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Use of Liquid Metal Embrittlement (LME) for Controlled Fracture

by James C. Hirvonen, Daniel J. Snoha, Jonathan S. Montgomery, Laszlo J. Kecskes, David M. Gray, and William S. de Rosset

ARL-TR-4976 **September 2009**

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14. ABSTRACT
A concept is described for using liquid metal embrittlement (LME) to cause fracture of steel. Three different approaches are taken to break the metal, all based on putting the liquid metal (LM) into a surface notch (or groove) and applying stress. Some success was achieved in breaking the metal in quasi-static tests of flat samples. However, for dynamic tests of flat samples, as well as tests of a specific cylindrical configuration, brittle fracture was not achieved.

15. SUBJECT TERMS
liquid metal embrittlement, 1038 and 4340 steels, drop tower, modified Charpy bar, fracture of steel cylinders

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1. Introduction

As a part of a program to investigate the possibility of making a fragmenting round with a selectable fragment distribution, several concepts have been explored that make use of the idea of filling in external grooves of a steel shell with a material that is capable of changing the properties of the shell. One of the first concepts was the use of a reactive (thermitic) material to heat external grooves in a steel shell and thereby alter the steel's material properties. The authors in reference 1 have developed this concept using a conventional aluminum and iron oxide powdered thermite mixture to react a boron carbide paste into a steel surface in order to produce a brittle iron boride on a steel surface (*1*). This approach was tried on a macroscopic V-notch by researchers at Texas Tech University, who found the resultant surface temperature increases at the steel surfaces to be too low for enabling metallurgical phase changes for the small volume of thermitic charge available in the V-notch volume (*2*). This result was confirmed by calculations conducted at the U.S. Army Research Laboratory (ARL) (*3*).

Another concept was the use of a reactive material cold-sprayed into external grooves of a steel shell (*4*). Here, the idea was to remove material from the groove by igniting the reactive material. However, ignition of the particular reactive material used in the tests did not result in its removal, and the changes in the reactive material after the reaction were insufficient to provide the desired result. Another idea that has been proposed recently is to use thermite to weld or fill in external grooves on a steel shell (*5*). Here, the thermite reaction would bridge and strengthen the grooves, inducing natural fragmentation. This concept is currently being pursued.

Another concept involves liquid metal embrittlement (LME). It has long been known that certain metals in the liquid state can embrittle other metals (*6–13*). For example, it is well documented that gallium and/or mercury can embrittle aluminum via grain boundary penetration. This has been used for the selective dissolution of grains in susceptible materials. In steels, LME effects have been observed for selective species under conditions that are not as well understood or documented as in the case of aluminum. Unfortunately, these embrittlement events have often occurred as catastrophic component failures associated with unintended elemental contamination and environmental conditions. Although the exact mechanisms of embrittling are complicated, the penetration by the embrittling agent is normally intergranular, and the requirements for embrittlement tend to vary depending on the materials involved. However, minimal conditions required for LME in steels include that:

1. The alloy to be embrittled must be in a state of tension, either locally or applied.
2. The surface of this metal must be clean and free of oxides (i.e., free of a barrier to the liquid metal [LM]).

3. The LM must intimately wet the metal surface. Crack growth rates of up to 1 m/s have been measured (*12*), which is rapid for small-scale components.

Table 1 (*12, 13*) shows various metals known to embrittle steel.

Table 1. Melting temperature of various LMs known to embrittle high-strength steels.

LM	Melting Temperature (F)
Mercury	-38
Gallium	85
Indium	313
Lithium	356
Tin	449
Cadmium	610
Lead	620
Zinc	787
Tellurium	841
Antimony	1167
Copper	1981

The embrittling effect is most pronounced in steels with high hardness values. This is shown in figure 1 (*6*). The drop in tensile strength as a function of material hardness is evident for HRC values in excess of 40. The data shown are for AISI 4130 steel, but it is expected that the approximate relation would hold for other steels.

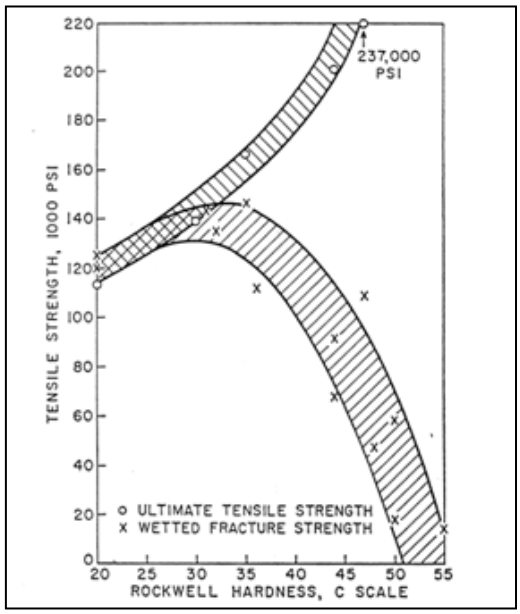

Figure 1. Effect of tensile strength on the embrittling process.

How fast materials fail due to LME depends on many factors. An indication of the role that applied stress plays is shown in figure 2 (*6*). As shown in the figure, fracture could possibly take place in under a second, given a high tensile stress. The material in question is AISI 4130 steel with a hardness value of HRC 44. The embrittling agent is molten lithium.

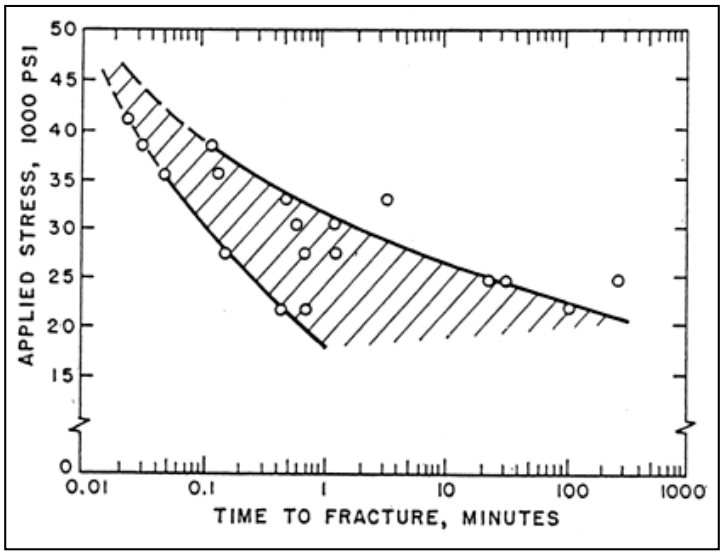

Figure 2. Effect of applied stress on time to failure using LM embrittlement.

For the application of an exploding cylindrical shell, the hoop tensile stress can be extremely high, so the LM embrittling process may cause fracture in a very short time. However, the longitudinal tensile stress may be small. Both components are needed to form a fragment with a pre-determined mass and geometry.

The use of LME agents for purposely reducing the fracture strength of steels via topical application has been investigated (*7*). The choice of potential LME agents for this study avoided mercury due to its poor environmental (toxic) effects and focused on the relatively benign, low melting gallium/indium/tin (Ga/In/Sn) eutectic that has a low melting temperature (~ 11 °C) and contains known embrittling elements (table 1).

Figures 3 and 4 show one possible embodiment of the LME concept. The embrittling agent is encapsulated and placed at the bottom of a notch machined in the exterior surface of a shell. The bottom of the notch is coated with an alloying metal (e.g., lead/tin eutectic) that is capable of forming a eutectic with the embrittling agent. The role of the thin lead/tin coating is to protect the underlying steel surface from oxidation and to make an oxide-free steel surface available to the molten embrittling elements when released. The rest of the notch is filled with a thermite material. A small reactive nanofoil will ignite the thermite material when a signal is transmitted to it. The thermite reaction melts the encapsulation, and the eutectic is released in a liquid state. In theory, wherever the LM is released, the shell is embrittled (figure 4). This embrittling process occurs before the shell is detonated. The metal fractures preferentially at the LM release

locations. This gives the desired fragment size and shape. If the LM is not released, the shell fractures in its natural mass distribution of fragments.

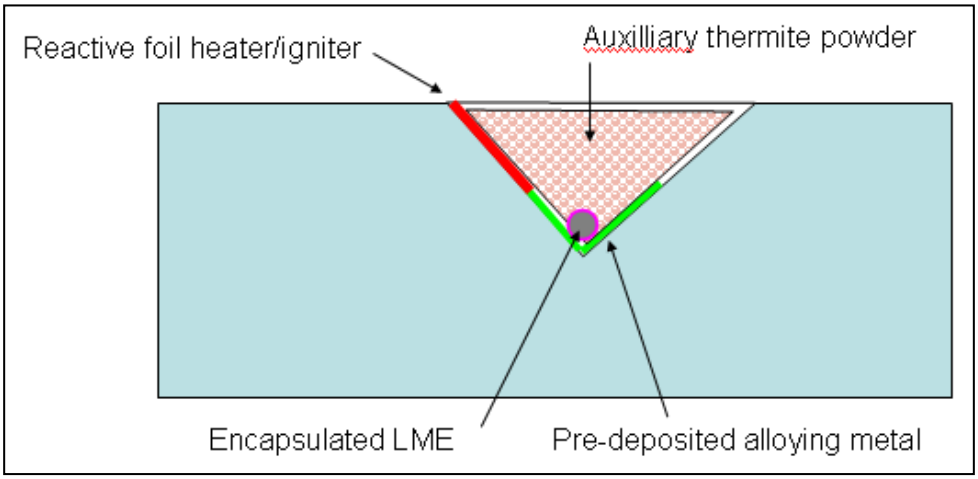

Figure 3. Embodiment of LME concept before activation of embrittling agent.

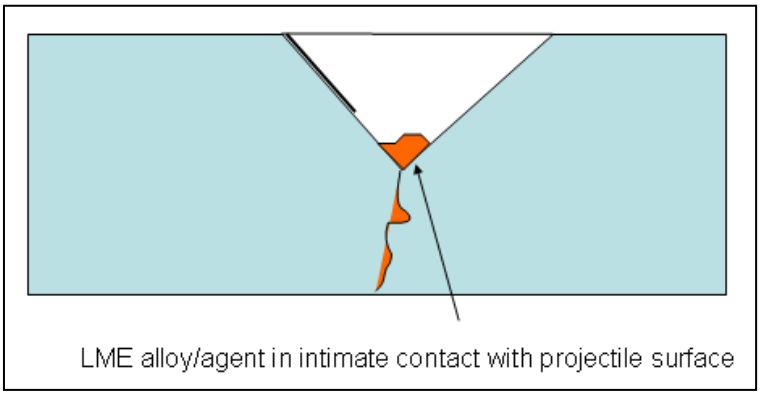

Figure 4. Embodiment of LME concept after activation of embrittling agent.

