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FINE STRUCTURE AND SUPERPLASTICITY IN ULTRAHIGH CARBON STEELS.(U)
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FINE STRUCTURE AND SUPERPLASTICITY IN ULTRAHIGH CARBON STEELS

BY

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J. WADSWORTH, J.T. LO, B. WALSER, R. robert CALIGIURI [redacted] O.D. SHERBY

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FINE STRUCTURE AND SUPERPLASTICITY IN
ULTRAHIGH CARBON STEELS

(Contract N00014-77-C-0149)

FIRST SEMI-ANNUAL PROGRESS REPORT
(Jan. 1, 1977 - June 30, 1977)

by

J. Wadsworth, J. T. Lo, B. Walser, R. Caligiuri and O. D. Sherby

This program centers on a study of the mechanical behavior of ultrahigh carbon (UHC) steels in the range 1% to 2.4% carbon. These UHC steels were developed at Stanford University during 1973-1976. They were shown to be superplastic at warm temperatures (600 to 800°C) and strong and ductile at room temperature. The objectives of our present study are threefold: (1) to alter the chemistry of the UHC steels in order to optimize superplastic characteristics at elevated temperature and strength and ductility characteristics at room temperature, (2) to investigate various thermal-mechanical processing procedures for obtaining the desired microstructure in the UHC steels and (3) to utilize superplasticity in solid state bonding of similar and dissimilar steels. Our program is sponsored by the Defense Advanced Research Projects Agency and is administered by the Office of Naval Research. We are grateful to Drs. Edward Van Reuth and Arden Bement of DARPA and Dr. Bruce MacDonald of ONR for their support, assistance and interest in our work.

During the first half year of our program we have concentrated on the chemistry of our UHC carbon steels. Specifically we have investigated the influence of small additions of Cr, V, Mn, Si, and Ni on the properties of fine grained UHC steels. We have discovered that Ni and Si are undesireable additions for they enhance graphitization leading to inferior warm and low temperature properties. Addition of Cr, V and Mn appears to be desirable for these elements stabilize the carbides that are formed and fine ferrite grains remain fine even after long time deformation at warm temperatures. Additions of as little as 0.1%V seems to be very beneficial in maintaining fine ferrite grains. The silicon conent should be at 0.2% or less, preferable at 0.1%, and the manganese content should probably be at about 1%. These changes in composition have lead to considerable improvement in superplastic behavior (~1000% elongation) and in room temperature ductility (~20% elongation) compared to our original UHC steels. The mode of failure at warm temperatures is also improved; whereas a finite reduction of area was noted earlier, the new UHC steels usually exhibit 100% R.A.

One of the co-inventors of our UHC steels, Dr. Bruno Walser, spent the month of July at Stanford on our steel program. The last section of this report describes his work at Sulzer Brothers, Winterthur, Switzerland on UHC steels.

Mr. Robert Caligiuri has just completed his doctoral thesis on our program, and his degree will be formally conferred in August of this year. He made excellent progress in his work on superplastic bonding of UHC steel powders, this work will be described in the annual report; the title of his thesis is "The Pressure Sintering Kinetics of Iron Powders and Superplastic Ultrahigh Carbon Steel Powders".

The following is an outline of the four sections that make up this first semi-annual report.

Superplasticity in 52100 tool steel

J. Wadsworth, J.T. Lo, and O.D. Sherby

ABSTRACT

Superplasticity has been successfully developed in type 52100 tool steel.

A variety of thermal-mechanical processing techniques has been developed to obtain fine grained structures - a necessary prerequisite for superplasticity. One group of techniques involved cold working the material after various heat treatments. The second group of techniques involved isothermally warm working the material at different temperatures after first hot working. Material in the as-received condition also was tested for comparison.

The results of tests at 650°C showed that 52100 material could readily be made superplastic, i.e. high values for the strain rate sensitivity exponent and high elongations to failure were possible. An elongation to failure of 731% was achieved in one case.

The development of fine grained microstructures was followed by optical metallography. The role of low angle boundaries has also been investigated. It has been illustrated that low angle boundaries are detrimental to superplastic properties but that they can readily be removed by a simple heat treatment. This heat treatment involves cycling through the A_1 transformation temperature. This converts the low angle boundaries to high angle boundaries thereby improving the superplastic properties.

INTRODUCTION

Superplastic materials are ideal materials in many ways. They are easily formable using small externally applied forces at warm temperatures during fabrication and the fine grain size which characterizes the fine structure superplastic materials is also beneficial at room temperature for strength and toughness.

It is now well established that ultrahigh carbon steels (UHC) can readily be made superplastic[1,2,3]. These steels generally contain between 1% and 2.3% carbon. On the iron-carbon phase diagram, Figure 1, they are seen to lie between high carbon steels and cast iron.

These steels, when in the as-cast condition, can be processed by any one of five routes[4] to develop a final, fine-grained structure. This structure consists of a dispersion of spheroidized cementite particles (0.1 - 0.5μm diam.) in a matrix of fine, equiaxed ferrite (~1μm grain size). This structure meets the requirements for superplastic materials established by Sherby[5]. These are:

1) Fine grain size, typically ASTM grain size number 20 (0.00002" to 0.0002").

2) Usually there are two phases present; this prevents grain growth during superplastic deformation.

3) For the two phases to effectively prevent grain growth they must have similar strengths in the superplastic state.

4) The strain rate sensitivity exponent, m, in the equation $\sigma = K\dot{\varepsilon}^m$, where σ is the stress, $\dot{\varepsilon}$ the strain rate and K a constant, must be high. Usually $m \approx 0.5$.

5) High values of m are usually only found at or above $0.5\text{-}0.7T_m$, where T_m is the absolute melting point. Above $0.5\text{-}0.7T_m$ rapid grain

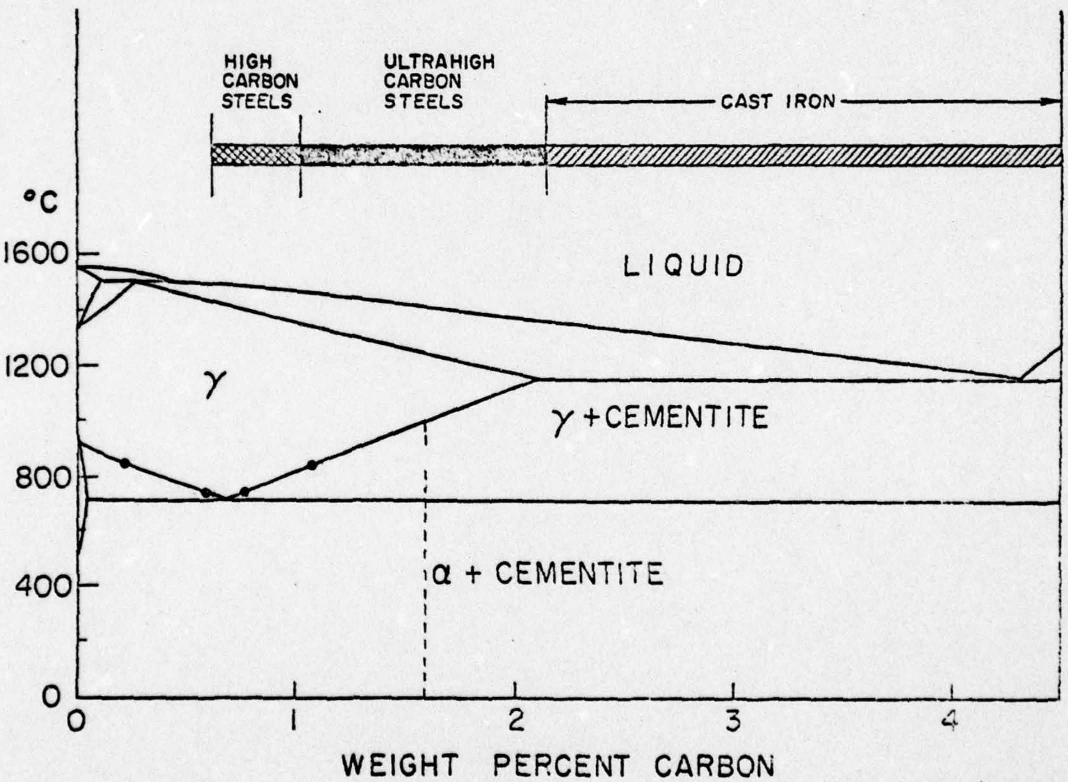


Figure 1. The iron-carbon phase diagram

growth can occur and below $0.5-0.7T_m$ dislocation mechanisms prevail.

6) The strain rate, $\dot{\varepsilon}$, is typically low (10^{-6} to 10^{-4} sec^{-1}) although this can be varied with grain size.

7) The grain boundaries should be high angle (disordered) boundaries.

8) The grain boundaries should be mobile in order to relieve stress concentrations.

9) The grains should be equiaxed.

Usually superplastic properties are assessed by determining both the strain rate sensitivity and the elongation to failure at an appropriate strain rate. Figure 2 shows a schematic illustration of the stress dependence of the strain rate that is often found in ultrahigh carbon steels. The slope of this curve is the stress exponent, n, which is the reciprocal of the strain rate sensitivity. Two regions are usually found. At slow strain rates (region 2) the material has a high m value and is superplastic. At high strain rates the strain rate sensitivity is low and the material is generally not superplastic. Ductilities in excess of 1000% have been found in certain ultrahigh carbon steels[6].

The influence of small alloying additions has been a source of interest since the success of plain ultrahigh carbon steels[1,2,3,5]. In particular, carbide stabilizing elements such as chromium, vanadium, and titanium are considered to be suitable additions. These additions have been shown to be of considerable benefit in improving the superplastic properties of UHC steels[6].

52100 type tool steel falls into the above category of an ultrahigh carbon steel containing a small amount (~1 1/2%) of chromium. It is a cheap, widely available tool steel and its investigation is worthwhile since it is a commercially used ultrahigh carbon steel.

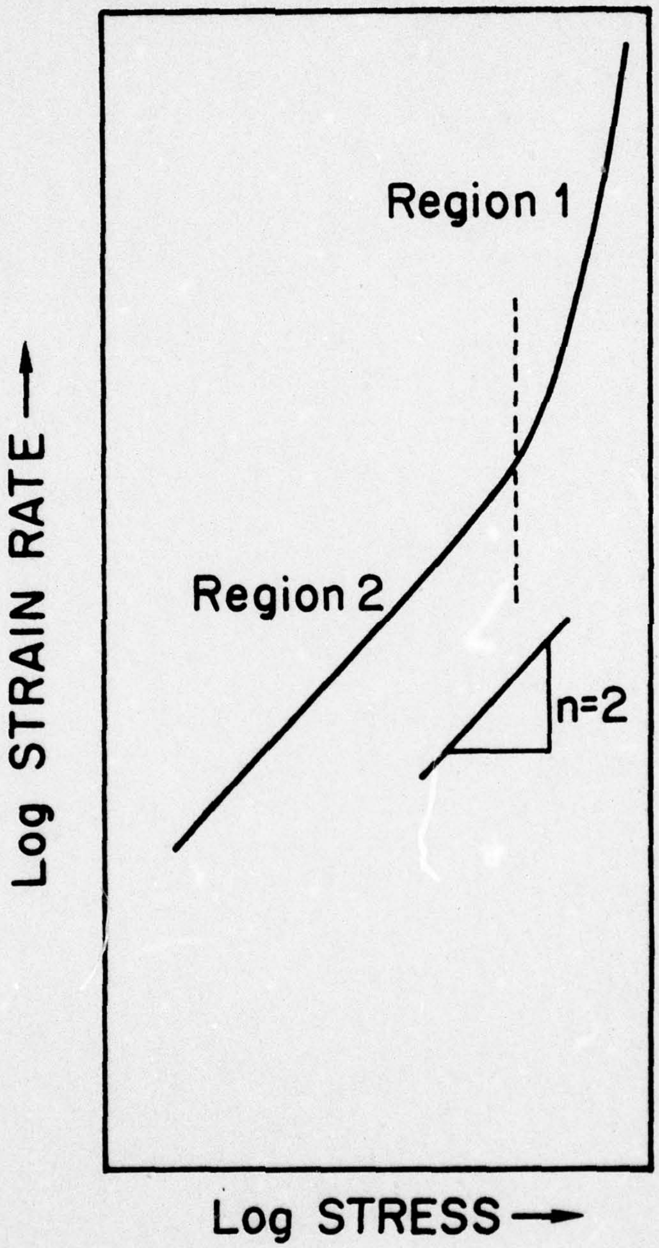


Figure 2. Schematic illustration of the strain rate-flow stress relationship in ultrahigh carbon steels.

MATERIALS AND PROCESSING

52100 material was purchased from Vasco Pacific in 25 lb. forged bars of approximate dimensions 12" x 2 3/4" x 1 1/2". The composition is given in Table 1. The as-received microstructure is one of coarse spheroidized cementite (~2μm) in a coarse ferrite matrix (~15μm).

The processing of the as-received material was designed to produce an ultrafine ferrite grain containing fine cementite particles - the single most important microstructural feature of superplastic UHC steels.

All of the methods developed involve mechanical working in the form of rolling. It is convenient to split the methods into two groups. In the first group, cold working of the steel is involved; in the second group, hot and warm working are involved. Details of all methods are shown in Table II.

Methods Involving Cold Rolling

i) 52100 material in the as-received condition was cold worked with intermediate anneals at 650°C. The reduction per pass was ~3%. The possibility of cracking during cold rolling necessitates the intermediate anneals which also cause recovery, recrystallization and refinement of the structure[4]. In an extension of this method, the material is again quenched from 770°C and further worked after annealing at 650°C.

ii) 52100 material is in this case quenched from 770°C (just above the A_1) to produce martensite containing cementite. Further annealing at 650°C produces a refined mixture of cementite in ferrite. This structure is then cold rolled at 3% per pass to a true strain of about $\varepsilon = -1.0$ with intermediate anneals where necessary.

iii) This method involves quenching the as-received 52100, but in this case from 900°C. At this temperature all the carbon is in solution

TABLE I

Composition of 52100, tool steel, weight percent

C	Si	Mn	Cr	S	P	Mo	Ni	Cu
1.005	0.28	0.41	1.42	0.001	0.01	0.05	0.15	0.12

TABLE II

Thermomechanical Processing Routes

A.	Cold Working Routes	Initial Thickness		Final Thickness	Strain, ε	Total Strain, ε_T
A1	As-Received 52100					
	Cold worked at 3%/pass	0.2503"	→	0.2090"	-0.18	
	Anneal at 650°C, 2 3/4 hr.					
	Cold worked at 3%/pass	0.2090"	→	0.1005"	-0.73	
						-0.91
A1a	As Above + 700°C water quench + anneal at 650°C, 1 hr					
	cold worked at 3%/pass	0.100"	→	0.0618"	-0.48	
						-1.39
A2	As-Received 52100					
	770°C Water quench					
	Anneal at 650°C, 1 hr.					
	cold worked at 3%/pass	0.2508"	→	0.2033"	-0.21	
	anneal at 650°C, 3 hr.					
	cold worked at 3%/pass	0.2033"	→	0.134"	-0.415	
	anneal at 650°C, 20 min.					
	cold worked at 3%/pass	0.134"	→	0.0985"	-0.308	
						-0.93
A3a	As-Received 52100					
	900°C Oil quench					
	anneal at 650°C, 1 hr.					
	cold worked at 3%/pass	0.2495"	→	0.224"	-0.108	
	anneal at 650°C, 3 hrs.					
	cold worked at 3%/pass	0.224"	→	0.115"	-0.667	
						-0.775
A3b	As-Received 52100					
	900°C Oil quench					
	Anneal at 650°C, 3 hrs.					
	Cold worked at 3%/pass	0.281"	→	0.226"	-0.218	
	anneal at 650°C, 2 1/2 hrs.					
	cold worked at 3%/pass	0.226"	→	0.124"	-0.600	
	anneal at 650°C, 1 hr.					
	cold worked at 3%/pass	0.124"	→	0.088"	-0.343	
						-1.16
A4	As-Received 52100					
	1120°C Oil quench					
	Anneal at 650°C, 2 hr.					
	cold worked, 10%/pass	0.45"	→	0.10"	-1.5	
	anneal at 500°C, 2 hr.					
						-1.5

TABLE II (cont'd)

B. Hot and Warm Working Routes

Warm Work Temperature	Hot Working				Warm Working				
	Initial Thickness	Passes	Final Thickness	ε_{α}	Initial Thickness	Passes	Final Thickness	ε_{α}	ε total
B1 650°C (1200°F)	1.36"	19 passes	0.223"	-1.81	0.223"	24 passes	0.0715"	-1.15	-2.96
B2 593°C (1100°F)	1.33"	14 passes	0.24"	-1.71	0.24"	23 passes	0.085"	-1.025	-7.735
B3 538°C (1000°F)	1.33"	19 passes	0.24"	-1.71	0.24"	23 passes	0.085"	-1.025	-2.735
B4 427°C (800°F)	1.33"	14 passes	0.479	-1.023	0.479"	30 passes	0.149	-0.761	-1.784
B5 427°C (800°F) followed by 316°C (600°F)	1.33"	14 passes	0.479"	-1.023	0.479"	12 passes at 800°F	0.319	-0.4065	
					0.319"	21 passes at 600°F	0.139"	-0.82	-2.25

but the temperature is comparatively low in the gamma-iron single phase field. The material is then annealed at 650°C and cold rolled to a true strain of about $\varepsilon = -1.0$ with intermediate anneals where necessary.

iv) This method duplicates the successful approach first attempted on UHC steels[4] where the material is quenched from a temperature high in the gamma region. Here, however, the 52100 was found to quench crack from 1120°C when oil quenched. However, some material was free of cracks and was cold worked after annealing at 650°C.

Methods Involving Hot and Warm Working

These methods are the most commonly used to develop fine structure in UHC steels.

The material is first homogenized at a temperature of 1100°C, in the γ range, where all the cementite is dissolved in the austenite matrix. This material is then continuously rolled in steps of about 10% per pass during cooling through the $\gamma + Fe_3C$ range to a temperature high in the $\alpha + Fe_3C$ range ($\sim$ 600°C $\rightarrow$ 650°C). This working comminutes the proeutectoid cementite into fine particles as it precipitates from solution. The material is then isothermally worked at warm temperatures, at between 5% and 10% per pass. In this investigation temperatures of 315°C (600°F) to 649°C (1200°F) were employed for isothermal rolling. This final rolling contributes further to the refinement of the cementite and the ferrite grains.

In both the cold and warm working approaches it is possible that a final structure containing both high angle and low angle (subgrains) boundaries results. The low angle boundaries cannot effectively contribute to superplastic flow. For this reason a final step was often added. This involves cycling the material through the A_1 transformation temperature ($\alpha \rightarrow \gamma$). This usually took the form of heating to 770°C and quenching into water. It is believed that this simple treatment converts the low angle boundaries to high angle ones and thus is beneficial to superplastic properties.

Specimens were machined from the final rolled strip as shown in Figure 3.

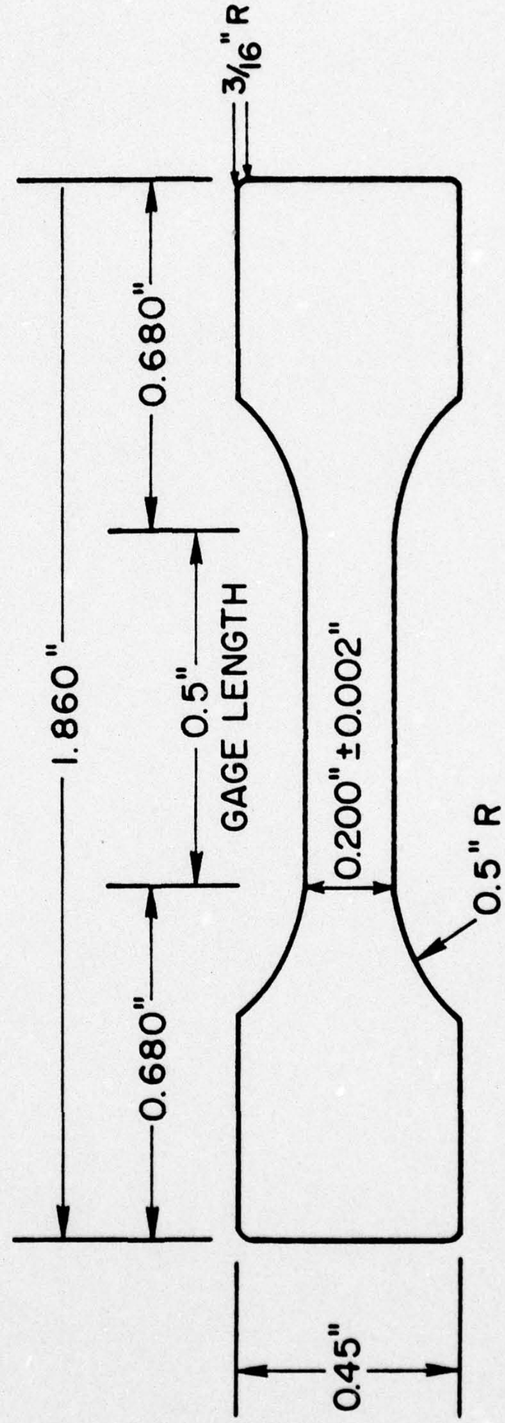


Figure 3, Specimen specifications for tensile testing apparatus.

EXPERIMENTAL PROCEDURE

Mechanical Testing

The warm temperature tension tests carried out to determine superplastic properties were done in an experimental set-up attached to an Instron machine[3]. In both types of test, the specimen was tested at 650°C in a reducing atmosphere of forming gas (90%N_2, 10%H_2). A dual-elliptical reflector, infrared furnace was used as the heating element.

In the first type of test the strain rate-stress relation is determined. This involves a change in strain rate test (or differential crosshead speed test) from which a value of strain rate sensitivity exponent can be determined. The steady state flow stress is measured at each of a number of imposed strain rates (10^{-5} sec^{-1} to 10^{-2} sec^{-1}). In these tests the specimen is first pulled at a low strain rate to about 20-30% deformation to establish a more or less constant structure (stable grain size etc.). The logarithm of flow stress, σ, is plotted as a function of logarithm of true strain rate, $\dot{\varepsilon}$. The slope of the resulting plot yields m, the strain rate sensitivity exponent.

In the second type of test, constant crosshead speed (decreasing true strain rate) tests to fracture were used to measure tensile ductilities. An initial engineering strain rate of 1%/min was used in these tests.

