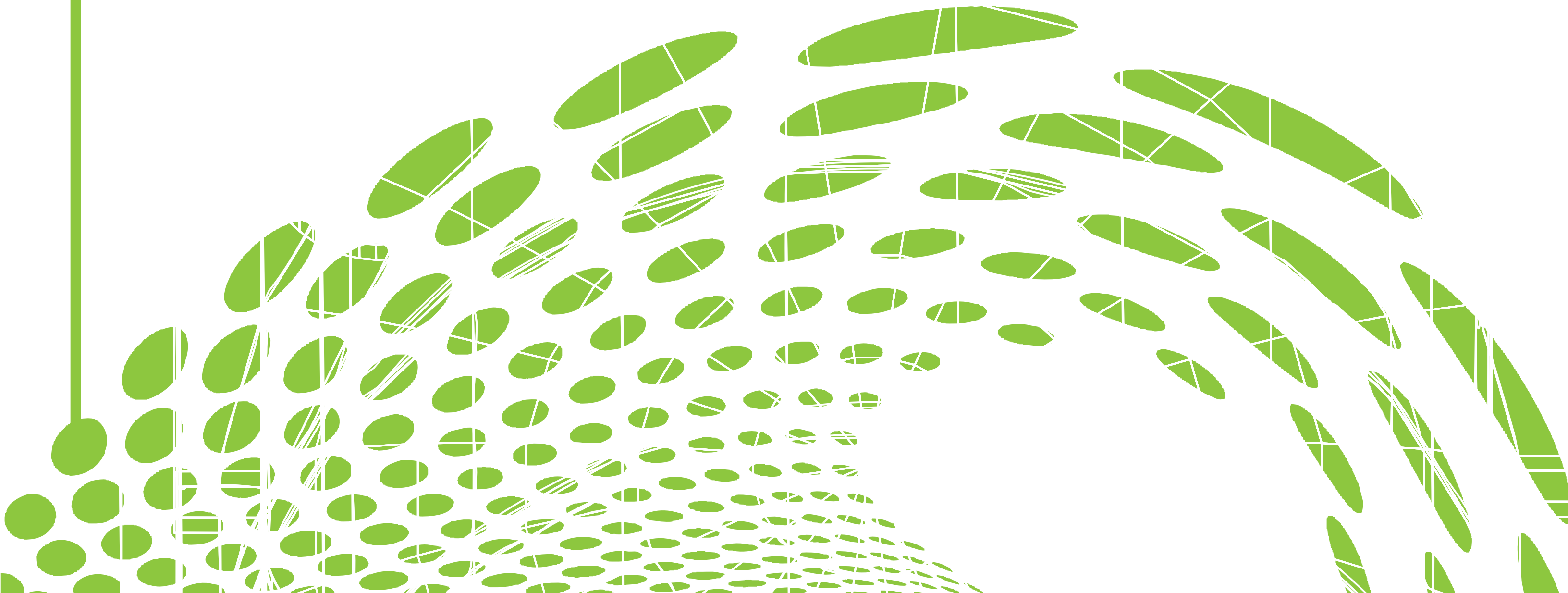
ASME STANDARDS TECHNOLOGY, LLC

STP-PT-075

Effect of Thickness on the Transformation Behavior of Grade 91 Steel



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EFFECT OF THICKNESS ON THE TRANSFORMATION BEHAVIOR OF GRADE 91 STEEL

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FOREWORD

This report evaluates the effect of thickness on the transformation behavior of Grade 91 steel alloy to further define the materials properties.

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ABSTRACT

The chemical compositions of six heats of Grade 91 steel were determined and two heats with furthest apart chemical composition were selected as test samples for the cooling rate tests and the Jominy hardness tests. A Jominy test apparatus was built in accordance with ASTM A255-10 [1] and successfully tested. Six Jominy bars were machined, three from each of the two heats. Thermocouples were welded at five centerline locations in radial holes drilled along the length of one bar from each heat. Jominy cooling rate determinations from 1940°F were made at the five locations from each heat. Jominy cooling rate determinations were essentially identical between the two instrumented bars. Two Jominy bars from each Grade 91 heat were austenitized at 1940°F and subjected to the Jominy test. Hardness per ASTM A255 was made on one bar, the second bar was tempered at 1410°F and then the hardness tests were made. Photomicrographs were made at the five locations in the Jominy bars where the thermocouples were located. All of the microstructures showed 100% martensite; there was no indication of transformation to a microstructure other than martensite.

1 INTRODUCTION

The Grade 91 alloy has been extensively studied, yet many questions still remain unanswered. The excellent elevated temperature properties of Grade 91 are dependent on the alloy achieving a tempered martensite microstructure after it is cooled from the austenitizing temperature. It must be tempered after cooling to assure an ideal microstructure. The question of the ability of Grade 91 to achieve such a microstructure through its entire thickness continues to be a subject of discussion. Grade 91 is a deeply hardenable alloy that should produce a martensitic when air-cooled in very thick section sizes. This project will allow the determination to what thickness such a microstructure can be assured. Based on past studies of this alloy, it should be able to achieve its maximum hardness through the entire four (4) inch length of a Jominy bar.

This research project could assist in determining what section sizes can provide a martensitic microstructure when properly heat-treated. Many of the organizations that are ASME Accredited and use Grade 91 in their fabrication practices will benefit from the results of this research. BPV Committee on Power Boilers (I), BPV Committee on Materials (II), and BPV Committee on Construction of Nuclear Facilities Components (III) should find this information invaluable. In particular, any organization involved in supercritical and ultra-super-critical boilers could be interested in this materials data.

2 TEST METHODOLOGY

2.1 Coupon Tests – or Jominy bar

The intent of this project was to determine whether a Jominy end quench test will permit the prediction of the microstructure produced when a Grade 91 bar is cooled from its austenitizing temperature. The test plan was divided into 7 tasks. The tasks were as follows:

- Task 1 was to build a Jominy Test apparatus and test it.
- Task 2 was to adapt a Jominy bar to accommodate five chromel-alumel thermocouples (TC) and to imbed the TCs at the mid-point of the longitudinal axis of the Jominy bar.
- Task 3 was to select two heats of Grade 91 whose product form had section sizes from which Jominy bars could be machined.
- Task 4 was to measure the cooling curves at the five locations in the Jominy bar.
- Task 5 was to conduct the Jominy tests on two bars from each heat.
- Task 6 was to prepare photomicrophs from the five locations in the Jominy bar where the cooling rates were measured.

2.2 Building and Testing a Jominy Test Apparatus

The first task was to build a Jominy test apparatus. This was done in accordance with ASTM A255-10 [1]. When the test apparatus was fabricated, an initial test was conducted to assure it was operating correctly. Figure 2-1 and Figure 2-2 represent photographs of the Jominy apparatus and the quench test. The upper photograph shows the test apparatus and a quench in progress. The bottom photograph shows the desirable conical shape of the water during the quench. This is exactly the water distribution required for an appropriate quench.

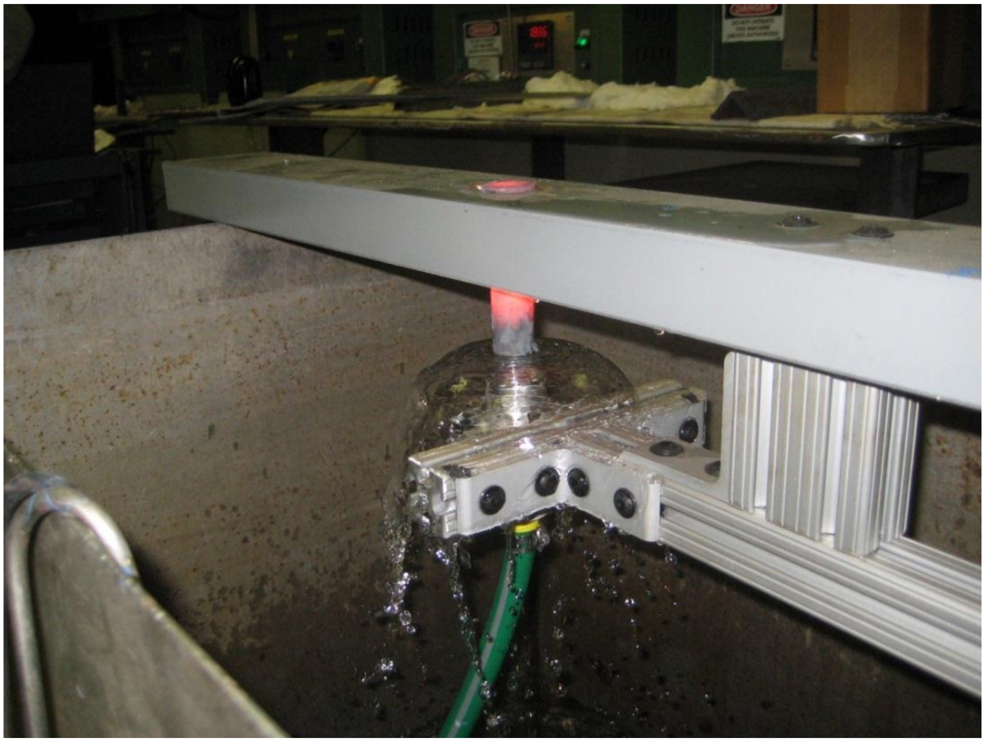


Figure 2-1: Initial Test of Jominy Bar Apparatus

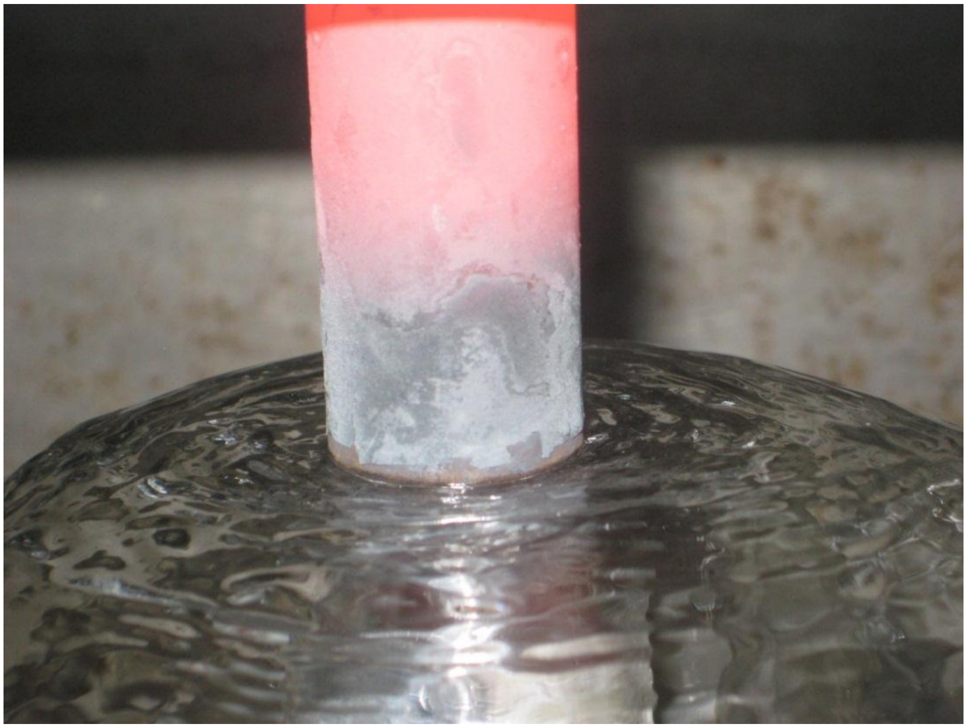


Figure 2-2: Desirable Conical Shape of Water during Quench

2.3 Designing a Jominy Bar

The second task was to design a Jominy bar, so it could accommodate the thermocouples required to measure the cooling rates at the five locations. The locations selected, measured from the quenched end, were 0.25 in., 0.75 in., 1.5 in., 2.5 in., and 3.75 in. Photographs showing the locations in a Jominy bar are shown in Figure 2-3. Figure 2-4 shows the manner whereby the TCs were embedded in the Jominy bar. The pairs of TC holes were drilled 0.10 inches apart and 0.093 inches in diameter to a depth of 0.375 inches. Then the holes were extended another 0.25 inches at 0.036 inches in diameter to accommodate the TC wires. Each hole was designed to accommodate a chromel or an alumel wire. The hole diameters were selected to accommodate ceramic insulation to the 0.375 in. depth and the bare TC wire beyond that depth. The TC holes were 0.10 inches apart straddling each of the five axial distances from the quenched end. The TC wires were electro-discharged welded into their respective holes. A chromel wire was welded at the bottom of one of the holes and an alumel wire was welded to the bottom of the adjacent hole. This match provided the chromel-alumel thermocouple needed to measure the temperature during the Jominy quench.

