Hydrogen Compatibility of Structural Materials for Energy Storage and Transmission Applications

(Semiannual Report for Period Through October 1, 1976)

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HYDROGEN COMPATIBILITY OF STRUCTURAL
MATERIALS FOR ENERGY STORAGE
AND TRANSMISSION APPLICATIONS
(Semiannual Report for Period Through October 1, 1976)

Sponsored by
ERDA Division of Energy Storage Systems

Compiled by
S. L. Robinson

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SUMMARY

Substantial support activities for Brookhaven National Laboratory (BNL) have been completed since the initiation of this program. We have assessed the suitability of commercial alloys for containment of hydride-dehydride reactions, and recommendations for materials selection based upon tensile and slow crack growth tests have been made. We have also prepared and installed in a test chamber at BNL a series of in-situ test specimens to be exposed to a cyclic iron-titanium hydride environment. Future BNL support activities will include welding/joining specification development and a post-mortem examination of the in-situ test specimens.

As our direct support of the Brookhaven National Laboratories hydride program diminishes, our efforts are becoming more oriented to materials development and to the development of specifications for the use of structural steels in hydrogen environment. Eighteen months of work have yielded several advances in materials compatibility, protective coatings, and experience in practical assembly and specification problems. Along with a summary of past and present work, we will address the question of what directions the continuing work should take.

1. Brookhaven National Laboratories' hydride storage program has been supported during the last six months by supplying 80 self-loaded tensile specimens for accelerated testing in an FeTiH_x test bed. The welding specification for the experimental pipeline was forwarded to BNL engineers for their information with the understanding that it is preliminary in nature.
2. Unexpected delays in assembly of the hydrogen pipeline have extended the date for full pressure operation to November 30. Components were found to contain unacceptable flawed welds. This general noncompliance went undetected by a commercial inspection firm. To guarantee personnel safety, we are repair welding and inspecting pipeline components at Sandia rather than at commercial vendors.
3. An experimental alloy test matrix has been designed, and preparation of heats is beginning. The test matrix to be applied is intended to optimize the hydrogen compatibility of relatively low-cost, low-strength steels while maintaining or increasing initial strength levels.

4. A preliminary welding specification for containment of hydrogen in structural mild steels has been developed, and we expect to improve this specification with data produced in current and future studies. A welding program is being developed to improve the compatibility of mild steels weldments. The program will focus upon the heat affected zone microstructures and weld metal chemistry. We plan to assess the compatibility of welds produced by joining methods such as electron beam, laser fusion, plasma arc, and explosive welding, in addition to the multipass metal arc processes.
5. Hydrogen permeation resistant coatings, applied by pyrolysis of silane, and by brush electroplating, have been developed. Studies of plasma spraying are underway to develop more rapidly and inexpensively applied permeation barriers. Self-loaded specimens with brush electroplated coatings were designed and prepared for accelerated hydride-dehydride cycling in an FeTiH_x bed at Brookhaven National Laboratories. Subatmospheric testing of hydrogen permeation inhibition by minor gaseous constituents was unsuccessful and has been discontinued. We are considering the initiation of high pressure permeation tests at Savannah River Laboratories.
6. Environmentally assisted, cyclic fatigue in high pressure hydrogen has been identified as a potential hazard to the integrity of flawed pressure vessels in materials where slow crack growth under constant load is not expected. A re-analysis of inspection requirements and of proof testing procedures is suggested. Experimental studies of cyclic fatigue in hydrogen, and leak before burst designs, are therefore indicated for flawed structures such as weldments. These studies would provide quantitative data to designers, and inputs to a comprehensive code for assembly and inspection of mild or structural steel pressure vessels for hydrogen containment. The problem of the growth of flaws in elastic/plastic materials, and of flaws growing through or emanating from arrays of defects will be treated through computer calculations, in order to guide experimentation and provide support for establishing codes.
7. Our efforts during FY 1976 will be apportioned as follows:

a. Experimental pipeline operation experimentation	~40%
b. Materials and hardware procurement	~15%
c. Welding development	~10%
d. Advanced mechanical testing	~15%
e. Advanced development of alloys	~15%
f. Brookhaven National Laboratory Support	~ 5%

INTRODUCTION

The intent of this study is to advance the safe and reliable utilization of structural materials in energy systems. Based upon testing of commercial materials, we have made recommendations to Brookhaven National Laboratories (BNL) concerning large scale containment of iron-titanium hydride reactions. In addition, we are modifying existing structural steels and joining methods with the purpose of optimizing hydrogen compatibility. Prototype testing of experimental materials and assemblies will be carried out in an experimental hydrogen pipeline now under construction.

We have completed most of the initial studies planned for the BNL hydride storage program. Slow crack growth tests have been conducted and the analysis is nearing completion. Tests of the potential damage to structural materials resulting from direct hydrogen damage by intimate contact with FeTiH_x have been completed. To allow assessment of long term materials behavior during accelerated hydride-dehydride cycling in an FeTiH_x bed, a series of self-loading tensile specimens have been supplied to BNL and placed in a test chamber. Following the six month test cycle planned, post-mortem analysis will be performed at Sandia Laboratories. During this test period at BNL, we will be concentrating on making the pipeline loop operational and pursuing materials development activities.

The date for full operation of the experimental pipeline loop has been delayed until November. While the reasons for this delay will be discussed in detail later in the text, they center about unsatisfactory welding of the initial components and our need to guarantee personnel safety prior to loop operation.

Several supportive materials programs have been initiated. These programs include: i) modifications to the chemistry of structural materials, ii) development of thermomechanical treatment (TMT) schemes for existing alloys, and the coupling of TMT and alloy modification, iii) welding development and specifications, iv) continued development of coating schemes for protection of vulnerable structures, and v) advanced mechanical testing.

Much data and experience have been accumulated during the past 18 months concerning problems associated with the use of structural steels for hydrogen containment. Much work remains to be done, and from an engineering point of view the next few years will be important in achieving a data base for developing service codes. A demonstration of hydrogen supplementation of

natural gas is scheduled to occur in 5 years*. In this application both pure hydrogen and natural gas-hydrogen mixtures must be handled safely at intermediate and possibly high pressures. Therefore, as we report the specific results of our studies, it is appropriate to consider the nature of future efforts needed to optimize the performance of structural materials for hydrogen service. Both cost and proven field performance of engineering structures will be important success parameters for developing efforts. A summary section will attempt to place in perspective to the general problem, the work completed and planned for this program.

HYDRIDE STORAGE SUPPORT

The tensile test program for screening of candidate structural alloys has been completed, and the results were presented in report SAND75-8040, covering the period through June 30, 1975. Additional tensile tests of the A516 and A106 in intimate contact with FeTiH_x have been completed, and those results were summarized in report SAND76-8206, for the period through January 15, 1976. For completeness, these results have been included in this report as Tables I and II, and Figures 1 and 2 respectively. Analysis of tensile ductility losses in H_2 , comparative fractographic studies, notch sensitivity studies, behavior of weld metals in H_2 , and weldability studies indicate that alloys A516-grade 70 and A106-B are suitable low cost structural steels for the intended application. Tensile tests in FeTiH_x indicated that intimate contact with FeTiH_x degrades ductility to about the same extent as does 1 atmosphere of pure H_2 gas. Therefore, hydride contact presents no apparent difficulties during short term (72 hours) exposure.

