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Publication Date

1966-03-01

UCRL-16761

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Proceedings of the
American Society of Metallurgists
November 12, 1966 - Cleveland, Ohio

UCRL-16761

UNIVERSITY OF CALIFORNIA
Lawrence Radiation Laboratory
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AEC Contract No. W-7405-eng-48

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March 1966

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ABSTRACT

The response of quenched and tempered, and ausform H-11 steel to strain aging was investigated. The straining temperature was the most important variable. Straining at elevated (up to 900°F) temperatures and retempering resulted in large increases (~ 30%) in yield strength without the loss in ductility associated with static strain aging. The yield to ultimate strength ratio approached unity in the strain aged steels, however, elongations of 8-12% were retained.

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INTRODUCTION

The demand for structural materials with improved properties has stimulated much research. As a result, the theoretical understanding of strengthening mechanisms has been sufficiently developed to be useful in the design of high strength materials. However, in the more critical area of fracture toughness, the metallurgist is still dependent on an empirical approach. In this investigation the effects of strain aging on the strength and ductility of a hot work die steel were studied. The objective was to find processing conditions which resulted in increases in strength without significant loss of ductility. It was hoped that such conditions would also maintain the fracture toughness of the material. This hope was borne out in subsequent work.¹

The static strain aging (straining at room temperature followed by aging) of quenched and tempered 4340 has been investigated by Stephenson and Cohen.² Although large increases in strength were obtained, the strain aged specimens exhibited mechanical instability at the yield point. The authors pointed out that this circumstance made the use of such materials impractical. Zackay and Gerberich³ investigated the static strain aging of ausform H-11 and found significant strengthening could be obtained without introducing mechanical instability.

The present investigation was primarily concerned with dynamic strain aging (i.e. straining at a temperature where aging may occur simultaneously). The principal variables studied were the temperature of pre-strain (so called to avoid confusion with the straining that occurs in the final tensile test) and the prestrain rate. The effect of tempering after pre-strain was studied in a separate group of experiments. Both conventional (quenched and tempered) and ausform steels were investigated. An attempt

was made to correlate the resultant properties with the distribution of carbide particles, as observed in extraction replicas.

MATERIALS AND EXPERIMENTAL PROCEDURES

Materials

All experimental studies were made on two heats of vacuum induction melted H-11 (Vascojet 1000) steel supplied by Vasco Corporation. The composition of these steels is shown in Table I. The properties of the vacuum melted steels were determined for two different thermomechanical treatments. Steel A was austenitized for 1/2 hour at 1850°F in an inert atmosphere, air-cooled, and tempered at 900°F for one hour. The latter tempering treatment was intended to minimize the embrittling effects of retained austenite. Steels having the above treatment will be referred to throughout the paper as "conventional steels".

The second heat (Steel B) was subjected to the ausform process through the courtesy of the Ford Motor Company, Dearborn, Michigan. The ausform process consists of deforming metastable austenite at an elevated temperature prior to transformation to martensite. Alloy steels processed in this manner exhibit superior strength and ductility than when conventionally heat treated and also possess better fatigue and impact strength.⁴ This steel was austenitized at 2100°F, deformed 87% at 1050°F, air cooled, and subsequently double tempered at 500°F. The tempering is necessary to prevent delayed cracking after quenching. Steels processed in the above fashion will be referred to as "ausform steels".

Tensile Tests

Tensile specimens of both the conventionally treated and ausform steels were precision ground to the dimensions shown in Fig. 1. Specimens

Table I-Compositions of the H-11 Vacuum Melted Steels

	<u>C</u>	<u>Si</u>	<u>Mn</u>	<u>S</u>	<u>P</u>	<u>Cr</u>	<u>V</u>	<u>Mo</u>
Steel A (conventional)	.40	.85	.33	.015	.011	5.06	.5	1.28
Steel B ausform	.40	1.00	.25	.008	.011	5.09	.5	1.4

were flooded with coolant during the grinding operation and the maximum depth of cut was limited to 0.001".

All tensile testing was done in an Instron machine. Elevated temperature tests were conducted in an inert atmosphere. The temperature difference between the upper and lower tensile grips was less than 4°F. The elongation of the specimen for both room and elevated temperature tests was sensed by means of a differential transformer coupled directly to a micrometer.