A series of experiments was undertaken to test the feasibility of this concept. The main question to be answered was under what circumstances steel could be made to fracture in a brittle manner by using LMs. The procedures used to test the feasibility evolved over a period of months. In the end, three different means to fracture the steel samples were used to establish conditions to achieve fracture. Each featured a notch machined into steel. Initial tests focused on drop tower tests with ensuing tests consisting of cyclic stress tests to assess LME susceptibility. Finally, cylinder tests were performed to assess the LME behavior in a non-planar geometry more representative of the final projectile application.

These means are described in section 2. Section 3 contains the results. Following the results is a discussion in section 4, including additional data in the appendix.

2. Experimental Procedures

2.1 Drop Tower Tests of Modified Charpy Bars

Fracture of metals can be examined by conducting tests with notched bars, known as Charpy bars. The approach taken here was to use a modified Charpy bar to investigate the fracture properties of metals that had or had not been treated with an embrittling agent. The modified Charpy bar used in these tests is shown in figure 5. The thickness dimension of this bar was based on dimensions of externally-grooved steel cylinders that had been used in a series of fragmentation tests (*14–16*). The cylinders used in these tests had grooves that ranged from 0.010 in deep to 0.050 in deep. The wall thickness of the cylinders was 0.10 in. The material used for the modified Charpy bars began as 1038 steel (0.38 wt % C), the same as was used for the cylinders used in the fragmentation tests. Later, the modified Charpy bars were made from 4340 steel in order to promote the enhanced embrittlement seen, for instance, in a high hardness steel (figure 1).

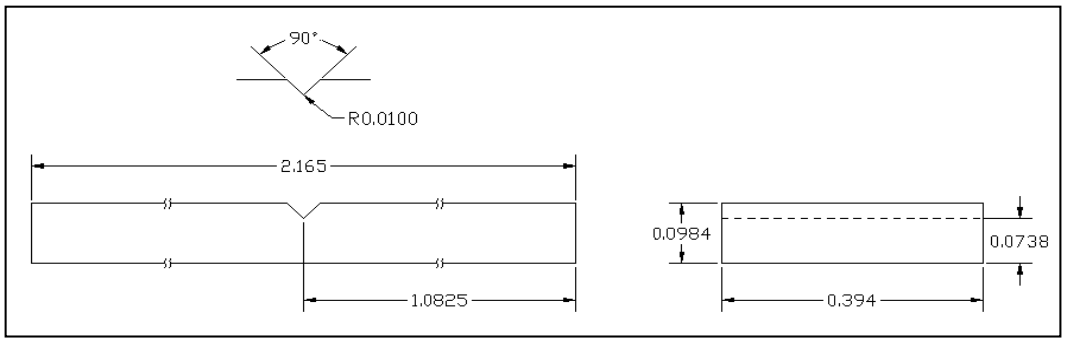

Figure 5. Modified Charpy bar.

The plan was to place the LM in the groove and determine what conditions were needed to embrittle the sample. The particular LM chosen for these tests was a Ga/In/Sn eutectic (liquid at room temperature). The weight percentages of the elements were 62%, 22%, and 16%, respectively. As the tests progressed, additional small amounts (less than 10%) of known embrittling elements such as lead, antimony, and zinc were added to this eutectic mixture. The fraction dissolved in the liquid state of the solvent Ga/In/Sn eutectic was not known.

The LM was applied to each type of test sample (bar or cylinder) in various ways. Generally, the LM would be placed in the notch after insuring that the notch surface was free of grit and dirt. At that point, a small, three-sided file (riffler file) would be used to scratch the surface of the notch below the LM to promote intimate wetting of the LM with the steel surface without air present, as well as induce localized stress raiser sites. At other times, a tungsten scribe was used. In other cases, sandpaper was used. In some instances, the modified Charpy bar was heated,

either with a hot-air blower or a hot plate. Temperatures of approximately 100 °C were achieved.

The first means by which the steel was fractured made use of a drop tower. Such parameters as the impact energy and energy to deform or fracture the sample were measured. Figure 6 shows a picture of the drop tower that was used. The modified Charpy bar is placed with the notch side down between two support rails. The striker, or tup, is raised a certain height above the bar and then released. The tup strikes the modified Charpy bar, which is then either partially or completely fractured and passes through the support rails to a collection box below.

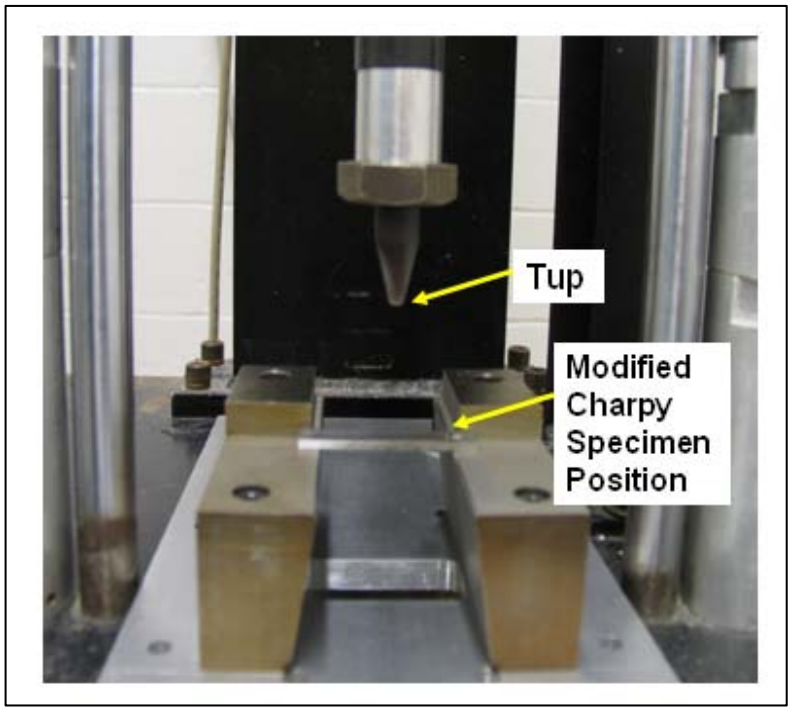

Figure 6. Drop tower test arrangement.

The steel bars were heat treated to different values of hardness, and a range of impact energies (determined by the drop height and weight of the tup) was used on untreated samples to establish input conditions where most of the tests would be conducted for the treated sample. Once this range was established, it was used on subsequent tests of treated samples.

The literature clearly states that LME will only occur when the sample is under tensile stress (*6*). The drop tower provides a tensile stress at the crack tip, but it occurs very rapidly. In order to induce a tensile stress state in the Charpy bar at a slower rate, a special jig was made by which the modified Charpy bar could be put under a tensile load. A picture of this jig is shown in figure 7. The Charpy bar is held down at its ends by two screws. Another screw (not shown in the picture) can be advanced against a hardened pin in contact with the back of the sample so as to cause a deflection in the bar. The amount of deflection is measured with a dial indicator. The

jig is positioned so that the LM can be applied to the notch while the sample is under tension. The idea was to induce the start of embrittlement before the sample is tested in the drop tower.

Figure 7. Picture of jig used to deflect the Charpy bar.

The pre-stressed samples could be sorted into five different groups according to the procedures applied to them, as indicated below.

1. Procedure A: The sample had a notch coated with LM. The notch was abraded, and the sample was then deflected by an amount equal to or greater than 0.055 in. The sample was then heated to 100 °C or more.
2. Procedure B: The sample had the same coating and abrading procedure as A. The sample was deflected less than 0.055 in, and it was heated to 100 °C or more.
3. Procedure C: The sample was chemically etched. The sample had LM applied to the notch (no abrasion), it was deflected equal to or more than 0.050 in, and it was heated to 100 °C or more.
4. Procedure D: The sample had the same coating and abrading procedure as A. The sample was deflected equal to or more than 0.055 in, and it was not heated.
5. Procedure E: The sample had the same coating and abrading procedure as A. The sample was deflected less than 0.055 in.

After the bars were processed, according to one of the five procedures, they were placed in the drop tower and tested. Hereafter, samples treated with Procedure A will be Group A samples, and so forth.

2.2 Static Deflection Tests

Additional deflection tests were made with the modified Charpy bar samples. The purpose of these tests was to determine what, if any, cracks were introduced into the samples by the deflection alone. Large variations in results were obtained. Part of the problem was traced to the difficulty in heat treating the 1038 steel samples to a uniform and consistent hardness. It was decided to change the sample material to 4340 steel, which could be easily heat-treated to the higher hardness values that were needed to conduct the experiments.

The original procedure for deflecting the modified Charpy bars was to advance the screw once to get the desired deflection. In the static tests, the modified Charpy bar deflection was cycled through larger and larger amounts. That is, instead of advancing the screw once, it was advanced and then returned to the original position. Each time the degree of deflection increased, generally by 5 or 10 mils. For example, the screw would be advanced to 10 mils of sample deflection and then returned to its original position. Next, the screw would be advanced to 20 mils and then returned to its original position. This sequence was repeated until the total deflection was 60 mils or the sample fractured. This procedure allowed a measurement of a permanent set in the Charpy bar before fracture.

2.3 Cylinder Tests

Eventually, the concept of using LME to provide a controlled fragmentation warhead would have to be translated into a shell-like configuration. As a first step, the cylindrical steel shell used in the fragmentation tests (*14–16*) was chosen to test the feasibility of the concept. In this case, the cylinder was made from 4340 steel.

The procedure that was found to be consistent in reducing the toughness of 4340 steel (see section 3.3) was used on an external circumferential notch in the cylinder. The cylinder was then pressurized to create a tensile stress on its outside surface in much the same way as the Gun Liner Emplacement with Elastomeric Material (GLEEM) process is carried out (*17*). The design of the cylinder is shown in figure 8. This is approximately the same configuration as used in the fragmentation tests. However, there is only one groove, and it is located in the center of the cylinder. Its depth is 0.025 in, the same as was used for the modified Charpy bars.

