A STUDY OF THE MECHANISM OF THE DELAYED YIELD PHENOMENON

BY T. VREELAND, JR., D. S. WOOD AND D. S. CLARK

Abstract

This paper presents the results of an experimental investigation of the behavior of an annealed low carbon steel subjected to a rapidly applied constant stress and to repeated short-duration stress-pulses. The test stresses were greater than the upper yield stress. The material was aged at various temperatures between stress-pulses, and the effect of the time of aging on the number of stress-pulses to induce yielding was determined.

Plastic and anelastic microstrain of the order of $30 \times 10^{-6}$ in./in. is observed prior to the onset of yielding in rapidly applied constant stress tests and in repeated stress-pulse tests. Aging of the specimens for a sufficient length of time at a given temperature between stress-pulses induces recovery in the material such that yielding does not occur in repeated stress-pulse and aging cycles. The activation energy of the recovery process corresponds, within the limits of the experimental accuracy, to the activation energies of carbon and nitrogen diffusion in iron.

These effects are discussed in terms of the dislocation theory of yielding. The delayed yield and the microstrain are attributed to the action of dislocations within the crystals of the material. The recovery process is attributed to the diffusion of carbon and nitrogen to the dislocations which have been displaced, thus stabilizing the array of dislocations for the particular stress condition.

The dependence of yield point and strain aging phenomenon on carbon and nitrogen in steel has been studied by other investigators. Muir (1) and Davenport and Bain (2) have demonstrated that the activation energy of strain aging (return of the yield point) is almost exactly that for the diffusion of carbon in alpha iron. Several other investigators have demonstrated that the presence of the upper yield point in low carbon steel is intimately related to the presence of carbon and nitrogen (3-5). The upper yield point and strain aging phenomenon are theoretically described.

1The figures appearing in parentheses pertain to the references appended to this paper.

Of the authors, T. Vreeland, Jr., is research assistant, D. S. Wood is assistant professor of mechanical engineering, and D. S. Clark is professor of mechanical engineering, California Institute of Technology, Pasadena, Calif. Manuscript received January 2, 1952.
by the concept of dislocations and their interaction with interstitial solute atoms, such as carbon and nitrogen in the steel. Such descriptions have been given by Cottrell (6, 7), Nabarro (8), and Cottrell and Bilby (9).

Previous investigations at the California Institute of Technology (10-12) have shown that a definite period of time is required for the initiation of yielding in annealed low carbon steel subjected to rapidly applied constant stress exceeding the static upper yield stress. In those investigations, the tests were continued at constant stress until yielding occurred. By considering the concepts of the dislocation theory, one may suspect that if the material is subjected to a stress greater than the static upper yield stress and that stress is released before yielding can take place, some conditioning action occurs that may have a permanent effect on the delay characteristics of the material.

The purpose of the investigation discussed in this paper is to determine whether or not the cumulative time at stress for a series of stress-pulses is the same as the delay time required for the initiation of yielding in a single rapid-loading test. The influence of aging at different temperatures for different intervals of time between the stress-pulses is considered in this study. Sensitive strain measurements are made during the stress-pulses and during rapidly applied constant stress tests in an attempt to detect any plastic or anelastic microstrain that may occur.

**Material, Test Specimens, and Treatments**

The specimens used in this investigation were machined from 5/8-inch diameter hot-rolled bars from a single billet. Three check analyses made by the mill on random bars all gave the following results:

<table>
<thead>
<tr>
<th>Element</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>0.12</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.43</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.019</td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.042</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.27</td>
</tr>
<tr>
<td>Copper</td>
<td>0.23</td>
</tr>
<tr>
<td>Tin</td>
<td>0.037</td>
</tr>
</tbody>
</table>

A drawing of the test specimen used in this investigation is shown in Fig. 1. The gage section was finished by grinding. A thin, flat gage section was employed to facilitate the application of SR-4 strain gages.

