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Metallurgical Department
American Brake Shoe Company
Mahwah, New Jersey

FINAL TECHNICAL REPORT
W. A. L. Report No. 316/45-27

Copy No. 44

MARTENSITIC HIGH CHROMIUM HEAT RESISTANT STEELS
Including Data Obtained In The Period
February-August, 1953

CONTRACT NO. DA-30-069-ORD-237
NEW YORK ORDNANCE DISTRICT

August 24, 1953

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John A. Fellows
John A. Fellows
Research Metallurgist

UNANNOUNCED

FINAL TECHNICAL REPORT Including data obtained in the period February 14, 1953 through August 14, 1953.

From: American Brake Shoe Company
230 Park Avenue, New York 17, New York

To: New York Ordnance District
180 Varick Street, New York 14, New York

Contract No.: DA-30-069-ORD-237

RAD No.: ORDTB-1-12041

O. O. Project No.: TB4-161C

WAL: Ex. O#91219-09

Title: MARTENSITIC HIGH CHROMIUM HEAT RESISTANT STEELS

Object: To develop high-chromium high-strength materials for heat and erosion resistant applications in ordnance. The particular intent is to establish the upper chromium limit for which a satisfactory martensitic structure may be achieved on quenching. This structure, to be acceptable, must upon tempering provide adequate yield strength, ductility, impact strength, hot hardness and erosion resistance for ordnance purposes.

General Summary, August 14, 1951 - August 14, 1953

Initial efforts under this research contract were devoted to studies of the types of structure and the responses to heat treatment that could be observed with limited stocks of test bar material available from assorted laboratory experimental heats already on file. While the processing of hardness surveys and metallographic preparation of samples was in progress, a first group of heats was melted specifically for this project. The aim points of chemical composition were adjusted on the basis of available information concerning the expected phase relations of high chromium structures. The microstructures, chemical analyses, details of heat treatment and hardness values obtained were reported in Interim Technical Report No. 1 dated November 18, 1952 (WAL Report No. 316/45-14). In general, the following observations were found to apply:-

1. The phase relationships between alpha, austenite and carbides did not conform to the predictions of the constitution diagrams for the Fe-Cr-C system, presumably by reason of the manganese, silicon, nickel and nitrogen present in these commercial type alloys.
2. Ductility and hardness were difficult to obtain in simultaneous satisfactory degrees since low carbon alloys were soft from excessive ferrite, high carbon alloys were ferrite-free but brittle because of carbide networks and no carbide-free, ferrite-free intermediate zone was discovered.
3. Elimination of carbides by solution treatments at extremely high temperatures appeared possible but subject to the disadvantage of over-stabilization of the austenite resulting in lack of hardening.

The second phase of the work under this project related to examination of an ordered sequence of experimental melts aimed at 21% and 24% chromium with 0.35% carbon. Variations were provided in nickel and nitrogen content to permit definition of phase relationship dependence on composition as well as trends in mechanical properties versus methods of heat treatment. The experimental data are contained in Interim Technical Report No. 2 dated July 29, 1953 (WAL Report No. 316/45-23). The general conclusions obtained were as follows:-

1. Prolonged duration of solution treatment was found to provide slightly more stable austenite but did not contribute to improved hardening. A reduction in tempering time was observed to be detrimental.
2. Quantitative relationships between phase ratios, solution temperatures and nitrogen and nickel contents were obtained for the 21% Cr alloys making possible a correlation on a modified basis with published Fe-Cr equilibrium diagrams.
3. Of three compositions at 21% Cr with hardness and structure favorable for mechanical testing, one (0.35% Cr, 2%Ni, min. 7% nominal composition) displayed attractive yield strength at the desired hardness but possessed inadequate ductility. This alloy was, therefore, scheduled for special trials of more complex heat treatments.