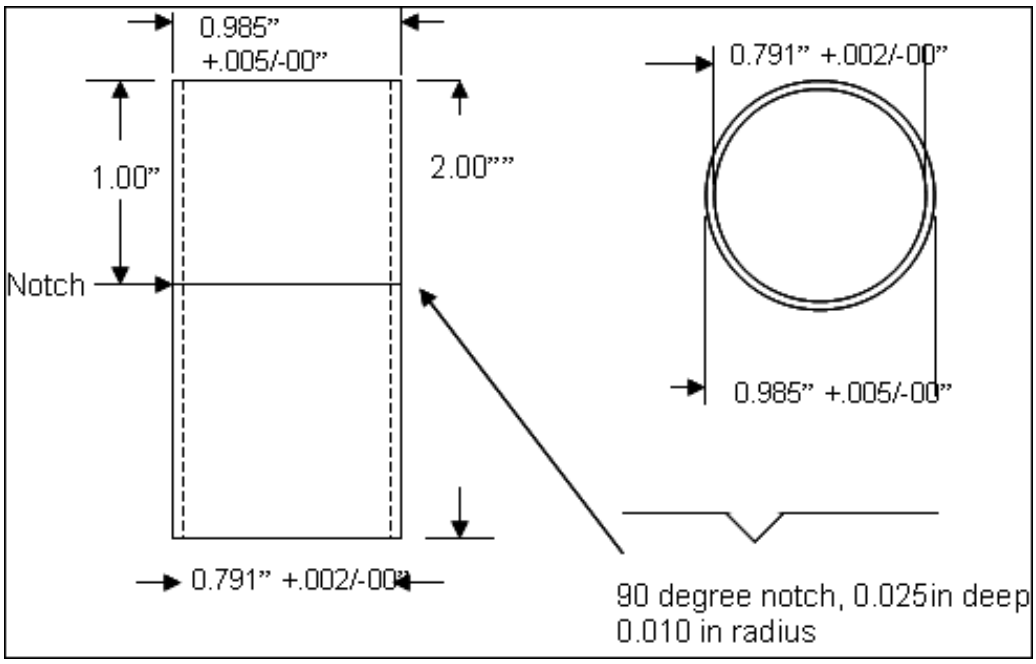

Figure 8. Design of cylinder for fracture test.

Pressure was applied to the center of the cylinder by filling the cylinder with an elastomeric material and applying pressure to that material. Figure 9 shows a picture of the test pieces. The green elastomeric material was shaped to fit the inside of the cylinder. After it was inserted into the cylinder, two nylon caps were used to seal the elastomeric material inside the cylinder. This figure shows the elastomeric material and the end caps outside the cylinder. Sitting on top of this arrangement is the steel piston used to push on the elastomeric material. Also shown is the cylinder with strain gages attached. After all the parts were assembled, the cylinder was put into a load frame and the pressurization took place. For this particular test, two 90° quadrants of the circumferential groove (on opposite sides of the cylinder) were coated with the LM mixture. The expectation was that these locations were where the fracture would initiate.

Figure 9. Experimental arrangement for pressurized cylinder test.

3. Test Results

3.1 Drop Tower Test Results

Figure 10 shows a picture of samples after the drop tower tests exhibiting partial to complete fracture.

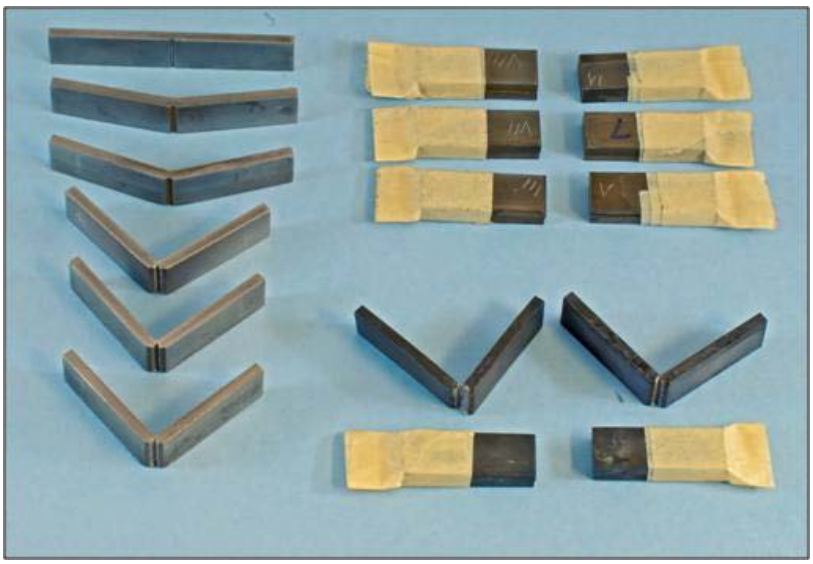

Figure 10. 1038 alloy samples after drop tower tests showing partial (left side) to complete (right side) fracture.

Data from the preliminary drop tower tests are plotted in figure 11. Figure 11 shows the impact energy versus sample hardness and whether the sample broke into two parts (complete fracture) or was intact (partial fracture). For high hardness values (~47 HRC) the samples broke at all impact energies, whereas at low hardness values (~11 HRC) the samples did not completely fracture over the entire range of impact energy applied to the sample. The dashed line in figure 11 separates, approximately, the partial and complete fractures.

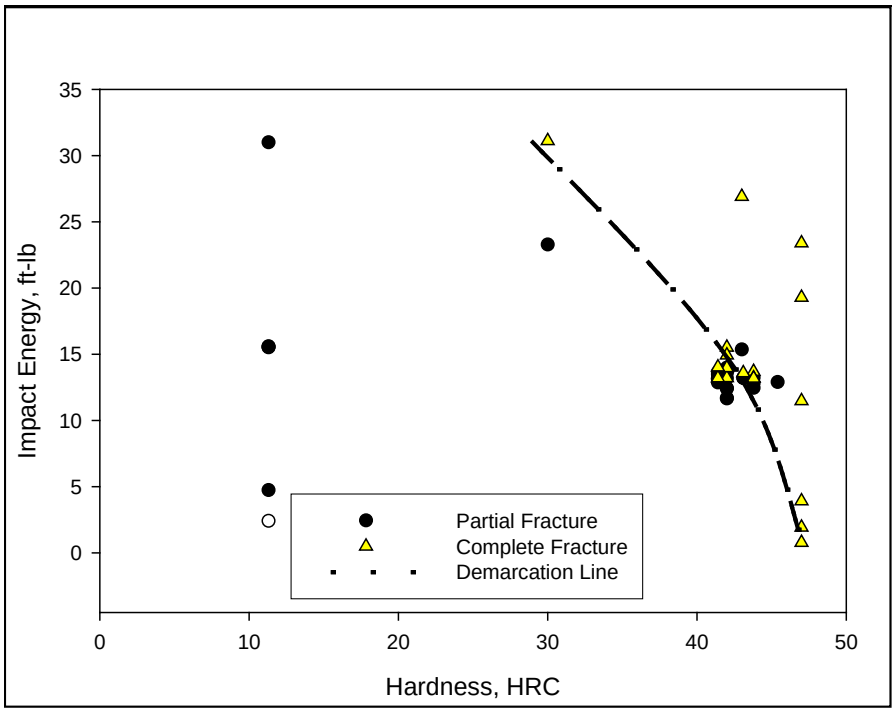

Figure 11. Drop tower test results for all modified Charpy bars not treated with LM; dashed line tends to separate complete and partial fractures.

It should be pointed out that if the strain rate is an important parameter, then it might be more appropriate to plot the hardness versus the impact velocity. However, the mass of the tup remains constant, so the impact energy is proportional to the square of the impact velocity. Thus, any major trends should be the same either way the data are presented.

By replotting the data in figure 11, ARL can focus on the region of hardness values between HRC 40 and 50. This range of hardness values was considered appropriate, based on data for another type of steel shown in figure 1. Figure 12 plots these data and shows that between hardness values of HRC 41 to 45 and above 14 ft-lb of impact energy, the Charpy bar will most likely completely fracture (note the one outlier at a hardness value of HRC 43.6). Below impact energy of 13 ft-lb, it is highly likely that the Charpy bar will not completely fracture. Between 13 and 14 ft-lb, there appears to be a zone of mixed results. (One might sense that there is a higher probability that fracture will occur in this region as the hardness value increases. However, the data are not convincing.) Expressed in different terms, if the sample has a low hardness value and the strain rate is low, then it is highly likely that the fracture will be arrested

within the sample. If the sample has a high hardness value and the strain rate is high, it is likely to fracture completely.

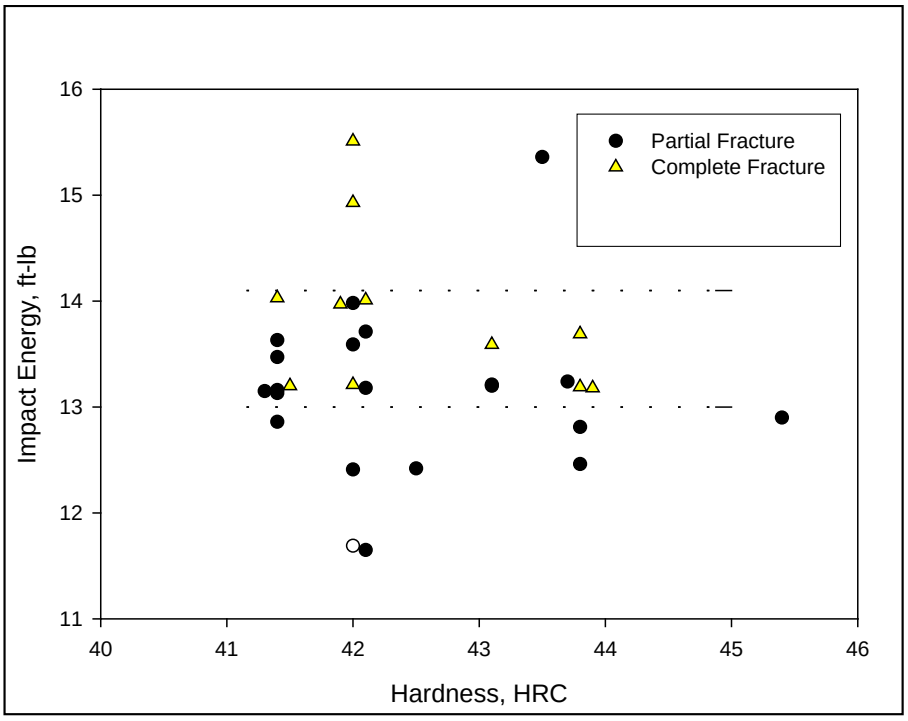

Figure 12. Enlarged region of drop tower test data from figure 10 (untreated samples); dashed lines enclose region of mixed results.

