An Investigation of Fatigue in an Fe-based Metallic Glass by Nanoindentation

by

Lisa Marie Witmer

Submitted to the Department of Material Science and Engineering in Partial Fulfillment of the Requirements for the Degree of Bachelor of Science at the Massachusetts Institute of Technology

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ABSTRACT

An Iron-based bulk metallic glass was studied using nanoindentation to examine the effects of fatigue on the onset of plasticity. Experiments were performed on samples in an as-received and 0.8T_g 12-hour annealed condition. The nanoindentation testing procedure focused on investigating fatigue of these samples through cyclic loading as well as investigating the maximum shear stress induced through varied loading in the as-received sample. With respect to the maximum induced shear stress, no clear correlation between induced stress and the onset of plasticity in this material was observed. The results of fatigue in the as-received sample demonstrate material strengthening upon repeated loading, while the effect is absent in the annealed sample. The results are discussed in relation to material structure and free volume, and analysis suggests that structural relaxation during annealing serves to inhibit material strengthening by fatigue in metallic glasses, while cycling in the as-received sample likely strengthens due to a local effect.

Thesis Supervisor: Christopher A. Schuh

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1. Introduction

Bulk metallic glass (BMG) is a class of materials which exhibits metallic bonding within an amorphous structure. This atomic arrangement is such that short-range order exists, but the material is without any discernable long-range order. This unique structure and the resulting properties set this material apart from crystalline metals and a desire to exploit these properties, which include high strength, hardness, and elastic limit [1] has increased interest in metallic glasses in recent decades. These materials have potential use in various structural applications, however realization of these applications has been limited by their less desirable properties, namely brittleness and low ductility, which cause cracks to quickly propagate through the material and result in catastrophic failure [2].

Before we are able to exploit the beneficial properties of metallic glasses we must first gain more understanding of the mechanisms of deformation in these materials, and specifically how they are affected by the material structure. There have been many efforts to examine the role of structure on plastic deformation of glasses. It has been established that the features of inhomogeneous plastic flow in metallic glasses are a result of shear localization with an applied stress at low temperatures [2, 3]. Figures 1a and 1b show a representation of the effect of applied stress on a shear transformation zone (STZ) and an image of shear banding, respectively. STZs are small clusters of atoms which act as the microscopic carriers of plasticity by atomic reorganization in response to an applied force. When an STZ is activated, the local atomic arrangement is disrupted resulting in a redistribution of free volume. If the stresses on the material continue to increase many of these STZs will be affected resulting in shear strain localization into a macroscopic displacement of material called a shear band [2]. Shear banding events create macroscopic plasticity through shear offsets in the material and often propagate unpredictably through the entire material to the point of fracture.
A strong correlation between plastic flow in metallic glasses and available free volume was established early in the study of this material [2, 3]. Areas of relatively larger amounts of free volume less closely packed and as a result, these areas require less shear stress to activate STZs into another configuration. Some groups have further explored the effect of free volume on the inhomogeneous plastic flow in glasses by altering the amount of free volume through the process of annealing, often resulting in severe embrittlement [5-7]. In this study nanoindentation is used to investigate the effects of thermal relaxation on shear banding in a localized area of material which allows the evolution of deformation in this material to still be observed.

STZs and shear banding represent the fundamentals with which to understand plastic deformation in metallic glasses, however the precise sequence of events which result in plastic flow are still unknown [8]. Complete comprehension of the plastic flow in metallic glasses might make it possible to control initiation of shear bands and their propagation through the material. This capability would be especially attractive for any metallic glass used in structural applications because it might allow the material to be engineered for safety and performance. Current efforts to control properties have been focused on changing the material composition and the development of composites to expand the shear band density and distribute the fracture energy over a larger volume.
of the sample [9]. Foreign-particle-reinforcement metallic glass composites may currently offer the
most control of microstructure and mechanical properties by adjusting the shape, size and volume
fraction of employed particles. A recent study [9] reports optimization of the combination of yield
strength and plastic strain in a zirconium-based glass by achieving 18.5% plastic strain and an yield
strength of 1.85 GPa through uniform distribution of 45-75 μm graphite particles at 3% volume
fraction. These findings are also in agreement with similar studies conducted in dendritic [10] and
crystalline phase [11] particle-reinforcement studies which the improved plastic strain is attributed to
the confinement and redirection of shear bands upon encounter with the second phase particles.

