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MECHANICAL PROPERTIES OF AN ULTRA HIGH STRENGTH STEEL

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MECHANICAL PROPERTIES OF AN ULTRA HIGH STRENGTH STEEL

by A. E.G. Schempp and W.A. Morgan

ABSTRACT

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Five percent chromium hot work tool steels have found application in the field of high strength steel design. A modification of Atlas Crovan was melted as an experimental 10-ton basic electric arc heat by Atlas Steels Limited, Welland, Ontario. The new material, designated as M-7-12, was tested at the Physical Metallurgy Division, Mines Branch, to determine its mechanical properties. especially with regard to transverse ductility and response to various heat treatments. It was found that this steel exhibits excellent tensile strength consistent with good tensile ductility and good impact strength over a wide range of temperature. This alloy possesses an ultimate tensile strength of 275,000 psi at room temperature, and of 190,000 psi at 1000°F. The impact strength at room temperature is 25 ft-lb, and at minus 100°F, 15 ft-lb. These properties make the alloy one of the most outstanding of new high strength materials.

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INTRODUCTION

Steel from the 10-ton basic electric arc heat No. B1140, M-7-12, was submitted to the Physical Metallurgy Division by Atlas Steels Limited, Welland, Ontario. The material was supplied in the form of rolled and annealed 1-1/8-in. round bar stock. Three discs cut from a 6-in. square forged billet were also received. Table 1 gives the chemical composition of the ladle sample analyzed by Atlas Steels. The table also gives the specified range of alloy contents of Crovan, compared with the modified experimental heat M-7-12 which contains more carbon and less vanadium.

TABLE 1

Material	С	Mn	Si	Cr	Мо	v	P	S
Crovan, Range	•30 •to •36	.20 to .50	.80 to 1.10	4.75 to 5.25	1.30 to 1.50	•70 to 1.00	<. 030	<. 030
M-7-12, Range	•37 to •43	.20 to .40	.80 to 1.00	4.75 to 5.25	1:20 to 1:40	•40 to •60	<. 030	<. 030
M-7-12, Heat B1140	•41	•38	1.00	4.92	1.35	•48	.012	.010

Chemical Analyses of Crovan and M-7-12, %

It was known that Crovan would not develop sufficient hardness to attain an ultimate tensile strength of about 280,000 psi. The increase in carbon was therefore planned to secure higher hardenability. On the other hand, the vanadium content was decreased because it is known that amounts of about one percent are more than sufficient to ensure high hardness. A hardening temperature of 1850°F has invariably been recommended for this type of steel, mainly for practical rather than metallurgical reasons. It is obvious that most of the vanadium carbide present in the material in the annealed condition will not go into solution at 1850°F and therefore could not contribute to the actual hardening. In the application of this alloy as a tool steel, the undissolved carbides of vanadium, present after hardening, contribute principally to wear resistance; however, this is not considered a necessary property for high strength structural applications. Another aspect, of no less importance, is the cost of the steel. It has to be competitive to the extent that improved properties must compensate for increased cost.

The literature on high strength steels has been so extensive in recent years that only a few important references will be mentioned here in order to provide a general survey of the subject. The first steels (1, 2, 3) used for high strength design were of the AISI 4340 type, modified later by additions of silicon and carbon to allow higher tempering temperatures and to obtain higher initial quenched hardnesses. The absence of secondary hardening and the necessity for oil quenching, however, are disadvantages unchanged by these modifications. Five percent chromium hot work steels have lacked sufficient strength to compete with the properties of the lower alloy AISI 4340 type steels. With slight modifications it was possible to improve the properties of the hot work steels to values at least equal to, and, at temperatures of about LOCO°F, better than the lower alloy type steels. Meferences 4 to 6 are publications on hot work steels and deal with their development and testing.

