LABORATORY EVALUATION OF COATINGS AND OVERLAYS FOR PREVENTION OF STRESS CORROSION CRACKING OF CONTINUOUS DIGESTERS

to

THE INSTITUTE OF PAPER CHEMISTRY

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PROPOSED RESEARCH PROGRAM

Proposal/Agreement No. 772-K-9097R

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BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

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INTRODUCTION

This proposal is a contribution to the TAPPI/IPC research program on stress corrosion cracking of continuous digesters. It is aimed at a comprehensive study of the effectiveness of weld overlays and coatings in mitigating stress corrosion cracking of steel in the near-weld regions in continuous digesters. The proposal is based on the input from the Steering Committee of the TAPPI/IPC research program at the December 1, 1982 meeting at Battelle.

Weld overlays and coatings are two of the most promising techniques to prevent stress corrosion cracking of continuous digesters. Both techniques can be used for new construction or as a part of the repair procedures for damaged digesters. In general, the corrosion resistance of the overlay and coating alloys themselves is judged to be sufficient for continuous digester service. The effectiveness of the overlays and coatings to prevent stress corrosion cracking of the carbon steel must be measured. Of particular interest is the behavior of the steel plate and welds adjacent to the protective layer.

OBJECTIVE

The effectiveness of weld overlays and coatings used at present, or those suggested by vendors, to cover welds in continuous digesters is as yet not clear. Weld overlays result in creation of new heat-affected zones in digester plate and, further, they give rise to additional residual stresses because of differences in the coefficients of expansion of the matrix and the overlay. Plasma- and flame-sprayed coatings are not expected to have these disadvantages, but the presence of holidays in unsealed coatings may actually accelerate attack on the substrate. In the case of sealed coatings, the service life of the sealing and, hence, the duration of protection afforded by it is uncertain. Further, undercutting of the coatings because of galvanic attack at the edges is also possible. As both overlays and coatings use stainless steels or nickel-base alloys, they are expected to have corrosion potentials that are noble to (more positive than) the corrosion potential of the base steel. It is, therefore, likely that substrate areas adjacent to the overlays or coatings may attain potentials in the stress corrosion susceptibility region because of galvanic effects. The present proposal is aimed at measuring the likelihood and severity of the above effects.

A laboratory program will be conducted in simulated digester environments to evaluate the effectiveness of the overlays and coatings to prevent stress corrosion cracking. The results will support the selection of effective overlays and coatings to be used in construction and repair of continuous digesters.

APPROACH

A number of techniques will be used in the laboratory to determine the performance of overlays and coatings under simulated digester conditions. Stressed and unstressed base metal specimens with overlays and coatings will be subjected to a long (four months) autoclave exposure to a caustic-sulfide environment at the temperature and pressure seen in continuous digesters to determine galvanic damage and stress corrosion cracking susceptibility with various overlays and coatings. The environment itself will be selected on the basis of an IPC review and preliminary experimentation.

One difficulty in determining the suitability of candidate digester materials is the lack of a standard environment for testing, such as those used in the ASTM tests for sensitized stainless steel or the NACE test for oil-country materials. For this reason, prior to the autoclave testing described above, caustic-sulfide environments that will raise the corrosion potential of digester plate material (A516 grade 70 steel) into the critical active-passive region of stress corrosion cracking will be determined using electrochemical techniques. Stress-corrosion tests on digester plate material will be performed in these environments to select one environment for the autoclave exposure.

The chemical composition of the environment in the autoclave exposure described above will be different from that of actual digester environments. To build a bridge between the data obtained in the autoclave test and the cracking observed in digesters, a series of stress corrosion cracking tests will be carried out on specimens of A516 grade 70 plate material with overlays and coatings in a severe digester environment selected on the basis of the IPC review. The technique to be used will be slow cyclic-load tests with stresses approaching the yield stress of the base material. These tests will be carried out at three applied potentials: at the free corrosion potential of the base metal in digesters, at a potential in the critical cracking region, and at a potential in the anodic passive region. The data obtained will be compared with those obtained from the autoclave tests. In addition, data from these tests will provide information on the effect of applying anodic protection to the base plate on the corrosion and stress corrosion of overlays and coatings.

