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SOME OBSERVATIONS ON NIOBIUM IN STEEL

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PHYSICAL METALLURGY DIVISION

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Some Observations on Niobium in Steel*

D. R. BELL** and *G. P.* CONTRACTOR***

Abstract:

The effects of niobium on silicon-killed, low carbon steel were investigated. Most of the data relate to 450-lb laboratory melts, forged and rolled to 3/4 in. plate. Data from one heat of commercial steel were also examined.

In the as-rolled condition, it was found that small additions of niobium increased the yield strength and raised the transition temperature markedly, but that larger additions were less effective. In the normalized condition, the effects on yield strength were similar to those in the as-rolled condition but the changes were less marked. The transition temperature was improved with all levels of niobium, the smallest additions (up to 0·04%) being most effective.

Commercial plate of a similar composition to the laboratory steel was procured with niobium contents of approximately zero, 0·010% and 0·023%, in 1/4 in., 3/4 in., and 1-1/2 in. thicknesses. The yield strength and transition temperature results obtained on the commercial material qualitatively corroborated the findings of the laboratory steels. Niobium was most effective in increasing the yield strength in the 1/4 in. plate, the effectiveness decreasing as the plate thickness increased. Rotating beam fatigue tests showed that niobium slightly increased the fatigue limit of smooth bars and had no effect on the fatigue limit of notched bars. The fatigue ratio was unaffected by niobium for smooth bars and decreased somewhat for notched bars.

Limited data, based on electrolytically extracted residues, indicated that the major bulk of the niobium added to the steels was partitioned to the carbide phase. Similarly, much of the nitrogen present in the steels was found to occur in the residues.

The evidence indicated that the effects of niobium on the yield strength and notch ductility of normalized carbon steel were qualitatively explicable in terms of grain refinement, and precipitation strengthening. The evidence did not account for the pronounced effects of small quantities of niobium on as-rolled steel.

1. Introduction

The material to be presented in this paper was obtained in the course of a program that originated as an investigation into the effects of small additions of several elements on killed low-carbon steel. The program developed into an urgent search for low-cost steel of excellent notch ductility with 45,000 psi yield strength in 3/4 in. plate, then reverted to its original research status. Hence, the investigation to date has been rather ad hoc, with an engineering emphasis on mechanical properties. As a consequence, many exploratory heat treatments and detailed experiments necessary to a systematic investigation into the effects of niobium on steel are yet to be carried out.

Most of the data pertain to tests and examination of induction melted steels prepared in the laboratory. Data from one heat of commercially-produced steel are included. Results of the analysis of phases electrolytically extracted from the carbon steels are outlined. Some preliminary results on the effect of niobium on an experimental low-alloy structural steel are also given.

2. Experimental Materials and Procedures

The laboratory carbon steels were air-melted in a 450-lb induction furnace. The steel was killed with silicon prior to pouring. No aluminum was added. The metal was cast into three 6' in. diameter, 135-1b, hot-topped, ingot moulds. In each melt, the first ingot was poured as a niobium-free reference base. Ferro-niobium was added to

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Table 1. Composition of laboratory steels (Element, per cent).

Sample	Nb	C	Mn	Si	S	P	N
High manganese group							
11	—	0·16	0·98	0·11	0·014	0·023	0·010
13HC*	—	0·27	0·98	0·15	0·012	0·009	0·006
15	—	0·18	1·36	0·10	0·011	0·013	0·009
16	—	0·19	1·21	0·12	0·009	0·012	0·007
15A	0·007	0·19	1·37	0·12	0·012	0·012	0·009
15B	0·017	0·19	1·39	0·14	0·012	0·013	0·010
13A HC	0·06	0·27	0·98	0·19	0·012	0·014	0·005
11A	0·07	0·16	1·16	0·14	0·014	0·019	0·009
13B HC	0·14	0·27	1·06	0·15	0·013	0·018	0·005
11B	0·15	0·16	1·16	0·22	0·013	0·020	0·009
16A	0·23	0·19	1·28	0·16	0·009	0·015	0·007
Base Av For group	—	0·20	1·18	0·16	0·012	0·017	0·008
Low manganese group							
6	—	0·17	0·54	0·26	0·024	0·026	0·009
7	—	0·16	0·75	0·18	0·024	0·022	0·009
8	—	0·17	0·89	0·11	0·022	0·021	0·005
8A	0·006	0·16	0·86	0·08	0·024	0·023	0·006
8B	0·02	0·16	0·81	0·05	0·016	0·019	0·005
7A	0·07	0·17	0·76	0·20	0·021	0·018	0·009
6A	0·13	0·19	0·56	0·10	0·024	0·028	0·004
6B	0·23	0·20	0·51	0·09	0·024	0·031	0·009
Base Av For group	—	0·17	0·70	0·16	0·022	0·025	0·007

* HC–High Carbon.

Table 2. Mechanical test results and estimated ferrite grain size of laboratory steels.

Sample	CV-15 T.T.*		Yield str.		As-rolled			Normalized			Grain size ASTM No.	
	AR	N	AR kpsi	N kpsi	UTS kpsi	El. % 4×D	R.A. %	UTS kpsi	El. % 4×D	R.A. %	AR	N
High manganese steels												
11	−15	−17	44·8	42·0	70·2	33	67·5	68·7	37	66·5	7	7
13HC	+15	− 8	51·6	53·0	86·1	28	62·7	81·5	32	62·0	7	7
15	−25	−45	46·9	50·0	75·9	33	70·8	74·5	35	67·0	7	8
16	−40	−62	46·1	48·0	72·6	37	69·1	71·3	36	70·0	7/8	8
15A	−13	−68	48·5	53·0	78·8	30	67·8	75·5	34	72·0	7	>8
15B	+11	−93	52·4	53·5	82·8	28	66·9	76·0	38	71·0	7	>8
13A HC	+90	−49	66·8	60·0	94·5	25	59·4	82·0	33	63·4	8	>8
11A	+70	−93	63·0	56·0	87·0	26	61·0	71·5	37	71·0	8	>8
13B HC	+37	−47	61·0	57·8	88·2	28	62·2	79·5	33	64·0	8	>8
11B	+45	−89	61·5	57·0	82·7	28	67·0	69·8	38	71·8	>8	>8
16A	−25	−110	57·1	53·0	75·6	32	70·8	71·0	37	70·0	8	>8
Low manganese steel												
6	+50	0	42·8	44·5	70·0	33	—	74·5	32	62·0	5	7/8
7	+18	−20	45·5	40·2	66·9	36	65·9	64·3	37	64·4	7	5/6
8	−10	−25	44·1	39·5	68·0	36	65·7	66·1	36	66·5	6	6/7
8A	0	−30	46·0	41·7	68·4	34	67·0	65·0	36	67·1	6/8	5/6
8B	+15	−25	50·3	46·2	69·9	32	65·6	64·3	37	68·6	7/8	8
7A	+50	−35	56·0	43·5	71·9	32	62·9	64·5	37	66·6	7	>8
6A	+68	−25	54·0	49·4	74·0	29	—	68·6	36	64·0	7	8
6B	+63	−27	49·0	47·6	69·0	31	—	66·4	35	65·0	7/8	>8

