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THERMAL EMBRITTLEMENT OF 4340 STEEL

Frank L. Carr, et al

Army Materials and Mechanics Research Center
Watertown, Massachusetts

December 1972

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TEMPER BRITTLENESS OF 4340 STEEL

FRANK L. CARR and THOMAS S. DeSISTO
METALS RESEARCH DIVISION

December 1972

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER
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13. ABSTRACT

Fracture appearance transition temperatures obtained with impact specimens were utilized to study the thermal embrittlement of 4340 steel between 950 and 1250 F (510 and 675 C). Embrittlement occurred at the higher temperatures but not in the vicinity of 1000 F within 16 days. The degree of embrittlement depended on both time and temperature. Reductions in toughness were correlated with changes in the morphology and size of ferrite grains as well as the size of carbide particles. These microstructural changes were similar to those observed in both 3140 and plain carbon steels by other investigators.

Conventional anisothermal procedures utilized to produce temper brittleness in low alloy steels also embrittle these steels by another mechanism. Thus, the degradation of toughness attributed to temper brittleness results from two different modes of embrittlement. Transitional behavior previously described as the retrogression of temper brittleness is concluded to result from thermal embrittlement. (Authors)

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Fracture appearance transition temperatures obtained with impact specimens were utilized to study the thermal embrittlement of 4340 steel between 950 and 1250 F (510 and 675 C). Embrittlement occurred at the higher temperatures but not in the vicinity of 1000 F within 16 days. The degree of embrittlement depended on both time and temperature. Reductions in toughness were correlated with changes in the morphology and size of ferrite grains as well as the size of carbide particles. These microstructural changes were similar to those observed in both 3140 and plain carbon steels by other investigators.

Conventional anisothermal procedures utilized to produce temper brittleness in low alloy steels also embrittle these steels by another mechanism. Thus, the degradation of toughness attributed to temper brittleness results from two different modes of embrittlement. Transitional behavior previously described as the retrogression of temper brittleness is concluded to result from thermal embrittlement.

CONTENTS

BACKGROUND

On several occasions the toughness of critical components of Army materiel fabricated from 4340 steel was determined to be relatively low for intermediate strength levels. Routine metallographic examination failed to reveal any cause for this degraded toughness. Although 4340 steel is considered to be immune to temper brittleness, the effects of tempering temperatures on the toughness of this steel apparently have not been studied. Jaffe and Buffum[1] reported two tempering regions in which temper brittleness developed in 3140 steel. These authors designated the resulting embrittlements as "lower nose" and "upper nose" temper brittleness. Later, Clancy and Norton[2] determined that the embrittling mechanism in 3140 steel occurring in the "upper nose" region was the change in morphology and size of ferrite grains concomitantly with the growth of carbides. Similar behavior in plain carbon steels was reported by Hyam and Nutting.[3] Both studies suggested that these microstructural changes be considered the fourth stage of tempering. Thus, there are strong indications that 4340 steel, although immune to embrittlement during tempering treatments in the vicinity of 1000 F (540 C), may be vulnerable to embrittlement at higher temperatures. This study is concerned with the development of any embrittlement in 4340 steel due to tempering between 950 F (510 C) and the critical temperature (A_{c1}).

PROCEDURE

Materials utilized for this study consisted of barstock of 4340 steel fabricated from three heats described below.

Barstock	Type of Heat	P content Weight %	ASTM Austenitic Grain Size #
9/16-in. square	Commercial Vacuum Arc Remelt (VAR)	0.006	9-11
5/8-in. round	Commercial Air Melted	.007	12-13
5/8-in. square	25# Laboratory Air Melted	.026	7-10

The chemical composition of these materials including minor elements is listed in Table I. These heats are comparable in major alloying elements. The higher contents of phosphorus and sulfur in the 25-pound laboratory heat were deliberate to study any possible effects of these elements on embrittlement. This laboratory heat is also slightly higher in carbon than the two commercial heats.

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[1]JAFFE, L. D., and BUFFUM, D. C. *Upper Nose Temper Embrittlement of a Ni-Cr Steel.* AIME Trans., v. 209, 1957, p. 8-16.

[2]CLANCY, W. P., and NORTON, M. R. *Microstructural Observations Pertinent to Tempering and Temper Brittleness.* ASTM Proc., v. 58, 1958, p. 912-930, also Ordnance Materials Research Office, OMRO 45, May 1958, AD 154-001.

[3]HYAM, E. D., and NUTTING, J. *The Tempering of Plain Carbon Steels.* J. of Iron and Steel Inst., v. 184, October 1956, p. 148-165.