The next step planned in this alloy qualification program was to investigate the possibility of slow crack growth under conditions of 4.14 MPa H_2 pressure and high stress intensity. The desired goal was to find a threshold stress intensity (K_{TH}) below which slow crack growth would cease. This stress intensity value would then dictate non-destructive inspection (NDI) criteria. The extended compact tension specimen (discussed in SAND75-8040) was used for these tests, and the test procedure was discussed in SAND76-8206. Welded and base metal specimens of 2 1/4Cr-1/2Mo, AISI 4340, A515 Grade 70, A516 Grade 70, A106-B, and C-95 have been exposed to 4.14 MPa H_2 for periods as long as seven months. The strength levels and air tensile test properties of these materials are summarized in Table III. The strength levels are well above guaranteed minima, except for the 4340 alloy which was annealed intentionally to a low strength to minimize embrittlement. Some indications of crack growth were obtained from the remote monitoring of the attached strain gauges while the specimens were in test, but these indications were later found to be due to temperature variations. Overall performance of the monitoring system was good. Upon removal from the test chamber, specimens were fatigued to mark existing slow crack growth zones, fractured and examined. No slow crack growth was observed in welded or unwelded specimens of A515 and A516 Grade 70, and A106-B to stress intensities (K_I) of 88 MPa $\sqrt{\text{m}}$.

*Two of the authors, S. L. Robinson and H. J. Saxton are members of the Ad Hoc Committee on Hydrogen as a Supplement to Natural Gas.

Clear interpretation of the above results is hampered by uncertainties related to the possible existence of incubation periods prior to the beginning of crack growth. Extended compact tension (EXTCT) specimens are loaded to a predetermined K_I value in air, placed in a pressure vessel, and then exposed to high purity H_2 gas (after cyclic evacuation and back filling to ensure the purity of the H_2 gas). Loading in air opens the fatigue precrack and allows an oxide layer to form at the crack tip. This thin oxide layer can act as a hydrogen permeation barrier, and possibly extend the incubation period before crack growth will begin. No feasible and economic testing method exists now which allows precracking, loading, and transferring of EXTCT specimens to a long term test chamber while maintaining a H_2 or inert environment. Incubation may also be caused by small overload of 1 or 2 $MPa\sqrt{m}$ required to insert the loading wedge. The temporary overload produces initially a slightly larger plastic zone at the crack tip, and the resulting compressive residual stresses can delay the onset of slow crack growth and slow the growth rate through the affected region.

Based on these possibilities, it can be argued that the lack of slow crack growth simply represents long incubation periods. One could disprove this argument by waiting a time equivalent to the service life of the structure being planned. This approach certainly is impractical for the applications considered for this program. In addition, no accepted theoretical model exists for calculating incubation times. Rice's discussions⁽¹⁾ of the phenomenon might lead us to believe that incubation times are relatively short for mild steels. A simple diffusional model for hydrogen permeation through an oxygen monolayer suggests that significant hydrogen contents can be reached near a crack tip in a matter of days. While atomically thin layers of iron oxide or adsorbed oxygen in mild steels may not greatly retard hydrogen permeation, coherent oxide layers of low hydrogen diffusivity (e.g. Al_2O_3 , or Cr_2O_3) can produce significant permeation rate decreases⁽²⁾. Since mild steels (A106 and A515 in our case) do not form permeation barrier type oxides, long incubation times should not result in delayed hydrogen transport to the crack tip region.

In addition, we believe that the second possible cause of crack growth incubation, a 1 or 2 $MPa\sqrt{m}$ overload, is far outweighed by the direct effects of crack blunting produced by loading specimens to stress intensities between 60 and 88 $MPa\sqrt{m}$. It can be argued for the low strength materials tested in this study (yield strength <690 MPa) that high degrees of plasticity blunt the crack tips of the slow crack growth specimens. The blunting reduces the effective stress intensity imposed on the EXTCT, and therefore its susceptibility to slow crack growth. Nelson (5,6) has loaded standard compact tension specimens of SAE 1020 steel in pure H_2 (1 atmosphere) and observed no sustained load cracking to $K_I = 50 MPa\sqrt{m}$, but observed cracking immediately upon beginning fatigue cycling. Fatigue cycling can sharpen the crack tip on every cycle, and thus crack growth occurs.

Our test observations and the above discussion lead to the following tentative conclusions. First the lack of slow crack growth observed in the steels tested either results from an inherent resistance to hydrogen assisted crack growth or from a reduction in effective stress intensity due to large scale yielding about the crack tip. While crack blunting would occur in

actual containment structures during proof testing, any load cycling during later system operation might lead to crack sharpening and environmentally assisted fatigue crack growth. Fatigue growth will be discussed in greater detail in the section titled Advanced Mechanical Testing. Several possible extensions of the work are discussed in that section.

As part of our support program at Sandia, we have designed, fabricated and loaded 80 preloaded tensile specimens for in-situ testing at BNL. These samples were delivered and installed in the hydride test reactor during the week of April 9, and accelerated testing designed to simulate in-service hydride-dehydride cycles has begun. Table IV summarizes the matrix of specimens under test. Post mortem analyses will be performed at Sandia after the six month test cycle is completed.

Drs. H. J. Saxton and S. L. Robinson of Sandia attended the 1st Annual Conservation Division Hydrogen Energy Contractors Meeting at BNL, held April 26 and 27. At that time, an informal discussion was held with BNL personnel concerned with the design, assembly and non-destructive inspection specifications for a large scale hydride containment vessel. As a result of the discussion, we have supplied to BNL the welding specifications and non-destructive inspection requirements used in the assembly of our experimental hydrogen pipeline. We have emphasized that the specification should be viewed as being conservative in nature, and that it will probably evolve as our experience in hydrogen compatibility of welded mild steels grow. The specification is discussed briefly in the following section, and the complete specification is reproduced in the Appendix.

PIPELINE LOOP CONSTRUCTION

The experimental hydrogen pipeline will circulate high pressure hydrogen at moderate rates through mild steel pipe. Specific materials experiments will be conducted in modules inserted into the line. At the time of the last report the components were in fabrication, e.g. the pump housing and the flanged pipe segments that connect the experimental modules. Initial operation was scheduled for the month of July. While the components were received to meet the schedule, further non-destructive examination at Sandia has revealed rejectible weld defects requiring either repair or replacement.

The pipe sections were welded by a commercial pipe manufacturing company by ASME certified welders and inspected by a commercial non-destructive inspection (NDI) firm. The specification was accepted by the manufacturer, and the inspection firm stated that the welds were sound after minimal repairs were completed. Spot examination of the welds by Sandia's NDI personnel detected deviations from the specifications and subsequently a 100% inspection was performed. A significant number of deviations from the specifications was subsequently detected during careful X-ray examination.

Prior to discussing the actual defects, it is desirable to list some of the high points of the specification and the reason for their existence. The principal points are:

1. No lack of penetration or incomplete fusion is allowed.
2. No cracks are allowed.
3. A minimal porosity is allowed (the charts and source are contained in the specification).
4. The inclusion count and spacing are stringently controlled.
5. NDI inspectors must attain documented proficiency levels.
6. Welders are ASME certified.
7. Low hydrogen electrodes as per AWS-E7018 are used.
8. Only certified (known chemistry) materials are used.
9. Post weld stress relief is specified.
10. All radiographs are to be performed to a 2-2T level.

Points 1 thru 4 concern the existence, initiation and potential growth of cracks, and reflect our incomplete knowledge concerning the behavior of certain flaws in hydrogen, e.g. porosity. One basic reason for building the pipeline is to facilitate gathering such data. Items 5 and 6 are required by the USA Standard Code for Pressure Piping, as is Item 2. Items 7 and 8 are our own additions and are intended to produce low residual hydrogen levels and to control the materials used. Item 9 is USA Standard Code and a legal requirement for materials with carbon equivalents $(C + \frac{1}{4} Mn) > 0.65$ w/o or ladle carbon analysis of 0.32 w/o or greater. The sensitivity level of the X-ray analysis is required to meet a specific quality level as per the ASME Boiler and Pressure Vessel Code - section V article 2 with some minor alternations to suit the specific circumstance.