Figure 2-3: Thermocouple Locations, 0.25 in., 1.5 in., 2.5 in., and 3.75 in. from Quenched End of Jominy Bar

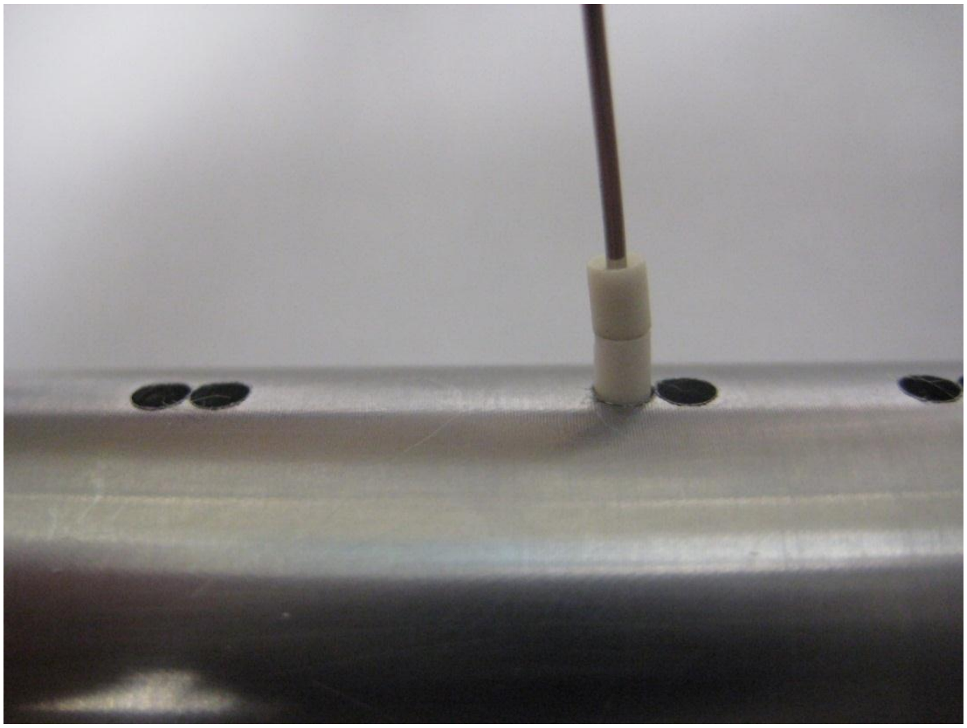


Figure 2-4: Individual Thermocouple (Chromel/Alumel) Embedded in Jominy Bar

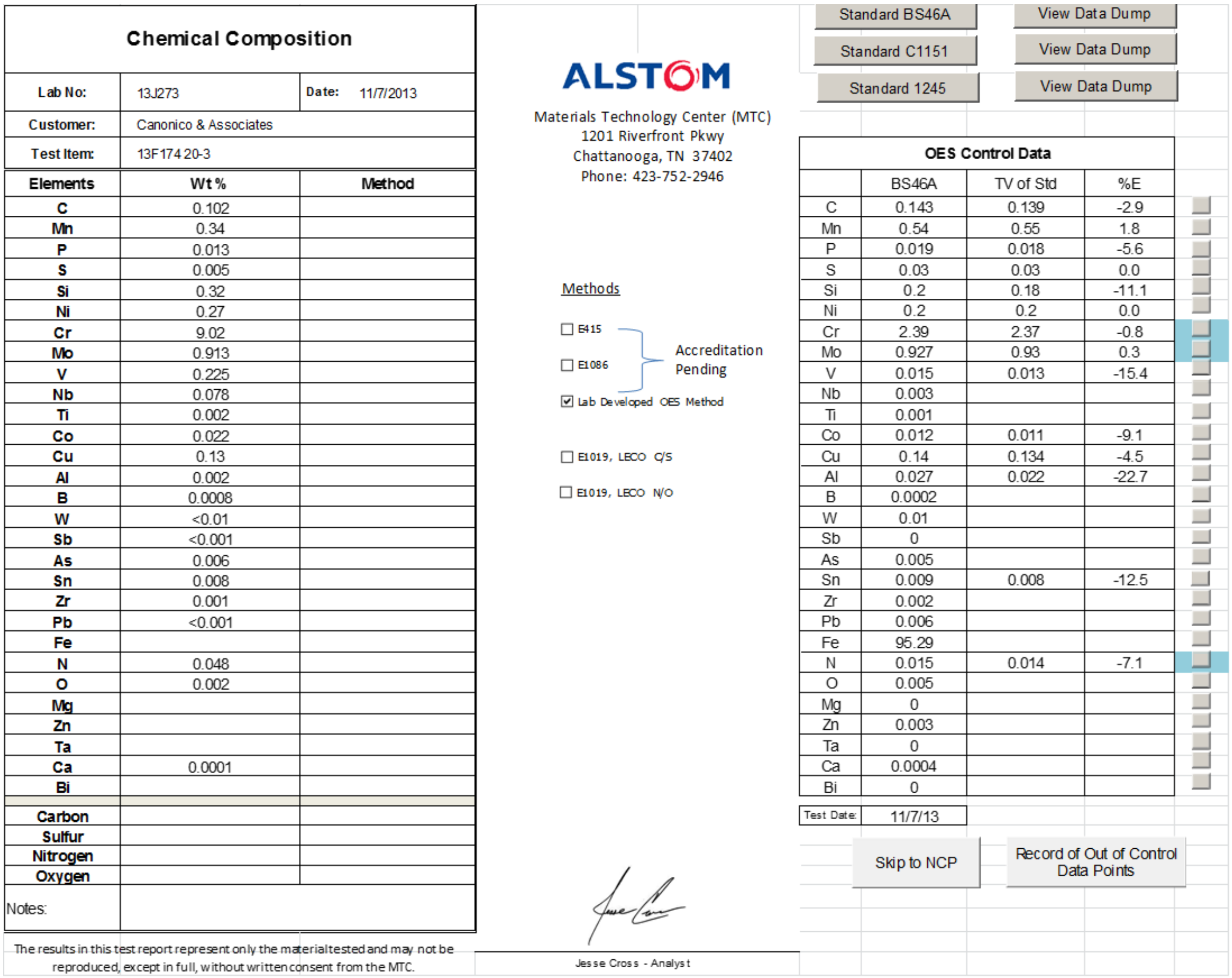
2.4 Selecting Two Heats of Grade 91

The third task was to select two Grade 91 heats to be used for Task 4 through 6. Alstom's Materials Technology Center (MTC) supplied six samples of Grade 91 alloy with six different heats. MTC conducted material test to determine the chemical composition of those six Grade 91 alloy samples. The Grade 91 alloys with the heats that had the further apart chemical composition were chosen for additional testing.

Test Bar No.	Heat	Supplier	Heat	Treatment	Test
1-1	High Heat	Wuhan Heavy Industry Casting & Forging Co., Ltd, China	13174 20-3	Austenitized at 1940ºF & Quenched	Cooling Rate
1-2	High Heat	Wuhan Heavy Industry Casting & Forging Co., Ltd, China	13174 20-3	Austenitized at 1940ºF & Quenched	Jominy
1-3	High Heat	Wuhan Heavy Industry Casting & Forging Co., Ltd, China	13174 20-3	Austenitized at 1940ºF, Quenched, Tempered at 1410°F	Jominy
2-1	Low Heat	IBF SpA, Italy	13CO90	Austenitized at 1940ºF & Quenched	Cooling Rate
2-2	Low Heat	IBF SpA, Italy	13CO90	Austenitized at 1940ºF & Quenched	Jominy
2-3	Low Heat	IBF SpA, Italy	13CO90	Austenitized at 1940ºF, Quenched, Tempered at 1410°F	Jominy

Their chemical compositions are shown in Figures 2-5 and 2-6.

As one might, expect the chemical compositions of all of the heats were quite similar; however, the sample bar from the Wuhan Heavy Industry Casting & Forging Co., Ltd., heat had a higher carbon content (Wt %) and higher Si, Ni, Mo, V, and B. The original plan was to machine three Jominy bars from one heat and two bars from the second heat. Because one thermocouple did not function correctly during the cooling of the first Jominy bar, it was decided to determine the cooling rates in a second Jominy bar. The second cooling rate test is shown in Figure 2-7. It may be that the shift from a smooth cooling curve is due to the transformation to martensite. The deviation from a smooth cooling curve is not seen because the thermocouple for unknown reasons became inoperative in this temperature range.



Chemical Composition

Lab No:	13J273	**Date:**	11/7/2013
Customer:	Canonico & Associates		
Test Item:	13F174 20-3		

Elements	Wt %	Method
C	0.102	
Mn	0.34	
P	0.013	
S	0.005	
Si	0.32	
Ni	0.27	
Cr	9.02	
Mo	0.913	
V	0.225	
Nb	0.078	
Ti	0.002	
Co	0.022	
Cu	0.13	
Al	0.002	
B	0.0008	
W	<0.01	
Sb	<0.001	
As	0.006	
Sn	0.008	
Zr	0.001	
Pb	<0.001	
Fe		
N	0.048	
O	0.002	
Mg		
Zn		
Ta		
Ca	0.0001	
Bi		
Carbon		
Sulfur		
Nitrogen		
Oxygen		
Notes:		

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ALSTOM

Materials Technology Center (MTC)
1201 Riverfront Pkwy
Chattanooga, TN 37402
Phone: 423-752-2946

Methods

☐ E415
☐ E1086
Accreditation Pending
☑ Lab Developed OES Method

☐ E1019, LECO C/S

☐ E1019, LECO N/O

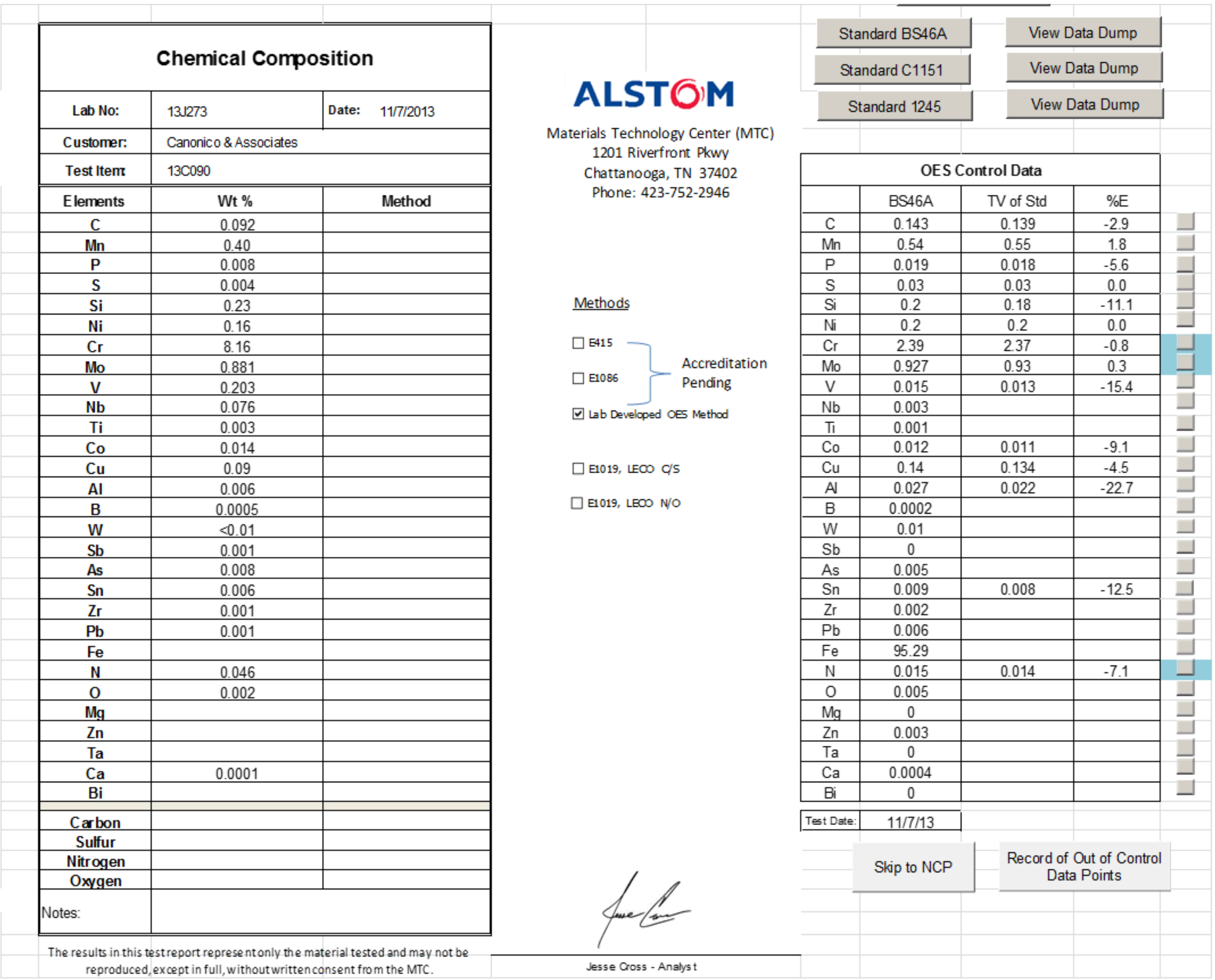
Jesse Cross - Analyst

OES Control Data

	BS46A	TV of Std	%E
C	0.143	0.139	-2.9
Mn	0.54	0.55	1.8
P	0.019	0.018	-5.6
S	0.03	0.03	0.0
Si	0.2	0.18	-11.1
Ni	0.2	0.2	0.0
Cr	2.39	2.37	-0.8
Mo	0.927	0.93	0.3
V	0.015	0.013	-15.4
Nb	0.003		
Ti	0.001		
Co	0.012	0.011	-9.1
Cu	0.14	0.134	-4.5
Al	0.027	0.022	-22.7
B	0.0002		
W	0.01		
Sb	0		
As	0.005		
Sn	0.009	0.008	-12.5
Zr	0.002		
Pb	0.006		
Fe	95.29		
N	0.015	0.014	-7.1
O	0.005		
Mg	0		
Zn	0.003		
Ta	0		
Ca	0.0004		
Bi	0		