* Charpy V-notch 15ft-lb transition temperature.

the furnace between pours to yield two more ingots with successively higher levels of niobium. The ingots were forged and rolled to 3/4 in. plate. The last pass of 1/4 in. reduction was made at 1010°C (1850°F). This temperature was reported as fairly typical of commercial practice. After rolling, the plates were placed on edge to cool in still air. It had been determined experimentally that this procedure approximated commercial hot bed cooling rates for 3/4 in. plate. Half the material was tested in the as-rolled condition, and the other half after normalizing from 900°C (1650°F). Duplicate room temperature tensile tests were carried out on 0·438 in. diameter × 2 in. gauge length specimens. Full Charpy V-notch curves of absorbed energy were established from which transition temperatures were determined. The microstructures were examined and photomicrographs of all steels were taken at ×100. Selected samples were examined in more detail.

The steels are considered in two groups based on manganese content with 0·90% the dividing line. Compositions and average base composition for each group are given in Table1.

It may be noted that the spread in manganese is 40 points as against 30 points in the AISI grades. The silicon content was deliberately held to the fairly low level of 0·16% average as any silicon in excess of that required to deoxidize the steel serves to reduce its notch toughness.

The succeeding Fig. 2 to 7, inclusive, show graphically the data of greatest interest for the laboratory induction steels. Detailed results are given below in Table 2.

2·1 Tensile Properties

Fig. 1 shows the yield strength of the high-manganese steels as a function of the niobium content. In general, small quantities increase the yield strength markedly, larger quantities being less effective. The curve peaks at about 0·07% niobium where the yield strength is increased from 47 kpsi average for the base composition to 63 kpsi. Normalizing reduces the effect but does not eliminate it.

The squares show data for commercial plate of the same thickness and similar composition. This material will be dealt with more fully later but these data are introduced at this stage to show that there is qualitative agreement between the laboratory induction steel and commercially-produced steel. Points marked HC refer to the high carbon melt-0·27% carbon compared to 0·020 % carbon average for the group.

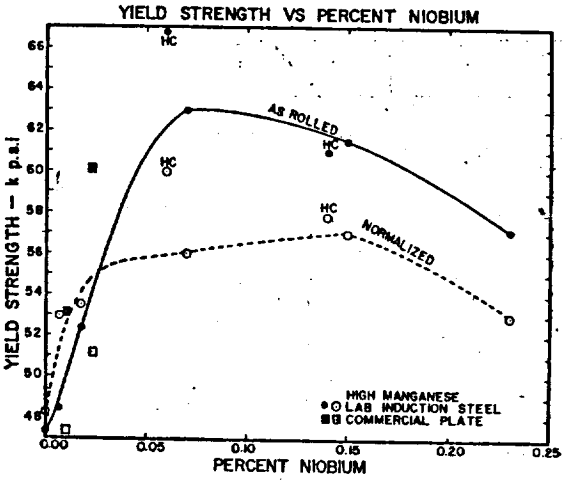

Fig. 1. The influence of niobium on the yield strength of low manganese steels.

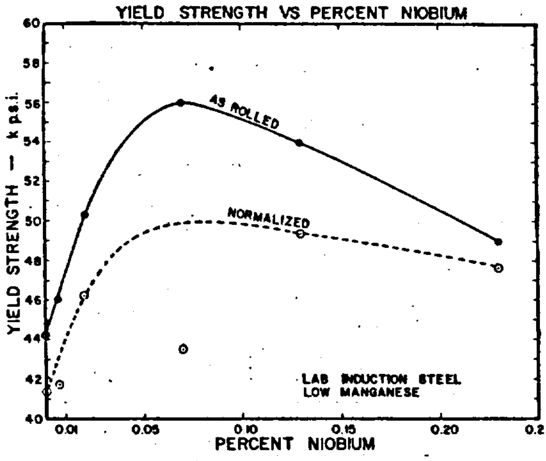

Fig. 2. The influence of niobium on the yield strength of low manganese steels.

Fig. 2 shows the same property for the low manganese group. The curves are similar to those for the high manganese series. The peak again occurs at about 0·07% niobium and the yield strength in the as-rolled condition is increased from about 44 kpsi to about 56 kpsi.

The curves in these two figures show the effects of changes in the base composition as well as effects due to differences in niobium content. The effect of niobium was isolated to some extent by plotting the change in yield strength from that of the base composition for each melt. The data on this basis for the high manganese series, Fig. 3, show increases in yield strength up to 40% in the as-

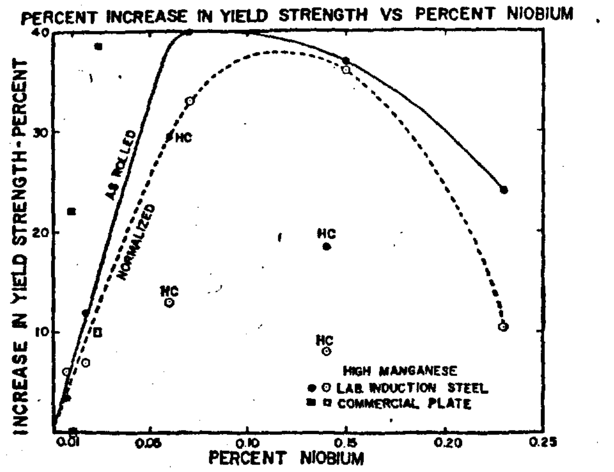

Fig. 3. The influence of niobium on the per cent change in yield strength of high manganese steels.

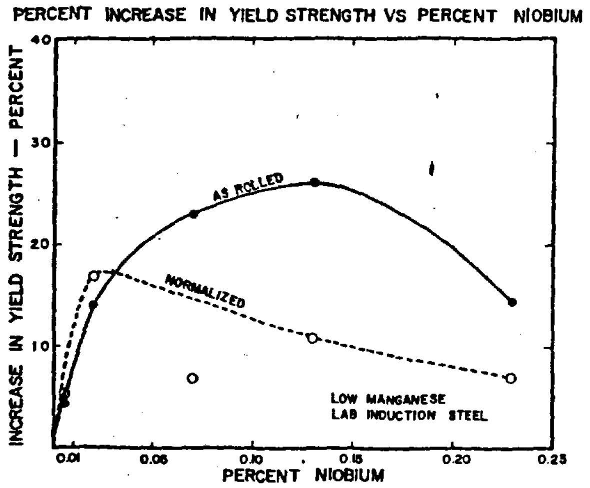

Fig. 4. The influence of niobium on the per cent change in yield strength of low manganse steels.

rolled condition, and, again, that higher levels of niobium are less effective.

