EFFECT OF ALLOYING ELEMENTS ON TEMPERED MARTENSITE EMBRITTLEMENT AND FRACTURE TOUGHNESS OF LOW ALLOY HIGH STRENGTH STEELS

January, 1971

C. Vishnevsky
TRW Inc.
Materials Technology Laboratory
Equipment Group
Cleveland, Ohio

Final Report - Contract DAAG 46-69-C-0060

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Prepared for
ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172
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ABSTRACT

A study was performed on the influence of various elements on the notch bend fracture toughness at 75°F and -100°F of 35CrMoV martensitic steels tempered between 400 and 800°F. The elements examined included C, Mn, Si, Cr, Ni, Mo, Co, V, and Al. The overall variation in room temperature yield and tensile strengths for twenty-four steels was 155-230 ksi yield strength and 188-288 ksi tensile strength.

Tempered martensite embrittlement was revealed by testing at -100°F, whereas 75°F tests were insensitive to this phenomenon. The elements C, Mn, Cr, Mo, and Co generally reduced toughness at both test temperatures and, particularly in large quantities, were undesirable on a toughness-yield strength basis.

The influence of Si, Ni, V, and Al was more complex. A steel containing 0.29% V exhibited excellent properties while Al in amounts of 0.18% and 0.30% offered no advantage over a level of approximately 0.05%. Increasing amounts of Ni in the range of 1.15% to 6.12% were highly beneficial to low temperature toughness at a sacrifice in yield strength. This element provided an improved toughness-strength balance on the basis of tensile strength but not yield strength. Particularly attractive properties were obtained with a steel which, except for a slightly lower C content (0.36%) and a higher level of Ni (3.05%), resembled the commercial alloy, 300M.

This study indicated a relatively slight dependence of fracture toughness on composition at 75°F, but a large overall variation in toughness at -100°F. Consideration should be given to this behavior in selecting steels for applications involving low service temperatures.
FOREWORD

This report, TRW ER 7384-1, presents the final results of a program performed by the Materials Technology Laboratory of the TRW Equipment Group for the Army Materials and Mechanics Research Center under Contract DAAG 46-69-C-0060, D/A Project IT06210S328. The work was conducted by C. Vishnevsky. Dr. F. R. Larson and Mr. F. L. Carr directed the program for AMMRC.

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I INTRODUCTION

High strength low alloy steels are utilized in advanced military and aerospace applications over a wide range of strength levels approaching approximately 300 ksi ultimate tensile strength. A limiting factor in most cases is insufficient crack propagation resistance or toughness at ultra high strength levels. Although high alloy steels are available which offer significant advantages in terms of toughness, considerable interest continues to exist in improving the performance of low alloy steels.

One such application area involves gun tube steels for large cannon, such as the 175mm M113, which are currently produced in the yield strength range of 160-180 ksi. No specific compositional requirements exist for these steels, although they usually are a modified 4335 composition containing about 3% Ni and 0.1% V (1). Qualification of steels for gun tube use is based on tensile and -40°F Charpy V-notch impact specifications. The firing pressure and range capability is in part limited by the yield strength. Because toughness tends to decrease as strength is raised, any improvements in performance through a strength increase would require maintaining a high toughness level.

Considerable data exist on toughness of various steels both in this strength range and higher strength levels. Reviews of the literature on compositional effects in low alloy steels and the relatively recently developed high alloy types appeared in previous reports (2,3). Although certain generalizations on alloying effects are possible, the complexity of steel compositions and the large number of steel types preclude the designing of new steels without additional experimentation.

Previous work had shown that the toughness of 160-180 ksi yield strength gun tube steels can be improved by compositional changes, without necessarily raising the total alloy content (4). The purpose of the present work was to provide further insight into compositional effects in martensitic .35C-3Ni-Cr-Mo-V steels tempered to strength levels above those currently utilized in large gun tubes. The elements examined included C, Mn, Si, Cr, Ni, Mo, Co, V, and Al. These were systematically varied at three levels and their influence on fracture toughness evaluated both at room temperature and -100°F.
II MATERIALS AND PROCEDURE

The effects of systematic changes in the levels of C, Mn, Si, Cr, Ni, Mo, Co, V and Al and interactions of Cr and Mo were studied using a total of twenty-four experimental steels whose compositions are given in Table I. Of these, heats 2 and 20 represented the base composition. Deliberate variations from this composition in the other steels are underlined in this table. The levels of individual alloying elements that were studied are summarized below together with the nominal or average values of Cr and Mo used in a full factorial three level study of these elements involving heats 2 and 20, 3, 4, 5, 6, 23, 24, 25, and 26.

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<tr>
<th>Element</th>
<th>Weight %</th>
<th>Cr-Mo Interactions (Full Factorial, Three Level)</th>
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<td>.37*   .43</td>
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<tr>
<td>Mn</td>
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<tr>
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<tr>
<td>V</td>
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<tr>
<td>Al</td>
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<td>.18    .30</td>
</tr>
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* Base composition; average of heats 2 and 20

The steels were produced in 40 lb. heats using a vacuum induction melting practice described previously (4). Ingots weighing approximately 25 lbs. were forged at 2000°F, using a 6 to 1 reduction, into bars having a cross section of 2 3/4 x .650 inches.
### Table I

**CHEMICAL ANALYSIS OF CAST HEATS**

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<th>Heat No.</th>
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* Underlined values are deliberate variations from base composition.
** Acid soluble
After forging the steels were normalized at 1650°F for 1 hour. Rough machined blanks for tensile and notch bend fracture toughness specimens were cut with the specimen axis parallel to the working direction, austenitized for 1/2 - 3/4 hour at 1550°F, quenched in agitized oil, and double tempered at 400, 500, 600, 700, and 800°F for 1+1 hours.

