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*SUSCEPTIBILITY OF TEMPER EMBRITTLED  
MATERIALS TO STRAIN-AGEING*

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PHYSICAL METALLURGY DIVISION

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SUSCEPTIBILITY OF TEMPER EMBRITTLED  
MATERIALS TO STRAIN-AGEING

by

R. F. Knight\*

ABSTRACT

A Ni-Cr low-alloy steel and a Ni-Cr-Mo steel which were, respectively, susceptible and insusceptible to reversible temper embrittlement, have been tested to determine their susceptibility to strain-ageing and strain-age embrittlement. Both materials were tested in embrittled and retoughened heat treatment conditions. Conditions of natural ageing and accelerated ageing were evaluated.

Natural ageing and a subsequent treatment at 100°C (212°F) resulted in only minimal changes in tensile properties and only a small degree of strain ageing. The Ni-Cr-Mo steel in the retoughened condition appeared to be somewhat more susceptible to strain ageing than the other materials.

Accelerated ageing at 205°C (400°F) resulted in a considerable amount of strain ageing in all the materials. This treatment resulted in a return of a marked yield point, higher yield strength and decreased tensile ductility. The relative degree of strain ageing of the four materials was the same as the degree of strain-age embrittlement shown by impact testing. The Ni-Cr-Mo steel in the retoughened condition was the most susceptible. For both heat treatment conditions the Ni-Cr-Mo steel was more susceptible to strain ageing than the Ni-Cr steel. For both steels the retoughening heat treatment resulted in the development of more strain ageing than did the embrittling heat treatment.

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## INTRODUCTION

The aim of this research was to evaluate and compare the degree of strain ageing developed in a Ni-Cr low-alloy steel which is susceptible to temper embrittlement and a Ni-Cr-Mo steel which is relatively insensitive to temper embrittlement processes. To this end, both steels have been tested in two heat-treated conditions, i.e., "embrittled" and "retoughened", under conditions of natural (room-temperature) and accelerated (elevated-temperature) ageing.

## MATERIALS

The experimental materials were prepared from a split 100-lb induction heat, melted under an argon cover and poured into two 50-lb ingots. A deoxidizing addition of 0.02% Al was added to the tap stream of both ingots. The chemical composition of the two steels was as shown in Table 1.

TABLE 1

Chemical Composition of Experimental Steels\*

Element, %	Steel A (Ni-Cr)	Steel B (Ni-Cr-Mo)
Carbon	0.32	0.305
Manganese	0.91	0.835
Silicon	0.41	0.41
Sulphur	0.019	0.018
Phosphorus	0.010	0.011
Chromium	0.64	0.64
Nickel	0.74	0.74
Molybdenum	0.018	0.425
Tin	0.008	0.008
Copper	0.040	0.046

\*Quantometer spectrographic report MS-SC-71-259.

The ingots were cropped and then forged and rolled to 1/2-in. thick plates from 1205°C (2200°F). The plates were normalized for 60 min at 900°C (1650°F), then austenitized for 30 min at 845°C (1550°F) and oil-quenched, then tempered for 60 min at 665°C (1230°F) and water-cooled, and finally tempered for 48 hr at 495°C (925°F) and air-cooled. After the above treatment the materials were in the "embrittled" condition, i.e., Condition E. Half of the material for both steels was then "retoughened", i.e., Condition T, by tempering for 30 min at 665°C (1225°F) and water cooling.

Tensile specimens, of 0.25-in. diameter, were obtained for both steels in the two conditions. The tensile properties were determined in duplicate for the heat-treated conditions, and for three strain-aged conditions. Charpy V-notch impact properties were determined for the heat-treated conditions and for two strain-aged conditions.

## MECHANICAL PROPERTIES

### A) TENSILE

The tensile properties of the experimental materials were as shown in Table 2 and Figure 1. The as-strained properties were calculated from the heat-treated values on the assumption that a pre-strained bar which is reloaded immediately would exhibit no ageing. The strain-aged bars were all pre-strained 2 1/2%. The properties of the steels in strain-aged condition I were determined by testing samples which had been aged naturally for 14 months. Samples were given a subsequent ageing treatment for 2 hr at 100°C (212°F) to give strain-age condition II. The remaining samples were given a similar treatment, but at 205°C (400°F) for strain-age condition III.