As before, the data for the commercial plate show qualitative agreement with those for the laboratory steels. It will be noted that the HC points in Fig. 3 are well below the curves and not above as in Figure 1. It would appear that higher carbon reduces the effectiveness of niobium in increasing yield strength. Fig. 4 shows the data on the change-in-yield-strength basis for the low manganese group. This Figure, compared with Fig. 3, clearly shows that niobium is considerably less effective in increasing yield strength with low manganese, the maximum change amounting only to about 27%.

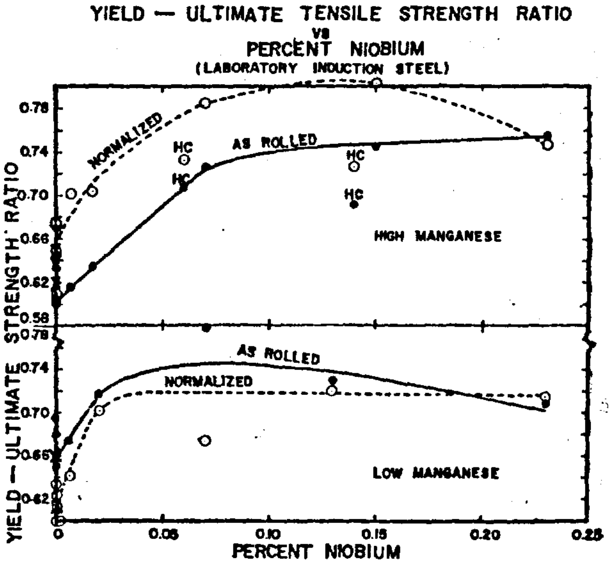

Fig. 5. The influence of niobium on the yield-ultimate ratio.

Fig. 5 shows the effect of niobium on the yield-ultimate ratio. Again, the influence of niobium on the high manganese steel is more marked than on the low manganese material. Also, the maximum effect is found at the low contents where the yield strength is markedly affected.

2·2 Impact Properties

The mechanical property of prime interest was notch toughness. The data as shown subsequently are based on the Charpy V-notch 15 ft-lb transition temperature. It is pointed out that this value is used due to its familiarity and not because it is considered a valid criterion for service performance

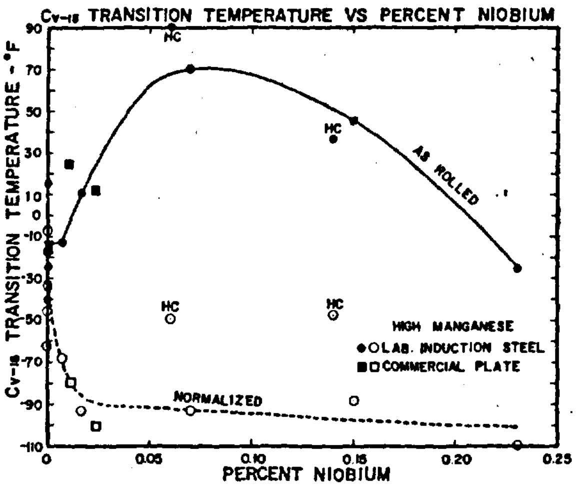

Fig. 6. The effect of niobium on the Charpy V–notch 15ft-lb transition temperature of high manganese steels.

for this type of steel. From a structural service viewpoint, a 25 to 35 ft-lb criterion would be more appropriate. Fortunately, the Charpy V-notch energy curves were generally similar in shape so that discussion based on change in transition temperature is unaffected by use of the 15 ft-lb rather than the 30 ft-lb criterion.

Fig. 6 shows the transition temperature as a function of niobium content for the high manganese series. In the as-rolled condition, the curve is similar to that for the yield strength vs. niobium content. However, for the normalized condition, while niobium increased yield strength, it decreased the transition temperature, a drop of about 33°C (60F°) being achieved with about 0·02% niobium.

The points indicated by squares refer to the previously mentioned commercial plate. Again, there is reasonable agreement with the results for the laboratory steels.

Fig. 7 shows the Charpy V-notch results for the low manganese group. The effect of niobium is much diminished as compared to the high manganese steels, especially for the nomalized material, where the drop in transition temperature amounts only to about 11°C (20°F).

Fig. 8 shows the transition temperature on the basis of change from base. As with yield strength, niobium has less influence on the low manganese group than on the high. The influence of higher carbon is quite evident in both the as-rolled and the normalized conditions. It might be mentioned that a single high manganese heat with a niobium of 0·42% was checked. In the as-rolled condition, the transition temperature was lowered 23°C (42°F) from base while the yield strength was virtually unchanged at 51.1 kpsi against 48.9 kpsi for the base material.

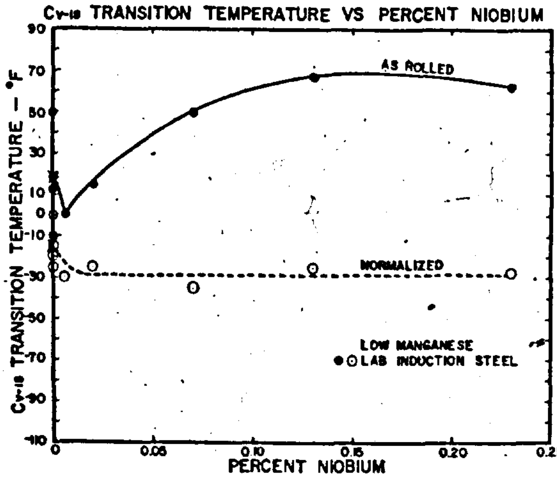

Fig. 7. The influence of niobium on the Charpy V-notch 15ft-lb transiton temperature of low manganese steels.

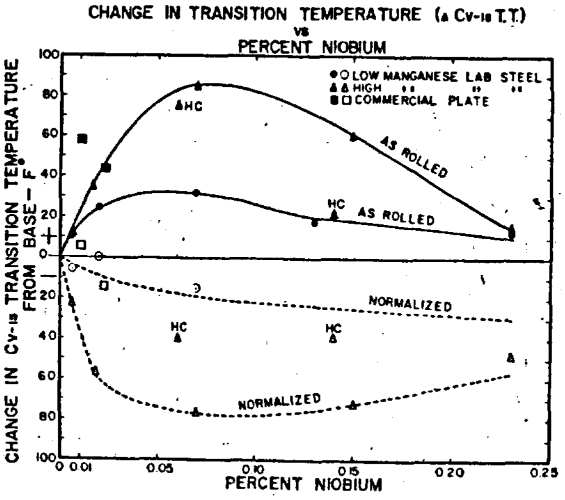

Fig 8. The influence of niobium on the change transition temperature of both high and low manganese steels.