Tensile tests at room temperatures and -100°F were conducted using the 1/4 inch diameter, one inch gage length specimens shown in Figure 1(a). The pulling speed in all cases was .01 inches per minute. An air environment was used for the room temperature tests while -100°F was achieved in a stirred acetone bath chilled with dry ice. Chart records of load versus extension up to necking were obtained at both test temperatures. For room temperature tests a strain gage beam type extensometer was directly attached to the specimen, while at -100°F a modified creep extensometer, attached to the specimen gage section, transmitted the deformation to the strain gage extensometer outside the bath. The results obtained from the tensile tests consisted of the tensile strength, yield strength, elongation in one inch, reduction of area and work hardening exponent (n) as defined by $\sigma = Ke^n$ where $\sigma$ = stress, $K$ = constant, and $e$ = true plastic strain. The work hardening exponent was calculated from a linear regression analysis of log $\sigma$ vs. log $e$. The procedure was identical to that previously described (4), and the standard deviations of n and linear correlation coefficients were such as to indicate good straight line fits, thus supporting the validity of using a simple power relation to describe the strain hardening of these steels.

Fracture toughness tests were performed at room temperature and -100°F using the notch bend specimen shown in Figure 1(b). In order to avoid possible damage to the clip gage used to monitor crack opening displacement, ethyl alcohol instead of acetone was used for the low temperature bath. Room temperature tests were performed in ordinary air.

The preparation of the test specimens consisted of cutting a narrow slot with a grinding wheel and extending its base approximately .050 inch by electric discharge machining. The width of this extension was approximately .015 inch. The length of the initial notch is denoted as $a_0$ in Figure 1(b). The notch was further extended at least an additional .050 inch by fatiguing the specimen in cantilever bending, so that the final total crack length, $a$, was within the limits $.45 - .55W$ where W is the specimen width, nominally 1.120 in.

The test techniques were in accord with the ASTM recommendations for plane strain fracture toughness testing (5). The procedure consisted of loading the specimens in three point bending and simultaneously recording load and crack opening displacement as measured by a sensitive strain gaged
Figure 1. Dimensions of tensile and fracture toughness specimens.
beam clip gage held by knife edges at the specimen surface. In these tests the knife edges were single edge razor blades spot welded to the specimen. The test configuration and the equation used to calculate critical stress intensity are illustrated in Figure 2.

Based on a graphical analysis of the test record, the details of which are described elsewhere (5), a load \( P_Q \) was obtained and used to compute a tentative plane strain fracture toughness value, \( K_Q \). In order to \( K_Q \) to be accepted as a valid \( K_{IC} \), a series of specific requirements must be satisfied. The most critical of these are that the test record of load versus crack opening displacement pass certain tests for linearity and that both the specimen thickness and crack length are greater than or equal to \( 2.5 \left( K_Q / \sigma_{YS} \right)^2 \), where \( \sigma_{YS} \) is the 0.2% offset yield strength. \( K_Q \) values that are not also \( K_{IC} \) should not be used in estimating \( K_{IC} \), but can be of value for screening purposes or indicating trends for specimens of the same type having the same thickness and crack length.
EQUATION 1): \[ K_Q = \frac{P_Q}{BW^{3/2}} \left[ 2.9 \left( \frac{a}{W} \right)^{1/2} - 4.6 \left( \frac{a}{W} \right)^{3/2} \\ + 21.8 \left( \frac{a}{W} \right)^{5/2} - 37.6 \left( \frac{a}{W} \right)^{7/2} + 38.7 \left( \frac{a}{W} \right)^{9/2} \right] \]

\[ P_Q = \text{LOAD OBTAINED FROM TEST RECORD, LBS.} \]
\[ B = \text{SPECIMEN THICKNESS, IN.} \]
\[ S = \text{SPAN LENGTH, IN.} \]
\[ W = \text{SPECIMEN WIDTH, IN.} \]
\[ a = \text{CRACK LENGTH (MACHINED NOTCH PLUS FATIGUE CRACK)} \]
\[ K_Q = \text{TENTATIVE PLANE STRAIN FRACTURE TOUGHNESS VALUES, PSI/IN.} \]
\[ K_{IC} = \text{KIC (PLANE STRAIN FRACTURE TOUGHNESS) IF ALL CRITERIA FOR VALID TEST ARE SATISFIED.} \]

Figure 2. Schematic representation of notch bend fracture toughness test setup and equation used to calculate fracture toughness.
The results of individual tensile and fracture toughness tests for all steels are presented in Tables A1 to A1V of the Appendix. The tensile data consisted of the tensile and yield strengths, elongation, reduction of area, and work hardening exponent. The fracture toughness test results in Tables A1II and A1IV include the tentative plane strain fracture toughness ($K_Q$) for all specimens. $K_Q$ values which satisfied all requirements for a valid plane strain fracture toughness number are further denoted as $K_{IC}$. At room temperature approximately 50% of the $K_Q$ values were not valid $K_{IC}$, but at -100°F only about 15% of the tests were only $K_Q$.

The primary causes for rejection of tentative $K_{IC}$ values were failure to meet crack length and specimen thickness requirements and excess plasticity as indicated by insufficient linearity of the test record. The failure of a large portion of the room temperature tests to satisfy the criteria for a valid $K_{IC}$ largely reflects insufficient specimen size for the yield strength and toughness of these steels. At -100°F toughness was lower, the yield strength was increased, and the number of rejections was substantially reduced.

The analysis of individual alloying elements was conducted with reference to a base steel which was the average of two heats (2 and 20) having the same nominal composition. The tensile and fracture toughness properties of these two base composition heats were averaged for comparison with other steels. The higher strength and lower toughness of heat 20 is probably the result of its slightly higher carbon content.