Figure 2 shows the relative susceptibility of the various materials to strain ageing, as indicated by the  $\Delta Y$  values, i.e., the difference in the flow stress before and after ageing. Figure 3 shows the changes in the ultimate tensile strength, percentage elongation and percentage reduction of area values ( $\Delta U$ ,  $\Delta E$  and  $\Delta RA$ , respectively) after ageing, as compared with the values for the as-strained condition.

TABLE 2

Tensile Properties of Experimental Steels

Steel	Heat-Treated Condition	Strain-Aged Condition	UTS kpsi	YS <sup>(1)</sup> kpsi	Y/U	% Elong in 4D	R.A., %	No. of Radial Fractures
A	E	unstrained <sup>(2)</sup>	110.8	89.6*	0.81	24.0	62.4	1
A	E	as-strained	113.6	101.7	0.90	21.5	60.8	-
A	E	I	114.3	106.6	0.93	23.0	64.2	2
A	E	II	116.1	108.7	0.94	22.5	63.1	2
A	E	III	114.5	112.8*	0.98	22.5	63.2	2
A	T	unstrained <sup>(2)</sup>	107.8	86.3*	0.80	25.0	67.6	0
A	T	as-strained	110.6	97.6	0.88	22.5	65.9	-
A	T	I	112.2	104.5	0.94	23.0	67.2	0
A	T	II	110.1	101.4	0.92	23.0	66.6	0
A	T	III	112.7	111.7*	0.99	21.5	64.5	0
B	E	unstrained <sup>(2)</sup>	136.7	119.9*	0.88	20.0	59.0	0
B	E	as-strained	140.2	128.7	0.92	17.5	57.5	-
B	E	I	140.0	133.4	0.95	20.0	61.3	1
B	E	II	140.6	132.9	0.95	20.0	59.4	0
B	E	III	142.5	141.9*	0.99	20.0	60.3	0
B	T	unstrained <sup>(2)</sup>	131.8	115.3*	0.88	23.0	63.0	0
B	T	as-strained	135.2	123.8	0.92	20.5	61.5	-
B	T	I	136.1	129.7	0.95	18.5	63.1	0
B	T	II	134.1	127.8	0.95	18.5	62.5	0
B	T	III	144.6	143.5*	0.99	15.0	59.3	0

(1) YS - for as-strained condition the value is average pre-strain flow stress; for other conditions the value is lower yield point where indicated by asterisk, and 0.2% offset for others.

(2) For the unstrained condition the values shown for YS are the average of 8 values, including the bars which were pre-strained for ageing tests.

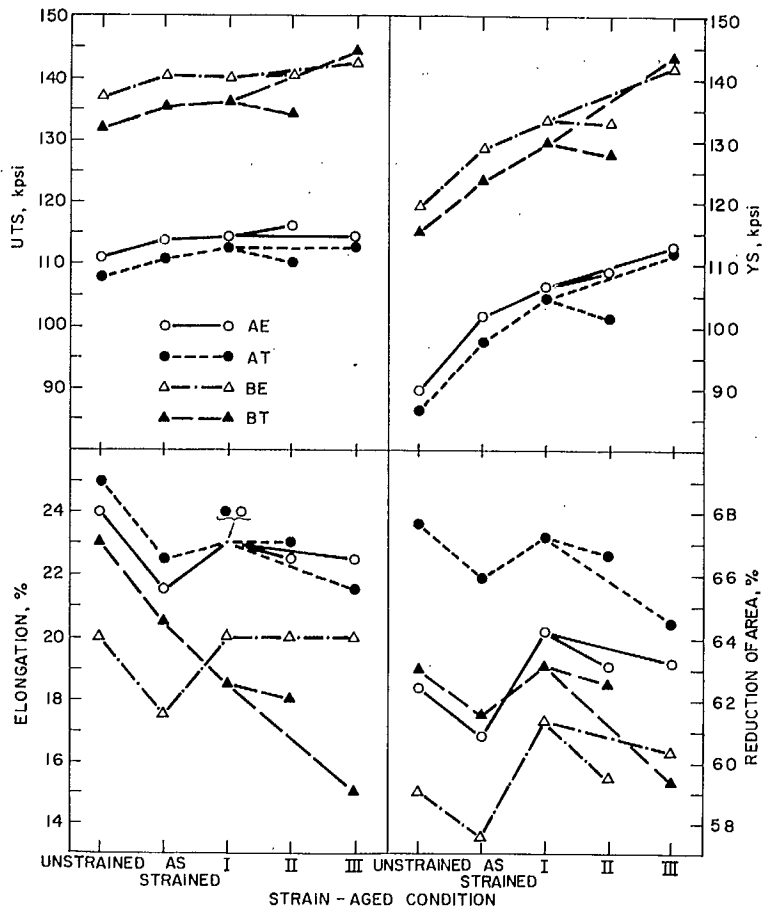


Figure 1. Tensile Properties of Experimental Materials in Unstrained, Strained and Strain-Aged Conditions.