The third interval of the program was devoted to completion of the survey of the effects of nickel and nitrogen upon the properties of the 24%Cr materials, to investigations of methods of improving ductility by various heat treatment schedules, to a brief trial of precipitation hardening of ferritic structures, and to experiments in attempts to eliminate carbide networks by titanium additions. The details of this work are covered in Interim Technical Report No. 3 dated August 14, 1953 (WAL Report No. 316/45-25). The results of this phase of the research may be summarized as follows:-

1. Complex solution treatments of a 21% Cr alloy were in general unproductive of better ductility but the practice of double tempering did offer attractive possibilities either with or without an initial isothermal anneal. Further work is recommended to explore the large number of possible combinations of such treatments with variations in temperature if this general program of research should at any time be resumed in the future.
2. Analysis of the structures of 24%Cr material made possible a quantitative expression of metallographic phase relationships in terms of solution temperature versus nickel and nitrogen contents. Approximate interrelations were then possible with available constitution diagrams for the Fe-Cr-C system on an adjusted basis.
3. Within the ranges of composition under study, the presence of nickel at 2% or higher appeared to provide a stability to the austenite which resisted transformation to a degree that prevented adequate hardening. At 1% nickel the desired hardness level could

just be obtained. For no combination of nickel and nitrogen with 24% chromium did there appear a possibility of satisfactory yield strength, due to the volume of alpha phase or of austenite as the case might be.

4. Attempts to harden a 100% alpha structure by carbide precipitation were unsuccessful. Such a technique requires that the initial particle size of the precipitate be sub-microscopic and that adjustment to proper hardness be then accomplished by agglomeration of the particles during a tempering cycle. In 30% Cr alloys dissolving approximately 0.25% C in the alpha phase at 2200°F., the carbide precipitate was too coarse after the initial quench for any appreciable hardening. No improvement in hardness was therefore possible through tempering.
5. A pilot trial of a titanium addition to a 0.75% C 24% Cr composition produced a structure of altered carbide shapes in a ferrite matrix in place of the dendritic cell patterns of carbide networks usually encountered for this material. Mechanical tests showed an encouraging level of ductility especially in view of the high carbon content. A further trial of this alloy combination was considered desirable.

The final period of experimental work has been concerned with the effects of titanium upon the structure of a 0.35% C, 24% Cr heat. A description of experimental procedure and results is contained in the body of this Final Technical Report. The following comments are pertinent:-

In the presence of 1/2% Mo and essentially 0% N, a 1-1/4% Ti addition created a structure of scattered carbonitrides in a matrix of ferrite containing a heavy and continuous grain boundary carbide network. Poor mechanical properties reflected the brittleness provided by these carbide envelopes. The contribution of the low nitrogen and the molybdenum addition to the creation of this unexpected structure could not be assessed from available data. Further trials of titanium were considered to offer some promise but appeared to require a lengthy program of research to define acceptable alloy combinations and structures.

From the results of the two years of work under this research contract, it appears that the development of a hard, ductile and tough cast alloy of high chromium content is a difficult assignment for levels of chromium above 21%. Considerable knowledge of structure and response to heat treatment as affected by composition has been gained for a variety of alloys. Achievement of the target properties appears most hopeful with a 0.35% C, 21% Cr, 2% Ni, low nitrogen composition. Even in this case, success depends on further investigation of possible heat treatment schedules. It should, at the same time, be recognized that this chromium level may be insufficient to provide the maximum desired improvement in erosion resistance. Successful controls of carbide shapes by special alloy additions may be possible but will not be simple to develop. Because of the long term research effort that seems necessary if this project were to be pursued to a final successful conclusion, it has been decided that this contract should be terminated at the end of the two year period, since justification of such a difficult program seems

doubtful in terms of the results to date. It is suggested, in the light of this experience, that a more fruitful approach to the problem might be through use of forged structures which should make possible a far more favorable combination of hardness and ductility.

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INTRODUCTION

A major difficulty encountered in the previous work under this project had been the very low ductility associated with compositions containing sufficient carbon to create the requisite volume of austenite for adequate hardening by transformation. This lack of ductility was primarily the result of the continuity of carbide envelopes which surrounded the austenite grains. Experimental additions of titanium to a high-carbon 24% chromium base analysis had provided evidence of a beneficial alteration of the carbide grain boundary films to a less interconnected shape and arrangement. Mechanical tests upon this high carbon material confirmed the inferences from the microstructure, displaying elongations of the order of 5% in contrast with values of 0.5% or less for unmodified compositions of much lower carbon content. Further investigation of the influence of titanium additions was therefore recommended, to be applied to a base analysis which had formed a part of the previous surveys of heat treatment. The scope of the further study of titanium modifications was however limited by a fundamental decision made in March 1953 regarding remaining work to be scheduled under this contract. It had been concluded from the experimental evidence accumulated, that attainment of the combination of properties specified as the target of this investigation, would be either very unlikely with an alloy bearing more than 20% Cr or at best would require a very prolonged research program. In view of these circumstances it was decided that it would be undesirable to continue this project to such a length. It was therefore agreed that this contract should be terminated in August 1953 on the completion of the second year of its existence. Because of the limited time thus remaining for experimental procedures as well as the large total of accumulated man-hours of effort already invested, it was determined that the remaining appraisal of the use of titanium should be restricted to a study of one additional experimental melt.