Because the effects of alloying element variations were studied at different test and tempering temperatures, an interpretation of the results only in terms of toughness would obviously be insufficient. It is particularly important to include the contribution of strength, as affected by composition, to toughness. In general, as strength increases the fracture toughness of low alloy steels is reduced. Ideally, an analysis of compositional effects on toughness would differentiate between a change in toughness intrinsically caused by alloying to that produced by a change in strength due to alloying. Although it is possible to relate $K_{IC}$ to strength for specific steel types (6), no satisfactory method exists for making an accurate strength effect correction.

Attempts to correlate $n$ values with fracture toughness proved unsuccessful, in contrast with earlier work on steels having a room temperature yield strength in the 160-180 ksi range (4). These data are included for possibly future use and reference purposes. Note the consistently sharp rise in $n$ at the 400°F tempering temperature. This reflects a leveling off or even drop in yield strength on tempering below 500°F, while the tensile strength continues to rise. A quantitative representation of $n$ as reflected by the tensile to yield strength ratio is shown in Figures 1A and 2A of the Appendix. The work hardening exponent increased linearly with increasing tensile to yield strength ratio and the degree of scatter was slight.
However, it is possible to analyze the effects of composition on strength and toughness on a combined basis, because from an engineering standpoint, the important factor is maximum toughness at a given strength level. Although it is possible to merely plot fracture toughness versus yield or tensile strength, a more useful representation can be made in terms of the crack size factor, \((K_0/\sigma_y)^2\) or \((K_{IC}/\sigma_y)^2\), versus strength. The crack size factor is directly proportional both to the plastic zone size and the size of a critical defect in any structure. The actual crack size is obtained by multiplying the crack size factor by appropriate geometry dependent terms for the structure in question.*

The effects of alloying elements on fracture toughness are presented in two sections. The first deals with single element variations from the base composition of C, Mn, Si, Cr, Ni, Co, Mo, V and Al, while the second describes the interactions of Cr and Mo.

A. EFFECTS OF INDIVIDUAL ELEMENT VARIATIONS

Fracture toughness results for single element series are presented in Figure 3 to 21. For each element the data are summarized in terms of plots of stress intensity factor, \(K_i\), versus tempering temperature and \((K_{IC}/\sigma_y)^2\) versus \(\sigma_y\). In the former graphs individual data points appear at 100°F intervals and distinguish between \(K_{IC}\) and merely \(K_0\) values. \(K_0\) or \(K_{IC}\) for the base composition are averages for heats 2 and 20. At a particular tempering or test temperature the average was denoted as \(K_0\) unless both values were valid \(K_{IC}\).

The plots of crack size factor versus yield strength are presented only in terms of \((K_0/\sigma_y)^2\) and individual \((K_{IC}/\sigma_y)^2\) data are not identified. The results for base heats 2 and 20 were not averaged. Furthermore, they appear as individual data points only in Figure 10 which shows the effect of Cr. In all other graphs of this type the data points for the base composition were omitted for clarity and were replaced by the trend line for the base composition (0.88%C) in Figure 10.

The following sections discuss the contribution of individual elements to fracture toughness.

**Carbon**

Carbon reduced toughness at all tempering temperatures as shown in Figure 3. In room temperature tests toughness was highest after tempering at 800°F. At the 0.43 and 0.37%C levels toughness dropped in an essentially continuous manner with decreasing tempering temperature, but

* Only the plane strain crack size factor \((K_{IC}/\sigma_y)^2\) should be used in design.
Figure 3. Effect of carbon on fracture toughness at two test temperatures.
with 0.29% C a slight trough in toughness was observed at room temperature. At -100°F a pronounced trough in toughness occurred at all three carbon levels in the tempering range of 500 to 600°F. This behavior is an indication of tempered martensite embrittlement and confirms the work of Kula and Ancill on 4340 steel, which demonstrated that $K_{IC}$ tests will reveal this form of embrittlement if testing is conducted at low temperatures (7). Room temperature $K_{IC}$ tests are known to be insensitive to this embrittlement.

There is a slight suggestion that at -100°F the embrittlement trough was shifted to lower tempering temperatures with increasing carbon content. The actual decrease in toughness caused by carbon was not concentrated in the embrittling range, but rather carbon produced a general reduction in toughness at all tempering temperatures.

Figure 4 shows the effect of carbon in terms of $(K_{IC}/\sigma_f)^2$ versus yield strength. Carbon exerted a strong strengthening effect and at equivalent strength levels it reduced toughness. This damaging effect was accentuated at -100°F. At the lower testing temperature an excellent strength-toughness balance was obtained with the 0.29% C steel after tempering at 400°F, as denoted in Figure 4. For most of the other alloying elements, as well, tempering at 400°F substantially raised the crack size factor, particularly at -100°F. These results are identified separately in the appropriate graphs of crack size factor versus yield strength.

**Manganese**

The effect of Mn on toughness at various tempering temperatures is shown in Figure 5. At room temperature, in the tempering range of 400 to 600°F, toughness was essentially constant and only slightly affected by Mn content, but at higher tempering temperatures toughness increased at all Mn levels. In this latter region, Mn reduced toughness slightly with the maximum variation in toughness at a given tempering temperature being approximately 7 ksi/√ft.

At -100°F the deleterious effect of Mn at the higher tempering temperatures was increased. A trough in toughness was observed at each Mn level with the lowest values occurring for the 1.52% Mn steel. At tempering temperatures of 400 and 500°F, Mn affected toughness only slightly.