Stress relaxation tests were carried out during both strain-rate-change and elongation-to-failure tests. This involved stopping the crosshead during testing and measuring the decay of load with time. Interpretation of these tests can be carried out after Lee and Hart[7] or by a method described by Kayali[3]. Both yield a value of m, the strain rate sensitivity.

Metallography

Samples for optical microscopy were prepared in the standard manner. They were sectioned longitudinally and mounted in Bakelite. They were then ground on SiC paper and finally on 1μm Al_2O_3 and 0.05μm Al_2O_3. Samples were usually etched in nital. Pictures were taken on an optical microscope at a variety of magnifications.

RESULTS AND DISCUSSION

Type 52100 in the as-received condition consists of large grained ferrite (~15μm) containing cementite particles of about 0.5-2μm (Figure 4). This is not fine enough to be considered a superplastic structure. To establish a basis from which to measure improvements in properties, this material was tested at 650°C. A strain rate sensitivity value of m = 0.105 was found and in a second test, an elongation of 154% at an initial engineering strain rate of $\dot{e}_i$ = 1%/min was achieved.

a) Methods involving cold work

A simple processing route consisting of cold working the as-received steel was attempted (route A1, Table II). In Figure 5 it can be seen that considerable refinement of the matrix has occurred, the grain size being difficult to resolve and probably about 3 or 4μm. The size of the cementite particles is little affected by this treatment however and is still rather coarse. This is not surprising since the material has not been taken above the A_1, i.e. no carbon has been taken into solution. The properties of this steel and other cold worked routes are shown in Table III. Although the m value has increased, the elongation to failure is still under 200%. This probably reflects the fact that most of the refinement of the structure due to cold work is nullified upon reheating at 650°C. If this material is now cycled through the α-γ transformation (A_1) then the elongation is improved to 300% (Table III) and with further cold work after cycling (Type A1a, Table III) to 333%. This improvement is probably due to the fact that at 770°C cementite dissolves to allow the ferrite to become high carbon content (~0.85%) austenite (Figure 1). Upon quenching, a high carbon, fine martensite containing cementite is formed. Upon

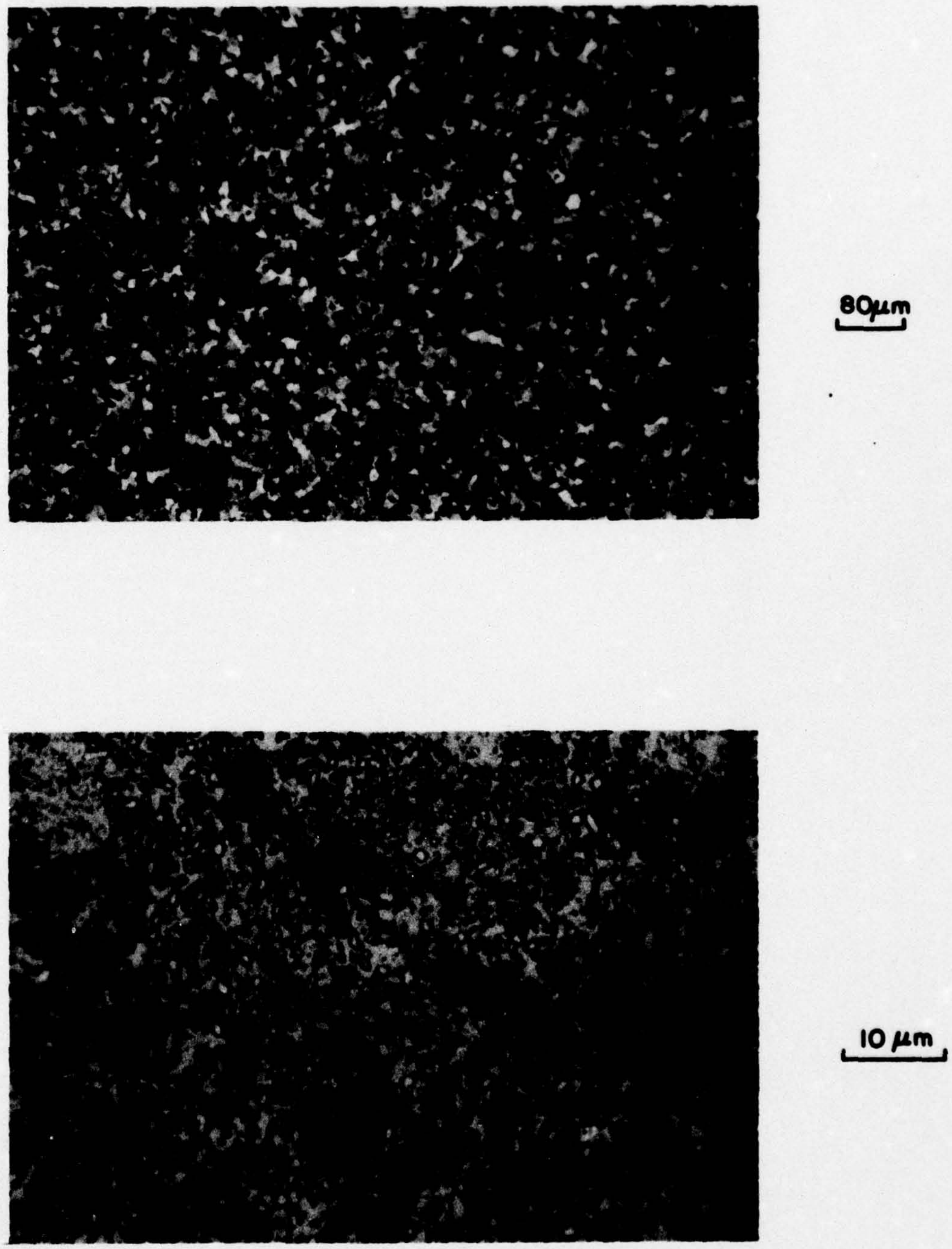


Figure 4. 52100 tool steel in the as-received condition. Coarse ferrite grains (~15μm) and coarse cementite particles (0.5-2μm).

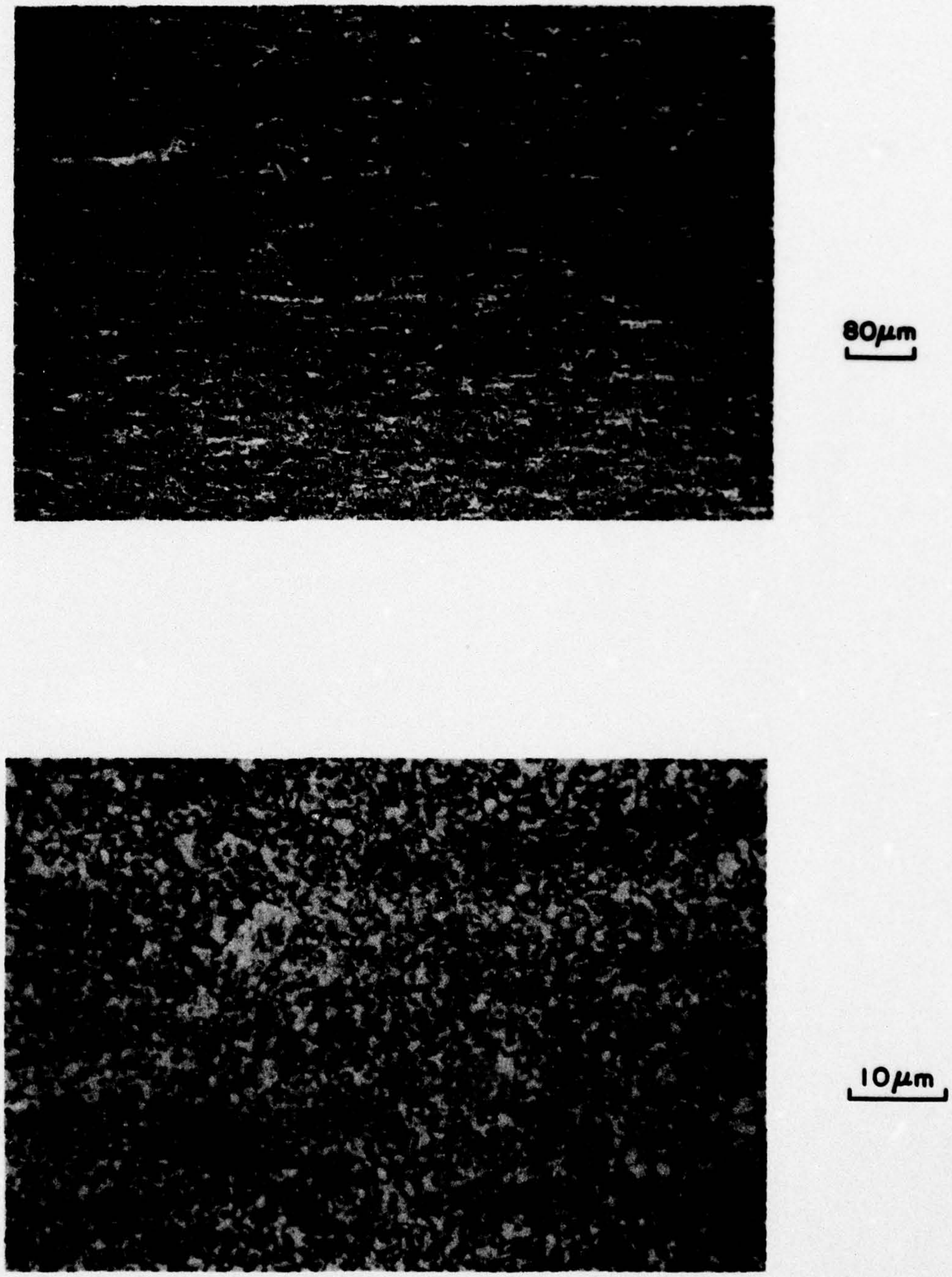


Figure 5. 52100 materials as received and then cold worked to a true strain of -0.91 (cold working route A1).

TABLE III

Superplastic Properties of 52100 after Cold Working Routes

Code	Processing	Strain Rate Sensitivity	Elongation %
A1	As-Received + Cold Work $\varepsilon = -0.91$	0.293	184
	As above + 1 cycle	-	300
A1a	As-Received + cold work ($\varepsilon = -0.91$) + 1 cycle + cold work ($\varepsilon = -0.48$)	-	333
A2	As-Received + 770°C cycle + cold work ($\varepsilon = -0.93$)	0.34	271
A3(a)	As-Received + 900°C cycle + cold work ($\varepsilon = -0.775$)	0.44	645
	As above + 1 cycle	0.48	500
A3(b)	As-Received + 900°C cycle + cold work ($\varepsilon = -1.19$)	0.47 0.49	547 527
A4	As-Received + 1120°C cycle + cold work ($\varepsilon = -1.5$)	0.4	505

annealing this structure the carbon precipitates as the martensite to ferrite transformation takes place and a refined structure results (Figure 6); upon further working, more pinning points (cementite) are available to prevent grain growth during the 650°C testing. It is also possible that high dislocation densities which form during cold working may form tangles of subgrains during heating after cold working, these low angle boundaries then transform to high angle boundaries upon heating above the A_1 and upon quenching retain their high angle nature thus further refining the structure since low angle boundaries cannot contribute effectively to superplasticity. This point will be raised again in discussing the warm worked steels.

It thus appears that at some stage during the processing the material must be taken above the A_1 transformation. This being so, Types A2, A3 and A4 structures (Table III) all involve first being treated at a temperature above the A_1 and quenched to cause carbon to enter into solution. A2 is quenched from 770°C. This temperature is just above the A_1. i.e. the least amount of carbon goes into solution and little grain growth of austenite occurs because cementite is present to restrict grain growth. Type A3 is quenched from 900°C; at this temperature, the material is fully austenitic and all carbon is therefore in solution. Type A4 is quenched from 1120°C, a temperature high in the γ range. In this case, quench cracking occurred but material was salvaged for rolling. After all these quenching procedures the material was cold rolled, after annealing, to a strain of about -1.0 to -1.5; these final structures are shown in Figures 7, 8 and 9. Intermediate anneals at 650°C were given where necessary (for details see Table II). All of the final structures are seen to be fine grained ferrite containing cementite.

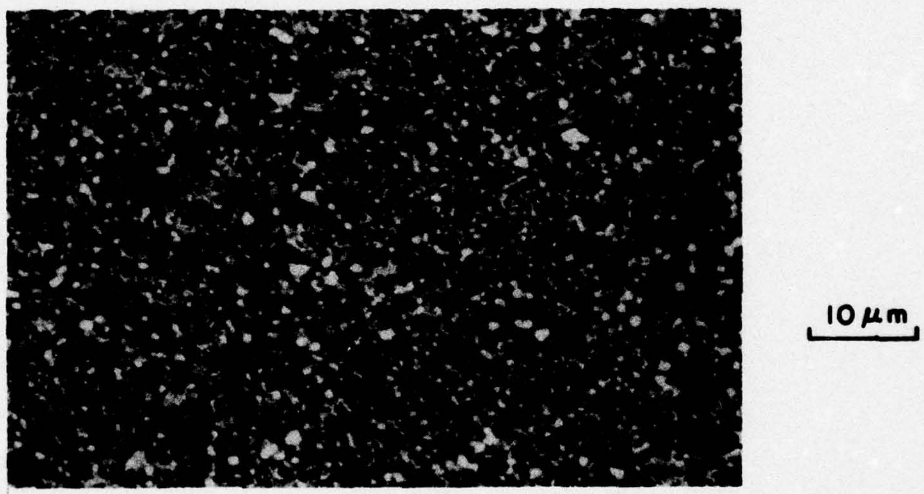


Figure 6. 52100 after cold working to ε = -0.91 plus one cycle (cold working route A1a).

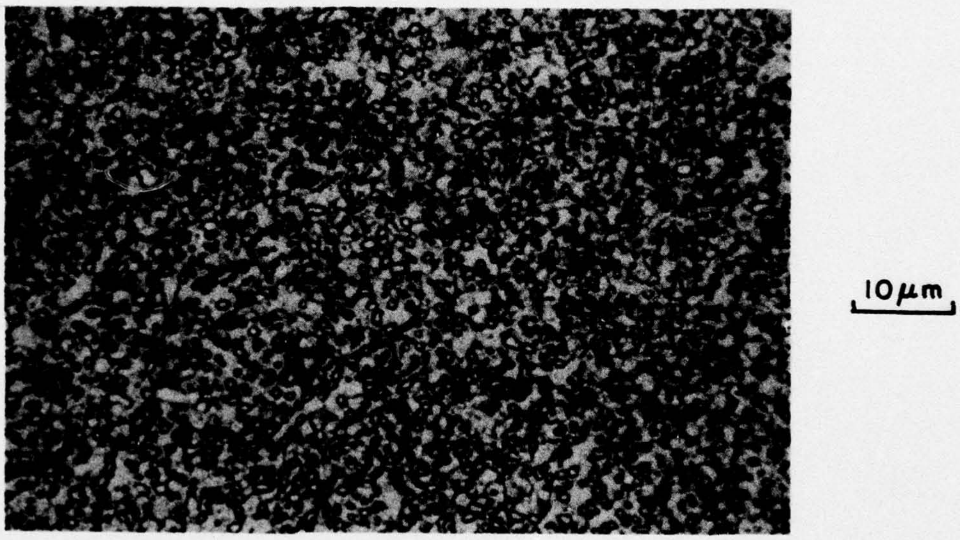


Figure 7. 52100 - cold working route A2. i.e. 770°C water quench followed by annealing and cold working to a strain of $\varepsilon = -0.93$.

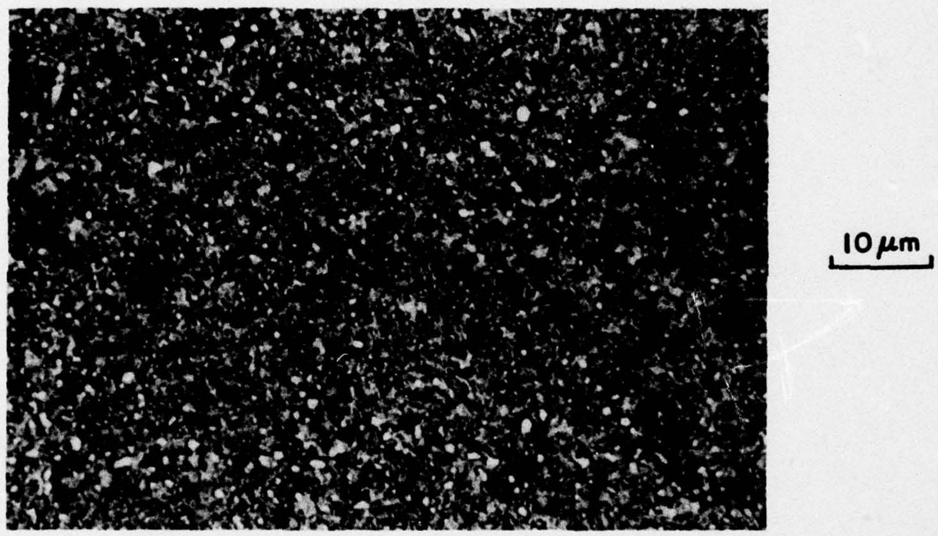


Figure 8. 52100 - cold working route A3a. i.e. 900°C oil quench followed by annealing and cold working to a strain of $\varepsilon = -0.775$.

Figure 9. 52100 - cold working route A4. i.e. 1120°C oil quench followed by annealing and cold working to a strain of ε = -1.5.

A series of micrographs illustrating the development of fine structure in the 900°C oil quenched series (A3) is illustrated in Figure 10. Upon quenching the as-received steel from 900°C, coarse martensite plates are produced. After annealing and cold work a more familiar ferrite + cementite structure is obtained. Upon cycling this material, a 'quasi' martensite is formed, i.e. a martensite that is so fine that it is not resolvable in the optical microscope[9]. Upon annealing this structure, a ferrite + cementite aggregate is once again produced.

The properties of materials initially quenched from above the A_1 prior to working are listed in Table III. The elongations-to-failure are seen to be improved considerably for material quenched from 900°C and 1120°C but not much improved for material quenched from 770°C. A maximum ductility of 645% was achieved for a material quenched from 900°C prior to cold working. The influence of cycling on these materials is not very significant, slight deterioration in elongation-to-failure being observed despite slight increases in values of strain rate sensitivity. This point will be further discussed in the next section on materials that have been warm worked.

b) Methods Involving Warm Work

The microstructures developed after hot and warm working are illustrated in the following figures and the accompanying properties in Table IV.

In these routes, all steels were deformed during cooling from about 1100°C to about 600°C. They were then isothermally rolled at one of five temperatures. The final microstructures for materials isothermally rolled at 649°C (1200°F), 593°C (1100°F) and 538°C (1000°F) are shown in Figures 11, 12 and 13. The structures are seen to be very fine for all three materials. Banding is observed also. These areas may contain some low

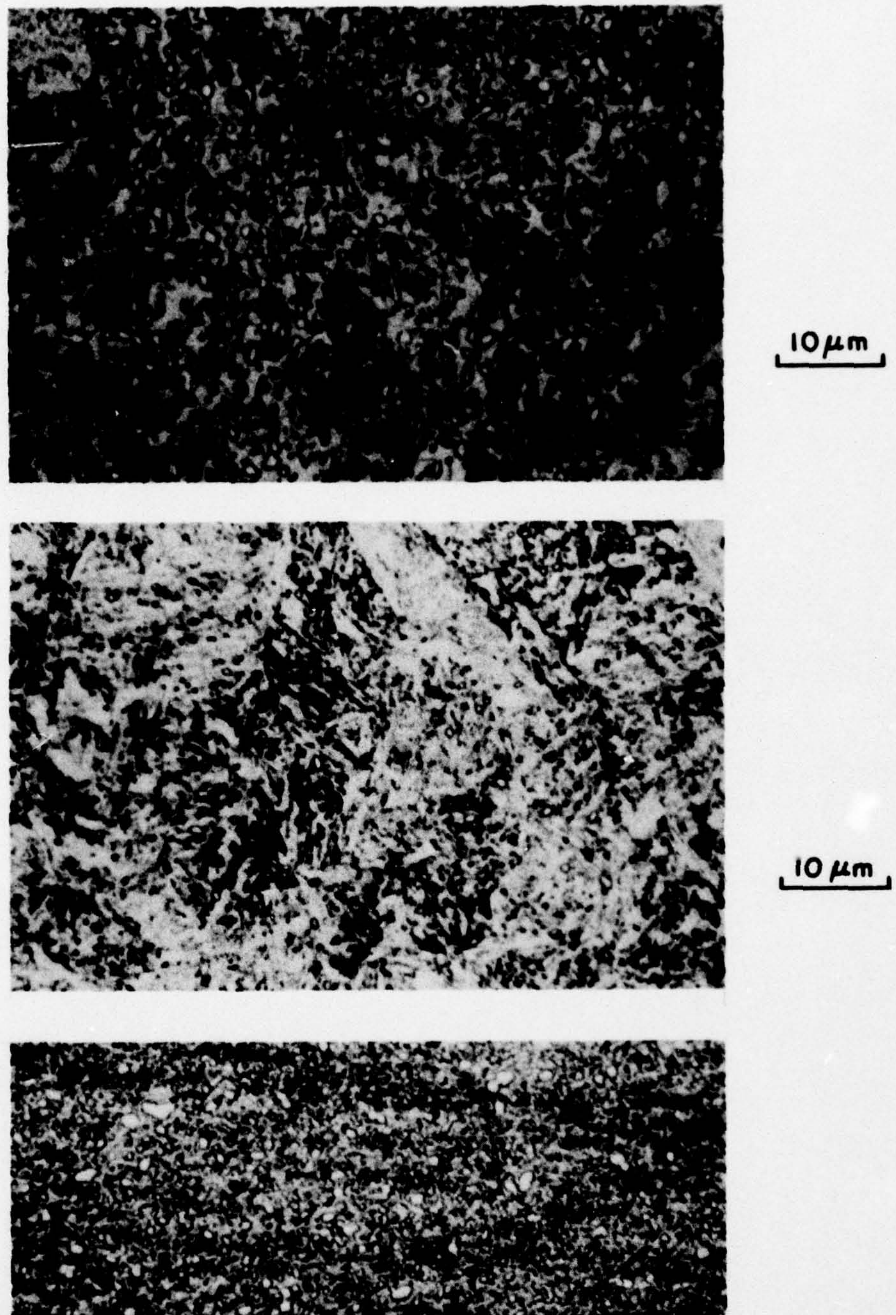
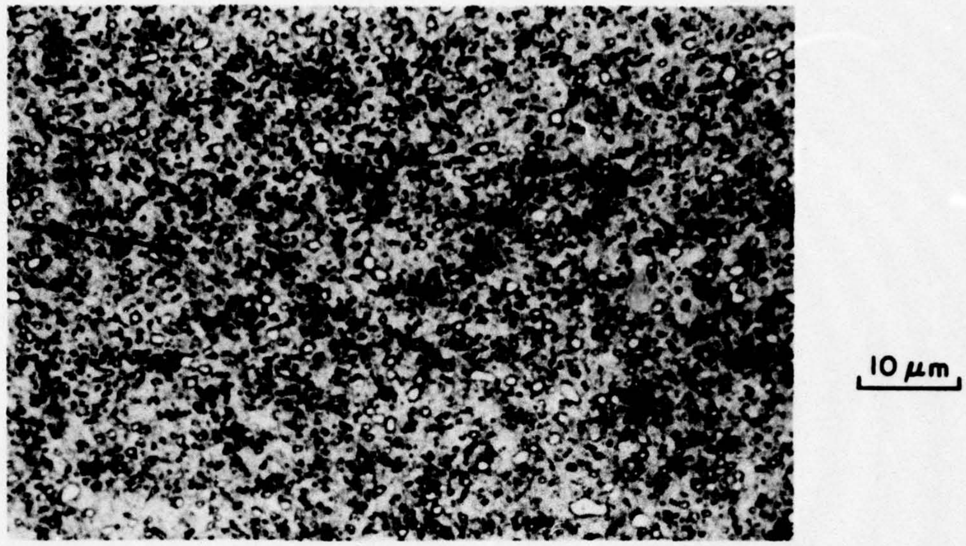


Figure 10. 52100 - Micrographs illustrating cold working method A3b. Top - 52100 As received - Center, Oil quenched from 900°C and Bottom, Annealed at 650°C and cold worked (ε = -1.16) -- (continued on next page).



