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An Analysis of Burst Disc Pressure Instability

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An Analysis of Burst Disc Pressure Instability

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Abstract

During the development stage of the 1X Acorn burst disc, burst pressure test results exhibited an unexpected increase of 8 to 14% over times of 90-100 days from initial fabrication. This increase is a concern where design constraints require stability. The disc material, 316L stainless steel sheet, is formed to a dome-like geometry and scored to produce a thin-walled, high-strength ligament. The fracture events controlling burst occur in that ligament. Thus it has been characterized both for tensile properties and microstructure through nanoindentation, magnetic measurements, optical and transmission electron microscopy. These results compare favorably with finite element simulation of the properties of the ligament.

The ligament exhibits a highly heterogeneous microstructure; its small volume and microstructural heterogeneity make it difficult to identify which microstructural feature controls fracture and hence burst pressure. Bulk mechanical test specimens were fabricated to emulate mid-ligament properties, and aged at both room and elevated temperatures to characterize and accelerate the temporal behavior of the burst disc. Property changes included yield and ultimate tensile strength increases, and fracture strain decreases with aging. Specimens were subjected to a reversion anneal identical to that given the burst disc to eliminate the martensite phase formed during rolling. Reversion-annealed samples exhibited no change in properties in room temperature or accelerated aging, showing that the reversion-anneal eliminated the aging phenomenon. Aging was analyzed in terms of diffusion controlled precipitate growth kinetics, showing that carbon migration to dislocations is consistent with the strength increases. A

vacancy-assisted diffusion mechanism for carbon transport is proposed, giving rise to rapid aging, which replaces interstitial carbon diffusion until excess vacancies from deformation are consumed. Mechanical activation parameters in stress relaxation were measured, indicating that the deformation structures formed at high strains typical of the score ligament are resistant to annealing, and mimick the behavior of athermal obstacles. This model also qualitatively explains the different rates of aging resulting from a range of levels of cold work.

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An Analysis of Burst Disc Pressure Instability

Introduction

The burst disc under consideration, shown in Figure 1, is a subassembly of the 1-X Acorn. Early development parts exhibited a burst pressure instability in which the burst pressure rose quickly in the first weeks following forming, and asymptotically approached a burst pressure some 8-14% higher than that measured immediately after forming. The presence of martensite in the 316L stainless steel disc was also identified in the region of the score ligament, by magnetic and by metallographic means. The presence of martensite in 316L is considered unusual since 316L is usually resistant to deformation-induced phase changes. A 732°C partial anneal was developed to revert the martensite to austenite, in order to improve hydrogen compatibility. As-formed discs were then subjected to this reversion-anneal immediately following forming. Subsequently, it was observed that the time-dependent burst pressure increase (referred to as aging) ceased, based on a six month observation. A long-term issue to be addressed is then whether the aging phenomenon has been conclusively stopped by the reversion anneal. Identification and description of the mechanism(s) responsible for the aging phenomenon is crucial to gaining confidence in the long-term stability of the burst pressure.

In a previous study¹ it was observed that the burst disc ligament immediately below the score mark controlled fracture, and hence the burst pressure, of the disc. When the disc is functioned, stable ductile rupture begins at the back or unscored surface¹, transitioning to an unstable shear fracture at a depth of roughly 10-15 μm . The shear fracture then rapidly propagates inward from the ductile fracture initiation site to the root of the score region. Simultaneously, the fracture moves rapidly outward along two fronts following the score, and terminating at the ends of the score.

The strength of the ligament must increase for an increase in burst pressure to occur. Experimentally we observe that, as the burst pressure increases, the dome (marked "disk" in Figure 1) grows plastically to a greater extent prior to the fracture event. The dome growth introduces work hardening in the dome, which, once strong enough, transfers load and deformation to the score ligament. This process implies a higher strength in the ligament¹.

The source of the strength increase with time is not immediately obvious, in that stainless steels such as low interstitial 316L stainless steel are not commonly considered to exhibit either thermal or strain aging at room temperature. We note, though, that the degree of deformation within the score ligament is quite intense and therefore, the aging response

¹ Memo, S.L. Robinson to Distribution, October 1, 1998, "Analysis of Burst Disc Failure Modes".

Figure 1. Burst disc assembly, metallographic cross section of burst disc score, and schematic of fracture cross-section.