Test Date: 11/7/13

Figure 2-5: Chemical Composition of Test Bar No. 1; Identified as "High Heat"



Chemical Composition

Lab No:	13J273	Date:	11/7/2013
Customer:	Canonico & Associates		
Test Item:	13C090		

Elements	Wt %	Method
C	0.092	
Mn	0.40	
P	0.008	
S	0.004	
Si	0.23	
Ni	0.16	
Cr	8.16	
Mo	0.881	
V	0.203	
Nb	0.076	
Ti	0.003	
Co	0.014	
Cu	0.09	
Al	0.006	
B	0.0005	
W	<0.01	
Sb	0.001	
As	0.008	
Sn	0.006	
Zr	0.001	
Pb	0.001	
Fe		
N	0.046	
O	0.002	
Mg		
Zn		
Ta		
Ca	0.0001	
Bi		
Carbon		
Sulfur		
Nitrogen		
Oxygen		
Notes:		

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Methods

☐ E415
☐ E1086
Accreditation Pending
☑ Lab Developed OES Method
☐ E1019, LECO C/S
☐ E1019, LECO N/O

Jesse Cross - Analyst

OES Control Data

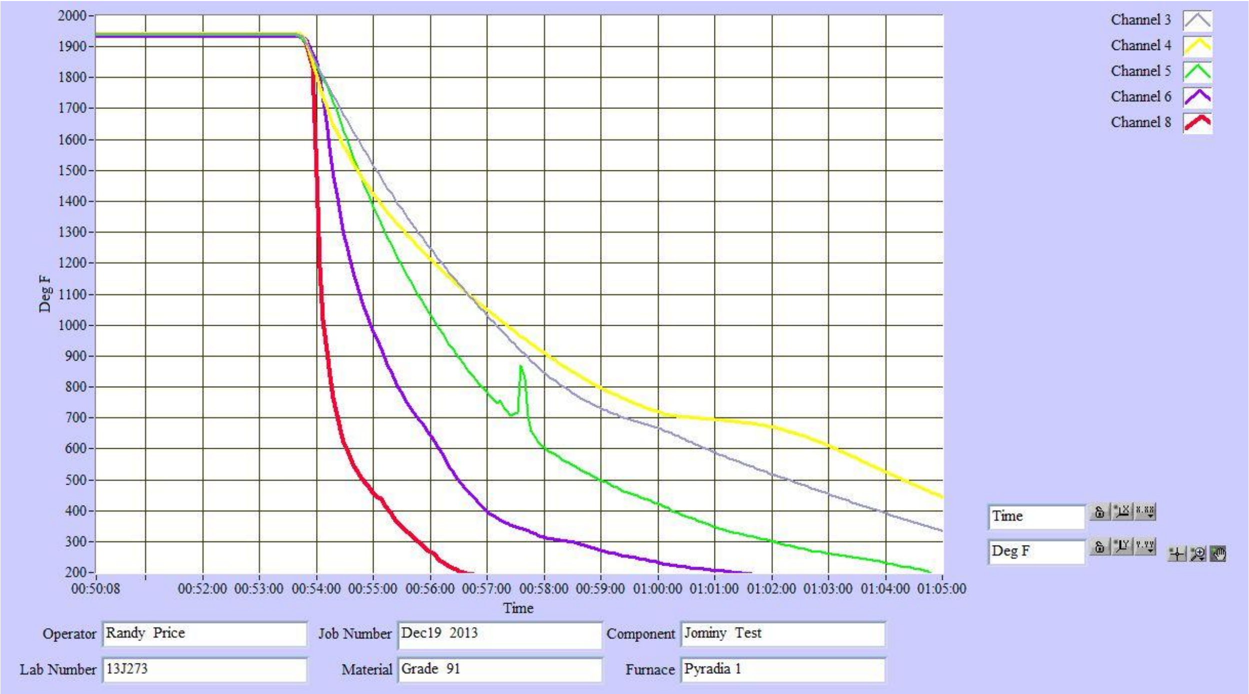
	BS46A	TV of Std	%E
C	0.143	0.139	-2.9
Mn	0.54	0.55	1.8
P	0.019	0.018	-5.6
S	0.03	0.03	0.0
Si	0.2	0.18	-11.1
Ni	0.2	0.2	0.0
Cr	2.39	2.37	-0.8
Mo	0.927	0.93	0.3
V	0.015	0.013	-15.4
Nb	0.003		
Ti	0.001		
Co	0.012	0.011	-9.1
Cu	0.14	0.134	-4.5
Al	0.027	0.022	-22.7
B	0.0002		
W	0.01		
Sb	0		
As	0.005		
Sn	0.009	0.008	-12.5
Zr	0.002		
Pb	0.006		
Fe	95.29		
N	0.015	0.014	-7.1
O	0.005		
Mg	0		
Zn	0.003		
Ta	0		
Ca	0.0004		
Bi	0		

Test Date: 11/7/13

Figure 2-6: Chemical Composition of Test Bar No. 2; Identified as "Low Heat"

2.5 Measuring the Cooling Curves

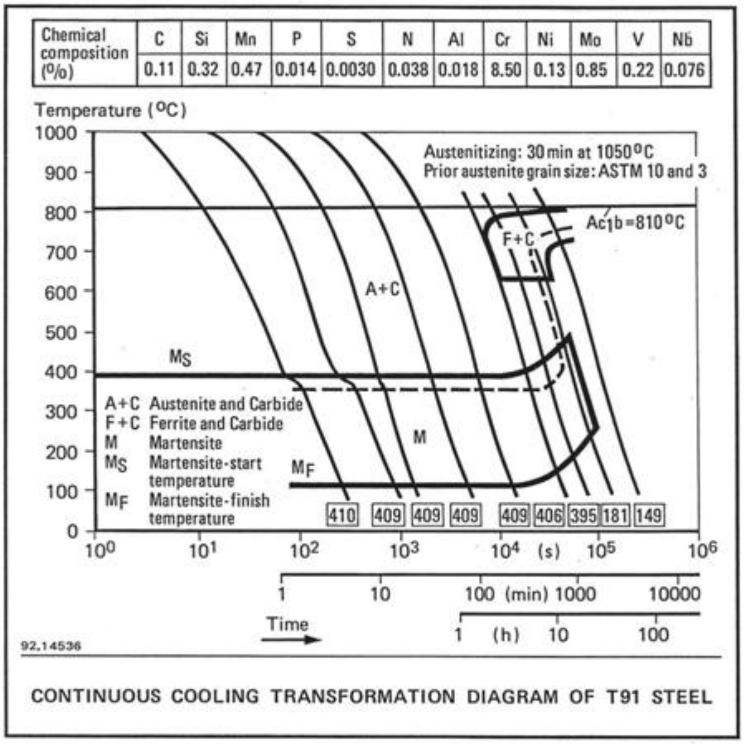
The fourth task was to measure the cooling rates at the five locations identified in the second task. The second cooling rate test is shown in Figure 2-7. There is evidence of recalescence in the 1.5 in. and 3.75 in. curves. The continuous cooling transformation (CCT) diagram obtained from Vallourec & Mannesmann [2] shown in Figure 2-8 indicates the onset of the Martensite (M_s) formation at about the same temperature (750°F). Because of the failure of one TC to function correctly, a second cooling rate test was conducted. The cooling curves from the first test (the dotted lines) are compared to those obtained from the successful second trial in Figure 2-9. The cooling curves are nearly identical, which places a great deal of credulence to their data. The cooling curves provided in both Figures 2-7 and 2-9 were used to estimate the time in seconds required to cool to a specific temperature. This data are shown in Figures 2-10 and 2-11.



Operator	Randy Price	Job Number	Dec19 2013	Component	Jominy Test
Lab Number	13J273	Material	Grade 91	Furnace	Pyradia 1

Figure 2-7: Cooling Curves for Five Thermocouples Embedded in Jominy Bars

Note: Thermocouple Locations are 0.25” (Red), 0.75” (Purple), 1.5” (Green), 2.5” (Lilac) and 3.75” (Yellow)



Chemical composition (%)	C	Si	Mn	P	S	N	Al	Cr	Ni	Mo	V	Nb
	0.11	0.32	0.47	0.014	0.0030	0.038	0.018	8.50	0.13	0.85	0.22	0.076

Figure 2-8: Grade 91 CCT Diagram [2]

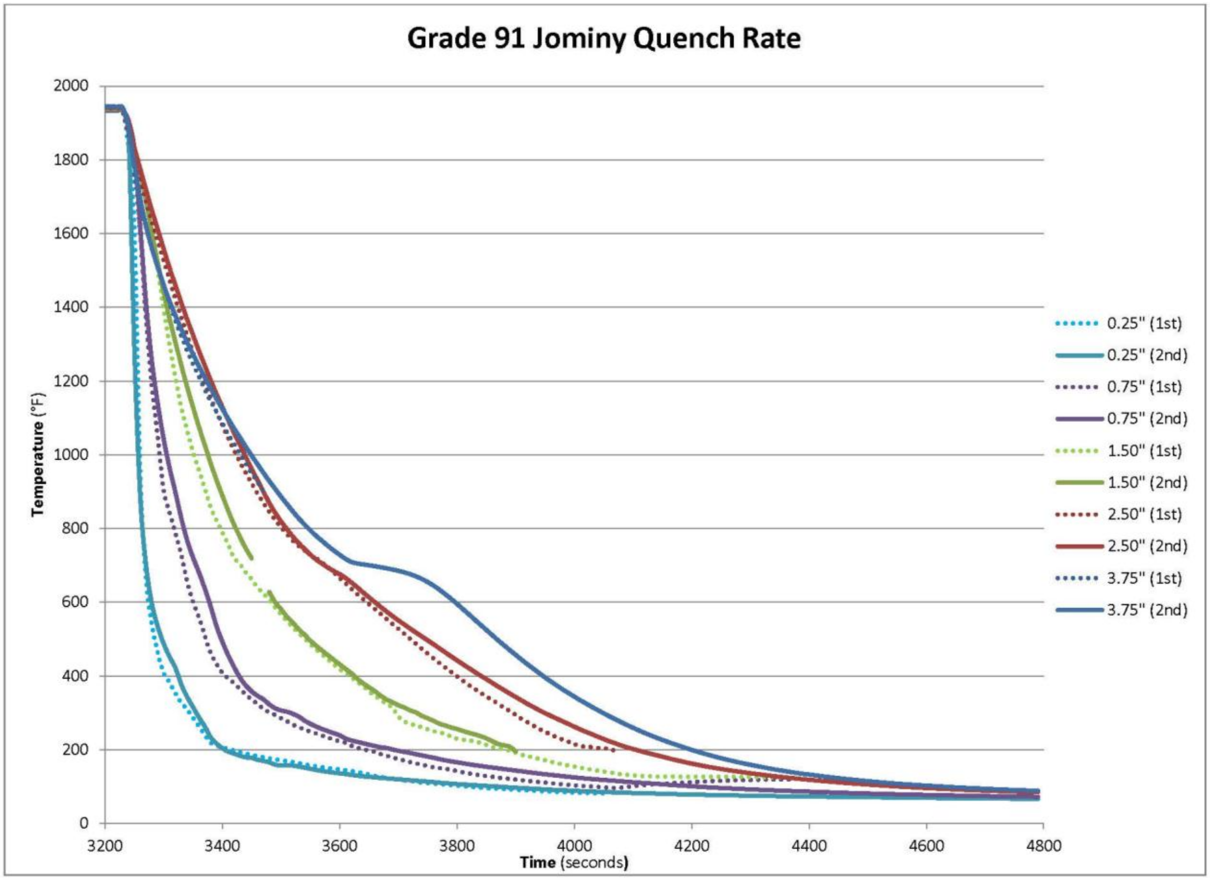


Figure 2-9: Comparison of Cooling Rates from the First Study, Which Had a Failed Thermocouple at the 3.75 in. Location and the Successful Second Test

Temperature °F	**Distance from Quenched End of Jominy Bar-inches Time to Temperature in Seconds**				
	0.25	**0.75**	**1.5**	**2.5**	**3.75**
1800	10	18	20	22	20
1600	20	25	50	58	42
1400	22	40	70	93	80
1200	25	50	90	140	135
1000	30	62	120	195	200
800	34	87	165	270	
600	42	120	255	420	
400	70	175	385	570	
200	180	400	640	800	

Figure 2-10: 1st Run Austenitized at 1940ºF

Note: Cooling rates; time (seconds) to reach specific temperature (°F) for the first cooling rate trial *above* and the second cooling rate trial *below*.

