**Nondestructive and Localized Measurements of Stress-Strain Curves and
Fracture Toughness of Ferritic Steels at Various Temperatures
Using Innovative Stress-Strain Microprobe™ Technology**

Final Report

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CONTENTS

ABSTRACT	1
INTRODUCTION.....	1
The SSM system and Its ABI Techniques:	2
ANTICIPATED BENEFITS.....	4
RESULTS AND DISCUSSION	4
Determination of the Indentation Energy to Fracture (IEF) for ferritic steels.	4
Determination of fracture toughness (K_{Jc}) ^{ABI} from ABI tests at various temperatures.	5
Determination of fracture toughness in the ductile/upper shelf region.....	9
Standardization of the ABI Test Techniques to Measure Stress-Strain Curves and Fracture Toughness of Ferritic Steels.....	9
MINIATURE SSM SYSTEM AND FIELD TESTING DEMONSTRATION	11
SUMMARY	13
PHASE III: TECHNOLOGY TRANSFER	14
REFERENCES.....	15
APPENDIX A.....	38

LIST OF FIGURES AND TABLES

Fig. 1 (a) Stress-Strain Microprobe system (Model SSM-M1000) configured for field testing of a pipe.	17
Fig. 2 Photograph of the SSM system in a laboratory configuration with an environmental chamber, a positioning table, and a video camera installed on its load frame (notice the LVDT/depth-transducer is mounted on a bracket outside chamber). The positioning table and video camera allow accurate ABI testing of any heat-affected-zone (HAZ) location.....	18
Fig. 3 Sixteen (16) Automated Ball Indentation (ABI) tests were made at different test temperatures...	18
Fig. 4 The room-temperature ABI test results on A533B-1 RPV steel (HSST Plate 02).....	19
Fig. 5 Indentation Energy to Fracture (IEF) versus ABI test temperature for ORNL 72W weld	19
Fig. 6 Example of Indentation load versus depth in an ABI test using a 0.51-mm (0.020-inch) diameter tungsten carbide indenter on X42 ferritic steel material.	20
Fig. 7 Comparison of Stress-Strain Curves from ABI and Tensile Tests on X42 ferritic steel.....	20
Fig. 8 ABI-Measured Stress-Strain Curves at Various Test Temperatures for A533B steel.	21
Fig. 9 Temperature variation of IEF for A533B steel.....	21
Fig. 10 Comparison between nondestructively ABI-measured (K_{Jc}) ^{ABI} and destructive 1T CT fracture toughness test results of 73W Weld of ORNL.	22
Fig. 11 Estimated K_{Jc} of SA508 Gr. 3 steels (base metals).....	22
Fig. 12 Estimated K_{Jc} of reactor pressure vessel weld metals.....	23
Fig. 13 Fracture toughness "Master Curve" data of Oak Ridge National Laboratory.....	23
Fig. 14 Fracture toughness Master Curve obtained from ABI tests on three RPV steels.....	24
Fig. 15 Effect of test temperature on the critical fracture stress and yield stress of HSST Plate 02.	24
Fig. 16 Effect of test temperature on the critical fracture stress and yield stress of ORNL 72W weld.	25
Fig. 17 Effect of test temperature on the critical fracture stress and yield stress of ORNL 73W weld.	25
Fig. 18 Effect of test temperature on the ratio of critical fracture stress to yield stress for HSST Plate 02.	26

Fig. 19 Effect of test temperature on the ratio of critical fracture stress to yield stress for ORNL 72W weld.	26
Fig. 20 Effect of test temperature on the ratio of critical fracture stress to yield stress for ORNL 73W weld.	27
Fig. 21 Effect of temperature on the critical fracture stress of three RPV steels.....	27
Fig. 22 Effect of test temperature on the ratio of the critical fracture stress to the yield stress for three RPV steels. The linear relationship of HSST Plate 02 has the lowest slope as compared to the two welds.....	28
Fig. 23 Fracture toughness Master Curves determined from 19 destructive fracture toughness tests	28
Fig. 24 Fracture toughness Master Curve determined from 22 ABI tests on the "Tasso" Brazilian steel.	29
Fig. 25 Fracture toughness Master Curve determined from 3 ABI tests on the "Alexandre" ferritic steel.	29
Fig. 26 Comparison between the reference temperature from localized ABI test and from destructive fracture toughness tests on six ferritic steel materials.....	30
Fig. 27 Example of ABI data and test results performed with the miniature SSM, Model SSM-M1000, on a ferritic steel pipe using 0.51-mm, 0.76-mm, and 1.57-mm (0.020-inch, 0.030-inch, and 0.062-inch) diameter tungsten carbide indenters.....	31
Fig. 28 The SSM system, Model SSM-M1000, shown inside ATC's laboratory with the testing head mounted on a 6-inch diameter carbon steel pipe using the manual magnets (with on/off switches)	32
Fig. 29 The SSM system (Model SSM-M1000) is used outdoors for field testing of a 6-inch diameter carbon steel pipe (from Columbia Gas Transmission Corporation).	33
Fig. 30 (a) Data from an ABI test performed, using the magnetic mounts and the battery pack, on a 6-inch diameter carbon steel pipe from Columbia Gas Transmission Corporation using a 0.020-inch diameter tungsten carbide indenter. (b) Comparison between the true-stress/true-plastic-strain curve from the ABI data shown in (a) with the curve from a miniature tensile specimen manufactured from the same pipe.	34
Table 1 ABI-measured fracture toughness results from three nuclear pressure vessel steels (Plate 02, 72W weld, 73W weld).....	35
Table 2 Comparison between reference temperature values determined from conventional/ destructive and ABI tests for various ferritic steels.	36
Table 3 Summary of test results from ABI tests (on the 6-inch diameter pipe and on the end tabs of miniature tensile specimens) and from tensile tests (including miniature and large size specimens) of a carbon steel pipe obtained from Columbia Gas Transmission Corporation.	37

Nondestructive Measurements of Stress-Strain Curves and Fracture Toughness of Ferritic Steels at Various Temperatures Using Innovative Stress-Strain Microprobe™ Technology

ABSTRACT

The results presented in this report demonstrate the capabilities of Advanced Technology Corporation's patented Portable/In-Situ Stress-Strain Microprobe™ (SSM) system and its Automated Ball Indentation™ (ABI) test techniques to nondestructively measure the yield strength, the stress-strain curve, and the fracture toughness of ferritic steel samples and components in a reliable and accurate manner.

The accuracy, reliability, and easy field applicability of the SSM technology to test reactor pressure vessel plates and welds and other ferritic steels have been demonstrated in this work on plate and weld samples obtained from Oak Ridge National Laboratory and from the University of Tennessee, Knoxville. The ABI-measured tensile and fracture toughness values are in excellent agreement with the results from destructive tests over the temperature range from -130°C/250°F to 288°C/550°F. Hence, this report provides the technical basis for any nuclear utility or any company from other industries to file a waiver or an exemption (with the appropriate US government regulatory office) to allow nondestructive ABI testing as an alternative to the destructive and expensive tensile and fracture toughness testing of the ferritic steel components under investigation. The laboratory version of the SSM system has been in commercial use since 1991 in three countries, and the Portable/In-Situ Stress-Strain Microprobe (SSM) system received a 1996 R&D 100 Award. Furthermore, a new miniature SSM-M1000™ system was developed and introduced in 1999 to provide even greater portability. Equipped with a portable battery pack and manual magnetic mounts, the SSM-M1000 will be a valuable test instrument for many industries.

Key Words: *in-situ*, nondestructive, ball indentation, microprobe, stress-strain curve, fracture toughness, master curve, reference temperature, pressure vessel, embrittlement, structural integrity

INTRODUCTION

The radiation-induced degradation of metallic nuclear structural materials is currently monitored via destructive testing of surveillance specimens (mostly tensile, Charpy impact, and fracture toughness if available). Long term operation of nuclear reactors (60 years instead of the 40-year design life of the current light water reactors) will rapidly exhaust available irradiated surveillance specimens. Hence, nondestructive techniques utilizing broken specimens and/or in-situ testing of the critical components in nuclear facilities are desired to ensure the structural integrity and to guarantee safe operation of nuclear power plants as well as to reduce maintenance cost and improve performance.

The major goal of this project was to develop new methods for determining fracture toughness of nuclear reactor pressure vessel (RPV) materials and their welds from Automated Ball Indentation "ABI" tests and using the appropriate fracture mechanism models. These innovative methods were validated by comparing the indentation-derived fracture toughness values with those from destructive tests on the same materials. The new nondestructive technology to determine fracture toughness from ABI tests (at various test temperatures) will allow nuclear utilities to monitor the radiation-induced degradation/embrittlement of structural materials over their design service life, following mitigation actions such as thermal annealing, and through service life extension, without the expensive machining of irradiated specimens. Furthermore, the ABI-determined fracture toughness values are not dependent on the specimen size or geometry such as the

case for conventional destructive fracture toughness tests. For the latter, testing Charpy-size specimens might produce invalid fracture toughness test results for many irradiated and irradiated+annealed conditions, hence, the limited number of surveillance specimens might be depleted without obtaining valid test results to determine the fracture toughness Master Curve and the reference temperature, " T_0 " (temperature at a fracture toughness level of $100 \text{ MPa}\cdot\text{m}^{0.5}$). However, each localized single ABI test produces both the stress-strain curve and the fracture toughness value (highly advantageous and desirable for heat-affected-zone areas).

Another important accomplishment of this project is the successful development of a miniaturized testing head of the SSM system for use inside a nuclear reactor. The electronics cabinet (containing the motor drive, signal conditioning modules and data acquisition interface) of the SSM system was also miniaturized. The new miniature SSM system, Model SSM-M1000, was successfully demonstrated on various curved surfaces using manual magnets to temporarily attach/secure the testing head to the pipe section. The SSM-M1000 system can be operated using a small portable battery pack which is useful in testing oil and natural gas pipeline in remote areas. Digital video files of field testing demonstrations are available on a CD from ATC or for viewing on ATC's web site: www.atc-ssm.com. Although most of the new developments in the SSM technology were initiated in response to the needs of the nuclear industry, several other industries will have immediate applications (e.g. natural gas and oil pipelines with unknown properties installed in service since the early 1900s, ships with unknown steel plates and welds, high pressure cylinders, steel bridges, etc.).

The ABI test technique is described in detail in many publications.¹⁻¹⁷ The ABI technique is nondestructive and localized, and is a sophisticated mechanical test technique which can be applied to small samples as well as to metallic components (such as pressure vessels and natural gas and oil pipelines) in the field. These capabilities of the ABI technique and the SSM technology are advantageous and desirable for testing aged components and for structural integrity evaluation. Furthermore, in addition to the ABI stress-strain curve measurements, the nondestructive and localized ABI technique of the Stress-Strain Microprobe™ (SSM) system provides fracture toughness properties. The determination of fracture properties from ABI tests is described briefly in References 4 through 6 and in detail in this report.

