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Small Specimen Test Techniques

William R. Corwin
Stan T. Rosinski, and
Eric van Walle, editors

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Foreword

This publication, *Small Specimen Test Techniques,* contains papers presented at the symposium of the same name held in New Orleans, Louisiana, on 13-14 January 1997. The symposium was sponsored by ASTM Committee E-10 on Behavior and Use of Nuclear Structural Materials, in cooperation with the European Network on Aging Materials Evaluation and Studies (AMES) and the TC5 Technical Subcommittee on Dynamic Testing at Intermediate Strain Rates of the European Structural Integrity Society (ESIS). The symposium chairman was William R. Corwin, Oak Ridge National Laboratory.

Overview

The Symposium on Small Specimen Test Techniques demonstrated the overall interest in ASTM Subcommittee E10.02 activities in radiation embrittlement for nuclear reactors. The symposium, which was held in New Orleans, Louisiana, on January 13-14, 1997, was organized to bring together, in a single meeting, the interests and capabilities of the scientific testing community and the needs of the commerical light-water-cooled power-reactor industry for improved methods to characterize component integrity. Technical interest in this topic was certainly demonstrated through the presentation of over 30 papers and 13 posters by experts representing 15 countries. A wide range of subjects was discussed during the symposium, concentrating on: (1) the use of unique small and miniature specimens, as well as nondestructive, nonintrusive, and in-situ test techniques for measuring mechanical and fracture properties; (2) the application of those test methods to assess irradiation-induced embrittlement; and (3) acutal examples of the use of these test techniques for determining material integrity and to evaluate potential component life extension. The strong interest in the topics results from the desire to improve miniature specimen testing technology and the recognition of the potential benefits in commerical nuclear power plant operation through improved integrity assessment.

This symposium was the third in a series of ASTM symposia on small specimen testing technology organized by ASTM Subcommittee E10.02 on Behavior and Use of Nuclear Structural Materials. The first symposium was held in Albuquerque, New Mexico in September 1983 and is summarized in *The Use of Small-Scale Specimens for Testing Irradiated Materials, ASTM STP 888*. The primary driving force was the need of the fusion reactor materials research community to assess effects of the very high levels of irradiation expected in the first wall of a fusion reactor. The limited volume of materials which can be irradiated in test reactors to high levels of embrittlement results in the need for small specimen technology. The second symposium, held in New Orleans, Louisiana in January 1992, explicitly included the needs of and applications to commercial nuclear power reactors, with direct application of small specimen test techniques to reactor pressure vessel annealing and life extension and the continued development of testing technology within the fusion reactor research community. The symposium is documented in *Small Specimen Test Techniques Applied to Nuclear Reactor Vessel Thermal Annealing and Plant Life Extension, ASTM STP 1204*. As a result of this keen interest in and obvious applications for the diversity of small specimen testing techniques, ASTM Subcommittee E 10.02 initiated an international testing exercise in 1994 to obtain a cross-comparison of material property measurements obtained from various types of subsize-specimen testing techniques. Sixteen organizations representing 10 countries initially participated. Objectives were: (1) to benchmark various subsize specimen testing techniques by comparing testing results with established material properties for the material tested, and (2) to provide information to participants to improve the correlation of subsize specimen testing results with material properties determined through standard ASTM methods. A variety of miniature testing techniques were utilized by the participants during this exercise on selected pressure vessel steel.

The recent symposium focused on the experimental, analytical, and computational aspects of small (and miniature) specimen test techniques, as well as on application to component integrity assessment. In addition, results of the cross-comparison exercise were presented.

The collection of papers within this special technical publication will provide a resource for both researchers and end users in this field. The symposium was organized into sessions covering the following small specimen test techniques: (1) miniature impact, (2) fracture toughness, (3) precracked round bars, (4) reconstitution, and (5) tensile and punch. In addition, a poster session presented additional information in these areas.

Two separate presentations reviewed international testing programs to compare miniature specimen testing results and investigate the impact of testing variables on the prediction of component behavior. These activities included various types of miniature test techniques and involved different materials and test conditions in order to provide a better understanding of small specimen behavior when compared to results obtained from standard ASTM specimen configurations.