of the alloy may be atypical. Data from Meyer² indicate the presence of a time-dependent burst pressure increase suggestive of strain-aging. Strain aging of austenite is now established as a known phenomenon³⁻⁶, although typically it is not observed in technological alloys, with the exception of 316 and AISI 330⁴. Rose and Glover³ examined iron-nickel-carbon alloys, and observed yield point phenomena in which the magnitude of the yield point, observed upon reloading from stress relaxation, was dependent upon prior strain. Carbon was found essential to the process. Jenkins and Smith⁴ observed serrated flow, evidence of a Portevin-LeChatelier effect in Fe/35Ni, and in type 330 stainless steel. In the temperature range 200-500°C, carbon was believed responsible for the interactions between solute atoms and dislocations. Hannula, Korhonen and Li⁵ observed yield point phenomena following prestraining and stress relaxation at room temperature in type 316 stainless steel. They attribute the effect to carbon-vacancy complexes interacting with dislocations, as did the previous authors, assuming that higher mobility would result from such a complex than for carbon alone. Their carbon concentration was 0.07w/o whereas the 316L under consideration has less than 0.03w/o carbon, raising the question of relevance to the behavior of 316L. Nevertheless, an extrapolation of their yield point results to conditions of prestrain and hold times typical of the burst disc predicts a strength increment similar to the lower bound of the observed burst pressure increase, that is, about an 8% increment. This similarity has lent support to the idea that the disc is experiencing strain aging. Ulitchney and Gibala⁶ explored internal friction and serrated flow behavior over the interval of 25°C to 400°C, in Fe/30Ni, Fe/36Ni, Invar, and 310 stainless steel. They correlated the presence of bound peak pair-caused (BPP) internal friction peaks (C-vacancy pairs) with jerky or serrated flow in plastic deformation, and demonstrated that not only is carbon essential to the process, but excess vacancy introduction enhances the BPP internal friction peak. The internal friction peak maximizes at about 250°C. From their results it may be inferred that at carbon concentrations typical of 316L, only extremely heavily worked structures of high dislocation density and thus high vacancy concentrations introduced by the deformation should exhibit BPP-derived internal friction, serrated flow, or strain aging. The necessary high strain deformation conditions may exist in the rupture disc score region. Thus, by introducing an extreme deformation state in the fracture zone of the disc (the score ligament), we may have introduced both strain-induced martensite and strain aging in a material that does not commonly exhibit either.

The fracture controlling zone of the burst disc is that region beginning approximately 10 μm (about 20% of the ligament thickness) below the outside or unscored surface of the disc and opposite the apex of the score. This region is very heterogeneous, exhibiting large gradients in cold work, in hardness and therefore in tensile properties. In addition, within this region the fracture transitions from a stable, ductile fracture mechanism to a

² B. Meyer, LANL, unpublished data, 1998.

³ K.S.B. Rose, S.G. Glover, "A Study of Strain-Aging in Austenite", *Acta Metall.* **14**, p. 1505, 1966.

⁴ C.F. Jenkins and G.V. Smith, "Serrated Plastic Flow in Austenitic Stainless Steels", *Trans. Met. Soc. Of AIME*, **245**, p. 2149, 1969.

⁵ S.P. Hannula, M.A. Korhonen, C.Y. Li, "Strain Aging and Load Relaxation Behavior of Type 316 Stainless Steel at Room Temperature, *Metall. Trans.A*, **17A**, p. 1757, 1986.

⁶ M.G. Ulitchney, R. Gibala, "Internal Friction and Strain Aging of Ferrous Austenite", *Metall. Trans.* **4**, p. 497, 1973.

high velocity shear fracture. In order to examine aging behaviors and their relationship to microstructure, it is desirable to emulate the appropriate (fracture-controlling) ligament microstructure in macroscopic specimens, which are more amenable to conventional examination. First, it is necessary to characterize the fracture zone. In the following, we describe the results of efforts to characterize the strength, microstructure and properties of the burst disc. These efforts will allow us to design an emulation technique for producing useful specimen geometries with appropriate properties. The specimens may then be examined for their aging response in real-time and accelerated aging modes.

Characterization of Burst Disc Microstructures

Finite element simulation of the formation of burst discs shows true strain levels up to 2.2 in the scored region⁷. Figure 2 shows these strain contours as computed by Chiesa⁷. The associated flow stress at a true strain of 1.4 (the midline of the ligament) is of the order of 1300 MPa (190 ksi). Magnetic examination of the back surface of the score region was conducted using a Foerster® "ferritegag". Readings of 0.2-0.3% were obtained (readings away from the score were zero); these values are uncalibrated since the sensor interrogates a 3 mm diameter volume in front of the tip; calibration requires a minimum thickness of 3 mm. We may approximate a correction factor based upon volume considerations. Using the affected volume of the foil divided by the interrogated volume of the instrument, we arrive at an approximate ratio of 1/30. Therefore a reading of approximately 0.20 to 0.33% would equate to 6 to 10% magnetic phase in the small volume. This is consistent with metallographic observations, although local concentrations might exceed 10% martensite.

Figure 2. Computed strain contours from Chiesa⁷ for the as-formed burst disc score. The midline is midway between the score root and the back surface.

⁷ M. Chiesa, Org. 8727, Sandia National Laboratories, to be published.

The strength and hardness distribution within the score region were characterized using nanoindentation. An example is shown below in Figure 3 for an intermediate anneal of 704°C (1300°F) / 0.75 Hr. The example is that of the annealed disc, in which flow stresses have been fully corrected for indentation size effects. Midline flow stresses of up to 1.4 GPa (203 ksi) are measured directly below the notch root⁸, in good agreement with the midline strain contour and stress computed by Chiesa⁷.

Figure 3. Nanoindentation results for flow stress beneath the score mark in a burst disc annealed at 704°C (1300°F) / 0.75 hour. The trace is situated midway between the score root and back surface of the disc.

Transmission electron microscopy of thin foils was attempted, in order to image the dislocation structures in burst discs. As anticipated, foil preparation to view the score ligament was difficult, and no useful results were obtained.