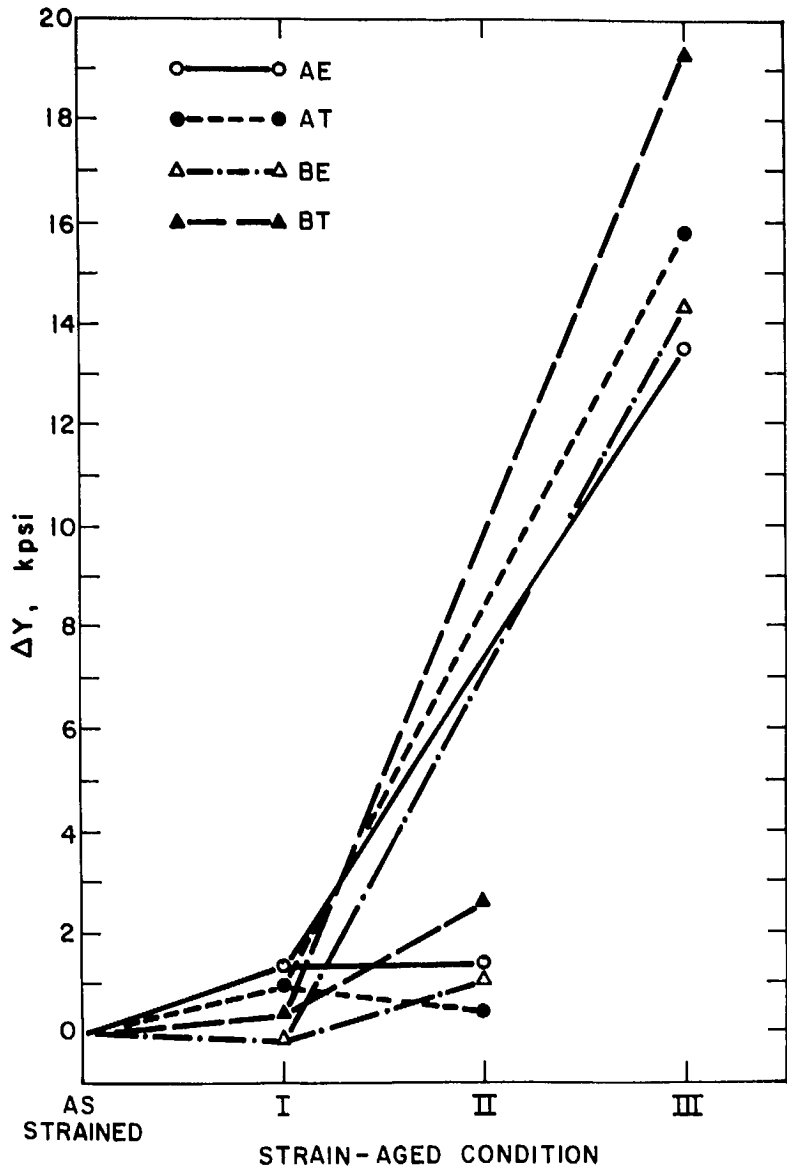


Figure 2. Susceptibility to Strain Ageing of Experimental Materials.