Temperature °F	**Distance from Quenched End of Jominy Bar-inches Time to Temperature in Seconds**				
	0.25	**0.75**	**1.5**	**2.5**	**3.75**
1800	12	20	20	25	27
1600	18	25	40	42	60
1400	20	40	80	77	100
1200	22	45	105	140	150
1000	23	78	142	215	220
800	35	100	193	280	320
600	44	145	260	425	560
400	95	200	400	600	720
200	170	440	660	860	970

Figure 2-11: 2nd Run Austenitized at 1940ºF

Note: The second cooling rate trial was conducted due to the loss of the thermocouple at the 3.75 inch location.

2.6 Continuing Jominy Tests on Two Bars from Each Heat

The fifth task was to conduct Jominy tests on two bars from each heat. The hardness tests on the first bar from each heat were to be conducted on the as-cooled bar (Austenitized at 1940°F and quenched). The second bar was austenitized at 1940°F, end-quenched in the conventional manner, and then tempered at 1410°F. The results of the hardness tests for both the as-quenched and quenched and tempered bars are shown in Figure 2-12. The slightly higher carbon content (0.102 Wt %) in the Test Bars No.1 heat (Vs the 0.092% C) in Test Bar No.2 heat is evident in the higher hardness values observed in Test Bar No.1 heat.

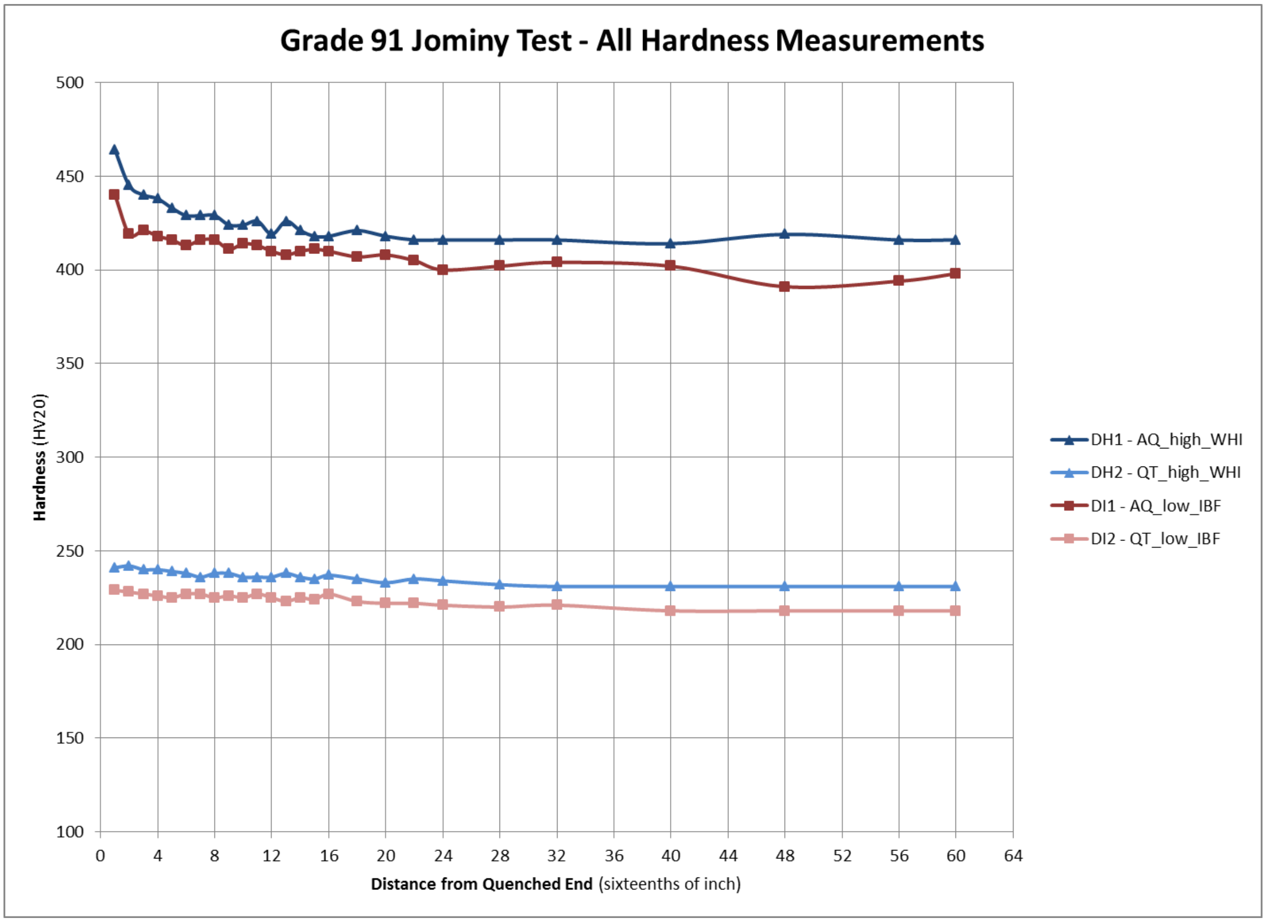


Figure 2-12: Jominy Hardness Data for High Heat Test Bars 1-2 & 1-3 (WHI) and Low Heats Test Bars 2-2 & 2-3 (IBF)

2.7 Preparing Photomicrographs at Five Locations

The sixth task was to prepare photomicrographs at the five locations where the TCs were imbedded in the Jominy bars. The photomicrographs were taken at the 0.25”, 0.75”, 1.5”, 2.5” and 3.75” locations in the Jominy bars. The photomicrographs are shown in Figures 13 through 2-22. Each Figure contains a 100X and a 500X magnification of the as-quenched Jominy bar and the quenched and tempered bar.

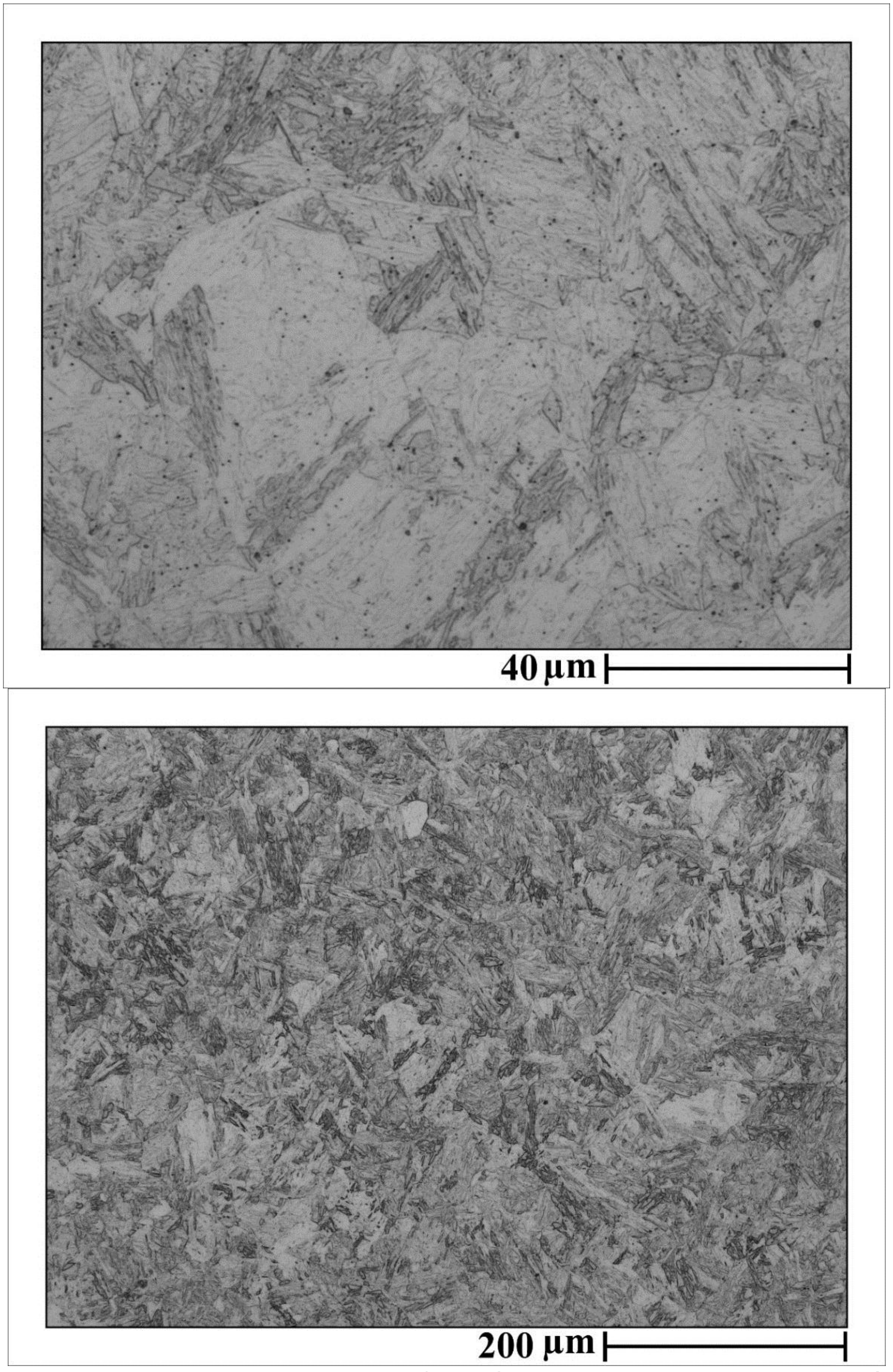


Figure 2-13: Microstructure at 0.25 in. from Quenched End; Austenitized (1940°F) and Cooled