The ABI test is based on progressive indentation with intermediate partial unloadings until the desired maximum depth (maximum strain) is reached and then the indenter is fully unloaded. The indentation load-depth data are collected continuously during the test using a 16-bit data acquisition system. The nonlinear spherical geometry of the tungsten carbide indenter allows increasing strain as the indentation penetration depth is increased. Hence, the incremental values of load and plastic depth (associated with each partial unloading cycle) are converted to incremental values of true-stress and true-plastic-strain according to elasticity and plasticity theories as described in Reference 3. The ABI test is fully automated and a single test is completed in two minutes or less depending on the desired strain rate. Sixteen publications including References 3 and 6 and this report are available for downloading from ATC's web site: www.atc-ssm.com.

The SSM system and Its ABI Techniques:

The patented Stress-Strain Microprobe (SSM) system (1996 R&D 100 Award Winner) utilizes an automated ball indentation (ABI) technique to measure local key mechanical properties including yield strength, flow properties (true-stress/true-plastic-strain curve), strain hardening exponent, strength coefficient, ultimate tensile strength, and fracture toughness. The SSM has been used for measuring the mechanical properties of several different materials including low-alloy pressure vessel steels (base metals, weld metals, and heat-affected zones "HAZs"), carbon steels, and austenitic stainless steels. The ABI technique requires a small amount of material and does not require any machining. The technique is essentially nondestructive because no material is removed from the surface tested, only a smooth shallow

indentation is left on the surface at the end of the test. The indentations are performed with a spherical tungsten carbide indenter whose diameter is in the range of 0.25 to 1.57 mm. The SSM system can be reconfigured for both field (in-situ component testing as shown in Fig. 1) and laboratory applications in a few minutes. Furthermore, for laboratory applications it can be equipped with an environmental chamber (Fig. 2) so that ABI or tensile tests can be performed from -196°C to +427°C. Additional information on the SSM system applications and several downloadable publications are available on ATC's Internet Web Site: www.atc-ssm.com.

The ABI technique is based on strain-controlled progressive indentations at a given point on the surface of a specimen.¹⁻³ Progressive indentations are performed with increasing loads and at the end of each indentation cycle, partial unloading is carried out so that the indentation depth associated with plastic deformation can be determined. Note that during each subsequent indentation, the amount of material experiencing plastic deformation increases which means that continuous yielding and work-hardening occur simultaneously. This is different from a tensile test where the amount of material experiencing plastic deformation remains the same (gage section volume) during the entire tensile test. Note that only a broken half of either a Charpy or a fracture toughness specimen is needed for measuring the stress-strain curves at several different temperatures (Fig. 3). A sample of ABI-measured stress-strain curves on unirradiated, irradiated, and irradiated+annealed specimens at room temperature is given in Fig. 4.^{2,18} Numerous ABI test results on archival reactor pressure vessel (RPV) steels are presented in the Phase I report of this Small Business Innovation Research (SBIR) project.¹⁹ The Phase I report includes results of many ABI tests conducted on unirradiated, neutron-irradiated, and irradiated+annealed plate and weld RPV materials.

The Stress-Strain Microprobe can also be used to determine Charpy energy and fracture toughness of metallic materials.^{5,6,19} This particular use of the ABI technique is not apparent because indentation does not induce any cracking in these metallic materials. In addition, the stresses at the center of the contact surface of the test specimen under the ball indenter are compressive, whereas the stresses in the front of a crack tip in a fracture specimen are tensile. However, elasticity theory and preliminary finite element analyses results show that the stress triaxiality present at a crack tip in a fracture toughness specimen and at the center of the contact surface under a spherical indenter are similar (Reference: F. A. McClintock and A. S. Argon, Editors, *Mechanical Behavior of Materials*, Addison-Wesley, 1966, pp.372-373).^{20,21} The material at the center of the contact surface under the indenter experiences a degree of constraint similar to that experienced by the material at the crack tip, and the deformation energy at the center of impression might be correlated to that at the front of a crack tip. Therefore, it has been postulated that the indentation energy per unit contact area up to a critical fracture stress is related to the fracture energy of a material. This parameter is referred to as Indentation Energy to Fracture (*IEF*), and it represents the fracture energy as determined from the ABI-measured stress-strain curves up to either the critical fracture stress or strain, depending on the controlling fracture micromechanism, which in turn depends on the test temperature. The concept of a critical fracture stress is applied to the lower shelf and the transition region (dominating cleavage fracture) and the concept of a critical fracture strain is applied to the upper shelf region (ductile fracture).

The variation of the *IEF* with respect to the ABI test temperature for unirradiated HAZ (ORNL 72W Weld)²² is shown in Fig. 5. Also, the figure shows the *IEF* curves for the base metal and the weld nugget. It shows a lower shelf, transition region and an upper shelf, similar to those present in the Charpy energy versus temperature curves for reactor pressure vessel materials. A preliminary correlation between *IEF* and Charpy energy curves has been developed. The concept of *IEF* was further refined and extended for determining the static fracture toughness, K_{Jc} , of pressure vessel steels. In Phase II of his project, the ABI-derived (K_{Jc})^{ABI} method was developed and validated for ferritic steels. The experimental work included unirradiated plate and weld RPV steels from Oak Ridge National Laboratory (ORNL) and some Brazilian steels from The University of Tennessee, Knoxville (UTK).

ANTICIPATED BENEFITS

The new nondestructive technology to determine fracture toughness from automated ball indentation tests will allow nuclear utilities to monitor the radiation-induced embrittlement of metallic nuclear structures over their design service life, following mitigation actions such as thermal annealing, and through life extension, without machining of irradiated specimens. This innovative technology (which is faster, better, and nondestructive) will improve safety, minimize radiation exposure to nuclear utility workers (ALARA), and will reduce maintenance costs in many other industries with metallic structures such as oil storage vessels, offshore platforms, ships, bridges, gas pipelines, etc.

RESULTS AND DISCUSSION

The test results of Phase I are briefly mentioned in this report while the accomplishments of Phase II are presented in more details below.

Determination of the Indentation Energy to Fracture (IEF) for ferritic steels.

An example of the indentation load-depth data and a comparison of the stress-strain curves from ABI and tensile tests are shown in Figures 6 and 7 for X42 ferritic steel material. A new ABI energy parameter called *Indentation Energy to Fracture (IEF)* was developed for ferritic steels.^{5,19} This *IEF* parameter allows the nondestructive determination of fracture energy from the ABI-measured true stress-strain curve up to the controlling micro-mechanical fracture mechanism of the critical fracture stress or the critical fracture strain (depending on the test temperature). The indentation load versus depth curves from ABI tests at various temperatures were used together with the critical fracture stress model to evaluate the temperature variation of fracture energy. The development of the *IEF* parameter is based on the following premise:

- (a) Fracture toughness can be interpreted as the deformation capability of the material under a concentrated stress field.
- (b) Indentation with a small ball indenter generates concentrated stress (and strain) fields near and ahead of the contact of the indenter and the test surface, similar to concentrated stress fields ahead of a crack albeit the indentation stress fields are mostly compressive. The high value of the stress under the ball indenter is sometimes called an example of *plastic constraint* where the rigid material surrounding the indentation volume that does the constraining.²¹ Hence, at a certain critical ball indentation depth there is a high state of transverse and lateral stresses similar to those in front of a sharp notch in an elastic material. Although, the conditions for crack initiation might be attained, the high degree of plastic constraint is the reason that cracks do not develop during ball indentation of ductile metallic materials. This explains that only initiation fracture toughness and no tearing modulus can be determined from ball indentation.
- (c) Monotonic tensile versus compressive stress-strain curves are similar; true for most homogenous metallic structural materials.
- (d) The cleavage fracture stress in ferritic steels is nearly temperature insensitive at low test temperatures in the transition and low shelf regions.
- (e) The deformation energy due to ball indentation up to a limit mean pressure level is related to the Charpy energy or fracture toughness; the limit stress, attained at a critical indentation depth in an ABI test, is proportional to the critical fracture stress of the test material.

The *IEF* is thus defined as,

$$IEF = \int_0^{h_f} P_m(h) dh \quad (1)$$

where,

$$P_m = \frac{4P}{\pi d^2} \quad (2)$$

In the above equation, P_m is the mean indentation contact pressure, P is the indentation load, h is the indentation depth, h_f is the indentation depth up to the cleavage fracture stress, and d is the chordal diameter of the indentation. As to be noted in Fig. 6, the indentation load versus depth curves are essentially linear in which case, the slope (S) of the curves can be used to calculate *IEF*,

$$P = Sh, \quad \text{and} \quad d = 2(Dh - h^2)^{0.5} \quad (3, 4)$$

where D is the indenter diameter, so that,

$$IEF = \frac{S}{\pi} \ln \left(\frac{D}{D - h_f} \right) \quad (5)$$

where "ln" is the natural logarithm.

The ABI-measured true stress-strain curves at various temperatures are superimposed with a representative true stress at fracture of 800 MPa (Fig. 8).³ The stress index of 800MPa is approximately one third of the critical fracture stress of A533B RPV steel. This assumed smaller value is required to account for the different tri-axial stress state in a smooth tensile specimen (the true-stress/true-plastic-strain curves from tensile and ABI test are identical) as compared to that in a sharp precracked specimen. However, it is clear from Fig. 8 that the area under the stress-strain curves up to the intersection of the representative/index stress (at the hypothetical indentation fracture limit) decreases with lower test temperatures, as expected from Charpy impact and fracture toughness tests. The indentation depth at fracture (h_f), is determined by interpolation or extrapolation (depending on the test temperature) from the plot of true stress versus indentation depth.¹⁹ An example of the *IEF* as a function of temperature is given in Fig. 9.

A preliminary correlation between the *IEF* values shown in Fig. 9 and the impact energy from Charpy V-notch specimens was found to be:¹⁹

$$CVN = 1.356 \times [e^{(0.003691 \cdot IEF)} - 1] \quad (6)$$

where CVN is in Joules and *IEF* is in mJ/mm².

Determination of fracture toughness (K_{Jc})^{ABI} from ABI tests at various temperatures.