The experimental procedure followed in the dynamic strain aging of the conventionally treated and the ausform steels is shown in Fig. 2. In the case of both steels, the term "pre-temper" refers to the heat treatment following transformation of the steel to martensite and preceding the straining operation.

After pre-straining and subsequent tempering, specimens were ground to a uniform diameter over the gage length and then tested to fracture at room temperature. A strain rate of $1.35 \times 10^{-4} \text{ sec}^{-1}$ was used for all room temperature tests. Strain rates of $1.35 \times 10^{-3} \text{ sec}^{-1}$, $1.35 \times 10^{-4} \text{ sec}^{-1}$, and $1.35 \times 10^{-5} \text{ sec}^{-1}$ were used for pre-straining.

EXPERIMENTAL RESULTS AND DISCUSSION

The discussion of experimental results will be centered on the variable of pre-strain temperature in view of the over-riding influence of this variable on the resulting mechanical properties. The influence of prestrain temperature and other variables such as the prestrain strain rate, and the pre- and post-tempering temperatures on (1) the strength and ductility, and (2) the shape of the stress-strain curve will be discussed for the conventional and the ausform steels, respectively.

Conventional Steels

(1) Strength and Ductility

All conventional steels were pre-tempered at 900°F. At lower pre-tempering temperatures there was insufficient ductility for pre-straining. Similarly, the majority of tests were conducted with a post-tempering temperature of 900°F since all specimens post-tempered at less than 900°F were brittle.

The response to strain aging of a conventional steel, pre- and post-tempered at 900°F, strained 2% at a strain rate of $1.35 \times 10^{-5} \text{ sec}^{-1}$ (the slowest employed in this study) as a function of pre-strain temperature is shown in Fig. 3. The room temperature properties of the strain aged steels, are shown in the upper three panels while the flow stress at two percent strain at the prestrain temperature is shown in the lowest panel. The room temperature properties of the unstrained steel, double tempered at 900°F, are indicated by arrows labeled "R.T. base".

As a consequence of the elevated temperature straining the yield strength was increased, on the average, about 80,000 psi, or 34%, over the range of pre-strain temperatures investigated. The ultimate tensile strength was increased about 14,000 psi, or 4.5%. Thus the yield-to-ultimate ratio was increased by strain aging.

The ductility of the strain-aged conventional steels was strongly dependent on the pre-strain temperature, as seen in Fig. 3. The maximum in uniform elongation at 750°F is less than that of the unstrained steel by about 2%, viz., the amount of pre-straining. Thus, at a pre-strain temperature of 750°F, it was possible to increase the strength by about 34% with a concomitant loss in uniform elongation of 20% and with no loss in reduction in area.

The flow stress at the pre-strain temperature decreased monotonically with temperature and reflected none of the undulations observed in the room temperature properties.

Similar plots are shown in Fig. 4 for specimens pre-strained at a higher strain rate ($1.35 \times 10^{-3} \text{ sec}^{-1}$). The dependence of yield and ultimate strength on the pre-strain temperature was nearly identical to that obtained at the lower strain rate; however, a maximum in uniform elongation was not observed. Unlike the specimens pre-strained at the slower strain rate, a large decrease in the elongation, i.e., 50% or more, was observed at all pre-strain temperatures below 750°F. There was also a decrease in reduction in area at the higher strain rate. The strength increase was about 50,000 psi, i.e., approximately 20%.

Tests conducted in the initial phases of this study indicated that low post-tempering temperatures usually led to poor ductility. For example, steels pre-tempered at 900°F, pre-strained 2% at 750°F, and post-tempered between 300° and 800°F consistently had less than 4% uniform elongation. Strength increases similar to those tempered at 900°F were observed but both uniform elongation and reduction in area values were always lower.

A minimum tempering temperature of 900°F for H-11 steel is generally recommended by producers for both the conversion of retained austenite and for sufficient tempering of the as-quenched martensite. The lack of ductility for dynamically strain aged specimens post-tempered below 900°F may thus be related to these factors in the conventionally heat treated steels.

(2) The Shape of the Stress-Strain Curve

The processing variables investigated, especially those of pre-strain and post-tempering temperature exerted a profound influence on the shape

of the stress-strain curve. The general types of stress-strain curves encountered in this investigation are shown in Fig. 5. The curve labeled Type I is typical of conventionally quenched and tempered steel viz., a relatively low yield-to-ultimate ratio, a work hardening rate which decreases smoothly with increasing strain and a large region of uniform plastic strain. This type of curve was observed on double tempered unstrained conventional H-11 steel.