2·3 Grain Size and Microstructural Characteristics

The approximate grain size of the laboratory steels was estimated by the visual comparison method and varied over the rather narrow range of ASTM 6 to 8 or finer. There was some variation in microstructure, which ranged from equiaxed ferrite with sharply defined lamellar pearlite colonies through irregular ferrite grains and mixed spheroidite-pearlite colonies, to a rather acicular pattern. The microstructural changes did not correlate with niobium content as did the mechanical properties. It can, however, be said that there was a trend to finer grain size compared to the base metal from 0·007% through 0·15% niobium with a reversion to a somewhat coarser grain size at 0·23%. This was generally true of both the normalized and the as-rolled conditions.

A qualitative correlation was noted between the variation in grain size and change in yield strength for the as-rolled steels. However, the grain refinement was accompanied by a rise in transition temperature, rather than a drop, which is the general case for plain carbon steels.

In the normalized condition, the same observation with respect to yield strength can be made but in this case grain refinement was accompanied by lowering of the transition temperature.

2·4 Commercial Plate

Thus far, the data have referred to plate of one thickness, finish rolled at the same temperature as nearly as possible and with the same cooling rate. Through the cooperation of Algoma Steel Corporation, a commercial heat of LD steel was produced with the base composition* very similar to the average of the high manganese laboratory steels. Ferroniobium was added to two 6·7 ton hot-topped ingots. Three ingots from this heat with zero, 0·01% and 0·023% niobium, respectively, were processed to standard commercial practice to plates of 1/4 in., 3/4 in., and 1—1/2 in. thicknesses. This material provided a check on the interaction between niobium content (over a small range) and plate thickness with the concomitant variations in finishing temperature, cooling rate, etc. Of equal importance, the 3/4 in. plate was required to check whether the properties of the laboratory steel were in any way similar to a commercial product. Data have already been presented to show that there is qualitative agreement. Figure 9 shows the effect of niobium on yield strength, transition temperature, and grain size for the commercial material. The solid lines refer to the as-rolled condition, the broken lines to the normalized.

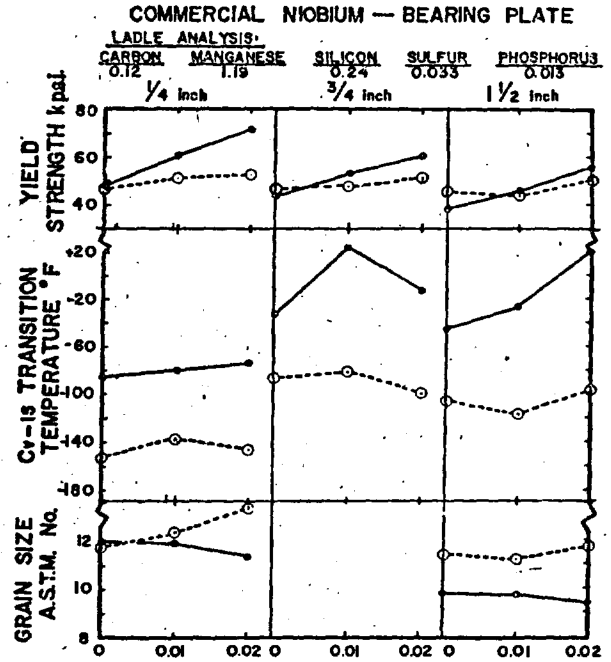

Fig. 9. The influence of niobium on the yield strength, the transition temperature and the grain size of commercial niobium-bearing plate.

With regard to the yield strength, in the as-rolled condition the increase at 0·023% niobium amounted to 47% for the 1/4 in. and 1—1/2 in. plate and 39% for the 3/4 in. plate. After normalizing, the increases amounted to only just over 10% for all thicknesses. The consistency of the yield strength increase in the normalized condition suggests that variation in rate of cooling from the normalizing temperature of 900°C (1650°F) did not influence the strengthening mechanism of niobium.

With regard to transition temperature, the situation is less simple. In the 1/4 in. plate, the transition temperature rises with niobium content, although only slightly. Incidentally, the so-called 15 ft-lb transition temperature for the 1/4 in. plate is derived from half size bars, 10 mm × 5mm, the test results being doubled and the 15 ft-lb transition temperature read from the curve. This procedure gives a spuriously low value. Hence, the data for the 1/4 in. plate are comparable amongst themselves but are not directly comparable to those for the 3/4 in. and 1—1/2 in. plate. The transition temperature curve of the as-rolled 3/4 in. plate shows a pronounced peak similar to that in the laboratory steels but at a much lower niobium content. Unfortunately, there was a good deal of Widmanstatten structure in the as-rolled 3/4 in. plate. This structure has such a strong effect on notch toughness that its influence probably masks the true effect of niobium. An obvious observation is that the variation for the as-rolled transition temperatures is much greater in the thicker plate than in the 1/4 in. where niobium had little effect. This is in contrast to yield strength where the effect of niobium did not vary appreciably with thickness. Within a given plate thickness, variations in grain size were slight (less than one ASTM grain size number). No correlations can be made between grain size and mechanical properties for the 1/4 in. plate. The marked rise in transition temperature with essentially constant grain size for the 1—1/2 in. plate is noteworthy and parallels the findings on the laboratory steels. The grain size of the 3/4 in. plate was not determined as the Widmanstatten structure made it irrelevant. Rotating beam (R. R. Moore) fatigue tests were carried out on the 3/4 in. and 1—1/2 in. plate.

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*	Carbon	Manganese	Silicon	Sulphur	Phosphorus
	0·12	1·19	0·24	0·033	0·013

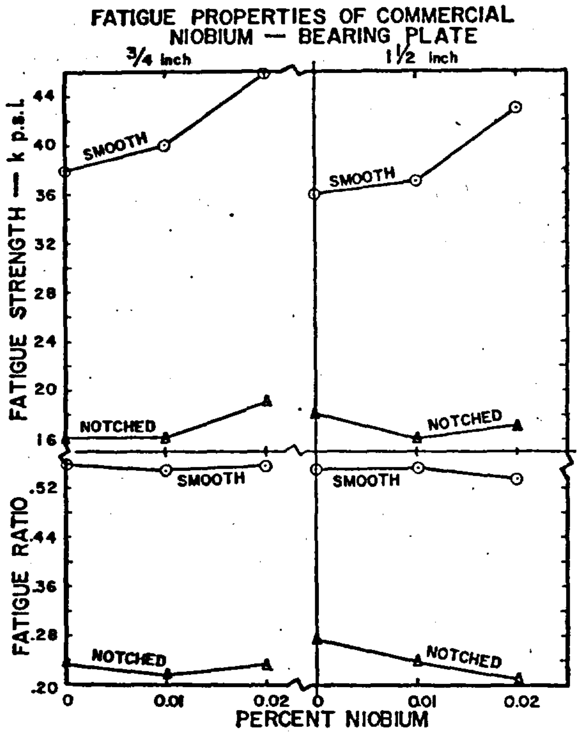


Fig. 10. The influence of niobium on the fatigue characteristics of commercially-produced niobium-beraring steel.