Two groups of specimens were employed in this investigation. The first group consisted of specimens which had been annealed for 1 hour in pure dry hydrogen at a temperature of 1700 °F (930 °C). These were used for a study of plastic and anelastic microstrain prior to yielding.
The results of a previous investigation (12) indicate that a homogenizing treatment at 1300 °F (700 °C) for a long period following the annealing treatment brings the material closer to a state of perfect equilibrium and considerably reduces the scatter in the rapid-load test data. Such a treatment was given to the second group of specimens, which were used for the static tests, rapid-load tests, and stress-pulse and aging tests. The hydrogen atmosphere was purified, and a pure hydrocarbon was introduced to prevent specimen decarburization. The heat treatment was as follows:

(a) Annealed at 1680 °F (915 °C) for 2½ hours.
(b) Homogenized at 1300 °F (705 °C) for 22 hours.

The homogenization treatment did not change the grain size or hardness of the material. The ASTM grain size was observed to be 6.7, and the average of 20 hardness determinations was 54 on the Rockwell B scale. Microscopic examination of the material showed that no detectable change occurred in the amount of pearlite present in the surface of the specimen as a result of the homogenizing treatment. The fact that the hardness of the material was not changed by this treatment also indicates that the carbon content was not changed appreciably.

**Equipment**

The tests were made with the rapid-load testing machine described in a previous paper (10). Suitable alteration of a part of the actuating mechanism permits the application of a stress-pulse to the test specimen.

The extensometers described in a previous paper (11) were used for tests in which only the initiation of yield strain was to be detected. Tensile and bending strains were measured by means of SR-4 resistance-sensitive wire strain gages. One Type A-5 gage was bonded to each side of the gage section of the test specimen.

Plastic and anelastic microstrain was measured by employing a strain bar which was attached to one end of the specimen. The nominal dimensions of the gage section of the specimen and the strain bar are the same; hence, the elastic strains in the specimen and the bar are nearly equal. The strain bar is made of X4130 steel, heat-
treated so that it remains elastic under the loads used in this investigation. Two Type A-5 gages are bonded to the gage section of the strain bar and electrically connected to the two gages bonded to the gage section of the specimen in such a manner that the elastic strain in the strain bar is subtracted from the total strain in the specimen. This arrangement permits the use of the maximum possible sensitivity of the recording system without exceeding the total strain-recording capacity of the system.

The load acting on the specimen was measured by means of a dynamometer employing Type AB-14, SR-4 strain gages with suitable temperature compensation.

The signals from the strain gage bridge circuits were recorded on photographic paper by a galvanometer-type recording oscillograph. A 3000-cycle-per-second carrier bridge amplification system was used.

**Test Procedure and Experimental Results**

**Static Tension Tests**—The static tension tests were performed in the rapid-load testing machine by manual operation of the pressure system. The load was applied to the specimen in increments, and each increment was held for 3 minutes before recording values of load and tensile strain. When the specimen yielded, the load was removed and then reapplied in increments to determine the lower yield stress. The test was discontinued when the bonded strain gages came loose from the specimen at a strain of approximately 2%. Static stress-strain curves for the annealed and homogenized material are presented in Fig. 2.
Rapid-Load Tension Tests—The rapid-load tension tests which were made to determine the stress—delay time characteristics of the material were performed in the manner described in a previous paper (10). The test results are plotted in Fig. 3.

The extent of bending in the specimen during rapid-load tension tests was investigated. The maximum bending strain found in nine tests was 6.2% of the tensile strain, and the mean was 2.9%.

Repeated Stress-Pulse Tests—The tests which were employed to study the effect of stress-pulses and aging on the delay time were made by imposing a stress-pulse of essentially constant magnitude and duration on the specimen. The pulse was as follows:

(a) Stress of 45,000 ± 800 psi applied in approximately 0.007 second.
(b) Stress held essentially constant for 0.029 ± 0.001 seconds.
(c) Stress removed in approximately 0.003 second.

The delay time for the material when subjected to a stress of 45,000 psi is approximately 0.050 second, which is greater than the duration of one stress-pulse and less than the duration of two stress-pulses. Thus, the material might be expected to yield during the second stress-pulse if there is no recovery between pulses. This procedure provides a method of detecting any recovery that may occur between stress-pulses.