Fracture Toughness determination in the Transition Region:

A new model has been successfully applied to ferritic steels.⁶ It utilizes the value of the critical fracture stress " σ_f ", the slope of the indentation load-depth " S ", the material yield parameter " A ", Meyer index " m ", the ratio of indentation maximum contact pressure to the mean contact pressure " μ ", and the ball indenter diameter " D " to calculate the Indentation Energy to Fracture *IEF* or W_T from the following equation instead of the earlier Equation No. 5.

$$IEF = \frac{2A^2 D^2}{\pi S} \left(\frac{\pi \sigma_f}{4\mu A} \right)^{\frac{2m-2}{m-2}} = W_T \quad (7)$$

The values of A , m , and S are determined from the ABI test results. The value of μ was found to be 1.1 from finite element analysis using ABAQUS code simulation.⁶ The value of the fracture stress is calculated from the following equation:²³

$$\sigma_f = \sigma_y \left[1 + \ln \left(1 + 2360 \left(\frac{K_{Ic}}{\sigma_y} \right)^2 \right) \right] \quad (8)$$

Where σ_y is the yield strength and C_{hic} is the cleavage fracture toughness in SI units.

The Charpy impact energy and the static fracture toughness, K_{Jc} , have non-zero lower shelves even at very low test temperatures. Hence, the fracture energy per unit area, W_f , can be given by:

$$W_f = W_0 + W_T \quad (9)$$

where W_0 is the lower shelf energy per unit area, and W_T is the temperature-dependent energy.

For ferritic steels (with yield strength of 275 to 825 MPa or 40-120 ksi) the fracture toughness (median value) versus temperature curve in the transition temperature region is expressed by the master curve (ASTM E-1921-97):²⁴

$$K_{Jc} (med) = 30 + 70e^{0.019(T-T_0)} \quad \text{MPa}\sqrt{\text{m}} \quad (10)$$

where T is the test temperature and T_0 is the reference temperature when $K_{Jc} = 100 \text{ MPa}\sqrt{\text{m}}$. From the above equation, the lower shelf fracture toughness is $30 \text{ MPa}\sqrt{\text{m}}$, hence, the lower shelf energy, W_0 , is calculated to be 2170 J/m^2 . The fracture toughness determined from ABI tests can then be calculated from:

$$(K_{Jc})^{ABI} = \sqrt{2E(W_T + W_0)} \quad (11)$$

where E is the elastic modulus.

Applying this equation to ABI tests on 73W weld from ORNL we obtained values in excellent agreement with those from 27 destructive 1T CT (1 inch thick compact) fracture toughness specimens as shown in Fig. 10. The T_0 values from the destructive K_{Jc} and the nondestructive $(K_{Jc})^{ABI}$ tests are -63°C and -65°C , respectively. The ABI tests were conducted using a 1.57-mm diameter tungsten carbide indenter at various low test temperatures. The ABI-determined fracture toughness test results were fitted with an exponential curve. The best fit curve was of the same shape as the fracture toughness master curve of Equation 10. Similar results were obtained from ABI tests on the HSSI 72W weld from ORNL.

The above model was used by Byun to determine fracture toughness from ABI tests on five forgings and four welds of Korean nuclear pressure vessel materials.⁶ Byun's results are in good agreement with the master curve (ASTM E1921-97) as shown in Figures 11 and 12. Byun's ABI tests were conducted using a 0.51-mm diameter tungsten carbide indenter.

Although the results shown in Figures 10 through 12 are very good, the ABI-determined fracture toughness values (calculated using equations 7, 9, and 11) are sensitive to the critical fracture stress values (calculated here from Equation 8). Currently, there is no standard test method to measure the critical fracture stress. In this project, we have developed a robust conservative method to determine the critical fracture stress for unirradiated ferritic steels as shown later. Furthermore, all the results shown in Figures 10-12 are from unirradiated materials. It is believed that the critical fracture stress does not change with irradiation. This can be verified by performing ABI tests at low temperatures on irradiated materials in any available hot cell.

The critical fracture stress was calculated from the yield strength at liquid nitrogen temperature (-196°C) and assuming a fracture toughness of $35 \text{ MPa}\cdot\text{m}^{0.5}$ for the fracture toughness at liquid nitrogen (slightly higher than the median value of the fracture toughness master curve at low temperatures). We have tested triplicate miniature tensile specimens at -196°C from three carbon steels from Brazil, and from one nuclear pressure vessel steel from Oak Ridge National Laboratory (ORNL). The yield strength values for all four materials varied from 965 MPa to 1007 MPa (140 to 146 ksi) with an average of 983 MPa (142.6 ksi). The yield strength values for these four materials at room temperature ranged from 379 MPa to 565 MPa (55 ksi to 82 ksi). This indicates that at very low temperatures the yield strength values become independent of the chemical composition, thermo-mechanical treatment, and the room temperature properties. This also explains the convergence, at low test temperatures, of the fracture toughness Master Curve²⁴ (ASTM Standard E 1921-97) for all ferritic steel materials. Hence, a value of 2344 MPa was calculated from Equation 8 for the critical fracture stress for all ferritic steel materials. Although, the critical fracture stress increases at temperatures above -100°C , this conservative value was used for all test temperatures ranging from 0°C to -150°C . We have tested the use of this critical fracture stress value on one plate (12-inch thick nuclear pressure vessel steel, ORNL HSST Reference Plate 02) and two commercial nuclear pressure vessel steel welds (ORNL 72W and ORNL 73W) from ORNL. These three materials were extensively studied and tested at ORNL in a multi-million dollar program to determine their T_0 (reference temperature corresponding to a fracture toughness level of $100 \text{ MPa}\cdot\text{m}^{0.5}$ as described in ASTM Standard E1921-97). The destructively-determined T_0 values for these three materials are shown in Fig. 13.²⁵ Our current and improved fracture toughness calculation from ABI tests in the transition range include the direct integration of Equation 1 instead of using Equation 7 which assumes a single linear relationship between the indentation load and depth throughout the entire ABI test. The new approach is particularly important for ABI tests at very low temperatures where the critical indentation fracture depth is reached early on the initial indentation load-depth curve. The latter is slightly nonlinear or has a lower linear slope than the rest of the load-depth data from the pre-load value up to the maximum indentation load. This was confirmed by calculating the load-depth curve in the initial region (zero load to the practical pre-load) from non-linear finite element ABI test simulations using the true-stress/true-plastic-strain curves measured from tensile tests on three steels. Dr. Kang Lee, consultant to ATC, performed the finite element analysis using the ABAQUS code.

The test results of determining the reference temperature, T_0 , from ABI test results and using the value of 2344 MPa for the critical fracture stress were -20°C , -49°C , and -61°C (see Fig. 14 and Table 1) while the destructive fracture toughness test results from ORNL were -21°C , -54°C , and -63°C for the HSST Plate 02, 72W, and 73W, respectively. These excellent results (T_0 agreement within 5°C) indicate that the use of 2344 MPa for the critical fracture stress is appropriate for all ferritic steels. Furthermore, similar to the ASTM fracture toughness Master Curve method, it is recommended that ABI tests be performed in at least triplicates at three test temperatures near the reference temperature, T_0 , of the test material. This new approach minimizes the conservatism of using a single value for the critical fracture stress for the three test temperatures (near T_0 , $T_0 - 10^{\circ}\text{C}$, and $T_0 + 10^{\circ}\text{C}$) and increases the probability that the ABI-measured fracture toughness values might fall between the 95 and the 5% confidence limit curves of the Master Curve.

Equation 8 was used to calculate the critical fracture stress from K_{Ic} and yield stress values at various temperatures for HSST Plate 02, ORNL 72W, and ORNL 73W RPV steels. Figures 15 through 17 show plots of the critical fracture stress and the yield strength as a function of temperature. These figures show that the critical fracture stress increases above -50°C and -100°C , for the plate and the welds, respectively. Furthermore, the figures show that the critical fracture stress increases while the yield stress decreases with increasing test temperature. Hence, the ratio of "critical fracture stress to the yield stress" increases with increasing temperature as shown in Figures 18 through 20 for the three RPV steels. Figure 21 shows a combined plot of the critical fracture stress as a function of test temperature for the three RPV steels. This plot shows that the use of 2344 MPa for the critical fracture stress for the three materials over the ABI test

temperature range is conservative. Furthermore, the lower curve of the HSST Plate 02 as compared to the 72W and 73W welds is consistent with its higher reference temperature value. This is also confirmed by the lower slope of the ratio of the critical fracture stress to the yield stress for the Plate 02 as compared to the 72W and 73W RPV steels (Fig. 22). It is interesting to note that the three materials show linear correlations with high regression values (0.949 to 0.986) as shown in Figures 18 through 20. Hence, if the ratio of the critical fracture stress to the yield stress is known for a particular steel, then the critical fracture stress can be calculated from the ABI-measured yield stress at the desired test temperature (near its reference temperature). Moreover, the conservatism of using a single value of 2344 MPa can be slightly reduced by using the slightly increased values of the critical fracture stress in the transition region (e.g. the conservative increasing values of the critical fracture stress between -100°C and 0°C of Plate 02 could be used for all ferritic steels).

The current ABI software of the SSM system allows the user to: (1) correct for the indenter and system compliance when testing at low temperatures where the depth transducer is located outside the environmental chamber, (2) correct for the initial ABI pre-load and its associated load-depth slope to produce a smoothly increasing mean indentation pressure with increasing depth, (3) input the critical fracture stress, and (4) select any number of ABI tests at the desired temperatures to be included in the fracture toughness Master Curve calculation and the determination of the reference temperature. Additionally, the software provides tabulated data and a graph of the fracture toughness Master Curve with its associated 95% and 5% confidence limits.

Three Brazilian steels were tested using a 0.76 mm (0.030 inch) diameter tungsten carbide indenter by ATC's subcontractor Dr. Kang Lee in order to evaluate: (1) the use of the new method for calculating fracture toughness values from ABI tests on ferritic steels other than RPV materials, (2) the degree conservatism in using a critical fracture stress value of 2344 MPa, and (3) the minimum number of ABI tests required to determine a reliable and accurate reference temperature from the ABI-determined fracture toughness Master Curve. The three materials are identified by the last names of the researchers who brought them to the United States (during their temporary assignments at The University of Tennessee, Knoxville) as ITO, Tasso, and Alexandre. The reference temperature values, as determined from destructive fracture toughness tests at ORNL, for the three materials are -69°C, -115°C, and -110°C for the ITO, Tasso, and Alexandre, respectively. The ABI-measured fracture toughness Master Curves for the ITO, Tasso, and Alexandre steels (Figures 23 through 25) were obtained from 44, 22, and 3 ABI tests, respectively. Nineteen (19) destructive fracture toughness tests were conducted at -90°C at ORNL on the Tasso Brazilian steel (Fig. 23). This figure shows that the ABI-measured fracture toughness values followed the median Master Curve except at high test temperatures where the values were lower than the median curve because of the use of a single value of the critical fracture stress over the whole transition temperature region. Hence, ABI tests should be conducted near the reference temperature (Fig. 24). Furthermore, Fig. 25 demonstrates that a minimum of three ABI tests at a single temperature (near the reference temperature) could be used to determine the median fracture toughness Master Curve. However, since ABI tests are fast and localized it is recommended that three to five tests each should be conducted at three temperatures near the reference temperature. The ABI-determined reference temperature values for the three materials were in excellent agreement with those from destructive fracture toughness tests as shown in Table 2 and in Fig. 26. Table 2 shows that the ABI-determined reference temperature values are consistently conservative (1 to 5°C higher than the destructively-determined values) for three RPV and three Brazilian steels over a wide range of T_0 values from -115°C to -21°C.