Table I. CHEMICAL COMPOSITION

Type of Heat	Weight Percent								ppm				
	C	Mn	Ni	Cr	Mo	Si	P	S	O	N	As	Sb	Sn
Commercial - VAR	0.38	0.74	1.79	0.83	0.25	0.22	0.006	0.004	5*	40	150	10	90
Commercial - Air	.38	.76	1.72	.83	.26	.30	.007	.008	65*	45	90	15	30
Laboratory #25 Air	.43	.68	1.79	.92	.24	.21	.026	.015	65*	160	102	16	40

*Average of 2 or more analysis

Coupons of suitable length for Charpy impact specimens were cut from the bar-stock of each of the three materials. These coupons were normalized and hardened as follows:

1650 F (900 C), 1 hour, air cooled
1550 F (845 C), 1 hour, oil quenched

Groups of these coupons from the two commercial heats were tempered in salt at individual temperatures between 950 and 1250 F (510 and 675 C) for various times between 1/4 and 384 hours. Groups of coupons from the small laboratory heat were limited to a single tempering temperature of 1250 F (675 C) for various times.

After heat treatment these coupons were machined into 0.384-inch-square standard Charpy V-notch impact specimens. Four Rockwell C hardness measurements were made on each specimen and the hardness values obtained on each group of specimens, representing an individual tempering treatment, were averaged. Values less than R_C 15 were obtained on groups which received prolonged tempering at high temperatures, but for the sake of uniformity other hardness scales were not utilized.

The various groups of impact specimens were tested over a range of temperatures on a pendulum-type machine having a capacity of 217 foot-pounds and a striking velocity of 16.8 feet per second. The fibrous fracture percentage was determined for each specimen according to the ASTM method[4] and was plotted as a function of testing temperature to obtain the 100% fibrous fracture transition temperature.

A specimen was chosen at random from each of five groups of the commercial air-melted steel representing various tempering times at 1250 F (675 C). These specimens were polished, etched, and replicated in the transverse orientation by the single-stage method. These replicas were examined by electron microscopy and a representative area was photographed and enlarged. The average diameter of the ferrite grains and carbides were determined by measurements on these pictures.

[4]ASTM Standards, supplement V to A370 part 4, 1968, p. 481.

RESULTS AND DISCUSSION

Transition temperatures and average hardness values obtained for each group of specimens representing the various tempering treatments are listed in the appendix for the three heats of 4340 steel. Transition temperatures for both commercial steels - air melted and vacuum arc remelted - are illustrated as a function of tempering time for various temperatures in Figures 1 and 2. For the

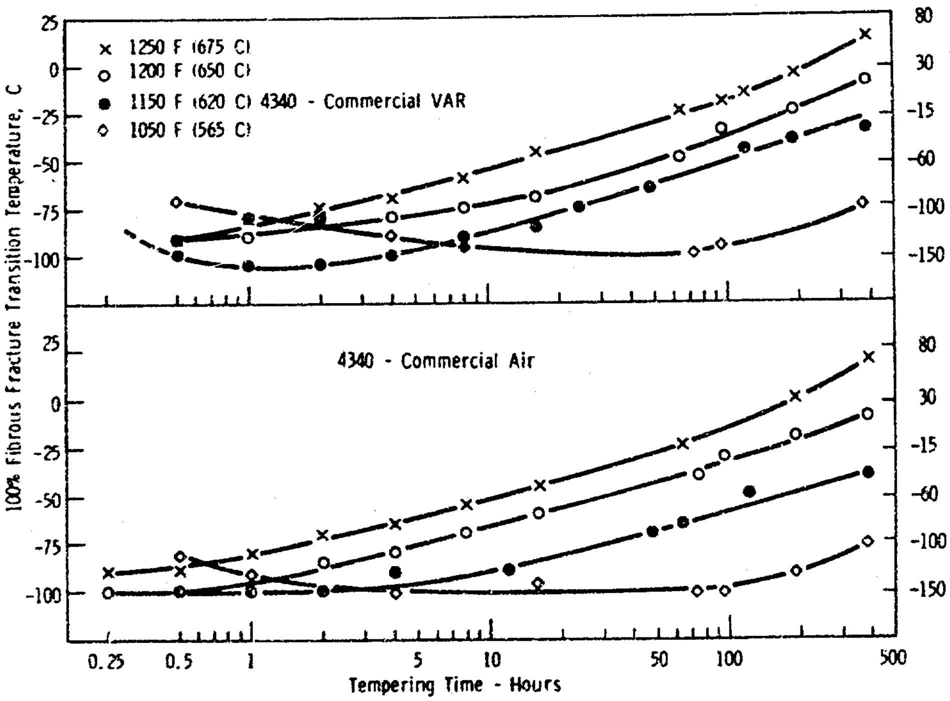

Figure 1. Transition temperature of 4340 steels versus tempering time at 1050, 1150, 1200, and 1250 F

