

3 4456 0002737 0

ORNL/TM-9691

OAK RIDGE
NATIONAL
LABORATORY

MARTIN MARIETTA

**Metallographic Examination of
Response of 3 Cr-1.5 Mo Steel
to Heat Treatment**

R. W. Swindeman
A. M. Nasreldin
R. L. Klueh
J. F. King

*Research supported by the
Morgantown Energy Technology Center,
U.S. Department of Energy*

OAK RIDGE NATIONAL LABORATORY
CENTRAL RESEARCH LIBRARY
CIRCULATION SECTION
ADMIN. ROOM 123
LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to use this
report, send in name with report and
the library will arrange a loan.

*Fossil
Energy
Program*

OPERATED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Printed in the United States of America. Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road, Springfield, Virginia 22161
NTIS price codes—Printed Copy: A03 Microfiche A01

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would *not* infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

METALS AND CERAMICS DIVISION

METALLOGRAPHIC EXAMINATION OF RESPONSE OF 3 Cr-1.5 Mo STEEL
TO HEAT TREATMENT

R. W. Swindeman, A. M. Nasreldin, R. L. Klueh, and J. F. King

NOTICE: This document contains information of
a preliminary nature. It is subject to revision
or correction and therefore does not represent
a final report.

Date Published — November 1985

*Research supported by the
Morgantown Energy Technology Center,
U.S. Department of Energy*

Prepared by the
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under Contract No. DE-AC05-84OR21400



3 4456 0002737 C

CONTENTS

ABSTRACT	1
INTRODUCTION	1
MATERIAL AND PROCESSING	3
2.25 Cr-1 Mo STEEL	8
3 Cr-1.5 Mo STEEL	11
METALLOGRAPHIC EXAMINATION OF WELDMENTS	20
SCANNING ELECTRON MICROSCOPY	26
DISCUSSION AND SUMMARY	26
ACKNOWLEDGMENTS	29
REFERENCES	29

METALLOGRAPHIC EXAMINATION OF RESPONSE OF 3 Cr-1.5 Mo STEEL
TO HEAT TREATMENT*

R. W. Swindeman, A. M. Nasreldin,[†] R. L. Klueh,
and J. F. King

ABSTRACT

The influence of austenitizing temperature, cooling rate, and tempering conditions on the microstructure of 3 Cr-1.5 Mo steels was examined in order to establish the heat treatment needed to produce fine-grained bainitic microstructures. Most of the work was performed on samples from an 8-ton commercial heat of a composition developed by the Climax Molybdenum Company that contained a manganese addition for hardenability and a vanadium addition for strength. This material was examined in both the normalized-and-tempered and the quenched-and-tempered conditions. The material was found to be fully bainitic in both the coarse- and fine-grained conditions. Additional studies were performed on another 8-ton commercial heat of a composition developed by the University of California, Berkeley, that contained more nickel and less manganese. This material also possessed adequate hardenability to produce bainitic microstructures for cooling conditions that would result in some proeutectoid ferrite in standard 2.25 Cr-1 Mo steel. Both compositions developed the microstructures and concomitant strength and toughness needed to be competitive with other developing microalloyed steels for use in hydrogen service.

INTRODUCTION

This interim report provides the results of metallographic studies performed on small commercial heats of 3 Cr-1.5 Mo steels that are candidate alloys for use in the construction of gasifier pressure vessels for service to 316°C (600°F). This class of steels is expected to have advantages over the 2.25 Cr-1 Mo steel (SA-387 grade 22 class 2) and other gasifier pressure vessel steels in the following ways: (1) better hardenability, resulting in good strength and excellent toughness; (2) more

*Research supported by the U.S. Department of Energy, Morgantown Energy Technology Center, Surface Gasification Materials Program, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

[†]Now with the Atomic Energy Establishment, Cairo, Egypt.

sluggish response to tempering, allowing a larger tempering window and more flexible heat-treating schedules; and (3) improved resistance to hydrogen attack at high strength levels with no significant tendency toward temper or hydrogen embrittlement. These expectations were based primarily on the results of evaluations of small laboratory heats at the Climax Molybdenum Company by Wada and coworkers^{1,2} and at the University of California, Berkeley, by Spencer and coworkers.^{3,4} The ability to scale the laboratory compositions to commercial heat sizes is a necessary step in the development of any new alloy, and reports^{5,6} describing this need have been prepared through the support of the U.S. Department of Energy METC Surface Gasification Materials Program.

The hardenability of low-alloy steels is known to be a function of composition and heat treatment.⁷ The addition of elements such as nickel and manganese suppresses the formation of proeutectoid ferrite during cooling from the austenitizing temperature and promotes the formation of a tough, bainitic microstructure. When present in high concentrations, however, these elements can present problems by increasing the tendency for martensite formation that can lead to cracking in weldments or increased tendency toward temper embrittlement. Partly to prevent such problems, the concentrations of these hardenability-promoting elements have been kept to a minimum. The grain size is also a factor that must be optimized. Strength and toughness are expected to increase with decreasing grain size, but proeutectoid ferrite, which is associated with lower strength and loss of toughness, tends to nucleate at the prior austenite grain boundaries. To prevent such ferrite microstructures very fine grain sizes should be avoided, and it is necessary to settle on a compromise for optimum properties.⁷ Some of the work reported here included a determination of the influence of annealing temperature on grain size.

Another consideration in developing specifications for heat treating schedules is the cooling rate from the austenitizing temperature. This can be important, even for alloys that do not develop proeutectoid ferrite upon cooling at rates typical of the center section of thick plates. Depending on the degree of hardenability, transformation products may be

subtle modifications of bainite or martensite. The coarseness of the lath structure in these constituents affects the upper shelf impact energies, and the presence of retained austenite in the martensitic microstructure can influence the transformation products during tempering.^{8,9} Detailed studies are under way, both in this activity and elsewhere, to examine the microstructural changes during the tempering.^{9,10} In this report, however, only the relatively coarse changes will be described. More detailed work, involving transmission electron microscopy of thin foil samples and extraction replicas, will be described at a later date.

Efforts to develop an understanding of the elemental constituents in the precipitates as a function of composition and tempering conditions have been undertaken by a number of researchers,^{10,11} and the recent work of Shaw has been a major contribution to understanding the physical metallurgy of these Cr-Mo alloys. The compositions examined by Shaw cover the steels described here; hence, only a modest effort was needed to confirm the findings for the commercial heats of 3 Cr-1.5 Mo steels. This work is close to being completed, and results will be provided in a separate report.

MATERIAL AND PROCESSING

The bulk of the work reported here was performed on an 8-ton commercial heat (Lukens heat A9349) of an alloy developed by Wada and coworkers,^{1,2} melted by Carpenter Technology, and processed by Lukens Steel Company. The composition is provided in Table 1. Major alloying elements consisted of 3% chromium for hardenability and resistance to hydrogen, 1.5% molybdenum for added strength, manganese and nickel totaling approximately 1% as the primary additives for hardenability, and 0.1% vanadium for strength. The carbon was selected at 0.1% to avoid potential cracking problems with welding that sometimes develop in vanadium-bearing steels. As indicated in the table the achieved composition came close to the aim. The heat was processed into 100-mm plate, which is thick enough to permit an evaluation of the thick-section weldability. The first lot of material was delivered in the nominally normalized-and-tempered

Table 1. Chemical composition of steels evaluated in study

Material	Content (wt %)													
	C	Mn	P	S	Cr	Mo	Si	Ni	Cu	Sn	Al	V	N	Sb
Lukens heat A9349	0.10	0.87	0.011	0.001	2.92	1.48	0.27	0.12	0.06	0.004	0.006	0.10	0.013	0.001
(Aim)	0.10	0.90	<0.012	<0.01	3.0	1.45	0.20	0.20	<0.12	<0.02	<0.025	0.10	0.01	<0.007
Lukens heat A9749	0.15	0.51	0.012	0.003	3.03	1.52	0.25	0.55	0.10	0.006	0.01		0.008	<0.01
(Aim)	0.15	0.50	<0.012	<0.01	3.0	1.5	0.20	0.50	0.1	<0.02	<0.025		<0.02	<0.01
Lukens heat A6660	0.13	0.33	0.012	0.02	2.34	0.99	0.05	0.12	0.15	0.008	0.004			
Cameron heat 56447	0.10	0.49	0.010	0.007	2.15	1.03	0.23							

condition. This involved an austenitizing treatment of 927°C, followed by air cooling, and subsequent stress relief at 677°C for 2 h. In this condition the grain size was ASTM number 5, and the microstructure was fully bainitic. Details of the fabrication history are described elsewhere.⁴ A second lot of material from the same heat was re-austenitized at 927°C for 2 h, water quenched, and stress relieved at 677°C for 4 h. In this condition the grain size was ASTM number 8 and the microstructure was fully bainitic.

The second heat of steel was based on a composition suggested by Spencer and coworkers.^{3,4} This heat was melted by the Electralloy Corporation and processed by Lukens Steel Company as heat A9749. The composition is provided in Table 1, where it can be seen that the aim and actual compositions were acceptably close. The composition was near that of heat A9349 with the following exceptions: the manganese was reduced and the nickel increased to maintain adequate hardenability yet reduce any tendency toward temper embrittlement. The carbon was increased to 0.15% and the vanadium eliminated to minimize possible problems with hot cracking in heavy-section weldments during postweld heat treatments. The heat was rolled to 100-mm- and 25-mm-thick plate products. These were austenitized at 927°C, air cooled, and stress relieved at 621°C for 4 h and 1 h, respectively for the two products.

For purposes of comparison, results from two heats of 2.25 Cr-1 Mo steel are presented where needed. Information on these heats is contained in Table 1. The first heat is Lukens heat A6660, which has been extensively studied in both 150-mm and 300-mm thicknesses.^{12,13} The second heat is a vacuum arc remelted (VAR) material, Cameron heat 56447, which was examined as a 50-mm-thick plate.¹⁴

The constituents in the as-cooled products may be predicted if one knows the cooling rate and the continuous cooling transformation (CCT) diagram for the alloy of interest. Figure 1 provides plots of log average cooling rate versus log thickness for typical commercial cooling procedures: water quenching, fan cooling, and air cooling.¹⁵ Two curves are needed for the case of water cooling, one to represent the quarter thickness (T/4) and the other the mid-section (T/2). The CCT diagrams to

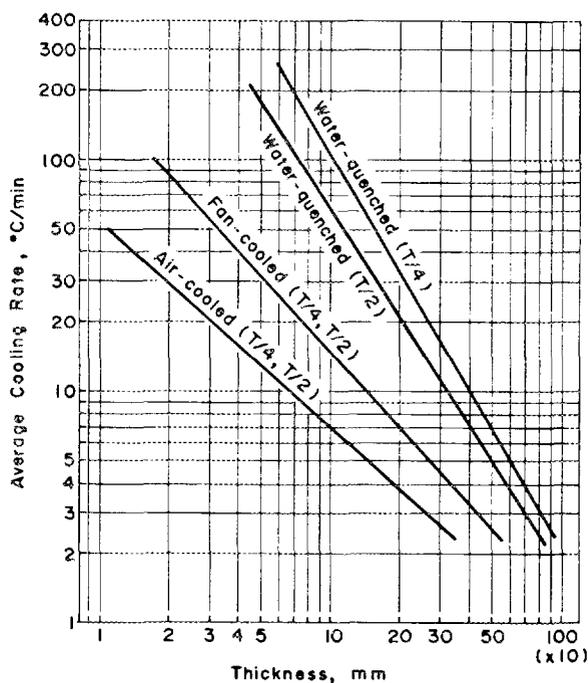


Fig. 1. Average cooling rates between the austenitizing temperature and 450°C for various cooling methods. *Source:* Y. Murakami, T. Nomura, and J. Watanabe, "Heavy-Section 2.25 Cr-1 Mo Steel for Hydrogenation Reactors," pp. 383-417 in *Application of 2.25 Cr-1 Mo Steel for Thick-Wall Pressure Vessels*, ed. G. S. Sangdahl and M. Semchyshen, STP 755, American Society for Testing and Materials, Philadelphia, 1982.

be used in conjunction with Fig. 1 are shown in Fig. 2 for 2.25 Cr-1 Mo steel and in Fig. 3 for heat A9349 (3 Cr-1.5 Mo-0.1 V steel). No similar curve is currently available for heat A9749, but it is known from the work of Spencer et al. that a cooling rate of 8°C/min produces a bainitic microstructure with some retained austenite.⁴ This degree of hardenability would correspond to the curves in Fig. 2 that are labeled 2.25 Cr-1 Mo-V-Ti-B.¹⁶ In the sections to follow we refer to the cooling rates, and the observed microstructures are referred back to these curves. In most cases we did not examine the material in the as-cooled condition. Instead, the plates were subjected to a low-temperature stress relief before sectioning and examination. Very little coarsening of the microstructure occurred as a result of this stress relief.