It is important to note here that researchers from three laboratories (namely, Advanced Technology Corporation, Korea Atomic Energy Research Institute, and The University of Tennessee, Knoxville) have successfully used three different size indenters (0.51 mm, 0.76 mm, and 1.57 mm diameter) on fifteen ferritic steel materials (including plate, weld, and forging). All laboratories have produced ABI-determined reference temperature values in excellent agreement with those from the destructive fracture toughness tests.

Determination of fracture toughness in the ductile/upper shelf region

The critical fracture stress model is not applicable for testing in the ductile (upper shelf) region. Hence, the following correlation was used to estimate the initiation fracture toughness, K_{JIC} , from the ABI-measured stress-strain data at room temperature and higher:¹⁹

$$K_{JIC} = \text{constant} (k \cdot d \cdot n \cdot \sigma_y)^{0.5} \quad (12)$$

where d is the grain size of the test material, k is the strength coefficient, n is the strain-hardening exponent, and σ_y is the yield strength. The grain size can be determined from archive RPV materials. For A533-B EPRI Heat 1bK, d was assumed to be 25 μm . The value of the constant in the above fracture toughness equation was 170. For ABI tests at room temperature and at 288°C (550°F), the following results were obtained:

ABI Test No. 1BK-RT-K2-2 (Room Temperature):

$\sigma_y = 411.6 \text{ MPa}$, $k = 972.9 \text{ MPa}$, $n = 0.198$

$$K_{JIC} = \text{constant} (k \cdot d \cdot n \cdot \sigma_y)^{0.5} = 239.3 \text{ MPa.m}^{0.5}$$

This is in good agreement with the measured fracture toughness value of 240.7 $\text{MPa.m}^{0.5}$ (Ref. 26).

ABI Test No. EBB-T31+550F (288°C/550°F):

$\sigma_y = 438.5 \text{ MPa}$, $k = 902.6 \text{ MPa}$, $n = 0.192$

$$K_{JIC} = \text{constant} (k \cdot d \cdot n \cdot \sigma_y)^{0.5} = 234.3 \text{ MPa.m}^{0.5}$$

This is in good agreement with the measured fracture toughness value of 205.5 $\text{MPa.m}^{0.5}$ (Ref. 26).

Also, using the miniature tensile test results, estimated fracture toughness values of 233.6 $\text{MPa.m}^{0.5}$ and 225.3 $\text{MPa.m}^{0.5}$ are calculated for the room temperature and the 288°C (550°F), respectively.

Standardization of the ABI Test Techniques to Measure Stress-Strain Curves and Fracture Toughness of Ferritic Steels.

A large ABI test data base was created and several papers were published in order to start a standardization process of the SSM technology and its ABI test techniques. ATC's principal investigator (Fahmy M. Haggag) and ATC's consultant (Prof. John D. Landes, ASTM Fellow) tried to commission a new Task Group for the creation of a new American Society of Testing and Materials (ASTM) standard test method for ABI testing to determine stress-strain curves and fracture toughness of ferritic steels. The new task group efforts, spanning two years under three ASTM Committees (E-28, E-10, and E-8), failed due to the fact that ATC holds a US Patent, No. 4,852,397, on its SSM system and its ABI techniques as proven by the minutes of the ASTM Subcommittee E08.08 meeting of May 1999 (Seattle, WA). A copy of Dr. Landes' request for the development of a new standard "Standard Test Methods for Automated Ball Indentation (ABI) Testing of Steel Samples and Structures to Determine Stress-Strain Curves and Fracture Toughness," and the minutes of

the meeting are included in Appendix A of this report to demonstrate the discrimination of ASTM against innovation and patented instruments and techniques. Current ASTM regulations with respect to patented items in their standards are vague, anti-patent, and are neither understood nor followed by ASTM staff and committee chairs. The completed ASTM Form "Request for the Development of a New Standard," dated March 26, 1999, was signed by Dr. John D. Landes as the Chairman of Subcommittee E08.08 on Elastic-Plastic Fracture, and he should be allowed (as subcommittee chairman) to initiate any appropriate new task group according to ASTM bylaws. However, neither Mr. Hopkins (E08 chairman) nor most of the executive subcommittee members attended the two technical presentations of E08.08 given by Haggag and Landes on the afternoon prior to the non-technical and anti-patent voting of the executive subcommittee E08.90.

ATC's initial standardization efforts with ASTM Committee E28 are summarized as follows: (1) at Haggag's request to the executive subcommittee, a new task group was commissioned as E28.90.02 in November 1997. (2) In May 1998, the first meeting of the new task group was sabotaged by members of Subcommittee E28.06 which is heavily dominated by the manufacturers of micro-hardness and nano-indentation test instruments. Task Group E28.90.02 was disbanded as a result of the conspiracy of the officers of E28 as proven by the minutes of their November 1997 (E28.90, E28.06, and E28.06.11) and the May 1998 meetings. (3) ATC has put ASTM on legal notice for any future violation of its subject US patent as a result of the improper activities of Task Group E28.06.11. This task group is trying to develop a new generic **Standard Practice** on instrumented indentation which might include ATC's patented ABI technology without any reference to ATC's patent as required by ASTM procedures. (4) Task Group E28.06.11 did not complete any ASTM form to start their activities, did not have a title for their commercially-driven standard practice, and did not follow the Form and Style Manual of ASTM for developing new standards. (5) Mr. John Newby, the Chairman of E28 Committee on Mechanical Testing, demonstrated his lack of knowledge of ASTM regulations by not knowing the difference between the types of ASTM standards, particularly the "**Standard Practice**" which does not produce test result and the "**Standard Test Method**" which produces a test result. (6) Mr. Newby again displayed his ignorance of ASTM Procedures and Bylaws and US Patent Laws when he disbanded the new task group E28.90.02 (which was commissioned to develop a **Standard Test Method**) using his **false claim** that it was a duplicate effort of the **non-comparable Standard Practice** of E28.06.11. Furthermore, Mr. Newby has blocked the initiation of any new task group for the SSM technology under the other two ASTM Committees E-10 (on Nuclear Technology and Applications) and E-08 (on Fatigue and Fracture) because of his political ties to the manufacturers of testing equipment and because of the poor performance and lack of leadership of the ASTM staff.

Our standardization activities under ASTM Committee E10 started by submitting the ASTM "P4 Form" for the development of a new standard to the E10.02 subcommittee. In January 1999, Haggag and Landes made two presentations on the status of the SSM technology and its ABI techniques and presented stress-strain and fracture toughness test results on various RPV steel plates and welds. Following the presentations, Dr. Landes made a motion to initiate a new task group and the motion was seconded by Thomas Mager (Westinghouse Electric Corporation), and was third by Art Lowe (consultant and retiree from Babcock and Wilcox Corporation). Stephen Byrne (chairman of Subcommittee E10.02) prevented the voting on the motion by stating that he wanted to consult with the Chairman of Committee E28. Landes and Haggag explained to Byrne what happened earlier with E28 and that John Newby did not want to allow the new task group under his committee. Byrne promised that if E28 would not allow the task group that we would continue our activities under E10.02. More than two months later, Byrne sent Haggag an e-mail message stating that Newby told him that we should include our Standard Test Method activities under Task Group E28.06.11 even though it was for a Standard Practice and not a Standard Test Method. **Stephen Byrne failed to keep his promise and supported the wrong position of John Newby.** Furthermore, our written complaint to Mr. Ken Pearson (Vice President of ASTM) was ignored. After two years of intensive efforts, including four meetings, ATC concluded that ASTM is not only anti-patent but encourages the competitor vendors to draft standards which encourage patent infringements. Hence, we concluded that if any DOE managers want to pursue this activity in the future, they should request it directly from ASTM.

Thousands of ABI tests were conducted by several organizations over the past ten years and numerous publications demonstrate the accuracy and reproducibility of the ABI-measured stress-strain curves and their agreement with those from tensile tests. Furthermore, four laboratories (namely ATC, Korea Atomic Energy Research Institute, North Carolina State University, and The University of Tennessee, Knoxville) have performed ABI tests at low and high test temperatures. The ABI-determined fracture toughness Master Curve results, produced by these four laboratories, are in excellent agreement with the destructive fracture toughness test results used in the development of the Master Curve of ASTM Standard E-1921-97. Hence, any nuclear utility can use the SSM technology and reference this report and/or the other publications in *Scripta Materialia* and *Journal of Nuclear Materials* when submitting their test results to the United States Nuclear Regulatory Commission (US NRC) for structural integrity evaluation (during license renewal or for service life extension). Currently, several government regulatory offices (e.g. Department of Transportation, Office of Pipeline Safety) allow the submission of waivers or exemptions to the Code of Federal Regulations for the use of a demonstrated technology such as the innovative SSM technology. A waiver submission might be a better and a faster approach as compared to the creation of a voluntary-consensus ASTM standard that might take many years (considering the poor attitude demonstrated by ASTM staff and committee chair toward the need for one of the major objectives of this Department of Energy project). Furthermore, any ASTM standard is not mandatory unless referenced in the particular government regulation. For example, ASTM standard E-1921-97 for the fracture toughness Master Curve, although its development was funded mostly by the US NRC, is not referenced by any US NRC regulation or guide. Most of the surveillance capsules of US nuclear utilities do not contain fracture toughness specimens. Hence, Charpy specimens could be precracked and tested in slow three-point bending as fracture toughness specimens according to ASTM standard E-1921-97. However, since Charpy specimens are 10-mm thick, some of their fracture toughness test results might be invalid according to the thickness validity requirement. Hence, such nuclear utilities might choose to use the innovative, faster, material-efficient, and cheaper ABI testing to produce fracture toughness results and a Master Curve that are localized and specimen-size independent.

MINIATURE SSM SYSTEM AND FIELD TESTING DEMONSTRATION

The initial ABI testing of a 6-inch diameter carbon steel pipe with a nominal thickness of 0.27 inch was conducted using three sizes of tungsten carbide indenters: 0.062-inch, 0.030-inch, and 0.020-inch diameter. The testing head of the SSM system was mounted on the pipe using four aluminum V-blocks as shown earlier in Figure 1. Test results on machined smooth areas were in very good agreement with those from locally polished areas. An example of the ABI data and test results using the three indenters is shown in Fig. 27. All ABI tests were carried out up to a maximum indentation depth of 30% of the indenter radius. The maximum indentation load for the three indenters was approximately 460 lbs, 120 lbs, and 60 lbs, resulting in approximate final indentation depths of 0.009 inch, 0.0045 inch, and 0.003 inch. As shown in Figure 27, the true-stress/true-plastic-strain curves from all three indenter sizes produced the same stress-strain curves despite the different test volume sampled in each ABI test.