In designing an emulation of the burst disc microstructure, we consider both flow stress and microstructure to be important. Magnetic moment, as a measure of transformation product, is an important aspect of that simulation. Schrinivas et al⁹ have shown that rolling of 316 is capable of producing martensite, the magnetic phase transformation product, although uniaxial tensile tests and other deformation processes do not typically induce transformation. It is necessary to introduce plastic deformation in three-dimensional space including rotations, for martensite to form in 316. We experimented with other techniques, including channel die-extrusion, and scale-up of the indenter and specimen size, and found only small (<1%) amounts of magnetic product. Therefore we designed a rolling procedure to produce specimens suitable for diagnostic experiments,

⁸ N.R. Moody, Org. 8725, Sandia National Laboratories, to be published.

⁹ V. Shrinivas, S.K. Varma and L.E. Murr, "Deformation-induced Martensitic Characteristics in 304 and 316 Stainless Steels During Room-Temperature Rolling, Metall. Trans. A, 26A, P. 661, 1995.

representing specific parts of the ligament region shown in Figure 2. These specimens are discussed under Experimental Procedures.

The full-scale specimens were intended for several analyses, including accelerated aging studies, and studies directed toward identifying thermally activated processes. The accelerated aging experiments would explore low temperature aging up to and including exposure, in vacuum, to a temperature of 250°C. Activation volume measurements, based upon the methods of Feltham et al¹⁰, were planned in order to determine the controlling deformation mechanism(s) in the emulated microstructure. Specimens are strained beyond yield to the flow stress regime and stress relaxation is then allowed to occur as total strain and temperature are held constant. The relaxations are then analyzed according to the relationship

$$V^* = - kT d\ln(t+t_0) / d\tau \quad (1)$$

In which V^* is the activation volume, k is Boltzmann's constant, T is the temperature (K), τ is the shear stress, t is the time of relaxation and t_0 , an integration constant, is taken¹⁰ as one second. Often, tensile stress (σ) is substituted in place of shear stress (τ). It is customary to obtain the area of the activated event through the relation $V^* = Ab$ in which b is the Burger's vector and A is the area swept out by the activated event. Alternatively, $V^* = b^2L$ in which the activated event is one Burger's vector in width and L in length which may then be computed. It is now common to graphically present the results in the form b^3/V^* vs. stress¹¹. Stress relaxation studies are the tool of choice in this study, since the available ductility, as will be seen, does not permit rate change experiments. The stress is taken as the instantaneous stress during relaxation and V^* is calculated from (1) using the slope at that stress during the relaxation. It is possible to discriminate deformation mechanisms according to the stress intercept at $b^3/V^* = 0$. For example, an intercept less than zero is associated with a frictional mechanism, an intercept of zero with dislocation intersection with dislocation multiplication and bypass, and an intercept greater than zero with the presence of athermal obstacles¹¹.

Experimental Procedures

A 15 cm diameter bar of 316L steel, hot finished, was sectioned first into plates 0.5, 1 and 1.25 cm thick and 25 cm long. The final rolled thickness was 0.025 cm, giving thickness reductions of 50, 75 and 80%. This produced true strains of $\epsilon = -0.7$, -1.4 , and -1.6 . Room temperature, multi-pass rolling was performed in order to reduce the plates to final thickness. Quenching between passes minimized temperature increases. The composition of that bar is shown in Table I. Specimens were then machined to final shape by Electro-Discharge Machining.

¹⁰ P. Feltham, G. Lehmann, R. Moisel, "Stress Relaxation in Nickel", *Acta Metall.* **17**, p. 1305, 1969.

¹¹ N.R. Moody, S.L. Robinson, J.E. Angelo, M.W. Perra, "Modeling the Effects of Temperature on Hydrogen-Induced Cracking in an Austenitic Superalloy", *Proceedings of the 4th Greek National Congress on Mechanics*, Vol 1, 310 (1995).

Table I. Composition of the 316L bar stock in wt. %.

Cr	Ni	Mn	Mo	Si	C	N	S	P
17.40	10.41	1.76	2.16	0.63	0.020	0.085	0.027	0.026

Metallographic sampling and hardness measurements confirmed that the microstructure of the hot-finished 15 cm diameter bar was uniform from center to edge. The carbon and nitrogen values are from LECO® analyses.

Mechanical Testing and Accelerated Aging

Testing was performed on a SATEC servomechanical test frame. Mechanically attached extensometers were used to both measure and control specimen strain. The Satec test frame is configured with a digital data acquisition and control system that can be programmed to execute both the monotonic tensile tests and the strain controlled relaxation tests that comprised this experimental program. In the latter case strain control on the order of $\pm 1 \mu\text{strain}$ was achieved with the use of a high-resolution extensometer.

Aging effects on the room temperature mechanical properties of the cold-rolled stainless steel were measured through a series of monotonic uniaxial tensile tests. Test specimens were flat “dogbone” tensile bars having 2.54 cm long gage sections that measured 0.635 cm in width and 0.25 cm in thickness. All specimens were instrumented with an attached 2.54 cm +50%, -10% extensometer for strain measurement. The mechanical properties tests were conducted at an initial strain rate of 0.02 sec^{-1} . Relaxation experiments were conducted with specimens of the same geometry but with a $2.54 \pm 0.0635 \text{ cm}$ extensometer for more precise strain control. Thermoelastic cooling was observed upon tensile straining. Therefore, in order to meet isothermal test requirements, data taken in the first few seconds after the crosshead was stopped were not used.