Both smooth (5—1/2 in. radius), and notched (60°C, 0·0002 in. radius, 0·075 in. depth) samples were tested.

Fig. 10 shows that the fatigue strength of the smooth specimens increases with niobium content. For the notched specimens, the pattern is inconsistent. Niobium has little effect on the fatigue ratio of the smooth specimens. In the case of the notched specimens, niobium has no effect on the 3/4 in. plate, but causes a small drop in the 1—1/2 in. plate. The fatigue ratio of all samples is in the order expected for plain carbon steels.

It was noted previously that there was relatively little variation in grain size of the laboratory steels rolled to 3/4 in. plate, although there was considerable variation in yield strength and transition temperature. For a given thickness, the same was generally true for the commercial plate. The indication is that niobium does not influence mechanical properties solely or perhaps even principally by its influence on grain size. Perhaps the best demonstration of the considerable influence of this element on transition temperature quite independent of its influence on ferrite grain size is shown in Fig. 11.

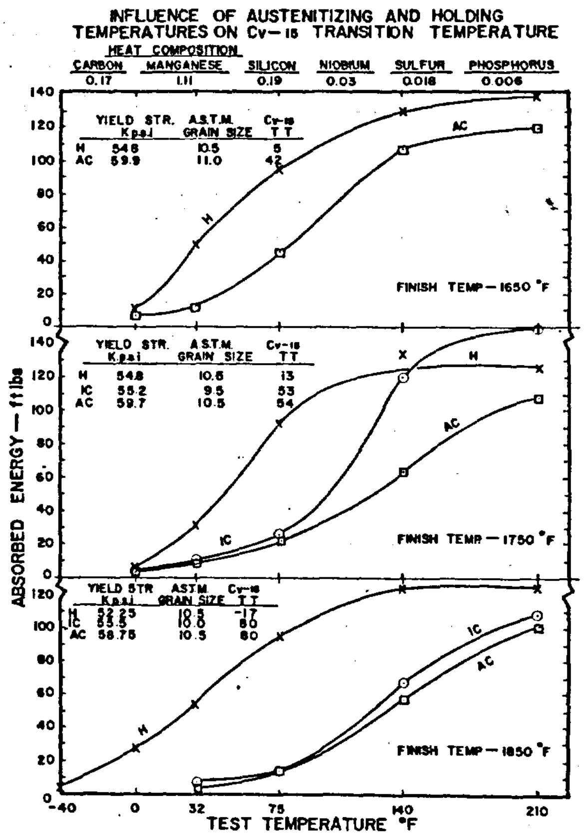


Fig. 11. The effect of finishing and holding temperatures on the yield strength, grain size, and impact transition temperature of niobium-bearing steel.

In this experiment, 1-1/4 in. plate containing 0·03% niobium was rolled in the laboratory to 3/4 in. in two passes. Three plates were finish rolled at 1010°C (1850°F), three at 950°C (1740°C and two at 900°C (1650°F). Prior to rolling, the samples were heated to 85°C (150°F) above the finishing temperature. After rolling, one plate was placed on edge to cool in still air (curves marked AC in the figure), one sample was held at the finishing temperature for one hour then cooled in still air (H curves in the figure) and, in the case of the two higher finishing temperatures, a further sample was held at 900°C (1650°F) for one hour then air cooled (IC curves). Tensile and impact tests were carried out as before. Grain sizes were determined by the Heyn Intercept Method. Attention is drawn to the bottom set of curves. The difference in transition temperature between samples held at the finishing temperature (H) and those directly air cooled (AC) is 54°C (97°F) although the grain size is identical. It will be seen that the

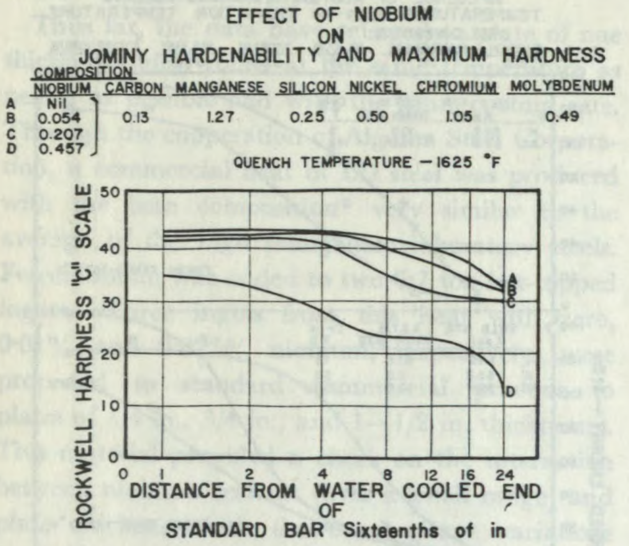


Fig. 12. The effect of niobium on the Jominy hardenability of an experimental low alloy structural steel.

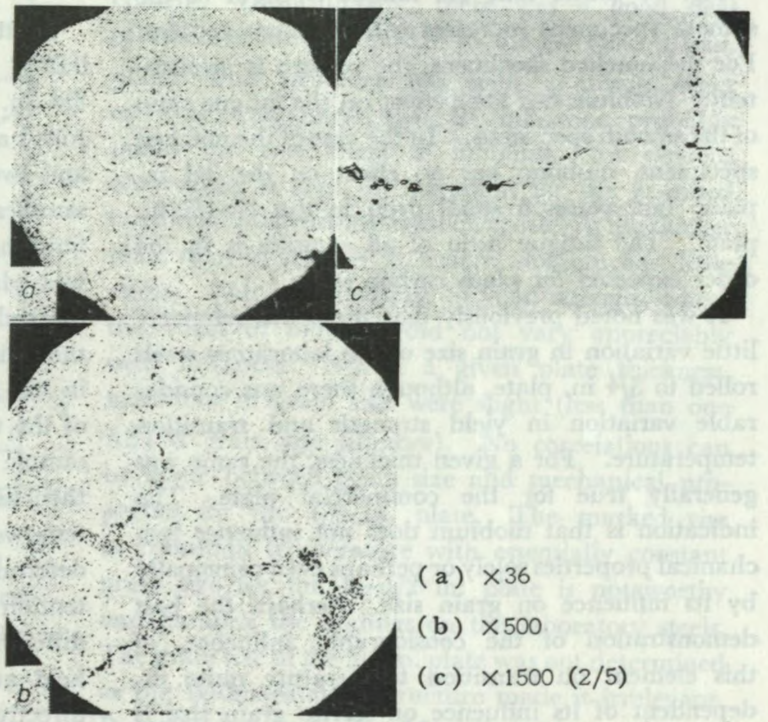
spread in the transition temperature curves increases as the finishing temperature increases.