The condition of the material was defined first in terms of the cooling conditions — annealed for slow cooling, normalized for rapid air cooling, and quenched for water cooling. The latter two cooling treatments

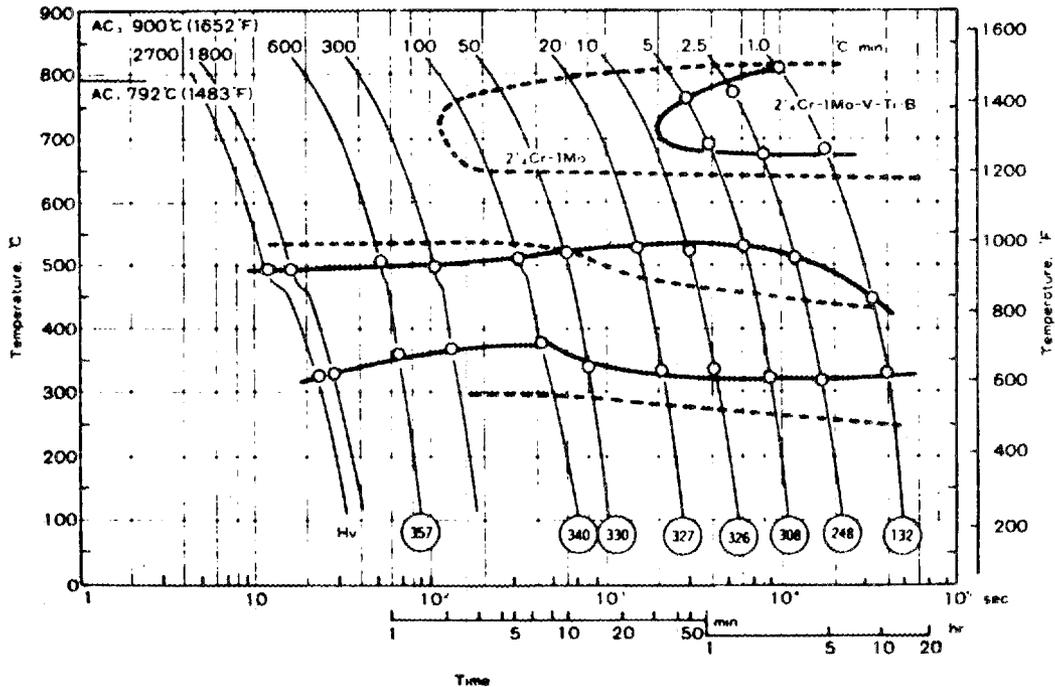


Fig. 2. Continuous cooling transformation (CCT) diagrams for 2.25 Cr-1 Mo steel and 2.25 Cr-1 Mo-V-Ti-B steel. Source: T. Ishiguro et al., "A 2 1/4 Cr-1 Mo Pressure Vessel Steel with Improved Creep Rupture Strength," pp. 129-47 in *Application of 2.25 Cr-1 Mo Steel for Thick-Wall Pressure Vessels*, ed. G. S. Sangdahl and M. Semchyshen, STP 755, American Society for Testing and Materials, Philadelphia, 1982.

required tempering, hence the three treatments are abbreviated below by the terms Ann, NT, or QT, respectively. The tempering condition involved different times at several temperatures, and the severity of the temper is given in terms of the tempering parameter (TP), as used by Hollomon and Jaffee,¹⁷ and defined by the equation

$$TP = T(20 + \log t) \times 10^{-3} ,$$

where T is tempering temperature in Kelvin and t is time at temperature in hours. As TP increases, the severity of the temper increases and the strength decreases.

A summary of the conditions examined by metallography is contained in Tables 2, 3, 4, and 5. These tables provide material identification; annealing, cooling, and tempering conditions; hardness; grain size; and limited tensile and impact data. Table 2 pertains to 2.25 Cr-1 Mo steel, Tables 3 and 4 pertain to heat A9349 of 3 Cr-1.5 Mo steel, and Table 5 pertains to heat A9749 of 3 Cr-1.5 Mo steel.

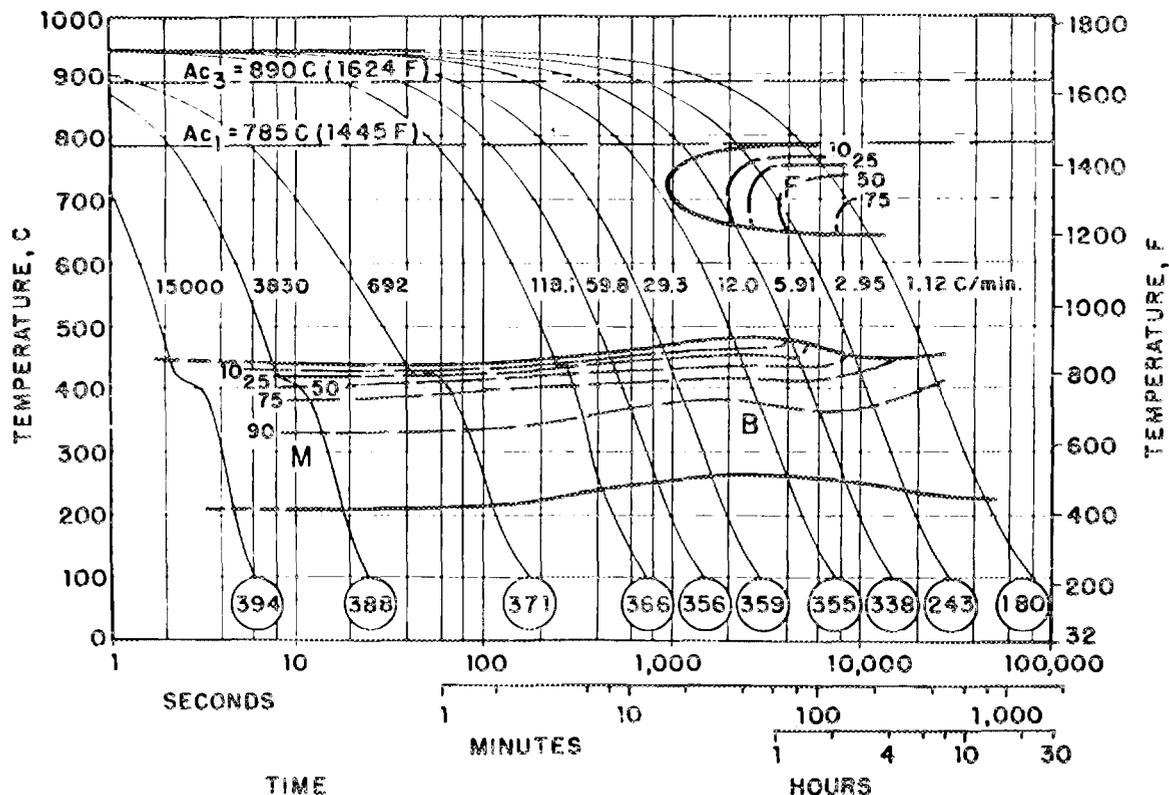


Fig. 3. Continuous cooling transformation diagram for 3 Cr-1.5 Mo steel, heat A9349. Source: T. Wada and T. B. Cox, "A New 3 Cr-1.5 Mo Steel for Pressure Vessel Applications," pp. 77-93 in *Research on Chrome-Moly Steels*, MPC-21, American Society of Mechanical Engineers, New York, 1984.

2.25 Cr-1 Mo STEEL

For reference purposes, the metallographic structures produced in 2.25 Cr-1 Mo steel are presented first. In Fig. 4, for example, the microstructures produced in the VAR heat are shown. Figures 4(a) and 4(b) reveal the combination of ferrite and pearlite that is produced by very slow furnace cooling — approximately 1°C/min. This microstructure has a hardness near DPH 140 (450 MPa ultimate strength). By fan cooling the 50-mm plate the cooling rate is increased to 30°C/min, and the type of microstructure shown in Fig. 5(a) is obtained. Consistent with expectations based on the cooling curves in Fig. 1, pearlite disappears but some proeutectoid ferrite occurs in combination with bainite. This microstructure has a grain size near ASTM 8 and a hardness of DPH 195 after cooling. Upon further tempering, the bainite coarsens as shown in Fig. 5(b) and softens to a DPH near 169.

Table 2. Mechanical properties of 2 1/4 Cr-1 Mo steel

Tempering conditions			Tensile properties				Impact properties		
Temperature (°C)	Time (h)	Tempering parameter ^a [$T(20 + \log t) \times 10^{-3}$]	Yield strength		Ultimate strength		Total elongation (%)	TT-54 ^b	USE ^c
			(MPa)	(ksi)	(MPa)	(ksi)		(°C)	(J)
Heat A6660									
690 ^d	12	20.30	325	47.1	523	75.9	24	21	95
690 ^e	12	20.30	506	73.5	630	91.4	20	-51	122
662 ^d	6	19.43	545	78.0	659	100.8	24		
649 ^d	8	19.27						-50	135
677 ^d	16	20.15						-65	154
Heat 56447^f									
621	1.5	18.05	628	91.0	768	111	22.4		
621	8	18.69	507	73.5	622	90.2	23.5	-67	358
649	8	19.27	465	67.4	599	86.9	24.9	-75	358
677	8	19.86	445	64.5	575	83.4	25.8	-60	358

^a T = temperature, K; t = time, h.

^b54-J (40-ft-lb) transition temperature.

^cUpper-shelf energy.

^dAir cooled.

^eWater quenched.

^fFan cooled.

6

Table 3. Mechanical properties of 3 Cr-1.5 Mo-0.1 V steel (heat A9349)

Tempering conditions			Hardness (DPH)	Tensile properties						Impact properties		
Temperature (°C)	Time (h)	Tempering parameter ^a [$T(20 + \log t) \times 10^{-3}$]		Yield strength		Ultimate strength		Elongation (%)		TT-54 ^b (°C)	FATT ^c (°C)	USE ^d (J)
			(MPa)	(ksi)	(MPa)	(ksi)	Uniform	Total				
Normalized and tempered												
566	2	17.03	387.0	1020	148.0	1309	190.0	6.6	16.6	98.07	159.8	169.5
663	8	19.57	239.3	675	98.0	774	112.0	5.6	17.7	-39.2	-11.52	218.4
663	16	19.85	225.1	624	90.6	731	106.0	5.8	17.6	-37.7	6.03	225.0
674	16	20.08	212.1	575	83.5	687	99.7	6.3	19.2	-55.18	-12.19	229.5
688	8	20.09	211.8	560	81.3	678	98.4	7.0	20.2	-60.34	-12.64	243.3
693	16	20.48	194.7	491	71.3	621	90.1	7.8	22.0	-58.47	-19.62	265.5
701	16	20.65	188.8	452	65.6	590	85.6	9.4	23.7	-72.64	-27.72	273.1
Quenched and tempered												
566	2	17.03	387.3	1078	156.5	1261	183.0	5.3	17.4	67.06	117.7	136.0
663	8	19.56	238.0	666	96.5	770	111.7	6.8	18.4	-73.48	-28.63	225.5
690	16	20.44	202.0	497	72.0	626	90.8	8.5	22.3	-70.15	-35.14	232.7
704	30	21.0	180.5	441	63.9	582	84.3	10.2	25.2	-77.7	-33.89	266.3

^a T = temperature, K; t = time, h.