In addition to room temperature testing, a series of elevated temperature accelerated aging tests were performed. The matrix of tests is shown in Table II. Following hold times (time 1 and time 2) at the indicated temperatures, specimens were cooled to room temperature and tested.

Table II. Accelerated aging studies test matrix.

Rolling Reduction	Temperature, °C	Time 1	Time 2
75%	150	14 days	28 days
75%	250	14 days	28 days
75%	732 1 Hr/250	14 days	28 days

Results

Not all rolled materials were suitable for testing. The 50% cold-rolled material exhibited low strength ($< 1030 \text{ MPa}$ yield strength) and no significant martensite formation. The

80% cold-rolled material began to show edge cracking during rolling, raising concerns that specimens would be vulnerable to defects. The 75% cold-rolled material was found to be the most satisfactory material to test. The martensite concentration was slightly below that of the actual burst disc score zone, e.g. only about 4-5% martensite was formed, compared to 6-10% in the score zone. [This result conflicts with the results of Shrinivas et al⁹ who obtained more than 30% martensite concentration at similar strains. The difference is probably due to the much higher carbon concentration in 316 of 0.08wt.% compared to less than 0.03 in 316L.] Nevertheless, the desired yield strength was obtained.

Mechanical Testing and Thermal Aging

The principal objectives of these tests were twofold. The first was to assess the influence of low temperature thermal aging on the as-rolled properties (specifically, strength and ductility) of the alloy. The second objective was to determine if the martensite reversion treatment reduced the susceptibility of the alloy to any thermal aging-induced changes in mechanical properties.

Since we expected to observe only subtle changes in the measured properties of the alloy it was important to determine the general reproducibility of the data. To that end, the stress-strain behavior for four specimens was determined in the as-rolled condition as shown in Figure 4. The three replicate tests reveal the good uniformity in cold work imparted to the alloy during rolling. Subtle yield drops are suggested by the data, although they are not visible at the resolution of the figure.

The results for 4 specimens tested in the 75% cold-rolled condition are summarized in Table III. Yield and ultimate strengths are reproducible to better than 1%. Strain measurements are also quite reproducible.

Table III. Tensile properties of replicate as-rolled specimen cold rolled to 75 thickness reduction %.

Sample #	Yield (MPa)	UTS (MPa)	Unif.Strain (%)	Frac. Strain (%)
1	1197	1302	2.16	12.34
2	1181	1303	2.13	12.51
3	1181	1291	2.28	12.62
4	1188	1301	2.23	12.34
Avg.	1187	1299	2.20	12.43
Std. Dev.	±7.6	±5.5	±0.07	±0.14

Aging the alloy at 250°C for 14 and 28 days after cold-rolling to 75% has a significant effect on both strength and ductility, as shown in Figure 5. The UTS of the alloy increased by approximately 10% (1299 MPa to about 1462 MPa after 14 days at temperature) while uniform strain (strain to UTS, the onset of plastic instability) and overall ductility decreased. After 14 days at 250°C, uniform strain fell by nearly 25% from 2.2% to 1.5% while strain to failure decreased from about 12.4% to about 8.5%.

Figure 4. Tensile behavior of rolled stainless steel specimens.

Figure 5. Effect of 250°C thermal age on the stress-strain curves for 75% cold-rolled stainless steel.

Only a small additional effect on mechanical properties was observed after 28 days at 250°C, suggesting a saturation of the thermal aging effect.

Room temperature aging of burst discs is more rapid than that observed in experimentally simulated midline material (at $\epsilon = -1.4$, identified in Figure 2 directly below the root of the score). Actual burst discs experience approximately 60% of their total burst strength increase in 60 days at room temperature, while the rolled midline-equivalent material experiences about a 10% (17.2 MPa) increase at room temperature. The reasons for this slow aging effect are discussed later.

As noted previously, the rolled specimens are intended to simulate the behavior of the score ligament, both in the as-rolled condition and following the aging schedule. The percentage strength increase observed in the rolled specimens is lower and develops more slowly than in the burst discs, but is consistent with the burst pressure increase observed after extended aging. Thus it is taken to simulate that behavior.

As the strength increase asymptotically approaching a limiting value, shown in Figure 6, the fracture strain approaches an asymptotic lower bound, as shown in Figure 7. Following aging at 150°C, strength and ductility of the worked alloy were affected to a lesser degree than at 250°C. Figure 8 shows the stress-strain behavior of the 75% cold-worked alloy after aging 15 days at both 150°C and 250°C. These curves are compared to a representative as-rolled curve. These tests reveal that the as-rolled stainless steel is thermally unstable at temperatures as low as $0.25 T_{MP}$. This result implies that the time-dependent burst pressure increase is derived from the same phenomena observed in the rolled specimens. Furthermore, at elevated temperature, short times are required to produce significant changes in properties; near room temperature, the time necessary for observable changes is greater, indicating that the mechanism is thermally activated.

The last series of tests were undertaken to test whether the thermal instability was also present in rolled specimens that had been subjected to the martensite reversion-anneal (732°C/1 hr). Figure 9 shows the tensile behavior of the 316L cold-rolled material after the 732°C reversion-anneal (duplicate specimens). The properties of the alloy are quite reproducible. As expected, the heat treatment results in a decrease in strength and an increase in ductility compared to the as-rolled specimens.