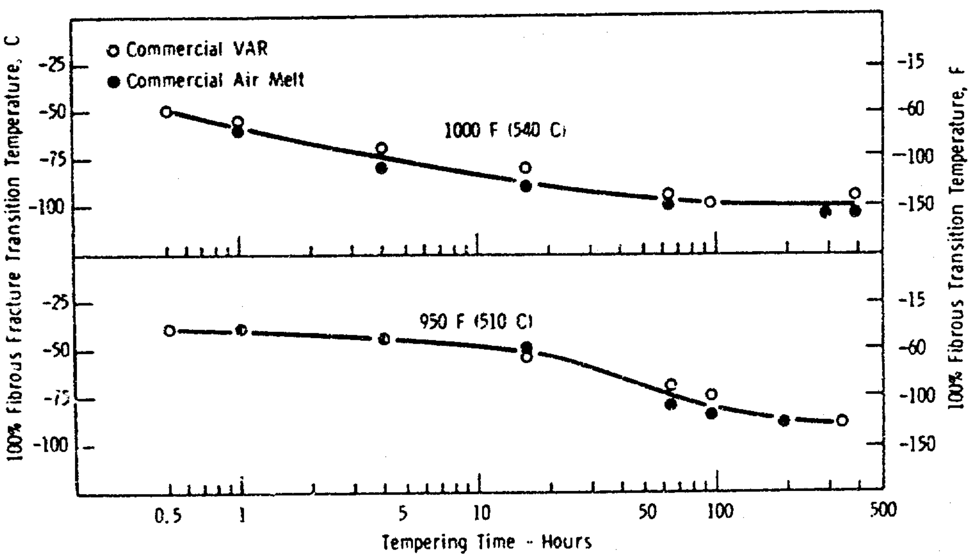

Figure 2. Transition temperature of 4340 steels versus tempering time at 950 and 1000 F

temperature range of 1050 to 1250 F (565 to 675 C) the transition temperature increased from about -150 F (-100 C) to values as high as 70 F (20 C) depending on both time and tempering temperature. Based on these data embrittlement commences in 4340 steel after the following approximate times and temperatures:

1250 F (675 C)	<1/2 hour
1200 F (650 C)	<1 hour
1150 F (620 C)	4 hours
1050 F (565 C)	100 hours

Thus, tempering beyond these times at the respective temperatures results in the degradation of toughness. This behavior is very significant with respect to critical components which receive prolonged tempering at relatively high subcritical temperatures. These high temperatures produce a significant decrease in the toughness of 4340 steel in a relatively short time. In contrast, a reduction in toughness due to this embrittlement requires more than 100 hours at 1050 F and does not occur at 1000 F in 384 hours (16 days).

Based on these results any isothermal or anisothermal treatment at high subcritical temperatures intended as a stress relief treatment may also cause some embrittlement of 4340 steel. In fact, any critical component fabricated from a low alloy steel which receives extended treatment between 1150 F and the A_{c1} should be suspected of embrittlement. Obviously the practice of double or triple tempering would not produce the optimum attainable toughness in this steel at these high temperatures.

Figure 2 presents the transitional behavior of both commercial heats of this steel at 950 and 1000 F (510 and 540 C). Decreasing transition temperatures with increasing times demonstrates that temper brittleness did not occur in these materials at these temperatures, at least for times of approximately 400 hours.

The immunity of this steel to temper brittleness has been known for some time. Knowledge of this immunity, in some cases, may be a source of false security unintentionally incorporated in the processing to obtain intermediate strengths.

Figure 3 illustrates the transitional behavior of these three heats of 4340 steel after tempering at 1250 F. Included in this figure are the data for 3140 steel obtained from the study by Jaffe and Buffum.[1] The rate of embrittlement is about the same for both types of steel. Despite significant differences in both phosphorus and sulfur contents the fracture transition temperatures of all heats of 4340 steel were the same, within experimental error, for similar heat treatments. This does not infer that either the shelf energy or individual energy values obtained at the same testing temperature were equal for these three materials. Both composition and processing exerted an influence on the energy values.

The average hardness values of specimens from the two commercial heats of 4340 steel which received various tempering treatments are plotted in Figure 4 as a function of tempering time for different temperatures. By cross plotting Figures 1 and 4 a correlation is obtained between hardness and transition temperature within the limits studied (R_c 10 to 40). This correlation results in a