X-ray analysis performed by Sandia personnel indicates non-compliance to the specification in about 75% of the welds. A variety of out-of-specification defects were observed. Drop-throughs in excess of 50% were observed in several cases, large numbers of cracks were found, voids of greater than 20% of wall thickness were observed, and the porosity levels were significantly out of specification in several cases. A detailed inspection report of the defects has been prepared by the Sandia NDI examiner. To initiate this evaluation process we evaluated the image resolution of the radiographs taken by the commercial NDI firm and found that they did not meet the 2-2T quality required. It can be concluded clearly that the commercial inspection was unsatisfactory. Whether poor quality commercial NDI is widespread is not known, but certainly this inspection was inadequate.

Because of possible parallels to actual service conditions, the source of this noncompliance to the specification needs to be pinpointed. In particular it is desirable to determine whether the difficulty resides in an overly demanding specification, poor welding performance by the commercial pipe company, or unsatisfactory performance by the NDI firm. Given this uncertainty in the quality of commercial NDI inspection, it is even more difficult to form conclusions about the day-to-day quality of commercial pipeline welds. A case in point is the existing controversy over the inspection of the Trans-Alaska pipeline(3,4). It is hoped that our efforts to

produce high quality pipeline welds and the experimental test programs will provide a realistic commercial welding specification consistent with reasonable construction costs and highly reliable hydrogen service.

In the meantime, it must be emphasized for the experimental pipeline at Sandia that every effort is being made to assure personnel safety. This safety consideration led us to inspect the commercially certified welds and decide to repair or replace pipeline components as needed. Weld repair, new welding, and final NDI will be performed at Sandia. Because of the above considerations, we do not expect full operation of the pipeline to begin until November 30.

MATERIALS DEVELOPMENT WORK

i) Alloy Modifications

The base material selected for the alloy modification work is A516 grade 70, a low strength steel being evaluated in the hydride storage program. The inclusion content of the material is relatively high when compared to high performance steels, with sulfur analyses as high as 300ppm. Existing primarily as manganese sulfide in A516, this sulfide inclusion exhibits high ductility during high temperature industrial processing (e.g., hot rolling). The inclusions can, therefore, elongate and form long planar particles along parallel planes (type II sulfides). Because the particles are less ductile at ambient temperatures, they can reduce ductility in the short transverse rolling direction.

The addition of rare earth elements to steel to form rare earth oxysulfides may diminish the inclusion elongation problem. The rare earth oxysulfides are less plastic at processing temperatures and separate to form small rounded particles rather than elongated stringers. This rare earth modification (REM) is not expected to reduce the existing volume fraction of inclusions*, but provides instead inclusion shape control (ISC). The first beneficial effect of ISC is the prevention of long cracks along sulfide/matrix interface area that accompanies particle size refinement at constant volume. This increase in internal surface area may function as a "trap" for the collection of hydrogen. Whether such traps are innocuous is a point of contention in theories of hydrogen embrittlement. However, increasing their number will reduce the internal pressure in any given void, thus reducing the growth rate of the voids⁽⁸⁾. Significant success in alloy modification has been achieved at Sandia by the application of trapping concepts to high performance stainless steels⁽⁸⁾. The third potential benefit is the interaction of particle morphology and processing parameters.

*Desulfurization of steel by REM is a ladle technique in which the removal of RE oxysulfides and RE sulfides is assisted by the steel making slag⁽⁷⁾. We will attempt the REM desulfurization of steel later in the program.

The preceding discussion has dealt with the potential benefits of inclusion shape control; an alternate course is to lower the inclusion content. In a steel such as A516, a large volume of inclusions exists; and if the inclusions strongly reduce hydrogen compatibility, a significant improvement should be produced. Obtaining very low inclusion content steels, such as A516, on a commercial scale is probably not feasible using present practices. For economic reasons, REM normally is not applied to low performance materials, but rather to high strength low alloy steels and to the current generation of microalloyed line pipe steels(7,11,12). For laboratory experiments, we will initially attempt to desulfurize A516 by electro slag remelting. Hydrogen performance results for this steel will then be compared with unmodified A516 steel. These experiments should help reduce the conflict existing in the literature concerning the effect of ISC(14,17,18,19) versus desulfurization(15,19) on hydrogen compatibility.

The matrix of alloy heats to be examined is summarized in Table V. Titanium(9,10), silicon(9,10), and copper additions have been included because preliminary work has suggested beneficial results. Our test matrix, therefore, includes these elements as additions to the A516 base metal. Rare earth additions and other alloy elements have been purchased, but significant delays in scheduling the casting and processing have been encountered. The casting of these experimental heats has been rescheduled and partial completion is expected near the end of November. Further modifications to the matrix and to the processing will be made as experience is developed.

A number of hydrogen embrittlement tests are applicable to assessing the compatibility of our alloys. The details of fracture mechanics considerations bearing upon the choice of tests are discussed in the Advanced Mechanical Testing section. Screening tests will be performed, consisting of smooth and notched tensile bars. Cyclic environmental fatigue tests will be either added to the screening tests, or used as a later qualifying test. Impact testing of the alloys is indicated as a necessary test. Current pipeline specifications (supplemental requirements of the API pipe specifications) calls for both the Charpy test, and ductile-brittle transition temperature measurement by the drop weight tear test (ASTM A436). The latter test will not be performed. Because of the inverse strain rate dependence of hydrogen embrittlement phenomena, the Charpy test probably does not need to be conducted on hydrogen charged material.

The goal of the chemistry modification is to produce more hydrogen compatible microstructures. For example, phase morphology, grain size and microchemistry are known to affect hydrogen compatibility, as pointed out by Thompson and Bernstein(9,16) in several papers. Thermomechanical treatment discussed in the next section has the same goal.

ii) Thermomechanical Treatment

Thermomechanical treatment studies reported previously in SAND76-8206 dealt with the high energy rate forming (HERF) of 1018 steel, and warm working of both high carbon (1.3 w/o C) and mild steels A515 and A516. These procedures modify the microstructures and hence the compatibility of the steels.

HERFing of 1018 steel produced high strength levels, but ductility losses were significant. The warm worked high carbon (1.3 w/o C) steel did not exhibit significant improvements in compatibility at high strength levels, and the testing variability made interpretation of the results difficult. Neither effort is being continued.

Warm working of the mild steels A515 and A516 produced significant improvements in strength and hydrogen compatibility (assessed by tensile testing). The tensile test data for the warm worked steels vs., as-received (mill finished) steels is summarized in Figure 3. The ductility (as % reduction of area) increased by 60% for A515 and by 30% for A516, as a result of warm working. Yield strengths were increased by about 50% and ultimate strengths by about 20% by warm working.

These modified materials appear to have a significant potential for application to hydrogen containment problems. It is our intent to assess their potential by performing further development work. Critical to this development work is a knowledge of i) the sources of the improved hydrogen compatibility, ii) the interactions of thermomechanical treatment and alloy chemistry, and iii) factors which might limit the acceptance of the finished product. These points will be discussed below.

Literature surveys by Thompson and Bernstein^(9,16) have summarized microstructural features which improve hydrogen compatibility in ferritic alloys, including mild steels. Fine grain size, large numbers of low angle boundaries, texturing (under proper orientation conditions), fine particle distributions of appropriate geometries, and reduction of lamellar pearlite were identified as desirable. Other authors^(14,15) have identified non-metallic inclusions as a source of fracture initiation and cracking in the high strength line pipe steels such as API-5L-X65, which are chemically similar to A515 and A516. Banded structures, in which pearlite and ferrite exist in alternating layers, and in which segregation of manganese exists, have been identified as a less preferred microstructure⁽²⁰⁾. Cracks are thought to follow the zone of high Mn concentration which is the pearlite band. This is especially troublesome since high bulk levels of Mn (added for solid solution strengthening) may increase hardenability in the segregated zone, producing a preferred cracking path. These banded structure segregation patterns have been stated^(20,21) to persist throughout heat treatment and hot working procedures.