^b54-J (40-ft-lb) transition temperature.

^c50% shear fracture appearance transition temperature.

^dUpper-shelf energy.

Table 4. Effect of cold rolling and austenitizing temperature on the grain size and hardness of 3 Cr-1.5 Mo steel (heat A9349)

Temperature		Time (h)	Cold work (%)	Hardness (DPH)	Grain size (ASTM No.)
(°C)	(°F)				
927	1700	2	10	359	12
			25	361	11
			40	374	10
954	1750	2	10	318	9
			25	345	9
			40	350	9
982	1800	2	10	314	8
			25	339	8
			40	351	8
1000	1835	2	10	370	8
			25	365	8
			40	364	8

Heat A6660 of 2.25 Cr-1 Mo steel was water quenched in the as-received condition, and the 150-mm thickness was fully bainitic at the T/4 location. Typical microstructures are shown in Fig. 6. The grain size was near ASTM number 6. Some coarsening of the substructure was noticeable, as the hardness dropped from DPH 212 in the stress-relieved condition to DPH 180 for a tempering parameter of 20.15, but the strength and fracture toughness of the material were adequate in both heat-treated conditions.

3 Cr-1.5 Mo STEEL

The microstructures in the as-received 100-mm plates of heat A9349 3 Cr-1.5 Mo steel are shown in Fig. 7. According to the curves in Fig. 1 the NT 100-mm plate would cool at a rate of approximately 7°C/min which, according to Fig. 3, should result in approximately 5% ferrite. The NT material is shown in Figs. 7(a) and 7(c), which reveal a bainitic constituent in a prior austenitic grain size of ASTM number 5. No ferrite was

Table 5. Mechanical properties of quenched and tempered 3 Cr-1.5 Mo steel (heat A9749)

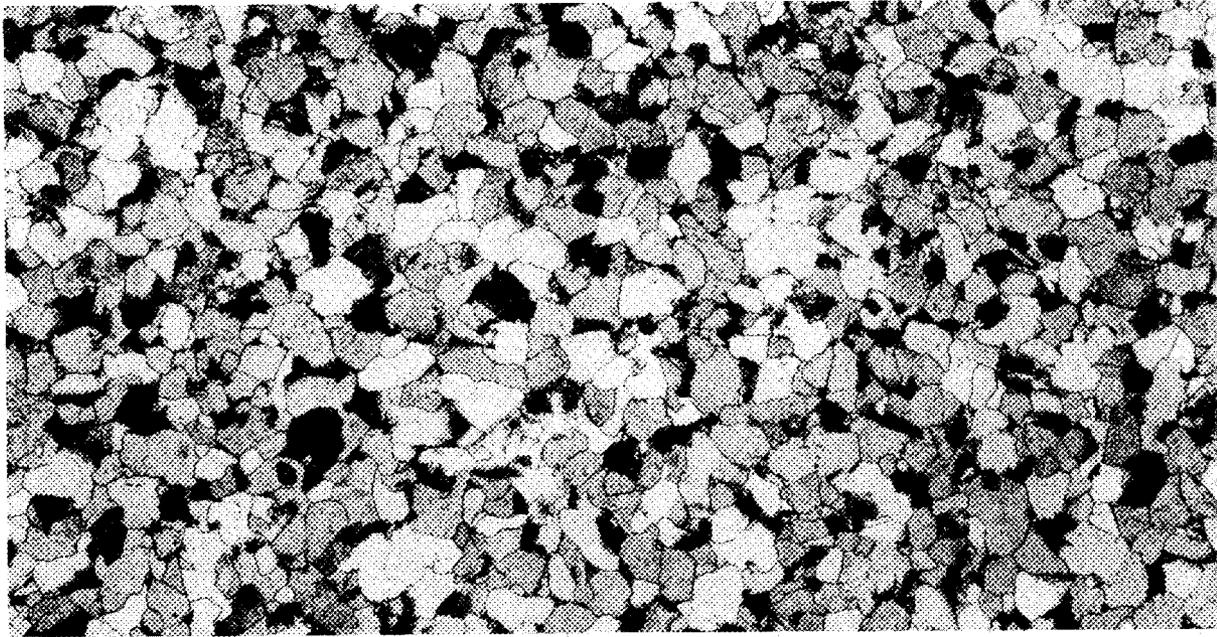
Tempering conditions			Hardness (DPH)	Tensile properties						Impact properties		
Temperature (°C)	Time (h)	Tempering parameter ^a [$T(20 + \log t) \times 10^{-3}$]		Yield strength		Ultimate strength		Elongation (%)		TT-54 ^b (°C)	FATT ^c (°C)	USE ^d (J)
				(MPa)	(ksi)	(MPa)	(ksi)	Uniform	Total			
566	2	As received	382	994	144	1170	170	5.2	22	85	130	132
663	8	19.56	239	646	93.7	771	111.8	7.4	22	-30	10	192
690	16	20.44	207	501	72.7	633	91.8	7.6	21	-60	-30	228
704	30	21.0	193	454	65.8	593	86.0	10.0	26	-60	-30	224

^a T = temperature, K; t = time, h.

^b54-J (40-ft-lb) transition temperature.

^c50% shear fracture appearance transition temperature.

^dUpper-shelf energy.



(a)

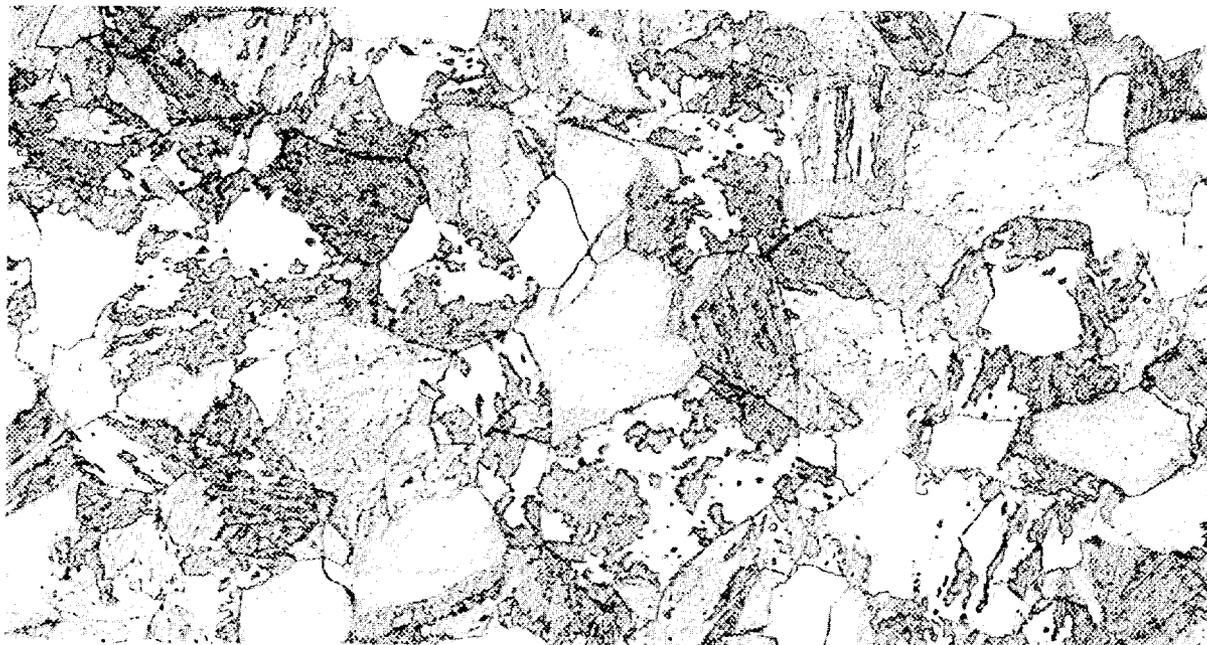
200 μm



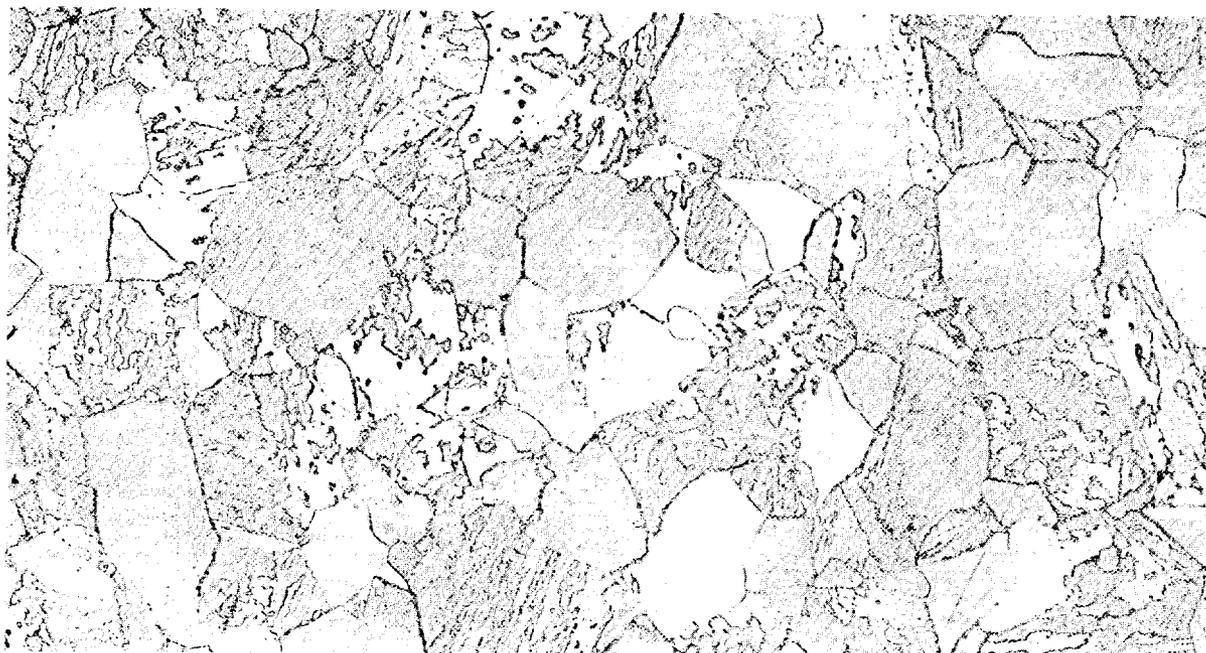
(b)

20 μm

Fig. 4. Microstructures produced by furnace cooling 2.25 Cr-1 Mo steel through the ferrite plus pearlite region of the CCT diagram: DPH = 140.