In terms of crack size factor, Mn reduced toughness at a given yield strength, as shown in Figure 6. Furthermore, a high Mn content decreased the yield strength. Optimum toughness-strength properties were obtained with 0.21% Mn, particularly after tempering at 400°F.
Figure 4. Influence of carbon on crack size factor versus yield strength at two test temperatures. Results at -100°F for the 0.29% C steel tempered at 400°F are identified separately.
Figure 5. Effect of manganese on fracture toughness at two test temperatures.
Figure 6. Influence of manganese on crack size factor versus yield strength at two test temperatures. The -100°F data for the 0.21% and 1.52% Mn levels tempered at 400°F are identified separately.
Silicon

The analysis of how Si affects toughness is complicated by its strong effect on tempering kinetics. Figure 7 shows that at -100°F, the embrittlement trough was shifted to higher tempering temperatures with increasing Si content. This shift is associated with a retardation in the onset of cementite formation caused by Si (8,9). The embrittlement trough in the 1.44% Si steel was shifted to approximately 800°F, the region in which toughness was highest for the two lower Si steels. At room temperature and -100°F toughness in this region increased with decreasing Si content. Because Si shifted the embrittling range, an increase in this element to 1.44% was highly beneficial to toughness at low tempering temperatures.

The beneficial effects of a high Si content are further illustrated in Figure 8. Both at room temperature and -100°F, 1.44% Si provided increased strength without sacrificing toughness. The virtual absence of Si was also more desirable than the presence of 0.35%.

Chromium

The highest level of Cr studied, 1.60%, resulted in the lowest toughness at all tempering temperatures, Figure 9. At room temperature, the toughness with 0.47% Cr was consistently higher than with 0.88%, while at -100°F this trend existed only in the tempering range of about 650-800°F. At all other tempering temperatures, -100°F tests showed a slight superiority in terms of toughness for 0.88% Cr followed by 0.47% Cr and 1.60% Cr.

However, when the results were examined on a strength basis (Figure 10) the lowest Cr content appeared to be preferred both from the standpoint of toughness and strength at room temperature and -100°F. The overall range in yield strength obtained with 0.47 and 0.88% Cr was similar. A Cr content of 1.60% resulted in a drop in peak yield strength as well as a general loss in toughness.

Nickel

Figure 11 shows the effect of Ni at various tempering temperatures. In room temperature tests Ni had little effect up to a tempering temperature of about 650°F, and toughness was essentially independent of tempering temperature. At higher temperatures Ni additions produced a drop in toughness. For -100°F tests Ni generally raised toughness. In the tempered martensite embrittlement range the addition of 6.12% Ni eliminated the trough present with 1.15 and 3.07% Ni.
Figure 7. Effect of silicon on fracture toughness at two test temperatures.
Figure 8. Influence of silicon on crack size factor versus yield strength at two test temperatures. The -100°F data for the 1.44% Si steel tempered at 400°F are identified separately.
Figure 9. Effect of chromium on fracture toughness at two test temperatures.
Figure 10. Influence of chromium on crack size factor versus yield strength at two test temperatures.
Figure 11. Effect of nickel on fracture toughness at two test temperatures.
The large beneficial effect of this element on toughness at low temperature was obtained at a sacrifice in yield strength, as shown in Figure 12. In fact both at room temperature and -100°F the 6.12% Ni steel gave the lowest yield strength values. At room temperature, optimum yield strength and toughness were obtained with 1.15% Ni followed by 3.07 and 6.12% Ni. At -100°F, 3.07% Ni provided the best crack size factor yield strength balance. The decrease in yield strength at high Ni levels was not reflected in the tensile strength which was essentially unchanged by variations in this element. For applications in which tensile strength rather than yield strength is the primary design parameter a plot of \((K_Q/\sigma_y)^2\) versus tensile strength would be more applicable and on that basis an increase in nickel content is desirable both at room temperature and -100°F, as shown in Figure 13.

**Cobalt**

Additions of 2.11 and 4.14% Co to the base cobalt-free steel caused a consistent decrease in toughness at both test temperatures, Figure 14. Cobalt also raised the yield strength, and the combined effect in terms of crack size factor versus yield strength appears in Figure 15. At room temperature the results for 2.11% Co coincide closely with the curve for 0.00% Co, although strength was increased, while 4.14% Co produced a further strengthening with a depression of the \((K_Q/\sigma_y)^2\) versus yield strength curve. At -100°F, Co was an undesirable addition at yield strengths up to 210 ksi. Above this strength, data for the cobalt-free composition were not available for comparison.

**Molybdenum**

Curves of fracture toughness versus tempering temperature for Mo alloying appear in Figure 16. Both at room temperature and -100°F, lowest toughness was obtained with the highest Mo content of 1.20%. At room temperature toughness consistently increased with decreasing Mo content, although this effect, particularly in the range .13 to .32% Mo was very slight. At -100°F an appreciable difference in toughness between 0.13 and 0.32% Mo did not exist, except possibly for a 700°F temper.

In terms of crack size factor versus yield strength (Figure 17), 0.13 and 0.32% Mo resulted in virtually identical properties at room temperature but 1.20% Mo degraded toughness slightly.

At -100°F the interpretation of the results is complicated by the exceptionally high toughness for the 0.13 and 1.20% Mo steels tempered at 400°F. In general at this testing temperature, 0.32% Mo gave the best properties while 1.20% Mo was undesirable from an overall strength-toughness standpoint.
Figure 12. Influence of nickel on crack size factor versus yield strength at two test temperatures.
Figure 13. Influence of nickel on crack size factor versus tensile strength at two test temperatures.
Figure 14. Effect of cobalt on fracture toughness at two test temperatures.
Figure 15. Influence of cobalt on crack size factor versus yield strength at two test temperatures. The -100°F test data for the 2.11% and 4.14% cobalt steels tempered at 400°F are separately identified.
Figure 16. Effect of molybdenum on fracture toughness at two test temperatures.
Figure 17. Influence of molybdenum on crack size factor versus yield strength at two test temperatures. The -100°F results for the 0.13% and 1.20% molybdenum steels tempered at 400°F are identified separately.
Vanadium

Previous work on the effects of alloying elements on the low temperature fracture toughness of .35% C, 3% Ni-Cr-Mo-V steels of the type used for the present study had indicated that at a tempering temperature of 800°F, toughness for three levels of vanadium decreased in the order 0.28%, <0.01% and 0.10% V(4). The same discontinuous effect of V was observed in this study, as shown in Figure 18. At -100°F, in the tempering range of about 630-800°F, the 0.29% V steel exhibited the best toughness, followed by <0.01% V and 0.13% V. In the tempered martensite embrittlement region 0.29% V was distinctly superior over the other levels, which possessed very similar properties. For all steels, toughness rose sharply when the tempering temperature was reduced to 400°F.