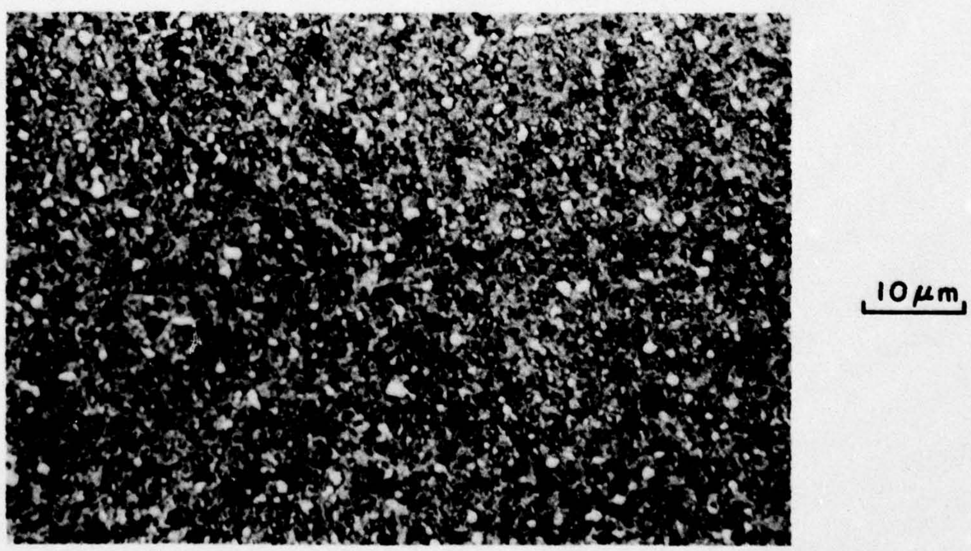


Figure 10. (cont'd)
Top - Quenched from 770°C
Bottom - Annealed at 650°C

TABLE IV

Superplastic Properties of 52100 after Hot and Warm Working Routes

Code	Processing	Strain Rate Sensitivity	Elongation %
B1	γ work + α work at 650°C(1200°F) cool	0.28	358
	+ 1 cycle	0.4	610
	+ 5 cycles	0.46	571
B2	γ work + α work at 593°C(1100°F) cool	0.22	310
	+ 1 cycle	0.37	533
B3	γ work + α work at 538°C(1000°F) cool	0.21	230
	+ 1 cycle	0.38	731
B4	γ work + α work at 427°C(800°F) cool	0.29	205
	+ 1 cycle	0.465	700
B5	γ work + α work at 427°C(800°F) cool	0.27	412
	+ α work at 316°C(600°F)		
	+ 1 cycle	0.44	565

Figure 11. 52100 - Hot and warm worked route B1 material isothermally rolled at 650°C (1200°F).

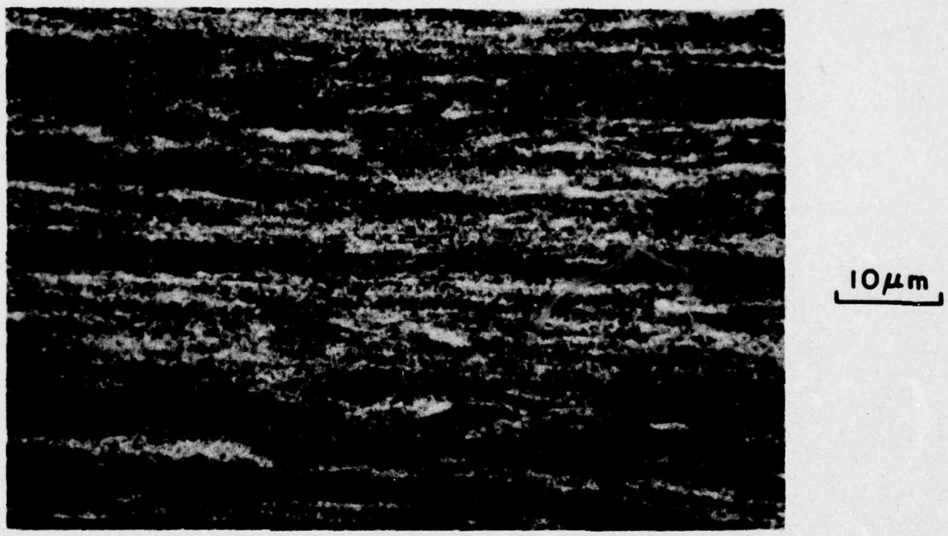


Figure 12. 52100 - Hot and warm worked route B2 material isothermally rolled at 593°C (1100°F).

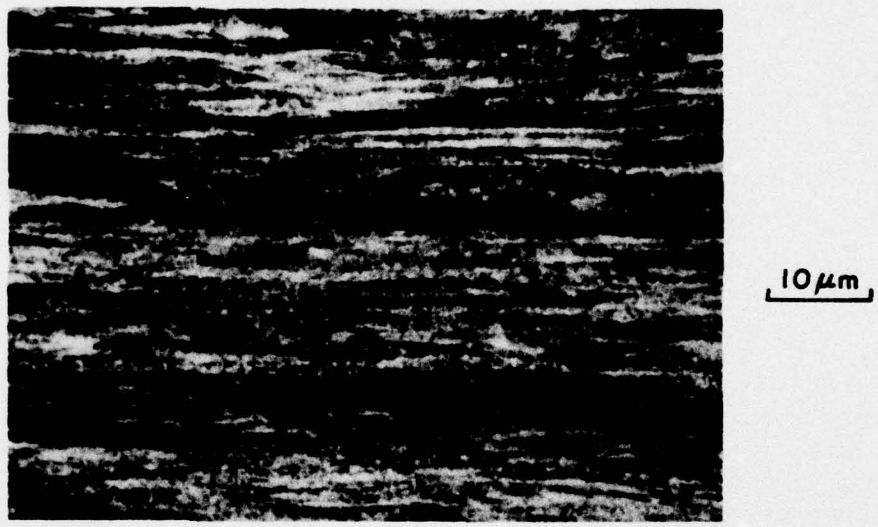


Figure 13. 52100 - Hot and warm worked route B2 material isothermally rolled at 538°C (1000°F)..

angle boundaries. It is believed that the conversion of these low angle boundaries to high angle boundaries should be beneficial to warm temperature properties (i.e. superplasticity) since low angle boundaries are unable to contribute to grain boundary sliding, the major mechanism of superplastic deformation. These bands and the means of their removal are shown in Figures 14 and 15 for materials isothermally rolled at 427°C (800°F) and 316°C (600°F). Upon cycling through the $\alpha \rightarrow \gamma$ transformation temperature at 723°C (i.e. the A_1) low angle ferrite boundaries are converted to high angle austenite ones. Quenching from above the A_1 prevents undersirable transformation products, such as pearlite, from forming. Upon annealing at temperature below the A_1, a fine grained ferrite + cementite structure containing high angle boundaries results and this should be better for superplastic behavior. The structures in Figures 14 and 15 are clearly seen to be more homogeneous after cycling through 770°C.

The superplastic properties listed in Table IV lend support to the above ideas.

Material rolled at 649°C (1200°F) has a rather poor strain rate sensitivity value of 0.28 and an elongation of 358%. Upon cycling once the 'm' value is increased to 0.4 and the elongation to failure nearly doubled (610%). This improvement is attributed to the conversion of subgrains to high angle boundaries. Further cycling, despite increasing the strain rate sensitivity value to 0.46 causes a decrease in the elongation to failure (571%). This is a curious result and as yet unexplained. Repeated cycling could be expected to coarsen the structure due to grain growth above the A_1. This coarsening however would be expected to manifest itself in the strain rate sensitivity measurements. The fact that it does not may reflect a shortcoming in the method of

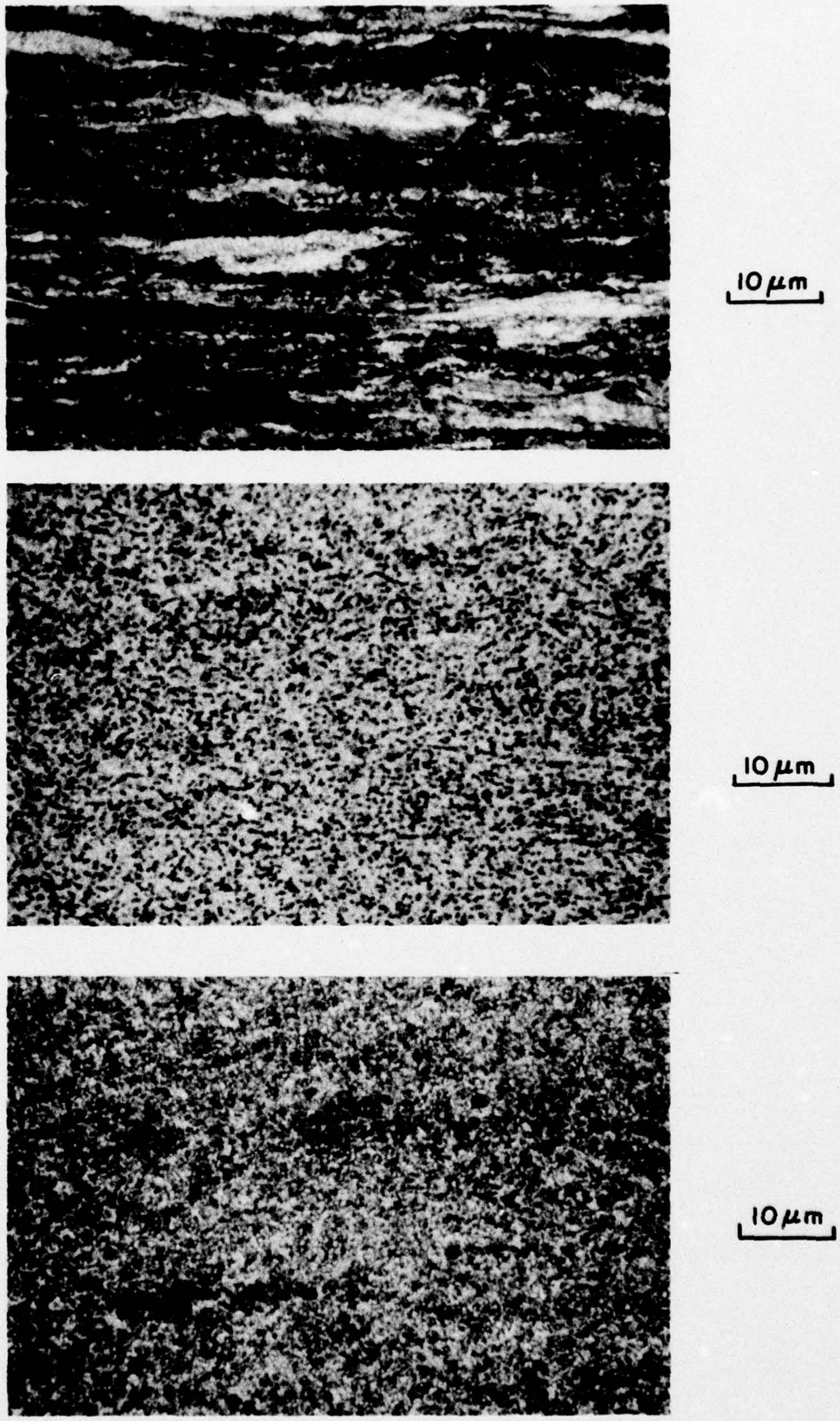


Figure 14. 52100 - Hot and warm worked route B4
Top - As rolled at 427°C (800°F)
Center - As quenched from 770°C
Bottom - After annealing at 650°C

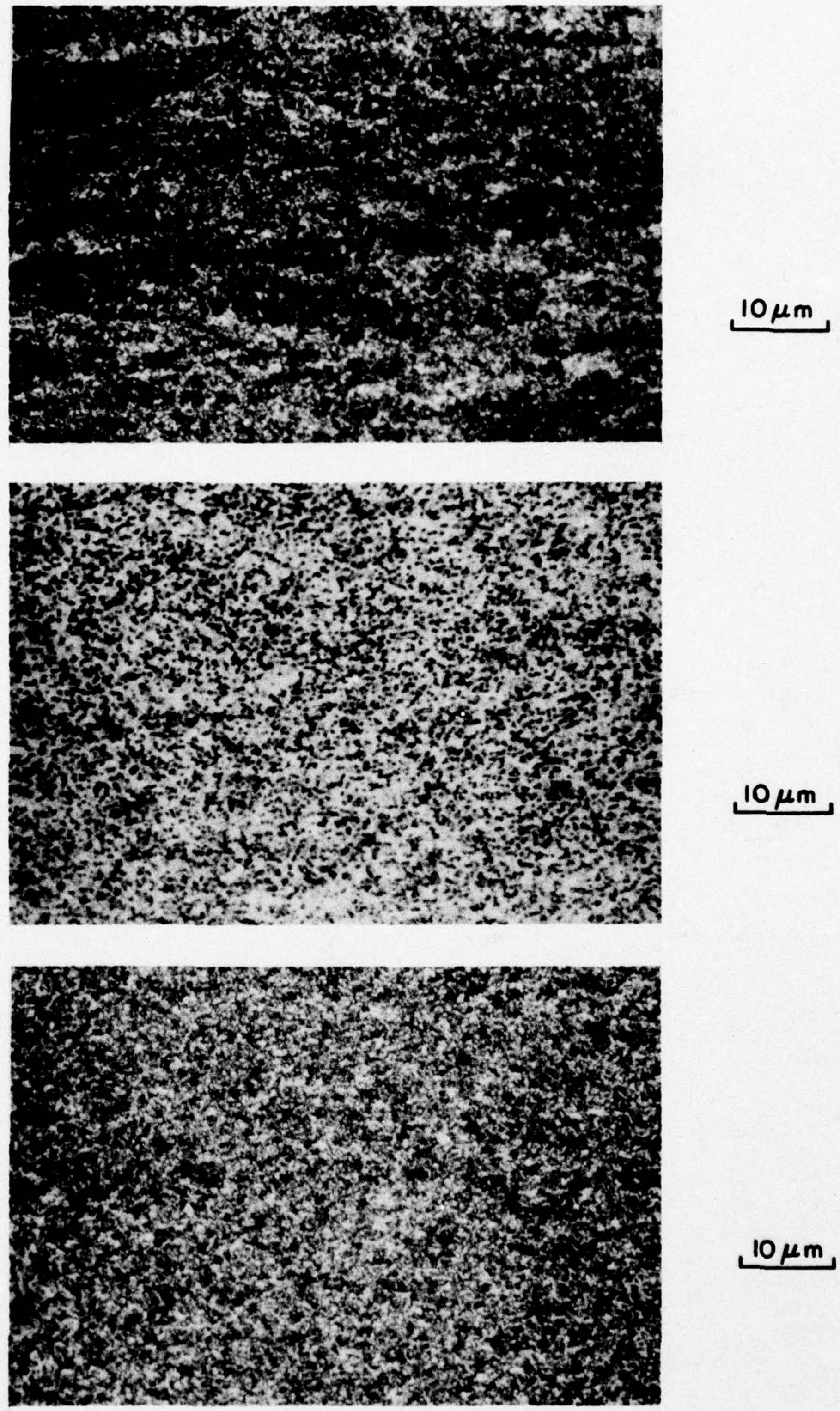


Figure 15. 52100 - Hot and Warm worked route B5.
Top - As rolled at 316°C (600°F)
Center - As quenched from 770°C
Bottom - After annealing at 650°C

determining strain rate sensitivities during short term tests as done here. It is hoped that current work involving strain rate sensitivity determinations from stress relaxations during elongation to failure tests will resolve this point. Figure 16 illustrates how strain rate sensitivity varies as a function of cycling. Here the flow stress is plotted as a function of imposed strain rate for material in the as-rolled at 649°C (1200°F), as-rolled at 649°C (1200°F) + 1 cycle and as-rolled at 649°C (1200°F) + 5 cycles. At high strain rates the differently treated materials have similar strengths. At low strain rates however the cycled material is considerably weaker. When tested at a constant crosshead speed the material which underwent 5 cycles has the lowest flow stress, Figure 17. This series of experiments has been repeated several times[8] and the same results occur. It is suggested that grain growth proceeds more rapidly in the material cycled five times and that this does not manifest itself in a short term test but in a long term test results in premature failure.

All the other warm worked materials were found to exhibit rather low ductilities in the as-rolled condition but excellent ductilities after a single cycle. A maximum ductility of 731% was found in material isothermally rolled at 1000°F and then cycled, Figure 18. A value of 700% was found in material isothermally rolled at 800°F and cycled. The improved microstructures apparent in Figure 14 plainly indicate that a more homogeneous structure is obtained after cycling. The removal of low angle boundaries is proposed as part of the reason for this improvement. It is envisaged that current transmission electron microscope work[10] will clarify this proposed explanation.

An attempt was made to correlate all the measured strain rate

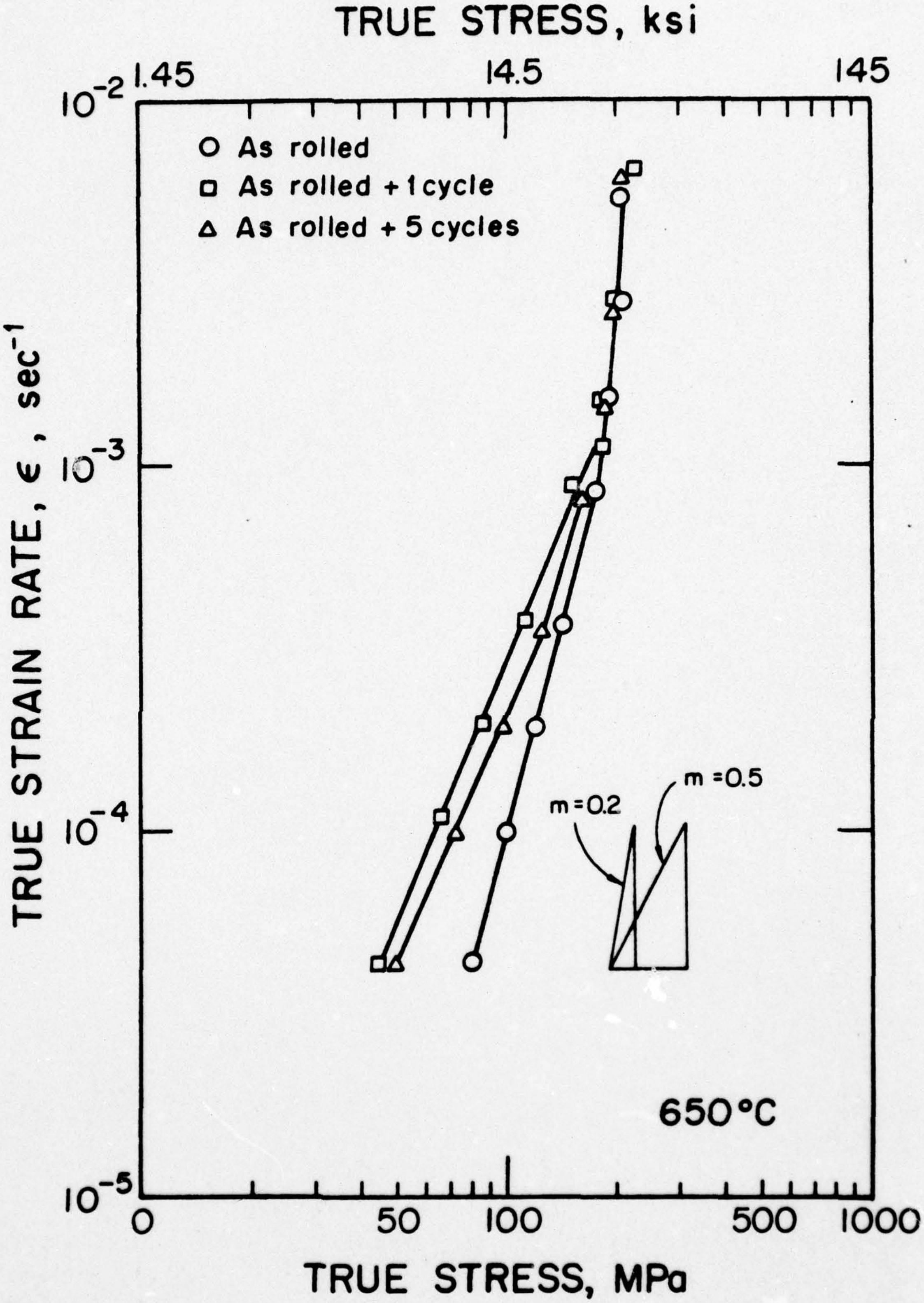


Figure 16. The flow stress, σ, as a function of imposed strain rate, $\dot{\varepsilon}$, for 52100 in the as rolled (B1 route), as rolled + 1 cycle and as rolled + 5 cycle conditions.