PROCEDURE

The base analysis for the final trial of a titanium addition was selected from the group of six experimental heats at 24% chromium reported in Interim Technical Report No. 3. Of these the most promising from the standpoint of hardness after heat treatment were the two compositions containing 1% nickel (Heats 52-751 and 52-754). The lower of the two nitrogen levels represented by this pair was chosen as perhaps permitting easier control of chemical analysis in melting. One modification was adopted through the introduction of a molybdenum addition to this final heat. This was made on the basis that additional stabilization of austenite might be required in conjunction with the presence of titanium and that molybdenum had been a favorable element for erosion resistance in ordnance service and was therefore preferable rather than increased nickel.

A 115 lb. heat was melted in a basic lined high frequency induction furnace using virgin materials in the charge. The titanium was added as titanium sponge, in part to the bath just before tap and the remainder to the stream as the heat was tapped into the ladle. Deoxidation in the ladle was provided by additions of .07% Ca (as CaSi), .07% Se (as FeSe) and .10% Al. The tapping temperature was Freeze Point plus 400°F. and pouring was begun at Freeze Point plus 300°F. Ten tensile test bar castings, three Charpy impact test bar blanks and one chemical analysis wedge were poured in baked core sand molds. The aim point and final chemical analysis of this heat are reported in Table I.

TABLE I

Chemical Composition of 24% Cr Steel With Ti and Mo Additions

Designation	Chemical Analysis											
	C%	Mn%	P%	S%	Si%	Cr%	Ni%	N%	Ti%	Mo%	Al%	Se%
Aim Point	.35	.80	min.	min.	.50	24.0	1.00	.10	1.50	.50	*	*
53-394	.36	.86	.014	.009	.64	23.63	1.06	.007	1.21	.53	.16	.044

*Added for deoxidation purposes - values reported are residuals.

Although the nitrogen proved to be unusually and undesirably low, the remainder of the constituent elements were deemed to be acceptably close to the aim point and there did not appear to be justification for remaking the heat on the basis of the nitrogen variation alone. Heat treatment of coupons was therefore scheduled for both tensile and Charpy impact tests, employing solution temperatures of 1900°, 2050° and 2200°F. with a subsequent tempering treatment at 1200°F. for all specimens. Standard .505" diam. by 2" gage tensile specimens were machined and tested at a cross-head speed of 0.1" per minute with the results listed in Table II.

TABLE III

Mechanical Properties of 24% Cr Steel With Ti and Mo Additions

Bar No.	Yield Strength - psi.			Tens. Str. psi	%	%	BHN	Heat Treatment
	0.01%*	0.1%*	0.2%*					
53-394-1 -2	3,000 2,500	- -	- -	10,500 10,000	0 0	0 0	187 187	{1900°F-2hrs-OQ; 1200°F-6hrs-OQ.
53-394-3 -4	** 50,000	** 57,500	** 59,500	** 62,400	** 1.0	** 3.5	** 187	{2050°F-2hrs-OQ; 1200°F-6hrs-OQ.
53-394-5 -6	48,000 52,000	58,500 60,500	60,500 62,500	65,200 65,100	2.0 1.5	0.7 0.2	196 196	{2200°F-2hrs-OQ; 1200°F-6hrs-OQ.

*Percent permanent offset

**Broken in stamping identification number after machining.

N.B. The following comment was recorded during tensile testing,

"53-394-1, -2, -4 & -6 showed cracks before testing".

Due to scheduling difficulties in routing other laboratory work through the machine shop, the completion of the machining of the Charpy impact specimens was delayed past the date on which the tensile tests were pulled. After inspection of the tensile test data, all further work on the impact test coupons was cancelled as being futile for such brittle material.

A metallographic specimen was prepared from the fracture of one of each pair of tensile test specimens with the polished face arranged parallel to the longitudinal axis. A representative structure is illustrated in Figure 2-6.