At room temperature in the tempering range of 700-800°F, toughness also decreased in the order 0.29%V, <0.01%, and 0.13% V, although the differences were slight. However, at lower tempering temperature the vanadium-free steel provided the best toughness, followed by 0.29% and 0.13% V.

The effect of V on crack size factor-yield strength curves is presented in Figure 19. The 0.13% V steel exhibited the highest yield strength. At room temperature it usually resulted in the lowest toughness, although at yield strengths below 185 ksi the <0.01% V steel was slightly inferior. The largest crack size factor values at yield strength above 200 ksi were observed for both the <0.01% V and 0.29% V steels tempered at 400°F. At -100°F 0.29% V generally provided the best toughness-strength balance.

Aluminum

Aluminum variations between 0.048% and 0.30% did not markedly affect 75°F toughness in the tempering range of 650-800°F, as shown in Figure 20. Below about 600°F toughness was highest with 0.30% Al followed by 0.18% and 0.048%. In tests at -100°F, the toughness of all three steels was similar after tempering at 400°F or 500°F. In the embrittlement region the 0.18% Al steel was toughest followed by 0.48% and 0.30% Al. Tempering at 800°F changed this order with toughness decreasing as aluminum content increased.

In terms of crack size factor the effect of Al, as shown in Figure 21, was slight at room temperature, although at yield strengths above 195 ksi additions of 0.18% and 0.30% resulted in higher toughness at the same strength level than exhibited by the base composition having 0.048% Al.

The superior strength-toughness balance offered by tempering at 400°F is again illustrated by the -100°F test results for alloying with 0.18% and 0.30% Al. However, at all other tempering temperatures, a high Al content lowered the crack size factor.
Figure 18. Effect of vanadium on fracture toughness at two test temperatures.
Figure 19. Influence of vanadium on crack size factor versus yield strength at two test temperatures. Results for the <0.01% and 0.29% V steels tempered at 400°F are identified separately.
Figure 20. Effect of aluminum on fracture toughness at two test temperatures.
Figure 21. Influence of aluminum on crack size factor versus yield strength at two test temperatures. The -100°F results for the 0.18% and 0.30% Al steels are identified separately.
B. COMBINED EFFECTS OF CHROMIUM AND MOLYBDENUM

A group of nine compositions were studied in which Cr and Mo were varied at three levels in all possible combinations. The nominal or average values of each element in this series were as follows:

Cr: .49, .88, 1.52%
Mo: .15, .32, 1.15%

The actual chemical analyses of these steels (Heats 2 and 20, 3, 4, 5, 6, 23, 24, 25 and 26) appear in Table I.

Figure 22 shows the effect of tempering temperature on fracture toughness of all nine compositions. The curves for the base composition (0.88Cr, 0.32Mo) are drawn through the average results of heats 2 and 20. Individual data points are omitted in this figure, but the curves were drawn to pass through the actual data points at 100°F intervals. Specific test results which distinguish between \( K_0 \) only and \( K_{IC} \) may be obtained from Tables All and AIV. At room temperature the overall variation in toughness for all steels was less than 20 ksi/\( \sqrt{\text{in.}} \). With the exception of steel heat 25, (0.51%Cr, 0.15%Mo), a pronounced embrittlement trough was not present at room temperature and the general trend was for toughness to decrease with decreasing tempering temperature.

At -100°F all steels exhibited a substantial drop in toughness on tempering at about 500-600°F. The overall variation in toughness was greater at -100°F than room temperature, being about 30 ksi/\( \sqrt{\text{in.}} \) for a 400°F temper, 20 ksi/\( \sqrt{\text{in.}} \) in the embrittlement region, and approximately 50 ksi/\( \sqrt{\text{in.}} \). after tempering at 800°F.

The toughness interactions of Cr and Mo at each tempering temperature are shown in Figures 23 to 27 as plots of \( K_0 \) versus nominal Cr content at each of the three nominal Mo levels. With the exception of one data point at 500°F, lowest toughness at a given Cr content was obtained at the highest Mo level of 1.15%. Similarly, a Cr content of 1.52%, with only two exceptions resulted in the lowest toughness at a particular Mo level.

The results in Figures 23 to 27 may be summarized by considering the trends in toughness as Cr and Mo were varied at three levels, two test temperatures, and five tempering temperature combinations (3 x 2 x 5 = 30). For Cr alloying at a constant Mo level, raising Cr from 0.49% to 0.88% reduced toughness in 19 out of 30 instances, while in the increments 0.49% to 1.52% Cr and 0.88% to 1.52% Cr these ratios were 28/30 and 29/30.
Figure 22. Variation of fracture toughness with chromium and molybdenum content at two test temperatures.
Figure 23. Effect of chromium and molybdenum on fracture toughness (tempered at 400°F).
Figure 24. Effect of chromium and molybdenum on fracture toughness (tempered at 500°F).
Figure 25. Effect of chromium and molybdenum on fracture toughness (tempered at 600°F).
Figure 26. Effect of chromium and molybdenum on fracture toughness (tempered at 700°F).
Figure 27. Effect of chromium and molybdenum on fracture toughness (tempered at 800°F).
For Mo alloying at constant Cr, an increase from 0.15% to 0.32% Mo reduced toughness in 16 out of a possible 30 times and for the increment 0.32% to 1.15% Mo and 0.15% to 1.15% Mo these values were 23/30 and 30/30 respectively. This comparison indicated that large quantities of Cr and Mo (1.52% and 1.15% respectively) were definitely undesirable from solely a toughness standpoint. However, the effects of Cr between 0.49% and 0.88% and Mo in the range 0.15% to 0.32% were not consistent, although there was a slight tendency for toughness to be reduced with increasing amounts of either. More specific information on the effects of these elements at each tempering temperature may be obtained by reference to Figures 23 to 27. The fact that the highest levels of Cr and Mo yielded the lowest toughness, while the results for the two other levels were not consistent was shown earlier when only the single element variations of these elements were described (see Figures 9 and 16).