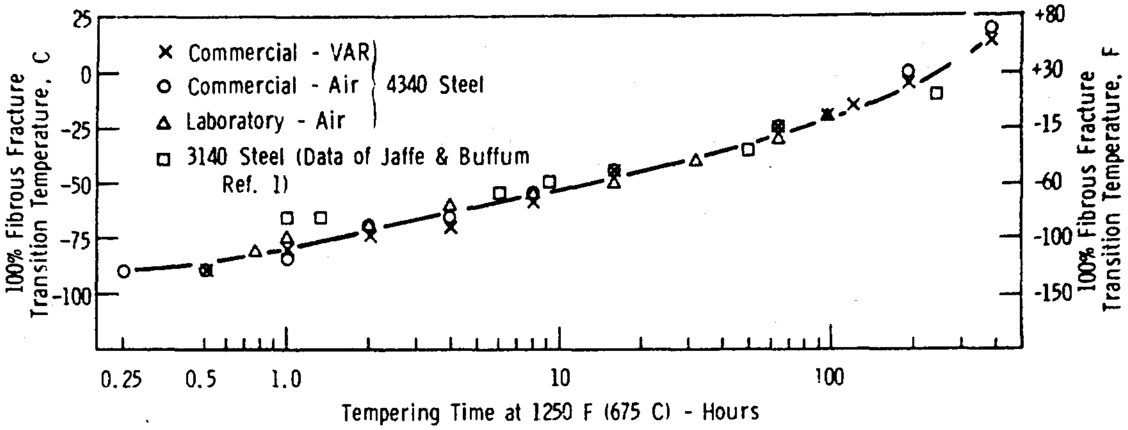

Figure 3. Embrittlement of 4340 and 3140 steels at 1250 F (675 C)

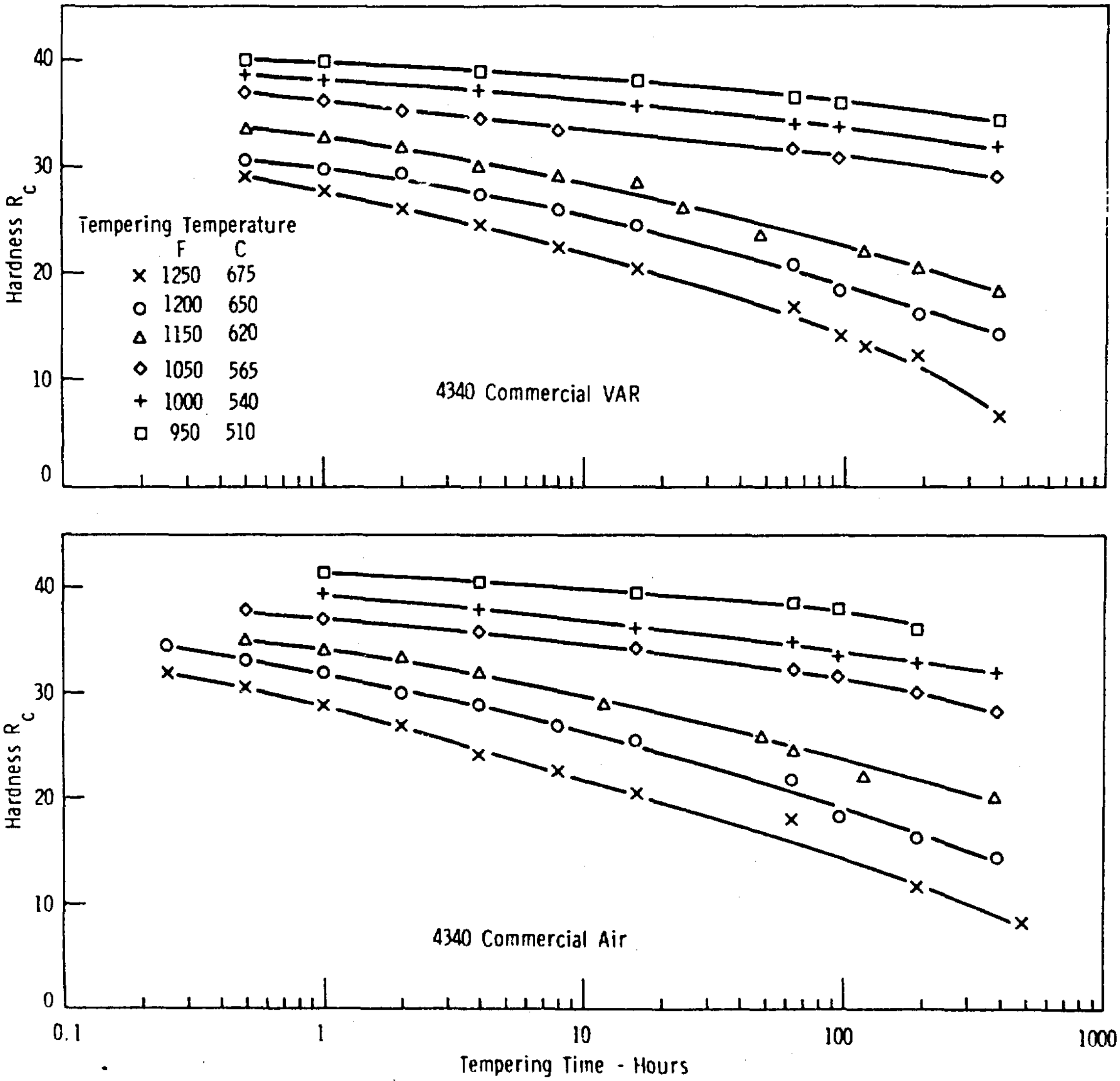

Figure 4. Hardness versus tempering time for various temperatures

C-type curve for both heats of steel depicted in Figure 5. Both curves indicate that optimum toughness is achieved at a hardness of R_c 30 to 35. For either increasing or decreasing hardness values beyond this range there is a continuous decrease in the relative toughness of this steel indicated by increasing transition temperatures (Figure 5).