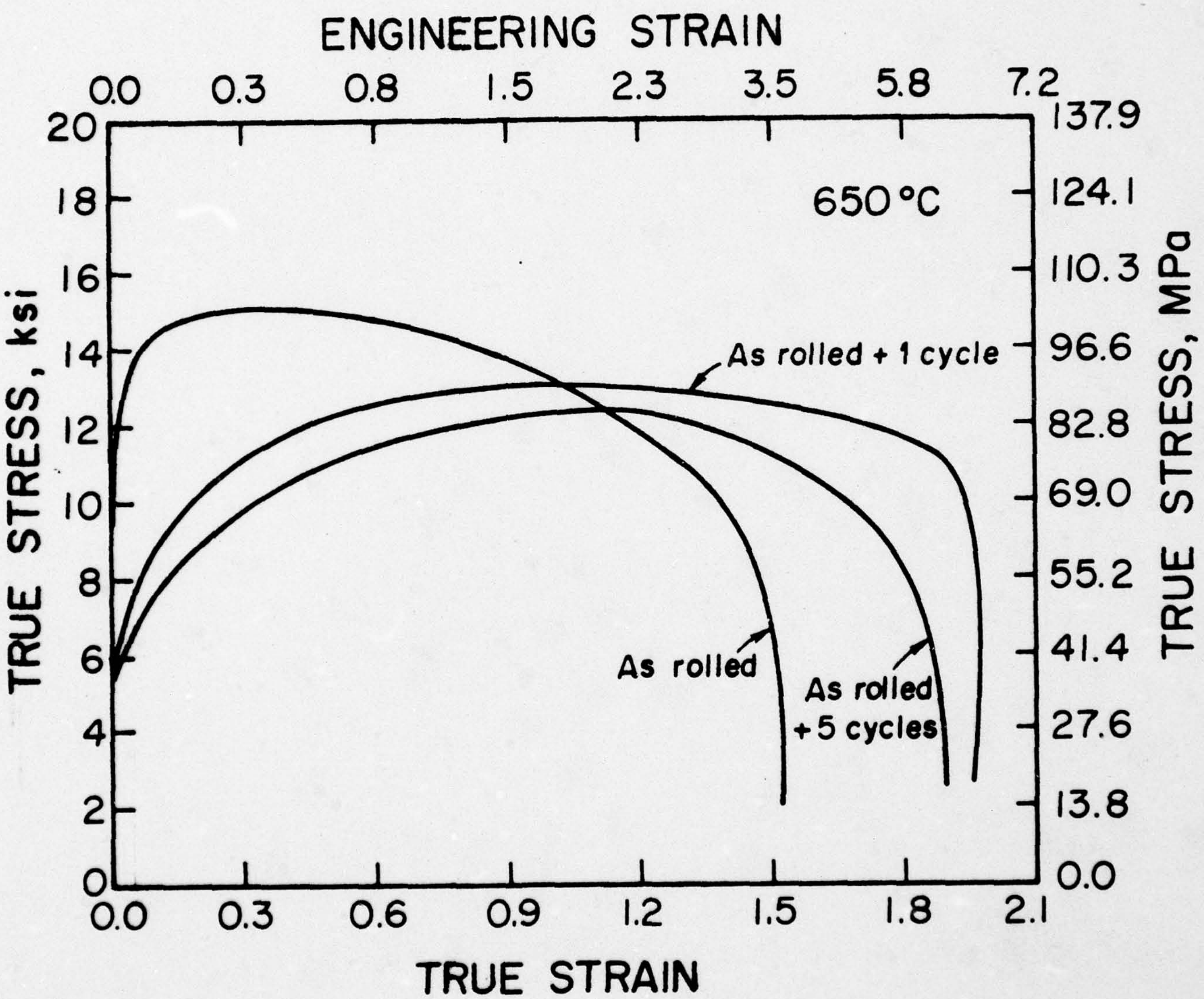


Figure 17. True stress versus true strain for elongation to failure tests in 52100 material in the as rolled (B1), as rolled + 1 cycle and as rolled + 5 cycle conditions.

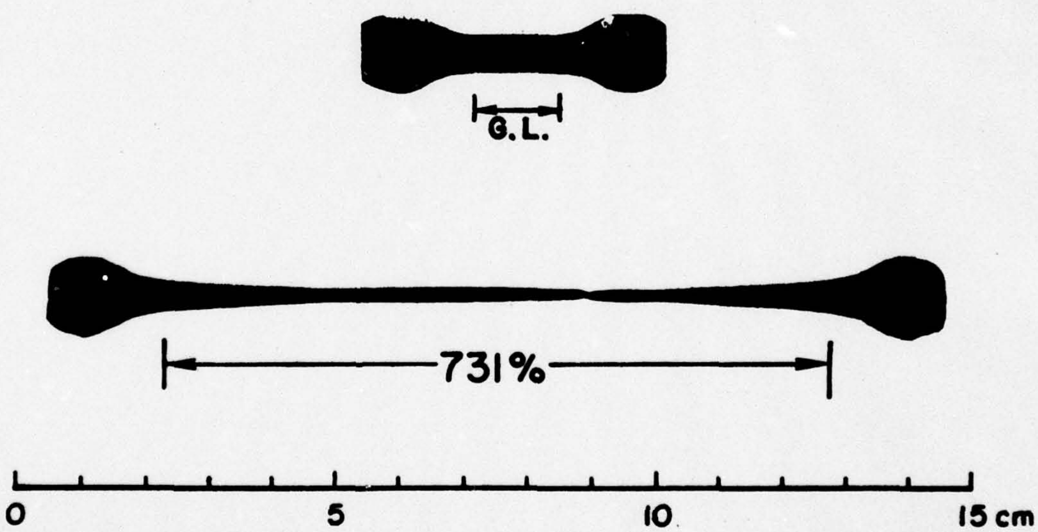


Figure 18. 52100 in the B3 + 1 cycle condition tested to failure at 650°C under an imposed initial engineering strain rate of $\dot{e}_i$ = 1%/min.

sensitivities with corresponding elongations-to-failure. Figure 19 shows that despite considerable scatter the expected trend is evident. Increasing strain rate sensitivities leads to higher elongations.

An interesting microstructural observation is that 52100 material exhibits necking down to a considerable degree during superplastic flow and reduction of area of nearly 100%. This is unlike some of the plain carbon steels where a relatively abrupt failure occurs despite high elongations[1,2] and a finite reduction of area is noted.

Finally it should be pointed out that these materials have excellent room temperature properties. They exhibit high yield strengths (typically 130 ksi), high ultimate tensile strengths (typically 150 ksi) and good ductilities (8% to 30% depending on whether the material is annealed or not). Work in progress shows that the properties are strongly influenced by finishing rolling temperatures.

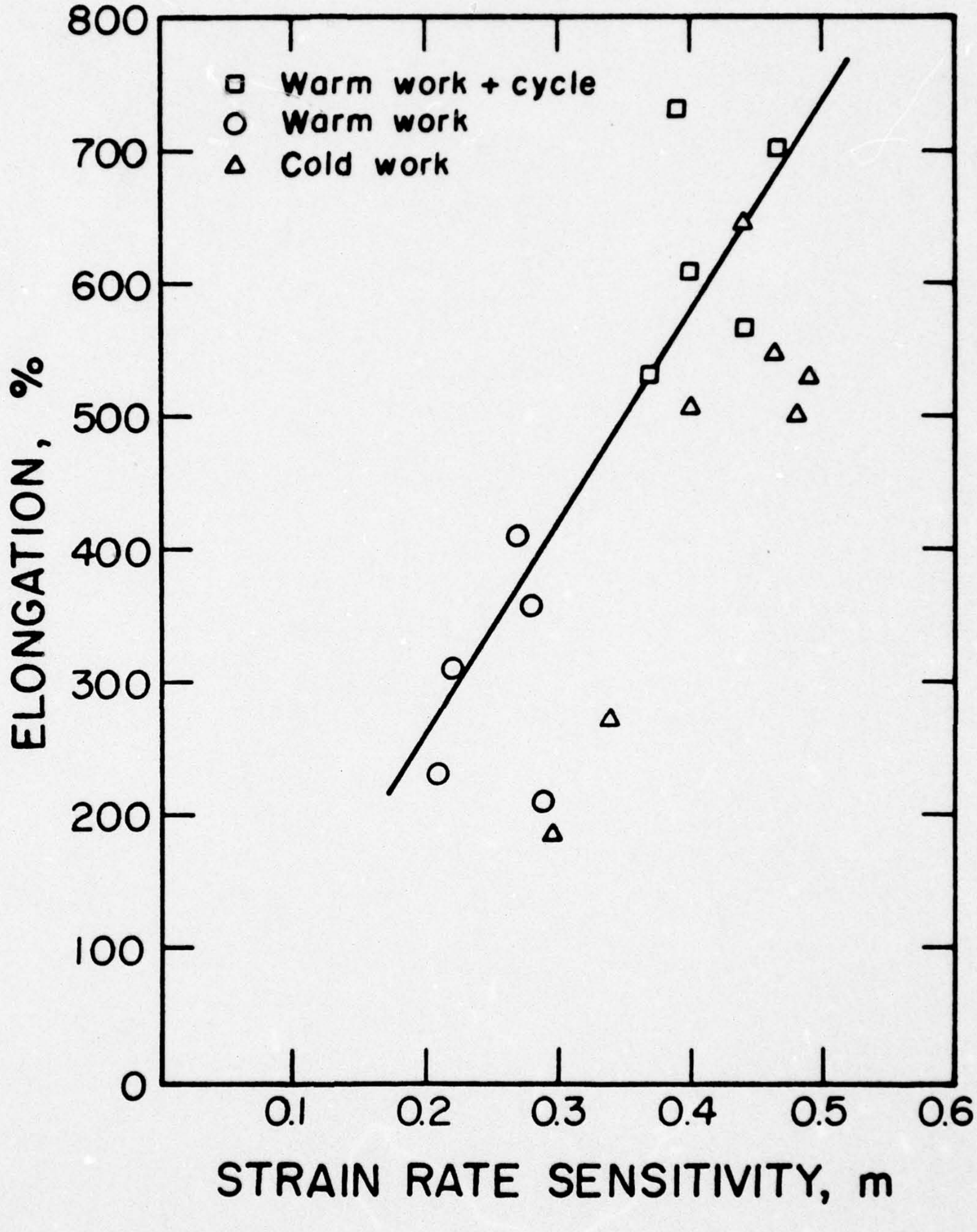


Figure 19. Strain rate sensitivity, m, versus elongation to failure for all tests.

CONCLUSIONS

1. The development of superplastic properties in 52100 type tool steel has been investigated.

2. The development of a structure that is superplastic can be readily achieved in this material by methods involving either a) cold working or b) hot and warm working.

3. This superplastic structure consists of a matrix of very fine ferrite grains containing very fine cementite particles. Typically the ferrite grain size is about 1μm and the cementite particle size about 0.1μm.

4. In methods involving cold work, it was found to be beneficial to take the as-received material to a temperature above the A_1 at some stage in the thermal mechanical processing to allow carbon to go into solution. An optimum elongation of 645% was found after a process involving annealing and cold work following a quench from 900°C.

5. In methods involving warm work it was discovered that subgrains formed. It is postulated that these do not contribute to superplastic deformation modes and hence are deleterious. This postulation is supported by the fact that thermal cycling above the A_1 - which converts the subgrains to high angle boundaries - enhances greatly the superplastic porperties. An optimum elongation of 731% was found for material isothermally warm worked at 538°C (1000°F) and then cycled.

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Influence of Ni, Cr, and V on superplasticity in ultrahigh carbon steels

J. Wadsworth and O.D. Sherby

ABSTRACT

The influence of small additions of nickel, chromium and vanadium upon the superplastic properties of ultrahigh carbon steels has been investigated.

Nickel is a graphite forming element whereas chromium and vanadium are carbide stabilizers. This difference is reflected in the microstructures both during the warm working leading to the fine structure development and during superplastic deformation. After extensive warm working, nickel-containing steels exhibit graphitization whereas chromium and vanadium-containing steels do not and very fine structures are developed in these latter steels.

Upon superplastic testing, graphitization is enhanced in nickel-containing steels and this is detrimental to properties. Increased grain growth of the ferrite is also observed due to cementite depletion as the graphitization occurs. Good superplastic properties were found in both the chromium and vanadium-containing steels. An elongation to failure of 817% was achieved in the latter steel at 650°C. All the steels were found to have better properties at temperatures below the A1 transformation temperature.

Room temperature properties have also been assessed. High strengths were found with good associated ductilities.

Influence of Ni, V and Cr on Superplasticity in Ultrahigh Carbon Steels.

J. Wadsworth and O. D. Sherby

INTRODUCTION

The development of cheap structural materials which will deform to large strains under small externally applied stresses during fabrication has been a constant goal for materials scientists. In theory, superplasticity can be said to represent the achievement of this goal. In practice,however, problems arise which have limited the range of use of superplastic materials and in general they cannot be said to have fulfilled their full engineering potential. Either the materials have low strength at room temperature (e.g. Zn - 22Al) or are expensive alloys (e.g. In 100, Ti-Al-4V). Recently however, a new class of materials which overcomes both of the above objections has been made superplastic[1,2,3]. These are steels containing 1.0%C to 1.9%C and the other usual steel-making elements, manganese, silicon and sulfur. Impressive room temperature properties combined with low cost make these ultrahigh carbon steels unique in the field of superplasticity. It is the purpose of this paper to describe the first attempts (not always successful ones) to improve further the properties of these materials by small alloying additions.

SUPERPLASTICITY

The word superplastic is used to describe the property of certain polycrystalline materials to deform to extraordinary elongations (several hundred percent) in tension. Two basic types of superplasticity exist, internal stress superplasticity and fine structure superplasticity. This latter type refers to the extensive elongations that can occur in metallic systems in which a fine grain structure exists. It is this type of superplasticity which is evident in the work to be described here and to which attention will now be directed.

A definition of superplasticity is contained in the equation relating flow stress, σ_f, and strain rate, $\dot{\varepsilon}$;

$$\sigma_f = k\,\dot{\varepsilon}^m$$

where k is a constant and m is the strain rate sensitivity.

Strain rate sensitivity is a fundamental parameter in an applied mechanics interpretation of plasticity since when $m = 1$ the material acts as a Newtonian fluid (such as glass, molasses etc.) with, in consequence, high plasticity. Usually materials exhibit a value of ~0.2. Typical values for superplastic materials lie in the range $0.4 < m < 0.6$. Strain rate sensitivity, m, has a reciprocal relationship with n, the stress exponent, found in high temperature flow equations.

To achieve the desirable high strain rate sensitivities at warm temperatures ($0.5 - 0.65T_m$), materials with a fine equiaxed grain structure are required. At warm temperatures it is difficult to maintain a fine grain size without the presence of a second phase. For this second phase to be effective it is necessary that the strength of the second phase and the matrix be similar at the temperature of superplastic flow. These prerequisites for superplasticity are often to be found in eutectic or eutectoid alloys. Such alloys can be made fine-grained by appropriate heat treatment and mechanical working. Steels containing amounts of carbon in excess of the eutectoid composition (> 0.8%C) have been made superplastic by producing particulate composite structures of cementite (~0.1μm) in fine grained ferrite (~1μm)[1,2,3]. Eutectoid steel contains about 12 volume % of cementite which is generally insufficient to effectively restrict grain growth although these alloys can be made superplastic[4]. By increasing the amount of carbon and hence cementite (1.3%C - 1.9%C corresponds to 20 vol.% - 30 vol.% cementite) grain growth is restricted and superplastic flow occurs to a greater degree[1,2].

It is of interest to note that pure alloys of iron and carbon cannot be made superplastic since the steel-making impurities (e.g. Mn, Si, S) are essential to prevent cementite and ferrite grain coarsening[4].

It should be noted that increasing the carbon level increases the possibility of agglomeration of the cementite or of its reversion to graphite during thermo-mechanical processing.

MATERIAL

Six ultrahigh carbon steels were chosen for this investigation. Four of them, containing 1% Ni, were supplied by Jones and Laughlin Steel Corporation. One of these steels also contained a small amount of vanadium and some rare earth additions. (mainly cerium and lanthanum). The silicon level of all the steels was inadvertently high and the manganese rather low. The fifth steel, a 52100 tool steel, was supplied by Vasco Pacific Steel Company and contains 1 1/2% of chromium. The final steel contained a small amount of vanadium the compositions of all the steels are shown in Table I.

The addition of nickel was made to the four high carbon steels because of this element's beneficial influence on hardenability. As has already been stated, the maintenance of a fine grain size is essential for fine structure superplasticity. The addition of vanadium was made in an attempt to precipitate vanadium carbide on grain boundaries thereby pinning them and restricting grain growth during superplastic deformation.

The chromium addition to 52100 tool steel enters the cementite to form a stable carbide of the form $(Fe,C)_3C$[5] which it is believed should be beneficial in the attainment and maintenance of a fine grain size.

EXPERIMENTAL

i) Preparation of Fine Grain Size

All steels were thermo-mechanically processed at Lockheed Inc. The

TABLE 1

COMPOSITIONS OF THE STEELS

C	Mn	Si	S	P	Ni	Cr	Cu	Ti	V	other
1.32	0.38	0.41	0.015	0.03	1.04	0.02	0.03	0.01	0.01	-
1.56	0.39	0.49	0.015	0.03	1.09	0.02	0.04	0.03	0.01	-
1.88	0.39	0.53	0.018	0.03	1.07	0.02	0.04	0.03	0.01	-
1.26	0.37	0.42	0.015	0.03	1.09	0.02	0.03	0.02	0.10	-
1.005 (52100)	0.41	0.28	0.001	0.010	0.15	1.42	0.12	-	-	Mo 0.05
1.37	1.04	0.22	0.005	0.01	0.07	0.03	0.01	0.01	0.12	-

treatment was a two-stage one. The first stage consisted of rolling the steel continuously in the $\gamma + Fe_3C$ range, during cooling to the $\alpha + Fe_3C$ range, in steps of ~ 10% / pass to a true strain of about 1. The homogenizing starting temperature in the gamma plus cementite range was about 1100°C and the finishing temperature about 600°C. This work, referred to as $(\gamma w)_{cool}$, breaks down the pro-eutectoid cementite into fine spheroidized form as it precipitates from solution and also refines the gamma grains. The second stage consists of isothermally rolling the steel high in the $\alpha + Fe_3C$ range (referred to as α work) to a final thickness of ~ 0.1" at reductions of ~ 8% / pass. This breaks down the pearlite structure formed during the austenite to pearlite transformation and further refines the cementite structure. The final structure is a fine particulate composite of cementite in ferrite. The total true strain involved in developing these structures is ~2.5 - 3.0.

Details of all the rolling schedules are shown in Table 2.

ii) Testing Procedure

Tests were carried out in tension on specimens machined from the final rolled strip. Gage lengths of 1" were used in the determination of strain rate sensitivity and 1/2" gage lengths in the determination of elongations-to-failure tests. All tests were carried out in an atmosphere of forming gas ($10\%H_2$, $90\%N_2$) on an Instron testing machine on apparatus described elsewhere(4). Tests were carried out at both 650°C ($\alpha + Fe_3C$ range) and 770°C ($\gamma + Fe_3C$ range)

a) Determination of strain rate sensitivity

Two techniques were used to determine strain rate sensitivity. In the first a standard testing procedure was used to determine the flow stress at each of a number of strain rates. The material is first deformed to a large strain to obtain a steady state structure. Change in strain rate tests are then performed over small strain increments with the assumption that little

TABLE 2

ROLLING RECORD

Composition	Gamma work - (γ_w) cool				Alpha work at 650°C-(α_w)650			Total Strain $\varepsilon_T = \varepsilon_\gamma + \varepsilon_\alpha$
	t_o, in.	t_1, in	Number of Passes	true strain in(γ_w)cool ε_γ	t_2, in	Number of passes	true strain in(α_w)650 ε_α	
1.32%C + 1%Ni	1.60	0.432	14	-1.3	0.099	23	-1.47	-2.77
1.56%C + 1%NI	1.52	0.23	16	-1.9	0.085	15	-0.99	-2.89
1.88%C + 1%Ni	1.43	0.206	15	-1.93	0.065	15	-1.15	-3.08
1.26%C + 1%Ni + 0.1%V	1.34	0.336	13	-1.39	0.097	21	-1.24	-2.62
1.0%C + 1.5%Cr (52100)	1.36	0.225	19	-1.81	0.070	24	-1.51	-2.96
1.37%C + 0.12%V	1.375	0.272	15	-1.62	0.079	14	-1.23	-2.856

t_o - starting thickness

t_1 - thickness after (γ_w) cool

t_2 - final thickness

grain growth occurs during these strain rate tests. Grain size is an important factor in determining the flow stress of superplastic materials since the stress, σ, is proportional to grain size$^{1.25}$. The choice of strain rates is governed by the available crosshead speeds of the testing machine. At each of the crosshead speeds used the specimen was extended to a strain at which the stress was known to achieve a constant value. Measurements were carried out and repeated at various values as indicated in the schedule below. Approximate values of engineering strain are shown in brackets for each of the crosshead speeds employed.

0.1"/min. (20%), → 0.002"/min. (1%), → 0.005"/min. (1.5%),
0.01"/min. (1%), → 0.02"/min. (2%), → 0.05"/min. (1.5%),
0.1"/min. (1%), → 0.002"/min. (1%), → 0.01"/min. (1.5%),
0.1"/min. (1%), → 0.2"/min. (4%), → 0.5"/min. (5%),
1"/min. (10%).

A value of m is determined from a plot of log σ (flow stress) v log $\dot{\varepsilon}$ (strain rate) since

$$m = \frac{d\ln \sigma}{d\ln \dot{\varepsilon}}$$

In the second technique m is determined from stress relaxation tests[4]. Stress relaxations were carried out at various stages of the strain-rate-sensitivity experiments and at least once during the elongations to failure tests. This involved stopping the crosshead and measuring the decay of stress with time.

b) Determination of elongations-to-failure

Elongations-to-failure tests were carried out at an initial engineering strain rate of 1%/min. Stress relaxations were measured after about 20% deformation. A hot zone of about 2-2.5" restricted accurate temperature control at the end of high elongations-to-failure tests and thus probably limits the upper meaningful ductility that can be achieved (using a 1/2" gage length). Also, grain growth and decreasing strain rate during the tests make interpretation of these stress-strain curves difficult. It should also be noted that the specimen thickness can affect the elongations achieved, thick specimens favouring high elongation.

c) Thermal Cycling

Previous work[4] has suggested that cycling through the γ-α transformation temperature (A_1) has a beneficial influence on superplastic properties certain specimens were cycled therefore to investigate this effect.