1, References are at the end of the report

EXPERIMENTAL PROCEDURE AND OBSERVATIONS

(1) Transformation Temperatures and Thermal Expansion

Samples machined to a diameter of 5/16 in. and a length of 2.560 in. were used in the annealed condition to establish the transformation temperatures and the coefficient of thermal expansion. The dilatometer apparatus includes a furnace with programme control, an inert gas atmosphere, and an electromechanical magnification device which records length changes at 500:1 magnification against a calibrated temperature scale. Table 2 lists the transformation temperatures at a constant heating rate of 270° F/hr and two cooling rates, 270° F/hr and 72° F/hr. The transformation during cooling takes place at higher temperatures if the rate of cooling is decreased.

TABLE 2

Transformation Points	270°F/hr heating and cooling rate °F	72°F/hr cooling rate
A _{c1}	1562;1565	~
A _{c3}	1616;1625	-
Ar3	1382	1440
^A r1	1292	1410

Transformation Temperatures of M-7-12

From the recorded temperature-expansion curves, the mean coefficient of thermal, linear expansion has been calculated for a number of temperature intervals. The results are listed in Table 3.

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inermal, Linear Expansio	n or M-7-12, Anneared
Temperature Interval, °F	Coefficient, $\frac{\text{in.}}{\text{in. x °F}} \times 10^{-6}$
80 – 200	6.06
80 - 400	6.21
80 ~ 800	6.79
80 - 1200	7.12
500 - 1200	7.56
800 - 1200	7.71

(2) <u>Hardenability</u>

Jominy hardenability tests were carried out on M-7-12, even though it is an air hardening steel, in order to show the influence of a higher hardening temperature as well as the small difference in hardness between the water quenched end and the air cooled portion of the samples. Figure 1 demonstrates that for both hardening temperatures the Jominy bar hardness gradient amounts to one to two points Rockwell "C" over a distance of 3 in.

A series of $\frac{3}{4}$ -in. round samples were hardened by air cooling from different hardening temperatures. Figure 2 shows how the hardness of the untempered pieces increases rapidly with increased hardening temperatures. The time at hardening temperature, 10 minutes, was kept constant. The samples were fractured and compared with a set of standard samples showing Shepherd grain size numbers. In Figure 2, the Shepherd fracture grain size curve is included. It can be seen from the hardness and grain size curves that the hardening





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temperature of 1850°F, usually used for Grovan, is not sufficient to obtain maximum solution of alloy carbides for highest hardness after transformation. A hardening temperature of 1950°F will produce material two to three points Rockwell "C" higher with little effect on the grain size. This additional hardness results in an increase of approximately 20,000 psi tensile strength. It may be added that considerable grain coarsening occurs above 1975°F, and that scaling becomes a problem only above 2050°F if the time at hardening temperature does not exceed 15 minutes and if a graphite muffle is used in the furnace to reduce oxidation.

(3) Tempering Characteristics

The most important property of five percent chromium hot work steels is the secondary hardening effect. The peak of hardness is known to be practically at the same tempering temperature of about 950°F (provided that the tempering time is kept constant at 2 hours) for a wide range of modifications in alloying elements. Carbon and molybdenum are known to increase the hardness at the peak. However, the rate of softening with increasing tempering temperatures seems, again, to be fairly constant with all modifications of the basic five percent chromium steel examined.

Tempering at temperatures above the peak hardness has two main advantages: (a) the steel may be used in service at temperatures up to the tempering temperature, and (b) more thorough stress relief is obtained at the higher tempering temperatures. These two aspects are the reason why five percent chromium steels are superior to AISI 4340 steels which not only require oil quenching, causing higher initial stresses, but also soften, despite high silicon contents, rather rapidly at temperatures over 500 to 600°F.



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Figure 3 shows tempering curves for M-7-12 for two hardening temperatures, 1850 and 1975°F. The steel was tempered for two hours at all temperatures (tempering was interrupted by air cooling after one hour). A double tempering cycle is normally recommended for this type of steel to ensure almost complete transformation of retained austenite. The first cycle will transform retained austenite to bainite, and the second cycle will temper this new transformation product, thereby yielding a structure as uniform and fully relieved from stresses as possible. In Crovan as well as M-7-12, retained austenite could not be observed microscopically. X-ray diffraction patterns also showed no evidence of more than four percent retained austenite after samples had been air cooled. However, double tempering is still a recommended technique to ensure optimum mechanical properties.