The prior austenitic grain sizes of these steels were fairly comparable, however, larger differences in this metallurgical characteristic could be expected to influence both hardness and transition temperature.

Figure 6 illustrates the microstructure of the commercial air-melted steel after tempering for different times at 1250 F. In this composite illustration a change from acicular to equiaxed ferrite grains occurs within a relatively short time. For longer times both ferrite grain size and carbide size increase with increasing time. This confirms the observations of previous investigators[2] who reported that changes in the mechanical properties of 3140 steel at these high tempering temperatures was the result of microstructural changes. In Figure 6 it is evident that rather drastic changes in microstructure are associated with the gross increase in transition temperatures illustrated in Figure 3. It is also evident by comparison of Figures 1 and 6 that significant reductions in toughness occurring after relatively short times result from relatively subtle changes in microstructure.

Cursory determinations of both the ferrite grain size and carbide particle size were made on five specimens of the commercial air-melted steel representing different tempering times at 1250 F (675 C). These results are compiled in Table II. Based on this limited data the dual relationship between the ferrite grain size and carbide size on one hand, and both hardness and transition temperature on the other hand, are illustrated in Figures 7 and 8. These figures indicate that the hardness decreases and the transition temperature increases linearly as the logarithm of the square root of the average diameter of both ferrite grains and carbides. It is not possible to discriminate the influence of each of these microstructural features on these mechanical properties. These results are in agreement with Hyam and Nutting[3] who studied the tempering of a series of plain carbon steels which differed in carbon content from 0.1 to 0.9 weight percent. These investigators found an inverse linear relationship between Vickers hardness and tempering time plotted on a logarithmic scale. In addition these investigators reported the following:

- For any one steel, simple isothermal tempering treatments which produce the same hardness produce the same microstructure.
- The ferrite grain size of steels tempered under the same conditions of time and temperature decreases as the carbon content increases.
- The measured activation energy of the tempering process is not equal to the activation energy for the diffusion of carbon in alpha iron (about 20 kcal/g mol) but corresponds with the value for the self diffusion of alpha iron (about 60 kcal/g mol).

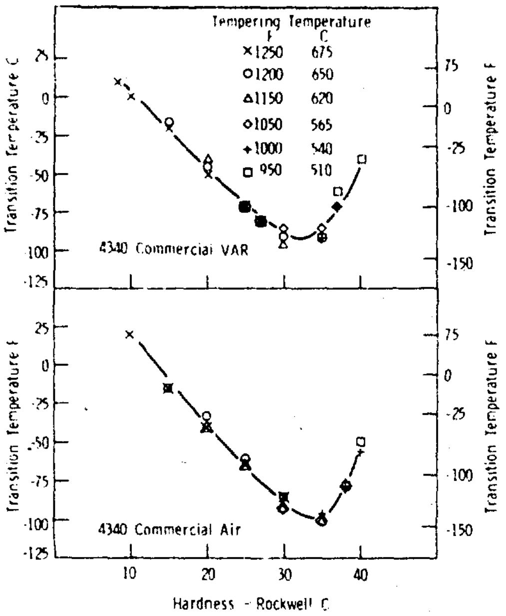

Figure 5. Hardness versus transition temperature

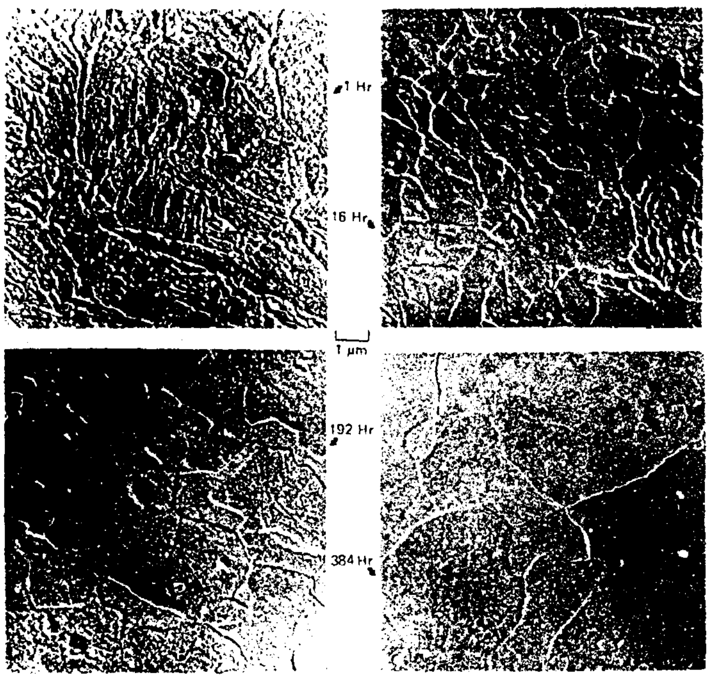

Figure 6. Effect of tempering at 1250 F (675 C) for various times on the microstructure - 4340 steel