The curve labeled Type II was characteristic of conventional steels strained at room temperature and is similar to those obtained by Stephenson and Cohen² on 4340 steel. The essential feature of this type of plastic behavior is the onset of plastic instability at the yield stress. The exact slope of the stress-strain curve at or near the yield appears to be related to rheological factors determined by the shape of the specimen as well as by the method of determining the strain, i.e., whether by measurement of cross-head movement or by the use of an extensometer. Type II curves were observed for conventional heat treated specimens strained at room temperature, for those pre-strained in the 600°F range, and for dynamically strain aged specimens subsequently post-tempered at a low temperature.

Type III curves were observed for the conventional steels that were dynamically strained and post-tempered at the highest temperature investigated, i.e., 900°F. These curves are characterized by a high yield-to-ultimate ratio, medium to large regions of uniform plastic strain, and by featureless stress-strain behavior beyond the yield stress.

Type IV curves exhibiting a yield drop followed by a very low rate of work hardening to fracture were observed for conventional steels dynamically strain aged at pre-strain temperatures on either side of the

600°F embrittling range, i.e., either between 300° and 500°, or between 750° and 800°F. Frequently, a slightly increased rate of work hardening would be evidenced at large strains as shown in a second example of Type IV curve in Fig. 5.

The variation in the type of the stress-strain curve at room temperature with pre-strain temperature is shown schematically in Fig. 6 for both the slow and the fast strain rates. For room temperature straining, Type I curves (unstable plastic behavior) were in evidence while in the middle temperature range Type IV curves (characterized by a yield drop) were obtained and, finally, at the highest temperature, Type III curves (relatively featureless with a relatively high yield strength and high uniform elongation) were exhibited.

A prominent feature of the room temperature stress-strain curve was that of the yield drop. The magnitude of this drop (measured from the peak load to the plateau of the curve beyond the yield point) was determined for various processing conditions. The influence of the pre-strain temperature on the size of the yield drop at several strain rates is shown in Fig. 7. The yield drop was exhibited over a pre-strain temperature interval of about 400°F and its magnitude was strongly affected by strain rate. The peak of the yield drop vs pre-strain curve for the slower strain rate is three times higher than that for the higher strain rate. The yield drop peaks for both strain rates were at 600°F. At 800°F there was virtually no discernable yield point shown at either strain rate. The presence of these yield drops at both strain rates is reflected of course in the yield strength vs pre-strain temperature curves as shown by the broad maxima centered about 600°F in the upper panel of Figs. 3 and 4.

Ausform Steels

In general, the response of the ausform steels to dynamic strain aging was similar to that of the conventional steels. As in the case of the conventional steel, the variables of pre-strain temperature and strain rate were investigated in greatest detail although limited studies were made of the influence of pre- and post-tempering temperatures.

The ausform steels were stronger and more ductile prior to dynamic strain aging, and for the most part suffered a less catastrophic loss in ductility at certain critical pre-strain temperatures than did the conventional steel. The pre-strained ausform steel was embrittled after being post-tempered in the approximate temperature range of 600° to 750°F. Consequently, most experiments were confined to specimens post-tempered at 900°F.

(1) Strength and Ductility

The variation of strength and ductility of the dynamically strained ausform steel with pre-strain temperature at the slowest ($1.35 \times 10^{-5} \text{ sec}^{-1}$) and the fastest ($1.35 \times 10^{-3} \text{ sec}^{-1}$) strain rates employed are shown in Figs. 8 and 9, respectively. All the data in these figures were from specimens pre-tempered at 500°F, strained 2% at the indicated pre-strain temperature, and post-tempered at 900°F.

The dynamically strain aged ausform steel specimens showed an increase in yield strength varying from approximately 50,000 to 100,000 psi. The yield strength increase was strongly dependent on the pre-strain temperature, especially at the slower strain rate. The ultimate tensile strength was relatively insensitive to pre-strain temperature. A peak in yield strength and ductility at approximately 525°F was evident in specimens tested at both strain rates. However, the effect was more pronounced at the slower strain rate, as shown in Fig. 8. At the peak of strength and ductility,

the yield strength and elongation for the slow and the high strain rates were, approximately 390,000 psi (a 36% increase) and 13%, and 359,000 psi (a 30% increase) and 12%, respectively. The increase in uniform elongation over that of the unstrained specimens that was exhibited at both high and low strain rates may not be statistically significant.