TABLE 4

Tempering Temperature °F	400	600	800	900	925	950	975	1000	1050	1100	1200
Hardness, R _C , after harden- ing at 1850°F, (a)	57호	56 <u>국</u>	57寺	58	58 <u>1</u>	58 <u>1</u>	57호	56 1	53	51	39쿨
Hardness, R _c , after harden- ing at 1975°F, (b)	59	58社	59	60	60 <u>1</u>	60쿭	59 3	· 58君	55쿺	53	42 <u>3</u>

Hardness of M-7-12, (Double Tempered^H)

A held 1 hr at temperature, air cooled, and held 1 hr at temperature
(a) 10 min at temperature, air cooled, untempered hardness R_c 59¹/₂
(b) 10 min at temperature, air cooled, untempered hardness R^c 62¹/₂

The higher hardening temperature of 1975°F results in a tempering curve practically parallel to but higher than the curve obtained after hardening at 1850°F. From the separation of the two tempering curves in the region above the peak hardness it may be seen that M-7-12 can be tempered 50°F higher when hardened from 1975°F instead of 1850°F, and the resulting hardness or the tensile strength will be the same. This increased tempering temperature might be useful in certain applications. However, as will be pointed out in the following chapter, a loss in tensile ductility occurs after the heat treatment at higher temperatures. Table 4 lists the hardness values obtained from numerous tests on specimens $\frac{3}{4}$ in, diameter and $\frac{1}{2}$ in. thick.

(4) <u>Tensile Properties</u>

Twelve tensile bars were rough machined from the 1-1/8-in. round material for the determination of longitudinal tensile properties at room and elevated temperatures. The bars received the standard hardening heat treatment of holding 10 minutes at 1850°F, air cooling, and double tempering (1 hr + 1 hr) at 1050°F, producing a hardness of R_c 53¹/₄. After this treatment, the bars were carefully ground to size, leaving a very small taper from the gauge points to the centre. (The maximum difference in diameters along the tapered, $1-\frac{3}{4}$ -in. long portion was kept to 0.005 in.) The bar dimensions of 0.375 in. diameter, $1-\frac{1}{2}$ -in. gauge length, and overall length of $5-\frac{1}{2}$ in. were chosen because standard bars with 0.505 in. diameter would have required the full load capacity of the 60,000-1b Universal Baldwin tensile machine used. Another three bars were hardened by air cooling from 1975°F and double tempered (1 hr + 1 hr) at 1100°F, thus producing a hardness of Rc 53¹/₄. Table 5 lists the longitudinal tensile properties obtained, and Figure 4 shows the same data graphically.

TABLE 5

Longitudinal Tensile Properties of M-7-12 at Room and Elevated Temperatures, Heat Treated to Rc 53

Test Temp.	Heat Treatment	U.T.S. kpsi	0.2 % Yield Strength knsi	<u>Yield</u> U.T.S. 100 %	Elong.in 4D=1 ^{1/2} in. %	Reduction in Area %
72 72	I I I	278.0 276.2	223.8 230.3	80.6 83.4	8.7 9.3	32.1 37.1
72	II	281.2	231.2	82.2	6.7	23.5
600	I	244•7	203.2	83.1	10.0	42.6
600	I	248•8	206.0	82.8	10.0	41.7
800 800	I I	232.8 228.7	186.8 195.0	80.2 85.3	12.0 11.7	46.5 46.5
1000	I	192.2	154.6	80.4	12.7	50.2
	I	195.8	155.0	79.2	13.3	51.2
1000	II	194.0	154.9	79.8	8.7	24.8
1000	II	190.3	143.1	75.2	9.3	26.4
1100	I	152.7	112.3	73.6	16.6	58.7
1100		152.6	112.0	73.	15.3	58.1
1200	I	75.6	52.1	68.9	33•3	87.8
1200	I	85.1	55.4	65.1	32•0	87.8