The microstructures of A515 and A516, prior to and after warm working are shown in Figure 4. Prior to warm working (mill finished condition) A516 is banded while the A515 is not. After warm working by rolling, the grain size has decreased from 35μ to less than 7μ , a texture is observed, a reduction in the amount of lamellar pearlite has occurred (including some spheroidization in the A515), and low angle boundaries appear to be present in both steels. Thus, a significant number of the desirable features listed by Thompson and Bernstein are present in the microstructures produced by warm working.

The fracture surfaces of the warm worked vs. mill finished A515 and A516 are reproduced in Figures 5 and 6. Significant differences exist between the A515 and A516 when tested in hydrogen, more so in the warm worked than in the mill finished materials. The fracture surfaces of the warm worked materials after testing in hydrogen show more dimples than their hot worked counterparts. The fractures are still quasi-cleavage fractures in which cleavage occurs ahead of a crack and the matrix then tears back by ductile processes.

In the warm worked materials, the A516 has clearly defined cleavage zones, separated by dimpled tear zones. The A515 does not display the clearly demarked fracture zones, but instead the cleavage and ductile zones are on a smaller scale and are more intimately interrelated. A number of microstructural features may be responsible for the differences between the warm worked steels, and for the compatibility improvement over that of the hot worked steels. Several of our initial results are discussed below.

We have investigated the effects of warm working on chemical segregation of the manganese, particularly as related to banding, and the subsequent redistribution caused by warm working. Figure 7 summarizes the results schematically. Segregation of manganese is found in both the mill finished and warm worked steels. Warm working decreased the degree of segregation and moved manganese into the bulk ferrite of the A516. Microprobe analysis is not yet complete for A515. Analysis of manganese segregation patterns may yield information concerning preferred crack paths. Analysis of tramp element segregation to prior austenite boundaries will also be attempted. The crack paths in the warm worked A515 were considerably more random; both microstructure and prior microsegregation are suspected to play a role. Patterns of Mn segregation may exist^(20,21) in the warm rolled steels; a reanalysis over larger areas typical of the prior segregation pattern will determine the presence of this segregation pattern. Tramp element segregation at prior austenite grain boundaries also may have a significant effect in the warm worked steels. This work is continuing in order to separate microstructural effects from chemical segregation effects.

The possible interaction of alloy chemistry and thermomechanical treatment has already been considered in the alloy matrix previously listed in Table V. Separate heats of the appropriate alloys will undergo both hot working and warm working schedules to test the applicability of REM to thermomechanical working as a means of increasing hydrogen compatibility.

Some of the factors which might limit the commercial acceptance of a warm worked product are listed below. First, mill capacity might limit the available thicknesses of product material and thus thick section applications may be impractical. Second, anisotropy of strength or ductility properties would require special design considerations. For example, a structure might be vulnerable to impact in a particular orientation. Third, joining of these materials may be difficult and exacting; this subject is discussed in the next section of this report. Fourth, no data are available concerning the impact energy to produce splitting in the short transverse rolling direction. The inventor of the warm rolling process believes that short transverse splitting will not be a problem⁽²²⁾, but any conclusions await experimental results. We will assess these potential limitations during future studies.

iii) Welding Development

Some tensile testing of welded mild steels was performed as part of the BNL support work previously reported. The slow crack growth behavior of welded mild steels has also been studied, and the results reported in the first section of this report. A preliminary specification for the welding of mild steels has been developed and applied to the experimental pipeline. The weld tensile test results will be discussed in order to illustrate several points. In the hydrogen environment (4.14 MPa H₂) tensile fractures in weldments occurred primarily in heat affected zones (HAZ) or in fusion zones (FZ) very near to the HAZ-FZ interface. The fracture surfaces usually exhibited both ductile dimples and cleavage. When tested in hydrogen, sound welds exhibited % RA's nearly equivalent to the unwelded metals, although the uniform elongations decreased. Welds displaying untypical fracture surfaces usually contained several medium sized inclusions. The A515 weld shown in Figure 8 is an example. A 44% RA was obtained, with 50% cleavage on the fracture surface, and three inclusions of about 150-200 μ m diameter which initiated large amounts of cleavage. Analysis by non-dispersive X-ray on the scanning electron microscope disclosed high silicon and calcium levels, confirming that the particles were from the weld rod flux. Particles about 1mm apart produced intersecting cleavage zones and cleaved fully 40% of the cross section and area while 2mm spacing did not produce interacting cleavage. Thus, the spacing of such inclusions is important to the fracture surface appearance. However, the measured tensile ductility was not appreciably decreased by the presence of 50% cleavage; and a significant amount of plastic deformation occurred. In the next example, an extreme case of loss of ductility is cited. A poorly welded A106 specimen was tensile tested in hydrogen after X-ray inspection detected several large inclusions in the FZ. The specimen failed at 1/3 of the yield strength with essentially 0% RA, and displayed extensive cleavage. The cleavage was due to inclusions from welding fluxes, which were again identified by non-dispersive X-ray on the scanning electron microscope. It is apparent from this discussion that the behavior of imperfect mild steel weldments in hydrogen is highly variable.

We plan to extend our welding parameter studies to low and moderate strength manganese-carbon mild steels, and to the warm worked steels discussed previously. This testing will include typical multipass metal arc processes and potentially applicable processes such as electron beam, laser fusion, plasma arc and perhaps explosive bonding. These latter methods have the potential of producing clean welds and thin weld zones. The number and distribution of welding defects and subsequent behavior will be analyzed to assist setting NDT codes and requirements.

The experimental stainless steel/mild coupling discussed in previous reports is in assembly, and is expected to be burst tested within several months. Results will be reported as they become available.

iv) Permeation Barrier Development

The following work has been completed:

- i) Deuterium permeation measurements as a function of temperature for (a) thin silicon films (100Å to 1200Å) deposited by pyrolysis of silane (b) thick zinc coatings (50µm) applied by both plating and brush plating.
- ii) An investigation of controlled motion and manual motion methods of coating application focused upon developing optimum coating parameters.
- iii) Testing for possible permeation inhibiting effects of small fractions (<1%) of carbon monoxide and carbon dioxide in deuterium.

Some of the lead-tin specimens fabricated for long-term in-situ testing at BNL displayed a few small blisters. These defects were caused by excessive tool speed, which may trap electrolyte or gases below the metal surface and give rise to later blistering. The significance of the blistering to protectiveness of the coating is not yet known. However, since the blistering occurs between layers of the coating and not between the coating and the base metal, a permeation barrier still exists over the base metal. Some protection should, therefore, be obtained even in the presence of blisters.

Coating effectiveness is measured both by permeation resistance, and by the coatings' ability to adhere to and protect a tensile bar as it is being deformed. Permeation data is summarized in Figure 9. The Pb-Sn alloy reduces the permeation by about 10^7 (extrapolated) below that of the base metal at 100°C, the alloy performing considerably better than either pure lead or tin coatings. The alloy coating also performed well in tensile tests, principally because it adhered well out to several % deformation and prevented hydrogen ingress until plastic deformation was well underway. A more satisfactory zinc coating procedure has been developed, but the permeation through this coating is still 5000 times faster than through the Pb-Sn, extrapolated to ambient temperatures.

The controlled motion series for zinc is shown in Figure 10, and is typical of most of the brush plated coatings. The most defect free structures and lowest permeabilities were obtained at low current densities and moderately low tool speeds using mechanically controlled motion.

Application of brush plated coatings is slow and likely to be expensive. The cleaning schedule used for brush plating in the laboratory is reproduced in Appendix B. A number of demanding steps are required. Also, the actual application of the coating is slow, e.g., a minimum of 20 minutes is required to apply an optimal 0.05mm coating to a tensile bar 25mm long by 5mm diameter. The pure metal solutions (e.g., zinc) may be reused, but the Pb-Sn electrolyte must be mixed fresh every day, and collecting spillage or overflow may poison the electrolyte. All of these conditions impose serious obstacles to the commercial application of brush electroplated coatings. Recognizing these needs, efforts are underway to accelerate the cleaning and coating procedures.