Specimens were aged in air at 770°C for ten minutes and quenched into water and then liquid nitrogen. The specimens were ground, or etched in nital, to remove any decarburized layers before testing.

d) Room Temperature Properties

Specimens of 1" gage length were tested at room temperature to determine the yield strength, U.T.S. and elongation to failure. Strain was measured using a 1/2" extensometer. All specimens were tested at an initial engineering strain rate of 5%/min.

iii) Optical Microscopy

Specimens were mounted in bakelite and ground on a conventional series of emery papers and alumina cloths. Etching was carried out in nital and photographs were taken at various magnifications.

RESULTS AND DISCUSSION

i) Microstructural Development of Fine Grain Size

The development of the necessary fine grain size is illustrated by optical micrographs. Figure 1 shows the 1.26% + 1%Ni + 0.1%V in the as-received, $(\gamma w)_{cool}$ and $(\gamma w)_{cool} + (\alpha w)_{650°C}$ conditions and is typical of the four high carbon steels containing nickel. The grain size is seen to decrease from 40μm to about 1μm. In these steels the structure is initially seen to

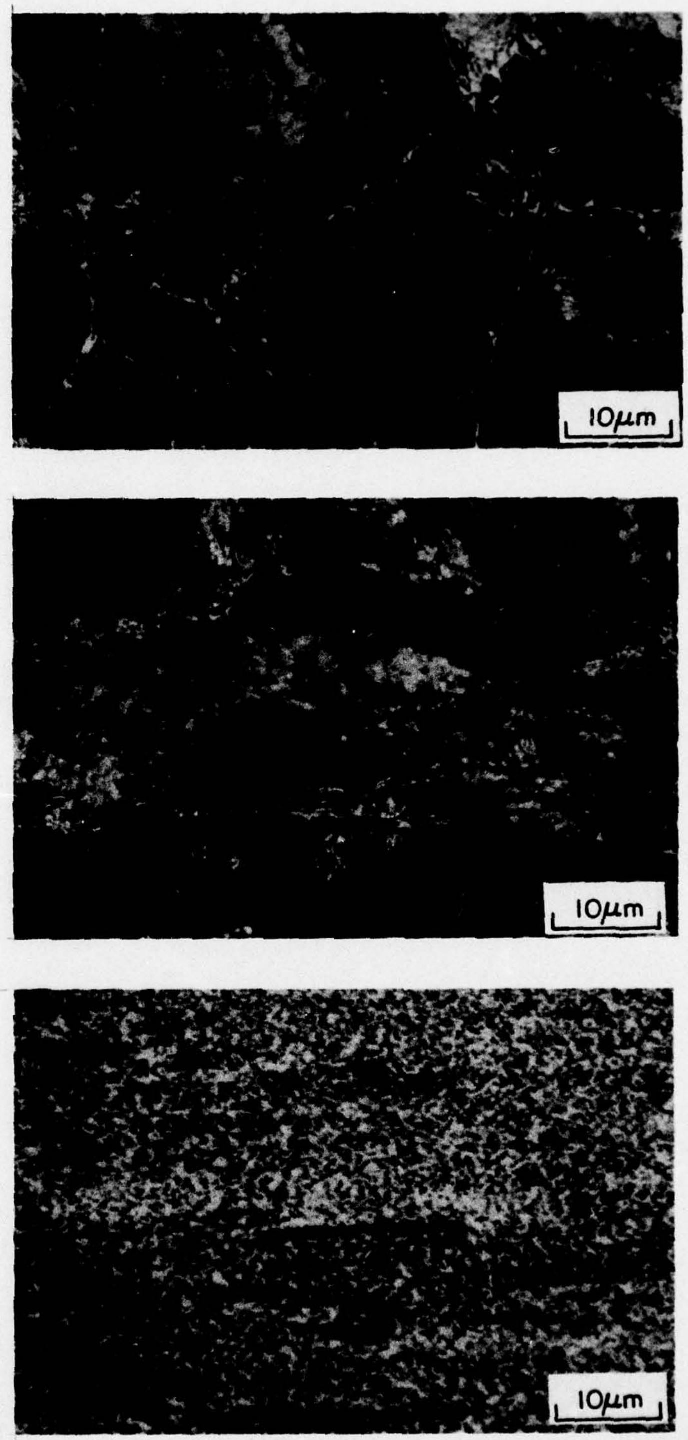


Figure 1. 1.26%C + 1%Ni + 0.1%V steel:
a) As cast
b) After $\gamma_{\text{work cool}}$
c) After $\gamma_{\text{work cool}} + \alpha_{\text{work}}$

consist of coarse pearlite grains with cementite at the grain boundaries. After thermo-mechanically treating the steels the structure is broken down into fine-grained ferrite with cementite particles at the grain boundaries. One disturbing feature is the appearance of small strings of graphite in these steels after extensive working. It should be pointed out that these steels undergo an enormous degree of strain greatly enhancing the possibility of graphitization.

Figure 2 shows micrographs for the 52100 material. Here the structure in the as-recieved condition is one of coarse-grained ferrite (~12.5μm) and spheroidized cementite. After thermal-mechanical treatment the grain size is almost too fine to resolve with the optical microscope (<1μm). No evidence of graphitization was observed either in the steel or in the one containing only vanadium, Figure 3. It should be noted that this beneficial influence of vanadium is overwhelmed in the nickel bearing steel previously described.

It is in this final, fine-grained condition that most tests were performed.

ii) <u>High Temperature Tests</u>

Strain rate change tests and elongation to failure tests were performed on all steels at 650°C ($\alpha + Fe_3C$ range) and all but the vanadium containing steel at 770°C ($\gamma + Fe_3C$ range). The results are summarized in Figures 4 and 5 and Tables 3 and 4. Constant stresses were achieved at each of the cross-head speeds employed; typical examples are shown in Figure 6 for the 52100 material (tested at 650°C after 1 thermal cycle).

Superplastic properties were found in most of the steels at both temperatures with the exception of the 1.3%C + 1%Ni steel. The strain rate range over which high m values are found is seen to be greater in the $\alpha + Fe_3C$ range than in the $\gamma + Fe_3C$ range. Walser and Sherby[6] have previously ascribed this difference to the fact that cementite is stronger in the $\alpha + Fe_3C$ range

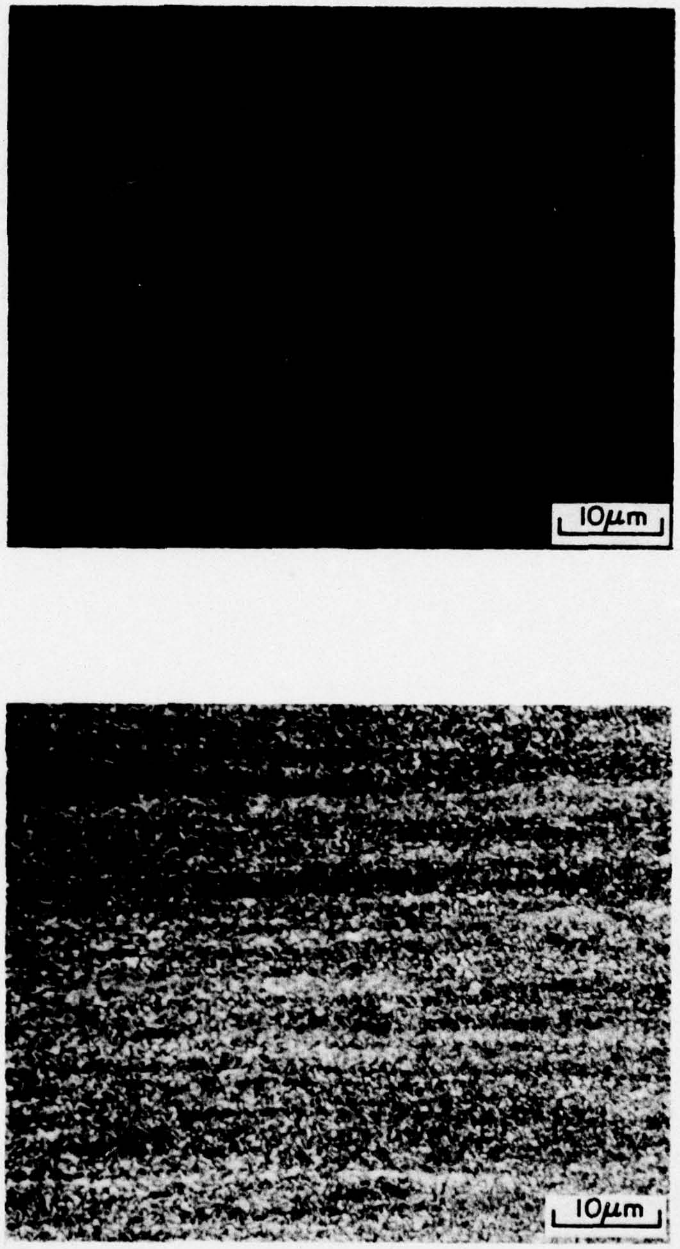


Figure 2. 52100 steel
a) As received
b) After $\gamma_{\text{work cool}} + \alpha_{\text{work}}$

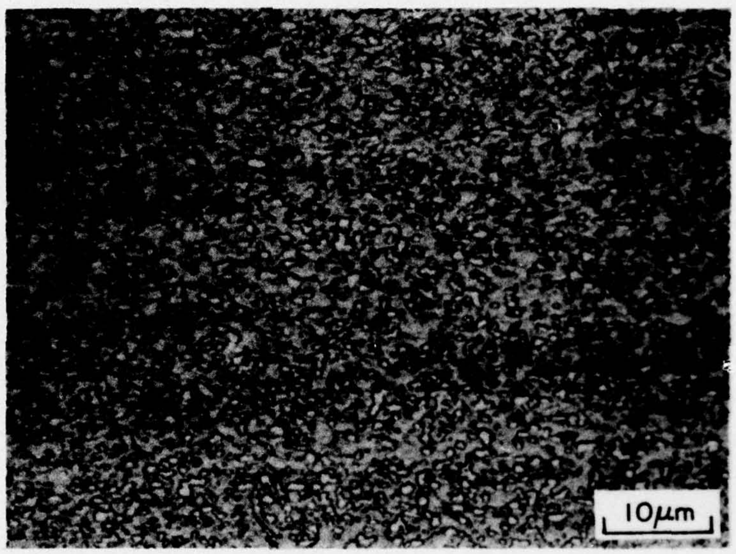


Figure 3. 1.37%C + 0.1%V steel in the $\gamma_{work\ cool} + \alpha_w$ condition.

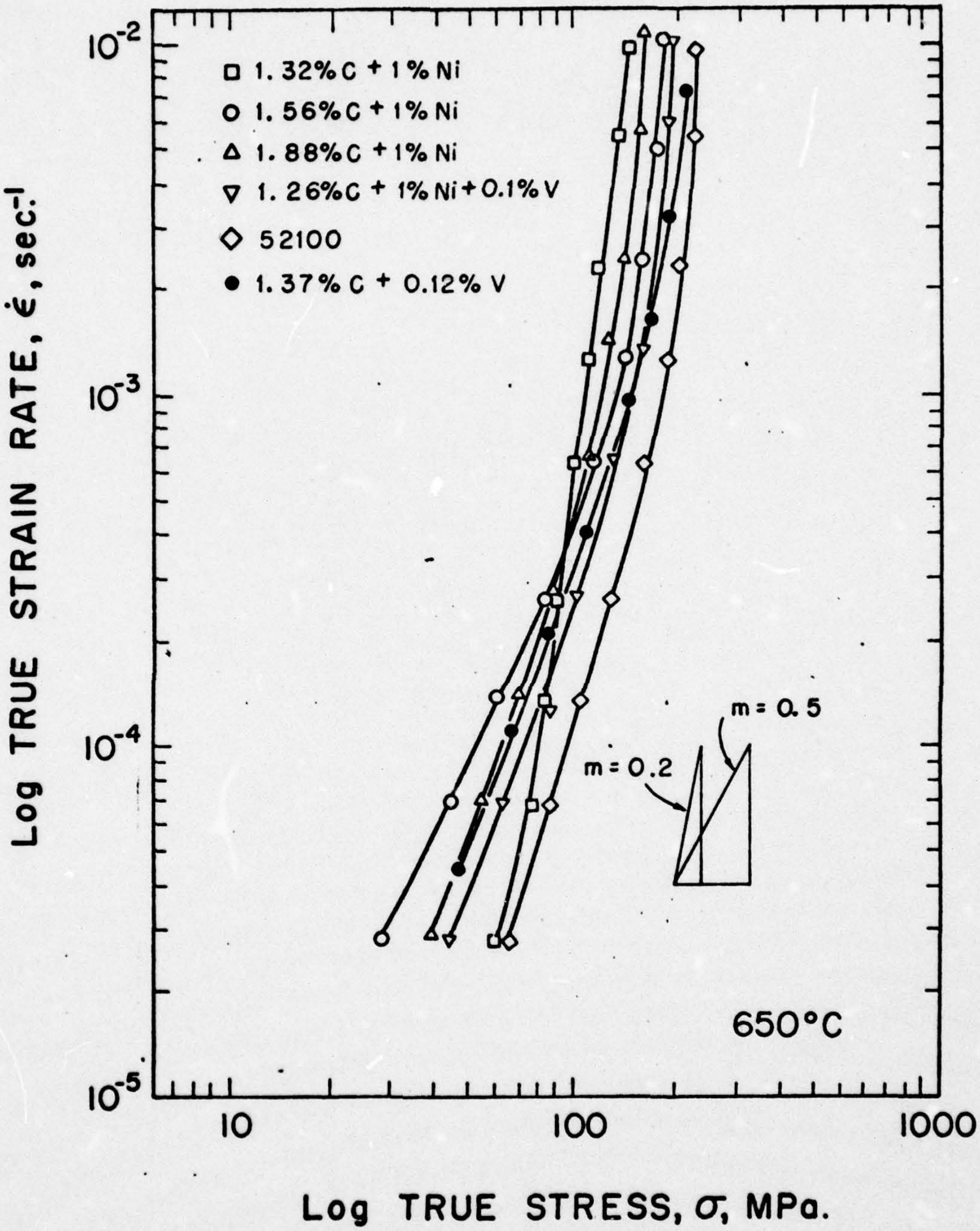


Figure 4. Flow stress, σ, V true strain rate, $\dot{\varepsilon}$, for all steels. Tests carried out in tension at 650°C.

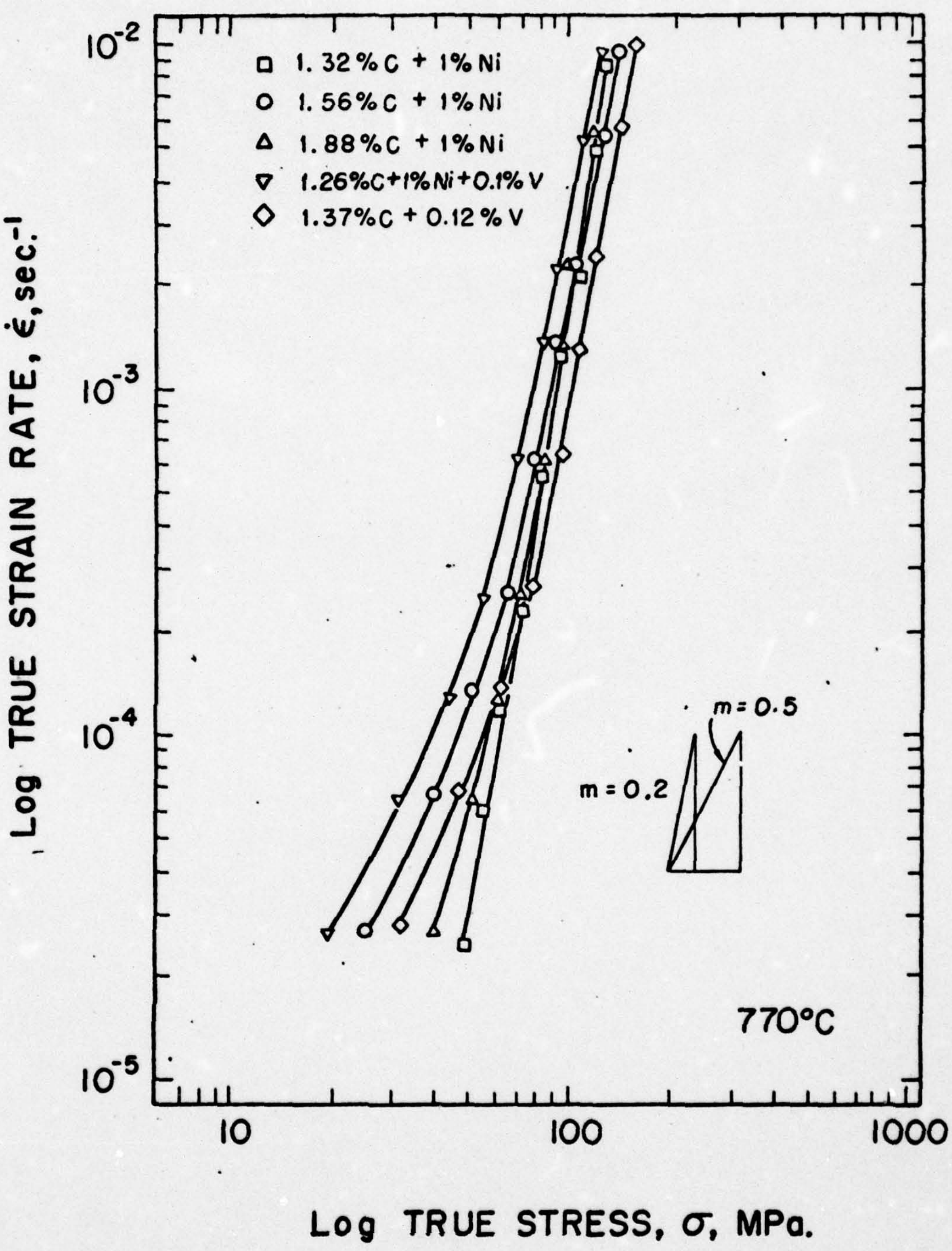


Figure 5. Flow stress, σ, V true strain rate, $\dot{\varepsilon}$, for all steels except the 1.37%C + 0.12%V. Tests carried out in tension at 770°C.

TABLE 3

STEELS IN THE AS-ROLLED CONDITION
TESTED AT 650°C

Composition	Strain Rate Change Tests		Corresponding Elongation to failure tests
	Condition and Temperature of test °C	strain-rate sentitivity $m = \frac{d \ln \sigma}{d \ln \dot{\varepsilon}}$	Elongation % at an initial strain rate $\dot{e}_i$ = 1%/min
1.32%C + 1%Ni	As rolled	0.2	84%
1.56%C + 1%Ni	As rolled	0.5	386%
1.88%C + 1%Ni	As rolled	0.37	114%
1.26%C + 0.1%V + 1%Ni	As rolled	0.43	194%
1.0%C + 1.5%Cr (52100)	As rolled	0.28	358%
1.37%C + 0.12%V	As rolled	0.42	817%

TABLE 4

STEELS IN THE AS-ROLLED CONDITION TESTED AT 770°C

Composition	Strain Rate Change Tests: Condition and Temperature of Test	Strain Rate Change Tests: strain-rate sensitivity $m = \frac{d \ln \sigma}{d \ln \dot{\varepsilon}}$	Corresponding Elongation to failure tests: Elongation at an initial strain rate of $\dot{e}_i = 1\%/\text{min}$
1.32%C + 1%Ni	As rolled	0.2	104%
1.56%C + 1%Ni	As rolled	0.5	160%
1.88%C + 1%Ni	As rolled	0.2	62%
1.26%C + 0.1%V + 1%Ni	As rolled	0.5	117%
1.00%C + 1.5%Cr (52100)	As rolled	0.41	355%

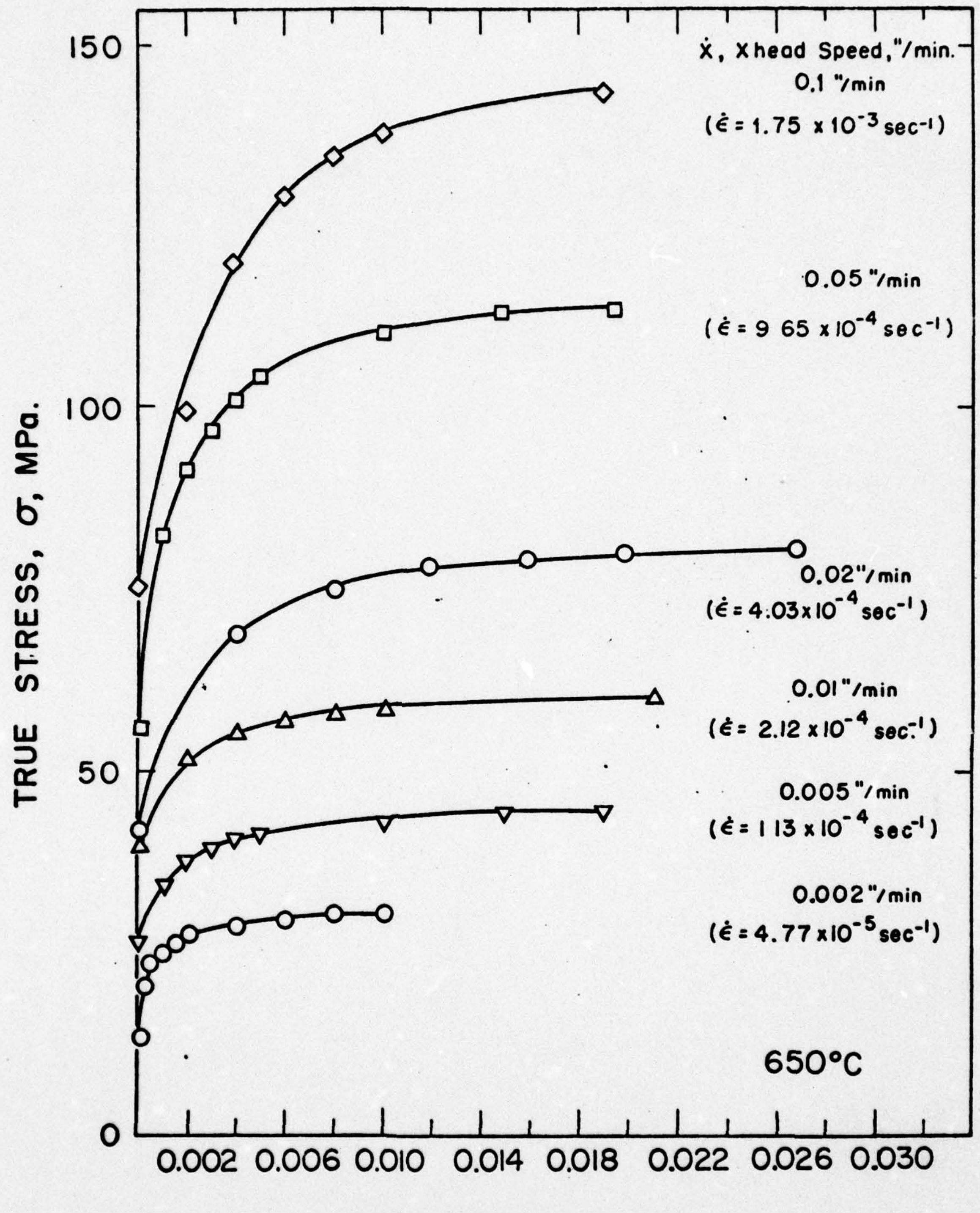


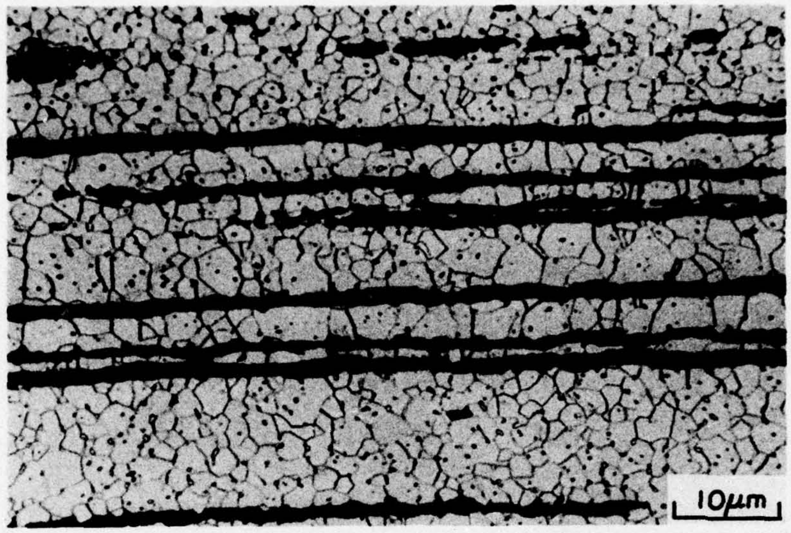
Figure 6. True stress, σ, V. true strain, ε, for 52100 in the as-rolled + 1 cycle condition in tension at 650°C for a variety of different crosshead speeds.

than it is in the $\gamma + Fe_3C$ range[6] making plastic flow by normal processes more difficult and hence extending the superplastic range. This is reflected in the better elongations-to-failure found at 650°C.