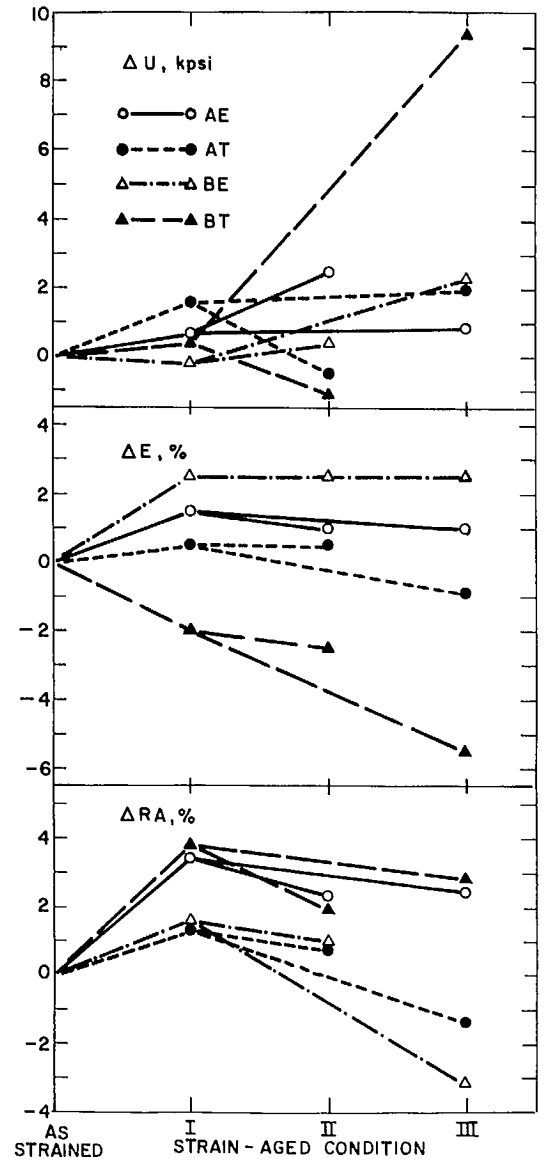


Figure 3. Effect of Strain Ageing on Change in UTS, % Elong and % R.A. Values.



### B) IMPACT

The Charpy V-notch impact properties of both steels in the two heat-treated conditions were determined. Plate-type tensile samples were pre-strained in tension, and Charpy specimens were machined from the strained portion of these bars. The actual degree of pre-strain used was 2.1% for Steel A and 3.0% for Steel B. The strained Charpy specimens were aged naturally for 14 months to duplicate the strain-aged condition I used for the tensile testing. Several of the bars were given a further treatment at 205°C (400°F) for 2 hr to duplicate the strain-age condition III. The results of impact testing are shown in Figure 4.