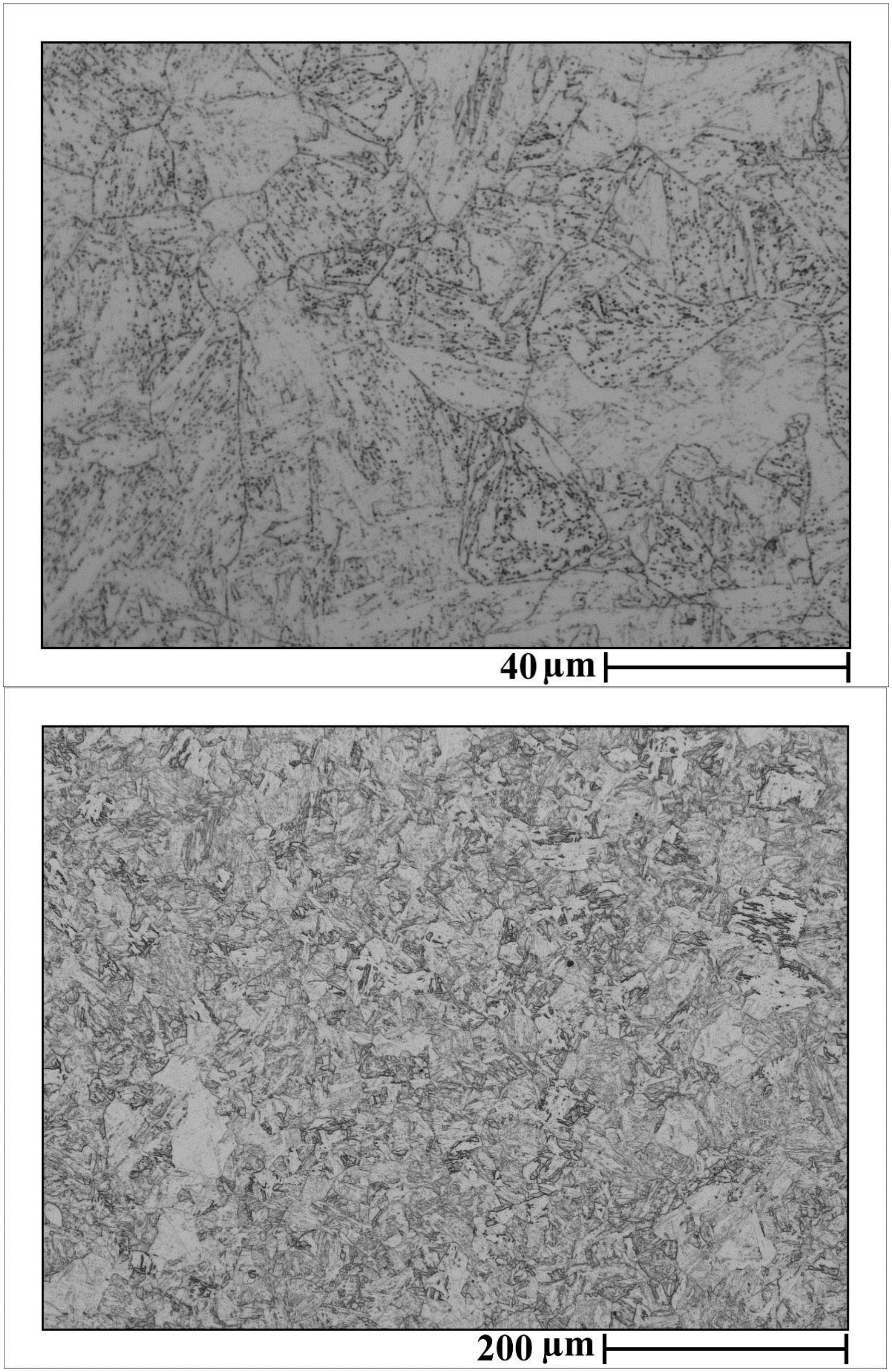


Figure 2-14: Microstructure at 0.25 in. from Quenched End; Austenitized (1940°F) and Tempered (1410°F)

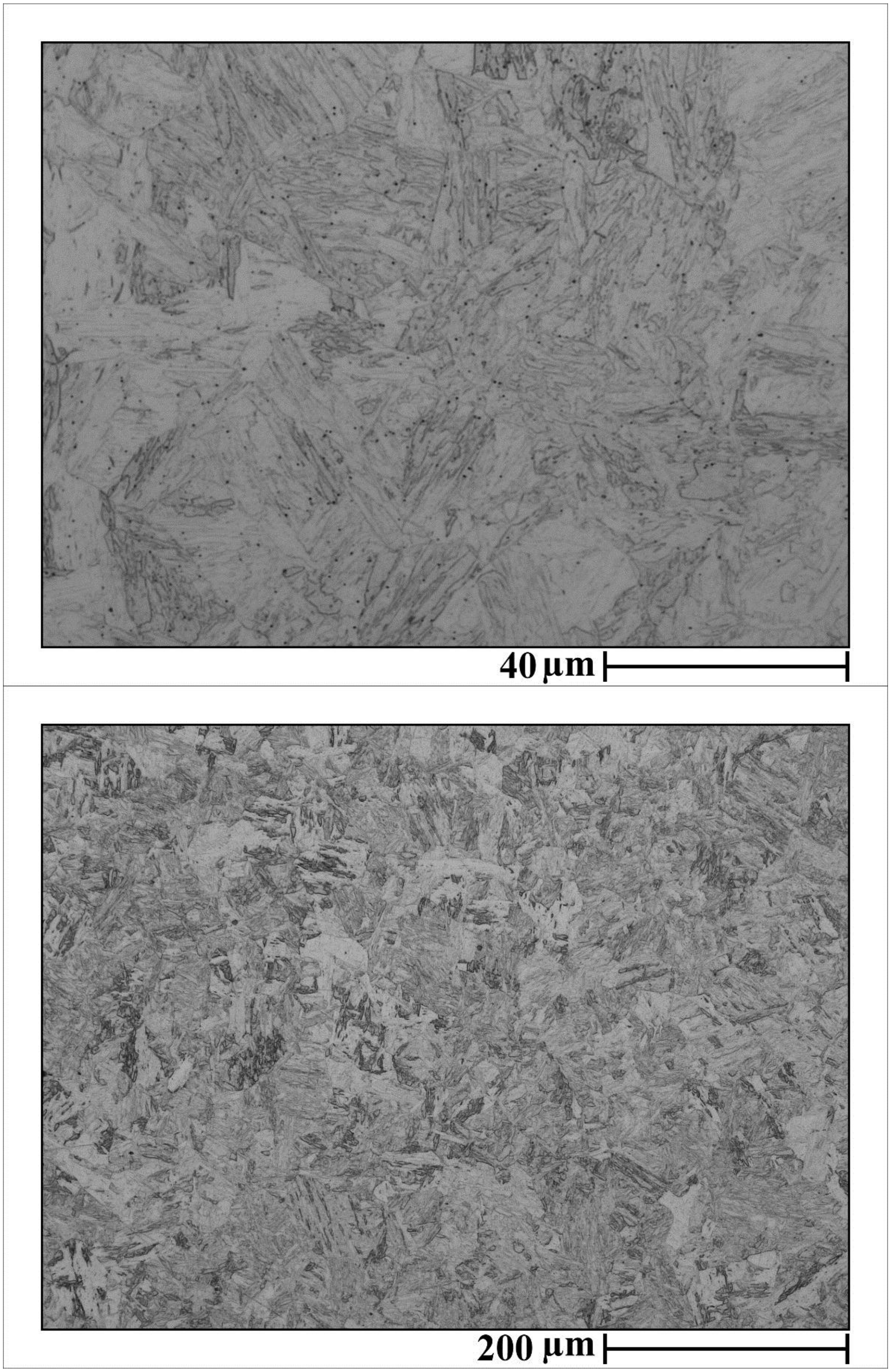


Figure 2-15: Microstructure at 0.75 in. from Quenched End; Austenitized (1940°F) and Cooled