Figure 13 plots the results for the samples that were treated with LM but not pre-stressed. Of the 13 tests run, only three resulted in complete fracture. It can be argued that the LM treatment lowered the bottom end of the zone of mixed results from 13 to 12.5 ft-lbs. However, this does not appear to be a very significant change. In addition, the hardness values of the fractured samples were near the upper end of the hardness range, so that the likelihood of complete fracture might be higher for those samples.

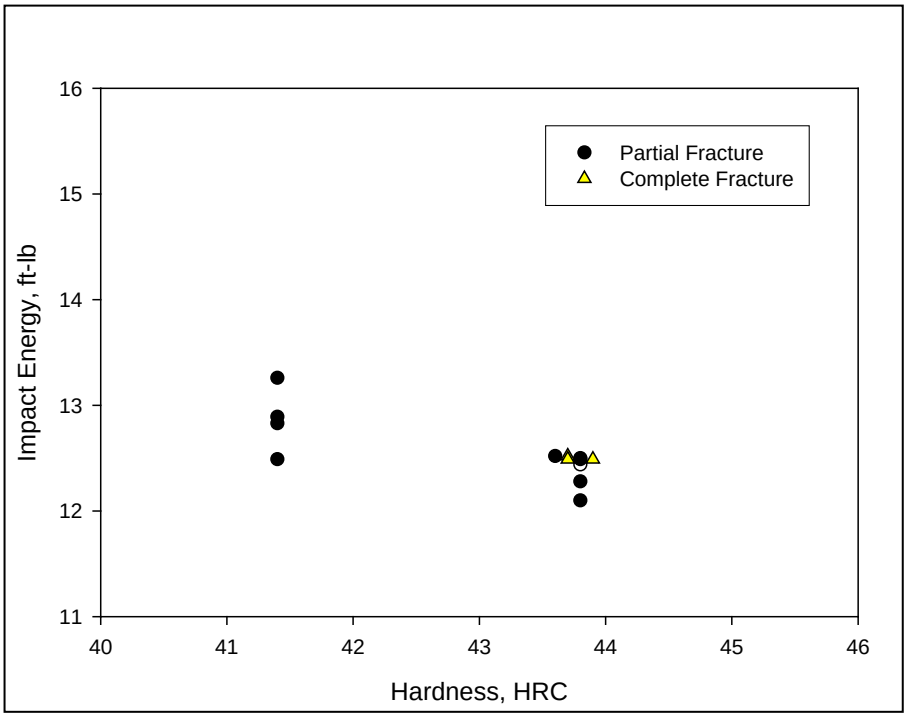

Figure 13. Drop tower test results of LM-treated modified Charpy bars.

Until now, there was concern with whether the sample completely fractured or not. For those samples that completely fractured, ARL would like to know if they would have completely fractured at a much lower impact energy (i.e., was the fracture brittle in nature). One indicator that is available from the drop tower test machine is the total energy expended on the sample, either to fracture or deform it. If the sample fractures with a low value of this energy, then it may be that it fractured in a brittle manner and that lower impact energy might have fractured it also. Figure 14 shows the total energy expended for the samples shown in figure 13. The figure shows that there is no gap between the energies required either to partially or completely fracture the sample. The fact that a sample separates in some cases and not in others for samples with similar hardness values is taken as experimental variation. For the untreated samples with a hardness value of HRC 47 (see figure 12), the absorbed energy ranged from 0.73 to 1.48 ft-lb. Thus, the absorbed energy appears to be more a function of sample hardness than prior treatment.

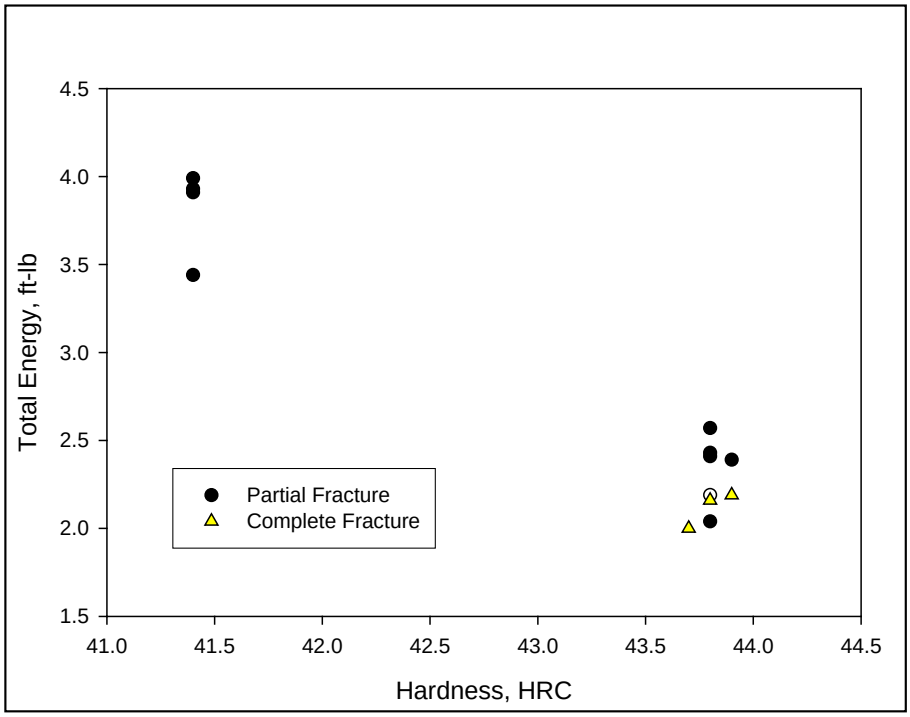

Figure 14. Total energy expended on the sample during the drop test for LM-treated samples.

Several tests were done initially to determine the effect of the deflection on the bar (no LM treatment). It was found that a deflection of 0.050 in or greater resulted in a permanent deformation or set to the bar. That is, the stress at the notch tip was great enough to cause plastic deformation. (As-received modified Charpy bars that had not been heat-treated acquired a permanent set at much lower deflections.) Sample deflections went as high as 0.070 in. In none of the tests was there any observation of cracking.

Baseline drop tower tests were conducted on the pre-stressed (but not treated with LM) Charpy bars. The drop tower was configured to provide an impact energy of 12.8 ft-lb in all cases. Some of the bars completely fractured and some did not. Two of the samples were filled with a riffler file. One of them completely fractured and one did not. Various degrees of deflection were used, up to and including a deflection of 0.060 in. The total energy to deform or fracture the samples as a function of sample hardness is shown in figure 15. These values are not a great deal different from those shown in figure 14. The most important point taken from this figure is that there is still no appreciable difference in total energy needed to partially or completely fracture these samples.

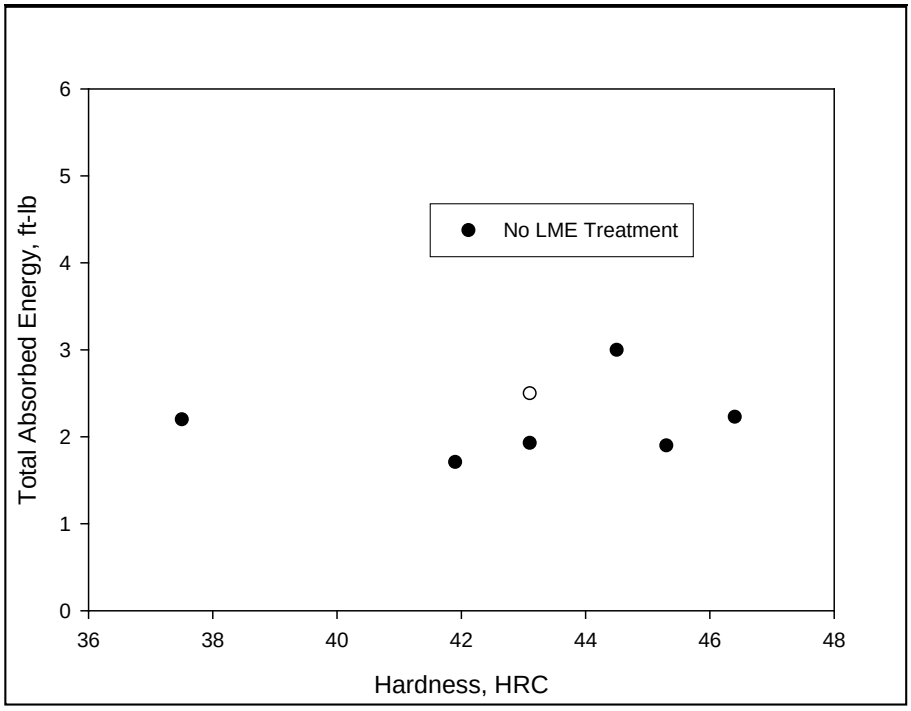

Figure 15. Drop tower test results for pre-stressed samples not treated with LM.

The drop tower test results are now examined for those samples that were both pre-stressed and treated with LM. Photographs of the fracture surfaces of the first three samples tested in this manner (100-PN, 101-PN, and 102-PN) are shown in figures 16a, 16b, and 16c, respectively. Sample 100-PN fractured completely, but the other two did not. However, the degree of fracture for the latter two samples was so great that the two ends of these samples were easily separated after the tests. The deflection of these particular samples prior to the impact tests were not measured as carefully as in subsequent tests. The deflection is estimated to have been approximately 0.050 in for each sample. (Note that without a precise measurement of the deflection, these samples were not included in any of the described procedures.) There was a varying degree of wetting by the LM on each of the samples' fracture surfaces. Sample number 100-PN was not wetted, sample number 101-PN was partially wetted, and sample number 102-PN was completely wetted. The absorbed energy appears to correlate with the degree of wetting. That is, sample number 100-PN had the greatest absorbed energy, sample number 101-PN had less absorbed energy, and sample number 101-PN had the least absorbed energy (see section A-3, tables A-2a through A-2c in the appendix.

Figure 16a. Non-wetted fracture surfaces (sample number 100-PN).

Figure 16b. Partially wetted fracture surfaces (sample number 101-PN).

Figure 16c. Completely wetted fracture surfaces (sample number 102-PN).