Numerous novel and improved methods for obtaining and applying data from small specimens were described in the various sessions on testing techniques. Improvements in both correlation methods with standard-size specimens and test techniques were described by the authors of papers dealing with impact testing of subsize Charpy V-notch type specimens. The impact of critical parameters in the reconstitution process, including welding technique, insert size, specimen orientation, and even striker tup geometry, were addressed. The potential for utilizing reconstituted Charpy samples on component integrity was discussed in view of future, enhanced surveillance methodologies for plant life management. The portion of the symposium related to fracture toughness provided new insight into both the potential limitations and possible ways to correct and utilize measurements made using very small fracture toughness specimens. Innovative experimental approaches to obtaining fracture toughness data with small amounts of test material included techniques for using very small-sized compact tension specimens as well as those describing new specimen designs. Of particular interest during this symposium was the application of the Master Curve method being standarized under ASTM Committee E 08 on Fatigue and Fracture for the determination of reference temperature in the transition region for ferritic materials. This technique may significantly enhance the assessment of reactor pressure vessel integrity, and several papers demonstrating application of the Master Curve approach were presented. A new addition to this symposium series was the feasibility of using small fatigue-precracked round bars to measure fracture toughness of pressure vessel materials and to refine and validate experiment and analysis procedures. Improvements and innovations in several of the punch and disk testing techniques, discussed in previous meetings in this series, were also reported.

As the level of overall accuracy and the degree of reproducibility of data generated by the small specimen test techniques improves, the techniques can be more fully evaluated among themselves and against other standardized tests. This analysis will ultimately improve the confidence regarding application of these techniques for the evaluation of reactor pressure vessels or any other structures. It is apparent from the papers presented in this symposium, and the technical information presented throughout this symposium series, that these testing techniques continue to mature. They provide a means of obtaining material property information for situations where extraction of samples from vessels or other structural components is not desirable or possible, or when the amount of available materials is too limited to utilize conventional, standardized techniques.

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Miniature Impact Techniques

Stan T. Rosinski[1] and William R. Corwin[2]

ASTM CROSS-COMPARISON EXERCISE ON DETERMINATION OF MATERIAL PROPERTIES THROUGH MINIATURE SAMPLE TESTING

REFERENCE: Rosinski, S. T. and Corwin, W. R., "**ASTM Cross-Comparison Exercise on Determination of Material Properties Through Miniature Sample Testing,**" *Small Specimen Test Techniques, ASTM STP 1329,* W. R. Corwin, S. T. Rosinski, and E. van Walle, Eds., American Society for Testing and Materials, 1998.

ABSTRACT: An international testing exercise was conducted under ASTM Committee E10.02 to obtain a cross-comparison of material properties obtained from various types of subsize-specimen testing techniques. Sixteen organizations representing 10 countries initially participated in the exercise. Objectives were to (1) benchmark various subsize specimen testing techniques by comparing testing results with established material properties for the material tested, and (2) provide information to participants to improve the correlation of subsize specimen testing results with material properties determined through standard ASTM methods. The testing material for the cross-comparison exercise was the ASTM A533 grade B class 1 plate designated as HSST plate 03 provided by Oak Ridge National Laboratory. A variety of miniature testing techniques were utilized by the participants during this exercise. A summary of the exercise and a general comparison of the results obtained are provided. Detailed discussions on individual participant testing programs will be presented by each organization later in subsequent papers.

KEYWORDS: Mechanical properties, A533B, miniature specimen technology

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In 1992 ASTM Subcommittee E10.02 on Behavior and Use of Nuclear Structural Materials sponsored the *Symposium on Small Specimen Test Techniques Applied to Nuclear Reactor Vessel Thermal Annealing and Plant Life Extension* [1]. The symposium was organized to bring together, in a single meeting, both the diverse interests and capabilities of the scientific testing community and the needs of the commercial light-water-cooled power-reactor industry. Topics discussed during this symposium included: (1) unique small and miniature specimens, as well as nondestructive, nonintrusive, and in-situ test techniques for measuring mechanical and fracture properties; (2) application of those techniques to assess embrittlement due to irradiation and high-temperature exposure; and (3) examples of the use of these techniques to verify results of thermal annealing of vessels and to evaluate potential reactor-vessel life extension.

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As a result of the interest in small-specimen testing for application to various aspects of the commercial power reactor industry, it was agreed to initiate a follow-on activity with the explicit purpose of evaluating the type of materials property data generated by these various methods. ASTM Subcommittee E10.02 initiated this activity with the primary goal to compare results from numerous nonstandard techniques with results from standardized tests and with each other. The information from such an exercise would support future standards development in the area of miniature specimen testing techniques.