DISCUSSION

The extreme lack of ductility evidenced by the data of Table II was quite unforeseen from the experience with the earlier heat (52-864) at 0.71% C reported in Interim Technical Report No. 3. Metallographic study of the structures revealed a complete and continuous grain boundary envelope of carbides. These were not of the branched complex shapes typical of the straight iron-chromium carbides but were made up of fairly simple sequences of titanium carbonitrides with chromium carbides interspersed. The matrix proved to be alpha phase with no evidence of austenite having existed at any of the solution temperatures. The majority of the titanium compounds were lavender-gray carbon-rich carbonitrides which existed both at the grain boundaries and scattered uniformly within the grains. In much less quantity were the golden-pink nitrogen-rich carbonitrides, frequently rod-shaped and usually situated adjacent to but not a part of the grain boundary envelopes.

There was some slight visual evidence after careful scanning that solution treatment at 2200°F. had provided a tendency toward more frequent discontinuities in the carbide envelopes than resulted from the 1900°F. treatment. This was in line with the small differences recorded in tensile elongation but the distinction proved to be difficult to present photographically because of the limited field of view at any useful magnification. A single structure (Figure 2-c) has therefore been reproduced as typical of all three solution treatments.

In analyzing the effects of titanium upon this composition, several accompanying influences must be taken into account. These include, in the general picture, the contributory effects of carbon, nitrogen and molybdenum in particular. The preliminary trial heat (52-864) contained 0.71% C and 1.49% Ti. This melt may be compared directly (as in Figure 1) with an early experimental heat (51-638 - reported in Interim Technical Report No. 1) of the same carbon level. Aside from the titanium addition the two analyses are essentially identical with the one exception of the nitrogen contents. The structures are quite different being a matrix of 100% austenite with grain boundary carbide envelopes in the high-nitrogen, titanium-free alloy whereas with the medium nitrogen and 1-1/2% titanium material an alpha phase matrix exists surrounding islands of austenite. These with their enclosed carbides account for approximately one-third of the volume. Not all the carbon is alloyed with the titanium but appreciable quantities exist as iron-chromium carbides. The shape of these carbides appears to have responded to the presence of the titanium by assuming a less continuous arrangement which shows perhaps to better advantage after heat treatment at 1900°F. as in Figure 12-b of Interim Technical Report No. 3. (A direct comparison of the two structures at 1900°F. is not available since this temperature was not employed with 52-638.) The net effect of the titanium addition with the decrease in nitrogen has therefore been a marked shift in phase-ratio toward a ferritic structure plus an alteration of carbide habit. In the first of these the two elements may be considered as having the same type of effect and therefore enhance each other. The second response would appear to be caused by the titanium alone.

Appraisal of the second titanium-modified heat requires a different set of standards of comparison because of lower carbon and nickel contents. Since no counterpart of the extremely low nitrogen level of 53-394 is available, a sequence of two heats has been chosen for contrast to illustrate a trend in this respect. Figure 2-a, illustrating the structure for 0.23% N, shows only 10% alpha in a matrix of austenite and exhibits very few excess carbides which existed at the solution temperature. At one-half this nitrogen content, Figure 2-b, the alpha phase volume has more than tripled and a dendritic pattern of excess carbides has appeared. The final step, Figure 2-c, with essentially no nitrogen and with the introduction of 1-1/4% Ti and 1/2% Mo, provides what is

essentially a complete counterpart of Figure 1-a, namely a continuous carbide network with a 100% alpha matrix instead of the austenite of Heat 52-638. Some contribution toward the formation of this ferrite may be attributed to all three of the variables (N, Ti, Mo) but the major effect seems likely to have stemmed from the drastic reduction in nitrogen content if the influence of this element on phase relationships as observed in earlier studies may be cited in comparison. This absence of nitrogen was, of course, unplanned but was an inadvertent melting loss that illustrates some of the difficulty in control of analysis of this class of alloys.

Unfortunately for comparison purposes, no example of a similar alloy carrying molybdenum but free of titanium is available. To classify the very unfavorable presence of the carbide network as the direct effect of molybdenum appears to be unwarranted. Without the advantage of further trials, the best that may be deduced at the moment would seem to be the assignment of this structure to the combined influence of the absence of nitrogen and the presence of molybdenum. This is a difficult interpretation of behavior at the moment simply from the lack of sufficient data. With opportunity for further research, there should be no problem in clarifying the true cause of the structure.