The elevated temperature flow stress at two percent strain of the ausform steel drops much more rapidly with temperature than that of the conventional steel. The curves are featureless - with the exception of the single questioned point at 525°F for the rapidly strained specimens.

A limited number of tests were made on specimens pre-strained at low temperatures (300° - 450°F) and post-tempered below 900°F. High strength and excellent ductility was observed for specimens pre-strained and post-tempered at or below 500°F. For example, at a pre-strain temperature of 300°F and a post-temperature of 350°F a yield strength of 397,000 psi and an elongation of 10% was obtained. However, specimens post-tempered between 500° and 800°F were brittle.

(2) The Shape of the Stress-Strain Curve

Unlike the dynamically strained conventional steel, there were no critical pre-strain temperatures which resulted in Type II (plastically unstable) stress-strain curves for the ausform steels. Type II curves were observed only for specimens that had been subsequently post-tempered between 500° and 750°F. Type IV curves (showing a yield phenomena) were seen for virtually all specimens pre-strained from 350° to 600°F while Type III curves (featureless with relatively high yield strength) were exhibited for specimens pre-strained between 750° and 900°F. The variation in the shape of the stress-strain curve with pre-strain for several strain rates is shown schematically in Fig. 10.

The magnitude of the yield drop and its response to strain rate (at the pre-straining temperature) in the ausform steel was similar to that of the conventional steel, as shown in Fig. 1. As in the conventional steel, the yield drop was greatest in those specimens strained at the slower rate. Although there is insufficient data to confirm the indicated trends, the peak for the maximum yield drop in the ausform steels appears to be at a lower pre-strain temperature (525°F) than that of the conventional steels (about 600°F).

MICROSTRUCTURE

An attempt was made to ascertain the structural changes caused by strain aging, and to relate such changes to the mechanical properties. Carbide extraction replicas were prepared by standard techniques^{*} and examined in an electron microscope. In the case of the ausform steels, no significant structural differences were observed as a function of processing. Carbide replication has however been successfully applied to dynamically strain aged ausform steels of different composition, notably ones lower in carbon content. The reason for this difference in replicating behavior is not known. Structural changes were observed, however, in the conventional steels, as is shown in Fig. 12. Agglomerates of large carbide precipitates were observed in the unstrained steel, Fig. 12A. In the strain aged steels, Fig. 12B and C, these have been replaced by a finer, more uniformly distributed, precipitate. The original precipitate has apparently redissolved, and the carbon reprecipitated. Similar observations were made by Stephenson and Cohen in statically strain aged 4340.² The dynamically strain aged structure, Fig. 12C, consists of a higher volume fraction of more uniformly sized precipitates than the statically strain aged case, Fig. 12B. Attempts to identify this precipitate by electron diffraction were unsuccessful.

The particle spacing does not account for the observed strength on the basis of the Orowan criterion for yielding.⁵ This criterion assumes that the yield strength is determined by the stress necessary to force

* A carbon film is deposited on an electropolished specimen, and subsequently removed by etching. Carbide particles, being insoluble in the etchant, are retained on the carbon film.

a dislocation between adjacent particles. This stress is inversely proportional to the spacing of the particles. At the observed (300,000 psi) strengths, the particle spacing would have to be about 100Å to satisfy the Orowan criterion. This is close to the resolution limit of the carbide extraction technique. Transmission microscopy, which has higher resolution is being employed in current studies.

SUMMARY AND CONCLUSIONS

The tensile properties of H-11 steel were significantly changed by dynamic strain aging. In both conventional and ausform H-11 steel, large increases in strength were obtained with little or no loss in ductility as measured by percent elongation. The effects of dynamic strain aging will be summarized separately for each type of steel.

Conventional (quenched and tempered) Steel

1. Straining at room temperature followed by aging resulted in mechanical instability at the yield point, as found in a previous study of 4340.

2. A 900°F tempering temperature was required for adequate ductility during prestrain. A similar post-tempering temperature was necessary to avoid embrittlement in the final test.

3. Prestrain temperatures of 300-450°F and 750-900°F produced good combinations of strength and ductility. Specimens strained at intermediate temperatures exhibited instabilities or embrittlement. A high strain rate (during prestrain) accentuated these effects.