Silicon films have been deposited on tensile bars and permeation membranes by pyrolysis of silane gas. Heating a specimen to 425°C for 24 hours in a closed vessel containing 195 torr SiH₄ and 5 torr H₂ resulted in a pure silicon film 100Å thick along with considerable interdiffusion of silicon and iron. Permeation reductions of several orders of magnitude were observed in the temperature range measured (250 to 350°C) and the high activation energy of the silicon coated sample implied a permeability reduction of $\sim 10^5$ at room temperature (see Figure 9). Thicker films ($\sim 1200\text{\AA}$) deposited at higher temperatures ($\sim 600^\circ\text{C}$) were less adherent and less effective as permeation barriers. Preliminary air tensile tests indicate excellent adhesion and hydrogen environment tests are being performed. Since the laboratory method used to deposit the films is far too energy intensive for use on large hardware, alternative methods for depositing silicon are being investigated.

Tests of thick zinc coatings applied by two different methods, brush-plating and bath-plating, indicate that coating uniformity may be a problem. For example, a brush-plated sample having a 50 μm thick zinc coating on one side had a substantially lower permeability than an electroplated sample having a 50 μm thick coating on both sides. In the temperature range measured, 250 to 350°C, the silicon films were more effective than the zinc coatings. Measurements will be extended to room temperatures using tritium as the permeating gas.

It has been reported that the addition of small quantities of gases such as carbon monoxide or carbon dioxide to hydrogen during tensile testing inhibits hydrogen embrittlement. It is thought that selective adsorption of these species on the metal surface reduces the quantity of adsorbed hydrogen, thereby lessening the rate of hydrogen transport into the metal. Though it was recognized that sticking coefficients are a very strong function of temperature, a few experiments were tried with carbon monoxide and carbon dioxide in deuterium at the elevated temperatures typical of the previously described permeation experiments. No effect on permeation rate was observed at partial pressures of 1 torr (mole fraction 0.01) CO or CO₂. No further low pressure, high temperature experiments are planned in this area. It seems more reasonable to study gaseous inhibitors using techniques amenable to ambient temperature testing such as high pressure permeation or tensile testing.

Current testing in progress includes:

- i) Brush electroplated coatings being evaluated in the BNL accelerated hydride cycling tests (see Hydrided Storage Support Work).
- ii) A comparison of the permeation behavior of bath and brush plated specimens having the Pb-Sn coating,
- iii) permeation and tensile test behavior of plasma sprayed coatings of aluminum and copper; selection, permeability and mechanical behavior testing of other potential plasma spray materials, particularly tungsten, silicon and germanium, based on unpublished research⁽²³⁾ on permeation of hydrogen. The plasma spraying method of coating offers speed and economy if dense coatings can be produced.

These results will be noted periodically in the bimonthly letters as the become available.

v) Advanced Mechanical Testing

In this program we have evaluated the hydrogen compatibility of several steels by conducting a series of comparative tensile tests and performing a number of slow crack growth tests. In a number of tests conducted on pre-cracked, wedge loaded samples, we have not been able to produce slow crack growth in 4.14 MPa H₂. In addition, Loginow and Phelps⁽²⁵⁾ found no crack growth in statically loaded WOL specimens (up to 55 MPa $\sqrt{m}$) fabricated from A106 even in 97 MPa H₂. Our reservations concerning total reliance on these tests to guarantee the growth of preexisting defects has been previously discussed.

It might be argued that hydrostatic proof testing of pipeline components will blunt the notch tips of preexisting defects prior to hydrogen exposure, and therefore, the EXTCT specimen tests described provide materials performance representative of real components.

However, as pointed out by Nichols and Cowan⁽²⁹⁾ and others proof testing can produce a false sense of security when the possibility of long term effects such as radiation embrittlement, stress corrosion, fatigue and corrosion fatigue may exist. Taking fatigue (especially near compressor sites) as a possibility, it has been found that fatigue growth rates (expressed as crack growth per load cycle, da/dn) of low strength steels are enhanced in the presence of hydrogen gas. It is conceivable then that fatigue conditions in the presence of hydrogen can resharpen a notch previously blunted during proof testing and propagate the crack at accelerated rates. Figure 11 summarized the data of Nelson⁽⁵⁾ on SAE 1020 steel at low pressures, and that of Phillips and Chandler⁽²⁴⁾ on SA-105 steel at high pressures. A surprising similarity of da/dn values at fixed (low) frequencies of load application and ΔK is observed for the two steels in spite of the large pressure difference between the two tests and compositional differences between the steels. A "threshold" K of about 15-20 MPa $\sqrt{m}$ appears to exist for cyclic crack growth in these steels at hydrogen pressures above 2 MPa. The data of Walter and Chandler show a significant frequency effect at high H₂ pressures and high stress intensities, which diminishes at low stress intensities. A large R-ratio effect is also observed.

A number of future activities are planned to fully investigate the possibilities of hydrogen assisted slow crack growth and hydrogen enhanced fatigue crack growth in gas transmission systems. First an analysis of the operational conditions that present transmission systems experience and future transmission systems may experience will be conducted. During this analysis the potential presence of structural defects, such as preexisting cracks and flawed welds, will be considered. Some specific items to be included in our analysis will be: the defects which can be realistically expected to exist in commercially produced and welded pipeline components, the stress intensities (including J integral considerations) developed in pipelines containing defects, the range of load cycling environments experienced in pipeline systems (ΔK and R considerations), an assessment of the degree of crack blunting which is expected in preexisting defects during proof testing of pipeline components, and the

electrochemical environment imposed on buried pipeline components being cathodically protected from corrosion.

As a result of this study, we expect to develop a quantitative basis for the design of relevant laboratory materials and prototype testing. We anticipate that the testing program will include, as appropriate, slow crack growth testing, environmental fatigue testing, J_{IC} testing, and burst testing of preflawed components.

Burst testing will provide experimental confirmation for the value of proof testing of embrittled or unembrittled structures and R-value management as ways of avoiding cyclic flaw growth. Leak before burst designs can also be tested in this way. Similar testing and analysis have already been performed at Sandia as part of a flawed pressure vessel study on high performance stainless steels.

	FY77	CALENDAR 1977		FY78			FY79
<u>BNL SUPPORT</u>							
POST MORTEM	•	•	•	•	•	•	•
CONTINUED		•	•	•	•	•	•
CONSULTATION		(welding, etc. as appropriate)					
<u>ALLOY DEVELOPMENT</u>							
EXP. HEATS,	▲	▲	▲	▲	▲	▲	▲
TMT, TESTS	△	△	△	△	△	△	△
ANALYSIS							
<u>WELDING DEVELOPMENT</u>							
PARAMETER STUDY	□	□	□	□	□	□	□
ALTERNATE PROCESSES	■	■	■	■	■	■	■
DEFECTED WELDS IN	□	□	□	□	□	□	□
PIPELINE							
<u>PIPELINE</u>							
DEFECTED WELDS	▲	▲	▲	▲	▲	▲	▲
INHIBITORS			△	△	△	△	△
ODORANTS					▲	▲	▲
<u>ADVANCED STUDIES</u>							
FATIGUE (inhibitors, etc)			•	•	•	•	•
PROOF & BURST			⊕	⊕	⊕	⊕	⊕
<u>STRUCTURE ANALYSIS</u>	•	•	•	•	•	•	•
<u>COATINGS</u>							
DEVELOPMENT	△	△	△	△	△	△	△
SCALE-UP			▲	▲	▲	▲	▲
TO PIPELINE

FIGURE 1: Hydrogen Environment Effects on the Ductility (%RA) of Steels in Smooth Bar Tensile Tests

FIGURE 2: Hydrogen Environment Effects on the Ductility (%RA) of Steels in Notched Bar Tensile Tests