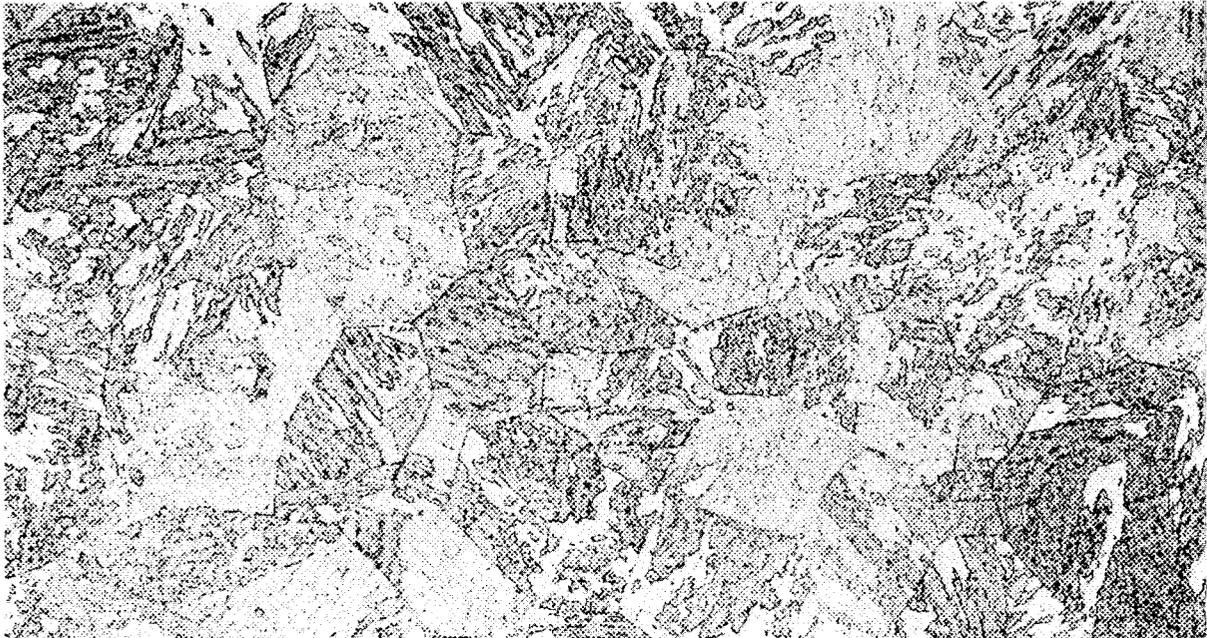
(a)

50 μm 

(b)

50 μm

Fig. 5. Microstructures produced by fan cooling 50-mm plate of 2.25 Cr-1 Mo steel through the ferrite nose and into the bainite region of the CCT diagram, then tempering: (a) $TP = 19.27$, $DPH = 195$; (b) $TP = 20.15$, $DPH = 169$.



(a)

50 μm 

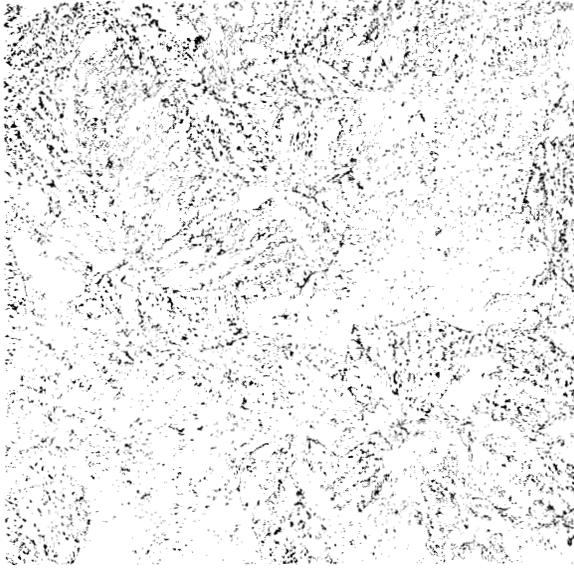
(b)

50 μm

Fig. 6. Microstructures produced by accelerated cooling of 150-mm plate of 2.25 Cr-1 Mo steel through the bainite region of the CCT diagram, then tempering: (a) $TP = 19.27$, $DPH = 212$; (b) $TP = 20.15$, $DPH = 180$.

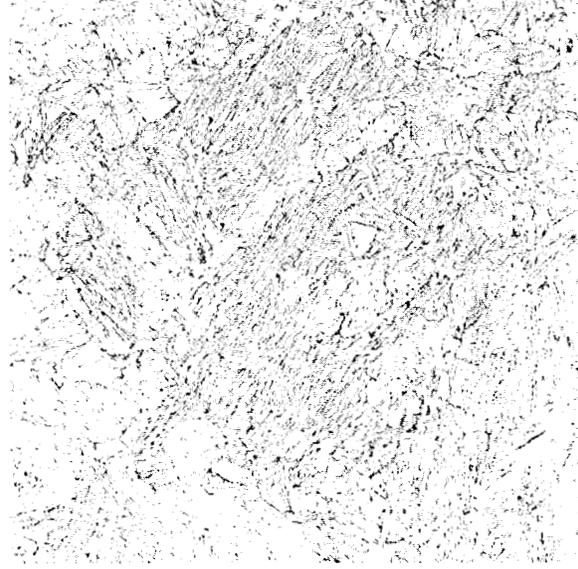
Y-203594

Y-203596



(a)

50 μm

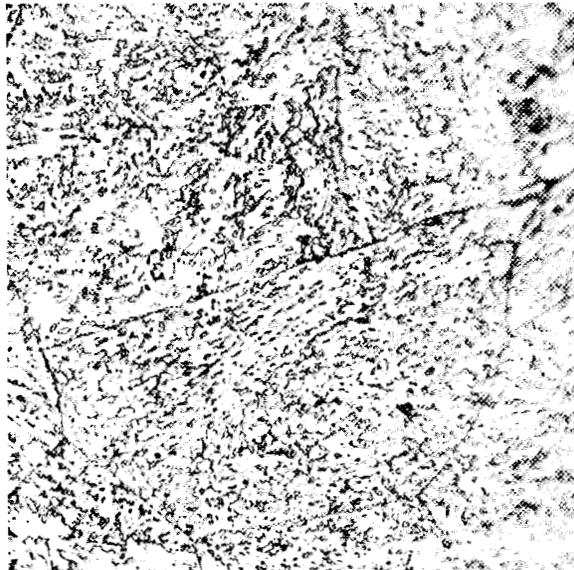


(b)

50 μm

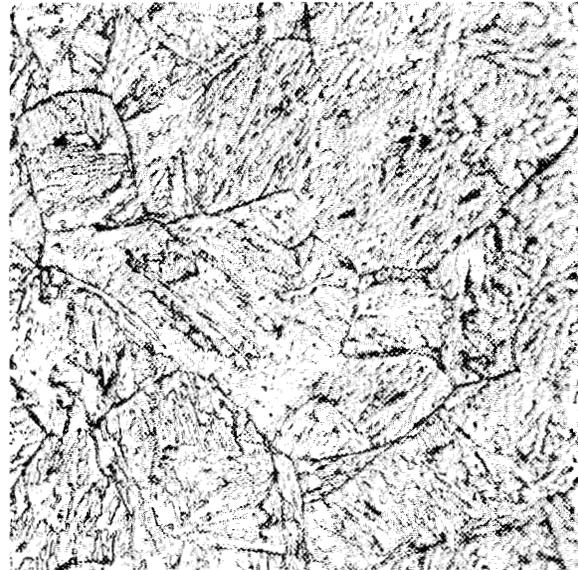
Y-203595

Y-203597



(c)

20 μm



(d)

20 μm

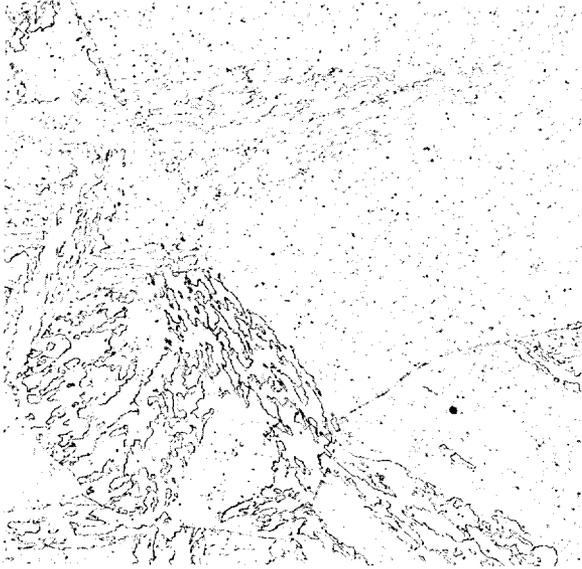
Fig. 7. Microstructures in the as-received 100-mm plates of 3 Cr-1.5 Mo steel (heat A9349), $TP = 17.03$: (a) and (c) for NT condition, $DPH = 387$; (b) and (d) for QT condition, $DPH = 387$.

found. The DPH is very high, about 387. The material that was heat treated again to the QT condition exhibited a fine grain size, about ASTM number 8, as revealed by Figs. 7(b) and 7(d). The water quench should produce cooling rates in the range 60 to 100°C/min. A fully bainitic microstructure would be expected, on the basis of the CCT diagram in Fig. 3, and this expectation is confirmed in Figs. 7(c) and 7(d). The hardness of this microstructure was virtually the same as that of the material in the NT condition.

The evolution of microstructure with tempering for the NT and QT conditions is revealed in Figs. 8 and 9, where direct comparisons may be made between the material in the NT condition and in the QT condition. For both starting conditions, there is a coarsening in the microstructure with increasing *TP*. The QT material tends to exhibit a more acicular bainitic lath structure than the NT material, but there is virtually no difference in the dependence of hardness on the TP for the two starting conditions. Thus, any change in hardness between the original normalized and quenched conditions must have been eliminated by the stress relief treatment introduced at the mill (566°C for 2 h). Any differences in the amount of retained austenite that could exist between the NT and QT conditions are not manifested at the magnifications available in light microscopy and do not affect hardness and tensile properties. The difference in the grain size could play a role, however, because the finer grain size in the QT material would have a strengthening effect and tend to balance the effect of any accelerated tempering of the QT material relative to the NT material.

In order to explore grain size effects for the same heat-treating conditions, a small study was performed to alter the grain size of heat A9349 by cold working and annealing. Limited microscopy, hardness, tensile, and impact tests were then performed. The data produced in the study are summarized in Table 4. Cold rolling to levels of 10, 25, and 40% had little influence on the resulting grain size after austenitizing at several temperatures. The austenitizing temperature was more important, producing the ASTM grain sizes 11, 9, 8, and 8 for austenitizing temperatures of

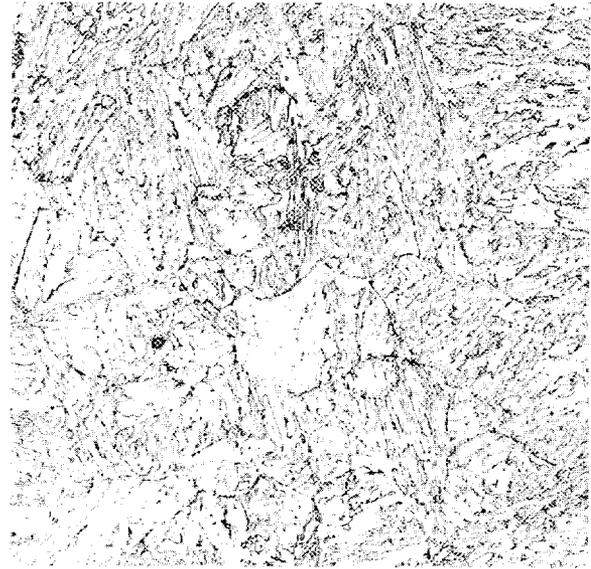
Y-203569



(a)

50 μm

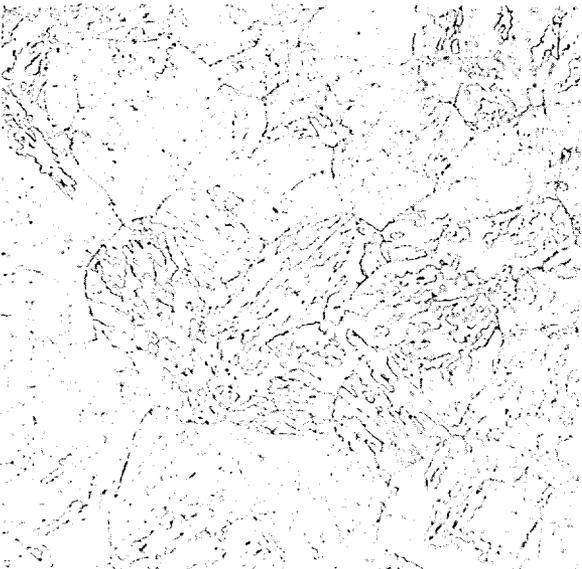
Y-203574



(b)