Theser results can grouped according to the procedures that were used to treat the samples. These test results are shown in figure 17. In this figure, there is no distinction between partial and complete fractures.

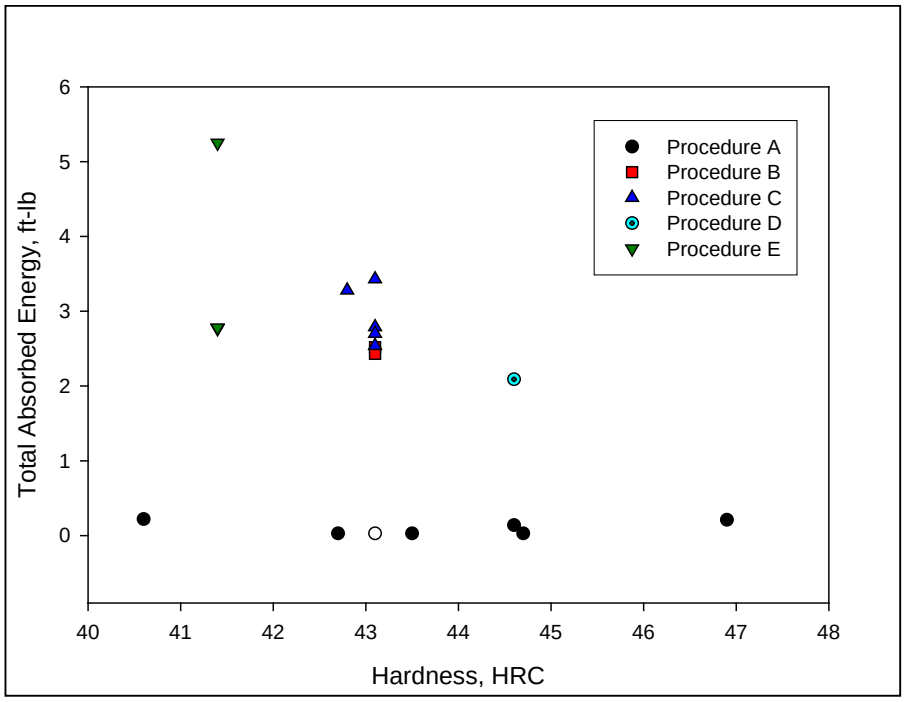

Figure 17. Drop tower test results for pre-stressed, LM-treated samples.

There is a clear gap in absorbed energy between results for samples treated by Procedure A and results for the other samples. The total absorbed energy for the Group A samples is effectively zero.

For several of the Group A samples, a distinct crack was heard while the sample was being pre-stressed. This sound was associated with the visible formation of a crack and occurred some time during the 30 min of pre-stressing. (For some samples, this occurred in less than three min.) The extent of cracking was difficult to discern, since the LM covered the surface where the crack would normally show. It is now thought that the Charpy bars in the lower energy band were already pre-cracked to some extent.

3.2 Static Deflection Test Results

Given that one or more cracks might be initiated during the pre-stress operation, attention was now focused on reproducing this result in a consistent manner. Special care was taken to insure that the LM was in contact with an oxide-free surface. For instance, solder was applied to the notch in the Charpy bar using an acid flux. When LM was applied to the soldered surface, it appeared to penetrate the solder, or at least dissolve it. When the Charpy bar was given Procedure A, it cracked while being deflected after 15 min.

Further tests on samples that had been heat-treated to HRC 44 began to show some variability in results. For instance, sample number 133 was given Procedure D and cracked within one minute of reaching a 0.060 in deflection. If tested in the drop tower, this sample would have appeared in the lower energy band. Sample number 134 was given Procedure D and did not crack; it was

then given Procedure A and still did not crack. The same result occurred for sample number 135. However, sample number 136 was given Procedure D and cracked in less than 30 sec at a deflection of 0.060 in. A conclusion that can be drawn from these tests is that it is possible to induce cracks in the samples with LM when the sample is under stress at room temperature.

An attempt was made to identify the source or sources of the test results variability. One possibility was the non-uniformity of sample hardness from location to location within a sample and from sample to sample within a batch. The 1038 steel used for these tests is especially difficult to heat treat to high hardness and obtain uniform hardness throughout the sample. A close examination of several samples revealed a variation in hardness from sample to sample could be as much as 16 points on the Rockwell C scale. Variations in a single sample could be as high as 10 points. For this reason, a switch was made to 4340 steel, which can be heat-treated to high hardness uniformly and should display a higher propensity for embrittlement as seen in figure 1. This material was used for the remainder of the tests. The appendix gives representative hardness measurements and shows the difference between the 1038 and 4340 steel samples.

More consistent results were obtained with the 4340 steel samples. The first three samples that were tested had a hardness of HRC 46.9. LM was applied to each of them, either with a riffler file or a tungsten carbide scribe, at room temperature. They all broke into two pieces at deflections of 55 mils or less. The remainder of the samples was heat-treated to HRC 53.9. Three of these samples were not treated with LM. They did not break into two pieces when deflected to 60 mils. The remaining nine samples were treated with LM, and all samples completely fractured at 55 mils deflection or less except one. Various methods were used to apply the LM. They all involved scratching the Charpy bar notch covered by the LM. The means used to scratch the surface included a riffler file, 400-grit sandpaper, and a tungsten carbide scribe. No approach appeared to be the best one.

A summary of these results is shown in table 2. In some cases the surface of the notch and side of the bar were sanded with abrasive paper before the LM was applied. The entries under “Surface Preparation” indicate if this was done. The means by which the LM was scratched into the surface of the notch is shown in the column “LM Application”. A complete fracture is indicated by a “Y” in the “Fracture Y/N” column.

Table 2. Data summary for 4340 steel Charpy bars.

Sample Number	Hardness (HRC)	Surface Preparation	LM Application	Fracture Y/N	Maximum Deflection (mils)
185	46.9	none	riffler	Y	50
186	46.9	none	riffler	Y	55
187	46.9	400 grit in notch	scribe	Y	55
189	53.9	400 grit in notch	riffler	Y	<55
190	53.9	400 grit in notch	riffler	N	60
190 (repeat)	53.9	400 grit in notch 150 grit on edge	400 grit	Y	55
191	53.9	400 grit in notch 150 grit on edge	400 grit	Y	55
192	53.9	400 grit in notch 150 grit on edge	scribe riffler	Y	35
193	53.9	400 grit in notch	N/A	N	60
193 (repeat)	53.9	none	400 grit	Y	50
194	53.9	400 grit in notch	400 grit	Y	40
195	53.9	400 grit in notch	N/A	N	60
196	53.9	400 grit in notch	N/A	N	60
197	53.9	400 grit in notch	400 grit	Y	30 - 40
198	53.9	400 grit in notch	400 grit	Y	30
199	53.9	400 grit in notch	400 grit	N	55

3.3 Cylinder Test Results

Two steel cylinders were tested. The first one was pressurized at a cross-head displacement rate of 0.1 in/min, and the second was tested at a higher rate, 1.0 in/min. Both cylinders split in the longitudinal direction at loads over 20,000 lb. The first cylinder is shown in figure 18. A large amount of permanent deformation has occurred in the middle of the cylinder. The local deflection at the center of the cylinder is on the order of that generally achieved in the flat modified Charpy bar samples. However, it was not sufficient to fracture the cylinder at the circumferential groove location. The fracture along a longitudinal direction is due to the very high hoop stresses generated by the internal pressure. There is a similar tendency for fracture seen along the longitudinal direction in the explosive experiments (*14–16*).

Figure 18. Cylindrical steel cylinder after pressurization test showing longitudinal fracture.

4. Discussion

The phenomenon of LME has been known for some time. Many investigations have been conducted to elucidate the mechanism by which the base metal loses ductility and becomes brittle. Reference 6 explores the complexity of this phenomenon and concludes that there is no underlying physical theory to explain the mechanism of LME. Lacking a basic theory, ARL attempted to induce the embrittlement on a trial-and-error basis that was based on approaches that have worked in the past.

Initial work began with 1038 steel samples. Variations in the test results were observed. Part of these variations might be ascribed to small differences in the treatments given the samples. For instance, figures 16a through 16c show quite different results in terms of the amount of surface wetting by the LM. These differences might be ascribed to small differences in the deflections given each sample, leading to large differences in the degree of fracture for the samples. These differences might also be ascribed to the inherent variable nature of the fracture process. Another possible source of variation was the difficulty in achieving uniform sample hardness, both within a given sample and from sample to sample. This variation was mitigated, to some extent, by switching to 4340 steel.

It was found that brittle fracture in 4340 steel modified Charpy bars could be obtained given certain conditions. The notch of the bar was coated with a gallium-indium-tin eutectic mixture, and then the surface was scribed with a sharp object. This procedure ensured that the LM was in contact with an oxide-free surface. The modified Charpy bars were then loaded quasi-statically in a test fixture. So long as the deflection was great enough, brittle fracture would occur. The fractures seen were not instantaneous. In some instances it occurred quickly (in a few sec), and

in other instances it took more than several min. The Charpy bar treated with the LM did not break in a brittle manner when subjected to the load of a drop tower, unless it was pre-strained past a critical value. The speed of the embrittlement brings into question the viability of the concept for a controlled fragmenting warhead. If it is diffusion controlled, then it may be too slow, even if accelerated with increased temperature.

If a quasi-static tensile stress is a needed to fracture 4340 steel, then the controlled fragmentation concept for a cylinder would require that the cylinder be pressurized a short time before being detonated. A small number of tests of the cylinders showed that with the particular geometry examined, there was not enough tensile stress normal to the circumferential groove to cause fracture at this location, even when filled with the LM mixture. The hoop stress dominates the material, and a fracture forms in the longitudinal direction. However, in an actual warhead that is in the configuration of a closed cylinder, pressurization might produce a higher the stress in the longitudinal direction than that for an open-ended cylinder.

The nature of the fracture in the longitudinal direction is quite different for the static and dynamic cases. Figure 18 shows a longitudinal fracture caused by a static internal pressure. It can be characterized primarily as a tensile failure. The failure mechanism of the cylinder due to explosive loading is more complex. At the early stages of the cylinder expansion, tensile cracks are formed in the longitudinal direction on the outer surface of the cylinder. As the cylinder expands further, these external tensile cracks link up to the inner surface of the cylinder by means of compressive shear failures. How LM would promote or enhance this process is unknown.