FIGURE 3: Influence of Warm Rolling on Hydrogen Compatibility of Pressure Vessel Steels

Hot Worked A515
200X

Hot Worked A516
200X

Rolling Direction Out of Page

Warm Worked A515
200X

Warm Worked A516
200X

Rolling Direction to right (→)

FIGURE 4: Microstructures of Mill Finished and Warm Worked Steels

HOT ROLLED
UNEXPOSED TO H

HOT ROLLED - THERMAL CHARGE
4.14 MPa H₂ TEST

WARM ROLLED
UNEXPOSED TO H₂

WARM ROLLED - THERMAL CHARGE
4.14 MPa H₂ TEST

FIGURE 5: Fracture Surfaces of Mill Finished and Warm Worked
A515 Grade 70 Steel

HOT ROLLED
UNEXPOSED TO H₂

HOT ROLLED - THERMAL CHARGE
4.14 MPa H₂ TEST

WARM ROLLED
UNEXPOSED TO H₂

WARM ROLLED - THERMAL CHARGE
4.14 MPa H₂ TEST

FIGURE 6: Fracture Surfaces of Mill Finished and Warm Worked
A516 Grade 70 Steel

Hot Worked A516
Relative Mn levels

A 1.000
B 1.120
C 1.074
D 1.0221

Warm Worked A516
Relative Mn levels

A 1.000
B 0.962
C 0.988
D 0.965

FIGURE 7A: Manganese Segregation Studies in Mild Steels
Hot Worked and Warm Worked A516

Hot Worked A515
Relative Mn levels

A 1.000
B 1.130
C 0.954

FIGURE 7B: Manganese Segregation Studies in Mild Steels
Hot Worked A515

25X

Si CaCa Mn Fe Fe

FIGURE 8: Fracture Initiation at Welding Flux Inclusions in Welded A515 Steel

FIGURE 9: Protective Coatings for Reducing Hydrogen Uptake and Permeation In Steels

FIGURE 10: Microstructures of Brush Plated Tin Produced by Manual and Controlled Motion at Variable Current Densities

FIGURE 11: Hydrogen Environment Assisted Cyclic Cracking in Carbon-Manganese Mild Steels

TABLE II. Comparison of Tensile Properties After Exposure to FeTiH_x and 1 Atmosphere H_2 Case

A516	Test	0.2% Yield	ULT Strength	RA	Uniform Elongation	Total Elongation
	Air	303 MPa	518 MPa	72.6%	20.8%	47.0%
	H_2	312	520	72.6	18.6	28.5
	Hydride Charged	306	512	72.6	16.7	27.6
*	$\text{H}_2/48$ Hrs.	287	307	66.7	21.4	39.7
A106	Air	300	524	67.2	20.1	39.0
	H_2	312	531	64.2	17.7	25.4
	Hydride Charged	312	527	65.2	18.2	24.7
	$\text{H}_2/48$ Hrs.	308	517	62.8	19.0	27.6

*Different Lot of A516 Material

H_2 : Test in 1 Atmosphere H_2

Hydride Charged: Baked 72 hrs. in FeTiH_x , and 1 Atmosphere H_2 , 72 hrs. @ 200°C
Tested in environment at room temperature

$\text{H}_2/48$ Hrs: Soaked 48 hrs. in 1 atmosphere H_2 prior to test

TABLE III

Unexposed Tensile Properties of Materials Tested for
Slow-Crack Growth Behavior in 600 psi H₂ Gas

Material	Condition	σ_y , MPa	σ_u , MPa	ϵ_u , %	%, RA
A516 Gr 70	Base Metal	303 (44)	510 (74)	21	76
	Welded	380 (55)	525 (76)	9.4	67
A515 Gr 70	Base Metal	359 (52)	552 (80)	20.0	63
	Welded	372 (54)	552 (80)	10.0	44
A106-B	Base Metal	384 (47)	517 (75)	20.0	68
	Welded*				
A151-4340	Base Metal	531 (77)	745 (108)	10.0	65
	Welded	Not Tested - Embrittled			
2½ Cr-½ Mo	Base Metal	365 (53)	545 (79)	13.7	79
	Welded	365 (53)	535 (77.5)	9.1	79
C-95					

The numbers in () are stresses in SKI. A106-B welded is not reported due to the wide variability of values. A number of examples are cited in the text. Sound welds should behave similarly to the A516 and A515 Gr 70 welds.

TABLE IV. Test Matrix for Long-Term In-Situ Self-Loaded Tensile Specimens

Condition	Material						
	A106 Proof Stress	A106 Design Stress	A516 Proof Stress	A516 Design Stress	6061 Proof Stress	Cr-Mo Proof Stress	304 Proof Stress
Smooth Matrix	2	2	2	2	2	2	
Notch Matrix	2	2	2	2	2	2	2
Smooth Weld	2	3	2	2	1	2	
<u>Notched Weld</u>							
Heat Affected Zone	2	2	2	2	2	2	
Fusion Zone	2	2	2	2	2	2	2
<u>Coated (Sn-Pb)</u>							
Smooth Weld	2	1	2	1		2	
Notched Weld	2	1	2	1		2	

TABLE V: Exploratory Alloy Matrix
Base Metal A516 Grade 70

HEAT	ADDITIVES	REFERENCES	CARBON EQUIVALENT (A) (APPROXIMATE)	WORKING SCHEDULE	SULFIDE CONTROL
1	None (Melt & Cast Only)		0.38 to 0.45	Hot Worked Warm Worked	None
2	(B) (X w/o rare earth R.E.)		unchanged	"	I.S.C.
3	Sulfur level reduction		unchanged	"	Volume Red. + I.S.C.
4	0.1 w/o Ti	9-13,16,20	slight decrease	"	None
5	0.3 w/o Ti	"	0.37-0.44	"	None
6	0.3 w/o Ti, X w/o R.E.	"	(A)	Warm Worked	I.S.C.
7	0.5 w/o Si	"		Hot Worked	None
8	0.5 w/o Si + X w/o R.E.	"	(A)	Hot Worked Warm Worked	I.S.C.
9	0.5 w/o Si + lowered S level	same, parti- cularly 20.21	(A)	"	Volume Repro- duction + I.S.C
10	0.30 w/o Cu	14,15	0.39-0.46	"	
11	0.3 w/o Cu + 0.3 w/o Ni	13-15		"	None

(A) Assume base metal is 0.25 w/o C, 0.8 w/o Mn. Sulfur is not calculated, but plays a role in weldability (21). Silicon probably raises C.E. No C.E. for Ti is available. C.E. is given by reference (13).

(B) X is a parameter to be calculated from the sulfur level of the base metal, see Sulfide Inclusions In Steel, A.S.M. 1975 Metals Park, Ohio.

			DWG CLASSIFICATION LEVEL																							
DESIGN AGENCY CONTROL NO.			REVISIONS										ENGINEERS													
			ISSUE	DESCRIPTION										PREPARED BY										DATE	SPEC CFL	PROD RBA
			C																					6-29-76	<i>LS</i>	<i>RBA</i>
SHEET INDEX	SHEET																									
	ISSUE																									
	SHEET	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21				
	ISSUE	C	C	C	B	B	B	B	B	B	B															
AGENCY APPROVALS												TITLE														
ORG	DATE											INITIALS	ARC WELDING OF CARBON STEEL PIPE, FLANGES AND FITTINGS													
		DWG CLASSIFICATION LEVEL										SIZE	CODE IDENT NO.	DWG NUMBER												
A-2		UNCLASSIFIED										A	14214	K93869												
														SHEET 1 OF 10												

LS 5300-A (12-75)

APPENDIX A. ARC WELDING OF CARBON STEEL PIPE, FLANGES AND FITTINGS, (Issue C, 6-29-76).