One further aspect of the titanium-modified alloys merits particular thought. The evidence of the microstructures suggests, in accordance with the recognized influence of titanium, that the greater affinity of carbon for titanium has displaced a quantity of chromium into solution in the matrix which otherwise would be separately compounded with iron in a carbide structure. In this sense the effect of titanium has been to increase the available chromium content of the matrix. This could have two possible results, namely accentuated formation of ferrite (and consequent softness) and a possible enhancement in erosion resistance. This leads to the conjecture as to whether, for a net desired effect of chromium, a smaller amount of this element might achieve the same final result in the presence of titanium that would otherwise require a much higher chromium content. If this reasoning is correct, it opens one hopeful approach to the problem of elimination of excessive volumes of ferrite while still retaining desirable carbide shapes and an unimpaired contribution toward erosion resistance.

It seems pertinent to record one aspect of the metallographic preparation of the samples of Heat 53-394. The standard "grain size reagent" (5 ml. HCl 1 gm. $\text{HOC}_6\text{H}_2(\text{NO}_2)_3$, 100 ml. $\text{C}_2\text{H}_5\text{OH}$) attacked the interface between the iron-chromium carbides and the matrix so vigorously that, even with minimum etching times a deep and wide fissure was created between them. This showed so prominently as a dark gap in the surface that proper interpretation of the structure was not possible from photomicrographs. Trials of a variety of etching reagents recommended for chromium base alloys were unsuccessful with this material, in some cases by reason of unfavorable selective attack and in others because of lack of uniformity of delineation. The final reagent used was a variation of the "grain size" formula with the hydrochloric acid reduced to 3 milliliters. This provided an acceptable if not ideal definition of structure. It was used without difficulty for the companion photomicrographs of Figures 1 and 2 to eliminate any possible differences in interpretation arising from divergent etching techniques.

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-A-

CONCLUSIONS

The addition of titanium to a 24% Cr base analysis, while successful in contributing to non-embrittling carbide shapes in a high-carbon medium-nitrogen preliminary heat, has resulted in an almost complete lack of ductility in a similar medium-carbon alloy further modified by absence of nitrogen and presence of molybdenum. The brittleness was caused by a continuous carbide network at the grain boundaries. The reason for this carbide envelope, which bears no resemblance to the structure of the previous titanium-modified melt, is not well understood on the basis of available data.

The use of titanium introduces directly or indirectly a pronounced tendency to form the alpha phase at solution temperatures. If adequate hardening by transformation is to be accomplished, this tendency must be counteracted by lower chromium, higher carbon, additional alloy content, or perhaps all three. Of these, the use of any appreciable quantity of alloy additions represents the most serious problem in that the creation of austenite in this fashion also frequently renders it extremely resistant to transformation.

The experience to date in the melting of titanium-modified alloys indicates that control of analysis, particularly with respect to nitrogen and titanium, may be somewhat difficult in production. The size and cost of the titanium addition also represents an appreciable expense, although it should be recognized that the work which has been possible under this project has not been sufficient to define the minimum effective titanium content.

It seems apparent that the use of titanium in high chromium alloys offers some possible advantages in control of carbide habits but that development of the proper alloy balance, the control of melting losses and the proper understanding of structures would require a prolonged program of research.

RECOMMENDATIONS

In view of the lengthy and involved research effort which appears unavoidable if the desired combination of properties is to be developed in a cast steel of more than 20% Cr content, it is recommended that no further investigations be conducted under this research contract. It is also suggested that a continuation of this project under any new contract may likewise be of doubtful benefit as far as cast materials are concerned. The definite requirement of a minimum hardness can only be met with a sufficient carbon addition to ensure that the soft alpha phase is present in minor quantity. If quite high chromium contents appear to be essential this necessary quantity of carbon (or carbon substitutes) must reach higher and higher percentages as chromium is increased. Such carbon levels unavoidably form grain boundary carbide networks in cast structures that are very resistant to spheroidization efforts through heat treatment and that forfeit nearly all chances for ductility.

It is therefore recommended that further efforts to develop a high chromium, high carbon steel of this nature be confined to forged structures. This treatment should make possible the breakdown of the carbide envelopes by hot work which should enhance to a major degree the attainable levels of ductility.