A final attempt was made to promote LME through the use of abusive grinding. The limited ad hoc study produced negative results. The work is described in the appendix.

In summary, a concept utilizing LME for the controlled fracture of a fragmenting warhead has been investigated. While brittle fracture of 4340 steel has been achieved with this approach, the conditions under which it works make a practical implementation of the process unlikely in the time scale available for the intended application. At a minimum, these conditions include need to deliver the LM to a clean, oxide-free surface and the need for a critical pre-stressing of the steel. In addition, unresolved issues, such as the speed at which the embrittlement takes place and the unknown effect of coupling the embrittling process to the manner in which the cylinder fragments, remain.

5. References

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Appendix. Additional Data

A-1. Samples

An attempt was made to understand the underlying phenomenology of liquid metal embrittlement (LME) through metallographic analysis. The photomicrographs shown in this appendix are a result of that investigation. In many cases, the photomicrographs raise more questions than they answer. They are offered here as a reference for any future works in this area.

Close examination of the fracture surfaces revealed some interesting features. Figure A-1 is a scanning electron microscope picture showing an end view of sample number 133. This sample was treated with LM and then pre-stressed. This produced a crack through a portion of the modified Charpy bar thickness. The crack does not appear to run straight along the notch bottom. Rather, it proceeds for a short distance along one side of the notch bottom and then jumps over to the other side. The intersection of the crack with the end of the bar shows the crack propagating from one side of the bottom of the notch. This may have been a result of uneven depth of scratches produced by the riffler file.

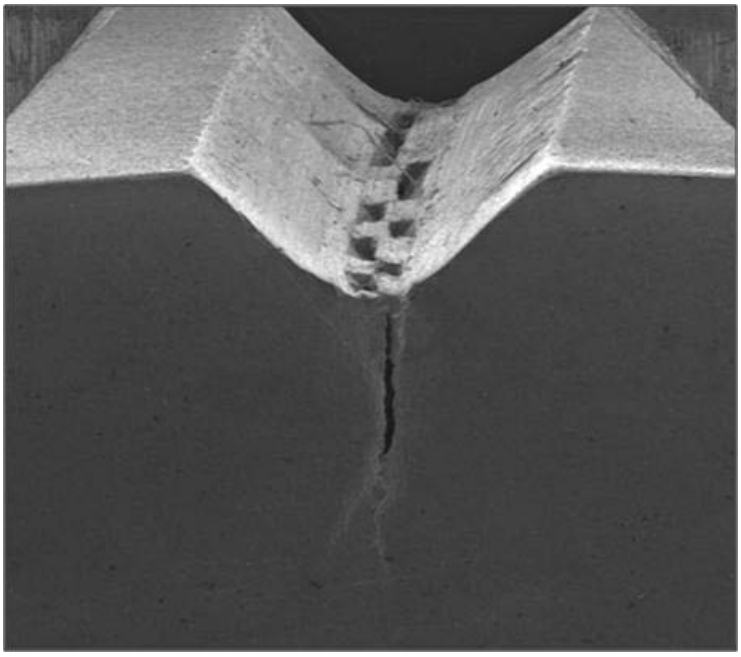

Figure A-1. Side view of notch in modified Charpy bar (sample number 133) after treatment with LM and pre-stressing (magnification 30×, back-scatter energy mode).

Figure A-2 is a photomicrograph of sample number 147. It had liquid metal (LM) worked into the notch with 150-grit sandpaper and then placed in the deflection device. It snapped at a deflection of 0.045 in. The picture shows the relatively smooth notch surface (top portion of

photograph) and the fracture surface (bottom portion of photograph). The smooth globules seen in the photograph are LM drops that are adhering to the surface of the crack.

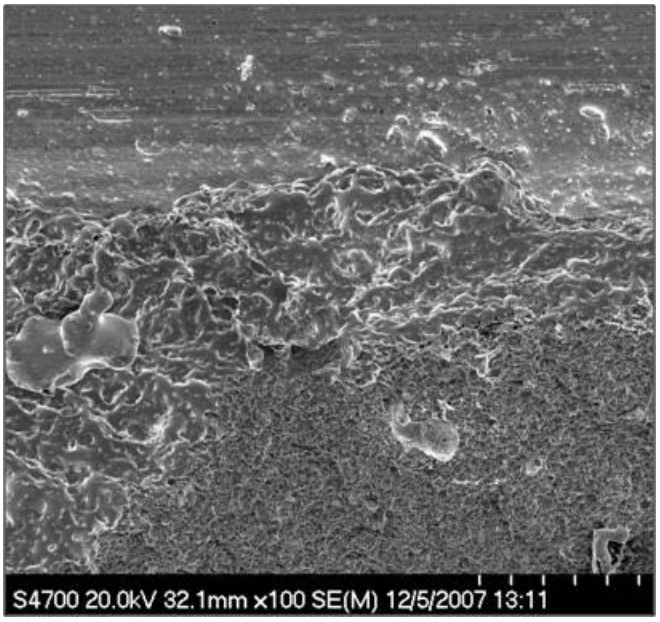

Figure A-2. Notch and fracture surface of sample number 147 (magnification 100×).

A-2. Hardness Measurements

One source of possible test variation was sample hardness. Small variations in this parameter might prove to be critical, especially in the range of HRC 40 to 50, based on the data presented in figure 11. An initial examination of the variation in hardness value from sample to sample was conducted to quantify the variation. Two measurements were made on each sample, taken from locations on the sample as shown in figure A-3. Hardness data for 19 samples are shown in figure A-4. All samples shown in this figure were made from 1038 steel. Individual measurements spanned a hardness value range from HRC 32 to HRC 48. There was almost a 10-point span of hardness for sample number 10 (127).

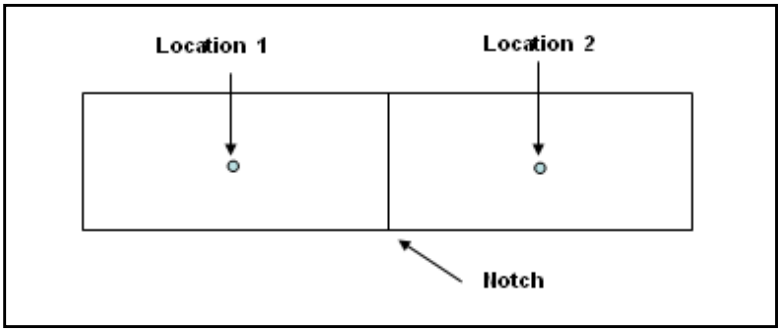

Figure A-3. Hardness measurement locations on the modified Charpy bar.

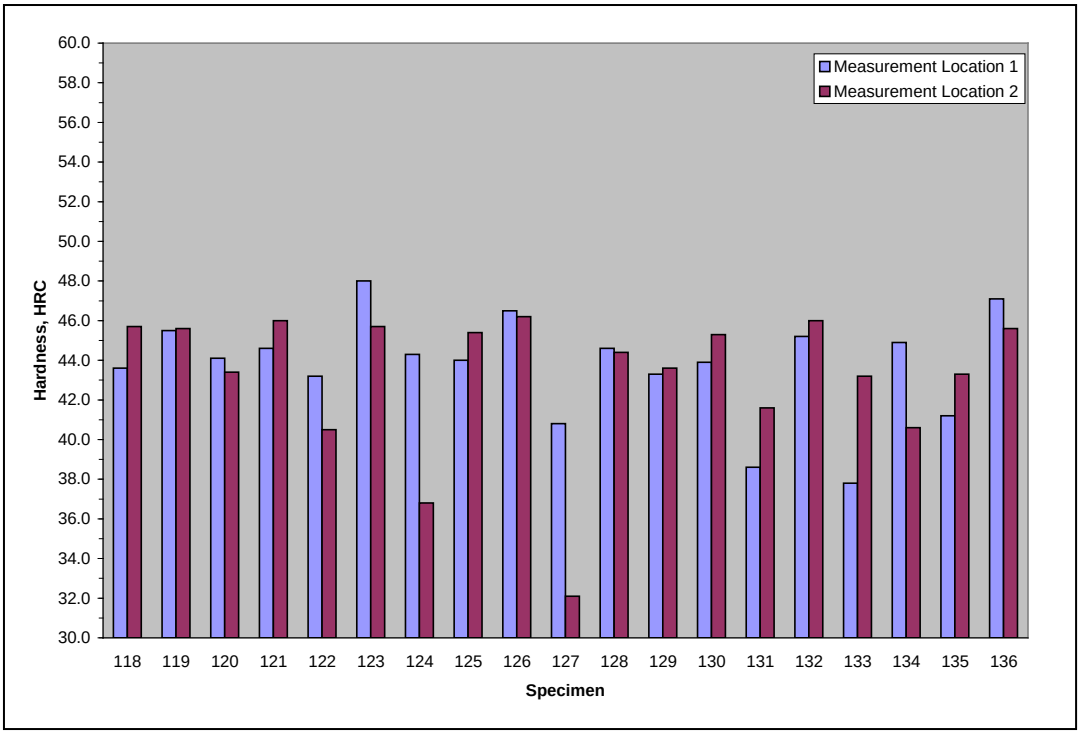

Figure A-4. Hardness data for 1038 steel sample numbers 118 through 136.

These measurements led to additional measurements on selected samples with the purpose of determining the range of hardness within a given sample. Hardness measurements were taken in close proximity of the central notch in the modified Charpy bar. Table A-1 gives the average hardness and standard deviation of the measurements. Samples were taken from two batches that had been heat-treated on different occasions.

Table A-1. Hardness measurements for selected 1038 steel samples.

Sample Number	Number of Readings	Average Hardness (HRC)	Standard Deviation
76	10	42.3	0.7
77	10	41.8	0.5
78	10	41.3	0.6
79	10	42.5	0.2
80	10	42.4	0.3
81	10	40.6	0.7
133	19	40.0	5.2
134	19	43.7	1.4
135	17	37.2	6.8
136	17	45.6	0.5

The second batch of samples (numbers 133–136) had a much higher standard deviation in the hardness measurements than the first batch (numbers 76–81). The variation in hardness in the second batch highlighted the difficulty in obtaining uniform results from heat treating 1038 steel, and the data provided the rationale for switching to 4340 steel.

More uniform hardness results were obtained when the switch was made to 4340 steel. This is shown in figures A-5 and A-6, where readings similar to those in Figure A-4 were taken. The figures represent two different batches of heat treatments.