The specimens were aged for various intervals of time between stress-pulses at temperatures of 70, 150 and 200 °F (21, 66 and 93 °C). The procedure of aging the specimen at 150 and 200 °F (66 and 93 °C) after a stress-pulse was as follows:

(a) Specimen removed from rapid-load machine and placed in an oil bath at desired temperature within 5 minutes after stress-pulse (temperature controlled to ±1 °F).
(b) Specimen removed from oil bath after desired aging period and immediately cooled in powdered dry ice (−109 °F or −78 °C).
(c) Specimen brought to approximately 70 °F in an alcohol bath 5 minutes prior to next stress-pulse.
(d) Stress-pulse applied when specimen reached 70 °F; specimen temperature determined by thermocouple and recording potentiometer.

The initiation of yielding was determined with the extensometers in the majority of the stress-pulse tests. A typical record of a stress-pulse is shown in Fig. 4.

![Fig. 4—Typical Record of a Stress-Pulse.](image)

The cumulative time at stress before yielding is plotted in Fig. 5 as a function of aging time between stress-pulses for the three aging temperatures. The specimens which were aged at 70 °F for 3 minutes
yielded during the second stress-pulse, and the cumulative time at stress before yielding was approximately equal to the normal delay time at the same stress. Aging at 150 °F and 200 °F for periods equal to or greater than a certain critical value induced recovery from the effects of the previous stress-pulse. This is shown by the fact that yielding did not take place in successive stress-pulse and aging cycles. The data show that recovery is accomplished by aging for a minimum of approximately 12 minutes at a temperature of 200 °F and 100 minutes at 150 °F.

Measurement of Plastic and Anelastic Microstrain—A typical record of microstrain in a rapid-load test is shown in Fig. 6. A correction must be applied to the data obtained from this record in order to obtain values of microstrain. This correction is made necessary by an imperfect balance between the elastic strain in the specimen and the strain bar. The sources of the inequality of elastic strain are:

(a) A difference in cross-sectional areas of the strain bar and the specimen.
(b) A difference in sensitivities of the gages on the strain bar and the specimen.
(c) Misalignment between specimen and strain bar at the threaded connection which can result in a difference of the bending moment in the specimen and in the strain bar. This will result in bending strain inequality.
(d) A difference in Young's modulus between the X4130 strain bar and the test specimen.

These sources of elastic strain inequality do not produce any change in the indicated strain while the stress remains constant. Therefore, the changes of indicated strain which occur during the period of substantially constant stress may be attributed to plastic and anelastic microstrain in the specimen. The microstrain shown in the records is indicated by a downward deflection of the trace, whereas an unbalanced elastic strain may deflect the trace in either direction. The
strain which is indicated on the record during the period of stress rise is subtracted from the total indicated strain; thus, any microstrain occurring during the period of stress rise is neglected.

Anelastic and plastic strains which may occur in the strain bar also introduce a subtractive error. Hence, the measured microstrain is always less than the true plastic and anelastic microstrain in the specimen. Although it is difficult to make a reliable estimate of the total error due to these effects, there is some justification for believing that it is small. First, the strain correction for elastic unbalance is normally a small portion of the indicated microstrain during the entire record. Hence, the microstrain which may occur during the period of stress rise is small compared to the total microstrain. Second, the stress in the strain bar is approximately one-fourth the value of the yield stress for this material; therefore, the anelastic and plastic strain in it should be negligible.

A mean value of microstrain of $30 \times 10^{-6}$ in./in. was found to take place prior to yielding in ten tests at various stresses. Although the microstrain varied from $20 \times 10^{-6}$ in./in. to $37 \times 10^{-6}$ in./in. in these tests, there is no systematic correlation with the magnitude of the stress. The microstrain rate during each test decreases with time until yielding starts. A recovery of approximately 50% of the microstrain was observed when the stress was removed. Successive stress-pulse and aging cycles showed decreasing amounts of microstrain in each stress-pulse cycle. When the aging conditions induced recovery, the decrease in microstrain for successive stress-pulse aging cycles was greater.