Because the effect of these two elements on toughness was similar, an attempt was made to correlate $K_Q$ with the weight % sum of Cr and Mo. Figures 28 to 32 illustrate the variations of $K_Q$ with % (Cr+Mo) at 75°F and -100°F together with the effects of these elements on yield strength. At each tempering temperature and test temperature, the overall tendency for $K_Q$ was to decrease with increasing % (Cr+Mo).

The effect of Cr and Mo on the yield strength for the 400°F, 500°F, and 800°F tempering temperatures (Figures 28, 29 and 32) was divided into two regions. For Cr+Mo levels involving only 0.15% or 0.32% Mo, strength was either essentially constant, Figure 32, or decreased slightly with increasing % (Cr+Mo), Figures 28 and 29. However, in the presence of 1.15% Mo the yield strength was substantially increased. The resulting curves based on three levels of Cr exhibit a drop in yield strength with Cr+Mo content, which is actually an increase in Cr at a constant Mo level. For the 600°F and 700°F tempering treatments, a similar trend existed to a lesser degree, and a single line was drawn through all the strength versus % (Cr+Mo) data as shown in Figures 30 and 31.

A more extensive presentation of how Cr and Mo interact to affect strength is presented in Figures 33 to 37 as plots of yield strength as a function of %Cr at each of three nominal Mo levels. The results may be summarized by again considering the incidence of certain trends for all combinations of three compositions, two test temperatures, and five tempering temperatures. At a constant Mo level, in the interval 0.49% to 0.88%, Cr decreased the yield strength in 28 out of 30 possible cases; for 0.88% to 1.52% Cr and 0.47% to 1.52% Cr the corresponding ratios were 21/30 and 28/30.
Figure 28. Variation of fracture toughness and yield strength at two test temperatures with combined chromium and molybdenum content (tempered at 400°F).
Figure 29. Variation of fracture toughness and yield strength at two test temperatures with combined chromium and molybdenum content (tempered at 500°F).
Figure 30. Variation of fracture toughness and yield strength at two test temperatures with combined chromium and molybdenum content (tempered at 600°F).
Figure 31. Variation of fracture toughness and yield strength at two test temperatures with combined chromium and molybdenum content (tempered at 700°F).
Figure 32. Variation of fracture toughness and yield strength at two test temperatures with combined chromium and molybdenum content (tempered at 800°F).
Figure 33. Effect of chromium and molybdenum on yield strength (tempered at 400°F).
Figure 34. Effect of chromium and molybdenum on yield strength (tempered at 500°F).
Figure 35. Effect of chromium and molybdenum on yield strength (tempered at 600°F).
Figure 36. Effect of chromium and molybdenum on yield strength (tempered at 700°F).
Figure 37. Effect of chromium and molybdenum on yield strength (tempered at 800°F).
However, Mo exerted the opposite effect. In the interval 0.15% to 0.32%, it raised the yield strength in 19 of 30 cases, and for 0.32% to 1.15% and 0.15% to 1.15%, an increase in Mo always produced a strength rise at a constant level of Cr.

The behavior of Cr and Mo may be generalized in the following fashion. Both of these elements displayed a tendency to decrease toughness per se, particularly when present in the largest quantities studied of about 1.15% Mo and 1.52% Cr. In the case of Mo, this decrease in toughness accompanied an increase in strength, but Cr additions had a tendency to reduce both toughness and yield strength. This suggests that an optimum balance of Cr and Mo requires using the lowest possible Cr content with Mo maintained at the lowest level consistent with strength requirements. In practice, additional considerations such as hardenability would also be involved.

These conclusions are illustrated in Figure 38 which summarizes the results for all Cr-Mo series steels in terms of \((K_{lc}/\sigma_s)^2\) versus yield strength. The data for the base composition (0.88% Cr, 0.32% Mo) are shown as average trend lines of \((K_{lc}/\sigma_s)^2\) curves from Figure 10. The highest yield strength values were achieved with a Mo content of 1.11% and low Cr (0.50%). At strength levels near the maximum, \((K_{lc}/\sigma_s)^2\) was usually highest for the 0.51% Cr, 0.16% Mo steel. A combination of low Cr (-0.50%) with either of the two lower Mo levels (0.15% or 0.32%) usually provided the best toughness, but the highest yield strength was achieved by the use of high Mo (-1.15%). The highest level of Cr (-1.5%), regardless of Mo content, significantly lowered toughness at all strength levels.

C. GENERAL CONSIDERATIONS

The preceding discussion of alloying effects on toughness of .35C, 3Ni-Cr-Ni-Mo-V steels has demonstrated that tempered martensite embrittlement can be observed in low temperature \(K_{lc}\) tests, an observation that is in accord with the prior work of Kula and Ancill on 4340 steel (7). At 75°F an embrittlement trough was generally not present after tempering at 400-800°F, but for -100°F tests a pronounced trough in toughness in the tempering range of 500-600°F existed for nearly all steels.

The most common method of revealing embrittlement involves room temperature impact tests conducted as a function of tempering temperature. In the tempered martensite embrittlement region, impact energy exhibits a trough which is also reflected as an increase in transition temperature (7,10).
Figure 38. Influence of chromium and molybdenum on plane strain crack size factor versus yield strength at two test temperatures.
There are a number of possible reasons why $K_{IC}$ tests at low temperatures are more sensitive in revealing embrittlement than room temperature tests. Phenomenologically, the embrittlement is probably a manifestation of transition-like behavior in $K_{IC}$ versus temperature curves.