A field demonstration was conducted using the miniature Portable/In-Situ Stress-Strain Microprobe system (Model SSM-M1000) outdoors where it was operated using a small booster battery pack and the testing head (weighing 25 lbs) was temporarily attached to a 6-inch diameter carbon steel pipe (obtained from Columbia Gas Transmission Corporation) with two small manual magnets having on/off switches. Photographs of the SSM-M1000 system and the pipe are shown in Fig. 28 (indoor for clarity of the photographs) and in Fig. 29 (outdoors for the actual field testing using the battery pack and the magnetic mounts on the pipe). Both the 0.020-inch diameter and the 0.030-inch diameter indenters produced successful ABI test results using the magnetic mounts.²⁷

An example of the field indentation load-depth curve (using a 0.020-inch diameter indenter) and a comparison of true-stress versus true-plastic-strain curves from ABI and tensile tests are shown in Fig. 30. This figure demonstrates that the ABI data using the magnetic mounts, the battery pack, and the 0.020-inch diameter indenter are very accurate and reliable. The success of this field ABI test is demonstrated by the excellent comparison of the true-stress/true-plastic-strain curves from ABI and miniature tensile tests shown in Fig. 30 (b). Table 3 shows a summary of (a) ABI test results on the pipe using a 0.020-inch diameter tungsten carbide indenter, (b) ABI test results on the end tabs of two miniature tensile specimens, (c) test results of two miniature tensile specimens, and (d) test results from two large tensile specimens. The table shows very good agreement between all ABI and tensile test results. This demonstrates the easy field applicability of the SSM system to measure the yield strength and the stress-strain curve of metallic structures in a nondestructive, reliable, and accurate manner. One of the field ABI tests was recorded using a video camera. The 5-minute digital video and the 2-minute computer screen capture file (showing the real-time ABI test data, analysis, and comparison with the true-stress/true-plastic-strain curve from miniature tensile test of the pipe material) are available on a CD from ATC or can be viewed on ATC's website: www.atc-ssm.com. The two video files demonstrate the easy applicability of the SSM-M1000 system to nondestructively test metallic structural components reliably and accurately.

SUMMARY

Evaluating the structural integrity of nuclear reactor pressure vessels (RPVs) through material properties determination is a critical safety assessment. Currently, the changes in mechanical properties of RPV materials, susceptible to radiation embrittlement, are monitored by periodic destructive testing of surveillance specimens (mostly Charpy impact and tensile specimens and fracture toughness if available). The patented Portable/*In-Situ* Stress-Strain Microprobe™ (SSM) system (1996 R&D 100 Award Winner), developed by Advanced Technology Corporation (ATC), utilizes an Automated Ball Indentation™ (ABI) test technique which is nondestructive and provides a localized direct measurement of the stress-strain curve of metallic materials. In Phase I of this project, new testing and analytical procedures were successfully developed and evaluated in various tasks: (1) ABI testing at higher strain rates, (2) innovative ABI testing at temperatures from -130°C/250°F to 288°C/550°F with excellent repeatability and agreement with stress-strain curves from tensile specimens, (3) the indentation load versus depth data were used to determine a new fracture parameter (Indentation Energy to Fracture, IEF) which was successfully correlated to Charpy impact energies as a function of test temperature using the critical fracture stress model for ABI tests in the transition temperature region, (4) estimation of ductile fracture toughness from ABI tests at room temperature and higher temperatures using the critical fracture strain model, (5) *in-situ* ABI testing of a steel plate from a commercial RPV (after partial removal of its thin inner stainless steel cladding), and (6) numerous ABI tests were conducted on archive commercial RPV steels at ATC as well as on irradiated and irradiated+annealed broken Charpy specimens (A533B plate and 73W weld) in the hot cell at Oak Ridge National Laboratory. The ABI test results on the irradiated and irradiated+annealed specimens successfully quantified the degree of embrittlement as well as the percentage of toughness recovery following thermal annealing.

In Phase II, the development of the IEF parameter was successfully completed to determine fracture toughness, K_{Jc} , values in the temperature transition region. ABI and tensile tests were conducted at liquid nitrogen temperature (-196°C) in order to determine the critical fracture stress for various ferritic steels. A new procedure was developed to determine the fracture toughness Master Curve from nine ABI tests at three test temperatures using a 0.51-mm diameter tungsten carbide indenter. The small indenter was very successful in testing narrow welds and heat-affected-zones (HAZs). Other indenter sizes of 0.76-mm and 1.57-mm were used on base and weld metal samples. The ABI-determined fracture toughness Master Curve produced reference temperature, T_0 , values in excellent agreement (within 5°C) with those from destructive fracture toughness tests on several nuclear RPV steels as well as on other ferritic steels (e.g. oil and gas pipeline materials, structural steels, etc.) covering a wide range of T_0 values from -115°C to -21°C. The innovative ABI-determined fracture toughness Master Curve technique will allow reliable and accurate determination of fracture toughness from very small volumes of material (highly desirable for small welds and HAZs which can not be tested by other conventional techniques). Furthermore, this will eliminate the need to machine, precrack, side groove, or reconstitute neutron-irradiated specimens. Hence, structural integrity evaluation, based on fracture mechanics analysis, of aged nuclear pressure vessels and other critical ferritic steel structures (e.g. pipeline, bridges, ships, high-rise steel buildings, etc.) will be easier, faster, better, and cheaper using the newly-developed innovative SSM technology.

Another important accomplishment of this project is the successful development of a miniaturized testing head of the SSM system for use inside a nuclear reactor. The electronics cabinet (containing the motor drive, signal conditioning modules and data acquisition interface) of the SSM system was also miniaturized. The new miniature SSM system, Model SSM-M1000, was successfully demonstrated on various curved surfaces using manual magnets to temporarily attach/secure the testing head to the pipe section. The SSM-M1000 system can be operated using a small portable battery pack which is useful in testing oil and natural gas pipeline in remote areas (digital video files of field testing demonstrations are available on a CD from ATC or for viewing on ATC's web site: www.atc-ssm.com). Although most of the new developments in SSM technology were initiated in response to the needs of the nuclear industry, several other industries will have

immediate applications (e.g. natural gas and oil pipelines with unknown properties installed in service since the early 1900s, ships with unknown steel plates and welds, high pressure cylinders, steel bridges, etc.).

PHASE III: TECHNOLOGY TRANSFER

This report successfully completes Phase II and most of Phase III (technology transfer) of this DOE SBIR project. The commercial state-of-the-art SSM systems (including the laboratory version which allows testing at various low and high temperatures and the new miniature system which allows easy field testing and can be adapted for remote testing) are currently offered by ATC for sale and testing services to US and international clients. Applications of the SSM technology are not limited to ferritic steels and covers the structural integrity evaluation and failure analysis needs in many industries. Recent examples of the innovative SSM technology applications include:

- (1) Testing of aged uranium alloys at the Y-12 weapons plant in Oak Ridge, Tennessee (1998).
- (2) Sale and commissioning of an SSM system at Los Alamos National Laboratory (May 1999).
- (3) SSM testing of ferritic steel vessels and pipeline welds to determine stress-strain curves and fracture toughness at low temperatures for several US and European companies (1998,1999).
- (4) Testing of automotive air bag inflator pressure vessels, Atlantic Research, Inc., Knoxville, TN, (1998, 1999).
- (5) Testing of automotive spot welds, Edison Welding Institute, Columbus, Ohio (1998, 1999).
- (6) Testing of very small solder bumps on a computer chip for automotive applications, Oak Ridge National Laboratory (1998).
- (7) Testing of stress-strain gradients in a welded aluminum sample (weld, heat-affected-zone, base metal) from the International Space Station, NASA AMES, Moffett Field, California (1997).
- (8) Testing of Nickel coating, TAFA, Inc., 1998.
- (9) Testing of various natural gas pipeline materials manufactured in 1931 through 1978. ATC submitted a comprehensive report No. ATC/DOT/990901 to DOT/OPS in September 1999. This report (funded solely by ATC) is entitled: Nondestructive Determination of Yield Strength and Stress-Strain Curves of In-Service Transmission Pipelines Using Innovative Stress-Strain Microprobe™ Technology. The SSM technology will provide a nondestructive testing alternative to the natural gas and oil pipeline industry for evaluating portions of pipelines with unknown properties as well as to improve the transmission and distribution efficiency safely and economically.
- (10) ATC will be performing a subcontract to Fleet Technology, Inc. (Canada) to determine the fracture toughness of two ship plates and welds from the Ship Structure Committee (SR 1385) using the innovative SSM technology. This will create a new application for structural integrity evaluation of many cargo ships and oil tankers.

The only remaining Phase III application (related to the nuclear topic of this SBIR project) is the ABI testing at low test temperatures to determine the fracture toughness Master Curve of irradiated and irradiated+annealed samples from nuclear utilities or from samples irradiated in test reactors. This will

verify the limited data available in literature indicating that the critical fracture stress of ferritic steels does not change with irradiation. Currently, no US nuclear utility has an immediate need for fracture toughness evaluation of its nuclear reactor pressure vessel. However, ATC is ready to accomplish this work when it receives a Phase III grant from DOE or a contract from any nuclear utility or vendor.