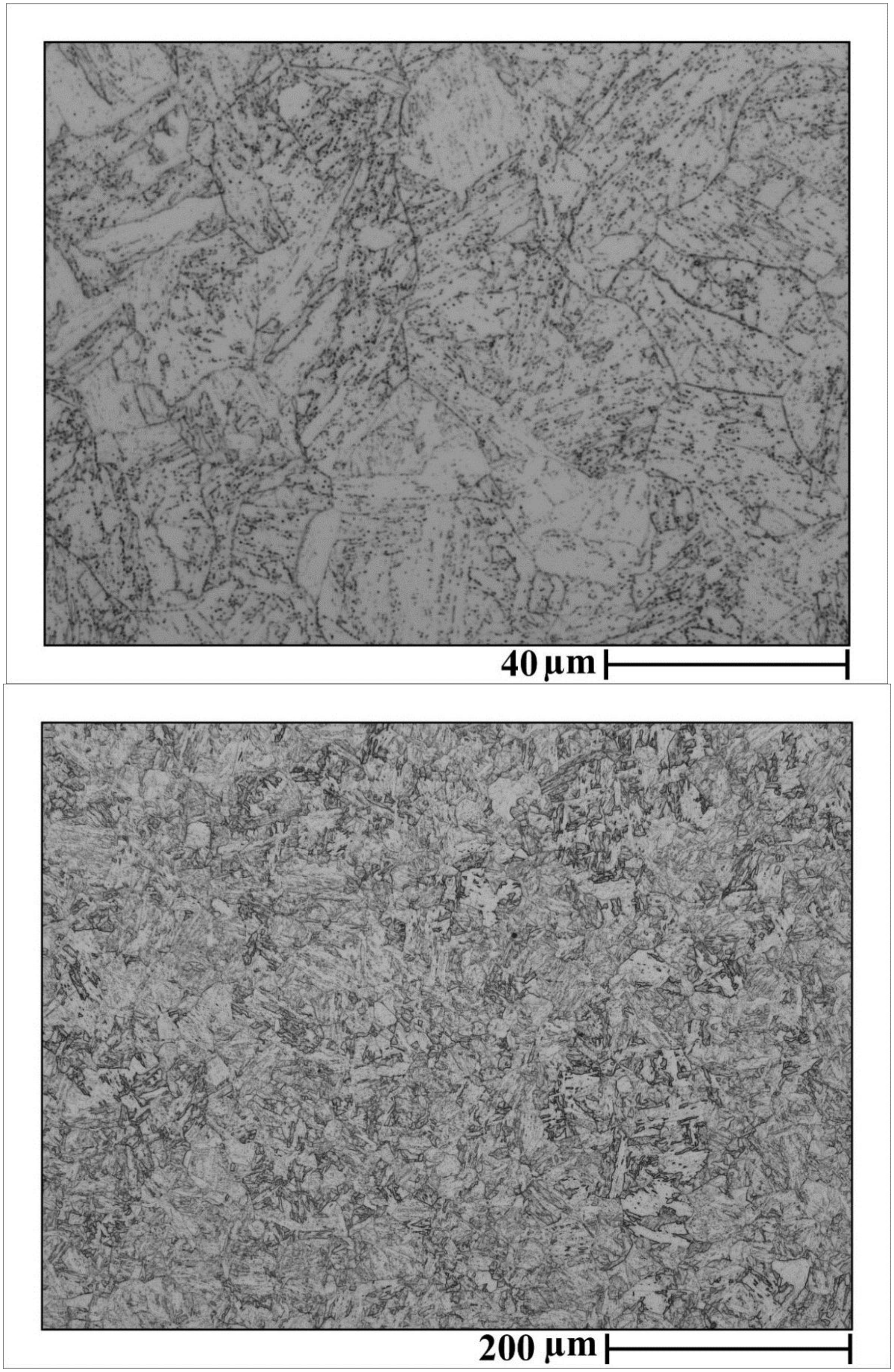


Figure 2-16: Microstructure at 0.75 in. from Quenched End; Austenitized

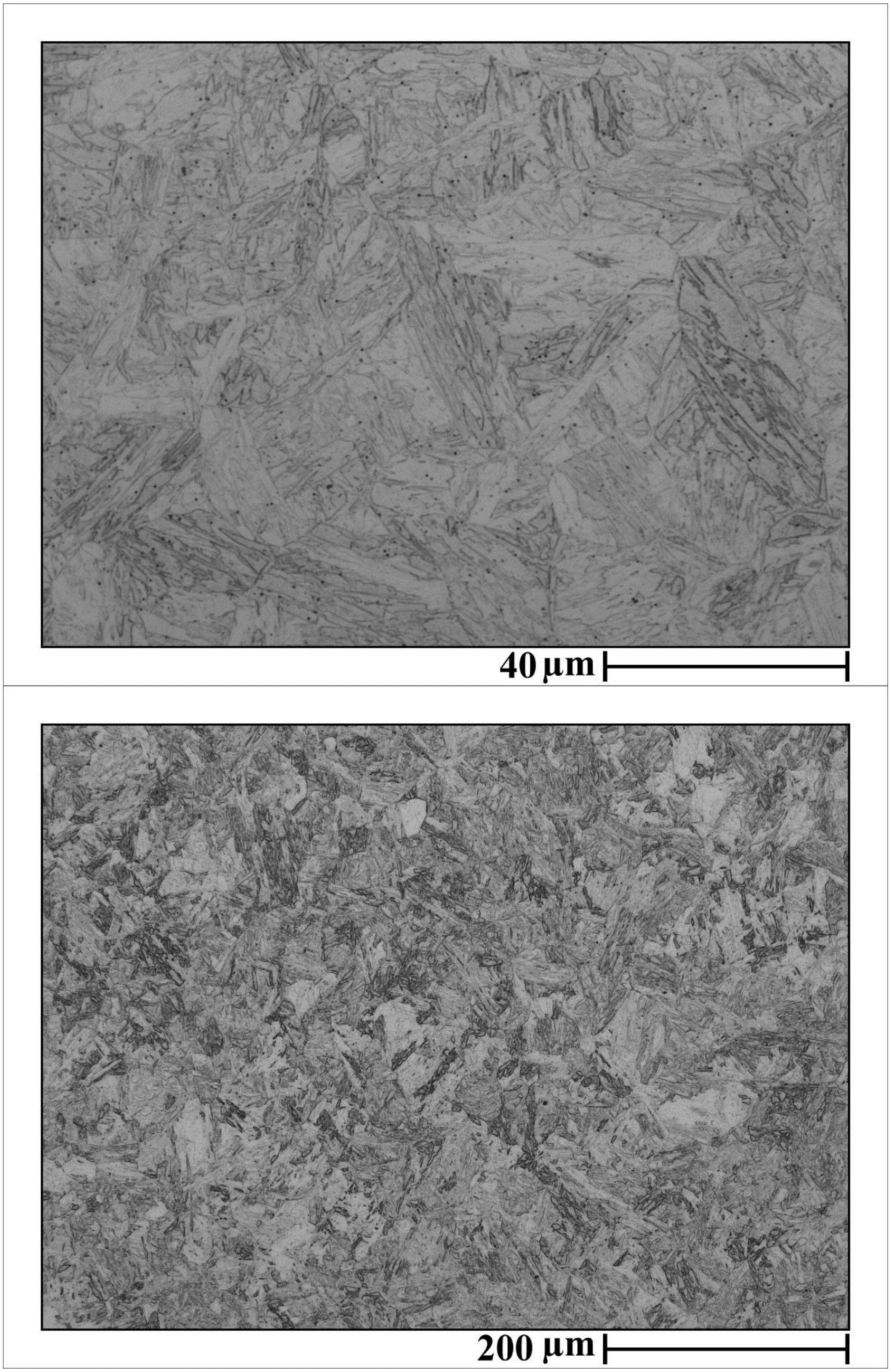


Figure 2-17: Microstructure at 1.5 in. from Quenched End; Austenitized (1940°F) and Cooled

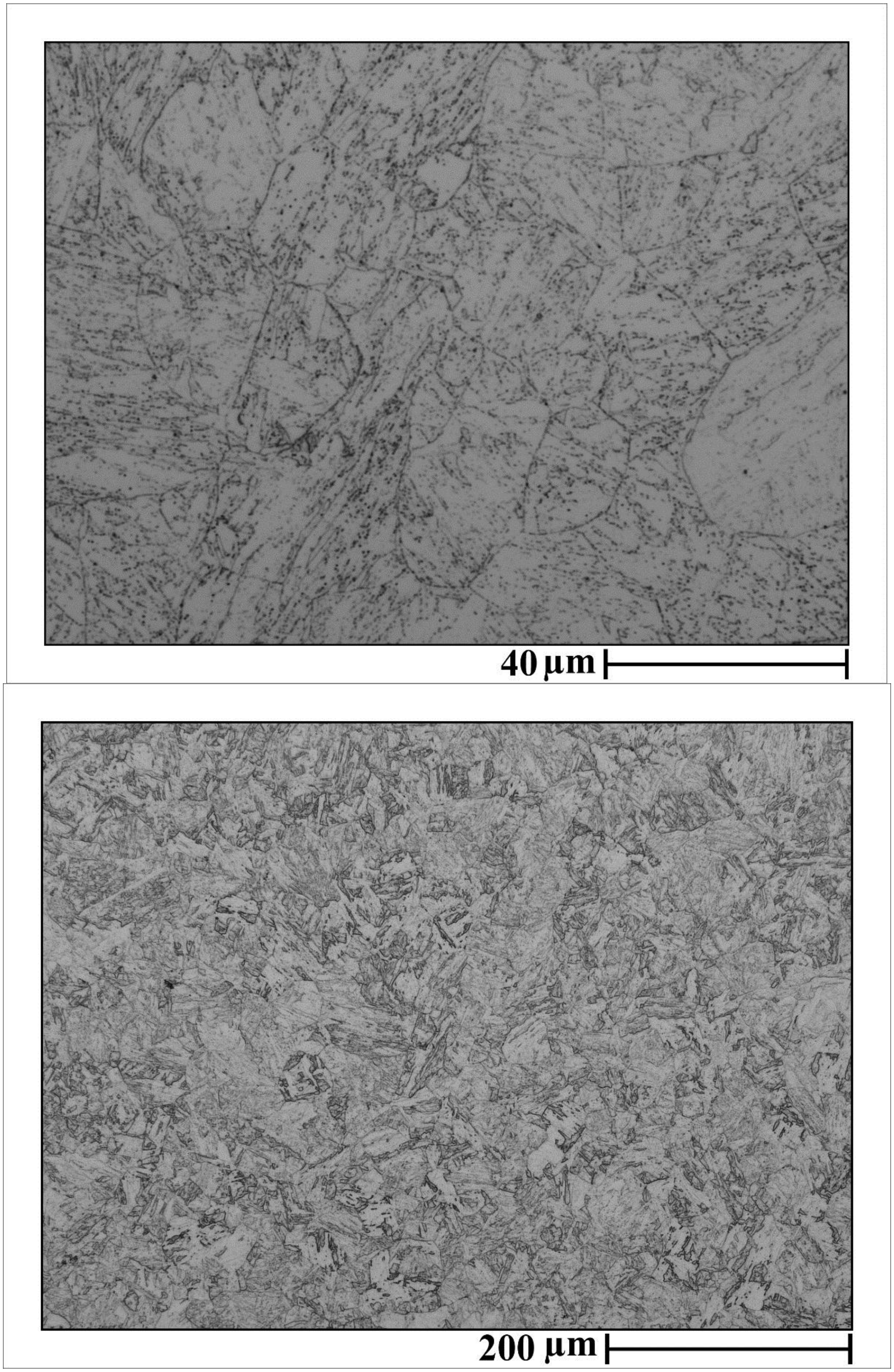


Figure 2-18: Microstructure at 1.5 in. from Quenched End; Austenitized (1940°F) and Tempered (1410°F)

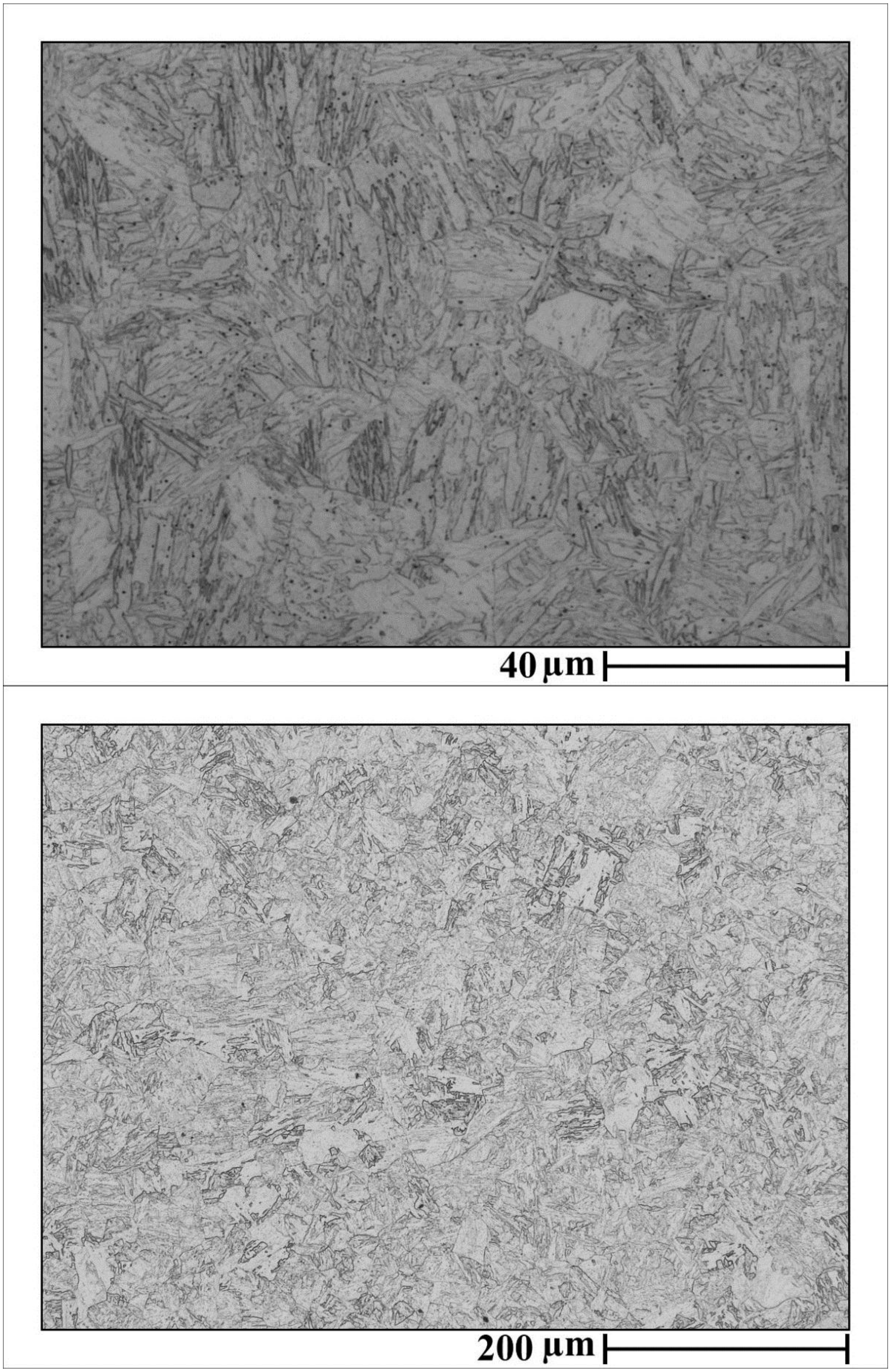


Figure 2-19: Microstructure at 2.5 in. from Quenched End; Austenitized (1940°F) and Cooled

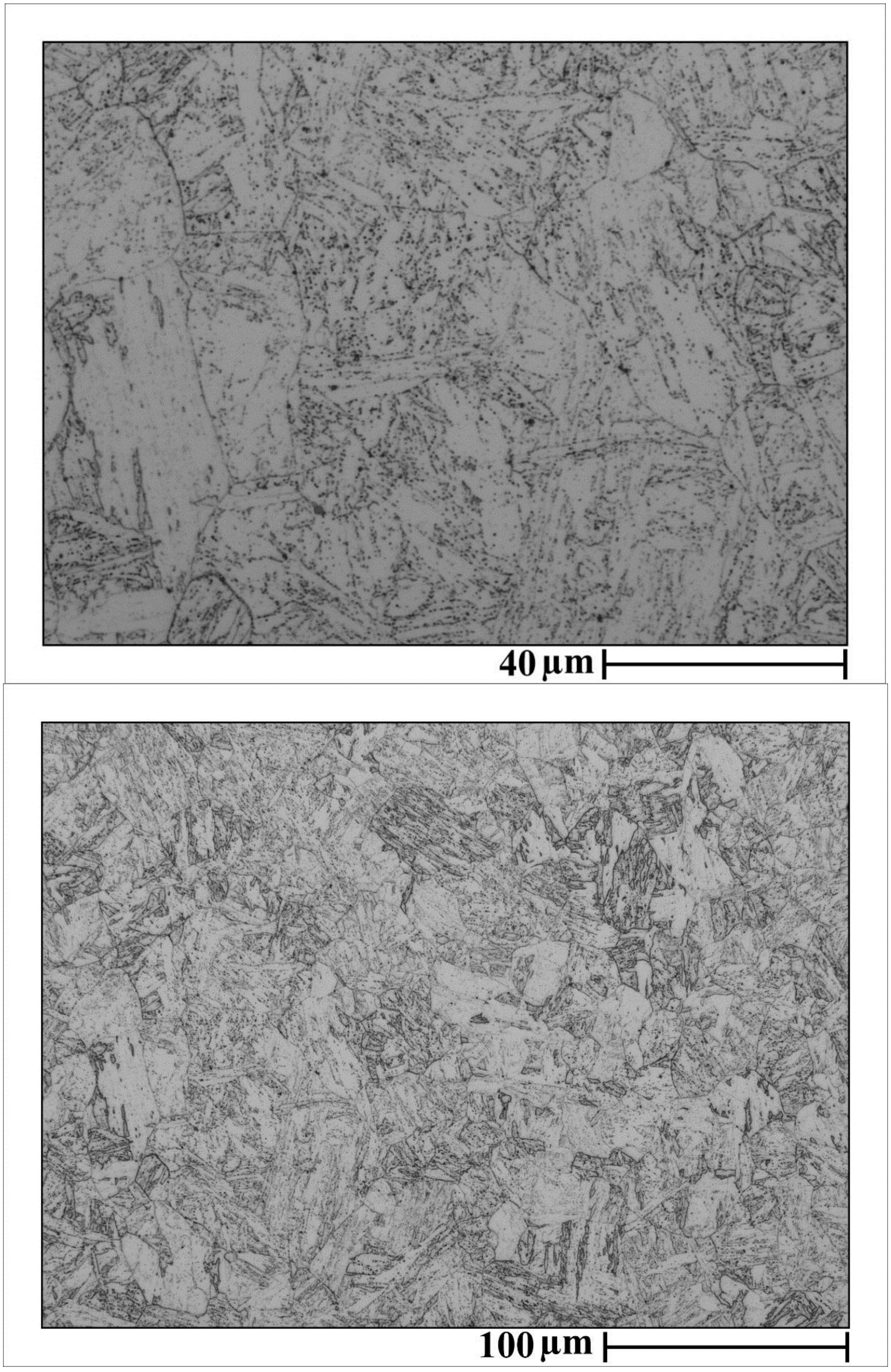


Figure 2-20: Microstructure at 2.5 in. from Quenched End; Austenitized (1940°F) and Tempered (1410°F)