4. Yield drops of up to 15,000 psi occurred during final testing after prestrain in the intermediate temperature range at the low strain rate ($\sim 10^{-5}$ sec⁻¹). No yield drops were seen after prestrain at the high

($\sim 10^{-3}$ sec $^{-1}$) strain rate.

5. Microstructure

Carbide extraction replicas indicated that strain aging resulted in solution of massive carbides and the formation of a finer, more homogeneous precipitate.

Ausform Steel

1. Mechanical instabilities were not observed after either static or dynamic strain aging.

2. An initial tempering temperature of 500°F was required to prevent delayed cracking after the ausform processing. Both high ($> 750^{\circ}\text{F}$) and low ($< 500^{\circ}\text{F}$) post tempering temperatures resulted in good combinations of strength and ductility. Tempering in the intermediate range resulted in embrittlement.

3. Prestrain temperatures up to 600°F resulted in useful strengthening, with a maximum at about 500°F. The strengthening effect decreased at higher temperatures. No embrittling range was observed.

4. The yield drop behavior was similar to that of the conventional steel.

Sample properties of strain aged conventional and ausform steels are tabulated below.

Conventional - All Specimens Pre- and Post-tempered at 900°F

Strain Temp (°F)	YS	UTS	% elong	% R.A.
---	236	306	9	15
300	308	316	8	13
750	319	322	8	15
900	289	330	6	18

Ausform - Pretempered 500°F

Strain Temp (°F)	Post Tempering Temp (°F)	YS	UTS	% elong	% R. A.
---	900	275	325	11	20
300	350	397	397	10	30
450	900	370	374	10	19
525	900	390	390	13	27
600	900	358	375	12	28
900	900	347	380	8	40

ACKNOWLEDGMENTS

The authors are grateful to Prof. E. R. Parker for helpful discussions.

This work was performed under the auspices of the United States Atomic Energy Commission.

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FIGURE CAPTIONS

- Fig. 1 Dimensions of tensile specimen.
- Fig. 2 Schematic diagram of thermomechanical processing.
- Fig. 3 The response of conventional (quenched and tempered) H-11 steel to strain aging as a function of prestrain temperature. The steel was pretempered at 900°F, strained 2% at a strain rate of $\sim 10^{-5} \text{ sec}^{-1}$ and posttempered at 900°F.
- Fig. 4 The response of conventional (quenched and tempered) H-11 steel to strain aging as a function of prestrain temperature. The steel was pretempered at 900°F, strained 2% at a strain rate of $\sim 10^{-3} \text{ sec}^{-1}$ and posttempered at 900°F.
- Fig. 5 The general types of stress strain curves encountered with different processing conditions.
- Fig. 6 The variation in type of stress strain curve with prestrain temperature for conventional H-11 steel at low and high strain rates.
- Fig. 7 The influence of the prestrain temperature (at low and high strain rate) on the magnitude of the yield drop of conventional H-11 steel during final testing.
- Fig. 8 The response of ausform H-11 steel to strain aging as a function of prestrain temperature. The steel was double pretempered at 500°F, strained 2% at a strain rate of $\sim 10^{-5} \text{ sec}^{-1}$, and posttempered at 900°F.
- Fig. 9 The response of ausform H-11 steel to strain aging as a function of prestrain temperature. The steel was double pretempered at 500°F, strained 2% at a strain rate of $\sim 10^{-3} \text{ sec}^{-1}$, and posttempered at 900°F.

Fig. 10 The variation in type of stress strain curve with prestrain temperature for ausform H-11 steel at low and high strain rates.

Fig. 11 The influence of the prestrain temperature (at low and high strain rate) on the magnitude of the yield drop of ausform H-11 steel during final testing.

Fig. 12 Carbide extraction replicas of conventional (quenched and tempered) H-11 steel;

- a) unstrained
- b) strained 2% at 70°F, retempered at 900°F
- c) strained 2% at 750°F, retempered at 900°F.

Magnification 12,000x.

MU-34743

Fig. 1

MUB-9953

Fig. 2

MUB-9954

Fig. 3

MUB-9955

Fig. 4

ZN-5495

Fig. 5

MUB-9956

Fig. 6

MUB-9957

Fig. 7

MUB-9958

Fig. 8

MUB-9959

Fig. 9

MUB-9960

Fig. 10

MUB-9961

Fig. 11

A

B

C

ZN-5494

Fig. 12

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