REFERENCES

1. Haggag, F. M., "Field Indentation Microprobe for Structural Integrity Evaluation," U.S. Patent No. 4,852,397, 1989.
2. Haggag, F. M., et al., "Use of Portable/In Situ Stress-Strain Microprobe System to Measure Stress-Strain Behavior and Damage in Metallic Materials and Structures," *ASTM STP 1318*, 1997, pp 85-98.
3. Haggag, F. M., "In-Situ Measurements of Mechanical Properties Using Novel Automated Ball Indentation System," *ASTM STP 1204*, 1993, pp. 27-44.
4. Haggag, F. M., et al., "Use of Automated Ball Indentation Testing to Measure Flow Properties and Estimate Fracture Toughness in Metallic Materials," *ASTM STP 1092*, 1990, pp. 188-208.
5. Haggag, F. M. et. al., "Indentation-Energy-to-Fracture (*IEF*) Parameter for Characterization of DBTT in Carbon Steels Using Nondestructive Automated Ball Indentation (ABI) Technique," *Scripta Materialia*, Vol 38, No. 4, 1998, pp 645-651.
6. Byun, T. S., et al. "A Theoretical Model for Determination of Fracture Toughness of Reactor Pressure Vessel Steels in the Transition Region from Automated Ball Indentation Test," *Journal of Nuclear Materials*, 252, 1998, pp. 187-194.
7. Byun, T. S., et al. "Measurement of Through-the-Thickness Variations of Mechanical Properties in SA508 Gr.3 Pressure Vessel Steels Using Ball Indentation Test Technique," *International Journal of Pressure Vessels and Piping*, 74, 1997, pp. 231-238.
8. Haggag, F. M., et al., "A Novel Stress-Strain Microprobe for Nondestructive Evaluation of Mechanical Properties of Materials," *Nondestructive Evaluation and Materials Properties III*, The Minerals, Metals & Materials Society, 1997, pp. 101-106.
9. Haggag, F. M., et al., "Using Portable/In-Situ Stress-Strain Microprobe System to Measure Mechanical Properties of Steel Bridges During Service," SPIE Proceedings on *Nondestructive Evaluation of Bridges and Highways*, Vol. 2946, 1996, pp. 65-75.
10. Haggag, F. M., et al., "Nondestructive Detection and Assessment of Damage in Aging Aircraft Using a Novel Stress-Strain Microprobe System," SPIE Proceedings on *Nondestructive Evaluation of Aging Aircraft, Airports, and Aerospace Hardware*, Vol. 2945, 1996, pp. 217-228.
11. Haggag, F. M. et al., "Characterization of Strain-Rate Sensitivity of Sn-5%Sb Solder Using ABI Testing," *Microstructures and Mechanical Properties of Aging Materials II*, TMS 1995, pp. 37-44.
12. Haggag, F. M., et al., "Application of Flow Properties Microprobe to Evaluate Gradients in Weldment Properties," *International Trends in Welding Sciences and Technology*, ASM, 1993, pp. 629-635.

13. Haggag, F. M., et al., "Measurement of Yield Strength and Flow Properties in Spot Welds and Their HAZs at Various Strain Rates," *International Trends in Welding Sciences and Technology*, ASM, 1993, pp. 637-642.
14. Haggag, F. M. et al., "Structural Integrity Evaluation Based on an Innovative Field Indentation Microprobe," *ASME PVP-Vol. 170*, 1989, pp. 101-107.
15. Haggag, F. M. and Nanstad, R. K., "Estimating Fracture Toughness Using Tension or Ball Indentation Tests and a Modified Critical Strain Model," *ASME PVP-Vol. 170*, 1989, pp. 41-46.
16. Haggag, F.M., et al., "The Use of Field Indentation Microprobe in Measuring Mechanical Properties of Welds," *Recent Trends in Welding Science and Technology*, TWR'89, ASM, 1990, pp. 843-849.
17. Druce, S. G., et al., "The Use of Miniature Specimen Techniques for the Assessment of Material Condition," *ASME PVP-Vol. 252*, 1993, pp. 58-59.
18. Iskander, S. K., Sokolov, M.A., and Nanstad, R.K., "Comparison of Different Experimental and Analytical Measures of the Thermal Annealing Response of Neutron-Irradiated RPV Steels," *ASTM STP 1325*, 1999, pp. 403-420.
19. Haggag, F. M., *In-Situ Nondestructive Measurements of Key Mechanical Properties of Pressure Vessels Using Innovative Stress-Strain Microprobe (SSM) Technology*, DOE/ER/82115-1, 1997.
20. Timoshenko, S. P., and J. N. Goodier, *Theory of Elasticity*, 3rd Ed., McGraw-Hill, 1970 pp. 409-414.
21. F. A. McClintock and A. S. Argon, Editors, *Mechanical Behavior of Materials*, Addison-Wesley, 1966, pp.372-373.
22. Nanstad, R. K., McCabe, D. E., Haggag, F. M., Bowman, K. O., and Downing, D. J., "Statistical Analyses of Fracture Toughness Results for Two Irradiated High-Copper Welds," *Effects of Radiation on Materials, 5th International Symposium*, ASTM STP 1125, R. E. Stoller, A. S. Kumar, and D. S. Gelles, Eds., American Society for Testing and Materials, Philadelphia, 1992, pp. 270-91.
23. J. Malkin, J. and Tetelman, A. S., "Relation Between K_{Ic} and Microscopic Strength for Low Alloy Steels," *Engineering Fracture Mechanics* 3, 1971, pp. 151-167.
24. ASTM Standard E 1921-97, "Standard Test Method for Determination of Reference Temperature, T_0 , for Ferritic Steels in the Transition Range," *Annual Book of ASTM Standards*, Vol. 3.01, 1998.
25. Merkle, J. G., et al., *Technical Basis for an ASTM Standard on Determining the Reference Temperature, T_0 , for Ferritic Steels in the Transition Range*, NUREG/CR-5504, ORNL/TM-13631, November 1998, page 1-25.
26. Server, W. L. and Oldfield, W., *Nuclear Pressure Vessel Steel Data Base*, EPRI Report NP-933, 1978.
27. Haggag, F. M., *Nondestructive Determination of Yield Strength and Stress-Strain Curves of In-Service Transmission Pipelines Using Innovative Stress-Strain Microprobe™ Technology*, ATC/DOT/990901, September 1999.

(a)

(b)

Fig. 1 (a) Stress-Strain Microprobe system (Model SSM-M1000) configured for field testing of a pipe. (Testing head weight is 25 lbs, 22 lbs electronics cabinet, and a 9 lbs notebook computer) (b) Details of the 6-inch diameter pipe, V-blocks, indenter chuck and indenter, indentation depth sensor (spring-loaded LVDT), and two indents made using a 0.030-inch diameter indenter.

Fig. 2 Photograph of the SSM system in a laboratory configuration with an environmental chamber, a positioning table, and a video camera installed on its load frame (notice the LVDT/depth-transducer is mounted on a bracket outside chamber). The positioning table and video camera allow accurate ABI testing of any heat-affected-zone (HAZ) location.

Fig. 3 Sixteen (16) Automated Ball Indentation (ABI) tests were made at different test temperatures on a small area of one broken half of a Charpy V-notch (CVN) impact specimen using a 0.76-mm (0.030-inch) diameter tungsten carbide indenter. The nondestructive/localized ABI tests allow multiple/repeated use of any specimen with smooth parallel surfaces. Furthermore, the innovative analysis of the new fracture parameter (Indentation Energy to Fracture "IEF" which is calculated from the indentation load-depth curves of ABI tests conducted at various temperatures) produces the fracture toughness Master Curve of ferritic steels.

Fig. 4 The room-temperature ABI test results on A533B-1 RPV steel (HSST Plate 02) shown in this figure demonstrate the capability of the ABI test to measure stress-strain curves of irradiated/embrittled and post-irradiated/annealed Charpy specimens and to quantify the degree of ductility recovery following thermal annealing (nondestructively and using minimal test material).
Reference: Haggag, F. M. et al., "Use of Portable/In Situ Stress-Strain Microprobe System to Measure Stress-Strain Behavior and Damage in Metallic Materials and Structures," ASTM STP 1318, 1997, pp. 85-98.

Fig. 5 Indentation Energy to Fracture (IEF) versus ABI test temperature for ORNL 72W weld (base metal, weld nugget, and heat-affected-zone).

Fig. 6 Example of Indentation load versus depth in an ABI test using a 0.51-mm (0.020-inch) diameter tungsten carbide indenter on X42 ferritic steel material.

Fig. 7 Comparison of Stress-Strain Curves from ABI and Tensile Tests on X42 ferritic steel.

Fig. 8 ABI-Measured Stress-Strain Curves at Various Test Temperatures for A533B steel.

Fig. 9 Temperature variation of IEF for A533B steel.

Fig. 10 Comparison between nondestructively ABI-measured $(K_{Jc})^{ABI}$ and destructive 1T CT fracture toughness test results of 73W Weld of ORNL.

Fig. 11 Estimated K_{Jc} of SA508 Gr. 3 steels (base metals).
Reference: *Journal of Nuclear Materials*, Vol. 252, 1998, page 191.

Fig. 12 Estimated K_{Jc} of reactor pressure vessel weld metals.
 Reference: *Journal of Nuclear Materials*, Vol. 252, 1998, page 191.

Fig. 13 Fracture toughness "Master Curve" data of Oak Ridge National Laboratory.
 Original data used to develop the Master Curve of ASTM Standard E1921-97. Reference No. 25.

Fig. 14 Fracture toughness Master Curve obtained from ABI tests on three RPV steels. A 0.51-mm (0.020-inch) diameter tungsten carbide indenter was used to perform 11 ABI tests on Plate 02 (the specimen on the top left of the figure), and 9 ABI tests each on the 72W and 73W weld samples (shown on the left and right lower part of the figure, respectively).

Fig. 15 Effect of test temperature on the critical fracture stress and yield stress of HSST Plate 02.

Fig. 16 Effect of test temperature on the critical fracture stress and yield stress of ORNL 72W weld.

Fig. 17 Effect of test temperature on the critical fracture stress and yield stress of ORNL 73W weld.

Fig. 18 Effect of test temperature on the ratio of critical fracture stress to yield stress for HSST Plate 02. (Although the fracture stress and the yield stress increases and decreases nonlinearly with increasing temperature as shown in Fig. 15, their ratio increases linearly with temperature.)

Fig. 19 Effect of test temperature on the ratio of critical fracture stress to yield stress for ORNL 72W weld. (Although the fracture stress and the yield stress increases and decreases nonlinearly with increasing temperature, their ratio increases linearly with temperature.)

Fig. 20 Effect of test temperature on the ratio of critical fracture stress to yield stress for ORNL 73W weld.
(Although the fracture stress and the yield stress increases and decreases nonlinearly with increasing temperature, their ratio increases linearly with temperature.)

Fig. 21 Effect of temperature on the critical fracture stress of three RPV steels.
The critical fracture stress of HSST Plate 02 changes slowly from -196°C up to -50°C and it has lower values than the two weld materials.

Fig. 22 Effect of test temperature on the ratio of the critical fracture stress to the yield stress for three RPV steels. The linear relationship of HSST Plate 02 has the lowest slope as compared to the two welds.

Fig. 23 Fracture toughness Master Curves determined from 19 destructive fracture toughness tests at -90°C and from 44 ABI tests at various temperatures on the "ITO" Brazilian steel.

Fig. 24 Fracture toughness Master Curve determined from 22 ABI tests on the “Tasso” Brazilian steel.

Fig. 25 Fracture toughness Master Curve determined from 3 ABI tests on the “Alexandre” ferritic steel.
The fracture toughness Master Curve is determined from a minimum of three ABI tests.

Fig. 26 Comparison between the reference temperature from localized ABI test and from destructive fracture toughness tests on six ferritic steel materials.