The distribution of the microstrain over the specimen gage length was investigated. The difference in strain between two regions of the gage section was measured by means of four Type A-8, SR-4 strain gages. Two gages were bonded to each region, one on each side of the gage section, and the four gages were electrically connected so as to measure the strain difference between the two regions. These strain gages have a gage length of $\frac{3}{8}$ inch, and the specimen gage length is $\frac{1}{2}$ inch. The specimen was subjected to a rapid load, and the resulting test record indicates no inhomogeneity in strain prior to the initiation of yielding. A second indication of the distribution of the microstrain was obtained in the following manner: The surface of a test specimen was mechanically polished, etched, and examined microscopically at magnifications of 100 and 600 diameters between successive stress-pulses in an attempt to detect the formation of slip lines. No slip lines were observed until yielding took place during the third stress-pulse. Slip lines were then observed within the single Luders' band which had formed.

Tests on Material Previously Subjected to Stress-Pulse and Aging Cycles—The influence of stress-pulse and aging cycles on the
static stress-strain relationship and on the stress–delay time relationship was investigated. Tests were conducted on specimens which had been previously subjected to stress-pulses followed by sufficient aging to produce recovery. Hence, none of these specimens had previously yielded.

The static upper and lower yield stress and the yield strain are not changed significantly by the stress-pulse and aging treatment. The results of the stress–delay time determinations are plotted in Fig. 3. These tests show that the delay times are greater by a factor of approximately 4 than the delay times for the original material for corresponding stresses.

**Discussion of Results**

The relations between delay time and stress for the annealed and homogenized material are given in Fig. 3. The scatter in the data was found to be reduced by the homogenizing treatment. This treatment also produced changes in the stress–delay time relationship which was previously reported for the original annealed material (12). The reduced scatter and the changes in the stress–delay time relationship can be attributed to a closer approach to the equilibrium state in the annealed and homogenized material. The amount of bending found in the rapid-load tensile tests is sufficient to introduce the scatter found in the stress–delay time determinations of the annealed and homogenized material.

The results of the repeated stress-pulse tests, presented in Fig. 5, show that a definite recovery from the effects of the previous stress-pulse takes place when the time and temperature between stress-pulses is sufficient. An activation energy for the recovery process can be found by assuming that the recovery follows a law of the form

\[ t \sim e^{Q/RT}, \]

where \( t \) is the critical aging time for recovery, \( Q \) is the activation energy of the recovery process, \( R \) is the gas constant, and \( T \) is the absolute aging temperature.

The recovery times of 12 minutes at 200 °F and 100 minutes at 150 °F give an activation energy of 18,800 cal/mole for the recovery process. There is an uncertainty of 10% in this value due to scatter in the critical aging time and the fluctuation of the aging temperature. This activation energy corresponds to the activation energy of strain aging (1, 2, 8), (18,100 cal/mole), and to the activation energy of the diffusion of carbon and nitrogen in alpha iron [carbon: 18,100 cal/mole (13), 19,800 cal/mole (14); nitrogen: 17,700 cal/mole (14)]. Thus, it is reasonable to assume that the mechanism of recovery from previous stress-pulses is related to the mechanism of the
return of the yield point and to the diffusion of carbon and nitrogen in the steel.

A microstrain of approximately \(30 \times 10^{-6}\) in./in. was measured prior to yielding for both rapid loading and repeated stress-pulses. The microstrain appears to be independent of the applied stress within the range of stress covered in this investigation. This value of microstrain agrees in order of magnitude with that found by Averbach (15) (50 to \(60 \times 10^{-6}\) in./in.) in static tests on polycrystalline samples of iron and steel.

Cottrell (7) has recently presented some promising additions to the previous concept of the mechanism for the initiation of yielding in polycrystalline low carbon steel. He extends the previous theory of dislocations anchored by interstitial solute atoms to include the following concepts:

(a) Releasing a dislocation from a grain boundary requires a larger force than releasing an anchored dislocation within a crystal.

(b) A few dislocations are released prior to yielding, and their movement is obstructed by interaction with other nearby anchored dislocations or grain boundaries. Yielding is not initiated until the resistance offered by obstructions to the movement of dislocations is overcome.