50 μm

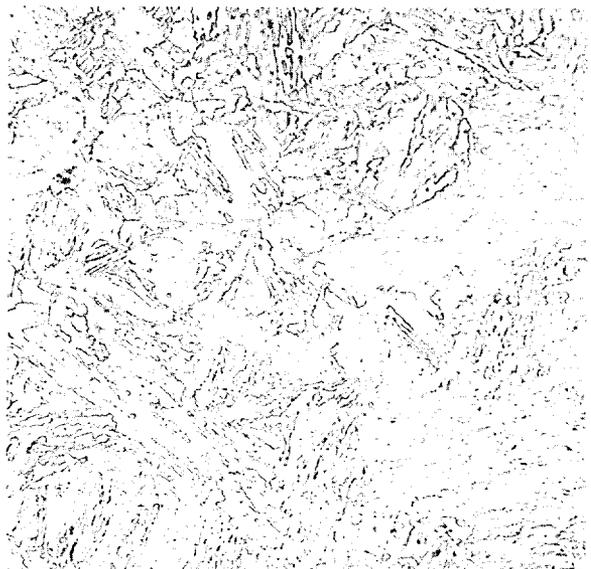
Y-203593



(c)

50 μm

Y-203573

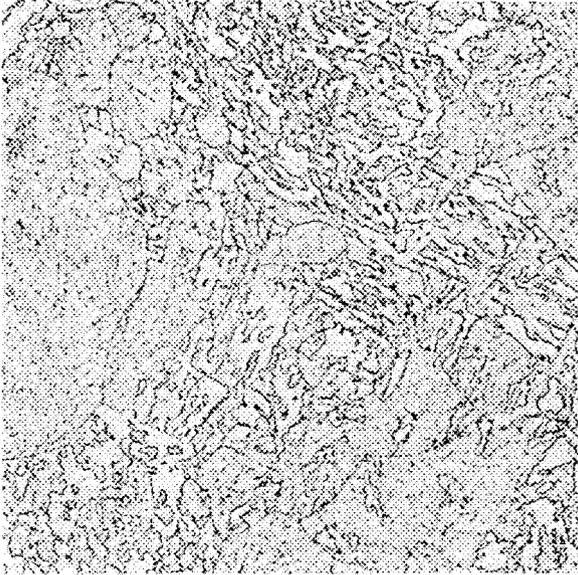


(d)

50 μm

Fig. 8. Change in microstructures with TP for 3 Cr-1.5 Mo steel (heat A9349): (a) NT, $TP = 19.57$; (b) QT, $TP = 19.56$; (c) NT, $TP = 20.08$; (d) QT, $TP = 20.44$.

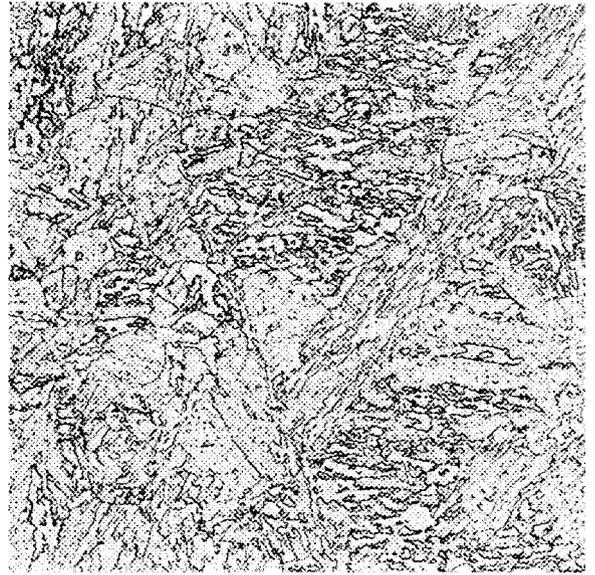
Y-203567



(a)

50 μm

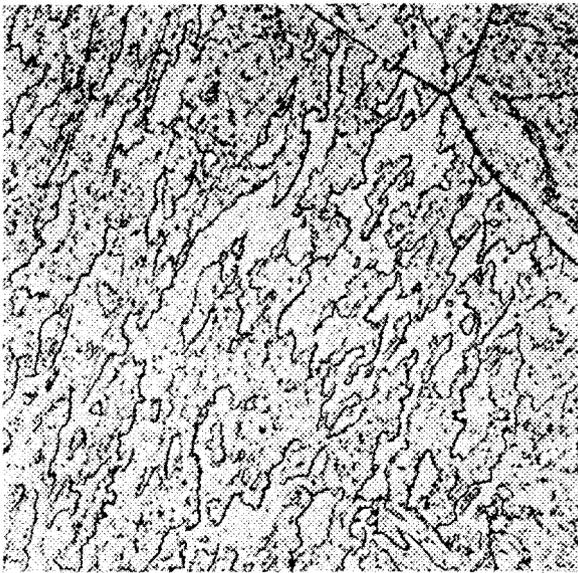
Y-203571



(b)

50 μm

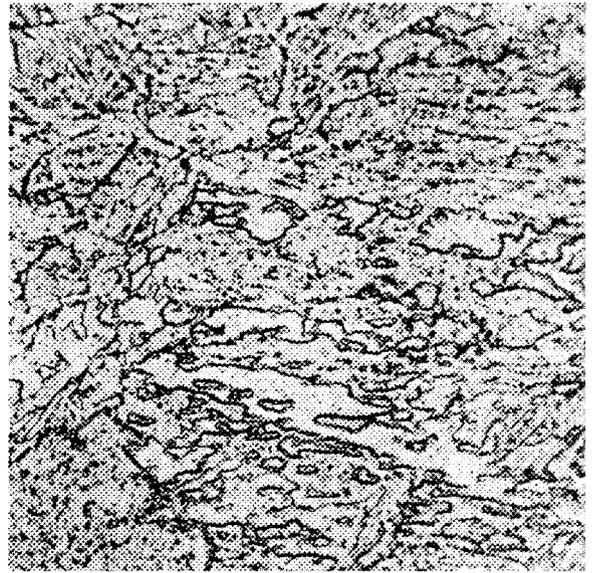
Y-203568



(c)

20 μm

Y-203572



(d)

20 μm

Fig. 9. Change in microstructures with TP for 3 Cr-1.5 Mo steel (heat A9349): (a) NT, $TP = 20.65$; (b) QT, $TP = 21.00$; (c) NT, $TP = 20.65$; and (d) QT, $TP = 21.00$.

927, 954, 982, and 1000°C, respectively. Typical microstructures are provided in Figs. 10 and 11. Thus, by re-austenitizing the QT material at 954°C, the original grain size of ASTM number 8 could be preserved and the material given a NT treatment. Comparison of data produced on this finer-grained NT material after tempering to $TP=20.90$ revealed that the hardness and strength were greater than those for the ASTM number 5 material. The QT material was then re-austenitized at two temperatures, which produced the microstructures shown in Fig. 12. A treatment of 1100°C produced a grain size that matched the original NT material of ASTM number 5. The material was then quenched and tempered to produce a coarser-grained QT material. At the TP value of 19.56 this material had higher hardness than the fine-grained NT material but had the same strength level. Although not conclusive, the results of this small study are consistent with the supposition that the similarities in properties of the original NT and QT materials were due to finer grain size in the QT material in combination with a more rapid rate of tempering.

The microscopy performed on heat A9749 of 3 Cr-1.5 Mo was limited to an evaluation of microstructural coarsening of the as-received material at three values of the TP : 19.56, 20.44, and 21.0. (See Table 5 for the data summary.) Typical microstructures are shown in Fig. 13. The grain size is near ASTM 8, and the material is bainitic, which is consistent with expectations based on the studies of Spencer and coworkers for an air cooling rate near 7°C/min.^{3,4,9} The material exhibits the same response to tempering as heat A9349, in terms of hardness and strength. Very detailed studies of the microstructural response of the heat are being undertaken by Spencer and coworkers,^{3,4} and the results of these studies will be available at a later date. It is important to recognize, however, that this composition is reported to have some retained austenite after rapid cooling.

METALLOGRAPHIC EXAMINATION OF WELDMENTS

The development of joining methods constitutes a major part of the effort to qualify new alloys for pressure vessel applications, and 3 Cr-1.5 Mo steel is no exception to this need. To date, heat A9349 has been

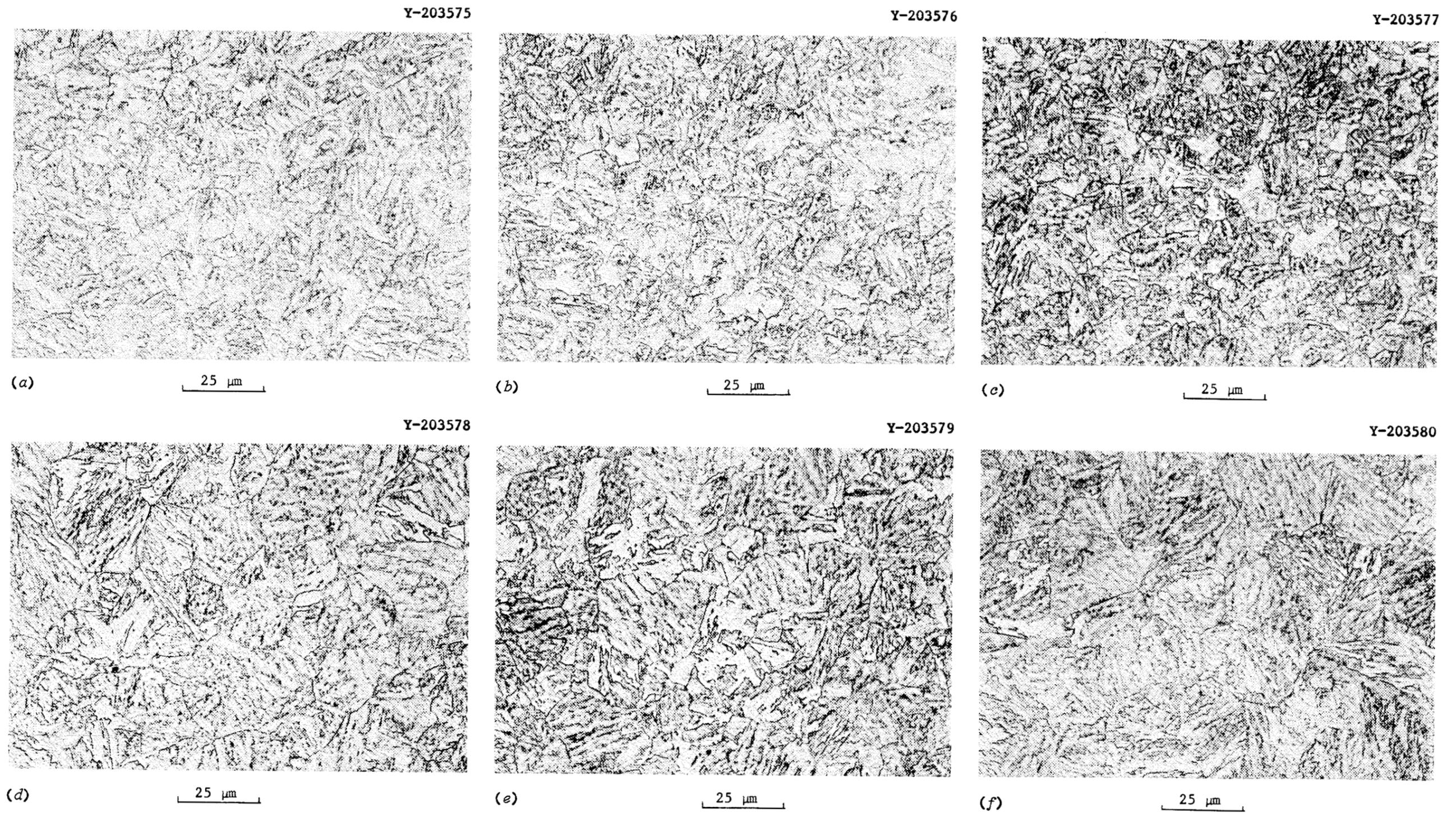


Fig. 10. Effect of cold rolling on the grain size of 3 Cr-1.5 Mo steel (heat A9349) after austenitizing at 927 and 954°C for 2 h: (a) 10% + 2 h at 927°C; (b) 25% + 2 h at 927°C; (c) 40% + 2 h at 927°C; (d) 10% + 2 h at 954°C; (e) 25% + 2 h at 954°C; (f) 40% + 2 h at 954°C.