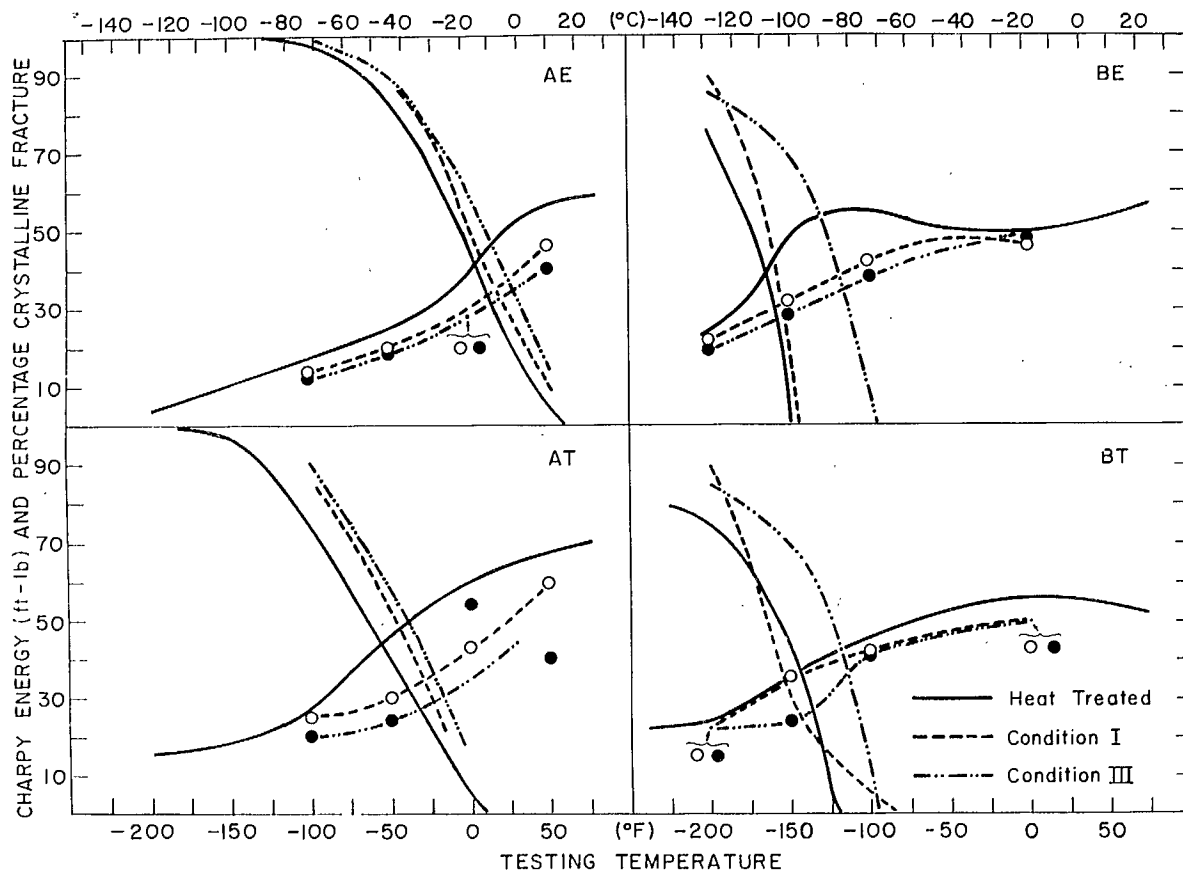


Figure 4. Impact Properties of Experimental Materials in Heat-Treated and Strain-Aged Conditions. The curves rising at higher temperatures are the impact-energy curves, and the other curves are the fracture appearance curves.

DISCUSSION

The relative susceptibility of the two steels to reversible temper embrittlement is revealed by the difference in impact transition temperatures in the embrittled and retoughened conditions. These transition temperatures and the degree of susceptibility are shown in Table 3 for the 35 ft lb energy level and the 50% crystalline fracture criteria.

TABLE 3

Impact Transition Temperatures and Susceptibility to Temper Embrittlement of Experimental Steels

Steel	Impact Transition Temperature, °F				Susceptibility Rating*			
	35 ft lb Criterion		50% Cryst Criterion		35 ft lb		50% Cryst	
	Condition E	Condition T	Condition E	Condition T	C°	F°	C°	F°
A	-9	-80	-4	-65	39.4	71	33.9	61
B	-170	-153	-173	-158	-9.4	-17	-8.3	-15

\*Susceptibility rating is the upward shift in impact transition temperature due to the embrittlement treatment.

It can be seen from Table 3 that the Ni-Cr steel shows a considerable susceptibility to temper embrittlement. The Ni-Cr-Mo steel shows no susceptibility; in fact, the "retoughening" treatment results in poorer impact properties. It should also be noted that, from the viewpoint of impact properties, the Ni-Cr-Mo steel is considerably superior to the Ni-Cr steel in that it maintains an adequate level of impact strength to considerably lower testing temperatures.

The relative degree of susceptibility of the two steels is also apparent from the fracture appearance of the tensile test specimens, as shown in Table 1. The Ni-Cr steel showed almost exclusively radial-split fractures in the embrittled condition. In the retoughened condition the fractures were cup-cone in appearance, as they were, with one exception, for all the Ni-Cr-Mo steel specimens in both conditions.

It can be seen from Figure 1, which shows the effect of various strain-ageing treatments on the tensile properties, that the Ni-Cr-Mo steel had a considerably higher strength and proportionately lower ductility than the Ni-Cr steel for both conditions of heat treatment and for all strain-aged conditions.

Comparing the as-strained condition with Condition I shows that on ageing at room temperature for 14 months both steels behaved in a similar manner in both conditions of heat treatment. They exhibited little change in ultimate tensile strength, a moderate increase in yield strength and, in general, an increase in tensile ductility. The only exception was the Ni-Cr-Mo steel in the retoughened condition which showed a decrease in percentage elongation.

Comparing Conditions I and II shows the effect of a further ageing treatment for 2 hr at 100°C (212°F). There was little change in ultimate tensile strength, with both steels in the embrittled condition showing a slight increase and both retoughened steels showing a slight decrease. The yield strength decreased slightly for all materials except the Ni-Cr steel in the embrittled condition. The tensile ductility decreased slightly for all materials.

Comparing Conditions I and III shows the effect of a supplementary ageing treatment at 205°C (400°F). Again, there was little change in ultimate tensile strength except for the Ni-Cr-Mo retoughened material which showed a significant increase. The effect on yield strength was more significant in that it was only after this treatment that there was a return of a marked yield point. All materials showed a significant increase in yield strength and also a decrease in tensile ductility. The latter was greater for the retoughened materials than for the embrittled materials.