In an effort to identify important deformation mechanisms in metallic glasses some groups have
explored standard testing procedures common to structural metals. One specific area of interest is
the effects of stress concentrators in this material. Stress concentrators are geometric
discontinuities, such as cracks, sharp corners and holes, which may cause a local increase in the
intensity of a stress field with an applied force [12] and one method of imposing and studying the
effects of localized stress on plastic deformation in metallic glasses is through nanoindentation.
Nanoindentation dynamically measures very small forces and displacements created using a diamond
tip and has been used to extract mechanical properties from the material [13, 14] or information
about discrete deformation events [15-18]. A typical load vs. depth (P vs. h) plot produced by
nanoindentation of an iron-based metallic glass at a load rate of 2500 μN/s is shown in Figure 2.
The arrow marks a rapid displacement burst called a ‘pop-in’ which corresponds to a shear banding
event below the indenter tip [18-21].
In light of their potential structural applications, a pertinent aspect of metallic glass deformation is the effects of fatigue. Fatigue is plastic deformation of a material through cyclic loading and unloading at stresses far below the yield stress of the material. A material subjected to dynamic loading is more likely to fail at lower applied loads especially if the load is repeated for a number of cycles [12]. The sub-critical loading leads to an accumulation of gradual deformation with each loading cycle which compounds and eventually causes failure. Until recently the inability to produce bulk specimens of metallic glass limited the investigation of fatigue on BMGs. Now standardized fatigue tests, such as tension, compression and bending tests, are able to be applied to bulk metallic glass samples. The fatigue work of Flores on BMGs focuses on the stress/life behavior between BMG alloys [11, 22] and a composite of the same material with results pointing to a sizeable increase in the endurance limit of the composite material as high as double the limit of a BMG in one study [22]. An important difference between current fatigue tests conducted in this field and those presented here is that this study features the fatigue cycles are carried out by nanoindentation to
induce localized stress in the material.

A preliminary study on the effects of fatigue by nanoindentation in a palladium-based bulk metallic glass of composition Pd_{40}Ni_{40}P_{20} has been conducted within the Schuh research group at MIT and has served as a springboard for investigation into the research presented here. The palladium study showed material strengthening by nanoindentation through fatigue load applications on this material. This sparked interest into whether the material strengthening was unique to the palladium glass or if it was a result of the material structure, and thus applicable to other BMGs. The present study primarily serves to investigate the effects of fatigue in an Fe-based metallic glass through nanoindentation in comparison to the Pd-glass results. Additionally, the effects of maximum shear stress and sub-$T_g$ annealing are explored.

2. Methods

The material used in this study was a Fe-based bulk metallic glass rod 5mm in diameter with composition Fe_{41}Co_{17}Cr_{15}Mo_{14}C_{15}B_{6}Y_{2}. The glass was produced by arc melting pure metals and casting them in a cooled copper mold under an inert atmosphere, as detailed in Ref. [23]. Two pieces of the glass were sectioned from the same bulk sample using a diamond-impregnated circular saw (Buehler, Isomet 5000). One sample was tested in an as received condition while another was annealed at 397°C ($T=0.8T_g$) for 12 hours in an argon atmosphere to prevent oxidation. It has been shown that annealing BMGs below the glass transition temperature ($T_g$) does not alter the amorphous structure of the glass [5, 6]. Both the as received (AR) and annealed (AN) samples were mounted in epoxy and mechanically polished to a surface finish of roughly 1nm.
Figure 3: a) a symmetric loading profile and b) an example of a cyclic loading profile, which features five preload cycles at a preload of 1250uN. The load rate for both curves is 2500 μN/s.

Nanoindentation tests were performed using a Hysitron TriboIndenter (Hysitron, Inc., Minneapolis, MN) with load and depth resolution of 0.1 μN and 0.2 nm, respectively. All tests were performed with a diamond tip in a depth range where the tip geometry could be reasonably approximated as a sphere. The radius of the tip is estimated as ~1100nm, according to standard procedures described in Refs. [24, 25]. Each loading and unloading segment was conducted at a constant rate of 2500 μN/s. Two characteristic loading functions were used in testing the AR and AN samples: symmetric loading and cyclic loading. The symmetric load function is a simple load to 4000 μN followed by complete unloading, as shown in Figure 3a and was used to understand the baseline behavior of the glass. An example of a cyclic loading function is shown in Figure 3b. The cyclic loading functions contain a series of low force loading and unloading cycles, the quantity and magnitude of which were varied to investigate of fatigue and stress conditions, respectively.