The reference temperature values from ABI tests are consistently conservative by less than 4°C.

Fig. 27 Example of ABI data and test results performed with the miniature SSM, Model SSM-M1000, on a ferritic steel pipe using 0.51-mm, 0.76-mm, and 1.57-mm (0.020-inch, 0.030-inch, and 0.062-inch) diameter tungsten carbide indenters.

Fig. 28 The SSM system, Model SSM-M1000, shown inside ATC's laboratory with the testing head mounted on a 6-inch diameter carbon steel pipe using the manual magnets (with on/off switches) shown in the detailed photo. The two magnets are connected with an aluminum bracket for alignment on the axial direction of the pipe while the top surface of the bracket is used as the reference surface for the spring-loaded LVDT (indentation depth sensor).

Fig. 29 The SSM system (Model SSM-M1000) is used outdoors for field testing of a 6-inch diameter carbon steel pipe (from Columbia Gas Transmission Corporation). The top photo shows the testing head mounted on the pipe, electronics cabinet, notebook computer, small booster battery pack, inverter (producing 300 Watts of 115VAC from the 12 VDC battery pack), and a small power surge protector. The middle photo shows that mounting the light-weight testing head to the pipe using the two magnets takes only few seconds. The bottom photo shows two small depressions in the pipe from two ABI tests made using a 0.020-inch tungsten carbide indenter.

(a)

(b)

Fig. 30 (a) Data from an ABI test performed, using the magnetic mounts and the battery pack, on a 6-inch diameter carbon steel pipe from Columbia Gas Transmission Corporation using a 0.020-inch diameter tungsten carbide indenter. (b) Comparison between the true-stress/true-plastic-strain curve from the ABI data shown in (a) with the curve from a miniature tensile specimen manufactured from the same pipe.

Table 1 ABI-measured fracture toughness results from three nuclear pressure vessel steels (Plate 02, 72W weld, 73W weld).

Test Name	Temp. [C]	Indenter Diameter, (D) [mm]	Load-Depth Slope, (S) [kN/mm]	Yield Parameter, (A) [MPa]	Yield Strength [MPa]	Strength Coefficient, (K) [MPa]	Strain-Hardening Exponent (n)	Meyer's Num. (m)	Critical Stress [MPa]	Critical Depth (ht) [mic]	Critical Depth Ratio (dt/D)	KJc [MPa*m ^{0.5}]
Plate 02												
69W135-10-2	-10	0.508	4.04	2956	680	1003	0.064	2.316	2353	2.29	0.14	120.6
69W135-10-3	-10	0.508	3.98	2845	654	943	0.061	2.301	2339	2.54	0.14	116.5
69W135-20-1	-20	0.508	3.77	2637	607	931	0.07	2.262	2345	2.79	0.15	90.4
69W135-20-2	-20	0.508	3.77	2865	659	967	0.064	2.303	2350	3.81	0.17	87.7
69W135-20-3	-20	0.508	3.38	2435	560	790	0.058	2.329	2350	12.45	0.31	102.5
69W135-30-1	-30	0.508	3.43	2581	594	845	0.059	2.368	2339	12.19	0.30	100.2
69W135-30-2	-30	0.508	4.24	3050	702	1000	0.06	2.324	2339	3.05	0.15	97.5
69W135-30-2a	-30	0.508	4.09	2901	667	972	0.062	2.270	2344	1.52	0.11	98.2
69W135-30-3	-30	0.508	3.87	2956	680	900	0.049	2.378	2348	6.60	0.23	82.9
69W135-10-1a	-30	0.508	3.92	2767	636	990	0.072	2.303	2348	3.81	0.17	78
69W135-42-1	-42	0.508	3.99	3197	735	954	0.046	2.375	2342	5.33	0.20	82.8
72W weld												
72W03-40-1	-40	0.508	3.32	2442	562	817	0.063	2.308	2339	10.41	0.28	97.9
72W03-40-2	-40	0.508	3.39	2451	564	821	0.063	2.285	2342	8.64	0.26	92.4
72W03-40-3	-40	0.508	3.07	2394	551	748	0.053	2.361	2346	18.54	0.37	118.8
72W03-50-1	-50	0.508	3.28	2506	576	784	0.052	2.346	2350	11.68	0.30	101.5
72W03-50-2	-50	0.508	3.24	2489	572	791	0.055	2.348	2350	13.46	0.32	104.9
72W03-50-3	-50	0.508	3.18	2425	558	778	0.057	2.316	2342	13.46	0.32	106
72W03-60-1	-60	0.508	3.16	2519	579	791	0.054	2.351	2339	12.45	0.31	102.6
72W03-60-2	-60	0.508	3.06	2426	558	755	0.053	2.358	2345	18.03	0.37	117.3
72W03-60-3	-60	0.508	3.62	2481	571	857	0.067	2.256	2341	4.06	0.18	75.2
73W weld												
73W00-50-2	-50	0.508	3.31	2387	549	779	0.059	2.298	2346	12.45	0.31	104.2
73W02-50-1	-50	0.508	3.09	2408	554	775	0.057	2.326	2344	14.22	0.33	107.7
73W02-50-3	-50	0.508	3.24	2521	580	793	0.054	2.334	2338	11.43	0.29	99.9
73W02-60-1	-60	0.508	3.22	2555	587	827	0.058	2.357	2340	12.19	0.30	102
73W02-60-2	-60	0.508	2.92	2464	567	754	0.049	2.413	2343	21.08	0.40	124
73W02-60-3	-60	0.508	3.36	2406	554	805	0.062	2.315	2356	13.46	0.32	107.4
73W02-70-1	-70	0.508	2.98	2527	581	720	0.04	2.407	2337	17.53	0.36	115.6
73W02-70-2	-70	0.508	3.38	2522	580	801	0.055	2.311	2346	8.38	0.26	90.9
73W02-70-3	-70	0.508	3.24	2573	592	792	0.05	2.369	2347	10.92	0.29	98.7

Table 2 Comparison between reference temperature values determined from conventional/ destructive and ABI tests for various ferritic steels.

Ferritic Steel Material	T ₀ (from Destructive Tests), °C	T ₀ (from ABI Tests), °C
A533B, HSST Plate 02	-21	-20
ORNL 72W Weld	-54	-49
ORNL 73W Weld	-63	-61
Brazilian Steel (ITO)	-69	-65
Brazilian Steel (Alexandre)	-110	-107
Brazilian Steel (Tasso)	-115	-114

Table 3 Summary of test results from ABI tests (on the 6-inch diameter pipe and on the end tabs of miniature tensile specimens) and from tensile tests (including miniature and large size specimens) of a carbon steel pipe obtained from Columbia Gas Transmission Corporation.

Test Name	Yield Strength (ksi)	Strength Coefficient (ksi)	Strain Harding Exponent	Ultimate Tensile Strength (ksi)
ABI tests using the field configuration of the Stress-Strain Microprobe System (Model SSM-M1000) operated with a 12 Volt D.C. battery and utilizing magnetic mounts				
Magnet-Battery-20-01	49.3	112.9	0.136	75.2
Magnet-Battery-20-02	48.2	120.4	0.147	78.4
ABI tests using the laboratory configuration of the SSM-M1000 system on the end tabs of a miniature tensile specimen				
20P6-1Col-1	53.1	128.2	0.142	84.3
20P6-1Col-2	51.5	125.2	0.143	82.2
Miniature tensile specimens manufactured with their axes in the circumferential direction				
Col-Gas6A1	50.8	124.7	0.161	81.7
Col-Gas6A2	52.3	134.4	0.178	82.3
Large tensile specimens manufactured with their axes in the axial direction				
906890	50.4			79.2
906891	51.0			79.2
906892	50.4			79.5

Notes: (1) All ABI tests were conducted using a 0.020-inch diameter tungsten carbide indenter.
(2) Results of the large tensile specimens were provided by Columbia Gas Transmission Corporation.

APPENDIX A

REQUEST FOR THE DEVELOPMENT OF A NEW STANDARD

(Completion of this form is required for the formation of a task group to develop a new standard. Some of the information may not be necessary or appropriate as determined by your subcommittee.)

SUBCOMMITTEE DESIGNATION (e.g. A01.22): E8.08

Date: March 26, 1999

Task Group Chairman: Dr. John D. Landes

Phone: (423) 974-7670

E-Mail: John-Landes@utk.edu

Address (only if non-ASTM member) ASTM Fellow; E-8 Committee Member

Proposed Draft Document Title: Standard Test Methods for Automated Ball Indentation (ABI) Testing of Steel
Samples and Structures to Determine Stress-Strain Curves and Fracture Toughness

Proposed Scope: 1.1 These methods cover the determination of the true stress versus true-plastic-strain curves of steel samples and structural components using an automated ball indentation (ABI) technique. It can be used for any material with thickness greater than 0.25 mm (0.010 in). It requires a surface that is free of oxide and debris, and that has a minimum distance of 0.50 mm (0.02 in) between free edges. 1.2 The ABI test can be conducted at temperatures ranging from -196 to 427°C (-320 to 800°F). 1.3 The true-stress versus true-plastic-strain curves that are measured with an ABI test have been shown to correlate with the stress-strain curves measured with a standard or a miniature tensile test conducted according to ASTM standards E-8-96 and E-646-93. 1.4 Initiation Fracture Toughness values are determined from the ABI-measured stress-strain curves and the critical fracture stress of the steel test material.

Important keywords that are not included above: Fracture Toughness Master Curve

Projected target date for first subcommittee ballot: June 1999

Explain why the standard is needed and how it will be used (i.e. adopted by government agency, procurement, quality assurance) It will provide new localized and nondestructive test methods for base metal, welds, and heat-affected-zones of nuclear pressure vessels, ships, pipelines, and other critical components. These test methods could be adopted by U.S. government agencies (e.g. Nuclear Regulatory Commission, Department of Transportation, and the Department of Energy) and other societies such as the American Society for Mechanical Engineers (ASME).

List other ASTM Committees or key outside organizations that you feel should be informed of this activity:

E10, E28

ASME- Sec. 11

List Proposed Task Group Members along with their affiliations:

Name	Affiliation	Name	Affiliation
1. Dr. Randy K. Nanstad	Oak Ridge National Lab	6. Dr. Thak S. Byun	Oak Ridge National Lab
2. Mr. Fahmy M. Haggag	Advanced Technology Corp.	7. Dr. Nick Panayotou	Knolls Atomic Power Lab
3. Dr. Vik N. Shah	INEEL	8. Mr. Doug Baldery	Knolls Atomic Power Lab.
4. Dr. Milan Brumovsky	NRI, Czech Republic	9. Dr. John H. Smith	NIST
5. Dr. Shafik K. Iskander	Oak Ridge National Lab.	10. Dr. Walt G. Reuter	INEEL

Are you interested in utilizing the ASTM Web-Based Interactive Standards Development Forums? ☒ Yes No

APPROVED AT SUBCOMMITTEE MEETING ON: _____ (date) OR

APPROVED BY SUBCOMMITTEE CHAIRMAN ON: March 29, 1999 (date)

NOTE: If approved by the chairman between meetings, the task group will be approved by the subcommittee at their next meeting.