		DWG CLASSIFICATION LEVEL							
<p>1. <u>G E N E R A L.</u></p> <p>1.1 <u>Scope.</u> This specification defines the minimum process and acceptance requirements for metal arc welding, i.e., GTA, GMA, or SMA of carbon steel pipe, flanges and fittings up to a maximum wall thickness of 1 inch.</p> <p>2. <u>D O C U M E N T S.</u></p> <p>The following documents form a part of this specification.</p> <p>ASTM-A-106 Specification for Seamless Carbon Steel Pipe for high-temperature service</p> <p>ASTM-A-105 Specification for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-temperature Service</p> <p>ASTM-A-181 Specification for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service</p> <p>ASTM-A-515 Specification for Carbon Steel Plates for Pressure Vessel for Intermediate and Higher Temperature Service</p> <p>AWS-A5.1 Mild Steel Covered Arc Welding Electrodes (E7018)</p> <p>AWS-A5.18 Mild Steel Electrodes for Gas Metal-Arc Welding (E70S-2)</p> <p style="padding-left: 100px;">ASME Boiler and Pressure Vessel Code - Section V, Non-Destructive Examination</p> <p>3. <u>R E Q U I R E M E N T S.</u></p> <p>3.1 <u>Material.</u></p> <p>3.1.1 <u>Base Metal.</u> The base material shall conform to ASTM-A-106, A105, A181, and A515.</p> <p>3.1.2 <u>Filler Metal.</u> The filler material to be used with the GTA or GMA welding processes shall conform to AWS E70S-2 of AWS filler metal specification number AWS A5.18. The filler material to be used with the SMA welding process shall conform to AWS E7018 of AWS filler metal specification number AWS A5.1. Material certification of chemical and mechanical properties is required.</p>									
ISSUE	C					DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT NO.	DWG NUMBER
						UNCLASSIFIED	A	14214	K93869
									SHEET 2 OF

LS 5300-AA (12-75)

	DWG CLASSIFICATION LEVEL	
<p>3.2 <u>Preparation of Base Material.</u> The preparation of base metal and joint design is the responsibility of the fabricator. If Plasma Arc cutting is used, 0.1 inch material must be machined or ground from the cut surface prior to welding.</p> <p>3.3 <u>Welding Technique.</u> The root pass on all full penetration welds shall be made using the GTA or GMA welding processes. Back side shielding must be used and maintained through the first filler pass. Filler passes may be made using the GTA, GMA, or SMA processes.</p> <p>3.4 <u>Certified Welders.</u> All welding shall be performed by welders certified per ASME Boiler and Pressure Vessel Code, Section IX, or API Standard 1104, Standard for Welding Pipelines and Related Facilities.</p> <p>3.5 <u>Written Procedures and Inspection Reports.</u></p> <p style="margin-left: 40px;">a. A written report, describing the welding procedures which were followed, shall be supplied to Sandia Laboratories.</p> <p style="margin-left: 40px;">b. Complete inspection reports and radiographs shall be supplied to Sandia Laboratories. These reports shall include the written procedures that are required in the standards for radiographic and magnetic particle inspection.</p>		
<p>4. <u>Q U A L I T Y A S S U R A N C E P R O V I S I O N S .</u></p> <p>4.1 <u>Visual Inspection.</u> The following is required of the weld to pass visual inspection.</p> <p style="margin-left: 40px;">a. The specified base metal and filler metal shall have been used.</p> <p style="margin-left: 40px;">b. The weld shall be free of cracks.</p> <p style="margin-left: 40px;">c. All craters shall be filled to the full cross-section of the weld and shall be free of cracks upon completion of the welding.</p> <p style="margin-left: 40px;">d. The face of the weld shall be at least flush with the outside surface of the joint and the weld shall merge smoothly with the base metal. Undercuts shall not exceed the values shown in Table 1 and shall not encroach on the required section thickness. Weld reinforcements are permitted per Table 1.</p> <p style="margin-left: 40px;">e. The root of the weld shall be examined and there shall be no evidence of cracks, incomplete fusion, or inadequate joint penetration. Concavity of the root surface is permitted within limits of Table 1 provided the resulting thickness of the weld is at least equal to that of the base metal. Maximum melt thru values are specified in Table 1.</p>		
ISSUE	C	DWG CLASSIFICATION LEVEL
		UNCLASSIFIED
SIZE	CODE IDENT NO.	DWG NUMBER
A	14214	K93869
		SHEET 3 OF

LS 5300-AA (12-75)

		DWG CLASSIFICATION LEVEL			
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TABLE 1 - LIMITS OF SURFACE IMPERFECTIONS				
MATERIAL NOMINAL THICKNESS	OUTSIDE SURFACE MAXIMUM UNDERCUT	MAXIMUM REINFORCEMENT THICKNESS	INSIDE SURFACE MAXIMUM ROOT SURFACE CONCAVITY	MAXIMUM MELT-THRU
up to 1/8, incl	1/64	3/32	1/32	1/16
Over 1/8 to 3/16 incl	1/64	1/8	1/32	3/32
Over 3/16 to 1/2 incl	1/32	5/32	1/16	1/8
Over 1/2 to 1 incl	1/32	3/16	1/16	5/32

NOTE: ALL DIMENSIONS ARE IN INCHES.

4.2 Radiographic Inspection. All welds are to be radiographed with a quality level of 2-2T (based on weld thickness) with a double film technique per ASME Boiler and Pressure Vessel Code - Section V, Article 2, with the following changes:

T-233 Density Limitations of Radiographs. The film density through acceptable weld metal shall be 2.0 minimum for single film viewing. Double film viewing is not acceptable.

T-262.3 Number of Penetrameters. Delete Item (d).

T-291 Requirements. Personnel performing the radiography shall meet the qualifications of NDT Level I; and the radiographic film interpreter shall meet the qualifications of NDT Level II as outlined in SNT-TC-1A, Supplement A.

4.2.1 Radiographic Inspection Acceptance Criteria. Welds having any of the following defects shall be judged unacceptable.

- a. Any type of crack.
- b. Incomplete fusion between the weld metal and base metal.
- c. Incomplete fusion between any of the weld passes.
- d. Inadequate joint penetration.
- e. Porosity and slag inclusions in excess of the amounts described in Section 4.2.2.

ISSUE	B					DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT NO.	DWG NUMBER
						UNCLASSIFIED	A	14214	K93869
									SHEET 4 OF

		DWG CLASSIFICATION LEVEL						
<p>4.2.2 Maximum Acceptable Areas of Porosity And Pore Dimension.</p> <p>a. The total area of porosity as determined from the radiographic film shall not exceed 0.060T square inch in any 6-inch length of weld, where T is the thickness of the weld. If the weld is less than 6 inches long, the total area of porosity will be reduced in proportion.</p> <p>b. The maximum pore dimension shall be 20 percent of T, or 1/8 inch, whichever is smaller.</p> <p>c. In any 1-inch length of weld or 2T, whichever is smaller, porosity may be clustered to a concentration four times that permitted by 0.060T. Such clustered porosity shall be included in the porosity in any 6-inch length of weld which includes the cluster.</p> <p>d. Aligned porosity shall be acceptable providing the summation of the diameters of the pores is no more than T in a length 12T, or 6 inches, whichever is less, providing each pore is separated by a distance at least six times the diameter of the largest adjacent pore. Aligned porosity indications shall be counted in the total area of permissible indications in any 6 inch length of weld.</p> <p>e. Dark images of a generally circular or oval shape will be interpreted as porosity.</p> <p>f. The porosity charts in Figures 1, 2, and 3 illustrates various types of assorted and uniform, randomly dispersed porosity indications. These charts for each thickness represent the maximum acceptable porosity. These charts represent full scale 6-inch radiographs and shall not be enlarged or reduced. The porosity distributions shown are not necessarily the patterns that may appear on the radiograph but are typical of the number and size of indications permitted. When porosity indications differ significantly from the porosity charts, the actual numbers and sizes of the pores may be measured and the total area of porosity calculated.</p> <p>Permissible porosity indications for weld thicknesses intermediate to those illustrated may be evaluated either by comparison with the next thinner material or by calculation as shown in Table 2.</p>								
ISSUE	B				DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT NO.	DWG NUMBER
					UNCLASSIFIED	A	14214	K03869
								SHEET 5 OF