The accentuated lowering of -100°F toughness test after tempering in the embrittlement region could result from a change in microscopic fracture mode or bulk properties. In the case of impact tests, embrittlement has been associated with an increasing amount of failure along prior austenite grain boundaries (11). Embrittlement is only observed in steels containing certain impurities such as P, As, Sb, Sn, N or Si (12) and occurs at a tempering temperature corresponding to the start of the third stage of tempering during which cementite forms. A number of theories have been proposed which consider embrittling interactions of segregated impurities with cementite, preferentially at grain boundaries (7, 13). It has also been suggested that embrittlement may arise because of changes in matrix properties unrelated to carbide films (14).

Recent work by Ronald (15) supports the notion that changes in bulk properties are primarily responsible for embrittlement. He suggested that the increased amount of intergranular fracture under plane strain conditions in embrittled material actually arises because plastic flow within the grains is hindered by whatever structural changes are related to embrittlement.

The present study does not, however, permit a critical analysis of these and other proposed embrittlement theories. The results do suggest that variations in the degree of embrittlement are not related in a simple fashion to changes in flow properties. An example of this behavior is found in the case of Cr and Mo. High levels of both elements lowered toughness, in the case of Mo at an increase in yield strength, while for Cr alloying the embrittlement accompanied a strength drop.

A number of investigators have examined the role of alloying elements on tempered martensite embrittlement, but the results with respect to major alloying elements are somewhat contradictory. Schrader et al, (16) investigated the effects of Mn, Ni, Cr, Mo and Al on impact properties at room temperature. Mo, V and W had no effect while Cr and Mn promoted embrittlement. Steels containing 1.5% and 5% Ni did not exhibit a trough in room temperature energy. Increasing Al from 0.04% to over 0.1% completely eliminated the embrittlement trough. However, Riedrich (17) found that, although Al could eliminate embrittlement at room temperature, the impact trough was present in low temperature tests.
In contrast with Schrader's results on Ni, Payson (18) observed embrittlement in 2.6% to 5% Ni steels. Steels with 4.0% Ni and 0.13% C and 2.6% Ni and 0.25% C exhibited an impact trough at room temperature. A 5% Ni steel containing only 0.06% C was not embrittled at room temperature, but the trough was present in -100°F tests.

Capus (12) found that tempered martensite embrittlement requires the presence of certain impurities. For example, in 1.5% Ni-Cr-Mo steels, N, P, As, Sb, Sn, Si, and Mn promoted embrittlement. Mn lowered toughness at all tempering temperatures above 350°C, and it was suggested that the embrittling mechanism for Mn is probably different from that of the other elements, (12). Very pure steels in which the levels of the other impurities were considerably lower than in commercial steels were not embrittled.

The critical role that minor impurity elements exert on tempered martensite embrittlement is analogous to their effect on reversible temper embrittlement which is observed on prolonged heating at or slow cooling through approximately 850-1000°F. Balajiva et al (19) showed that a high purity 0.3C, 3Ni-.75Cr steel was not embrittled. Subsequently, Steven and Balajiva (20) determined that the impurities P, As, Sb, Sn, Si and Mn could all induce embrittlement in the same steel. In the case of temper brittleness Low, et al (21) have demonstrated that interactions exist between impurities and alloying elements. For example, a plain carbon steel containing 0.08% Sb was not embrittled while additions of Cr and Ni induced embrittlement, particularly when both elements were present.

It is likely that interactions between impurities and alloying elements also exist in tempered martensite embrittlement, but there is little in the present work to suggest that it can be eliminated by regulating the major alloying elements. Si in large amounts merely shifted the embrittlement range to higher tempering temperatures, thereby permitting tempering at 500°F. A high V content of 0.29% and increasing amounts of Ni raised toughness in the embrittlement region, and the 6.12% Ni steel did not exhibit a trough at -100°F. This is consistent with nickel's well known toughening effect at low temperatures. Thus, the role of alloying elements appears to be secondary to that of impurities indigenous to commercial purity steels, although toughness at a particular test temperature can be affected by alloying, probably through a shift in transition temperature.

The question of whether a certain alloying element raises or lowers toughness should also be considered in terms of the effect of a particular compositional change on strength as well as toughness. Accordingly, the
previous sections have provided an analysis of alloying effects in terms of crack size factor versus yield strength. A summary of valid plane strain fracture toughness \( (K_{lc}) \) versus yield strength and tensile strength for all steels appears in Figures 39 and 40 and provides a convenient basis for comparing the results of this study with existing data on fracture toughness of steels. The choice of yield strength or tensile strength depends on which strength parameter is more important in a particular engineering application. In contrast with previous graphs the toughness both at room temperature and \(-100^\circ F\) is plotted versus room temperature yield or tensile strength.

The overall results for all steels for which valid \( K_{lc} \) data were obtained show that at \( 75^\circ F \) \( K_{lc} \) was not strongly affected by composition. The variation in toughness at \( 75^\circ F \) was generally less than \( \pm 15\% \) from an average trend line. In terms of tempering temperature it appears that \( 400^\circ F \) is generally optimum with respect to fracture toughness.

At \(-100^\circ F\) alloying elements exerted a more substantial effect on toughness. No general trend existed in these results, and at a constant strength level the variation in toughness was significantly higher than at \( 75^\circ F \). Appreciable reductions in toughness from the highest values are possible with the compositional variations used in the current work, and fracture toughness testing at \( 75^\circ F \) is clearly inadequate for sensitively discriminating between steels which will encounter subzero service temperatures.