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Table II. FERRITE GRAIN SIZE AND CARBIDE SIZE

Tempering Time at 1250 F, hr	Hardness R_c	Transition Temperature C	Average Grain Diameter inch, d	$\sqrt{d}$	Average Diameter of Carbides inch, c	$\sqrt{c}$
1	28.8	-80	3.94×10^{-5}	6.3×10^{-3}	4.42×10^{-6}	2.1×10^{-3}
4	24.1	-65	5.06	7.1	6.53	2.5
16	20.4	-45	6.16	7.9	9.19	3.0
192	11.6	0	9.89	9.9	1.52×10^{-5}	3.9
384	8.2	20	1.25×10^{-4}	1.1×10^{-2}	1.97	4.4

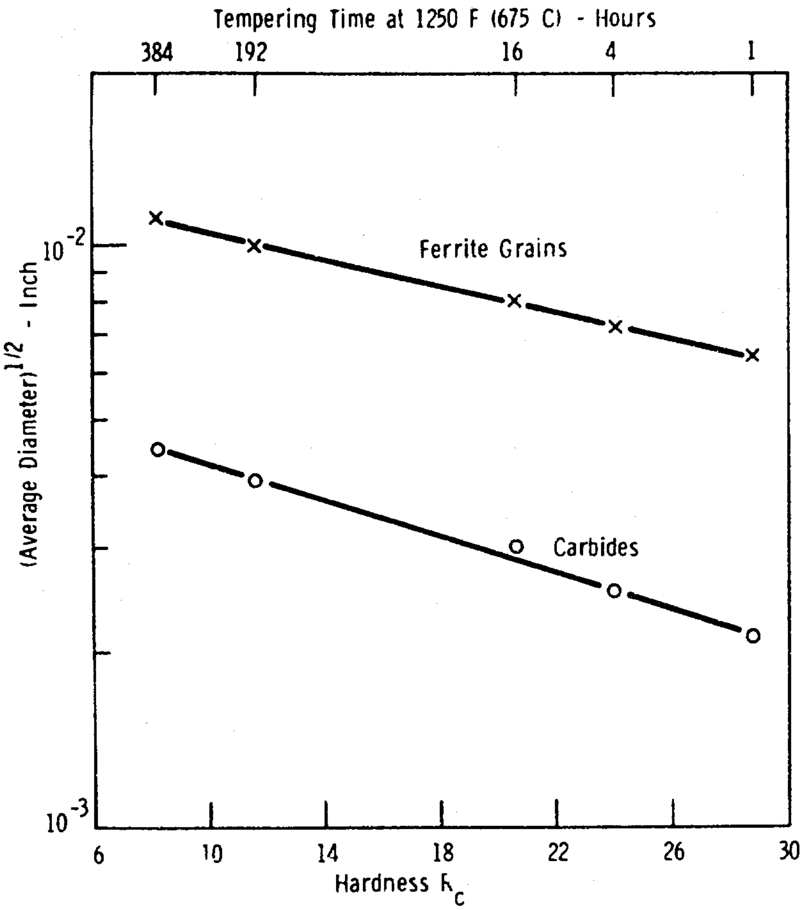

Figure 7. Hardness versus size of ferrite grains and carbides

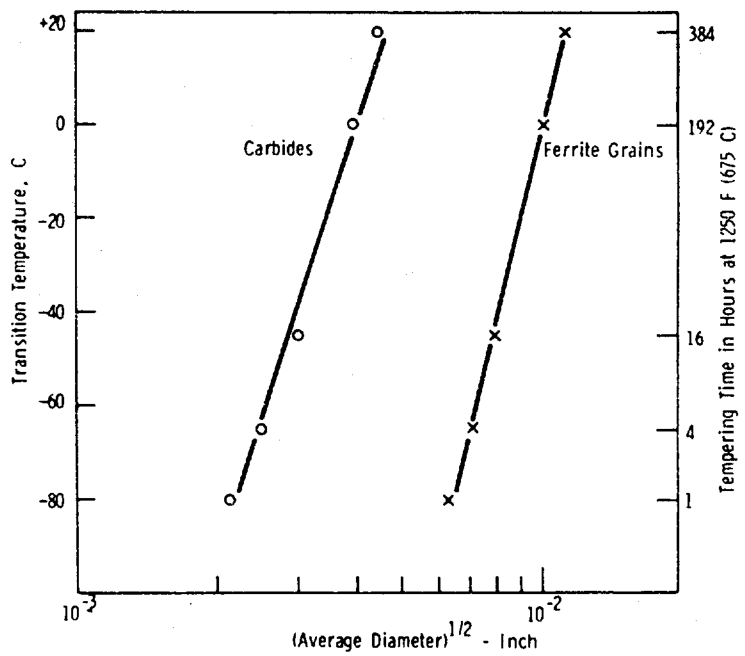

Figure 8. Transition temperature versus size of ferrite grains and carbides

In steels of different carbon contents tempered to the same hardness the size frequency distribution of the carbide particles is not the same.