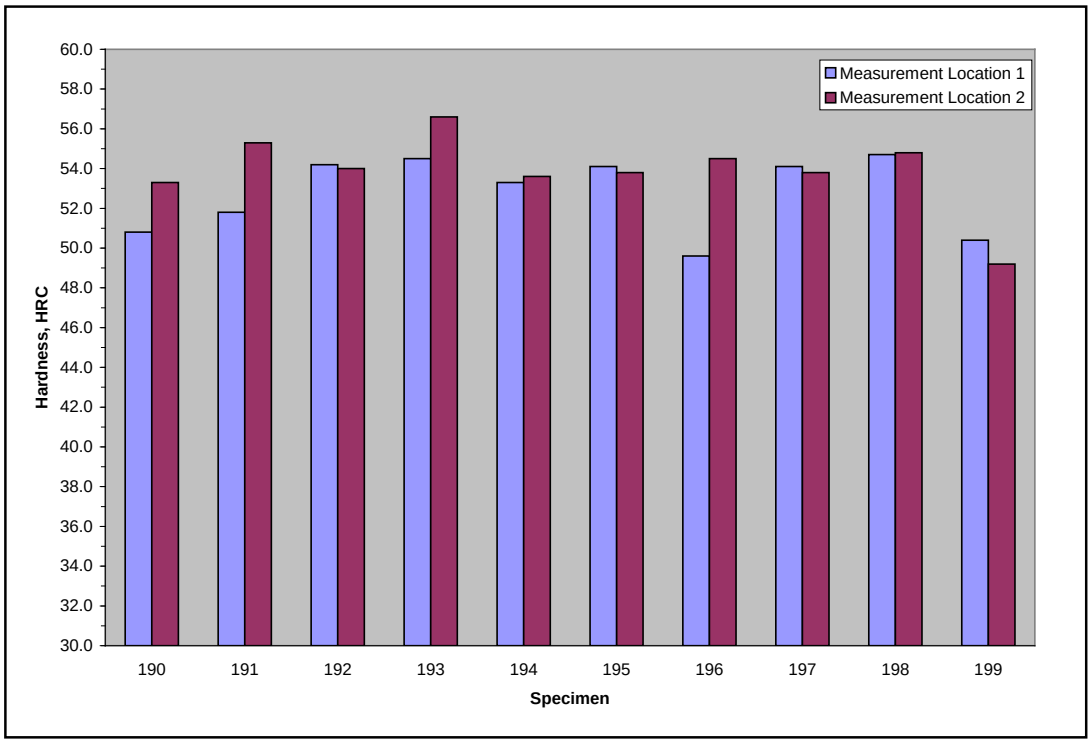

Figure A-5. Hardness data for 4340 steel sample numbers 190 through 199.

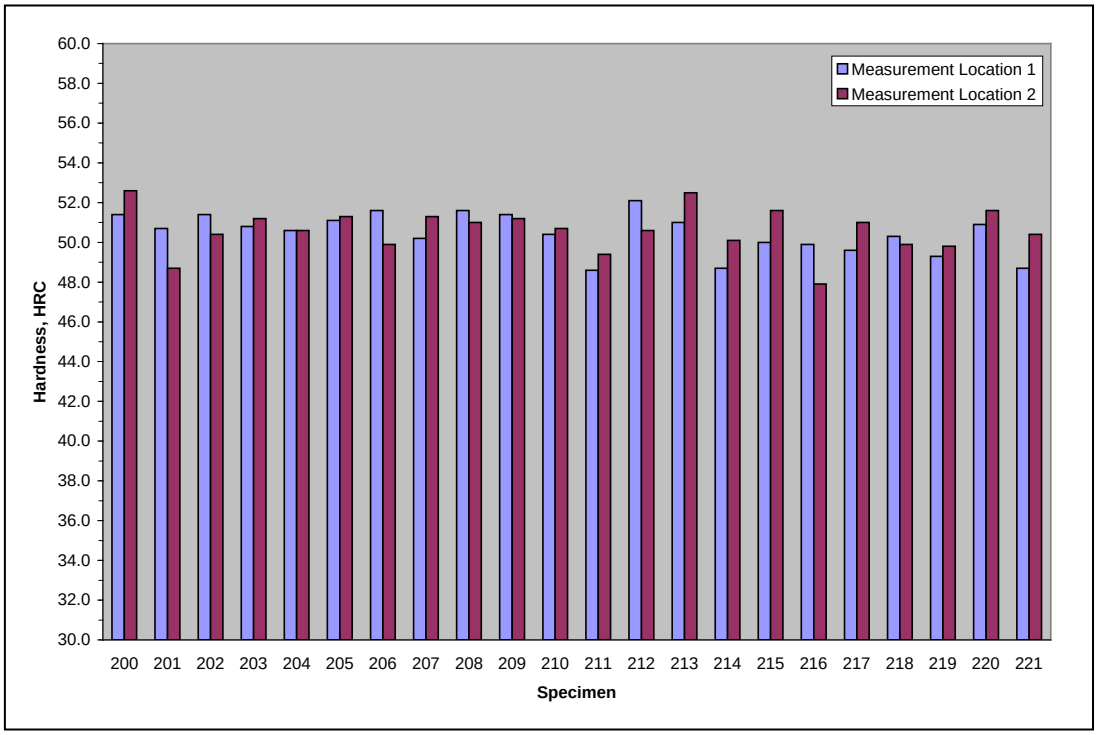

Figure A-6. Hardness data for 4340 steel sample numbers 200 through 221.

A-3. Charpy Impact Data

Tables A-2a through A-2c provide additional data for those modified Charpy bars that were tested in the drop tower. Values of impact energy and total energy have been summarized for selected samples in the body of this report. The table also shows the impact velocity of the tup, the maximum load sustained by the modified Charpy bar, and the amount of energy expended up to the time the maximum load was achieved. Notes are also provided to indicate special treatment of the modified Charpy bars.

Table A-2a. Drop tower test data, part 1.

Test Date	Sample Number	Hardness, HRC	Impact Energy, ft-lb	Impact Velocity, ft/sec	Maximum Load, lb	Energy to Max. Load, ft-lb	Total Energy, ft-lb	Sample Fractured? Yes / No
26-Sep-06	1-AN	11.3	2.41	4.65	203.68	1.62	2.47	No
26-Sep-06	2-AN	11.3	4.74	6.52	231.04	2.65	4.92	No
26-Sep-06	3-AN[1]	11.3	9.46	8.04	397.42	10.16	9.74	No
26-Sep-06	4-AN	11.3	15.59	10.33	228.50	3.18	5.35	No
4-Oct-06	11-AN[1]	11.3	15.56	10.31	809.17	14.62	16.72	No
4-Oct-06	12-AN[1]	11.3	15.56	10.31	774.82	14.98	16.76	No
10-Oct-06	13-AN	11.3	15.52	10.30	223.65	2.52	5.37	No
10-Oct-06	14-AN	11.3	15.55	10.31	222.44	2.91	6.17	No
10-Oct-06	5-PN[2]	11.3	15.49	10.29	401.43	1.59	3.22	No
10-Oct-06	7-HT	43.0	15.36	10.25	223.76	2.86	6.15	No
12-Oct-06	17-AN	11.3	31.00	14.56	234.98	2.96	5.81	No
12-Oct-06	10-HT	30.0	31.11	14.59	346.85	2.40	3.96	Yes
20-Oct-06	6-PN[3]	11.3	22.81	12.49	242.56	2.53	5.53	No
20-Oct-06	8-HT	43.0	26.90	13.56	428.20	1.73	3.13	Yes
20-Oct-06	9-HT	30.0	23.28	12.62	332.80	2.48	4.38	No
13-Dec-06	27-HT	47.0	23.39	12.65	389.67	1.05	1.48	Yes
13-Dec-06	28-HT	47.0	19.28	11.48	345.78	0.73	1.06	Yes
13-Dec-06	29-HT	47.0	11.44	8.84	377.99	0.94	1.21	Yes
14-Dec-06	30-HT	47.0	3.91	5.17	309.63	0.59	0.73	Yes
14-Dec-06	31-HT	47.0	1.93	3.63	342.51	0.80	0.91	Yes
14-Dec-06	26-HT[4]	47.0	11.84	9.00	317.57	4.67	5.01	Yes
29-Jan-07	33-HT	42.0	15.51	10.30	411.64	1.63	2.21	Yes
29-Jan-07	34-HT	42.0	11.69	8.94	411.64	1.69	2.35	No
29-Jan-07	35-HT	42.0	11.65	8.93	430.74	1.89	2.30	No
29-Jan-07	36-HT	42.0	12.42	9.22	377.30	1.31	2.34	No
29-Jan-07	37-HT	42.0	12.41	9.21	416.53	1.50	2.47	No
29-Jan-07	38-HT	42.0	13.21	9.50	416.35	1.48	1.88	Yes
29-Jan-07	39-HT	42.0	13.18	9.49	416.99	1.55	2.16	No
29-Jan-07	40-HT	42.0	13.59	9.64	424.08	1.58	2.44	No
29-Jan-07	41-HT	42.0	14.03	9.79	435.50	1.67	2.23	Yes
29-Jan-07	42-HT	42.0	13.98	9.78	425.10	1.57	2.36	No
29-Jan-07	43-HT	42.0	14.01	9.79	416.48	1.69	2.13	Yes
29-Jan-07	44-HT	42.0	13.97	9.77	417.50	1.83	2.30	Yes
29-Jan-07	45-HT	42.0	13.71	9.68	426.68	1.66	2.40	No

AN = Annealed PN = Poisoned HT = Heat-Treated

[1] Invalid test - double hit, [4] Invalid test - velocity sensor too close to impact

[3] Poisoned, 130 °C / 90 hr

[2] Poisoned, Room Temp / 90 hr

Table A-2b. Drop tower test data, part 2.