First, the AR and AN samples were characterized using the symmetric loading curve to describe the effect of annealing on the onset of plasticity in the glass. Next we investigated the
effect of maximum shear stress in the material during the cyclic preloading. Each of these tests was conducted at five preload cycles with the maximum cycle load of each cycle 1250 µN, 156 µN, or 11 µN. Finally the effect of locally induced fatigue was investigated by holding the maximum cycle load constant at 1250 µN for both samples and varying the number of preload cycles. The results from this testing parameter were compared to the baseline characterization of both samples.

Figure 4: A typical P vs. h plot of nanoindentation of an Fe-based metallic glass is shown (top). The velocity differential plot is shown (bottom) and the peaks of the velocity plot directly correspond to the pop-in events in the image above.
A procedure involving three main parts was used to analyze the individual indentation curves to distill the data into a useful form. First, each load vs. depth plot was examined visually and an approximate load for the point of the first pop-in was recorded. In an effort to avoid bias in the visually collected data, the P vs. h plots were analyzed further to identify pop-in events more precisely. The final loading segment to a maximum load ($P_{\text{max}}$) of 5000 $\mu$N of each indentation was isolated as this section of data contains the pop-in event. The data was then analyzed to identify deviation from elastic behavior upon loading by comparing the data to the Hertzian elastic prediction [26, 27] for the load-displacement relationship given by

$$P = \frac{4}{3} E_r R^\frac{3}{2} h^\frac{3}{2}$$

where $P$ is the load, $R$ is the radius of the indenter tip, $h$ is the indentation depth, and $E_r$ is the reduced modulus. The reduced modulus is given by

$$\frac{1}{E_r} = \frac{1 - \nu^2_{\text{sample}}}{E_{\text{sample}}} + \frac{1 - \nu^2_{\text{indenter}}}{E_{\text{indenter}}}$$

where $E_r$, for our purposes is calculated to be 217 GPa, based on indentation results. The Hertzian elastic prediction is indicated by the red curve in Figure 4 and since the data in blue follows the elastic prediction at low loads, the behavior exhibited in this region is elastic. A velocity difference profile was also developed by subtracting the predicted indenter velocity for an ideal Hertzian contact from the true measured indenter velocity ($dh/dt$). Points on the $P$ vs. $h$ plot were flagged as possible pop-in events where this velocity difference peaked above the characteristic noise level of a given set of tests as shown by the yellow points in Figure 4. The event corresponding to the peak in the velocity difference profile on the velocity profile exactly corresponds to the pop-in event shown
on the P vs. h plot. Finally, these load estimation data were collected and a double-check method was implemented in which the first set of recorded data was directly compared to the estimated pop-in loads produced. Where these values coincided within ±30 μN the indentation was considered a good test and was included in the final results. If these two values did not coincide, the indentation curve results were not considered in the results analysis. There are a few common reasons which may have caused the deviation such as inelastic behavior during the preload cycles or environmental noise resulting in a poor velocity differential jump, which occurs when there is not enough distinction of the velocity differential spike from baseline velocity to merit deviation.

For each set of experimental conditions, many nominally identical indentations were performed and analyzed. All results presented here consist of greater than 80 good data points for each set of testing parameters to ensure that we have statistical representation of the material behavior. It is important to note that the first pop-in event does not occur at the same load every time, but instead there is a considerable distribution, which can spread over ~1800 μN. This work presents the entire population of good values in cumulative fraction plots in which each point represents the load at first pop-in of a single indentation. Cumulative fraction plots are useful for examining the entire range of pop-in loads as well as visualizing the effect of different experimental conditions. Additionally, data for each test is represented on an average load chart which is useful for identifying the statistical significance of the trends with a change in test parameters. The average load at first pop-in was calculated using the middle 90% of data in an effort to represent the most frequently observed results. The tails of the spectrum were removed in this analysis because there is a slight possibility that these data may be less reliable, despite being approved by the double-check method. For instance, the velocity spike in pop-in events that occur at high loads are easier to distinguish above the machine noise and are easier to see as they are commonly associated with a larger displacement bursts in the P vs. h plots. It is possible that a first pop-in event at a lower load
could have been overlooked. Likewise, the pop-in events that occur at lower loads have lower velocity spikes and thus it is possible that a spike of machine noise could have been mistaken for a pop-in event.