Subcommittee E08.08 Chairman: Dr. John D. Landes

Signature of Subcommittee Chairman: John D. Landes

By completing this form, the task group chairman acknowledges that all copyrights to this document, as a draft and as an approved ASTM standard, are the sole and exclusive property of ASTM, in accordance with the Intellectual Property policies of the Society.

Signature of Task Group Chairman: John D. Landes

→ Forward completed form to your Committee Staff Manager

E08.08 - ELASTIC - PLASTIC FRACTURE

J. LANDES

Subcommittee E08.08 Chairman John Landes stated that John Merkle recently retired and therefore stepped down as co-chairman of Task Group E08.08.03. John Landes made a motion that a new task group be formed on Automated Ball Identification Testing for Tensile Properties and Fracture Toughness of Steel Alloys. The motion was seconded by Ashok Saxena. Extensive discussion pursued. Such a task group was initially considered two years ago with emphasis on tensile testing. Committees E28 and E10 were pursued as more appropriate homes than E08 for the potential task group. Noting that the ABI testing results could be used in obtaining fracture toughness, E08 was recently reconsidered as the appropriate home. The scope of the proposed task group is as follows: "1.1 These methods cover the determination of the true stress versus true-plastic-strain curves of steel sample and structural components using an automated ball

004

indentation (ABI) technique. It can be used for any material with thickness greater than 0.25 mm (0.010 in). It requires a surface that is free of oxide and debris, and that has a minimum distance of 0.50 mm (0.02 in) between free edges. 1.2 The ABI test can be conducted at temperatures ranging from -196 to 427°C (-320 to 800°F). 1.3 The true-stress versus true-plastic-strain curves that are measured with an ABI test have been shown to correlate with the stress-strain curves measured with a standard or a miniature tensile test conducted according to ASTM standards E-8-96 and E-646-93. 1.4 Initiation Fracture Toughness values are determined from the ABI-measured stress-strain curves and the critical fracture stress of the steel test material." Concern was expressed that because ABI is a patented name, ASTM would appear to be giving a seal of approval to a patented item, resulting in a competitive business advantage. This issue was discussed in detail. After much further discussion, a vote was taken regarding the formation of the proposed task group in E08: 3 affirmative, 6 negative and 9 abstain. Hence, the motion did not carry. Subcommittee E08.08 Chairman John Landes was asked to send a letter to the requestor stating that the request was voted down.

005

Nondestructive and Localized Measurements of Flow and Fracture Properties of Pressure Vessel Steels Using Innovative Stress-Strain Microprobe Technology

Significance and Background Information

Identification and Significance of the Problem or Opportunity, and Technical Approach

Continued availability of nuclear power requires proper characterization and demonstration of materials performance for structures and components considered critical for safe operation of the nuclear facility. Characterization of integrity of nuclear reactor pressure vessel (RPV), through material property determination, is a critical safety assessment. One method for structural integrity evaluation of RPVs is to monitor mechanical property changes of the materials susceptible to radiation embrittlement. Currently, the changes in mechanical properties of RPV materials are monitored by periodic destructive testing of surveillance specimens (mostly Charpy impact and tensile specimens and fracture toughness if available). Thermal annealing and lifetime extension programs increase the need for additional surveillance specimens and demand multiple use of available test materials. Hence, the development of in-situ nondestructive evaluation technology to determine key mechanical properties (tensile, fracture toughness, and Charpy impact) of RPV materials is greatly needed. The newly developed in-situ technology will be used to **monitor** the structural integrity of RPVs subjected to radiation embrittlement as well as to verify and **quantify the recovery** of key mechanical properties following RPV thermal annealing.

Currently no apparatus exists that can characterize, nondestructively in-situ, a RPV by **measuring directly its localized key mechanical properties** (including base metal, welds, and their heat-affected zones). The objective of this project is to develop and evaluate an in-situ nondestructive technology to determine key mechanical properties of reactor pressure vessel (RPV) materials subjected to radiation embrittlement. This will be accomplished by using innovative hardware and software and newly developed test techniques, models, and analytical procedures. A novel portable/in-situ stress-strain microprobe (SSM) system was developed recently by Advanced Technology Corporation to test minimal material and determine several mechanical properties (e.g., yield strength, elastic modulus, flow properties, strain-hardening exponent, strength coefficient) of metallic structures including their welds and heat-affected zones. **This SSM system has received the 1996 R&D 100 Award in October 1996.** The SSM system utilizes a nondestructive automated ball indentation (ABI) technique to provide a localized direct stress-strain curve measurement. Although, thousands of successful SSM ABI tests have been performed on various materials, all tests were conducted at room temperature and at quasi-static strain rates. The capabilities of the SSM system will be greatly increased in this project to accomplish all the goals of this solicitation.

Anticipated Benefits/Commercial Applications

Phase I results proved the scientific and technical merits of the *in-situ* nondestructive SSM

technology to determine key mechanical properties of reactor pressure vessels (RPVs). Nuclear applications will include testing of RPVs before and after their postirradiation thermal annealing (to evaluate their embrittlement condition and to quantify recovery), vessel supports, core internals, etc. This will ensure safe operation and avoid premature decommissioning of power plants and other expensive structural components. Furthermore, the SSM technology will be used to monitor aging and assess the integrity of structural components over their design service life and in lifetime extension evaluations in many industries.

The newly developed in-situ nondestructive technology will allow effective use of surveillance specimens since hundreds of ABI tests can be conducted, at various strain rates and at different temperatures, on a single broken half of a Charpy impact specimen or a fracture toughness specimens. Furthermore, new analyses will be developed and evaluated to determine static and dynamic toughness properties from the ABI-measured stress-strain data and the use of critical fracture stress and critical fracture strain models. It should be emphasized here that the fracture toughness and transition temperature shifts, determined using the new analyses, should be more reliable than those from other nondestructive methods. This is true since these values are based on directly-measured ABI stress-strain test results which are highly accurate and reproducible at all test temperatures and strain rates.

The resultant product from all phases of this project will be an improved Stress-Strain Microprobe (SSM) system with increased capabilities for nondestructively testing minimal volume of materials and providing all key mechanical properties (tensile, Charpy impact, and fracture toughness) at various test temperatures as well as at various strain rates. This will significantly increase the market of the SSM system and provide numerous industries with a greatly needed nondestructive monitoring technology.

The innovative in-situ nondestructive technology will be used to assess the integrity of many structural components over their designed service life and in lifetime extension and weld repair evaluations. Commercial application will include nuclear pressure vessels, piping, vessel supports, core internals, turbine casings, rotors, aircraft, aerospace vehicles, submarines, ships, oil platforms, tank cars, bridges, etc. This will result in a national benefit by providing a reliable nondestructive/in-situ technology to avoid unexpected/catastrophic failures and to prevent premature decommissioning of critical/expensive engineering structures.

Accomplishment of Phase I Objectives

The Department of Energy (DOE) grant application was solicited for the development of: "an in-situ nondestructive evaluation technology which tests the minimal amount of material required to determine mechanical properties (Charpy impact, and tensile and fracture toughness) of RPV materials subjected to irradiation embrittlement". A copy of the Phase I application is attached to each copy of this Phase II application.

In Phase I of this Small Business Innovation Research (SBIR) project, new testing and analytical procedures were successfully developed and evaluated for: (1) ABI testing at high strain rates, (2) innovative ABI testing at various temperatures from -130°C/-250°F to 288°C/550°F where all

ABI-measured stress-strain curves showed excellent repeatability and agreement with those from miniature tensile specimens, (3) the indentation load versus depth data were used with appropriate critical fracture stress and critical fracture strain models to determine Charpy impact energies and fracture toughness as a function of test temperature, (4) *in-situ* ABI testing of a steel plate from a commercial RPV (after partial removal of its inner cladding) using magnetic mounts, and (5) numerous ABI tests were also conducted on archive commercial RPV steels at ATC as well as on irradiated and irradiated+annealed broken Charpy specimens (A533-B plate and 73W weld) in the hot cell at Oak Ridge National Laboratory. The ABI test results on the irradiated and irradiated+annealed specimens successfully quantified the degree of embrittlement as well as the percentage of toughness recovery following thermal annealing. All tasks of this project were highly successful and numerous test results and analyses were generated and presented in the Phase I Report No. DOE/ER/82115--1, March 1997 (a copy of this report is attached to each copy of this Phase II application). The technical feasibility of all tasks was demonstrated to be 100% successful and the continued success and completion in Phase II will benefit the nuclear industry as well as many other industries (e.g., fossil power plants, oil, chemical, transportation, bridges, ships, aerospace, etc.). The success of Phase I is graphically illustrated in Figures 1 and 2.

The Phase II Project

Technical Objectives

In Phase I, successful demonstration of ABI testing at low and high temperatures was accomplished. These results were used to calculate a new ABI energy parameter called Indentation Energy to Fracture (IEF) for one heat of A533-B (EPRI Heat 1bK). In Phase II, this success should be extended to cover several other reactor pressure vessel materials and their welds. Effects of strain rate at low test temperatures will be further evaluated for all these commercial RPV steels. Further developments of generic critical fracture stress and critical fracture strain values for these RPV steels will be used to generate a large database for a new valuable American Society of Testing and Materials (ASTM) standard. The new ASTM standard will cover ABI procedures for ABI testing at various temperatures and strain rates and for ABI data analyses (using critical fracture stress and critical fracture strain models and establishing new reliable/accurate indices for the ductile-to brittle transition temperature) to determine impact and static and dynamic fracture toughness values from ABI test results. The standardization process of the ABI/IEF techniques will allow the nuclear utilities and other industries to use the nondestructive SSM technology in structural integrity evaluation of nuclear pressure vessels and other complex/expensive structural components. The new ASTM standard will: (1) allow the regulatory authorities (such as the Nuclear Regulatory Commission, Federal Aviation Administration, etc.) to approve test results and lifetime extensions in a timely manner, (2) increase the market of the new/improved SSM technology, and (3) provide national economic benefits in integrity evaluation of aging structures and in lifetime extensions. Finally, a miniaturized testing head of the Stress-Strain Microprobe (SSM) system will be developed and manufactured for use with robots inside a nuclear pressure vessel. Also, techniques for remote/robotic extraction of small cylinders (e.g. 0.5 in diameter by 0.5 in high) from the inside surface of the vessel will be investigated and evaluated.