The relative strengths of the steels depend upon the temperature and strain rate. This reflects the complex factors influencing the strengths of these steels at high temperature (e.g. the grain size, number of α/Fe_3C boundaries, degree of spheroidization of cementite, carbon content, graphitization etc).

All the materials were microstructurally investigated after testing. Graphitization was found in the four steels containing nickel. This explains the low ductilities (~100%) found in some of these steels. Figure 7 illustrates the problem in the 1.26%C + 1%Ni + 0.1%V after testing at 650°C and is typical of all the four high carbon steels containing nickel. Comparison with Figure 1 reveals that graphitization worsens during testing at 650°C. Graphitization is deleterious in its own right but also because the depletion of cementite leads to grain growth. The growth is from ~1μm to ~7μm in the case of the 1.25%C + 1%Ni + 0.1%V steel compared to growth from submicron grain size to about 1 1/2 - 2μm in 52100 material. The 1.3%C + 1%Ni steel probably suffers most since it contains the least amount of cementite after graphitization and this explains why the poorest superplastic properties were found in this material-normally a very successful superplastic composition in the absence of nickel[1,2] (the 1.26%C steel contains 0.1%V which is probably beneficial to a certain extent in limiting the grain growth). Despite the graphitization in the 1.56%C + 1%Ni steel an elongation of 356% at 650°C was achieved suggesting that very high elongations could be achieved in the absence of graphite. Also, it should be noted that very thin specimens were used (0.06" - 0.09") and this adversely affects ductility measurements[6].

In the 52100 material, the strongest steel tested, no graphitization was



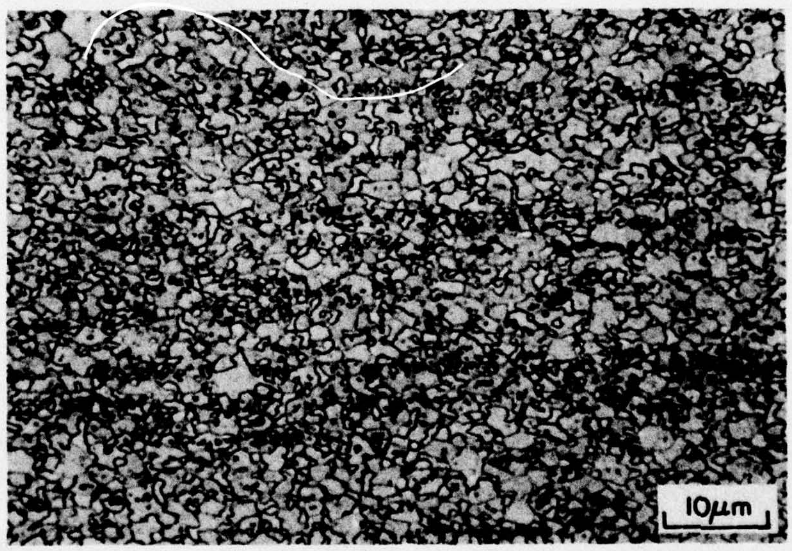


Figure 7. a) 1.26%C + 1%Ni +0.1%V after testing to 194% at 650°C in tension. b) 52100 after testing to 300% at 650°C in tension.

observed and the grain size after testing was uniform and about 1.5μm in size (Figure 7). Elongations of 358% and 355% were found at 650°C and 770°C. No graphitization occurred in the 1.37%C + 0.12%V steel and a very impressive elongation-to-failure of 817% was found at 650°C.

Graphitization is known to be promoted by high silicon and low manganese levels[7] and also by nickel. Current work at Stanford now indicates that the problem can be readily overcome by increasing the manganese to ~1% and reducing the silicon level to <~0.2% in unalloyed steels. In this way, elongations of over 800% have been achieved. In the case of 52100, chromium enters the cementite to form a stable carbide $(Fe,Cr)_3C$ and this prevents graphitization as does the addition of vanadium. This chromium or vanadium addition provides an alternative solution to graphitization and the discovery that a common tool steel can be made superplastic is an exciting one. Future work however will concentrate on developing graphite free superplastic properties in high hardenability steels without expensive alloying additions.

iii) Influence of cycling

Despite the high elongations found in these steels, recent work[4] has suggested that even higher elongations could be achieved by cycling through the ferrite/austenite transformation (A_1 temperature). It is believed that this is due to the removal of low angle boundaries formed during warm working. A mixture of low and high angle boundaries is not an optimum one since low-angle boundaries are not able to contribute effectively to superplastic deformation.

The influence of cycling is summarized in Figure 8 for the 52100 material. The increase in slope (m value) and decrease in flow stress can be clearly seen after 1 cycle. The beneficial influence of one cycle may be due to removal of low-angle boundaries or to further refinement of the

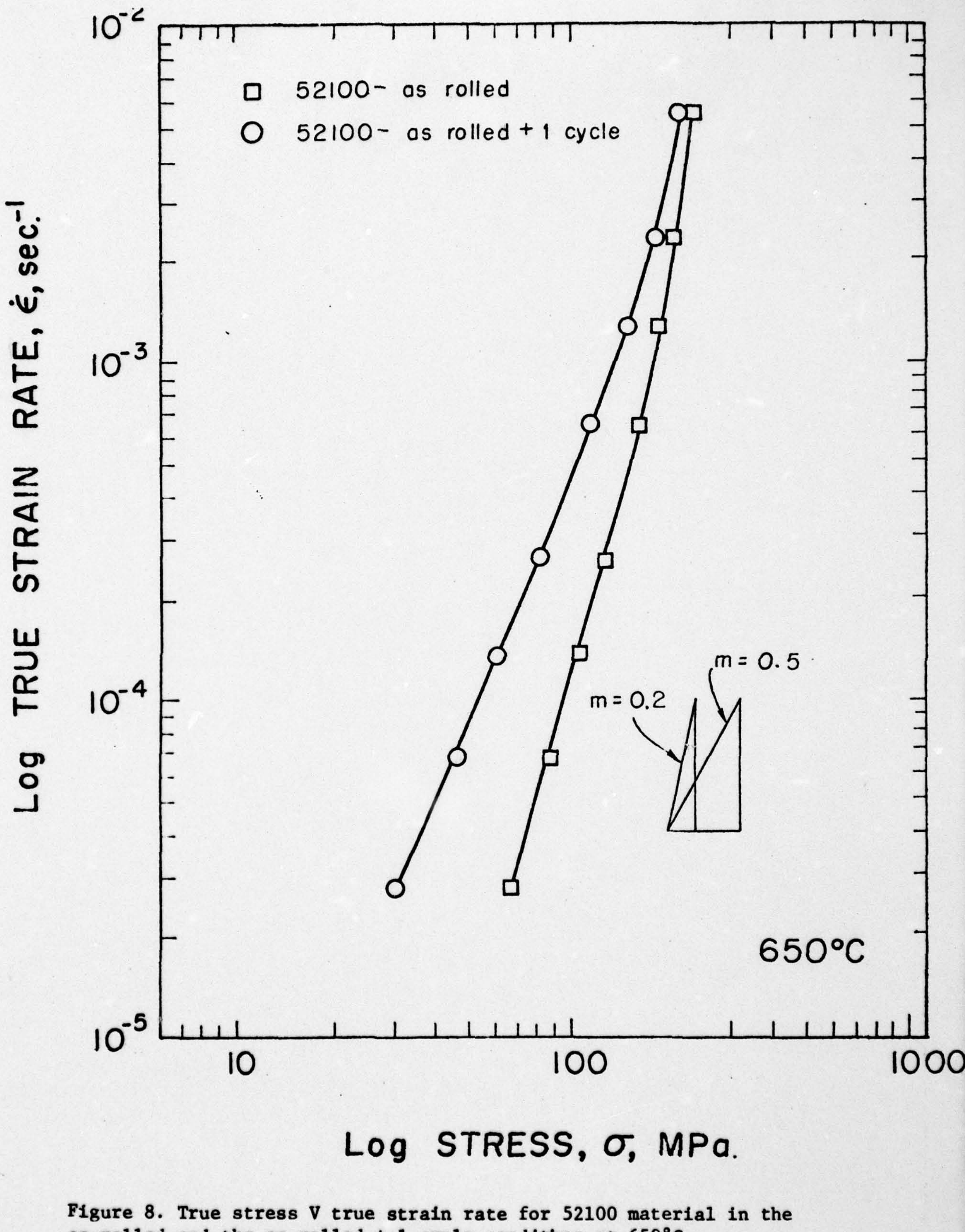


Figure 8. True stress V true strain rate for 52100 material in the as-rolled and the as-rolled + 1 cycle conditbns at 650°C.

as-rolled structure. The elongation-to-failure was also improved to 610% (1 cycle) from 358% (as rolled). The influence of further cycling was found to be deleterious in elongation-to-failure tests because grain growth occurs after many cycles and this reduces the elongation to failure.

iv) Room Temperature Properties

Tension tests were carried out at ambient temperatures on all the steels in the as-rolled and in the as-rolled plus annealed (650°C for 20 mins) conditions. The results are summarized in Table 5. In the nickel containing steels, the yield strengths and UTS values are low compared with previous work on ultrahigh carbon steels[1,2,3]. This is believed to be due to the deleterious influence of graphitization. (It is however worth noting the ductilities in the annealed condition highlighting the attractive combination of high temperature formability with room temperature properties in these materials.)

The properties of the 52100 material and the 1.36%C + V steel are impressive however and compare favorably with other UHC steels[1].

Conclusions

1. Extremely fine grained structures (<~1μm) have been developed in six ultrahigh carbon steels containing small alloying additions. These fine grained structures were achieved by a thermo-mechanical processing route.

2. Steels containing 1%Ni, inadvertantly high silicon and inadvertently low manganese, graphitized during thermo-mechanical processing. This undesirable feature can be eliminated by stabilizing the cementite with appropriate alloying elements or by balancing the steel making additions.

3. Despite graphitization, strain rate sensitivities in the range $0.3 < m < 0.6$ were achieved in most cases.

4. A common tool steel, type 52100, was successfully made superplastic and showed very little grain growth during superplastic deformation.

TABLE 5

STEEL	CONDITION	YIELD STRESS ksi	U.T.S. ksi	% El
1.32%C + 1%Ni	As rolled	98	109	11.8
1.32%C + 1%Ni	Annealed	74	88	19.3
1.56%C + 1%Ni	As rolled	127	140	8.3
1.56%C + 1%Ni	Annealed	118	126	17.2
1.88%C + 1%Ni	As rolled	124	127	2.4
1.88%C + 1%Ni	Annealed	96	102	7.2
1.26%C+V + 1%Ni	As rolled	123	138	10.8
1.26%C+V + 1%Ni	Annealed	118	129	17.5
52100	As rolled	134	148	11.9
52100	Annealed	128	137	18.7
1.37%C + 0.12%V	As rolled	138	147	10
1.37%C + 0.12%V	Annealed	126	127	22

5. A steel containing 1.37%C and 0.12%V was successfully made superplastic, an elongation of 817% being achieved at 650°C.

6. Cycling through the α-γ transformation (A_1) improves the superplastic properties by reducing the flow stresses and increasing the strain rate sensitivities. It is believed that this is due to the removal of low angle boundaries formed during warm working. One such cycle, after standard processing, improved the elongation to failure in 52100 material to 610% from 358%.

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Ultrahigh carbon steels-The influence of Chromium

J. Wadsworth and O.D. Sherby

ABSTRACT

The influence of chromium additions to ultrahigh carbon steels has been investigated. A common tool steel (52100 containing 1%C and 1.5%Cr), a 1.6%C + 1.5%Cr steel (designated 52160) and a plain 1.6%C steel have been compared.

Chromium is found to greatly enhance the superplastic properties. This is because the chromium enters the cementite and thereby stabilizes it. This in turn allows very little grain growth in ferrite to occur during superplastic deformation. A value of 1223% elongation to failure in 52160 was found at 650°C at an initial engineering strain rate of 1%/min. The influence of strain rate on the elongation to failure has also been investigated in this material.

Ultrahigh Carbon Steels - The Influence of Chromium

J. Wadsworth and Oleg D. Sherby

It is now well established[1,2,3] that steels containing between 1%C and 2%C can be readily processed to possess an attractive combination of properties at both warm (0.35 - 0.6T_m) temperatures (i.e. superplasticity) and at room temperature. This communication describes early results concerning the influence of small additions (~1 1/2%) of chromium.

Type 52100 tool steel, a cheap, widely available material, falls into the above category of an ultrahigh carbon (UHC) steel containing a small amount of chromium. Early experiments suggested that this material could readily be made superplastic[4]. A recent innovation has been to upgrade this steel to a carbon content of 1.6%C. This new steel is thus designated 52160. Increasing the carbon content increases the volume fraction of cementite and this leads to the further prevention of ferrite grain growth during superplastic flow.

UHC steels are processed into a fine-grained, final form by any of one of five routes[3]. The final structure in all cases is one of extremely fine cementite particles (0.1 - 0.5μm) in a matrix of fine-grained ferrite (1 - 2μm). Chromium is known to act as a carbide stabilizer entering the cementite, up to 9 wt %, to form a complex carbide $(Fe,Cr)_3C$[5,6]. It was believed that this improved stability, through small chromium additions, should improve the warm temperature superplastic properties and also the room temperature properties of UHC steels.

Both 52100 and 52160 were processed by a combination of hot rolling during cooling from 1100°C through the γ and $\gamma + Fe_3C$ range and isothermally rolling in the $\alpha + Fe_3C$ range at 650°C (Figure 1). For comparison throughout, a plain 1.6%C UHC steel is included. Compositions are shown in Table I and

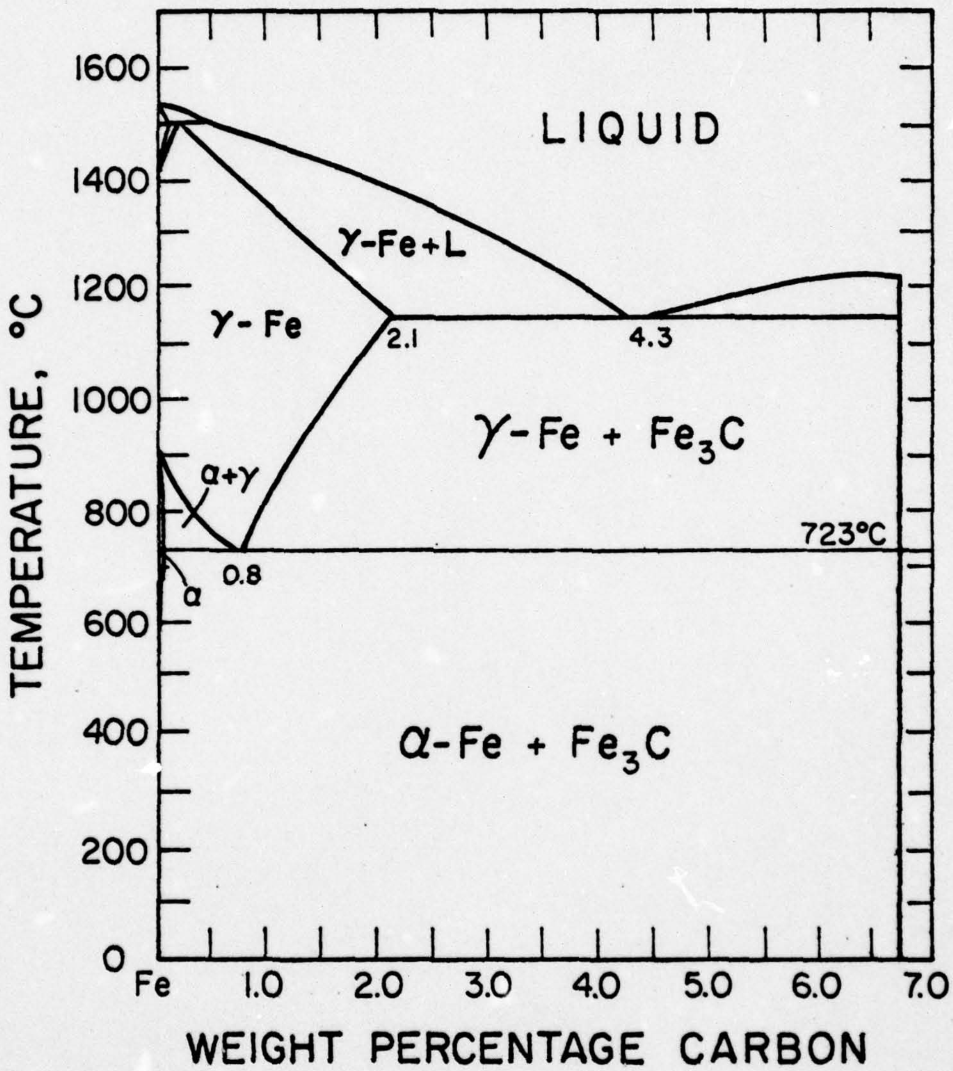

Figure 1. The Iron-carbon phase diagram.

TABLE I

COMPOSITIONS

STEEL	C	Cr	Mn	Si	S	P	Ni	Cu	Ti
52100	1.005	1.42	0.41	0.28	0.001	0.01	0.15	0.12	-
52160	1.640	1.61	1.00	0.22	0.006	0.01	0.07	0.01	0.01
1.6C	1.600	0.02	1.03	0.21	0.006	0.01	0.07	0.01	0.01

details of the thermomechanical processing in Table II.

Using tensile coupons machined from final rolled strip (~0.1" thick), superplastic properties were evaluated by measuring the elongations-to-failure during tensile testing at 650°C, at an initial engineering strain rate of $\dot{e}_i$ = 1%/min., on an Instron machine on equipment described elsewhere[7]. Tests were also carried out to determine the strain rate sensitivity at 650°C by measuring the flow stress at each of a number of imposed strain rates (Figure 2). The results of these tests are summarized in Table III. All three steels were found to be superplastic, i.e. m values were in the range $0.3 < m < 0.8$ and elongations to failure were high (>300%). In Figure 2 it may be seen that 52100 material is stronger than either 52160 or the plain 1.6%C material at low strain rates. Also, the 52160 material exhibits a high value of strain rate sensitivity at higher strain rates than do the other two materials. It is noted that the 52160 material exhibits a higher elongation than does the plain 1.6%C material despite having a lower measured value of strain rate sensitivity. This probably reflects the fact that it is the 'terminal' strain rate sensitivity, i.e. that near fracture, and not the one measured in a short term test, that controls elongation at fracture - a point made recently by Ghosh and Ayres[8].

An elongation to failure value of 1155% was found in the case of 52160 as shown in Figure 3. In a repeat test, for material for metallographic examination, a value of 1223% was recorded. This is the first time in the brief history of these materials that values in excess of 1000% have been achieved. These high values for 52160 are atributed to the beneficial influence of the increased quantity of cementite through increasing the carbon content (c.f. 52100), and to the improved stability of the cementite through the chromium additions (c.f. 1.6%C steel).