Test Date	Sample Number	Hardness, HRC	Impact Energy, ft-lb	Impact Velocity, ft/sec	Maximum Load, lb	Energy to Max. Load, ft-lb	Total Energy, ft-lb	Sample Fractured? Yes / No
15-Feb-07	48-HT	43.8	13.69	9.68	374.76	1.19	1.54	Yes
15-Feb-07	50-HT	43.8	12.46	9.23	432.58	1.95	2.48	No
15-Feb-07	51-HT	43.8	13.24	9.52	316.81	1.58	4.59	No
15-Feb-07	52-PN[1]	43.8	12.44	9.22	432.19	1.45	2.19	No
15-Feb-07	53-PN[1]	43.8	12.52	9.25	416.16	1.54	2.41	No
15-Feb-07	54-PN[1]	43.8	12.49	9.24	343.37	1.33	2.43	No
15-Feb-07	55-PN[2]	43.8	12.50	9.25	422.56	1.51	2.39	No
15-Feb-07	56-PN[2]	43.8	12.52	9.25	416.99	1.40	2.19	Yes
15-Feb-07	57-PN[2]	43.8	12.49	9.24	423.01	1.52	2.00	Yes
16-Mar-07	61-HT	43.8	13.19	9.50	424.61	1.67	2.24	Yes
16-Mar-07	62-HT	43.8	12.81	9.36	432.23	1.57	2.30	No
16-Mar-07	58-PN[3]	43.8	12.49	9.24	418.17	1.57	2.16	Yes
16-Mar-07	59-PN[3]	43.8	12.10	9.10	430.28	1.60	2.04	No
16-Mar-07	60-PN[3]	43.8	12.28	9.16	439.05	1.82	2.57	No
6-Apr-07	65-HT	41.4	13.20	9.50	400.98	1.54	2.79	Yes
6-Apr-07	66-HT	41.4	12.86	9.38	436.13	1.57	2.93	No
6-Apr-07	73-HT[4]	41.4	12.89	9.39	378.48	1.39	3.99	No
6-Apr-07	70-PN[5]	41.4	12.49	9.24	348.04	1.39	3.44	No
6-Apr-07	71-PN[5]	41.4	12.83	9.37	376.96	1.47	3.91	No
6-Apr-07	72-PN[5]	41.4	13.26	9.52	386.94	1.41	3.93	No
20-Apr-07	67-HT	41.4	13.13	9.48	407.83	1.72	3.20	No
20-Apr-07	68-HT	41.4	13.16	9.49	433.01	1.90	3.14	No
20-Apr-07	82-HT	41.4	13.15	9.48	428.26	1.96	2.87	No
20-Apr-07	85-HT	41.4	13.47	9.60	358.22	1.38	3.36	No
20-Apr-07	87-HT	41.4	13.63	9.65	414.23	1.62	2.54	No
20-Apr-07	88-HT	41.4	14.03	9.79	403.10	1.53	2.32	Yes
20-Apr-07	83-PN[6]	41.4	13.24	9.51	399.23	1.98	2.77	No
20-Apr-07	84-PN[7]	41.4	13.12	9.47	420.86	2.00	2.78	Yes
20-Apr-07	89-PN[8]	41.4	12.48	9.24	302.72	1.38	5.25	No
14-May-07	97-HT	43.1	13.20	9.50	393.70	1.95	3.99	No
14-May-07	98-HT	43.1	13.59	9.64	460.06	2.11	2.85	Yes
14-May-07	99-HT	43.1	13.21	9.50	430.67	2.01	2.52	No

HT = Heat-Treated PN = Poisoned

[1] Poisoned, Room Temp / 93 hr

[2] Poisoned, 107 °C / 93 hr

[3] Poisoned, 163 °C / 93 hr

[4] Hot Plate, 285 °C / 69 hr

[5] Poisoned, 285 °C / 93 hr

[6] Poisoned, Room Temp, Deflected 0.027" (est.)

[7] Poisoned, Room Temp, Deflected 0.035" (est.)

[8] Poisoned, Room Temp, Deflected 0.040" (est.)

Table A-2c. Drop tower test data, part 3.

Test Date	Sample Number	Hardness, HRC	Impact Energy, ft-lb	Impact Velocity, ft/sec	Maximum Load, lb	Energy to Max. Load, ft-lb	Total Energy, ft-lb	Sample Fractured? Yes / No
15-May-07	101-PN[1]	41.5	12.89	9.39	98.71	0.19	0.68	No
15-May-07	102-PN[1]	43.4	12.89	9.39	46.55	0.06	0.16	No
15-May-07	103-PN[1]	39.3	12.76	9.34	407.47	1.46	2.19	Yes
31-May-07	111-HT[2]	43.1	12.84	9.37	427.19	1.94	2.79	No
31-May-07	104-PN[2]	43.1	12.74	9.33	405.34	1.95	2.70	Yes
31-May-07	105-PN[2]	43.1	12.78	9.35	377.81	1.82	3.43	Yes
31-May-07	117-PN[2]	43.1	12.75	9.34	419.55	1.85	2.64	Yes
4-Jun-07	106-PN[3]	43.1	12.76	9.34	415.81	1.61	2.52	No
4-Jun-07	107-PN[3]	43.1	12.81	9.36	438.63	1.71	2.43	Yes
4-Jun-07	108-PN[4]	43.1	12.88	9.38	35.77	0.02	0.03	Yes
6-Jun-07	110-HT[5]	43.1	12.77	9.34	351.12	0.92	1.93	Yes
6-Jun-07	116-HT[5]	43.1	12.81	9.36	440.41	1.51	2.50	No
6-Jun-07	96-PN[6]	44.6	12.78	9.35	419.52	1.57	2.09	No
12-Jun-07	119-HT	45.4	12.90	9.39	448.93	2.06	2.47	No
12-Jun-07	120-HT	43.8	13.18	9.49	439.32	1.54	2.24	Yes
12-Jun-07	94-PN[4]	42.7	12.82	9.36	37.93	0.01	0.03	Yes
12-Jun-07	95-PN[7]	42.8	12.84	9.37	440.06	2.08	3.28	No
13-Jun-07	121- HT[8]	45.3	12.82	9.36	447.01	1.46	1.90	No
13-Jun-07	122- HT[8]	41.9	12.79	9.35	426.62	1.34	1.71	Yes
13-Jun-07	123-PN[9]	46.9	12.86	9.38	60.59	0.07	0.21	Yes
13-Jun-07	124-PN[10]	40.6	12.84	9.37	50.51	0.07	0.22	Yes
13-Jun-07	125-PN[11]	44.7	12.85	9.37	38.39	0.01	0.03	Yes
13-Jun-07	126-HT[8]	46.4	12.82	9.36	469.45	1.61	2.23	No
13-Jun-07	127-HT[8]	37.5	12.86	9.38	359.82	1.39	2.20	Yes
13-Jun-07	128-HT[8]	44.5	12.74	9.33	447.72	2.09	3.00	No
14-Jun-07	129-PN[4]	43.5[10]	12.68	9.31	36.42	0.02	0.03	Yes
14-Jun-07	130-PN[9]	44.6[10]	12.85	9.29	51.59	0.06	0.14	Yes

HT = Heat-Treated PN = Poisoned

[1] Riffler Filed, Poisoned, 100-110 °C / 30 min, Deflected 0.050" (est.)

[2] Chemically Etched, Poisoned, 100-110 °C / 30 min, Deflected 0.050"

[3] Riffler Filed, Poisoned, 100-110 °C / 30 min, Deflected 0.050"

[4] Riffler Filed, Poisoned, 100-110 °C / 30 min, Deflected 0.060"

[5] Deflected 0.060"

[6] Riffler Filed, Poisoned, Room Temp, Deflected 0.060"

[7] Chemically Etched, Poisoned, 100-110 °C / 30 min, Deflected 0.060"

[8] Riffler Filed, 100-110 °C / 30 min, Deflected 0.060"

[9] Riffler Filed, Poisoned, Hot-to-touch, Deflected 0.060"

[10] Riffler Filed, Poisoned, Hot-to-touch, Deflected 0.055"

[11] Riffler Filed, Poisoned, Room temp / 30 min, Deflected 0.060"

A-4. Abusive Grinding Tests

It is known that abusive grinding of steel will result in a region near the surface that exhibits a tensile stress (*18*). It was reasoned that this tensile stress would promote LME. In addition, the abusive grinding would also eliminate the need for pre-stressing the modified Charpy bar in a test jig. Consequently, a special grinding wheel was purchased from Saint Gobain Abrasives, Worcester, MA. The wheel's cutting edge had an included angle of 90° and used cubic boron

nitride (CBN) as the cutting agent. This design allowed the cutting of a groove with the same depth and angle as in the modified Charpy bars used for the main part of this study.

A batch of modified Charpy bar blanks was heat-treated to HRC 50.6. The grinding wheel was then used to make the notch at the center of each bar. The notch was made with a single pass with a wheel speed of approximately 6000 rpm. The LME mixture was applied immediately after the grinding so as to prevent oxidation of the newly-ground surface. No cracks or sample separation were observed on any of the modified Charpy bars.

There was some concern that the procedure for grinding the notch was not abusive enough. Consequently, it was decided to try slower wheel speeds. A second batch of modified Charpy bar blanks was obtained with an average hardness of HRC 46. The parameters associated with this batch are shown in table A-3. After four bars were ground, some damage to the cutting wheel was noted. A fifth bar was produced, but in attempting to make the sixth bar the CBN came off the grinding wheel and was embedded in the sample. As before, the LME mixture was applied immediately after grinding. Again, no cracks were observed on any of the modified Charpy bars.

Table A-3. Grinding parameters.

Sample Number	Wheel Speed (rpm)	Depth of Cut (in)	Travel Speed (in/min)
1	100	0.025	20
2	100	0.025	10
3	100	0.025	5
4	50	0.025	5
5	50	0.025	10
6	50	0.025	20

The modified Charpy bars were tested in a drop tower according to the procedure presented in section 2.1. A baseline was established by testing three samples (numbers 222, 223, and 224) from the first batch that had been ground but not treated with the LME mixture. Next, five samples from the first batch that had been treated with the LME mixture were tested (numbers 225 through 229). Finally, five samples (numbers 1 through 5) from the second batch were tested. The energy absorbed by each sample is shown in table A-4 along with the wheel speeds.

Table A-4. Drop tower test results for abusively-ground samples.

Sample Number	Wheel Speed (rpm)	Absorbed Energy (ft-lb)
222	6000	2.89
223	6000	3.20
224	6000	3.16
225	6000	11.39
226	6000	3.08
227	6000	2.89
228	6000	2.62
229	6000	3.12
1	100	2.61
2	100	2.47
3	100	2.12
4	50	2.53
5	50	3.32

The absorbed energy for the baseline samples was in the same range as that of Groups B through E (~2–3 ft-lb) as seen in figure 17. The same can be said about the remaining samples. None of the treated samples achieved the reduction in absorbed energy demonstrated by the Group A samples. Consequently, this limited study of abusive grinding produced negative results.

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