ORGANIZATION

The exercise was sponsored by ASTM Subcommittee E10.02 and coordinated by Stan T. Rosinski, Electric Power Research Institute, and William R. Corwin, Oak Ridge National Laboratory (ORNL). An invitation to participate in this exercise was forwarded in March 1994 to those attendees of the 1992 symposium discussed above and other individuals. The stated objective was to obtain a cross-comparison of material properties obtained from various types of subsize-specimen testing techniques. Specifically, the exercise was designed to: (1) benchmark various subsize specimen testing techniques by comparing testing results with established material properties for the material tested, and (2) provide information to participants to improve the correlation of subsize specimen testing results with material properties determined through standard ASTM methods.

Test material was distributed to all participants in February 1995 and the cross-comparison exercise initiated. Duration of the exercise was anticipated to be approximately 2 years with results to be presented in a future ASTM symposium (this proceedings).

The test material investigated during this exercise was provided by ORNL to each participating organization. No attempts were made to restrict the types of miniature specimen testing techniques utilized. However, in order to maintain an effective comparison base, proposed testing techniques and material properties to be measured were coordinated through the exercise coordinators. Testing techniques utilized by the organizations participating in this exercise included miniature tensile and Charpy V-notch (CVN), small punch, automated ball indentation, miniature fracture toughness (miniature disk compact tension, notched round bar) and other techniques. In general, properties measured included tensile, impact, hardness (microhardness), and fracture toughness. The initial participating organizations and proposed testing techniques are shown in Table 1.

To ensure that a sufficient level of comparable data was obtained from a majority of the participants during this exercise, minimum fabrication and testing recommendations were established, where practical. Inasmuch as the principal goal of this exercise was to obtain a means of comparing fracture properties obtained with various types of small, nonstandard test specimens, it was requested that, as a first priority, all participants obtain test results that can be used to determine the temperature range in which the transition from ductile to brittle fracture occurs in the test material. How the transition temperature is defined and determined was left to the individual researcher. Determinations of transition temperature are traditionally made using various levels of energy, lateral expansion, or fracture appearance of Charpy impact specimens, as well as at different levels of fracture toughness. Other indices are possible.

Table 1--Participating Organizations and Proposed Testing Techniques

		Proposed Testing Technique[1]							
Organization	Country	ABI	Bulge	CVN	DC(T)	Fatigue	Fracture	Punch	Tensile
SCK•CEN	Belgium			X					
University of Toronto	Canada							X	
Atomic Energy Canada Limited	Canada							X	
VTT Research	Finland			X					
Fraunhofer Institut fur Werkstoffmechanik	Germany			X					X
CISE	Italy			X	X			X	X
Kyoto University	Japan			X	X				X
Japan Atomic Energy Research Institute	Japan		X	X		X	X		
Kurchatov Institute	Russia			X				X	X
Tecnatom	Spain			X					X
Institute of Nuclear Energy Research	Taiwan			X					X
Advanced Technology	U.S.	X							
MPM Research	U.S.			X			X		
Johns Hopkins University	U.S.								X
Failure Analysis Associates	U.S.							X	
Battelle Pacific Northwest Laboratory	U.S.			X					

[1]Proposed Testing Techniques:
ABI = Automated Ball Indentation
Bulge = Miniature Bulge
CVN = Charpy V-notch; includes full-size and miniature, notched-only and precracked
DC(T) = Miniature Disk Compact Tension
Fatigue = Miniature Fatigue
Fracture = Miniature Fracture Toughness
Punch = Small Punch Test
Tensile = Full-size and miniature tensile, includes notched and smooth samples

As a second priority, it was requested that estimates be made of ductile fracture resistance, again with the goal of intercomparison with each other, as well as with more traditional measures such as Charpy upper-shelf energy, J_{Ic}, tearing modulus, etc. It was further requested that fracture properties be measured in the LT orientation, as a minimum. This would allow for comparison with available mechanical properties for the test material established through standard ASTM testing techniques.