The effect of the 250°C/15 day aging treatment on the properties of a reversion-annealed specimen is shown in Figure 10. Unlike the as-rolled specimens, the aging treatment has no appreciable effect on ductility and strength; the strength may be even slightly reduced. The thermal instability exhibited by the as-rolled material is eliminated by the reversion-anneal. As such the alloy no longer is susceptible to the time or temperature induced changes in mechanical properties, within the boundaries of time and temperature examined.

A sample room temperature stress relaxation curve is shown in Figure 11. Note that maintaining stable temperature is difficult due to thermoelastic cooling; accordingly, data was only used when temperature was constant within 0.5°C. Activation volume

Figure 6. Evolution in strength with aging time at 250°C.

Figure 7. Strain to onset of plastic instability increases with increasing aging time.

Figure 8. Influence of aging temperature on mechanical properties of 75% cold-worked stainless steel.

Figure 9. Tensile properties of reversion annealed cold worked stainless steel.

Figure 10. Tensile properties of reversion annealed stainless steel are unaffected by thermal aging.

Figure 11. A sample stress relaxation curve for 75% cold rolled stainless steel.

measurements are shown in Figure 12A for 50 and 75% cold rolled 316L, and for reversion-annealed 75% rolled material. The 75% cold rolled state exhibits no significant difference in slope (b^3/V^* vs stress), or stress axis intercept of b^3/V^* at $b^3/V^* = 0$, when tested after one week of aging. The offset in the stress axis intercept between as-formed and reversion-annealed material may be due to the elimination of the martensite. Pre- and post-anneal specimens appear to behave similarly to IN903 (Figure 12B), that is, an intercept greater than zero is obtained at $b^3/V^* = 0$.

Discussion

Assuming that we have adequately emulated the microstructural state of the midline of the ligament, these results lead to the practical conclusion that the burst disc will not exhibit further aging after experiencing the reversion-anneal. Limitations to the microstructural emulation include 1) a slightly lower volume fraction of martensite in the rolled product, 2) a reduced rate of room temperature aging compared to the burst disc and 3) differences in the strain rates and total strain between forming the burst disc ligament and the rate of deformation imposed during cold-rolling of the tensile specimens. In order to understand the roles played by these factors, it is important to clarify the mechanisms controlling deformation and aging rate. In the following we discuss the deformation mechanisms.

Most authors cited³⁻⁶ have demonstrated or assumed that carbon is crucial to the aging process. We have shown, on the basis of yield drops and reaction kinetics that the effects observed in this study are consistent with the diffusion of carbon to dislocations, there either to precipitate or to form atmospheres in the dislocation strain field. Our hypothesis concerning the source of the aging effects is therefore straightforward, and builds upon previous studies of strain aging in stainless steels. First we summarize the observations and the phenomena that a model must describe. We observe that extreme states of deformation are required to observe aging in low carbon stainless steels, in agreement with previous investigators³⁻⁶. The deformation sensitivity is such that a high strain microstructure ($\epsilon=1.4$) appears to age while a microstructure formed at lower strain ($\epsilon=0.7$) seems not to age at all. Aging appears to be thermally activated, and occurs at appreciable rates at or near room temperature, thus requiring a low activation enthalpy in any model proposed. Furthermore, the extent of the yield strength increase appears to saturate, and low temperature (732°C) annealing not only halts this increase but reverts the yield strength to approximately its original value. Finally, the activation volume behavior appears to be athermal in nature, arising from an intensely cold worked structure, rather than from a precipitation strengthened microstructure.

Analysis of the Activation Energy

Since the strength increase is thermally activated, measurement of the temperature sensitivity of the event is needed to identify the controlling mechanisms. Fitting an Arrhenius rate equation to the ultimate tensile strength data of Figure 8 shows that $\Delta H = 18$ kJ/mole (4.3 kcal/mole) for the 75% cold rolled 316L with a goodness of fit (R^2) of 0.995. Ulitchney and Gibala⁶ cite ΔH_{SD} for interstitial carbon self diffusion in austenitic

Figure 12. A Reciprocal activation volume normalized to b^3 as a function of flow stress for as-rolled and reversion annealed 316L stainless steel. B. Activation volumes for other alloys are shown for comparison [from Moody et al¹¹] at small values of b^3/V .

steels as 141.4 kJ/mole (34 ± 5 kcal/mole), indicating that interstitial carbon diffusion is not the rate controlling event for aging. The low value of ΔH explains how the aging phenomenon can occur at room temperature; however it fits no known single diffusing species. The lower ΔH for aging vs. self-diffusion may be due to vacancy complexing, as discussed by a number of authors³⁻⁶. A detailed analysis of the temperature sensitivities of interstitial carbon self-diffusion and carbon vacancy complex diffusion in the presence of excess (non-thermal) vacancies, arising from intense cold work, may be found in the appendix.