A limited amount of work has been done investigating the effect of niobium on low alloy structural steel. Fig. 12 shows that it reduces the hardenability of a low carbon 1% manganese, 1% chromium, 0·5% nickel, 0·5% molybdenum steel, although the effect is not very pronounced except at about 0·5% niobium.

The reduction in maximum hardness at this level undoubtedly reflects the reduction in matrix carbon due to the presence of niobium carbides, which are insoluble at the low austenitizing temperature of 885°C (1625°F). Initial results from dilatometer tests on this steel, run at heating and cooling rates of 100°C/hr (180°F/hr) indicate that niobium raises the upper critical temperature slightly. At 0·05% niobium there was no effect, 0·02% raised Ac3 by 20°C (36°F) and 0·45% niobium raised it by 40°C (72°F).

2·5 Metallographic Examination

The metallography of these steels was not very revealing. As noted previously, grain size was not of paramount importance. Niobium did have some influence on the pearlite, tending to disperse the lamellar carbide of the base composition pearlite to a mixed lamellar-spheroidal form. The effect was not pronounced. At higher niobium levels relatively massive carbides occur. Photo. 1(a) shows the general distribution of these carbides. Photo. 1(b) shows the eutectic pattern of the carbides at the triple boundary points, and Photo. 1(c) illustrates the occurrence of strings of single carbides linking the triple boundary points. It is emphasized that these carbides are not associated in any way with the ferrite grain boundaries, nor do they appear to be located at prior austenite grain boundaries. It is probable that they have persisted from the freezing stage despite the fact that the steels were heated to 1095 to 1200°C (2000 to 2200°F) for forging, reheated to 1095°C (2000°F) for slabbing and finally reheated to 1065°C(1950°F) for finish rolling. In addition, the normalized material was reheated to 900°C (1650°F). The 1000°C (1830°F) section of the Fe–C–Nb ternary diagram according to *Eggers* and *Peter* * indicates complete solubility of niobium for steels with up to at least 0·40% carbon and 0·75% niobium. It is probable that insufficient time was allowed for the



(a) ×36

(b) ×500

(c) ×1500 (2/5)

Photo. 1. Photomicrographs of laboratory carbon steel containing 0·21% niobium. Relief polished, unetched.

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* *R. A.* Grange, *F. J.* Shortsleeve, *D. C.* Hilty, *W. O.* Binder, *G. T.* Motock and *C. M.* Offenhauer–"Boron, Calcium, Columbium and Zirconium in Iron and Steel"–John Wiley and Sons, Inc., New York (1957).

niobium carbides to go into solution. Of more importance is the fact that at levels of 0·07% niobium or less, where this element is most influential on the mechanical properties of carbon steel, these features are not prominent.

Recourse to the electron microscope yielded some further information. Photo. 2 shows an electron micrograph of a normalized 0·23% niobium steel.

There is some preferential precipitation in a grain boundary that may be the prior austenite grain boundary. Photo. 3 shows an electron micrograph of a normalized 0·07% niobium steel. The precipitate shows no preference for the ferrite grain boundary. The particles appear to be primarily spherical although a few cubes can be seen.

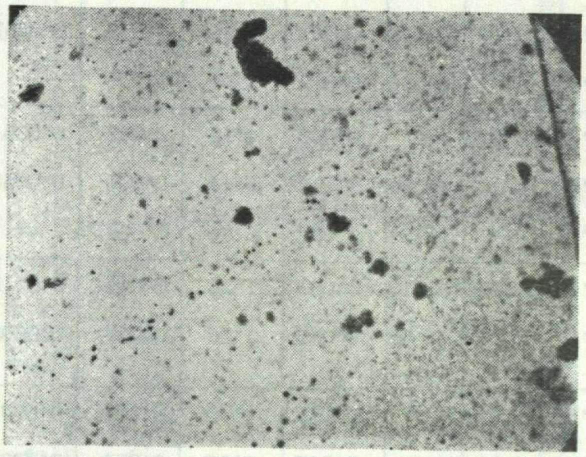
×40,000 (4/5)

Photo. 2. Electron micrograph of normalized 0·23 % niobium steel.
Carbon extraction replica.

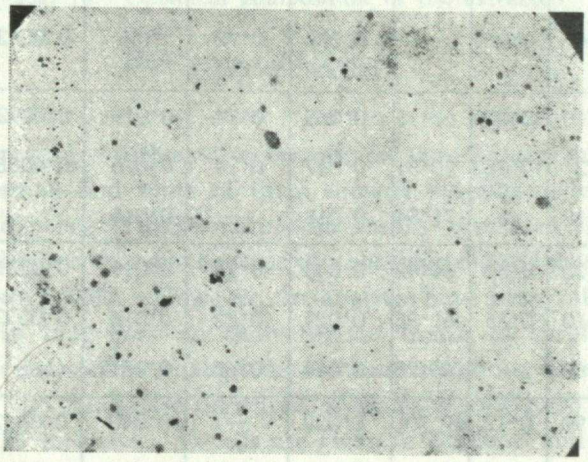
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Photo. 3. Electron micrograph of normalized 0·07 % niobium steel.
Carbon extraction replica.

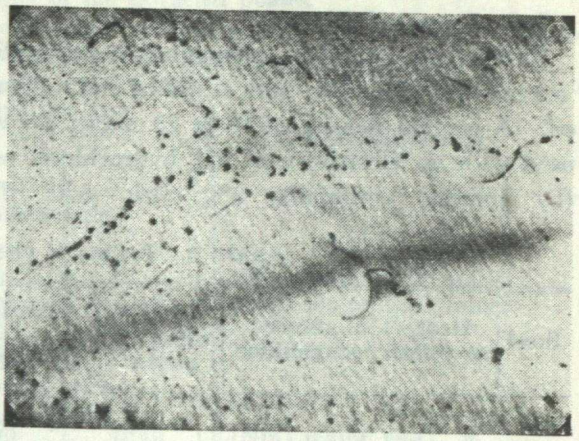

Photo. 4. Electron micrograph of normalized 0·023% niobium steel.
Electron transmission,

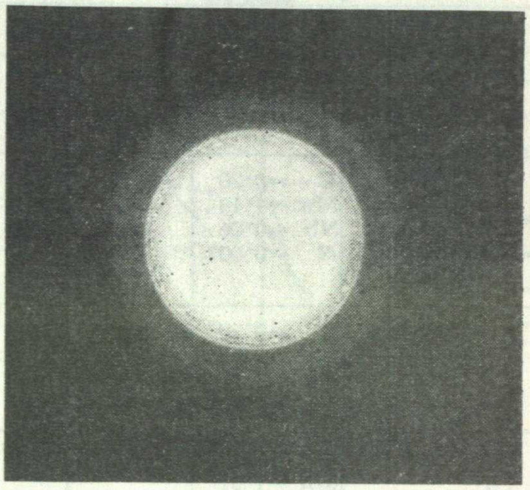

Photo. 5. Electron diffraction pattern of 0·023% niobium steel.