Baseline material property data for the material investigated in this exercise was not provided to the participants prior to their individual testing activities. Participants were requested to perform the testing and report results to ASTM coordinators for comparison with established properties for the material investigated. This approach was followed to ensure an unbiased application of correlation methodologies to predict standard ASTM material properties obtained through miniature sample testing. This paper provides the baseline properties of the material investigated and performs a preliminary comparison of the testing results obtained to date. The information presented will provide a relative assessment of the ability of various miniature specimen testing techniques to reliably predict mechanical properties otherwise measured via standard ASTM techniques. Additional details of individual participating organizations' test programs are included in this proceedings.

TEST MATERIAL

The material investigated in the cross-comparison exercise was the ASTM A533 grade B class 1 plate designated as Plate 03 in the ORNL Heavy Section Steel Technology (HSST) Program. The HSST plate measured 3.05 m (10 ft.) wide by 6.1 m (20 ft.) long by 305 mm (12 in.) thick. Its fabrication history has been documented elsewhere [2]. Numerous through-thickness samples have been removed from this plate for subsequent material characterization studies, including several sections provided to the International Atomic Energy Agency for use as a correlation monitor material [3]. For the samples provided during this exercise, the outer 2.54 cm (1 in.) of material from both surfaces of the plate were removed to eliminate the impact of edge effects on mechanical properties.

A thorough characterization of the HSST Plate 03 was performed at ORNL and documented [4]. The through-thickness variation of the chemical composition for Plate 03 is given in Table 2.

Table 2 -- Chemical composition of HSST Plate 03

Through-Wall Fraction	Composition (weight %)									
	C	Mn	P	S	Si	Ni	Mo	H	N	O
0	0.26	1.33	0.011	0.016	0.24	0.65	0.51	0.0003	0.0072	0.0012
1/4	0.26	1.36	0.011	0.015	0.26	0.62	0.52	0.0004	0.0070	0.0009
1/2	0.25	1.27	0.010	0.020	0.28	0.70	0.49	0.0003	0.0069	0.0025
3/4	0.26	1.37	0.010	0.019	0.25	0.62	0.43	0.0004	0.0070	0.0016
1	0.25	1.41	0.010	0.016	0.28	0.61	0.50	0.0003	0.0075	0.0012

Tensile and Charpy V-notch baseline tests performed on Plate 03 are described in detail elsewhere [4]. Tensile tests were conducted in accordance with the requirement of ASTM Standard Test Method and Definitions for Mechanical Testing of Steel Products (A 370). Charpy V-notch impact tests were conducted in accordance with ASTM Standard Test Methods for Notched Bar Impact Testing of Metallic Materials (E 23). A summary of the room temperature through-thickness variations in tensile properties is shown in Figure 1. The through-thickness variation of Charpy impact properties in the ASTM L-T orientation is shown in Figure 2. In 1995, an ASTM Subcommittee E10.02 round robin exercise on Charpy V-notch sample reconstitution was conducted to investigate the impact of ASTM and ISO-designed tups and reconstitution insert size on the resulting impact properties. As part of this exercise a large number of Charpy specimens from HSST Plate 03 were tested with both ASTM and ISO tups. Preliminary results of these tests are shown in Table 3.

Table 3--Average Charpy properties of HSST Plate 03 at two test temperatures with ASTM and ISO tups

	Temperature		Number of	Energy		Lateral Expansion		Shear
Tup	°C	°F	Specimens	J	ft-lb	mm	mils	%
ASTM	-12.2	10	46	40.93	30.19	0.649	0.026	10
ISO	-12.2	10	46	38.05	28.06	0.630	0.025	10
ASTM	93.3	200	23	163.6	121.7	1.257	0.049	100
ISO	93.3	200	23	151.4	111.7	2.269	0.089	100

Fracture toughness characterization of the HSST plate 03 was performed in accordance with ASTM Standard Test Method for Plane-Strain Fracture Toughness of Metallic Materials (E 399) with compact specimen sizes ranging from 1T to 6T. Test results have been previously discussed [5,6,7]. The variation of K_{IC} values through the thickness of Plate 03 is shown in Figure 3. The temperature dependence of fracture toughness is shown in Figure 4.

RESULTS

Upon completion of testing activities each participating organization was requested to provide results to ASTM. To date, approximately one-half of the participating organizations have either provided their test results to ASTM (these will be briefly discussed below) and will be discussed in further detail later in this proceedings or are being presented in this proceedings for the first time.

Preliminary results presented are organized according to the mechanical property measured. Although various methods may be used to determine a specific property, e.g., small punch and miniature fracture toughness specimens to measure material fracture toughness, emphasis is placed on measuring the specific mechanical property utilizing any of various testing techniques, and not focusing on any particular technique itself.