Analysis of the Kinetics of the Rate of Strength Increase

An analysis of aging kinetics based upon the kinetics of diffusion controlled growth allows a measure of the degree of acceleration obtained by the elevated temperature aging. Furthermore, it demonstrates why aging rates differ exponentially between microstructures, such that some age rapidly while others appear stable. We assume that carbon is diffusing to dislocations, and that the dislocations are spaced uniformly. The form of the deformation obstacle which carbon takes upon arrival at a dislocation need not be specified in this analysis, since diffusion is rate controlling. The theory of the kinetics of diffusion controlled growth describes kinetic phenomena in the form

$$X = 1 - \exp(-(t/\tau)^m) \quad (2)$$

where X is the extent of completion of the reaction with bounds of 0 and 1, t is the time, τ is a characteristic time at which 0.63 ($1 - e^{-1}$) of the reaction is complete, and m describes the order of the mechanism. For example, this expression is also useful in describing the growth of second phases. For X the degree of completion of the reaction, we use in this case the fraction of the total strength increment developed at time t . Using the 250°C data (the ultimate tensile strength) of Figure 6, and plotting $\ln \ln (1/(1-x))$ against $\ln t$ gives $m = 1.02$. Although only three data points are available, the goodness of fit is 0.999.

Ham¹² showed that $m=1$ describes the long-term collection of atoms by dislocations, when tetragonality of defects is considered. An exponent of 1 is also appropriate to the growth of needle-shaped precipitates¹³. These results are consistent with the previous thermal activation data, indicating that the strength increase is controlled by the diffusion of carbon atoms to dislocations. The result is consistent with carbon precipitation on dislocations¹². Thus the hypothesis of carbon transport to dislocations is supported by the agreement between theoretical and observed kinetics of the rate of strength increase. The burst disc data of Meyer² could not be analyzed in this framework due to the heterogeneous nature of burst disc microstructures.

Further discussion of the mechanisms mentioned above may be found in the appendices.

Limitations of the Study

A limitation in this study is that the multipass-rolled microstructure may differ in fundamental ways from the scored disc microstructure. The disc score is formed rapidly at room temperature, in about one second, followed by a hold for 10-15 seconds. The rolling process required about 30 minutes of multipass rolling, with repeated water baths to minimize temperature rise to the 40-50°C range. Aging and recovery are likely to have occurred in that 30-minute interval. Thus the zero time measurements in the emulated microstructure may already include some pre-aging (at slightly elevated temperature), and the kinetic phenomena may be altered. Pre-aging would be expected to reduce the room temperature response, to have some effect on the accelerated aging response, and potentially to alter the activation enthalpy. Differences in aging rates attributable to dislocation structures formed at high rates versus low rates are discussed in Appendix D.

¹² F. S. Ham, "Stress-Assisted Precipitation on Dislocations", *Journal of Applied Physics* **30**, 915-926 (1959), and "Diffusion-Limited Growth of Precipitate Particles", *Journal of Applied Physics*, **30**, 1518 (1959).

¹³ M.E. Fine Phase Transformation in Condensed Solids, McMillan, New York New York, 1964.

¹⁴ L.E. Murr and F.I. Grace, Residual Structure and Mechanical Properties of Alpha Brass and Stainless Steel Following Deformation by Cold Rolling and Explosive Shock Loading, *Trans. AIME*, 2225, vol 245, 1969.

¹⁵ C. Donadille, R. Valle, P. Dervin and R. Penelle, "Development of Texture and Microstructure During Cold-Rolling and Annealing of F.C.C. Alloys: Example of an Austenitic Stainless Steel, *Acta Metall.* **37**, 1547-1571, 1989.

No attempt has been made to compensate for these potential specimen preparation effects.

Conclusions

1. The stability of the tensile properties, and by extension the burst pressure of post reversion-anneal burst discs, have been demonstrated through microstructural emulation, accelerated aging, and modeling of both the microstructural sensitivity and temperature dependence of aging. We emulated two of the most important microstructural features of the burst disc ligament, that is, martensite production and flow stress (dislocation structure) characteristic of the midline of the ligament. Some differences still exist between the behavior of the simulated material and burst discs. While the emulated material ages, it does so more slowly than actual burst discs. This may indicate that the midline-simulating structure does not represent the burst pressure-controlling microstructure in the burst disc. It may also indicate that the preparation methods compromised the aging kinetics and magnitude of the effect. A desirable step to achieve full confidence in these conclusions is to reproduce these results using reversion-annealed burst discs.

2. The measured activation enthalpy of 18 kJ/mole (4.3 kcal/mole) for ultimate tensile strength increase is inconsistent with diffusion of carbon. An alternative mechanism, the (substitutional) diffusion of C-V complexes was investigated. The diffusion activation enthalpy of C-V complexes was modeled as $Q_{\text{eff}} = E_{\text{M}}^{\text{V}} - E_{\text{b}}^{\text{C-V}}$ (in the presence of non-thermal excess vacancies), predicting a carbon-vacancy binding energy of the order of 83 kJ/mole, which is at the upper bounds of reported values [summarized in reference 6]. The reaction kinetics are consistent with transport of a species such as carbon to dislocation lines, presumably pinning the dislocations and enhancing tensile strength. Applying a mathematical solution to the long-term diffusion problem of defects to dislocations due to Ham¹² has allowed a microstructurally consistent understanding of the differing effects that cold work has on the rate of aging of unstable microstructures. The model is consistent with the behavior of burst discs; however, it is unable to account for the effects of a highly inhomogeneous dislocation density in which aging kinetics may be controlled by high local densities rather than average dislocation densities.