3. Results and Discussion

3.1 Characterization of the AR and AN samples

Figure 5: a) The cumulative fraction plot of the symmetric loading tests shows that the onset of plasticity in the AR and AN sample occur at similar loads. The shape difference between the two plots suggests that the average force at first pop-in for the AN sample is more evenly distributed. B) The average force at first pop-in shows similarities between the two samples with the AN samples having a larger standard deviation.

The symmetric loading curve was used to characterize the AR and AN samples and the results aim to identify the effect of annealing the sample on the onset of plasticity in the material. The cumulative fraction plot and the average load at first pop-in chart are shown in Figures 5a and 5b, respectively. The cumulative fraction plot shows that the two sets of data roughly overlap one another, cover a comparable range of pop-in loads and the median value of each curve shown are also similar. This suggests that annealing had little to no effect on the average load of the first pop-
in. The statistically overlapping error bars in the average load at first pop-in chart indicate that the load at first pop-in in both materials is essentially the same and that annealing the sample did not have any systematic effect on the average first pop-in load as evident by lowering or increasing the average load at first pop-in.

One interesting difference between these two plots is the change in shape of the cumulative fraction plot. The points in the annealed sample have a more linear form as opposed to the sigmoidal AR plot, implying that the load at first pop-in for the AN sample was more evenly distributed over the range of values. The shape of the AN curve suggests that although the weak points remain relatively unchanged, which is evident by the fact that the AR and AN pop-in load values are similar at loads less than the median pop-in load at $F = 50\%$, the stronger points in the upper half of the plot seem to strengthen upon annealing as evident by the slight shift to the right.

Based on the current understanding of the relationship between STZs and free volume, one possible explanation for the more evenly distributed pop-in load values is this: In any sample there exist locations which have a varied amount of free volume. The amount of free volume dictates the strength or weakness of an area because it is this property which allows for more or less rearrangement of atoms in reaction to an applied load. There will always be a certain amount of inherently weak points in any material because the free volume is not evenly distributed throughout the material. Thus, there are regions in which the atomic mobility is high due to a relative abundance of free volume and the material in these regions will tend to deform more readily with an applied load because there are many different lowest-energy atomic configurations possible. There are also areas in the material with markedly less free volume and thus, less low-energy atomic configurations are possible to achieve when stress is applied. These regions tend to activate STZs less easily because a much higher applied stress is necessary to achieve atomic reorganization. Upon annealing the overall free volume in the material is decreased which acts as a method of material
strengthening by limiting the number of atomic configurations possible. Thus, areas of the annealed sample appear to have strengthened and the amount of free volume in the AN sample is more evenly distributed.

3.2 The effect of applied stress on the load at first pop-in in the AR sample

Here we aim to investigate the maximum shear stress in the material which is the highest shear stress experienced by any point in the material during the preload cycles. A complete analysis of the stress fields for our indentation parameters, that is elastic contact of a spherical indenter on a flat plate, is given by the Hertz Model [28]. Simplification of this model combined with the location of maximum shear stress yields the following approximation for the shear stress in a metallic glass under this type of contact:

\[ \tau_{\text{max}} \approx 0.47 P_m. \]  

(3)

\( P_m \) in this result is the mean contact pressure given by

\[ P_m = \frac{4 E_r a}{3 \pi R}, \]  

(4)

where \( R \) is the tip radius, \( E_r \) is the reduced modulus (Eq. 2) and \( a \) is the contact radius between the indenter tip and the surface as described by

\[ a = \left( \frac{3PR}{4E_r} \right)^{\frac{1}{3}} \]  

(5)

A contour plot of the stress fields under the indenter tip is shown in Figure 6, which shows that the maximum shear stress does not occur at the surface of the material where the material and indenter are in direct contact. Rather, the area of maximum shear stress is located beneath the indenter tip ~0.5\( a \) into the material. The shear stress in the material decreases radially from this point outward.
As is suggested in the calculation of $\tau_{\text{max}}$ and Eq. 4 above, the units of maximum shear stress presented here are normalized by the mean contact pressure.