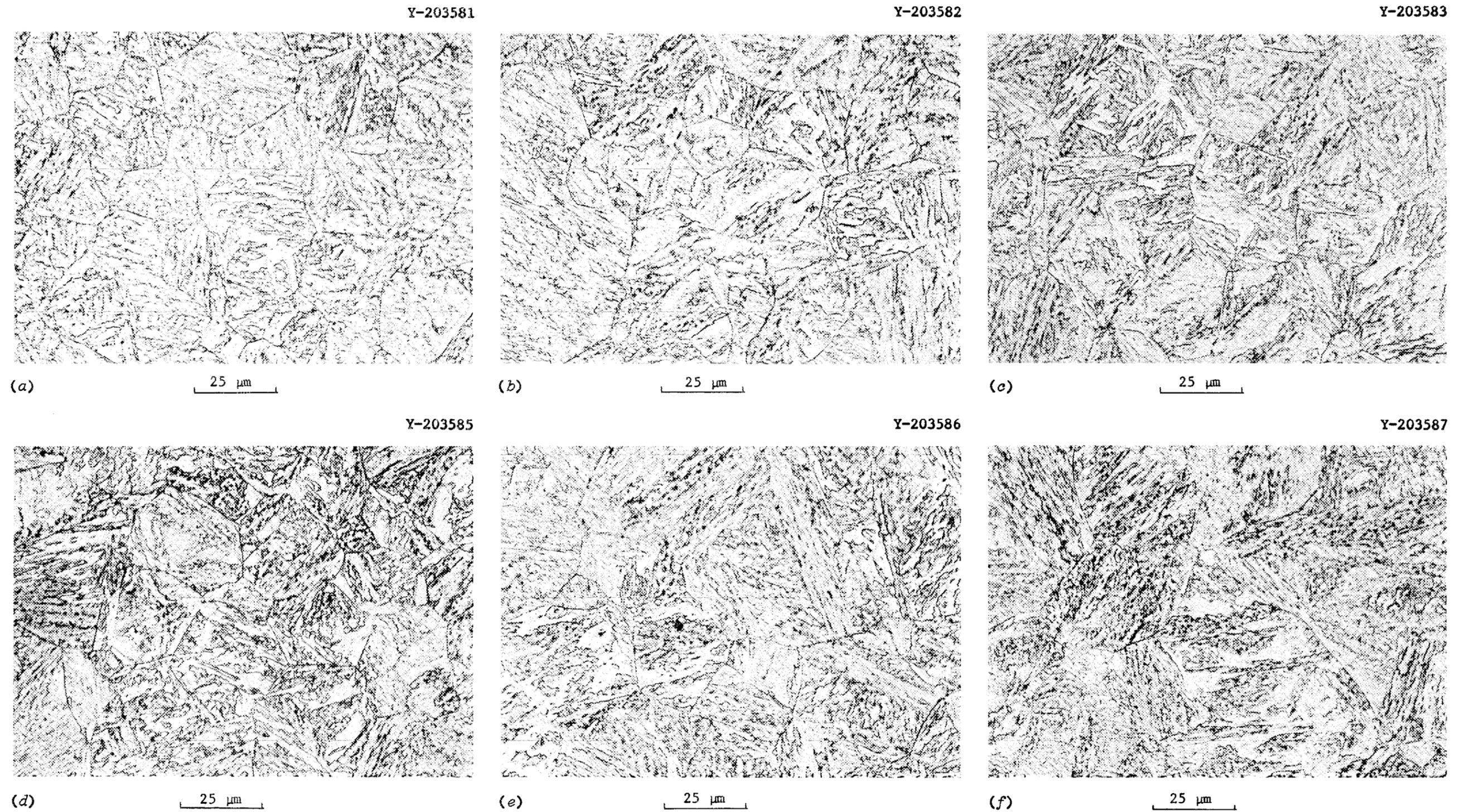
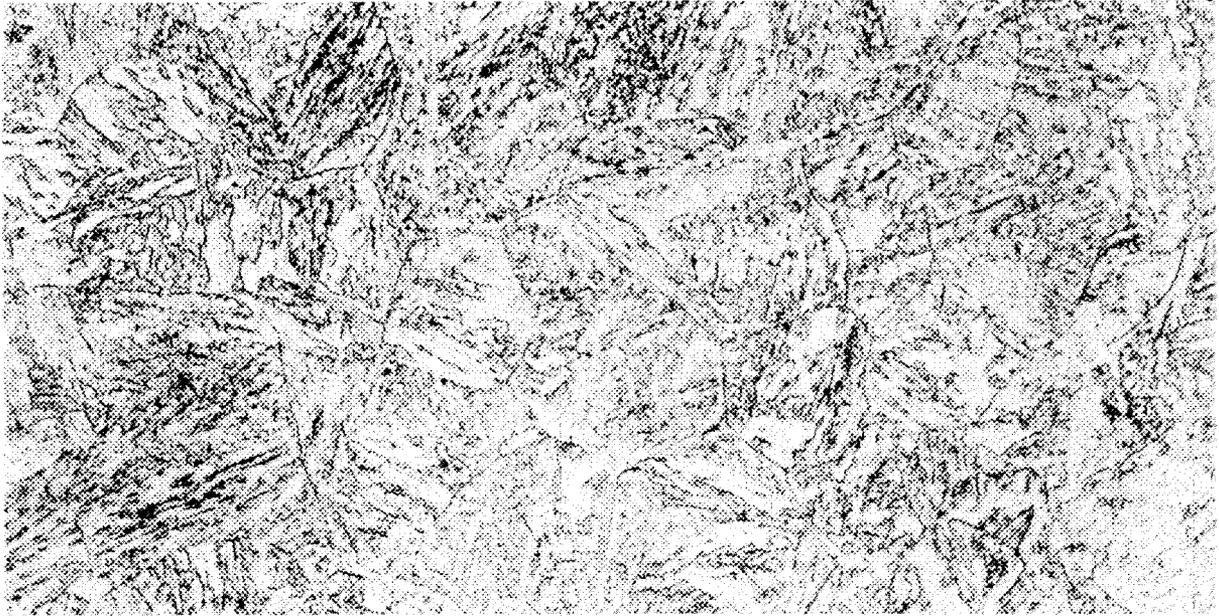
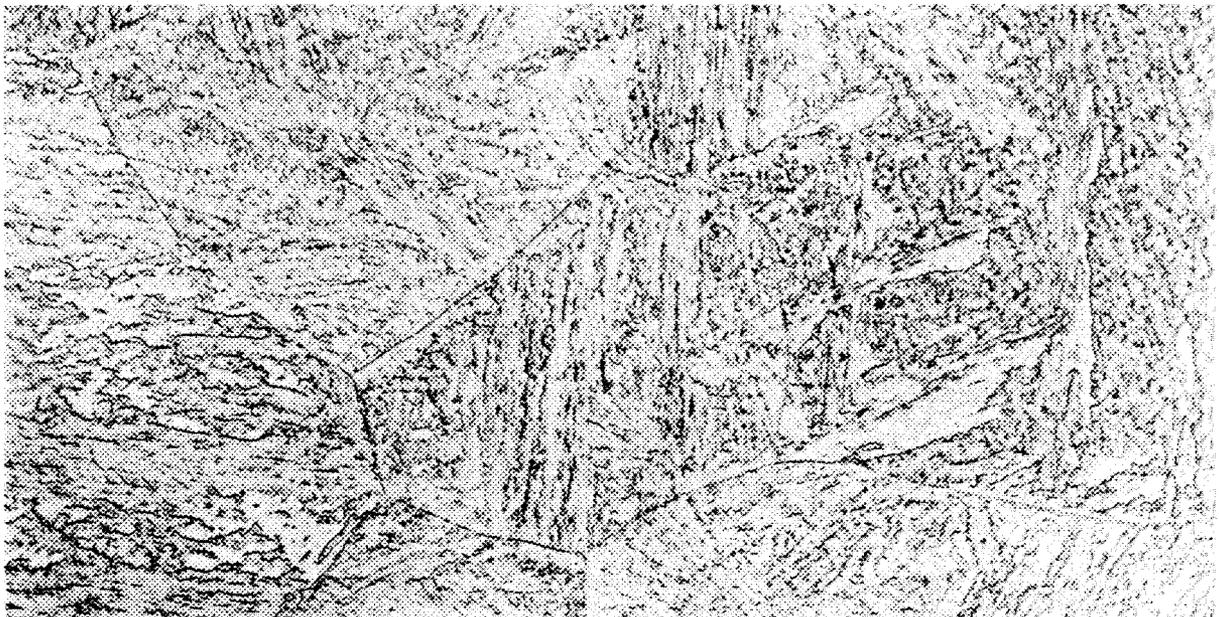


Fig. 11. Effect of cold rolling on the grain size of 3 Cr-1.5 Mo steel (heat A9349) after austenitizing at 982 and 1000°C: (a) 10% + 2 h at 982°C; (b) 25% + 2 h at 982°C; (c) 40% + 2 h at 982°C; (d) 25% + 2 h at 1000°C; (e) 25% + 2 h at 1000°C; (f) 40% + 2 h at 1000°C.



(a)

25 μm

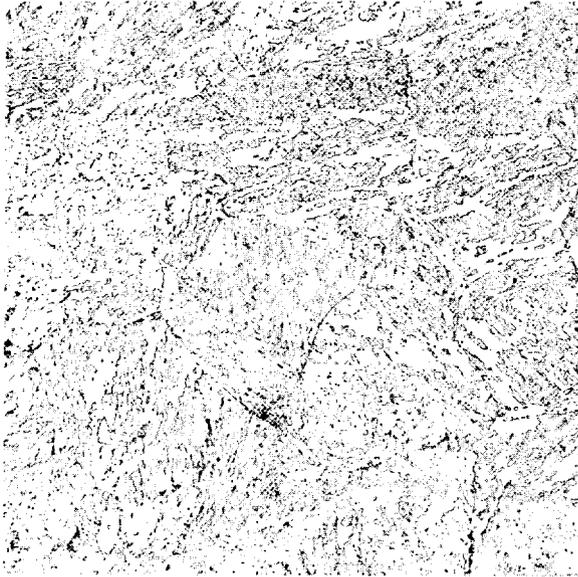


(b)

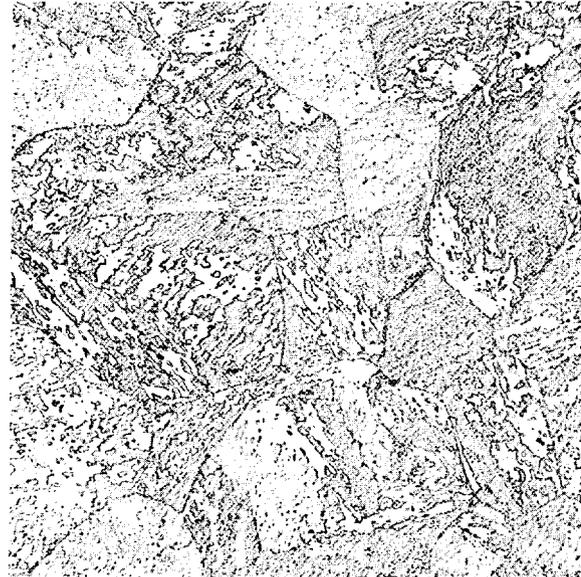
25 μm

Fig. 12. Microstructures produced in QT material by re-austenitizing:
(a) 1000°C for 2 h, (b) 1100°C for 2 h.