Bars Held 1/2 hr at Test Temperature

* I - 10 min at 1850°F, air cooled, tempered l hr + l hr at 1050°F, R_c 534

II - 10 min at 1975°F, air cooled, tempered l hr + l hr at 1100°F, $R_c 53\frac{1}{4}$



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The higher hardening temperature, although not far in the grain coarsening range, resulted in some loss of tensile ductility at room temperature. At 1000° F, the ultimate tensile strength is the same as after the lower hardening and tempering temperatures; the yield strength is slightly lower, and ductility values are only about the same as obtained with room temperature tests. In comparison to this, the ductility of bars with the standard heat treatment is l_2^1 . times better at 1000° F than at room temperature. As a result of this, it can be stated that M-7-12 has an ultimate tensile strength limited to 280,000 psi. A higher strength at room temperature can be obtained; however, ductility values then would be lower than are generally considered acceptable at present.

In order to produce transverse tensile properties as close to the longitudinal ones as possible, the effect of special homogenizing treatments before the hardening and tempering treatment was studied. Two discs from a 6-in. square, forged billet received different homogenizing treatments before test specimens were machined from them. The treatment given to disc 1 has been recommended for forgings of similar steels by the N.A.C.A. (U.S. National Advisory Committee for Aeronautics) in order to meet properties called for by severe aircraft specifications. The homogenizing treatment is as follows:

- Preheat at 1575°F for 1 hour, heat to
 1975°F, hold 2 hours, air cool.
- (2) Preheat to 1300°F, equalize, heat to1700°F, hold 1 hour, air cool.
- (3) Repeat step (2).
- (4) Heat to 1450°F, hold 1 hour, air cool.

The temperature in step (4) has been changed to 1380°F for M-7-12 to avoid partial transformation on cooling which would produce material difficult to machine. Disc 2 received a treatment that is based on T.T.T. curves of similar alloy steels. It is faster and consists of the following three steps:

- (1) Preheat at 1300°F, equalize, heat to 1975°F, hold 15 minutes, air cool.
- (2) Preheat at 1300°F, equalize, heat to
 1650°F, equalize, transfer into furnace
 at 1380°F, hold 2 hours, air cool.
- (3) Repeat step (2).

This treatment is a double, isothermal annealing operation, and the hardness of disc 2 was R_B 91, compared to R_B 94 of disc 1 after the treatments. Two tensile bars from each disc were machined from a mid-radius location. All four bars were hardened and tempered together at the standard temperatures (heat treatment I in Table 5). The bars were ground to size and tested at room temperature. Table 6 lists the properties and shows that the isothermal annealing operation produced a slightly lower reduction in area. All other values, including the hardness of R_c 53¹/₄, are practically the same.

When comparing the transverse with the longitudinal tensile properties, the only noteworthy difference is in the reduction of area. It will be seen from Tables 5^{A} and 6 that the value of the reduction in area of the transverse test is at a level of 21 percent about 60 percent of the average longitudinal value of 34.6 percent. This can be considered very good for forgings with a reduction of about 5:1, e.g. from a 16-in. round ingot to a 6-in. square billet.