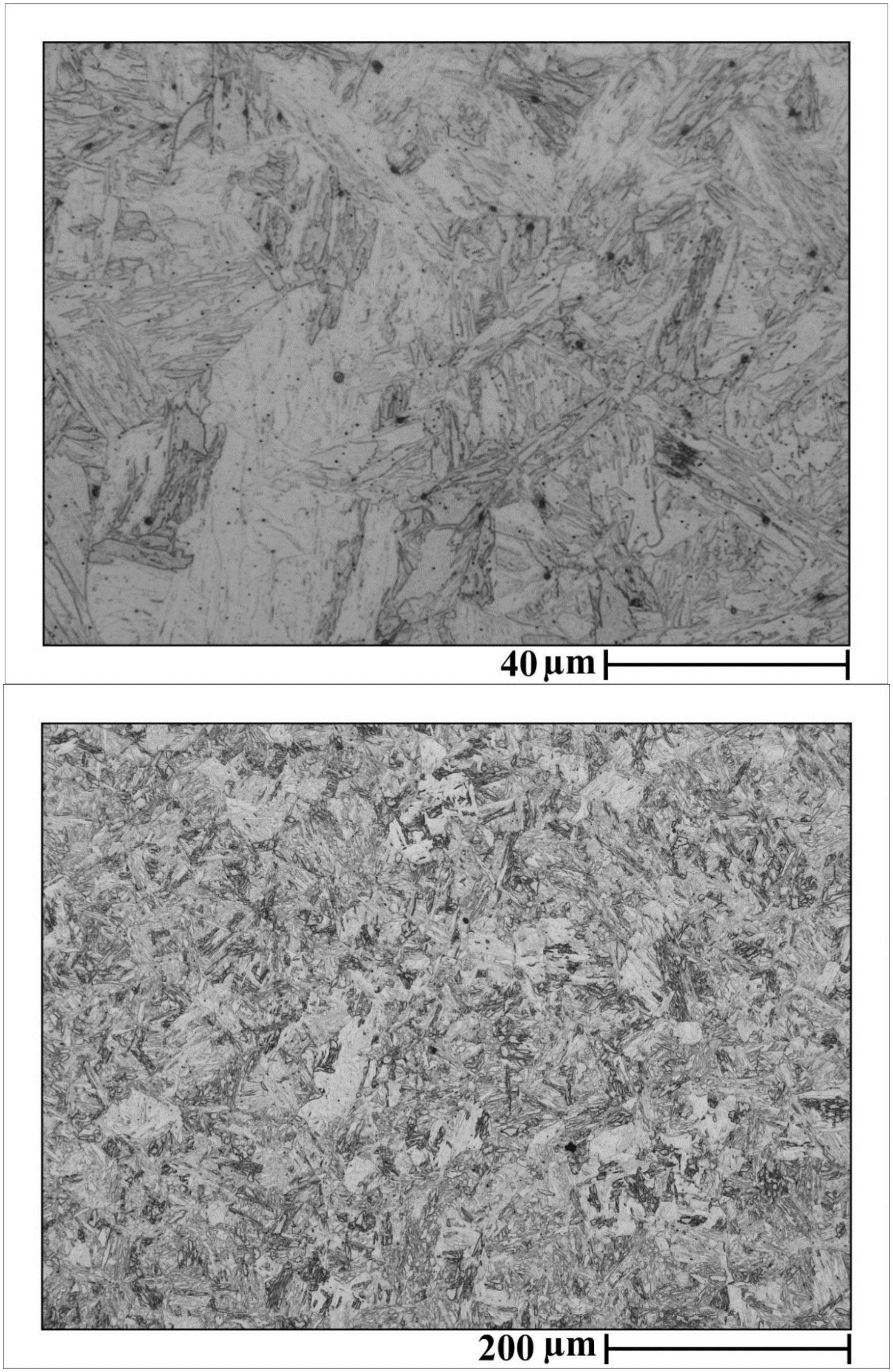


Figure 2-21: Microstructure at 3.75 in. from Quenched End; Austenitized (1940°F) and Cooled

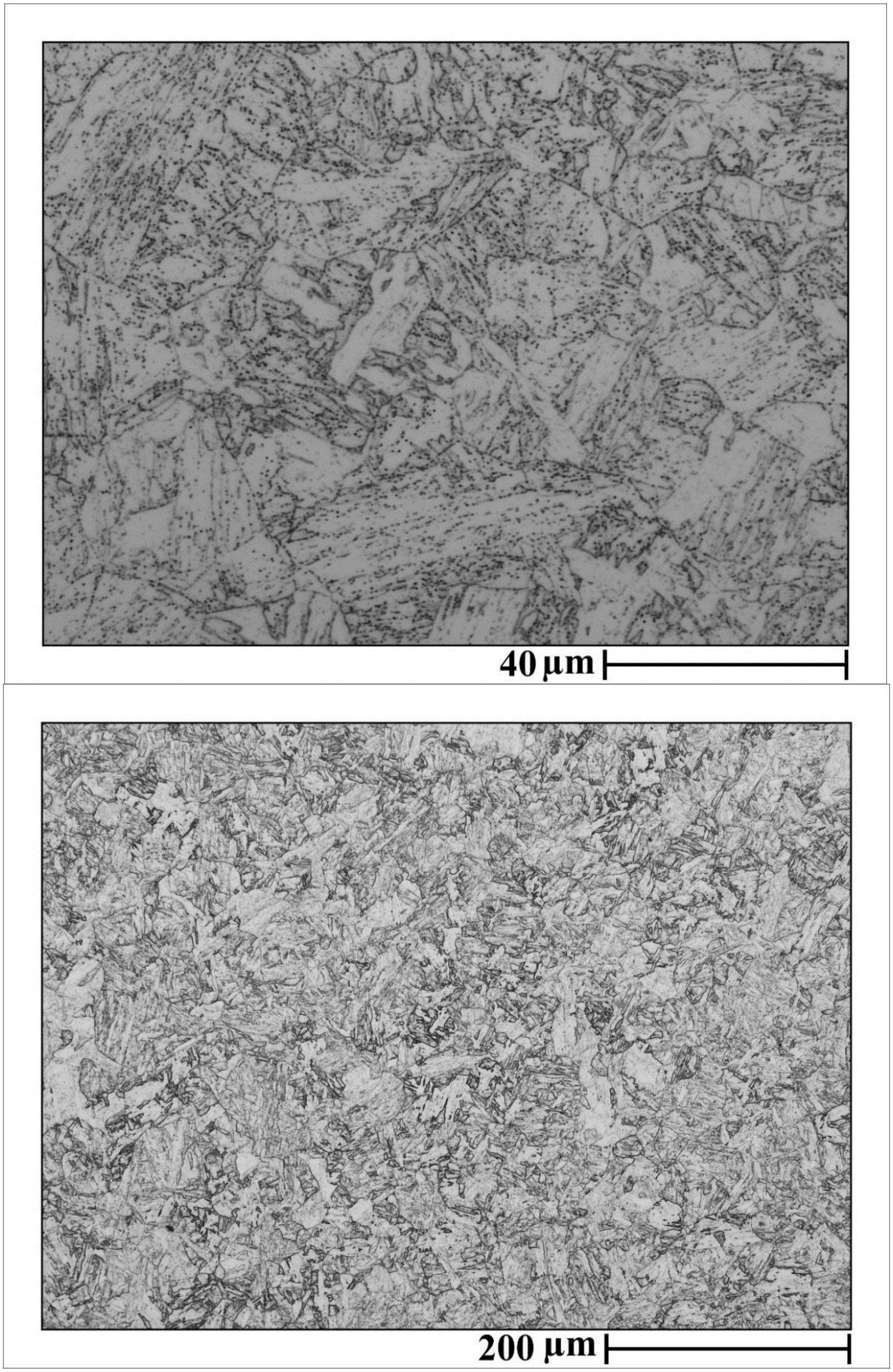


Figure 2-22: Microstructure at 3.75 in. from Quenched End; Austenitized (1940°F) and Tempered (1410°F)

3 DISCUSSION OF THE RESULTS

In 1980, while the Modified 9 Cr-1 Mo (Grade 91) was being optimized, Oak Ridge National Laboratory (ORNL) conducted Jominy bar tests that permitted a comparison between $2^1/_4$ Cr-1 Mo and Grade 91 steel. These results are shown in Figure 3-1. The results of that study, which are shown in Figure 3-1, were included in the data package submitted to the Section I Committee for the ASME Boiler and Pressure Vessel Code (BPVC) in June 1982, when ORNL requested a Code Case for Grade 91. The cooling rates for those Jominys were not measured, nor were any metallographic studies made of those samples. Those studies proved the extreme hardenability of the Grade 91 alloy.

The Grade 91 CCT diagram shown in Figure 3-1 is quite similar to that presented for Grade 9 in the Atlas of Continuous Cooling Transformation Diagrams for Engineering Steels [3]. This CCT diagram is shown in Figure 3-2. It suggests that the onset of transformation to a microstructure other than martensite will occur at an overall cooling rate of about 0.8 °F per second. (Austenitizing temperature is 1832°F the temperature cooling range to 200°F is 1632 degrees); this assumes that the change from a horizontal straight line to an inflection is an indication of transformation to a product other than martensite. It was initially assumed that that cooling rate was possible at the far end of a Grade 91 Jominy bar which is four inches from the quenched end and is essentially air-cooled, which did not prove to be true. The cooling curves shown in Figure 2-7 show a rate from 1200°F to 1000°F (i.e. the cooling rate at 1100°F) of about 4°F per second; a rate much faster than the 0.2 F per second shown in Figure 2-8. The overall cooling rate indicated in Figure 2-8 is near 0.02°F per second versus the near 3°F per second in Figure 2-7.

Also, of interest is the blip in the cooling curves at approximately 700°F, which relates fairly well with the onset of the martensite transformation in Figure 2-8. It is believed this blip is due to recalesence when the transformation to martensite occurs. The results of both cooling curves are presented in Figure 2-9. They are, for all practical purposes, identical. The similarity is also apparent in the cooling data presented in Figures 2-10 and 2-11.

The two grade 91 heats are sufficiently different (see Figures 2-5 and 2-6) in that they provide individual hardness curves as is evident in Figure 2-12. The carbon difference, 0.01%, is reflected in both the as-quenched Jominy bars and the quenched and tempered bars. The conversion of the as-quenched Vickers (HV) values for the Test Bar 1-2 (WHI) heat is 48 Rockwell C (RC) and the Test Bar 2-2 (IBF) heat is about 45 RC. The difference is the same for the tempered hardness readings, 22 RC and 20 RC (Test Bar 1-3 and Test Bar 2-3), respectively).

In reviewing all of the photomicrographs from the five locations on the Jominy bars, Figures 2-13 through 2-22, there is not a discernible difference from one photomicrograph to the other along any one bar. All as-quenched photomicrographs show 100% martensite, and all quenched and tempered photomicrographs show 100% tempered martensite.