As stated above, one of the primary objectives of this exercise was to provide information to participants to improve the correlation of subsize specimen testing results with material properties determined through standard ASTM methods. Given the nature of this objective participating organizations will not be specifically identified when comparing results to baseline properties for the HSST Plate 03.

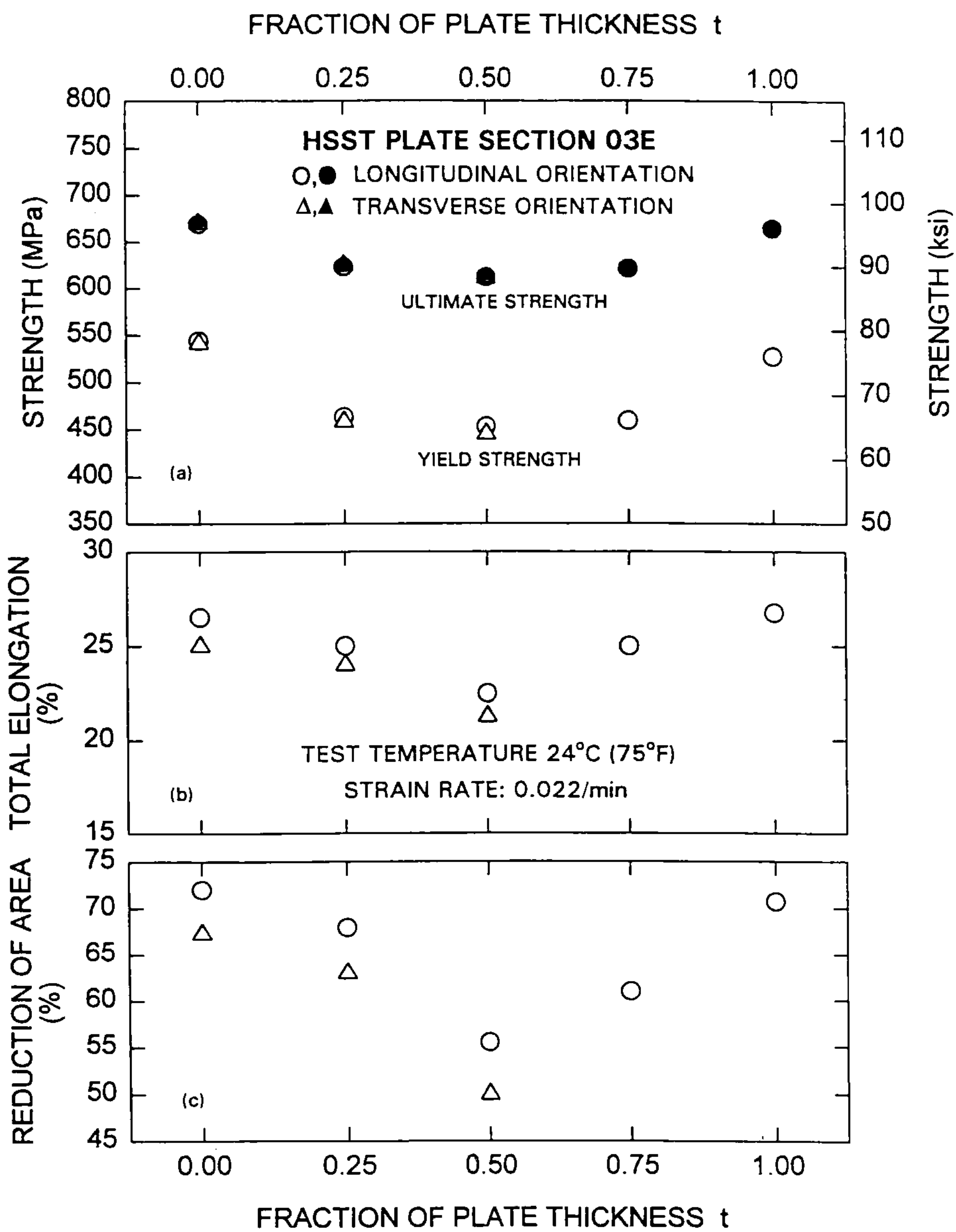

Figure 1 --Through-thickness variations of tensile properties of HSST plate 03.