3. The activation volume measurements demonstrate behavior which resembles that of precipitation-strengthened materials such as IN903, in which athermal obstacles control dislocation flow. The measurements also demonstrate the high stability of the worked microstructure against aging and thermal annealing. Furthermore, the activation volume measurements establish that the martensitic phase *per se* does not control the plastic flow of the worked metal. The reversion-annealing behavior is consistent with known microstructural effects at high cold-rolled strains in 316L, in which persistent dislocation structures that are resistant to annealing are observed. The activated event L is computed to be less than or equal to 1b, suggesting dislocation intersection and bypass as the deformation controlling event, an apparent contradiction of the athermal behavior noted.

4. This analysis leads to the conclusion that prevention of the undesired aging phenomenon, without resort to partial annealing, requires avoiding the extreme deformation states that give rise to the aging phenomenon.

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Appendices

A. Hypothesis for the Mechanism Controlling Time Dependent Strengthening

In order to explain the aging phenomenon we hypothesize a time dependent strengthening mechanism which depends upon carbon redistribution to dislocations wherein pinning points are formed. We assume that once carbon arrives at the dislocations, formation of obstacles is rapid. The rate of carbon redistribution is assumed to be greatly accelerated by the presence of excess vacancies produced by cold work, and the model exhibits strong dependence upon sink spacing (mean free path) which is related to the degree of cold work. The observed temperature sensitivity (low activation enthalpy) requires the formation of a large excess of vacancies from cold work (above the thermal equilibrium concentration), and the association of carbon with these vacancies into C-V pairs exhibiting a low migration energy. During the 732°C reversion anneal, excess vacancies are assumed to be annihilated while carbon is moved into solution, removing the strengthening effect of precipitated carbon on dislocations. Self interstitials formed by cold work are assumed immobile to higher temperatures, in agreement with other investigators³⁻⁶.

B. Analysis of the Activation Energy Utilizing Vacancy-assisted Accelerated Diffusion

This work draws upon that of Ulitchney and Gibala⁶. An explanation for the low temperature sensitivity of the aging process may be derived from an understanding of vacancy partitioning, when both free vacancies and carbon-vacancy (C-V) complexes exist. Assuming that C-V pairs are mobile, and are in dynamic equilibrium with interstitial carbon and free vacancies then the total vacancy concentration may be written as $C_v^T = C_v^F + C^{C-V}$, in which C_v^F is the free vacancy concentration, and C^{C-V} is the concentration of C-V pairs. Following the derivation of Ulitchney and Gibala⁶ the effective diffusion coefficient of vacancies can then be written

$$D = C_0^2 v Z_1 C_C \text{Exp}(E_b^{C-V}/kT) \text{Exp}-(E_F^V + E_M^V)/kT \quad (3)$$

In which C_0 is the initial vacancy concentration, v is a vibrational frequency, Z_1 is a crystallographic coordination number, C_C is the initial carbon concentration, E_b^{C-V} is the binding energy of carbon atoms to monovacancies, and E_F^V and E_M^V are the formation and migration energies for monovacancies in the metal, respectively. C_0 may be replaced by C_v^T to describe a variable diffusivity during the aging, as excess vacancies are consumed.

The effective temperature dependence of the carbon-vacancy diffusion in (2) is then

$$Q_{\text{EFF}} = E_F^V + E_M^V - E_b^{C-V} \quad (4)$$

which states that the effective activation enthalpy is equal to the vacancy self diffusion energy minus the binding energy of carbon to vacancies. When the number of vacancies

greatly exceeds the thermal contribution, as hypothesized to result from extreme levels of cold work, we may ignore E_F^V . During the aging transient we then obtain $Q_{EFF} = E_M^V - E_b^{C-V}$. From literature values, $E_M^V \sim 0.4Q_{SD} \sim 100$ kJ/mole (24 kcal/mol) in stainless steel. Using $Q_{EFF} \sim 18$ kJ/mole (4.3 kcal/mole), the binding energy E_b^{C-V} is approximately 82-83 kJ/mole (~ 20 kcal/mole). This value is at the upper bound of reported values⁶, indicating rough agreement with experiment. Following the end of aging when the vacancy excess has been consumed, thermal equilibrium would again control the vacancy population, the number of vacancies would become small, and the diffusion rate would become slow. At that time, the effective activation enthalpy Q_{EFF} would become larger by the value of E_F^V . As a result, Q_{EFF}^{C-V} exceeds $Q_{SD}^{(C)}$, and interstitial carbon diffusion would control the rate of transport of carbon diffusion to dislocations.

The effect of the reversion anneal is to consume excess vacancies, thus slowing the rapid transport of carbon. Thus the above diffusion argument explains why the strength increase stops following the reversion anneal. In addition, the anneal removes carbon already transported to dislocations, putting it back into solution, thereby removing the strength increase caused by aging.