Photo. 4 shows an electron transmission micrograph of the commerical 0·023% niobium 1/4 in. plate in the normalized condition. This material was first machined and ground to 0·050 in. then chemically thinned to approximately 0·006 in. before being reduced to foil by the standard electropolishing technique. The precipitate in this commercial steel was found to be uniformly dispersed. The particles are generally irregular in shape. The particle size in all cases ranged upwards from about 50 Angstroms.

Positive identification of the precipitate was made by electron diffraction of the extraction replicas. In Photo. 5 the outer continuous ring is due to Fe_3O_4, the dotted ring just inside it to niobium carbide.

No precipitate could be detected in the as-rolled steel by examination of either replicas or foil.

2·6 Extracted Phases

Electrolytic extraction techniques were applied o a number of samples, both as -rolled and normalized. A current density of 15 to 25 ma/sq cm was used with an electrolyte of 3% hydrochloric acid (by volume) containing 3% citric acid. The anode efficiency of the cell varied over the range 1.02 to 1.08 g/amp/hr. The extracted residues or phases were chemically analyzed and a few samples were also examined by X-ray diffraction.

Regarding the chemical analyses of the residues, it may be mentioned that these results should be

Table 3. Results of chemical analysis of extracted phases.

Steel	Heat treatment	Steel composition	Wt % of extracted phases	Analysis of extracted phases, %					Calculated matrix composition, %			
				C	Mn	Nb	N	Fe	C	Mn	Nb	N
High manganese group												
5A	AR	C =0·19 Mn =1·37 Nb =0·010 N =0·009	1·63	7·41	2·63	0·07	0·032	70·95	0·070	1·34	0·0089	0·0085
	N		1·60	8·77	2·88	0·23	0·11	66·38	0·050	1·34	0·0064	0·0073
5B	AR	C =0·19 Mn =1·39 Nb =0·02 N =0·010	1·81	6·56	2·00	0·19	0·081	71·08	0·072	1·37	0·018	0·0086
	N		1·77	1·00	2·82	0·54	0·19	66·68	0·067	1·36	0·009	0·0067
	Sph		2·37	7·52	9·50	0·51	—	—	0·012	1·19	0·008	—
2B	AR	C =0·20 Mn =1·18 Nb =0·06 N =0·007	1·90	7·27	2·21	2·00	0·078	—	0·063	1·16	0·022	0·005
	N		1·82	7·08	2·83	3·02	0·12	68·50	0·072	1·14	0·005	0·0048
	Sph		2·80	6·89	8·49	2·02	—	69·24	0·007	0·96	0·0035	—
2A	AR	C =0·20 Mn =1·27 Nb =0·16 N =0·005	1·59	7·42	1·69	9·10	0·24	63·19	0·083	1·26	0·015	0·0012
	N		2·06	6·90	2·73	7·60	0·21	63·08	0·055	1·23	0·003	0·0008
6B	AR	C =0·19 Mn =1·31 Nb =0·52 N =0·007	1·97	7·90	1·80	22·50	0·25	—	0·035	1·30	0·078	0·002
	N		1·77	8·10	2·20	25·30	0·38	49·71	0·047	1·29	0·073	0·0002
Low manganese group												
14	AR	C= 0·18 Mn =0·76 Nb =Nil N =0·005	1·23	6·89	3·15	—	0·015	—	0·096	0·73	—	0·0048
	N		1·37	7·22	2·98	—	0·049	—	0·082	0·72	—	0·0043
9A	AR	C =0·15 Mn =0·77 Nb =0·02 N =0·010	1·14	7·47	3·20	0·10	0·073	67·15	0·065	0·74	0·019	0·0092
	N		1·52	6·80	2·37	0·2	0·082	70·51	0·047	0·74	0·016	0·0088
	Sph		2·03	6·49	—	0·18	—	71·94	0·018	—	0·016	
10A	AR	C =0·18 Mn =0·75 Nb =0·21 N =0·008	1·97	6·98	1·24	9·70	0·30	64·42	0·043	0·74	0·019	0·0021
	N		2·13	7·10	1·97	9·10	0·35	61·95	0·029	0·72	0·016	0·0005
	Sph		2·42	6·84	4·40	8·63	—	65·28	0·014	0·65	0·0011	—
10B	AR	C =0·18 Mn =0·74 Nb =0·51 N =0·004	1·89	7·99	—	24·90	0·12	49·70	0·029	—	0·040	0·0017
	N		2·02	8·47	0·92	23·40	0·15	41·94	0·0091	0·73	0·038	0·0009
	Sph		2·29	7·38	3·92	22·20	—	47·23	0·011	0·67	0·0012	—

AR-As-rolled; N-Normalized; Sph-Spheroidized.

Table 4. Results of X-ray diffraction analysis of the extracted phases.

Sample	Steel, per cent		Extracted phases			
			Chemical analysis, per cent			X-ray diffraction
	Nb	N	C	Nb	N	
			High manganese series			
5A*	0·007	0·009	7·41	0·07	0·032	Fe$_3$C
5B	0·017	0·010	7·00	0·54	0·19	Fe$_3$C major NbC trace
2B	0·06	0·007	7·08	3·02	0·12	Fe$_3$C major NbC minor
2A	0·16	0·005	6·90	7·60	0·21	NbC major Fe$_3$C 〃
6B	0·52	0·007	8·10	25·30	0·38	NbC major Fe$_3$C 〃
			Low manganese series			
10B	0·51	0·004	7·99	24·90	0·12	NbC major Fe$_3$C 〃

* As-Rolled, all others normalized.

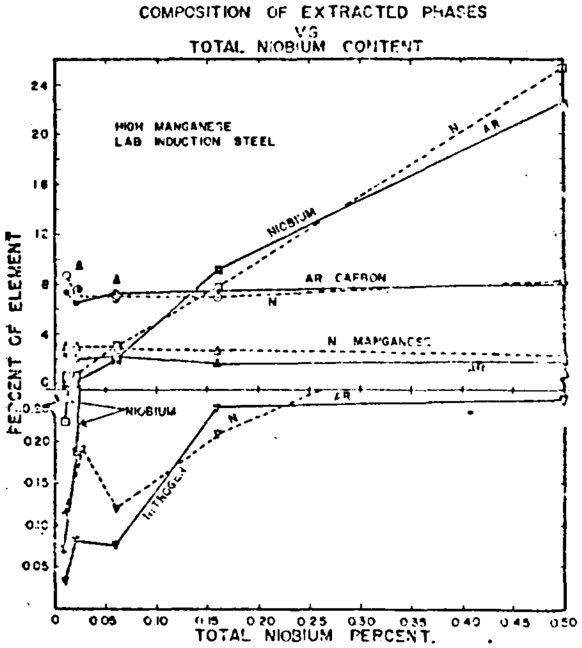

Fig. 13. Chemical composition of the extracted phases.
High manganese group.