The final part of the proposed program will investigate quantitatively the galvanic corrosion between base plate and overlays and coatings. Specimens of base plate material with overlays and coatings will be exposed to three digester environments and short-circuited with a zero-resistance ammeter to determine the galvanic corrosion currents. In addition, their corrosion potentials will be monitored and compared with IPC data to ensure that the test conditions simulate actual digester conditions.

The results for these corrosion studies with weld overlays and coatings will provide data for comparison of the effectiveness of the various protection techniques in mitigating stress corrosion cracking in continuous digesters.

RESEARCH PLAN

The research plan is divided into six tasks: Task I will be selection of an environment for autoclave exposure, Task II will be autoclave exposure of stressed and unstressed specimens with overlays and coatings, Task III will be cyclic-load tests of specimens with and without overlays and coatings under applied potential, Task IV will be galvanic effects evaluation, Task V will be compilation and evaluation of all results, and finally, Task VI will be meetings and reports. The experimental details and activities schedule are given below for each task.

Task I. Selection of a Caustic Environment for Autoclave Exposure

The objective of this task will be to select an environment for the autoclave exposure. The starting point for this task will be the IPC survey, which will be critically reviewed for selection of two of the more aggressive digester environments. The survey and other literature on caustic cracking of carbon steels will be used to determine additives to these environments that will raise the free corrosion potential of A516 grade 70 steel into the critical cracking region. Fast and slow potentiodynamic polarization scans will then be obtained for A516 grade 70 steel in these environments and in a number of modified environments. These results will be used to select two or three candidate environments. Slow strain rate tests of A516 grade 70 material will then be carried out in these environments at the free corrosion potential to determine if stress corrosion cracking does occur under these conditions. Specimens that showed cracking will be metallographically examined, and an environment that leads to cracking similar to the cracking observed in digesters will be selected for the autoclave exposures. It is realized that considerable experimentation might be necessary before a suitable environment can be selected. The activity schedule for this task is given below.

Schedule

Task I - Selection of a Caustic Environment

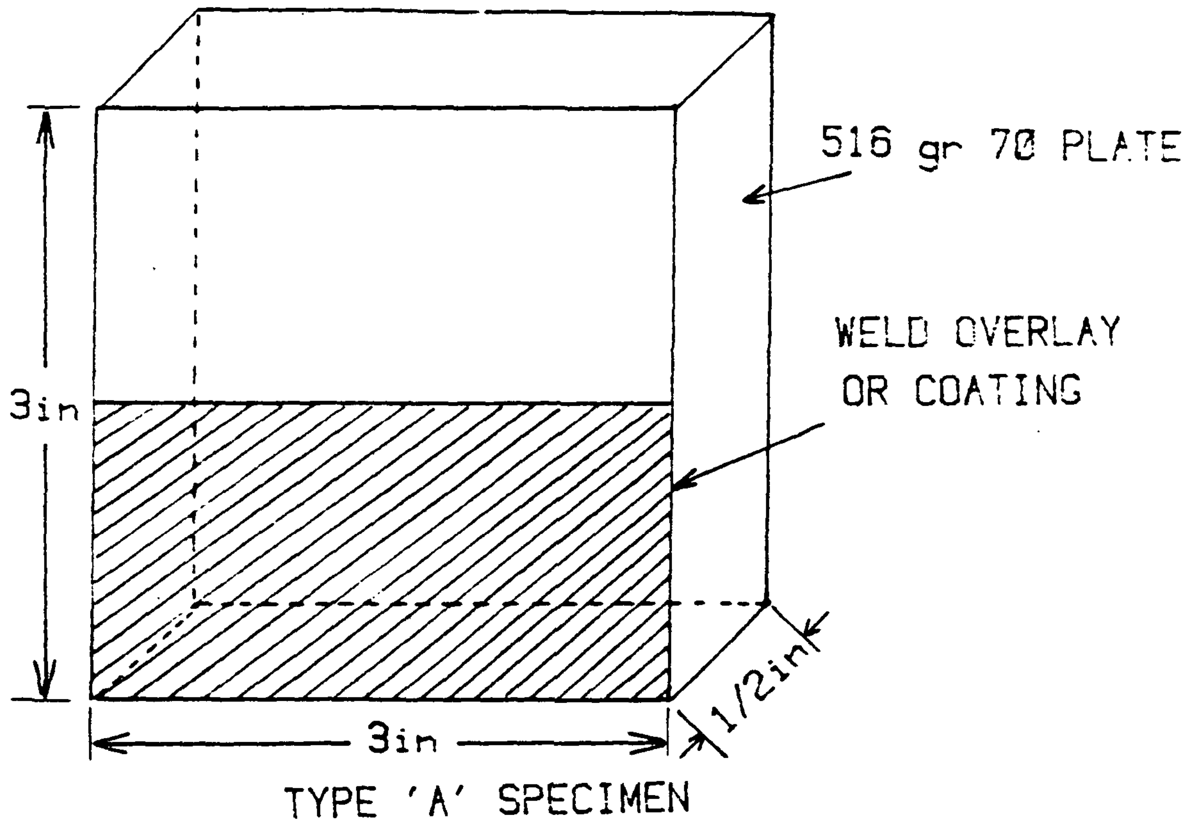
Work Schedule (Completed by Week Indicated)	Activity
4 weeks	1. Review of IPC survey to select two environments and literature survey to determine modifications
10 weeks	2. Polarization scans in 2 IPC environments and 8 modifications (maximum of 10 tests)
20 weeks	3. Slow strain rate tests of A516gr.70 steel in six modified environments
22 weeks	4. Metallographic examination of cracked specimens
24 weeks	5. Assessment of results and selection of one environment for autoclave test