C. Quantification of Thermal Acceleration of Aging

At the beginning of the discussion, we hypothesized that the effect of the reversion anneal was to consume excess vacancies. From the preceding discussion, we see that we can define the degree of acceleration of the aging at 250°C using the characteristic time τ , and the acceleration factor τ^{25} / τ^{250} . Using $m=1$, the data of figure 8, and 1σ of 7.6 MPa (see Table III), we obtain $\tau^{25} = 741 \pm 40$ days and $\tau^{250} = 7.1 \pm 0.7$ days, from which we obtain $\tau^{25} / \tau^{250} = 106 \pm 16$, which is the degree of acceleration of aging at 250°C compared to 25°C. Previously we asserted that annealing resulted in cessation of aging; we may now quantify that statement. We annealed 28 days at 250°C, and saw no strength increase ($X \sim 0$). From the relationship $t^{25}/\tau^{25} = t^{250}/\tau^{250}$, 28 days at 250°C is equivalent to 2968 ± 448 days at 25°C. Thus no aging effect should be observed for that period of time (8.1 ± 1.2 years).

D. Kinetics of Aging in Different Microstructures

Of particular interest is why heavily cold-worked microstructures such as found in the burst disc ligament age at high rates at room temperature, while our 75% cold-rolled specimens age more slowly, and the 50% rolled specimens appear to not age at all. Using the model provided by Ham¹² we compare the aging rates of different microstructures arising from a variety of levels of cold work. In this model, we use the dislocation density at different levels of cold work as a parameter. We assume that a defect such as C-V complexes arising from excessive cold work must 'sink' to the dislocation structure. Once there, (unidentified) pinning points are formed, increasing the flow strength of the microstructure. Solution of the long-term diffusion problem for both edge and screw dislocations, and for both regular and random arrays provides a relationship between the sink radius r_s , and the characteristic time (τ) of the diffusion problem. Ham assumed a cylindrical array of dislocations in which the sink radius (r_s) is related to dislocation

density by $r_s = (\pi\rho)^{-1/2}$ in which ρ is the dislocation density in length per unit volume. He then related the parameter $D\tau\pi\rho$, where D is the diffusion coefficient of the species in question, to the variable $(1/R(\rho)^{1/2})$, in which R is the capture radius of the sink.

Using Ham's relationship we may now illustrate both aging trends and the sensitivity of τ to dislocation density (ρ). We use the dislocation density data of Murr and Grace¹⁴ for brass, since data for 316L over a wide range of cold work are unavailable. A change in dislocation density from that of a 5-10% cold worked material ($10^{12} - 10^{13} \text{ m}^{-2}$) to the highly worked level typical of the burst disc material (greater than 10^{15} m^{-2}) reduces the characteristic time by about a factor of 400. Thus to reach a fraction reacted X ($1-1/e$) which requires 60 days in a burst disc will require decades in material cold worked to moderate strains, and the material would be seen as not aging. (This assumes that the necessary vacancy excess, postulated here and elsewhere, continues to exist. At small strains and low deformation rates, this will not be true, further diminishing the magnitude of the effect.) Consistent with the example, even our 50% reduced 316L exhibited no measurable aging within the bounds of the experiment.

Similarly, the difference in rate of aging between our 75% cold-rolled material and the burst disc behavior may be examined using equation (2). Recall from Figure 2 that very high strains, to $\epsilon > 2$, are predicted in the ligament. Based upon Murr's dislocation densities¹⁴, and Ham's¹² first eigenfunction solution, the characteristic time should decrease by a minimum factor of 2.5. This trend is correct in direction, that is, the aging rate increases with greater cold work, however it underpredicts the relative aging rates. The differences may arise from the extreme inhomogeneity of the dislocation density within the microstructure, as was shown by Donadille et al¹⁵, compared to the uniform structures assumed within the model. We know also that differences exist between microstructures formed at high rates vs. those formed at low rates. The rolled microstructures would tend to have more well-defined dislocation walls and fewer free dislocations between cell walls than would high-rate-formed microstructures (burst disc scores). Thus the high rate microstructure might have a greater volume of material to age, providing a greater overall rate and extent of strengthening. A much more detailed description of dislocation structures and an understanding of the obstacles formed by carbon precipitation are required to refine this analysis, and we must be satisfied with illustrating trends.

E. Activation Volume

Specimens were not aged sufficiently to allow activation volume measurements to detect the potential effects of carbon precipitation on V^* . The following observations apply only to unaged or short-term aged cold worked material.

The results in Figure 12A indicate that the activation volume behavior is strongly athermal, and exhibits a high degree of microstructural stability against thermal perturbations. The observations of Donadille et al¹⁵, who observed inhomogeneous recovery and recrystallization in heavily worked 316L, are consistent with the above observations of microstructural stability. Donadille observed microbands¹⁵ at strains

exceeding 70%, as well as rhombohedral shaped structures, which were very resistant to recovery and recrystallization, and which would not have recovered at the temperature of the reversion-anneal¹⁵. These apparently highly integrated dislocation structures may be the source of the athermal behavior. Much more detailed descriptions of these structures are required to clarify the occurrence of dislocation intersections as deformation-controlling events in the presence of these structures.

The length of the microstructurally controlling activated event L may be computed from $V^*=b^2L$. Since b^3/V^* is typically about one at the beginning of stress relaxation, L is of the order of one burgers' vector or smaller. This observation suggests that flow is controlled by dislocation intersection and bypass, which presents a difficulty in interpretation. A value of L less than $1b$ appears to require cooperative motion of dislocations in which a bypass event is partially compensated by a back-relaxation of other dislocations in the tightly woven dislocation structures. This issue cannot be resolved with the current information.

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