![Figure 6: Contour plot of the maximum shear stress in units of Pm induced by nanoindentation. The area of maximum shear stress in the material is directly below the indenter tip and ~0.5a beneath the material surface.](image)

The results presented in this section aim to examine correlations between an applied shear stress below the indenter tip during cycling to the average load at first pop-in. These tests were carried out using the cyclic loading curves with 5 preload cycles with varying peak loads which results in different maximum stress levels in the material. Peak loads of 1250 $\mu$N, 156 $\mu$N and 11 $\mu$N were applied to induce 6.6 GPa, 3.3 GPa, and 1.4 GPa of stress, respectively. The cumulative fraction plot and the average load at first pop-in chart are shown in Figure 7a and 7b. The cumulative fraction plot shows a variation in position for each curve; however, a clear correlation between stress level and average load at first pop-in can not be established. There exists no clear trend in the average load at first pop-in in response to the declining applied stress on the system. The average force at first pop-in load chart reinforces the inconclusiveness of this data because these
loads are similar and are accompanied by overlapping error bars.

Figure 7: a) The results of the maximum induced shear stress in the material are inconclusive with no clear trend in the data. b) The average force at first pop-in for 3.3 GPa and 6.6 GPa suggest that there might be a trend, however taking the data as a whole no clear trend is identifiable and the set of experiments thus far is inconclusive.

One might be inclined to predict that cycles of preloads at a higher stresses would result in a higher load at first pop-in based on the magnitude of perturbation imposed on the STZs in the sample. It is clear from these results, however, that no clear correlation between an induced stress and the load at first pop-in can be established. It is believed that this result could be related to the physical response of the material because consistency in the experimental design, execution and reproducibility has established confidence in the testing method used.

An alternative explanation which may have more merit than the one just offered, however, is that the results presented for the 1.4 GPa test are not an accurate representation of what is happening within the material due to machine sensitivity. Upon examination of the recorded loading cycles of the indenter tip there were several unexpected tip oscillations indicating that the machine transducer, or motion control mechanism, was expressing heightened sensitivity. The
expressed sensitivity contributed to greater amount of machine noise in the recorded data. Thus, identifying pop-in events above the machine noise was markedly more difficult and evidence of the difficulty in extracting usable data was observed in the total number of indents required for this loading curve. For every other testing condition presented in this study a maximum of 200 or 300 indents were conducted to obtain a yield of at least 27%. For the 1.4 GPa test, however, a total of 600 indents were conducted to obtain a yield of only 24%. It is likely that the good points identified exhibit high loads at first pop-in because evidence of the actual first pop-in event was clouded by the noise of the machine. A plausible explanation for the sudden increase in sensitivity with this specific test is that the sensitivity increased as a result of the transducer being programmed to cycle at such a low load during the preload cycles.

3.3 The effect of fatigue on the load at first pop-in in the AR and AN samples

![Effect of Fatigue in AR Sample](image1)

![Average Load at First Pop-In](image2)

Figure 8: a) Cumulative fraction plot of the effects of fatigue in the AR sample and b) average load at first pop-in. The cumulative fraction plot shows a consistent shift to the right with increased number of cycles and the average load at first pop-in shows and statistically significant increase in the load with increasing number of cycles.
This set of experiments was carried out by varying the number of preload cycles at a peak load of 1250 μN for all tests. The results presented in this section aim to examine the effects of fatigue at subcritical loads on the load at first pop-in in the AR and AN samples. First the results of fatigue tests for the AR sample will be discussed.

One, five, and ten fatigue preload cycles were conducted on the AR, and the cumulative fraction plot along with the average load at first pop-in chart for the AR sample are shown in Figure 8a and 8b, respectively. For comparison, the results of the symmetric loading function are also shown. It can be seen from these plots that one preload cycle does not impart any statistical shift to the data; however further cycling corresponds to an increase in the average load at first pop-in ~400 μN. The average load at first pop-in chart reinforces these results with the load at first pop-in showing significant increase with an increase in the number of cycles.

The results presented here suggest that fatigue by nanoindentation of the AR sample has a strengthening effect on the material. From our understanding of metallic glass structure and deformation mechanisms it seems possible that as the number of preload cycles increases, the local STZs near the indentation site respond by readjusting the atoms and free volume multiple times upon loading and unloading which then results in a hardening effect where a larger applied load to initiate plastic flow in the material is required. The strengthening effect observed may hint at a localized reduction of free volume with increased cycling that may affect the local shear band path, resulting in a shift of the entire curve to higher loads on the cumulative fraction plot.