Some of the \( K_{lc} \) values in Figures 39 and 40 compare favorably with the properties of such widely utilized steels as 4340, D6AC and 300M (22). The tougher steels evaluated are identified by the elements which were varied from the base composition. Particularly interesting from a strength-toughness standpoint was the 1.44\% Si steel which is essentially a lower carbon, higher Ni version of 300M. The compositions of these steels are compared below:

<table>
<thead>
<tr>
<th>Composition, Wt. %</th>
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<tbody>
<tr>
<td>Heat 12 C Mn Si Cr Ni Mo V</td>
</tr>
<tr>
<td>.36 .61 1.44 .83 3.05 .27 .09</td>
</tr>
<tr>
<td>300M C Mn Si Cr Ni Mo V</td>
</tr>
<tr>
<td>.41/.60/ 1.45/.70/ 1.65/.30/ .05/</td>
</tr>
<tr>
<td>.46/.90/ 1.80/.95 2.00/.50/ .10/</td>
</tr>
</tbody>
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55
Figure 39. Variation of plane strain fracture toughness with room temperature yield strength for all steels.
Figure 40. Variation of plane strain fracture toughness with room temperature tensile strength for all steels.
A study was performed on the influence of various elements on the notch bend fracture toughness at 75°F and -100°F of .35%C, 3Ni-Cr-Mo-V martensitic steels tempered between 400 and 800°F. The elements examined included C, Mn, Si, Cr, Ni, Mo, Co, V and Al. The overall variation in room temperature yield and tensile strengths for twenty-four steels was 155-230 ksi yield strength and 188-288 ksi tensile strength.

The results indicate a relatively slight dependence of fracture toughness on composition at 75°F, but a large overall composition effect at -100°F. At 75°F, toughness, in the tempering range of 400-600°F, was usually constant, but increased at higher tempering temperatures. At -100°F, for all except a 6.12% Ni steel, toughness decreased on tempering above 400°F, then increased above about 600°F. The resulting trough in fracture toughness indicated tempered martensite embrittlement. Alloying elements strongly influenced strength as well as toughness, and the results were also analyzed on the basis of crack size factor versus yield strength. The elements C, Mn, Cr, Mo, and Co reduced toughness per se, both at 75°F and -100°F. High levels of these elements were also undesirable from a toughness versus yield strength standpoint. Interactions of Cr and Mo were also studied and although their general affect was to reduce toughness, under certain conditions where high strength is needed, a high Mo content (1.15%) can be used in conjunction with low Cr (.50%).

The influence of the other elements was appreciably more complex. On the basis of maximum toughness at a constant yield strength, steels containing .14% Si or .29% V exhibited excellent properties. Al in levels of 0.18% or 0.30% offered no advantage over the base level of 0.048%. Nickel was evaluated at 1.15, 3.07, and 6.12. At -100°F it improved toughness in the embrittlement range, but on the basis of yield strength was undesirable in large amounts. However, it was shown that Ni may be an attractive addition for applications utilizing tensile rather than yield strength in design.

In addition to providing information on the effects of systematic alloying variations, this study demonstrated that compositional effects at low temperatures are substantially larger than at room temperature and consideration must be given to this behavior in selecting steels that will encounter low service temperatures.
LIST OF REFERENCES


17. G. Riedrich, Discussion to reference 16, p. 27.


VI APPENDIX
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### Table A1 (Continued)

**SUMMARY OF SMOOTH TENSILE PROPERTIES AT 75°F**

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## TABLE A11

### SUMMARY OF SMOOTH TENSILE PROPERTIES AT -100°F

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### TABLE A111

**SUMMARY OF FRACTURE TOUGHNESS RESULTS AT 75°F**

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* Tentative plane strain fracture toughness, ksi/μin
** Plane strain fracture toughness ($K_Q=K_{IC}$), ksi/μin
*** Crack size factor, μin.
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**SUMMARY OF FRACTURE TOUGHNESS RESULTS AT 75°F**

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* Tentative plane strain fracture toughness, ksi/in.
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*** Crack size factor, in.
### SUMMARY OF FRACTURE TOUGHNESS RESULTS AT -100°F

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**Table AIV (Continued)**
TABLE AIV (Continued)

SUMMARY OF FRACTURE TOUGHNESS RESULTS AT -100°F

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### TABLE A1V (Continued)

**SUMMARY OF FRACTURE TOUGHNESS RESULTS AT -100°F**

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Figure 1A. Dependence of work hardening exponent on tensile to yield strength ratio. Results for all steels tested at 75°F.
Figure 2A. Dependence of work hardening exponent on tensile to yield strength ratio. Results from all steels tested at -100°F.
ABSTRACT

A study was performed on the influence of various elements on the notch bend fracture toughness at 75°F and -100°F of 0.35%C, 3Ni-Cr-Mo-V martensitic steels tempered between 400 and 800°F. The elements examined included C, Mn, Si, Cr, Ni, Mo, Co, V and Al. The overall variation in room temperature yield and tensile strengths for twenty-four steels was 155-230 ksi yield strength and 188-288 ksi tensile strength. Tempered martensite embrittlement was revealed by testing at -100°F, whereas 75°F tests were insensitive to this phenomenon. The elements C, Mn, Cr, Mo, and Co generally reduced toughness at both test temperatures and, particularly in large quantities, were undesirable on a toughness-yield strength basis. The influence of Si, Ni, V, and Al was more complex. A steel containing 0.29% V exhibited excellent properties while Al in amounts of 0.18% and 0.30% offered no advantage over a level of approximately 0.05%. Increasing amounts of Ni in the range of 1.1% to 6.12% were highly beneficial to low temperature toughness at a sacrifice in yield strength. This element provided an improved toughness-strength balance on the basis of tensile strength but not yield strength. Particularly attractive properties were obtained with a steel which, except for a slightly lower C content (0.3%) and a higher level of Ni (3.0%), resembled the commercial alloy, 300M. This study indicated a relatively slight dependence of fracture toughness on composition at 75°F, but a large overall variation in toughness at -100°F. Consideration should be given to this behavior in selecting steels for applications involving low service temperatures.
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