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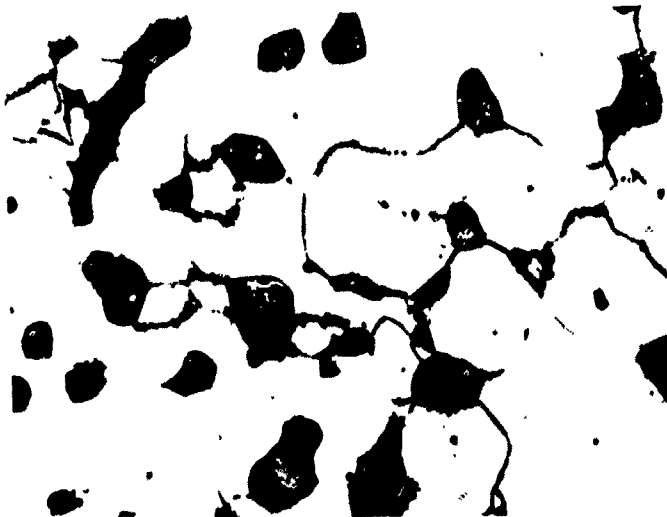
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NITROGEN, TITANIUM AND MOLYBDENUM INFLUENCES ON STRUCTURE

Etchant: Modified Grain Size Reagent

Magnification: 250X



52-754-3B

U-304302

Treatment: 2200°F-2hrs-OQ;
1100°F-6hrs-OQ

24% Cr Steel - High N, No Ti, No Mo
0.39%C 23.4%Cr 1.14%Ni 0.23%N

A matrix of austenite encloses an island system of alpha phase areas that measure 10% of total volume. Carbides in solution in the ferrite at 2200°F have precipitated and agglomerated to produce the dark speckled appearance. Numerous bands of fine pearlite are visible at grain boundaries and bordering alpha islands.

Figure 2-a



52-751-3B

U-303702

Treatment: 2200°F-2hrs-OQ;
1100°F-6hrs-OQ

24% Cr Steel - Medium N, No Ti, No Mo
0.39%C 23.4%Cr 1.12%Ni 0.11%N

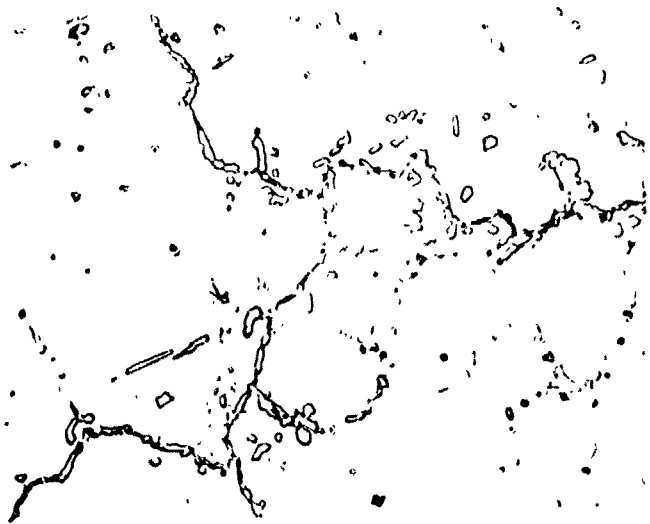
Lower nitrogen has increased the alpha phase volume to 32%. Precipitated carbides darken the color of the ferrite. A change in carbide solubility in the austenite has produced a network of excess carbides in a dendritic pattern. Narrow channels of fine pearlite at grain boundaries connect many of the alpha islands. Microhardness and magnetic tests indicate some transformation in matrix in original quench and after the temper following a segregated pattern.

Figure 2-b

NITROGEN, TITANIUM AND MOLYBDENUM INFLUENCES ON STRUCTURE (Cont.)

Etchant: Modified Grain Size Reagent

Magnification: 250X



53-394-6

W-203701

Treatment: 2200°F-2hrs-OQ;
1200°F-6hrs-OQ

24% Cr Steel - Low N, With Ti and Mo
0.36%C 23.6%Cr 1.06%Ni 0.01%N 1.21%Ti 0.53%Mo

Grains of alpha phase are outlined by grain boundary carbide networks. White, sharply outlined particles are iron-chromium carbides. Interspersed in network and scattered through grain interiors are titanium carbonitrides that have not been outlined by the etch and which appear lavender-gray when viewed through the microscope. Little evidence is found of carbides precipitated from the ferrite as in the unmodified Cr alloys. No transformation products are present since no austenite existed at the solution temperature.

Figure 2-c

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