TABLE II

THERMOMECHANICAL PROCESSING

Steel	true strain, ε, during working from 1100°C → ~ 650°C	true strain, ε, during isothermal rolling at 650°C	Total Strain
52100	$\varepsilon = -1.81$ in 19 passes	$\varepsilon = -1.15$ in 24 passes	-2.96
52160	$\varepsilon = -1.48$ in 15 passes	$\varepsilon = -1.35$ in 18 passes	-2.83
1.6%C	$\varepsilon = -1.71$ in 16 passes	$\varepsilon = -1.15$ in 13 passes	-2.86

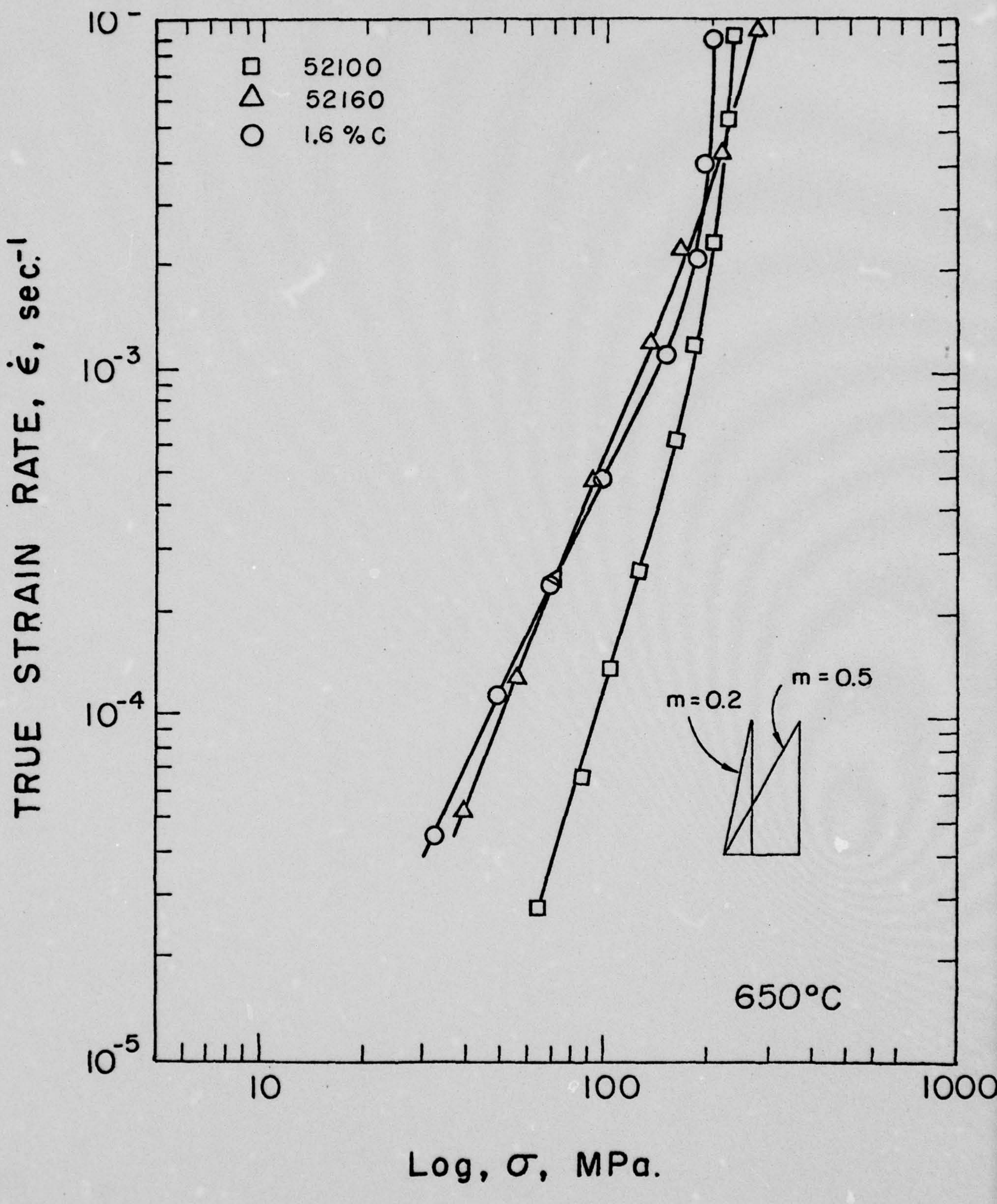

Figure 2. True stress-true strain relationships for 52100, 52160 and 1.6%C steels at 650°C.

TABLE III

SUPERPLASTIC PROPERTIES

Steel	Strain Rate Sensitivity, m,	Elongation at 650°C ($\dot{e}_i$ = 1%/min)
52100	0.3	329
52160	0.39	1155,1223
1.6%C	0.46	470

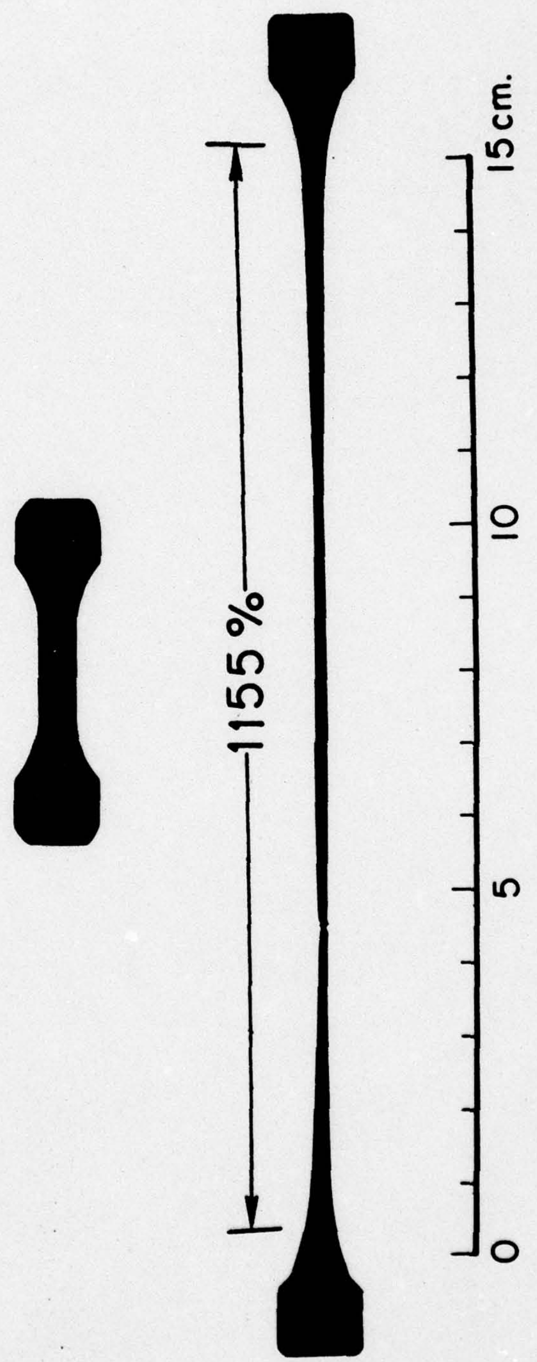

Figure 3. 52160 steel tested in tension at 650°C at an initial engineering strain rate of 1%/min.

This conclusion is supported by metallographic observation of the three steels. The steels are shown in Figure 4 in the as-rolled and as-deformed conditions. Also shown are the specimen heads-which experienced no strain and hence have effectively simply been annealed. Grain growth can be seen to be a minimum for the 52160 material. Even after 1000% deformation, grain growth is only from submicron size in the as-rolled condition to about 3μm. By comparison, the plain carbon material exhibits grain growth to 4-5μm after only 470%.

Because of the high elongations found, and the retention of good strain rate sensitivity at high strain rates, 52160 was tested over a wide range of strain rates (0.4% - 1000%/min.) at 650°C. The results are shown in Figure 5 and reflect the extremely high formability of this material in the fine grained condition over a wide range of strain rates.

Room temperature properties of these materials have also been assessed and are summarized in Table IV. These values represent the averages of several tests. Materials were tested in the as-rolled condition and also after an annealing treatment at 650°C for 20 minutes. Specimens were tested in tension on an Instron machine at an initial engineering strain rate of 2%/min. An extensometer was used to record specimen elongation. The room temperature properties are seen to be impressive.

Chromium is seen to have a beneficial effect upon room temperature properties reflecting the influence of chromium on refinement of the grain and carbide size.

In conclusion, the addition of chromium has a significant, beneficial influence on UHC steels that already posses a unique and attractive combination of warm temperature formability and high strengths at room temperature with associated ductility and cheapness of production.

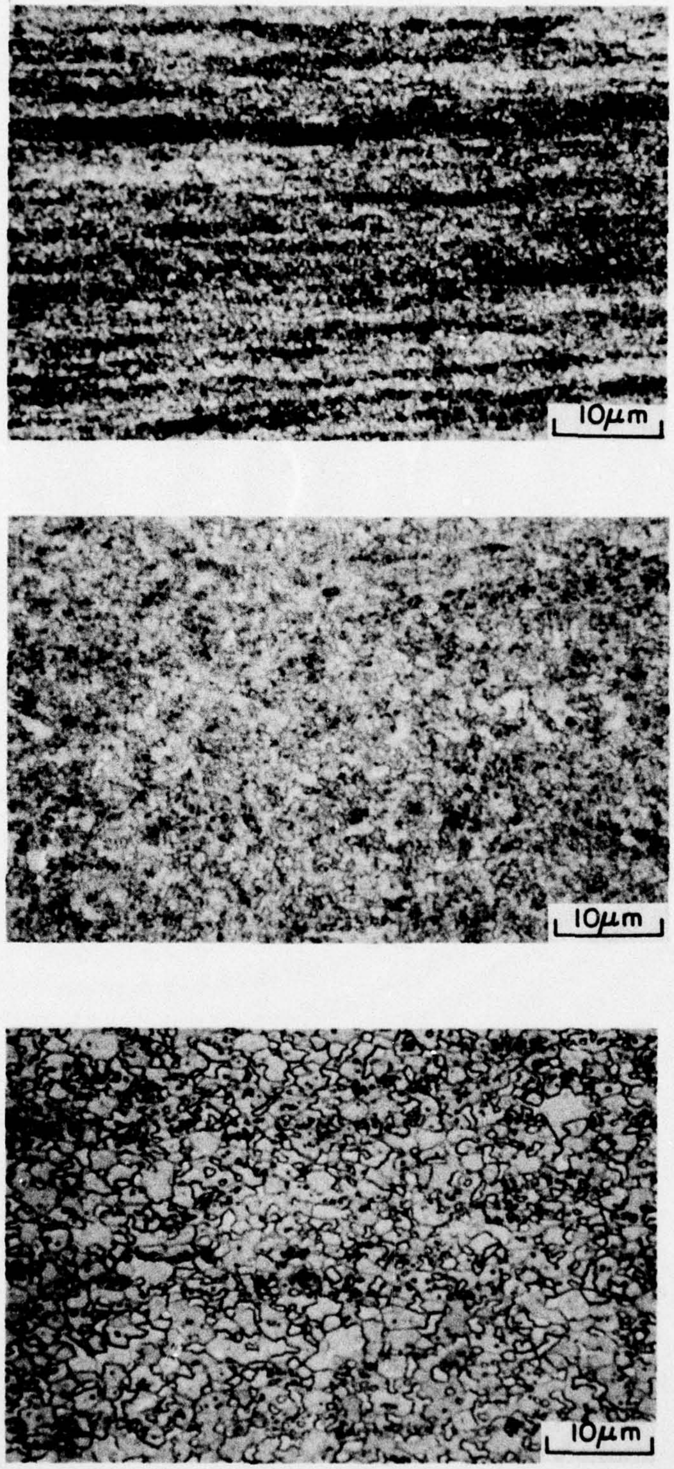

Figure 4a. 52100 steel, $\gamma_w + \alpha_w$
Top - As rolled
Center - Undeformed head of specimen tested to 286%
Bottom - Gage length of specimen tested to 286%.

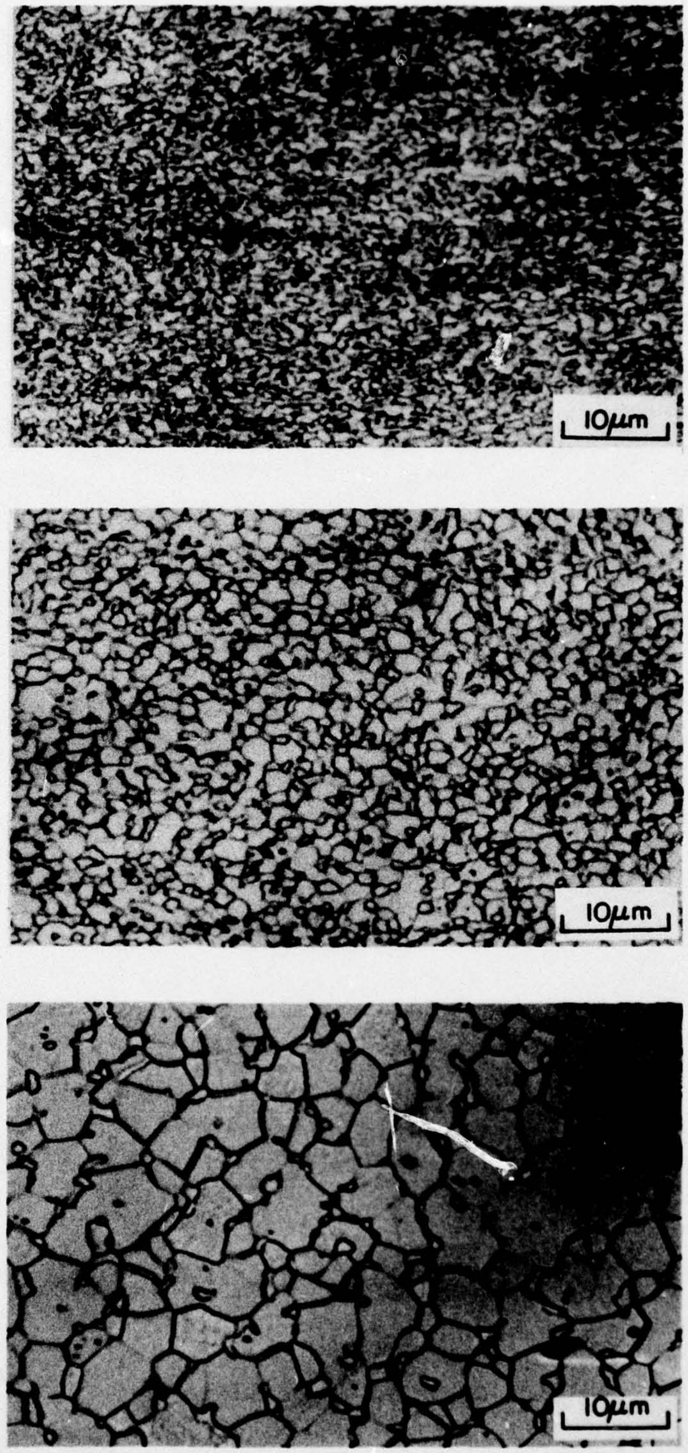

Figure 4b. 1.6%C steel $\gamma_w + \alpha_w$
Top - As rolled
Center - Undeformed head of specimen tested to 470%
Bottom - Gage length of sepcimen tested to 470%.

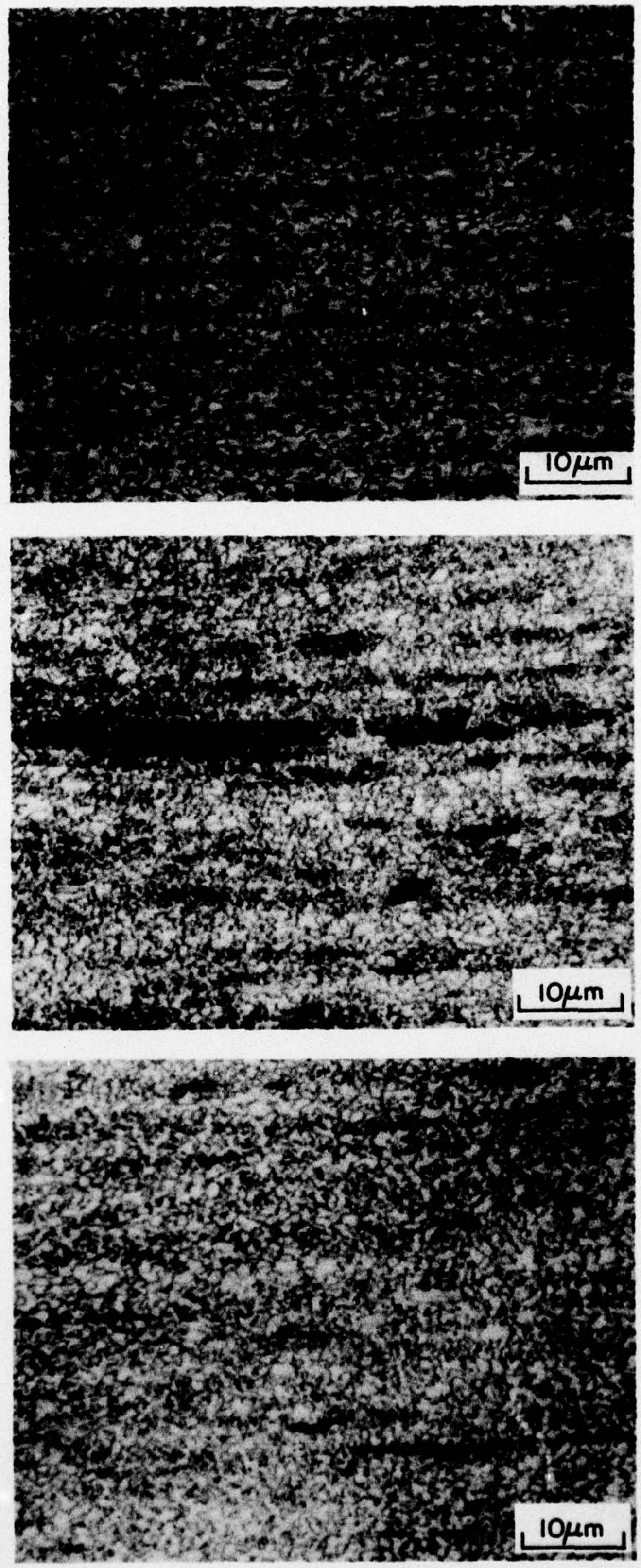

Figure 4c. 52160 steel, $\gamma_w + \alpha_w$
Top - As rolled
Center - Undeformed head of specimen tested to 200%
Bottom - Gage length of specimen tested to 200%.

Figure 4d. 52160 steel, $\gamma_w + \alpha_w$
Gage length of the specimen tested to over 1000%.

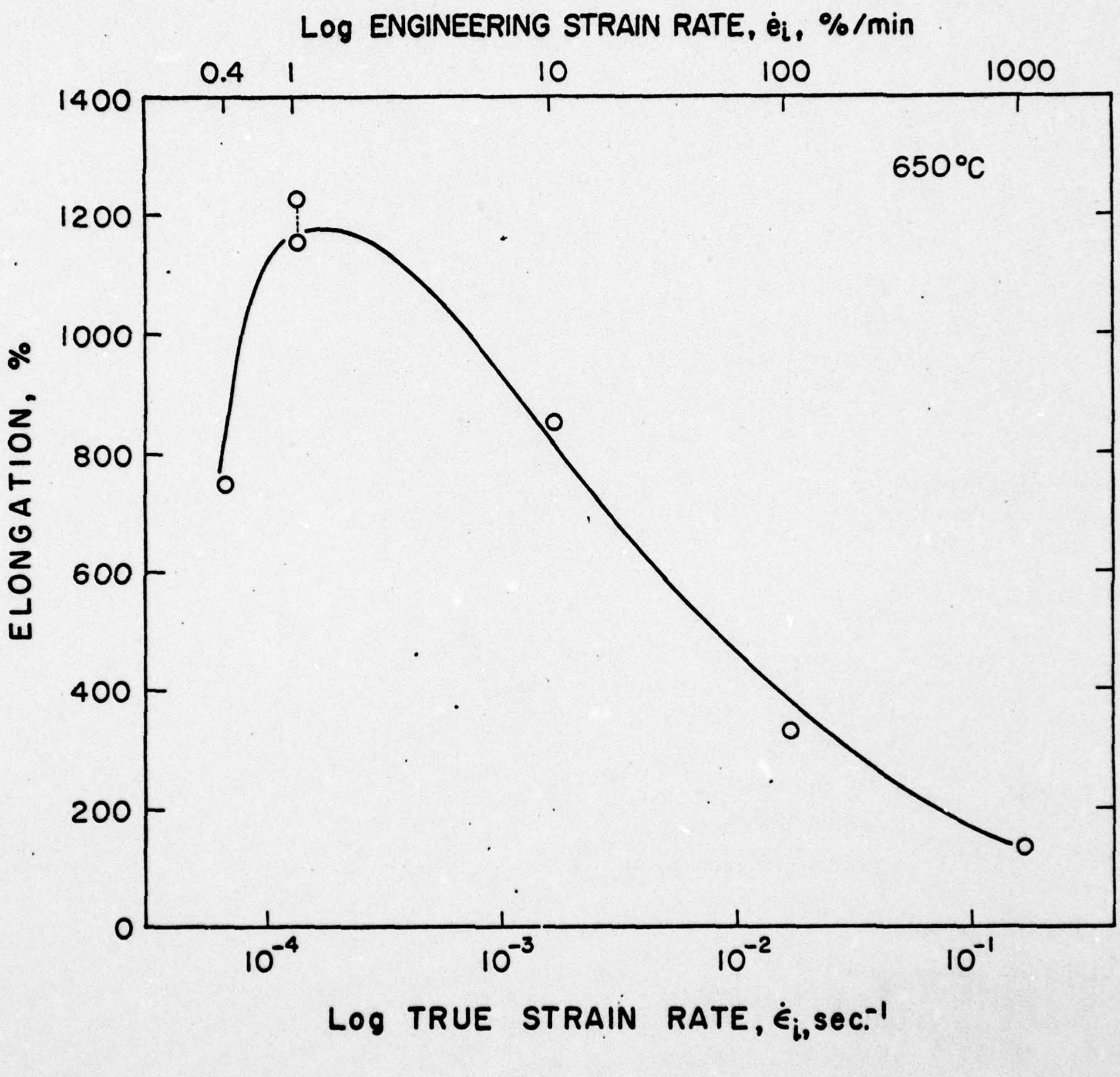

Figure 5. Percent elongation as a function of initial strain rate for 52160 at 650°C.

TABLE IV

ROOM TEMPERATURE PROPERTIES

Steel	As Rolled			As rolled + Annealed at 650°C		
	YPt ksi	UTS ksi	% El.	YPt ksi	UTS ksi	% El.
52100	134	148	11.9	128	137	18.7
52160	149	168	6.4	144	158	11.7
1.6%C	130	144	8.6	121	124	18

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Experimental work performed on ultrahigh carbon steels
at Sulzer Brothers, Switzerland

B. Walser

ABSTRACT

The forgeability of ultrahigh carbon steels has been investigated. The best isothermal forging temperature is found to be 650°C.

Graphitization is not apparent - even after extensive warm working - in steels containing less than 0.1%Si. Microstructural evaluation of refinement clearly shows that the fineness of the structure increases with strain during isothermal forging.

Additions of chromium and/or vanadium lead to a finer structure than for plain carbon steels but the addition of molybdenum makes structural refinement very difficult.

The wear properties of these steels have also been investigated and compare fovourably with conventional wear resistant materials.