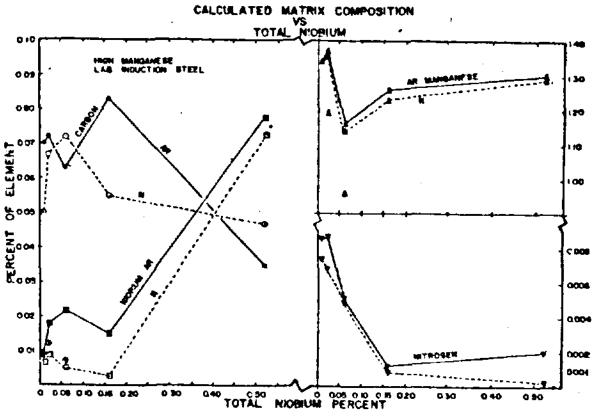

Fig. 14. Calculated matrix composition.
High manganese group of steels.

regarded with caution because the samples were very small and some of them showed pyrophoric tendencies. Furthermore, the results presented are limited in scope because of a shortage of the laboratory steels. However, the analyses have some value in that they do show certain trends of carbide composition change in niobium-containing steels. The results are portrayed graphically in Fig. 13 to 21 inclusive. Details are given in Tables 3 and 4.

Fig. 13 shows that the niobium content of the extracted phases in the high manganese group increases as the level of niobium in the steel rises. The same holds for nitrogen in the normalized steel, although it appears to reach a maximum of 0·24% at 0·15% niobium in the as-rolled steel. Carbon and manganese contents are relatively constant.

Fig. 14 shows the calculated matrix composition for the same group of steels. The niobium content rises, drops slightly, and then rises again with increasing total niobium on the steel. It is interesting that the first peak in the as-rolled condition corresponds roughly to the peaks in the yield strength and transition temperature curves.

Carbon shows an initial rise, then drops. The location of the points for carbon in the as-rolled condition at 0·06% and 0·16% niobium are considered doubtful. Both as-rolled and normalized steels show a sharp drop in matrix nitrogen at low levels of niobium. The curves for the low manganese group, Fig. 15 and 16, are generally similar to those for the high manganese group, especially

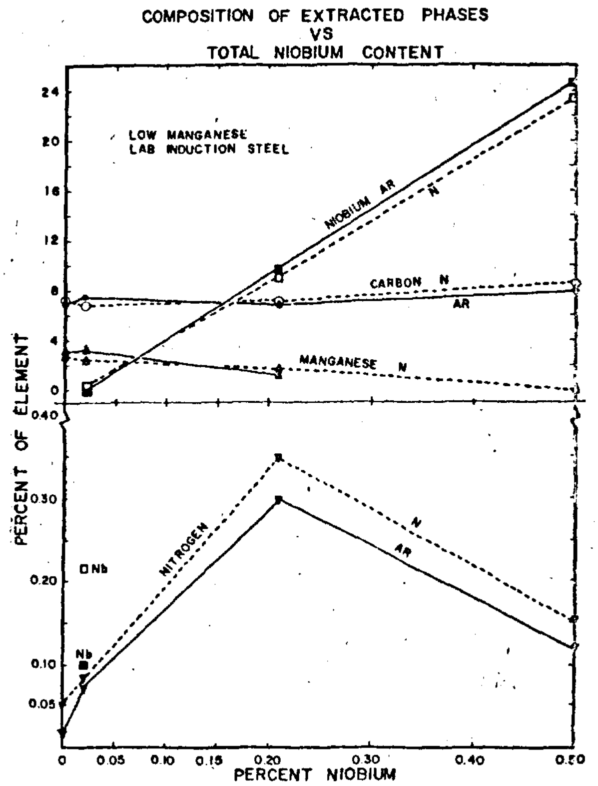

Fig. 15. Chemical composition of the extracted phases.
Low manganese. group.

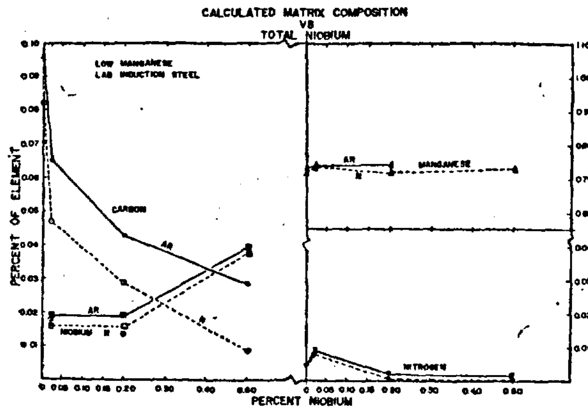

Fig. 16. Calculated matrix composition.
Low manganese group.

for the niobium, carbon and manganese contents of the extracted phases, although the nitrogen curves differ. Probably the most notable discrepancy is the form of the curve for calculated carbon content of the matrix. For these low manganese steels, the carbon content drops steadily with increasing total niobium, rather than increasing then decreasing as was found for the high manganese group.

X-ray diffraction analysis of the extracted phases, Table 4, identified the niobium carbide with quantities ranging from trace to 0·02% niobium steel to the major constituent in the 0·16% and 0·52% niobium steels. Iron nitride was not detected. The carbide and nitride of niobium are isomorphous so the increase in nitrogen found by wet analysis would not be detected by diffraction.

3. Summary

To summarize the principal points briefly, niobium raised the yield strength markedly in the as-rolled condition, less so after normalizing. The transition temperature was raised in the as-rolled state, but lowered after normalizing. The results of tests on commercially-produced steel agreed qualitatively with those of the laboratory steels. The effect of niobium on yield strength did not vary appreciably with plate thickness. However, its effect on transition temperature was influenced by change in thickness. It had no appreciable influence on the fatigue ratio. Niobium slightly reduced the Jominy hardenability of an alloy structural steel and about 0·5% also reduced the maximum hardness. In quantities over about 0·10%, it raised the upper transformation temperature somewhat.

Optical metallography showed niobium tended to decrease the ferrite grain size, although the degree of refinement was small in these steels. It also revealed the presence of relatively massive carbides, which were insoluble and which are considered to have had little or no direct effect on mechanical properties. Electron metallography showed a fine precipitate in the normalized steels but not in the as-rolled material.

In conclusion, it would seem from the evidence presented that the effects of niobium on the yield strength and transition temperature of normalized carbon steel are explicable, at least qualitatively, in terms of grain refinement and precipitation strengthening. The same cannot be said of the as-rolled state, where the change in yield strength is large, the change in grain size is small and no precipitate was detected. Furthermore, the transition temperature was raised markedly, despite slight grain refinement. A solid solution strengthening mechanism would be expected to raise the transition temperature to some degree but would not be expected to account for the large change in yield strength.

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