	DWG CLASSIFICATION LEVEL											
<p>4.3 <u>Magnetic Particle Inspection.</u></p> <p>a. All welds are to be inspected with the Dry Powder Magnetic Particle Inspection technique using the Prod Method per ASME Boiler and Pressure Vessel Code - Section V, Article 7, with the following changes:</p> <p style="padding-left: 40px;">Delete the following sections:</p> <table style="margin-left: 40px; border: none;"> <tr><td style="padding-right: 20px;">T-725.2</td><td>Wet Particles</td></tr> <tr><td>T-725.3</td><td>Fluorescent Particles</td></tr> <tr><td>T-732</td><td>Coil Method</td></tr> <tr><td>T-733</td><td>Direct Contact Method</td></tr> <tr><td>T-734</td><td>Yoke Method</td></tr> </table> <p>b. The welds shall be tested for longitudinal and transverse cracks with the magnetization current running:</p> <ol style="list-style-type: none"> In the weld direction. Perpendicular to the weld direction. <p>c. The non-destructive examination personnel responsible for the magnetic particle inspection shall meet the qualifications of NDT Level II as outlined in SNT-TC-1A, Supplement B.</p> <p>4.3.1 <u>Magnetic Particle Inspection Acceptance Criteria.</u> A weld shall be judged unacceptable if there is an indication of of any type of crack or other linear discontinuity.</p> <p>5. <u>REPAIR OF UNACCEPTABLE WELDS.</u></p> <p>Unacceptable defects in welds may be repaired only after approval of Sandia Laboratories is obtained for the method and extent of repairs. The inspection of repaired welds shall be repeated as required for the original weld. Defective welds that cannot be satisfactorily repaired shall be rejected.</p>			T-725.2	Wet Particles	T-725.3	Fluorescent Particles	T-732	Coil Method	T-733	Direct Contact Method	T-734	Yoke Method
T-725.2	Wet Particles											
T-725.3	Fluorescent Particles											
T-732	Coil Method											
T-733	Direct Contact Method											
T-734	Yoke Method											
ISSUE	B	DWG CLASSIFICATION LEVEL										
UNCLASSIFIED	A	CODE IDENT NO.										
		14214										
		DWG NUMBER 93869										
		SHEET 6 OF										

ISSUE

A	B
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DWG CLASSIFICATION LEVEL
UNCLASSIFIED
 SIZE
A

CODE IDENT NO.
 14214

DWG NUMBER
 K93869

SHEET 7 OF

THICKNESS (INCHES)	TOTAL AREA OF PERMITTED POROSITY (SQUARE INCHES)	LARGE PORE SIZE (INCH)			MEDIUM PORE SIZE (INCH)			FINE PORE SIZE (INCH)		
		APPROX FRACTION	DECIMAL VALUE(1)	NO.	APPROX FRACTION	DECIMAL VALUE(1)	NO.	APPROX FRACTION	DECIMAL VALUE(1)	NO.
1/8	.0075							1/64	.014	49
1/4	.015				3/128	.025	31	1/64	.0138	100
1/2	.030	3/32	.10	4	1/32	.031	40	3/128	.0195	101
3/4	.045	1/8	.125	4	1/32	.034	50	3/128	.024	99
1	.060	1/8	.125	5	5/128	.039	50	1/32	.0275	101

(1) These values used for calculating total area of permissible porosity given in second column

**TABLE 2 - MAXIMUM PERMISSIBLE POROSITY INDICATIONS IN RADIOGRAPHS PER 6-INCH
LENGTH OF WELD**

DWG CLASSIFICATION LEVEL

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SF 5300-AA (5-67)

		DWG CLASSIFICATION LEVEL	
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NOTE:

The above charts show the typical number and size of pores permitted in any six-inch length of weld with a thickness of 1/2 inch. The total pore area permitted is 0.030 square inch.

FIGURE 1 - POROSITY CHART FOR ONE-HALF INCH THICK WELDS

ISSUE	A	B			DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT NO.	DWG NUMBER
					UNCLASSIFIED	A	14214	K93869
								SHEET 8 OF

		DWG CLASSIFICATION LEVEL	
--	--	--------------------------	--

Dimension		No. of Pores
.024		35
.034		19
.125		1

Assorted

.125		4
------	--	---

Large

.034		50
------	--	----

Medium

.024		99
------	--	----

Fine

NOTE:
The above charts show the typical number and size of pores permitted in any six-inch length of weld with a thickness of 3/4 inch. The total pore area permitted is 0.045 square inch.

FIGURE 2 - POROSITY CHART FOR THREE QUARTER INCH THICK WELDS

ISSUE	A	B			DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT NO.	DWG NUMBER
					UNCLASSIFIED	A	14214	K93869
								SHEET 9 OF

		DWG CLASSIFICATION LEVEL	
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Dimension No. of Pores

.0275		38
.039		21
.125		1

Assorted

.125		5
------	--	---

Large

.039		50
------	---	----

Medium

.0275		101
-------	--	-----

Fine

NOTE: The above charts show the typical number and size of pores permitted in any six-inch length of weld, one inch weld thickness. The total pore area permitted is 0.060 square inch.

FIGURE 3 - POROSITY CHART FOR ONE-INCH THICK WELDS

ISSUE	A	B			DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT NO.	DWG NUMBER
					UNCLASSIFIED	A	14214	K93869
								SHEET 1 OF 1

APPENDIX B

CLEANING AND PREPARATION STEPS PRIOR TO BRUSH ELECTROPLATING

The following procedure is a condensation of that recommended by Vanguard Pacific Inc. for the Vanguard Process. Other procedures are dictated for other base metals and high strength steels.

BASE METAL - Mild Steel, Carbon Steel, Carburized & Nitrided Steel			
Step No.	Operation	Etching Polarity: Forward (+) or Reverse (-)	Remarks
<hr/>			
1	Preclean		Mechanical, Acid Pickling, Degreaser Solvent
2	Electroclean †	+	20-30 Seconds, 10-20 Volts
3	Rinse		Check for water breaks
4	Activator #2 †	-	10-20 Volts, until uniform gray to black color
5	Rinse		
6	Activator #3 †	-	10-20 Volts, uniform matte white color
7	Rinse		
8	Plate	+	Nickel (neutral) under Pb-Sn alloy, no copper acid high speed

† Proprietary Solution and/or procedure

In order to obtain the proper pattern of tool/workpiece (ANODE/CATHODE) movement; round parts should be rotated, an orbital motion should be used on flat parts. Slow speed of movement gives coarse grained deposits and insufficient speed gives porous deposits. Excessive speed leads to loss of current efficiency, and may exceed a critical velocity where no deposition takes place. Some satisfactory velocity ranges are indicated below.

<u>Solution</u>	<u>Anode-Cathode Relative Velocity (Feet/Min)</u>
Lead	30 - 50
Zinc	30 - 120
Nickel (neutral)	20 - 80
Nickel (acid)	20 - 50

The electrocleaning, etching, and activating operations should be carried out at moderate speeds (20-60 fpm).

Both area and current dictate deposition rates, as does operating temperature. Some deposition rates are given below.

<u>Solution</u>	<u>Average Time to Deposit 0.0001" (Seconds)</u>
Lead	12
Zinc	8
Nickel (neutral)	24
Nickel (acid)	24

Use of an Ampere-Hour meter is recommended to control deposition thickness, as is the use of as large a tool as possible, consistent with movement patterns, edge effects, temperature constancy and operator efficiency, whether manual or mechanized.

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