An anchored dislocation within a crystal may move when its total energy is greater than the potential energy of the anchoring barrier. The potential energy of the barriers changes with the applied stress. For a given stress, the number of dislocations which can move in a given time is determined by the statistics of the fluctuations in thermal energy of the dislocations. Therefore, a definite period of time would be required for a particular stress to release a given number of dislocations. When a sufficient number of dislocations are released and move to an opposing obstruction, they may overcome the restraint offered by the obstruction and initiate an avalanche of dislocations which results in yielding. The delayed yield phenomenon may be explained by this mechanism. Furthermore, the relatively constant value of microstrain observed before yielding in this investigation can also be qualitatively explained by these concepts.

No inhomogeneity in microstrain was found prior to yielding. This indicates that the microstrain does not result from the progressive formation of the Luders' band which appears on yielding. For a quantitative treatment of the dislocation theory of the observed microstrain, the number of dislocations which can be moved or the distance which these dislocations can move before being stopped by obstructions must be assumed. If the density of dislocations which are moved is \(p\) per unit area and if the mean distance which the dislocations move is \(L\), the movement of the dislocations causes a tensile strain of about \(pL\lambda_0/2\), where \(\lambda_0\) is the magnitude of the dislocation slip vector. If the obstacles which stop the movement of a dislocation within the
crystal are assumed to be the mosaic boundaries with a spacing of about \( L = 10^{-4} \) cm \((18)\), and taking \( \lambda_0 = 2.5 \times 10^{-8} \) cm for alpha iron, then by the above formula, between \( 10^7 \) and \( 10^8 \) dislocations per cm\(^2\) must be moved to produce a tensile strain of \( 30 \times 10^{-6} \) in./in.

When the stress is rapidly removed before the initiation of yielding, approximately 50\% of the microstrain is recovered. This indicates that some of the dislocations which were displaced by the applied stress return toward their original positions when the stress is removed, as might be expected.

The microstrain rate, prior to the initiation of yielding, decreases with time. This might be accounted for by a depletion of the reservoir of dislocations which may be moved by the applied stress. This may also account for the decreasing amount of microstrain induced by successive stress-pulses when recovery does not take place. When the aging treatment between stress-pulses induces recovery, a greater decrease in the microstrain in successive stress-pulses is found. The recovery mechanism may be explained by the diffusion of carbon and nitrogen to the dislocations which have been displaced. The resulting array of dislocations, anchored by atmospheres of carbon and nitrogen, may be expected to be more stable than the original array under the particular stress condition. Successive stress-pulse and aging cycles then produce less microstrain, and yielding does not take place. The more stable configuration of dislocations for the particular stress condition is also indicated by the results of the rapid-load tensile tests on the material which had previously been subjected to stress-pulse and aging cycles in which recovery took place. The delay times for the initiation of yielding are increased by a factor of approximately four over the delay times for the original material for corresponding stresses.

**Summary and Conclusions**

The results of the repeated stress-pulse tests on annealed low carbon steel show that a definite recovery from the effects of the previous stress-pulse takes place when the combination of time and temperature between stress-pulses is sufficient. For aging periods longer than the critical recovery time at a given aging temperature, repeated stress-pulse and aging cycles do not produce yielding. For aging periods shorter than the critical recovery time, the specimen yields when the cumulative time at stress is approximately equal to the normal delay time. This recovery process is found to be associated with an activation energy corresponding to the activation energy of strain aging and diffusion of carbon and nitrogen in the steel. The repeated stress-pulse and aging treatment stabilizes the material such that the delay times are increased for the same stress.

Sensitive strain measurements under conditions of rapid loading
and stress-pulses indicate that a plastic and anelastic microstrain of approximately $30 \times 10^{-6}$ in./in. precedes the yield strain. The delayed yield and the microstrain may be attributed to the action of dislocations within the crystals.

The recovery phenomenon and the increase in delay times for the previously stressed and aged material may be attributed to the diffusion of carbon and nitrogen to the dislocations which have been displaced, thus stabilizing the array of dislocations for the particular stress condition.

**Acknowledgments**

This investigation was conducted under the sponsorship of the Office of Naval Research. The rapid-load testing machine used in this investigation was constructed by the California Institute of Technology under a contract with the United States Air Force. Appreciation is expressed to the U. S. Air Force for permission to use the machine.

**References**