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TABLE 6

Transverse Tensile Properties of M-7-12, Heat Treated To Rc53, after two Homogenizing Treatments Hardened at 1850°F, Air Cooled, and Double Tempered at 1050°F

Test No.	Homogenizing Treatment	U.T.S. kpsi	0.2 % Yield Strength kpsi	Yield U.T.S. 100 %	Elong.in 4D=1½in. %	Reduction in Area %
Disc 1 (1) (2)	N.A.C.A. N.A.C.A.	283.7 283.7	238.7 239.5	84.2 84.4	8.1 7.3	27.1 23.0
Disc 2 (1) (2)	Iso-Anneal Iso-Anneal	280.5 277.0	237.0 233.3	84.5 84.2	7.3 7.3	21.1 21.1

TABLE 7

Longitudinal and Transverse Charpy V-Notch Impact Strength of M-7-12, Heat Treated to Rc 53

(a) Longitudinal Samples

Test Temperature °F	Impact Strength ft-lb
212	25, 28, 27, 26
72	24쿨, 26쿨, 25쿨
-40	21, $22\frac{1}{2}$, 21
-100	15, $18\frac{1}{2}$, 14, $18\frac{1}{2}$, 19

(b) Transverse Samples

Test Temperature °F	Impact Strength ft-lb Disc 2 ^A Disc 1 ^{AA}
72	12, 15, 18 ¹ / ₂ , 16 11, 9, 7
-100	7, 6, 3, 3 4, 4, 3½, 4

A With double isothermal anneal for homogenizing AA With N.A.C.A. homogenizing treatment

(5) Impact Properties

Charpy V-notch samples received the standard hardening treatment of air cooling from 1850°F and double tempering at 1050°F. The rough machined samples were ground to size after the heat treatment, and the notches were cut with carbide tipped tools. A 60-pound Olsen machine was used to break the specimens at temperatures between 212 and minus 100°F. The results are tabulated in Table 7 and graphically represented in Figure 5.

The results from the longitudinal impact tests (specimens machined from 1-1/8-in. round bars) show that M-7-12 displays no sharply defined transition temperature; there is no sudden decrease in impact strength at temperatures down to minus 100°F, where the energy absorbed was 15 ft-1b.

Transverse impact test data were obtained from bars machined from the 6-in. square discs previously mentioned. After the different homogenizing treatment and after rough machining the samples from a mid-radius location, the standard heat treatment was carried out. The results show that the transverse impact strength decreases with lower temperatures more rapidly than the longitudinal impact strength.

Of the two homogenizing treatments, the treatment given to disc 2 (double isothermal anneal) proved to be the better one with an impact strength about 5 foot pounds higher at room temperature, and about 3 foot pounds higher at minus 100°F. This difference in impact strength can be mainly attributed to the difference in Shepherd grain size which was 7 on bars from disc 2, compared with 5 on bars from disc 1. In general, impact strength measurements are more dependent on grain size than are tensile ductility measurements.

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Another series of longitudinal samples were hardened by air cooling from 1850°F and tempered at different temperatures up to 1100°F. The impact vs. tempering curve is shown in Figure 6. The most interesting part of the curve is the trough in the temperature zone of the peak of secondary hardness. The hardness vs. tempering curve is included in Figure 6 to illustrate this. No tests on samples of M-7-12 tempered in the range between 900 and 1000°F were carried out. Tests on Grovan samples, however, were carried out to establish its impact vs. tempering curve. This curve is almost identical with the curve of M-7-12, shown in Figure 6. In the zone of secondary hardening, impact results from Grovan samples were widely scattered. Some bars absorbed only 4 foot pounds in the impact test.

The fracture surfaces of the impact bars were fine grained (except for bars of disc 1) and flat. Most fractures showed a pattern of corrugations diverging from a central point at the bottom of the notch, towards the edge where the hammer struck. Another, deeper corrugation was observed running parallel to the notch and close to the edge of the hammer side. Only a slight lateral contraction could be seen. Bars broken at minus 100°F show no lateral contraction and no pattern of corrugations on the fracture surface. Similarly, bars broken at room temperature but tempered in the secondary hardening zone did not show a pattern of corrugations.