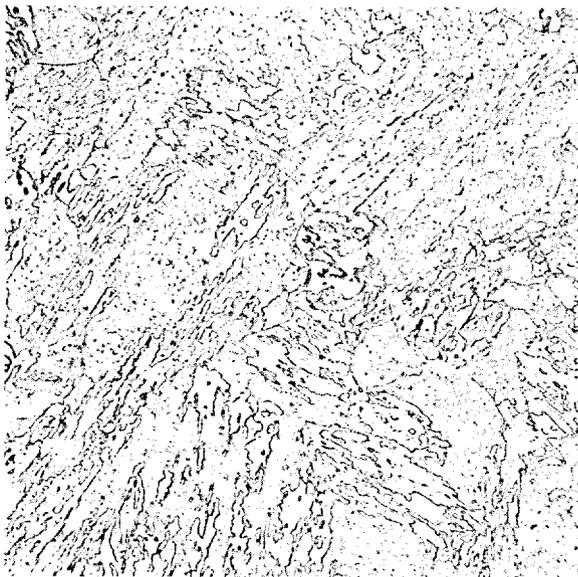
Y-203598

(a) 50 μm

Y-203599

(b) 50 μm

Y-203570

(c) 50 μm

Y-203600

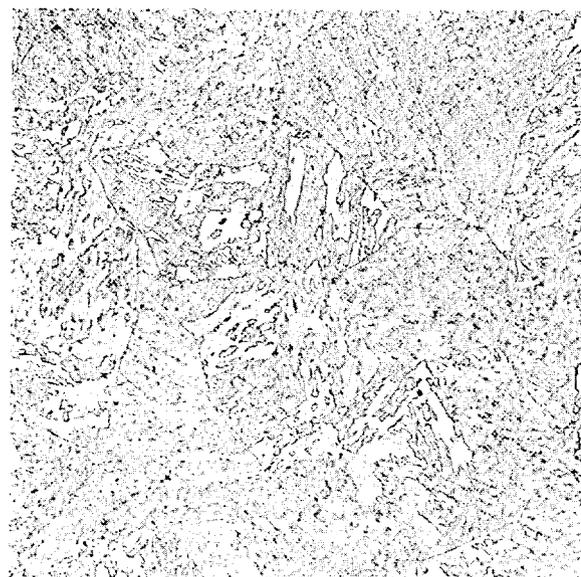
(d) 50 μm

Fig. 13. Microstructures produced by tempering 3 Cr-1.5 Mo steel (heat A9749): (a) $TP = 17.03$, $DPH = 382$; (b) $TP = 19.56$, $DPH = 239$; (c) $TP = 20.44$, $DPH = 207$; (d) $TP = 21.0$, $DPH = 193$.

welded in 25- and 100-mm thicknesses. The thinner section was used to evaluate the shielded metal-arc process at three levels of carbon and vanadium, while the 100-mm plate was used to examine welding by the submerged-arc process with wire having two levels of carbon and vanadium. Further, the 100-mm plate was welded by the electron-beam process. Numerous tempering treatments have been introduced, producing a large number of samples that could be metallographically examined. In addition to the weld metal, the heat-affected zone (HAZ) in the base metal provides additional material for metallurgical examinations. The metallurgical studies on the weldment samples are still in progress, and results will not be covered in this report except for the brief section contained below.

A typical submerged-arc weld deposit microstructure is shown in Fig. 14. This microstructure resulted from a weld deposited by the

Y-195774



Fig. 14. Typical submerged-arc weld metal microstructure in 3 Cr-1.5 Mo-V steel after postweld heat treatment at 663°C for 8 h.

submerged-arc process with a heat input of 22 kJ/cm, followed by a post-weld heat treatment of 663°C for 8 h ($TP = 19.56$). The weld metal hardness for this tempering was DPH 315. The microstructure was bainitic with no evidence of proeutectoid ferrite. This was typical of all the weldments produced to date regardless of the carbon and vanadium levels. Upon tempering, the weld metal hardness was about the same as that of the base metal, but the tensile strength was lower. The lower strength was attributed to a lower carbon content in the weld metal deposit compared with the base metal. Optical metallography at magnifications up to 200 times revealed little or no difference in the weld microstructures as a function of tempering conditions; hence, high-resolution techniques will be needed to more fully characterize the metallurgy of weldments containing filler metal. This work is under way.

Results of an optical metallographic examination of the HAZ in the submerged-arc weld were reported earlier.⁶ The possibility of martensite was identified, but confirmation by transmission electron microscopy is needed.

SCANNING ELECTRON MICROSCOPY

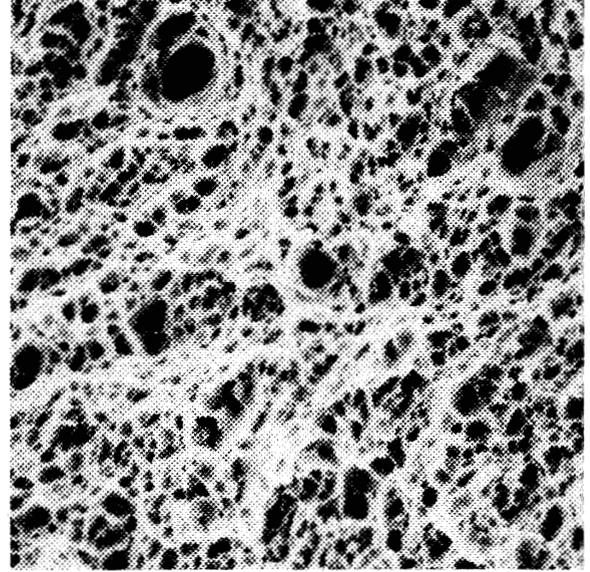
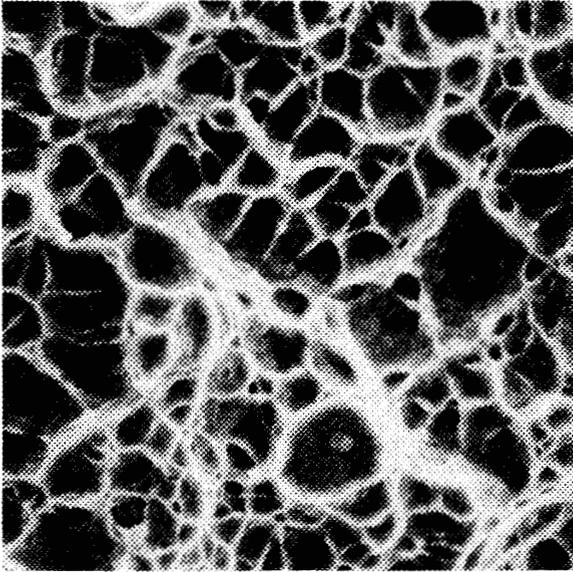
Scanning electron microscopy was undertaken to obtain a better understanding of how the fineness of the bainitic structure affects the fracture surface and upper shelf energy (USE) in the Charpy V impact test. Typical fracture surfaces are shown in Fig. 15. They were produced by testing the NT and QT conditions of heat A9349 at different values of the TP. As shown in Fig. 15 the higher USE fractures are associated with fracture surfaces in which the shear or tear dimples are more closely spaced. This spacing is controlled by the fineness of the precipitates and lath structure in the bainite.

DISCUSSION AND SUMMARY

The results of the studies described in this report demonstrate the high level of knowledge that has accumulated regarding the design of Cr-Mo

YP-622

YP-625



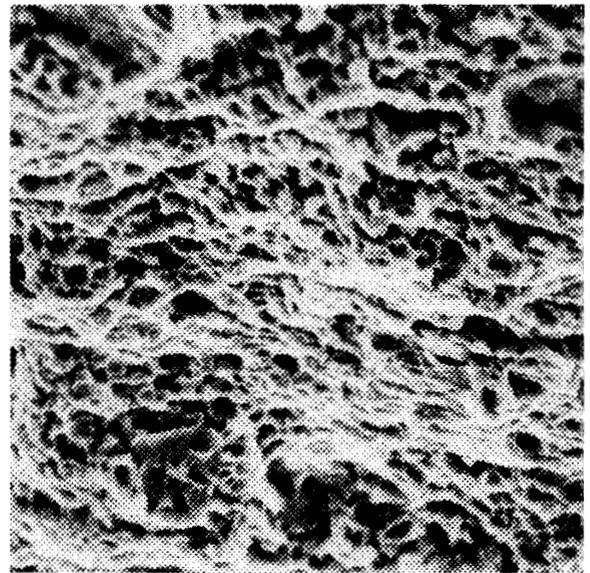
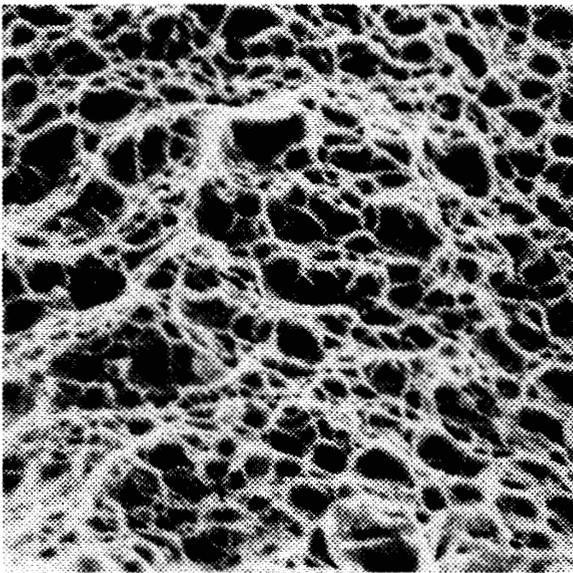
(a)

(b)

20 μm 20 μm

YP-623

YP-624



(c)

(d)

Fig. 15. Scanning electron micrographs of Charpy V specimen fracture surfaces at upper shelf temperatures, 300°C, heat A9349: (a) NT, $TP = 17.03$, 169.5 J; (b) NT, $TP = 20.65$, 273 J; (c) QT, $TP = 17.03$, 136 J; (d) QT, $TP = 21.00$, 266 J.

alloys for pressure vessel applications. Alloys can be developed in the laboratory and scaled easily to commercial heat sizes. The desired microconstituents can be obtained by control of the cooling rate from the austenitizing temperature, and grain sizes in the medium to fine range can be produced by standard mill fabrication practices.

For gasifier pressure vessel applications, the 3 Cr-1.5 Mo steels will require very little further development. In contrast to the needs for steels in high-temperature hydrogen service, there is less concern about the elemental constituents in the carbide precipitates because service for alloys in all but a few gasifier concepts will be limited to temperatures where temper embrittlement, hydrogen attack, and long-term material degradation are unlikely.⁵ The main concerns are that the alloy (1) exhibit minimum variability as a function of minor compositional changes, (2) offer a wide window for heat treatment that will result in a fine bainite microstructure for standard cooling rates, (3) offer a broad range of tempering conditions that will result in a combination of good strength and toughness, and (4) be readily weldable by standard practices such as the shielded metal arc and submerged-arc practices.

The metallographic examinations summarized above suggest that all of these targets can be met in the 3 Cr-1.5 Mo series of alloys. To be sure, there is much to be learned about the tendency to form retained austenite and to understand how this constituent transforms during tempering or long-term, high-temperature service. Also, it appears from the work of Richey and Shaw that some compositions within the series of the 3 Cr-1.5 Mo steels are more susceptible to reheat cracking and hydrogen embrittlement than originally anticipated.^{8,10} These are subjects still under investigation, however, so it would be premature to make recommendations concerning them.

The information summarized by this report will be provided to industrial groups that are responsible for the eventual selection of compositions to be developed into alloys approved by the ASME Boiler and Pressure Vessel Code for pressure vessel service.