Hyam and Nutting proposed a mechanism which required the diffusion of vacancies to be the rate-controlling factor governing the growth of carbides.

Compositional differences other than carbon could influence the kinetics of this reaction. Similar isothermal treatments were utilized in a recent study of embrittlement in 3% nickel steel.[5] At both 1150 and 1200 F significantly longer times were required for an increase in transition temperatures for this nickel steel than for these 4340 steels. Apparently the larger amount of carbide-forming elements (chromium, molybdenum, and vanadium) in the 3% nickel steel influenced the kinetics of this reaction.

Temper Brittleness

The decrease in toughness resulting from the described microstructural changes should not be confused with reductions in toughness due to temper brittleness in the vicinity of 1000 F. In the light of these findings the terminology applied to the former - "upper nose temper brittleness" - should be discontinued to avert confusion. It is also very probable that conventional procedures which utilize either slow furnace cooling or "step cooling" after an initial temper at a high subcritical temperature embrittle the steel by both mechanisms. This may also account, at least in part, for large differences in the measured values of the activation energy of temper brittleness contained in the literature.

A new interpretation replacing the retrogression of temper brittleness is obtained by applying the results of this study in retrospect to work performed by Jaffe and Buffum.[6] Figure 9 is a reproduction of the illustration of these investigators depicting temper embrittlement, de-embrittlement, and the retrogression of temper brittleness in 3140 steel. Current data justifies the following interpretation:

Data point 1 (Figure 9) represents tempered 3140 steel which is first isothermally embrittled (#2) then de-embrittled (#3) by holding at 1250 F for one hour followed by quenching. If the hold at 1250 F is extended or prolonged (#4 and #5) there is a reduction in toughness due to microstructural changes - increase in the size of ferrite grains and carbide particles. Comparable results are obtained by a single prolonged hold at 1250 F (#6). The slightly higher transition temperature at data point 5 compared to data point 6 is due to the additional subcritical thermal treatments received by these specimens. Thus this second embrittlement is due to changes in microstructure and not due to the regression of temper brittleness.

[5] CARR, F. L., and LARSON, F. R. *Thermal Embrittlement of Steel for 175-MM Gun Tubes*. Army Materials and Mechanics Research Center, AMMRC TR 69-16, June 1969.
[6] JAFFE, L. D., and BUFFUM, D. C. *Retrogression of Temper Brittleness*. Revue de Metallurgie, v. 58, 1951, p. 609, also Watertown Arsenal Laboratory, WAL 801/1-6, April 1950, PB 127-548.

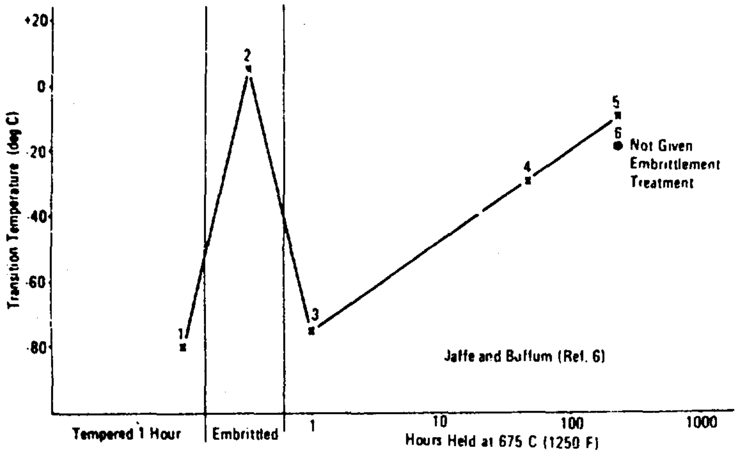

Figure 9. Effect of heat treatment upon transition temperature

SUMMARY

Based on results obtained with three heats of 4340 steel, this material is susceptible to reductions in attainable toughness due to isothermal treatments at high subcritical temperatures. The severity of this loss in toughness is very dependent on both tempering time and temperature. The 4340 steel, however, is immune to any embrittlement at either 950 or 1000 F (510 or 540 C) at least for times as long as 384 hours (16 days).

Confirming the results obtained by other investigators who utilized 3140 and plain carbon steels, these reductions in toughness are associated with microstructural changes. The morphology and size of ferrite grains together with the size of carbide particles controlled material toughness.