From a study of microstructures of Grovan impact bars, it appears certain that the fractures are of the intergranular type, whether tempered at 600°F, at 965°F in the peak hardness zone, or at 1050°F. This type of fracture can be attributed to the precipitation of carbides in the preferred location of the grain boundaries. It is not fully understood why secondary hardening produces an apparent



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brittle condition, although it is generally reckoned that the increase in hardness on tempering is caused by an increased amount of finely dispersed alloy carbides precipitated within the grains and in the grain boundaries. The lattice of the precipitate is initially coherent with that of the ferrite matrix, and the increase in hardness is associated with a lattice strain at the surface of the precipitate. The strain increases until coherency is destroyed by continued growth of the precipitate at higher temperatures or after longer tempering times. The structure softens, and the impact strength as well as the tensile ductility increase rapidly. It is not known at present how to produce a steel of this type with higher impact strength at this high hardness level, except by grain size control.

(6) Fatigue Properties

Several Krouse fatigue samples were rough machined from the 1-1/8-in. round bar stock, heat treated to R_0 53 by the standard heat treatment of air cooling from 1850°F and double tempering at 1050°F, ground to size, and finally hand polished. The machine used was a rotating beam unit of the cantilever type (10,000 rpm), and the dimensions of the specimen were as follows: length 3 in., minimum diameter 0.200 in., diameter on both ends 0.375 in., and radius (to produce the minimum diameter in the centre of the bar) 2 in. As shown in Figure 7, the endurance limit is about 122,000 psi at 10 million reversals; this is about 45 percent of the static ultimate tensile strength and can be considered a very satisfactory ratio for a steel with a hardness of R_c 53. Also, this ratio indicates a high degree of freedom from residual stresses.



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CONCLUSIONS

(1) The newly developed five percent chromium steel M-7-12 can be heat treated to a tensile strength of 280,000 psi by air cooling from 1850°F and tempering at 1050°F. This high strength condition of the steel exhibits very good tensile ductility combined with good impact properties.

(2) Tempering between 900 and 1000°F is not favoured because of a resulting loss in tensile ductility and impact strength. This brittle condition disappears when material is tempered above 1000°F. Tempering at 1050°F produces the most attractive combination of mechanical properties. The brittleness is not of the reversible type, e.g. not like the so-called blue brittleness of certain steels. Therefore it appears that M-7-12 can be used for high strength components exposed to all temperatures up to 1000°F over relatively long periods without danger of embrittlement or softening. The yield strength at 1000°F is 155,000 psi.

(3) Results from transverse test specimens compare favourably with results from longitudinal tests, however, a homogenizing treatment is recommended for forgings in order to minimize the effects of directionality. This treatment consists of hardening from a high temperature, followed by a double isothermal annealing operation.

(4) M-7-12 exhibits no sharply defined impact transition temperature above minus 100°F. Longitudinal tests at minus 100°F showed an impact strength of 15 foot pounds.

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(5) A high endurance limit of 122,000 psi, 45 percent of the static tensile strength, was measured. This is a good indication of the highly stress relieved condition after heat treating.

(6) Machinability of M-7-12 appeared to be good. Based on experience gained with Crovan, it can be said that welding, plating, and nitriding of the newly developed steel should be as feasible as with Crovan.

EGS/RB

REFERENCES

- (1) P. Payson and A.E. Nehrenberg, "New Steel Features High Strength and High Toughness", Iron Age, <u>162</u>, (1948), 64
- (2) A.G. Allten and P. Payson, "The Effect of Silicon on the Tempering of Martensite", Trans. ASM, <u>45</u>, (1953), 498
- (3) J.W. Sands and O.O. Miller, "Ultra High Strength Steels", Materials and Methods, <u>43</u>, (1953), 94
- (4) A.E. Powers, "Effect of Mo, W, and V on the High Temperature Rupture Strength of Ferritic Steel", AIME Trans., <u>206</u>, (1956), 1373
- (5) J.C. Hamaker, Jr., "Die Steel Useful for Ultra High Strength Structural Requirements", Metal Progress, <u>70</u>, (1956), 93
- (6) R.J. Nekervis, C.H. Lund, and A.M. Hall, "Survey Hot-Work Tool Steels for Aircraft and Missiles", Iron Age, <u>181</u>, (1958), 99 and 120.