ACKNOWLEDGMENTS

The report was reviewed by R. R. Judkins and R. K. Nanstad. It was edited by O. A. Nelson, and the final manuscript was prepared by D. L. Northern of the Metals and Ceramics Publications Office.

REFERENCES

1. T. Wada and T. B. Cox, "3 Cr-1.5 Mo Steel for Pressure Vessels in Hydrogen Service," pp. 81-98 in *Advanced Materials for Pressure Vessel Service with Hydrogen at High Temperatures and Pressures*, MPC 18, ed. M. Semchyshen, American Society of Mechanical Engineers, New York, 1982.
2. T. Wada and T. B. Cox, "A New 3 Cr-1.5 Mo Steel for Pressure Vessel Applications," pp. 77-93 in *Research on Chrome-Moly Steels*, MPC-21, American Society of Mechanical Engineers, New York, 1984.
3. E. R. Parker et al., "An Advanced 3-Cr-Mo-Ni Steel for Hydrogen Service," pp. 109-16 in *Research on Chrome-Moly Steels*, MPC-21, American Society of Mechanical Engineers, New York, 1984.
4. P. N. Spencer et al., "Design of Low Alloy Steels for Thick Walled Pressure Vessels," pp. 59-68 in *AR&TD Fossil Energy Materials Program Quarterly Progress Report for Period Ending Sept. 30, 1984*, ORNL/Fmp-84/4, November 1984.
5. R. W. Swindeman et al., *Assessment of the Need for an Advanced High-Strength Chromium-Molybdenum Steel for Construction of Third-Generation Gasifier Pressure Vessels*, ORNL/TM-8873, November 1984.
6. D. P. Edmonds et al., *Evaluation of 3 Cr-1.5 Mo-0.1 V Steel as a Gasifier Pressure Vessel Alloy*, ORNL-6106, January 1985.
7. J. H. Hollomon and L. D. Jaffe, *Ferrous Metallurgical Design*, John Wiley & Sons, New York, 1948.
8. M. W. Richey, *The Weldability of Cr-Mo Steels for Fossil Energy Applications*, M.S. thesis, University of Tennessee, March 1984.
9. R. O. Ritchie et al., "A New Series of Advanced 3 Cr-Mo-Ni Steels for Thick Section Pressure Vessels in High Temperature and Pressure Hydrogen Service," *J. Mater. Energy Syst.*, **6**(3), 151-62 (1984).

10. B. J. Shaw, "A Study of Carbides Formed in Low Alloy Cr-Mo Steel," pp. 117-28 in *Research on Chrome-Moly Steels*, MPC-21, ed. R. A. Swift, American Society of Mechanical Engineers, New York, 1984.
11. R. L. Klueh, work in progress at the Oak Ridge National Laboratory.
12. R. A. Swift and J. A. Gulya, "Evaluation of A387-22 Steel Modified for Improved Toughness," pp. 93-110 in *Advanced Materials for Pressure Vessel Service with Hydrogen at High Temperatures and Pressures*, MPC-18, ed. M. Semchyshen, American Society of Mechanical Engineers, New York, 1982.
13. R. W. Swindeman, M. K. Booker, and W. J. McAfee, *Design Methodology and Materials Data Base for Solvent Refined Coal Dissolver Vessels*, ORNL/TM-8577, August 1983.
14. R. W. Swindeman et al., *Effect of Tempering on the Strength and Toughness of 2.25 Cr-1 Mo Steel Weldments*, ORNL/TM-9307, October 1984.
15. Y. Murakami, T. Nomura, and J. Watanabe, "Heavy-Section 2.25 Cr-1 Mo Steel for Hydrogenation Reactors," pp. 383-417 in *Application of 2.25 Cr-1 Mo Steel for Thick-Wall Pressure Vessels*, ed. G. S. Sangdahl and M. Semchyshen, STP 755, American Society for Testing and Materials, Philadelphia, 1982.
16. T. Ishiguro et al., "A 2 1/4 Cr-1 Mo Pressure Vessel Steel with Improved Creep Rupture Strength," pp. 129-47 in *Application of 2.25 Cr-1 Mo Steel for Thick-Wall Pressure Vessels*, ed. G. S. Sangdahl and M. Semchyshen, STP 755, American Society for Testing and Materials, Philadelphia, 1982.
17. J. H. Hollomon and L. D. Jaffee, "Time-Temperature Relations in Tempering Steels," *Trans. Am. Inst. Min. Metall. Eng.* **162**, 223-49 (1945).

INTERNAL DISTRIBUTION

- | | | | |
|--------|-------------------------------|--------|-----------------------------|
| 1-2. | Central Research Library | 25. | P. J. Maziasz |
| 3. | Document Reference Section | 26. | H. E. McCoy |
| 4-5. | Laboratory Records Department | 27. | R. K. Nanstad |
| 6. | Laboratory Records, ORNL RC | 28. | G. M. Slaughter |
| 7. | ORNL Patent Section | 29-33. | R. W. Swindeman |
| 8. | K. W. Boling | 34-36. | P. T. Thornton |
| 9-10. | R. A. Bradley | 37. | P. F. Tortorelli |
| 11. | C. R. Brinkman | 38. | J. M. Vitek |
| 12. | W. R. Corwin | 39. | R. J. Charles (Consultant) |
| 13. | R. S. Crouse | 40. | G. Y. Chin (Consultant) |
| 14. | J. A. Horak | 41. | H. E. Cook (Consultant) |
| 15. | R. R. Judkins | 42. | Alan Lawley (Consultant) |
| 16. | J. R. Keiser | 43. | W. D. Nix (Consultant) |
| 17-21. | J. F. King | 44. | J. C. Williams (Consultant) |
| 22-24. | R. L. Klueh | | |

EXTERNAL DISTRIBUTION

45. P. M. Brister, Route 3, Box 31, Carnesville, GA 30521
46. R. L. Smith, P.O. Box 98, Chassel, MI 49916
47. ALLEGHENY LUDLUM STEEL CORPORATION, Brackenridge, PA 15014
G. L. Houze
48. AMERICAN WELDING SOCIETY, 550 LeJeune Road, Miami, FL 33126
H. G. Ziegenfuss
49. ASTM, Standards Development Division, 1916 Race Street,
Philadelphia, PA 19103
M. E. Lieff
50. BABCOCK & WILCOX COMPANY, 20 S. Van Buren Ave., Barberton,
OH 44203
Michael Gold
51. BADGER AMERICA, INC., 1 Broadway, Cambridge, MA 02142
W. P. Webb
52. C. F. BRAUN AND COMPANY, 1000 S. Freemont, Alhambra, CA 91802
G. R. Prescott

53. CBI INDUSTRIES, 800 Jorie Blvd., Oak Brook, IL 60521
W. R. Mikesell
54. CHEVRON CORPORATION, P.O. Box 4012, Richmond, CA 94804
A. G. Imgram
- 55-57. CLIMAX MOLYBDENUM COMPANY, 1600 Huron Parkway, Ann Arbor,
MI 48106
T. B. Cox
Marion Semchyshen
T. Wada
58. COMBUSTION ENGINEERING, INC., 1000 Prospect Hill Road, Windsor,
CT 06095
E. R. Steen
- 59-60. COMBUSTON ENGINEERING, INC., 911 W. Main Street, Chattanooga,
TN 37401
D. A. Canonico
B. W. Roberts
61. CORNELL UNIVERSITY, Ithaca, NY 14850
Che-Yu Li
- 62-63. ELECTRIC POWER RESEARCH INSTITUTE, P.O. Box 10412, Palo Alto,
CA 94303
R. I. Jaffee
R. Viswanathan
64. EXXON RESEARCH AND ENGINEERING COMPANY, P.O. Box 101, Florham
Park, NJ 07932
G. Sorell
65. FOSTER WHEELER DEVELOPMENT CORPORATION, 12 Peach Tree Hill Road,
Livingston, NJ 07939
W. R. Apblett, Jr.
66. GA TECHNOLOGIES, P.O. Box 81608, San Diego, CA 92138
D. I. Roberts
67. GENERAL ELECTRIC COMPANY, Turbine Materials Engineering, 1 River
Road, Schenectady, NY 12345
R. M. Curran
68. GETTY REFINING & MARKETING COMPANY, Delaware Refinery, Delaware
City, DE 19706
D. B. Bird

69. JAPAN STEEL WORKS AMERICA, INC., 200 Park Ave., New York,
NY 10168
T. Kondo
70. LUKENS STEEL COMPANY, Coatesville, PA 19320
J. A. Gulya
71. McDERMOTT, INC., 20 S. Van Buren Ave., Barberton, OH 44203
J. P. Rowe
- 72-73. THE METAL PROPERTIES COUNCIL, INC., 345 E. Forty-Seventh Street,
New York, NY 10017
M. Prager
A. O. Schaefer
74. MONSANTO COMPANY, 800 N. Lindbergh Blvd., St. Louis, MO 63166
D. P. Peng
75. SHELL OIL COMPANY, Engineering Services, P.O. Box 3105, Houston,
TX 77001
E. L. Creamer
- 76-77. STANDARD OIL COMPANY (INDIANA), P.O. Box 400, Naperville,
IL 60540
R. W. Howe
S. Ibarra
78. STEARNS CATALYTIC, P.O. Box 5888, Denver, CO 80217
P. E. Dempsey
79. STEEL FOUNDERS' SOCIETY OF AMERICA, 455 State Street,
Des Plaines, IL 60016
J. M. Svoboda
80. TENNESSEE VALLEY AUTHORITY, 1000 Chestnut Street Tower II,
Chattanooga, TN 37401
S. O. Hilton
81. R. D. THOMAS AND COMPANY, INC., 103 Avon Road, Narberth,
PA 19072
R. D. Thomas
82. U.S. STEEL CORPORATION, Applied Research Laboratory, 125 Jamison
Lane, Monroeville, PA 15146
C. E. Spaeder

83. UNIVERSAL CYCLOPS SPECIALTY STEEL DIVISION, 650 Washington Road,
Pittsburgh, PA 15228
H. L. Black
- 84-85. UNIVERSITY OF CALIFORNIA AT BERKELEY, Department of Materials
Science, Berkeley, CA 94720
E. R. Parker
R. O. Ritchie
86. UNIVERSITY OF CALIFORNIA AT SANTA BARBARA, Department of
Chemical and Nuclear Engineering, Santa Barbara, CA 93106
G. R. Odette
87. UNIVERSITY OF CINCINNATI, Department of Materials Science and
Metallurgical Engineering, Cincinnati, OH 45221
J. Moteff
88. UNIVERSITY OF ILLINOIS, Department of Mechanical and Industrial
Engineering, Urbana, IL 61801
D. L. Marriott
89. UNIVERSITY OF TENNESSEE, Department of Chemical,
Metallurgical, and Polymer Engineering, Knoxville, TN 37916
C. D. Lundin
90. OAK RIDGE ASSOCIATED UNIVERSITIES LIBRARY, Oak Ridge, TN 37831
MERT Division
- 91-93. DOE, MORGANTOWN ENERGY TECHNOLOGY CENTER, P.O. Box 880,
Morgantown, WV 26505
J. M. Hobday
A. J. Liberatore
J. S. Wilson
94. DOE, OAK RIDGE OPERATIONS OFFICE, P.O. Box E, Oak Ridge, TN 37831
Office of Assistant Manager for Energy Research
and Development
- 95-96. DOE, OFFICE OF FOSSIL ENERGY, Washington, DC 20545
J. P. Carr (FE-24, C179/GTN)
S. J. Dapkunas (FE-14, B127/GTN)
- 97-123. DOE, TECHNICAL INFORMATION CENTER, Office of Information
Services, P.O. Box 62, Oak Ridge, TN 37831
For distribution by microfiche as shown in DOE/TIC-4500,
Distribution Categories UC-90c (Coal Conversion and
Utilization-Coal Gasification) and UC-90h (Coal Conversion
and Utilization-Materials and Components).