In conclusion, the project did not provide the ability to delineate the onset of transformation to a microstructure other than martensite. It was known that the Grade 91 steel is extremely hardenable. The cooling rate at the essentially air cooled end of the Grade 91 Jominy bar was not sufficiently slow to result in the onset of a microstructure other than martensite. This study did provide baseline cooling rate data from which slower cooling rates can be suggested. If a Datatrak, or a similar device, can be located it would be possible to simulate slower cooling rates, those that simulate thicker section sizes. Those specimens would allow the opportunity to obtain samples from which metallographic studies could be obtained and samples from which tensile bars could be machined. This would allow the development of a correlation between microstructure and strength and other studies such as the influence of bainite and ferrite on the stress rupture properties of Grade 91.

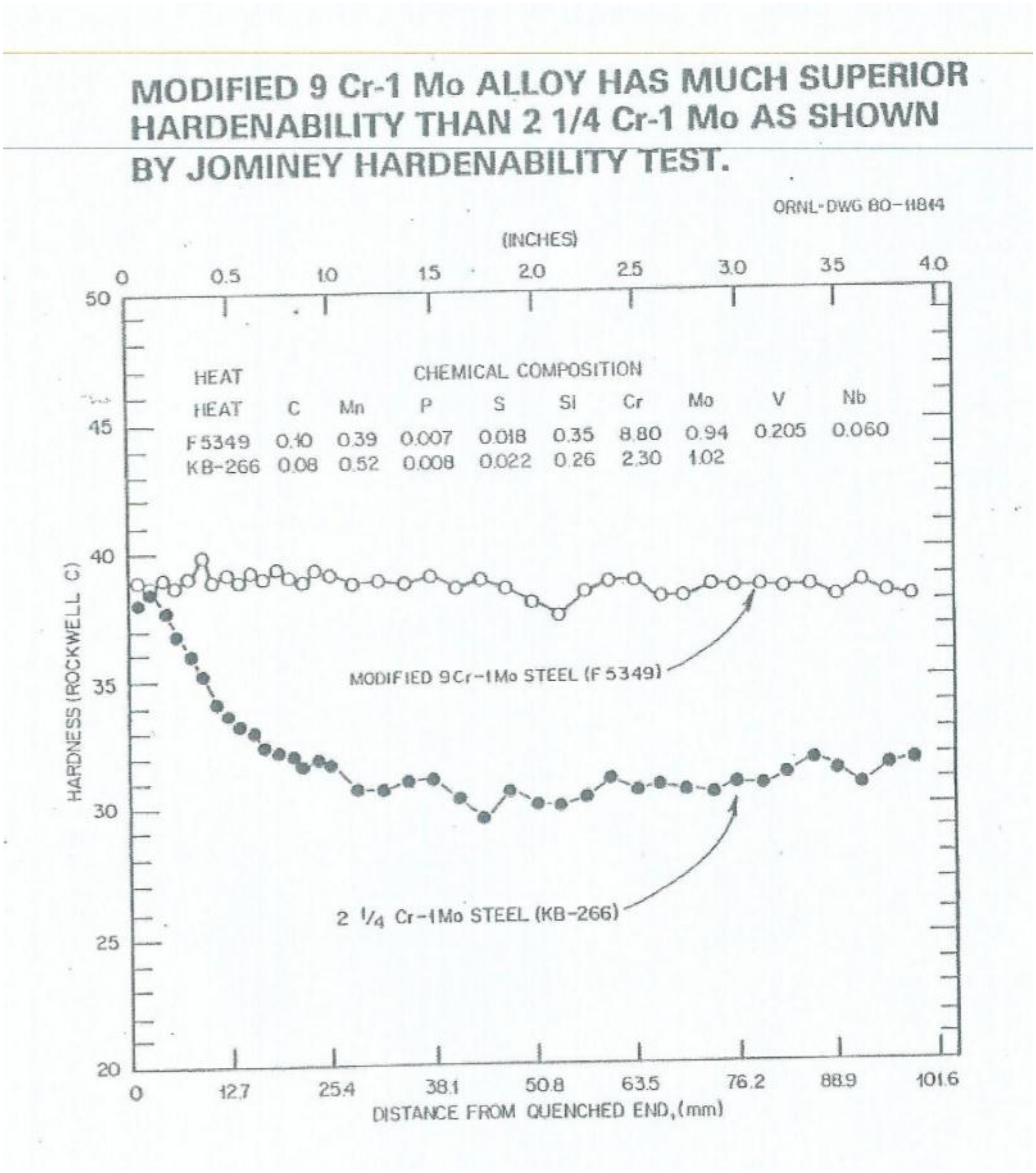


Figure 3-1: Oak Ridge National Laboratory Jominy Test, Circa 1980 [4]

The ORNL Jominy study (Figure 3-1) did not provide cooling rate data or microstructural information. From that point of view this project did deliver a great deal of useful information regarding the transformation of Grade 91 austenite to a transformation product other than martensite. As a minimum this study does provide data that show that the cooling rate must be slower than that which is possible with a standard Jominy bar. The V&M CCT [2] suggests that it is necessary to cool Grade 91 at an overall rate slower than 0.02°F per second or a rate at 1100 °F of 0.4°F per second to obtain a transformation product other than martensite. Those rates are considerably slower than what can be achieved with a standard Grade 91 Jominy bar.

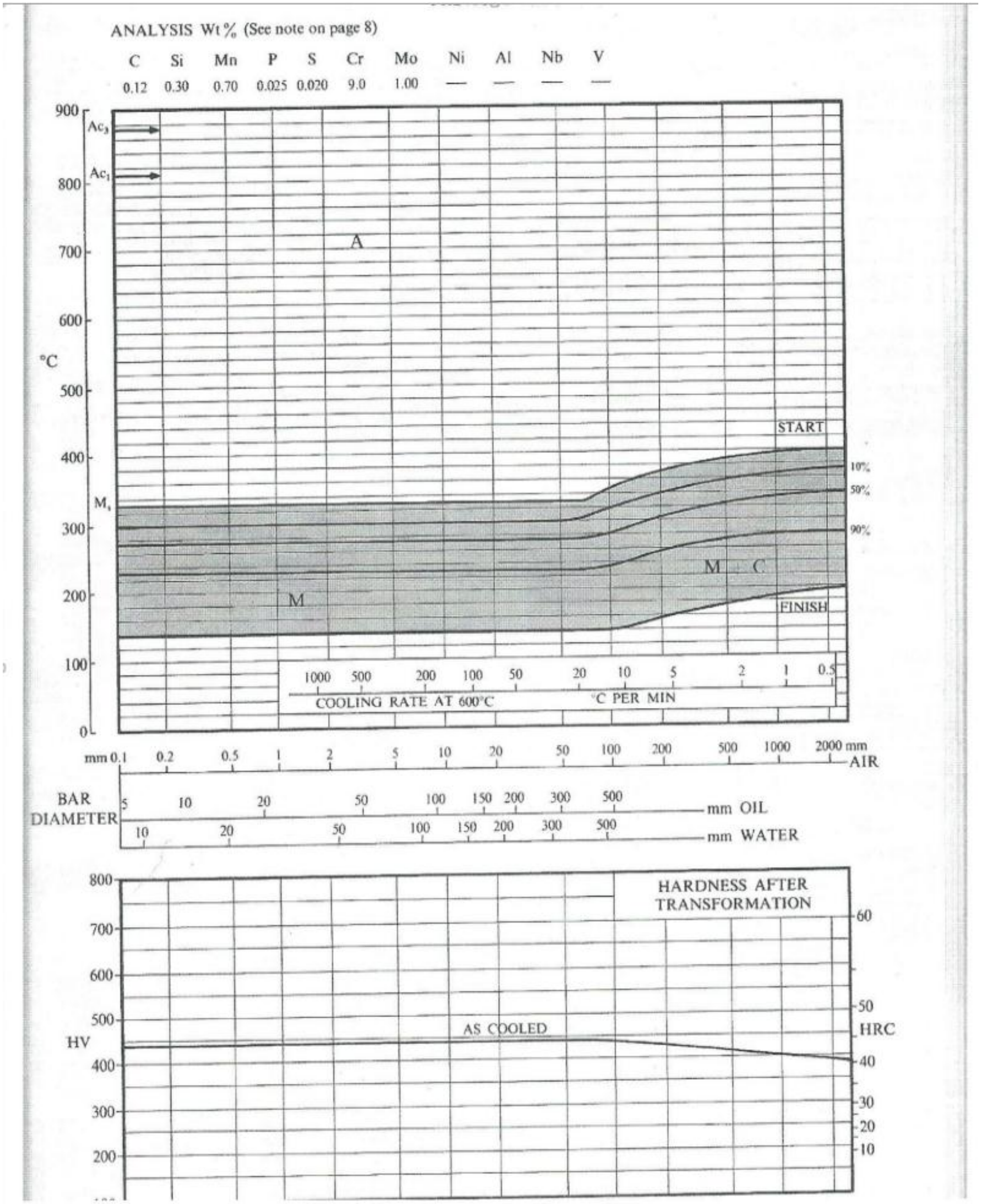


Figure 3-2: Continuous Cooling Transformation Diagram for 9 Cr – 1 Mo: Austenitized at 1832°F

REFERENCES

[1] ASTM A255-10 Standard Test Methods for Determining Hardenability of Steel
[2] Vallourec & Mannesmann Tubes “The T91/P91 Book” 1999
[3] Atlas of Continuous Cooling Transformation Diagrams for Engineering Steels, Atkins M., American Society for Metal, 1980
[4] Oak Ridge National Laboratory Jominy Test, circa 1980

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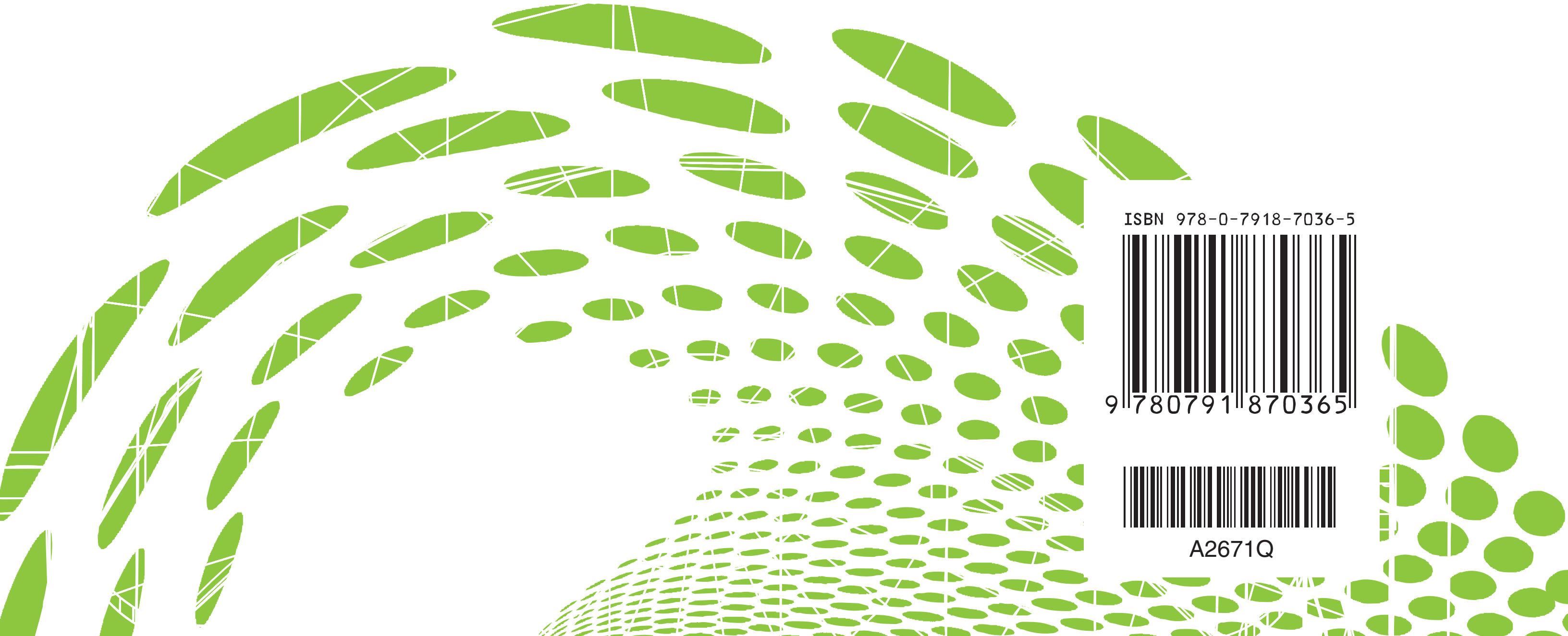

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