It is significant to note that this strengthening effect of fatigue by nanoindentation has been observed through preliminary studies conducted on a Pd-based metallic glass in the Schuh research group. The cumulative fraction plot for the Pd-based glass fatigued at 200 μN for one, five, and ten cycles compared to the baseline plot is shown in Figure 9. Similar to the results presented for the iron-based glass, the cumulative fraction plot of fatigue testing on the palladium glass also shifts to
higher loads with increasing number of cycles. The combination of this data with that presented in this paper suggest that strengthening of metallic glasses due to subcritical fatigue by nanoindentation may apply to the entire class of materials.

The experiments conducted for the fatigue tests on the AN sample were one cycle and five cycles and, as in the AR test, the cyclic results were compared to the symmetric loading curve. The cumulative fraction plot along with the average force at first pop-in chart for the AN sample are shown in Figures 10a and 10b. The cumulative fraction plot shows that the curves overlap each other very closely and that unlike the AR sample, the AN sample has a distribution of pop-in loads that is essentially invariant with the number of preload cycles. The average force at first pop-in chart echoes this finding with all three average force values falling within 100 μN of each other and well within the error bars of a given set of experiments. It is evident that the process of annealing at 80% T_g for 12 hours serves to inhibit any material strengthening by fatigue.
The extent of atomic reorganization as a result of the annealing process outlined here is difficult to definitively describe as the time scale and temperature necessary to produce a marked change in the atomic structure in BMGs is unclear. Many of the results of sub-$T_g$ annealing of metallic glasses focus on some degree of topological relaxation through a reduction in free volume \cite{5, 7} while studies carried out at shorter time scales point to short range compositional ordering followed by topological relaxation \cite{29}. A study in which a La-based BMG was annealed at $0.8T_g$ for up to 24 hours suggests significant changes in material properties at annealing times as short as $10^3$ seconds and strong evidence of structural relaxation at annealing times beginning at $10^4$ seconds \cite{6}. In light of this, the strong effects of annealing seen presently in the Fe-based glass may suggest that structural relaxation might have occurred to the extreme point that mechanical stressing through fatigue is incapable of producing further relaxation and thus no strengthening effect is observed here. If the behavior of the Fe-based glass is similar to the glass in the Ramamurty study, it might be assumed that shorter annealing times of $10^2$ or $10^3$ seconds may result in an intermediate
degree of structural relaxation and thus, the strengthening effect in the AN sample may occur, but proof of this requires further testing.

4. Conclusions

This research served in part to characterize the AR and AN samples as well as to investigate any possible correlation between the maximum shear stress induced in the AR sample and the onset of plasticity. A similar load at first pop-in ranges and median values suggest strong similarities between the AR and AN samples. A noteworthy difference between these two samples was the shape of the cumulative fraction plot which hinted at a more even distribution of free volume within the AN sample. The investigation into the effect of stress within the AR sample yielded no significant correlation between the maximum shear stress induced and the load at first pop-in. Further investigation into this area, however, is of interest considering the 3.3 GPa and 6.6 GPa tests show a slight trend while the 1.4 GPa test may have been affected by heightened machine sensitivity.

This research largely served as an investigation of fatigue in an AR and AN iron-based bulk metallic glass by nanoindentation. Plastic deformation was initiated in the samples after cyclic loading and unloading with the resulting shear banding events being characterized by the load at first pop-in. The results suggest material strengthening in the AR sample as a result of the local adjustment of atoms and free volume with little to no effect of fatigue on the annealed sample.

The results of the annealed sample suggest that it was unaffected by the fatigue loading and unloading of the material. This result could be due to the fact that annealing the sample may have caused atomic relaxation to the extent that there was no 'give' or leeway in adjusting the atoms and free volume upon fatigue. Thus, plasticity initiated in the material at a very similar load regardless of the number of fatigue cycles. Further investigation into the causes of the annealing effects
presented here and possibly quantifying the amount of reorganization of atoms due to annealing are also of interest.

It is of interest to determine whether or not the strengthening effect by fatigue in the AR sample will saturate at a certain number of cycles and the results of any additional cycling will resemble those of the AN sample. This possibility is attractive for structural applications because this may indicate that as the material is put into use, the material might strengthen due to structural relaxation and increase the load required to initiate plasticity in the material making the material more attractive for load-bearing applications. This strengthening effect has not yet been demonstrated in uniaxial compression tests, so the reasons for this discrepancy merit further investigation as well.

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