No significant difference in the fibrous fracture transition temperature was observed after similar thermal treatments on 4340 steels having different processing histories. Similiarly, different amounts of phosphorous and sulfur had no significant influence on the fibrous transition temperature.

The results of this study indicate that embrittlement previously attributed to the retrogression of temper brittleness is actually the results of thermal embrittlement.

Prolonged treatments at high subcritical temperatures aimed at relieving residual stresses can be deleterious to material toughness.

These results indicate that conventional procedures utilized to produce temper brittleness cause reductions in toughness by two distinct embrittling mechanisms. A change in conventional terminology is recommended to distinguish reductions in toughness due to microstructual changes (thermal embrittlement) from those associated with temper brittleness.

ACKNOWLEDGMENT

The authors acknowledge with gratitude the assistance of Frank Cotter and Andrew Connolly who performed the electron metallography and the size determinations of ferrite grains and carbide particles.

APPENDIX. TRANSITION TEMPERATURES AND AVERAGE HARDNESS VALUES FOR 4340 STEEL

Heat	Tempering Temperature		Tempering Time, hr	Transition Temp, deg C	Hardness R_c
Commercial VAR - 0.006% P	1250 F	675 C	0.5	- 90	29.0
			1	- 80	27.9
			2	- 75	25.9
			4	- 70	24.4
			8	- 60	22.2
			16	- 45	20.2
			64	- 25	16.7
			96	- 20	14.0
			120	- 15	13.1
			192	- 5	12.3
			384	15	6.6
	1200	650	0.5	- 90	30.6
			1	- 90	29.7
			2	- 80	29.5
			4	- 80	27.4
			8	- 75	25.9
			16	- 70	24.4
			64	- 50	20.7
			96	- 35	17.4
			192	- 25	16.3
			384	- 10	14.3
	1150	620	0.5	-100	33.5
			1	-105	32.9
			2	-105	31.9
			4	-100	30.3
			8	- 90	29.3
			16	- 85	28.5
			24	- 75	26.2
			48	- 65	23.6
			120	- 45	21.8
			192	- 40	20.4
			384	- 35	18.5
	1050	565	0.5	- 70	37.1
			1	- 80	36.4
			2	- 80	35.4
			4	- 90	34.7
			8	- 95	33.4
			64	-100	31.9
			96	- 95	30.9
			384	- 75	29.1
	1000	540	0.5	- 50	38.7
			1	- 55	38.2
			4	- 70	37.5
			16	- 80	35.7
			64	- 95	34.0
			96	-100	33.7
			384	- 95	32.0
	950	510	0.5	- 40	40.0
			1	- 40	39.7
			4	- 45	38.8
			16	- 55	37.8
			64	- 70	36.5
			96	- 75	36.0
			384	- 90	34.7

APPENDIX. TRANSITION TEMPERATURES AND AVERAGE HARDNESS VALUES FOR 4340 STEEL (Cont'd)

Heat	Tempering Temperature		Tempering Time, hr	Transition Temp, deg C	Hardness R_c
Commercial Air Melt - 0.007% P	1250 F	675 C	0.25	- 90	31.8
			0.5	- 90	30.4
			1	- 80	28.8
			2	- 70	26.9
			4	- 65	24.1
			8	- 55	22.5
			16	- 45	20.6
			64	- 25	17.9
			192	0	11.6
			384	20	8.2
	1200	650	0.25	-100	34.4
			0.5	-100	33.2
			1	-100	31.9
			2	- 85	30.0
			4	- 80	28.7
			8	- 70	26.9
			16	- 60	25.7
			64	- 40	21.9
			96	- 30	18.2
			192	- 20	16.3
			384	- 10	14.4
	1150	620	0.5	-100	35.0
			1	-100	34.1
			2	-100	33.4
			4	- 90	31.8
			12	- 90	28.9
			12	- 90	28.7
			48	- 70	25.9
			64	- 60	24.4
			120	- 50	22.2
			384	- 40	20.3
	1050	565	0.5	- 80	37.9
			1	- 90	36.9
			4	-100	35.8
			16	- 95	34.1
			64	-100	32.4
			96	-100	31.7
			192	- 90	30.2
			384	- 75	28.3
	1000	540	1	- 60	39.4
			4	- 80	37.8
			16	- 90	36.2
			64	-100	34.8
			96	-100	33.4
			192	-105	32.8
			384	-105	32.1
	950	510	1	- 40	41.5
			4	- 45	40.5
			16	- 50	39.5
			64	- 80	38.5
			96	- 85	37.9
			192	- 90	36.0
Laboratory Air Melt - 0.026% P	1250	675	0.75	- 80	27.3
			1	- 75	26.5
			2	- 70	25.6
			4	- 60	23.9
			8	- 55	21.6
			16	- 50	19.8
			32	- 40	17.3
			64	- 30	15.7
			96	- 20	14.6