Experimental work performed on UHC-steels at Sulzer Brothers, Switzerland

Bruno Walser

We have investigated six different castings of ultrahigh carbon steels of the following nominal composition:

#	C (%)	Mn (%)	Si (%)	Cr (%)	V (%)	Mo (%)
1.	1.4	0.7	0.25	-	-	-
2.	1.4	0.7	0.25	2	-	-
3.	1.4	0.7	0.25	2	0.5	-
4.	1.4	0.7	0.25	-	-	2
5.	1.4	1.0	<0.1	-	-	-
6.	1.4	1.0	<0.1	2	-	-

1. WARM WORKING

Warm working of the steels was performed by high speed (hydraulic hammers) forging and on an experimental rolling mill. In order to get information on the forgeability of the plain UHC steels, casting #1 was forged in the temperature range 1100°C to 650°C [(γ_w)C] from a round billet (Diameter 180mm) to a square bar (100mm x 100mm). This corresponds to a strain of $\varepsilon = -0.6$. This bar was cut into three pieces each of which was then isothermally forged to "step samples" at 600°C, 650°C and 700°C. The forgings are shown in Figure 1. The best forging temperature was 650°C.

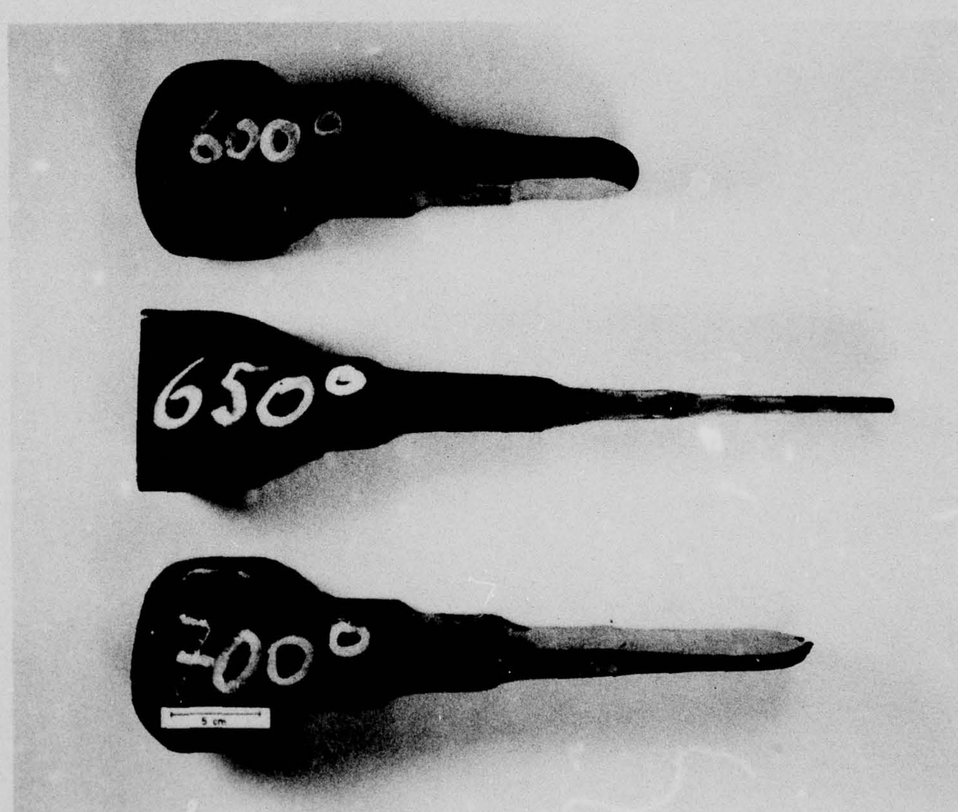

Figure 1. Steel #1 - 1.4%C, 0.7%Mn, 0.25%Si, isothermally forged at the indicated temperatures after $\gamma_{w\ cool}$ forging.

We were able to forge from 100mm down to 8mm ($\varepsilon = -2.5$) before the first cracks appeared on the tip of the sample. At 600°C cracking occured much earlier. The crack development is probably due to the formation of graphite. Steels containing high silicon (>0.2%) and low manganese (<1.0%) are susceptible to graphitization. Under the very high tensile forces at the tip of the forging, the graphite lamellaes probably act as crack initiators.

In the second forging experiment, steels #5 and #6 were deformed from 200mm round to 100mm square during (γ_w) cooling. The isothermal working at 650°C resulted in plates of 40mm thickness ($\varepsilon_\alpha = -0.9$) and 10mm thickness ($\varepsilon_\alpha = -2.3$). In these steels the high Mn to Si ratio prevents graphitization. Neither steels exhibited cracking during these severe forging operations reflecting the fact that the control of chemical composition leads to improved warm working characteristics.

Steels #1 to 4 were also rolled on our experimental rolling mill. The most difficult to deform was steel #4 containing 2% molybdenum. After a certain amount of deformation it always cracked. Alloying with chromium and vanadium made the rolling somewhat more difficult, but we were able to reduce billets from 30mm to plates of 2mm ($\varepsilon_\alpha = -2.7$). Steels containing carbide stabilizers such as Cr or Mo do not graphitize.

2. MICROSTRUCTURAL EVALUATION

Steel #1 showed strain induced graphitization as its analysis would suggest. We found that the graphite exists as stringers aligned in the rolling direction. The addition of alloying elements fully prevented the graphite formation, even for steels of the same basic composition as steel #1. The plain carbon steel #5 did not show graphitization since we kept the silicon content below 0.1%.

The refinement of the microstructure as a function of warm working strain is shown in Figure 2. The different prints are related to the steps in the 650°C forging shown in Figure 1. The top micrographs are longitudinal sections in the rolling direction from left to right and the bottom micrographs are transverse section. The fineness of the structure is clearly enhanced by strain. Also, the graphite lamellar are closed up by extensive deformation. In order to get a high quality product the amount of manganese sulfide has to be controlled. Past experience shows that stringers of inclusions are very detrimental to the toughness of warm worked products.

Metallography of the warm rolled plates showed that alloying with chromium and/or vanadium leads to a finer structure than the plain carbon steel for the same amount of warm working strain. The structural refinement of the molybdenum containing steel was very difficult and after a strain of $\varepsilon_\alpha = -1.3$ (40 → 4mm) only half of the carbides were spheroidized.

3. <u>WEAR RESISTANCE TESTING</u>

The wear resistance was tested by having a rotating roll pressed against a sample of UHC steel. The rotating roll was manufactured from 0.15% mild steel and then carburized (case hardened). Figure 3 shows the resulting wear of the partners after 20 hours of testing. For comparison, the wear properties of two carburized 0.15% steels are also shown. The UHC steel was heat treated the following way

Quenched only: 760PC/ 1 hr/ H_2O

Quenched + anneal 760°C/ 1hr/ H_2O + 300°C/ 1 hr/ air

In general the wear properties are in the order of the 0.15% steel combination; in some cases they are much better. More systematic studies are underway.

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FINE STRUCTURE AND SUPERPLASTICITY IN ULTRAHIGH CARBON STEELS.(U)
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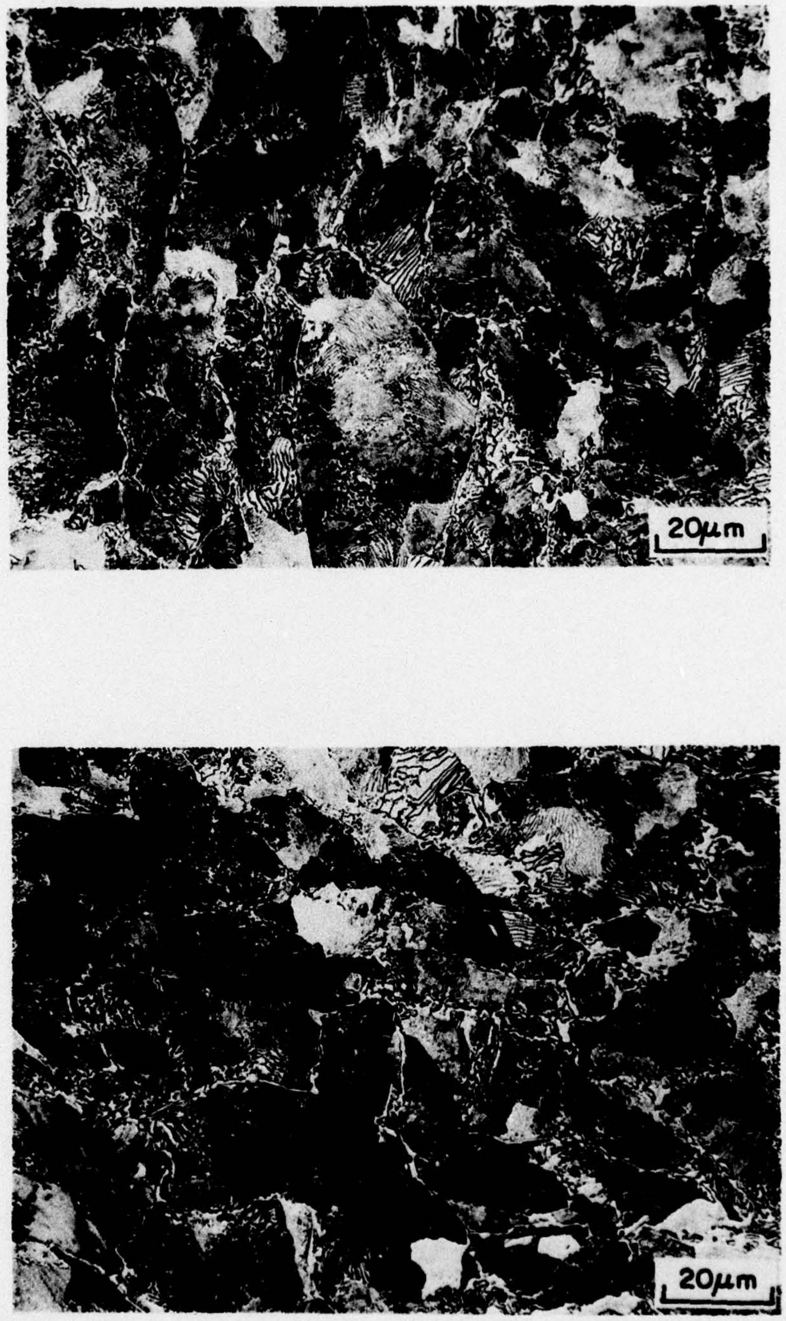


Figure 2. $\gamma_{w_{cool}}$ only, $\varepsilon = -0.6$

1.4%C - steel #1, forged to the shape shown in Figure 1. Micrographs represent the different steps and show the development of the structure as a function of the warm strain.

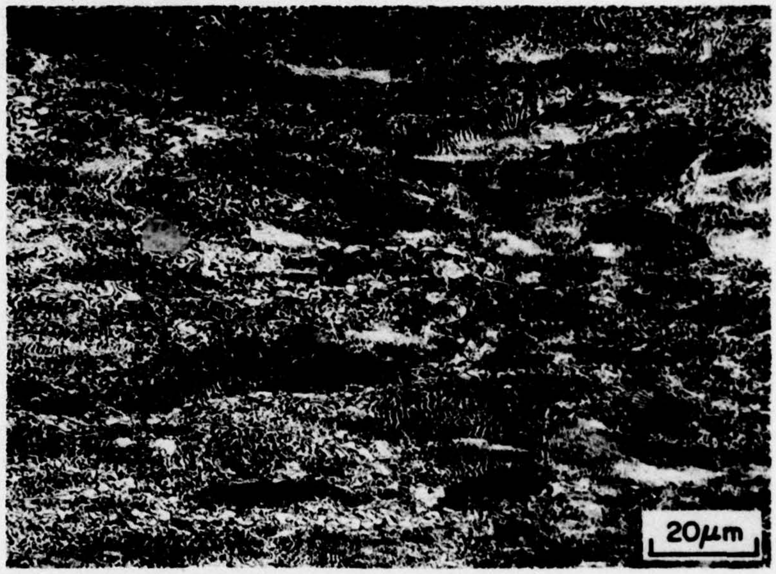


Figure 2 cont. α_w 650°C, ε_α = -0.7.

Figure 2, cont. α_w 650°C, ε_α = -1.2.

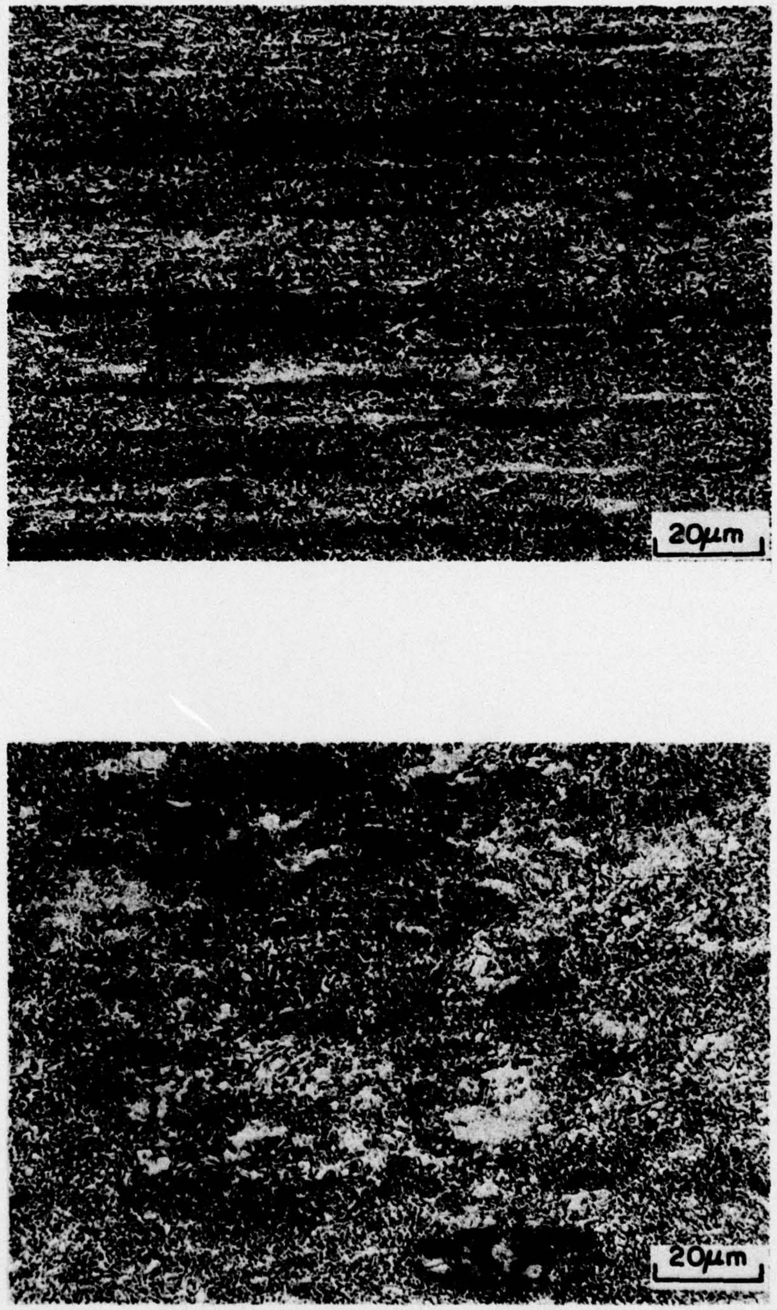


Figure 2, cont. α_w 650°C, ε_α = -1.9.

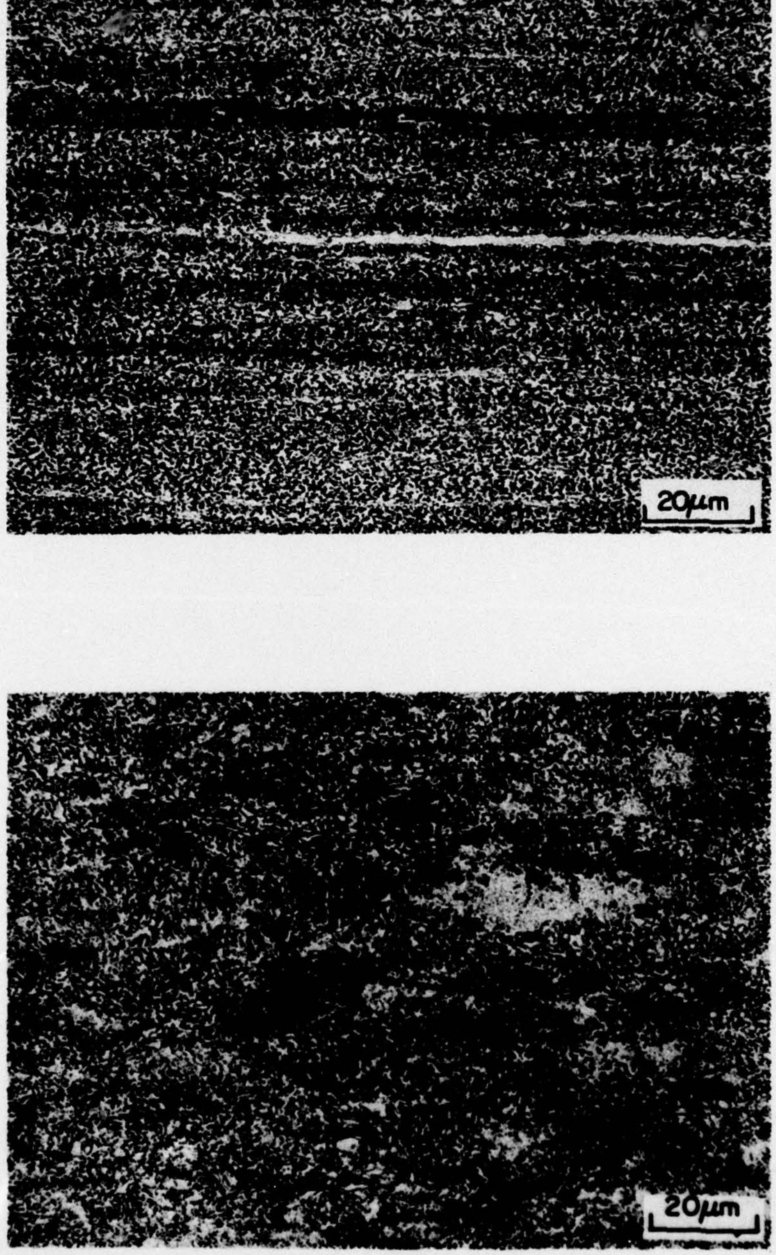
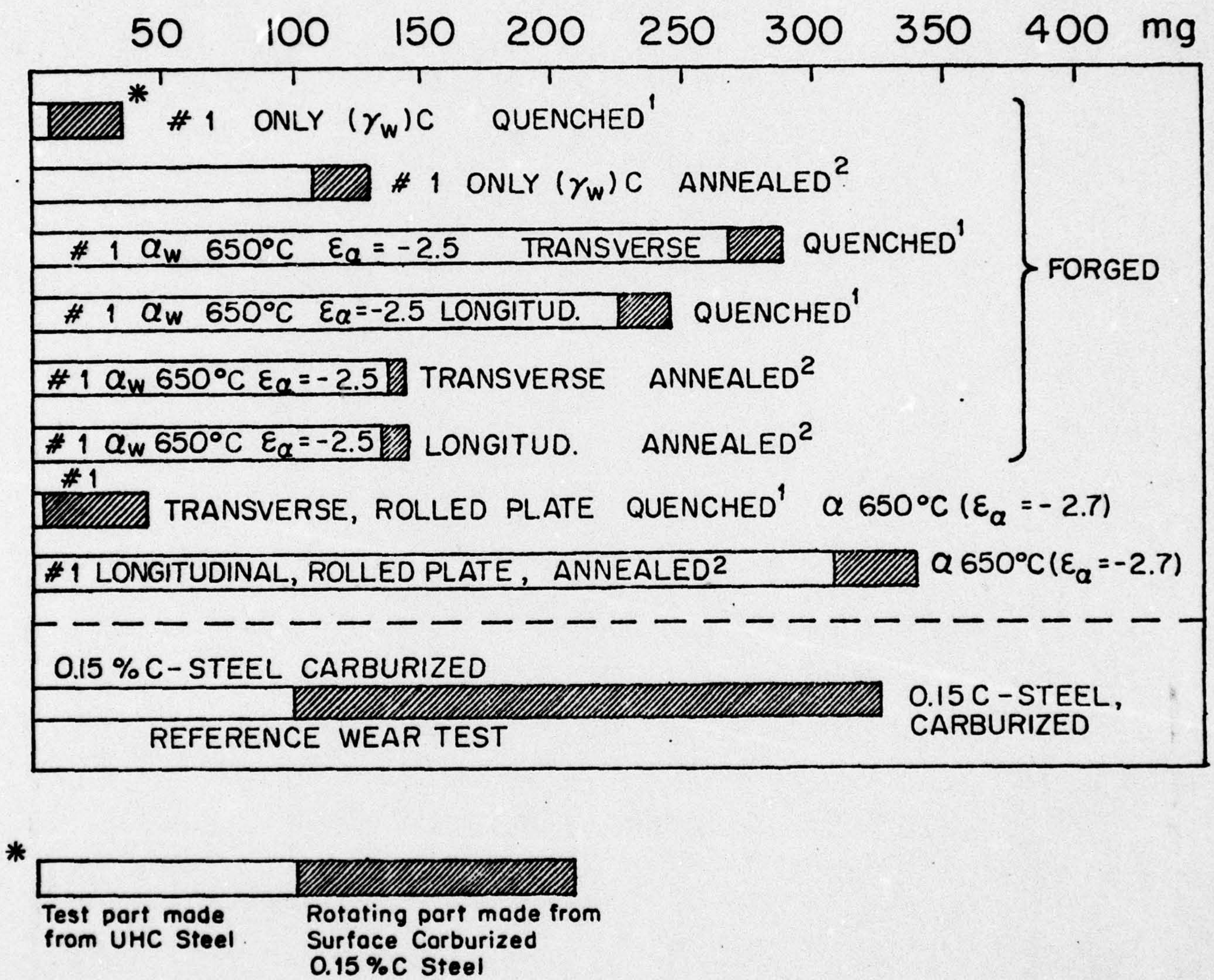


Figure 2, cont. α_w 650°C, $\varepsilon_\alpha = -2.5$.

WEAR AFTER 20 HOURS



[1]QUENCHED: 760°C for 1 hr. ⟶ H_2O Quench

[2]ANNEALED: 760°C for 1 hr. ⟶ H_2O Quench + 300°C for 1hr, air cool

Figure 3. Wear properties of plain UHC steel - 1.4%C.