It can be seen from Figure 2 that very little strain ageing occurred for any of the materials during holding at room temperature for 14 months. The Ni-Cr steel, particularly in the embrittled condition, appeared to be slightly more susceptible to material ageing than did the Ni-Cr-Mo steel. The subsequent ageing treatment at 100°C (212°F) is understood to be simply an extension of natural ageing because, up to that temperature, only the nitrogen content of the steels is effective on the strain-ageing reaction. This treatment resulted in little change for the Ni-Cr materials, but caused an increase in the susceptibility index for the Ni-Cr-Mo materials, which, although slight, would indicate them to be more susceptible than the Ni-Cr materials to natural ageing over a longer period of time. Figure 3, which shows the change in other tensile properties from the as-strained values, also indicates only minimal effects attributable to that portion of the strain-ageing phenomenon associated with the nitrogen content.

The treatment at 205°C (400°F), which introduces effects attributable to the carbon present in the steels into the strain-ageing mechanism, can be seen to have resulted in a significant degree of strain ageing in all the experimental materials. It is apparent that the Ni-Cr-Mo steel is more susceptible to accelerated strain ageing than the Ni-Cr steel, and that the retoughening heat treatment renders both steels more susceptible to strain ageing than does the embrittling heat treatment. The retoughened Ni-Cr-Mo steel, which appears to be the most susceptible to strain ageing of the four materials as indicated by the  $\Delta Y$  value, also shows the greatest increase in strength and decrease in tensile ductility after the 205°C (400°F) treatment.

The effect of strain ageing on the impact properties is shown in Figure 4. In all cases, the strain-aged properties are inferior to those of the heat-treated materials. However, to some extent, this is to be expected because the straining operation itself raises the strength and lowers the ductility. The amount of embrittlement due to natural ageing for 14 months is not apparent from Figure 4 because the impact properties for as-strained material were not obtained. However, the effect of straining and ageing for 14 months on the shift in impact transition temperatures from those of the heat-treated materials is as shown in Table 4.

TABLE 4

Increase in Impact Transition Temperature  
Due to Straining and Ageing for 14 Months

Material	Increase in Transition Temperature			
	35 ft lb Criterion		50% Cryst Criterion	
	F°	C°	F°	C°
AE	26	14.5	6	3.3
AT	50	27.8	18	10.0
BE	35	19.5	10	5.6
BT	4	2.2	-8	-4.4

The embrittlement caused by the additional ageing treatment at 205°C (400°F) is as shown in Table 5.

TABLE 5

Further Increase in Impact Transition Temperature  
Caused by Additional Ageing at 205°C (400°F)

Material	Increase in Transition Temperature			
	35 ft lb Criterion		50% Cryst Criterion	
	F°	C°	F°	C°
AE	10	5.6	12	6.7
AT	28	15.6	9	5.0
BE	20	11.1	35	19.5
BT	31	17.2	42	23.4

In terms of the impact energy criterion, the degree of embrittlement is in the same order for the various materials as that of the degree of susceptibility to strain ageing shown by the tensile tests. The degree of strain-age embrittlement due to the 205°C (400°F) treatment is, particularly in terms of fracture appearance, significantly less for the Ni-Cr materials than for the Ni-Cr-Mo materials. It is also apparent that the degree of strain-age embrittlement is greater for the retoughening heat treatment than for the temper-embrittling heat treatment.

SUMMARY AND CONCLUSIONS

Natural ageing and ageing at 100°C (212°F) caused only minimal changes in tensile properties because the presence of aluminum prevented significant ageing effects due to nitrogen. The 205°C (400°F) treatment introduced carbon effects into the strain-ageing phenomenon, and a significant degree of strain ageing was observed for all materials.

The molybdenum-bearing steel exhibited a greater degree of strain ageing than did the Ni-Cr steel, particularly for the accelerated ageing treatment. It would appear from this that molybdenum slows down the rate at which carbon can precipitate as carbide, and thus leaves more carbon in solution to interact with dislocations.

For both steels, under conditions of accelerated ageing, there was a greater ageing effect when they were in the retoughened condition. The higher tempering temperature used for the retoughening treatment would allow more carbon to remain in solution than did the lower temperature used to attain the temper-embrittled condition.

As a consequence of these trends, the greatest degree of strain ageing was found for the Ni-Cr-Mo steel in the retoughened condition. It exhibited the highest  $\Delta Y$  value for the 100°C (212°F) ageing treatment. It had a  $\Delta Y$  value of 19 kpsi after the accelerated ageing treatment, as well as a significant increase in the ultimate tensile strength and decrease in percentage elongation. It showed an increase of 31F° (17.2C°) in the 35 ft lb impact transition temperature due to the accelerated ageing treatment.

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