Task II. Autoclave Exposure

The objective of this task will be to determine, in a severe simulated digester environment, the worst-case corrosion and stress corrosion damage of various stressed and unstressed specimens of A516 grade 70 steel containing overlays and coatings. The environment will be selected on the basis of the results from Task I. The temperature and pressure will be those typical of continuous digesters (∿ 300 F and 150 psi). The unstressed specimens will be 3 in. x 3 in. in size and made from 1/2-in. plate of A516 grade 70 steel. These specimens will be of two types. The first type (called Type A) will carry overlays and coatings over one-half of one surface without any underlying weld. The second type (called Type B) will carry overlays and coatings over one-half of one surface with an E7018 weld at right angles to the coating or overlay. These specimen configurations are shown in Figure 1. Type A specimen will represent an overlaid or coated horizontal weld and Type B will represent conditions where a horizontal overlay or coating meets a bare vertical weld. Two types of overlays (309 and 182), two types of plasma-sprayed coatings (both sealed and nonsealed), and one flame-sprayed coating will be tested.

In addition to these specimens, stressed specimens also will be exposed. These will be thin strips of A516 grade 70 steel 1/2-in. wide x 1/8-in. thick x 4 to 6-in. long with overlay or coating covering half of one wide surface. The strips will be mounted on a carbon steel pipe of a diameter such that when stretched over the pipe, the curvature in the strips will result in stresses close to the yield stress over the entire length of the strips. The same types of coatings and overlays as used in Type A specimens will be tested. The specimens will provide information on the effect of working stresses on corrosion and stress corrosion damage at and near overlays and coatings under a severe condition.

Four, 1 month exposures will comprise the total autoclave exposure of 4 months. Duplicate specimens will be used for each type of specimen, and these will be insepcted for damage every month. In addition to the specimens described above, a specimen of A516 grade 70 material will be exposed and its corrosion potential will be monitored regularly with a suitable reference electrode to check that the environment has not changed. The solution will be refreshed every two weeks or if feasible, whenever the corrosion potential changes by more than ± 20 mv, whichever is sooner. At the end of the 4-month exposure, all specimens will be examined visually and selected specimens will be examined metallographically and under an SEM for evaluation of corrosion and stress corrosion damage. The environment composition will be analyzed periodically.



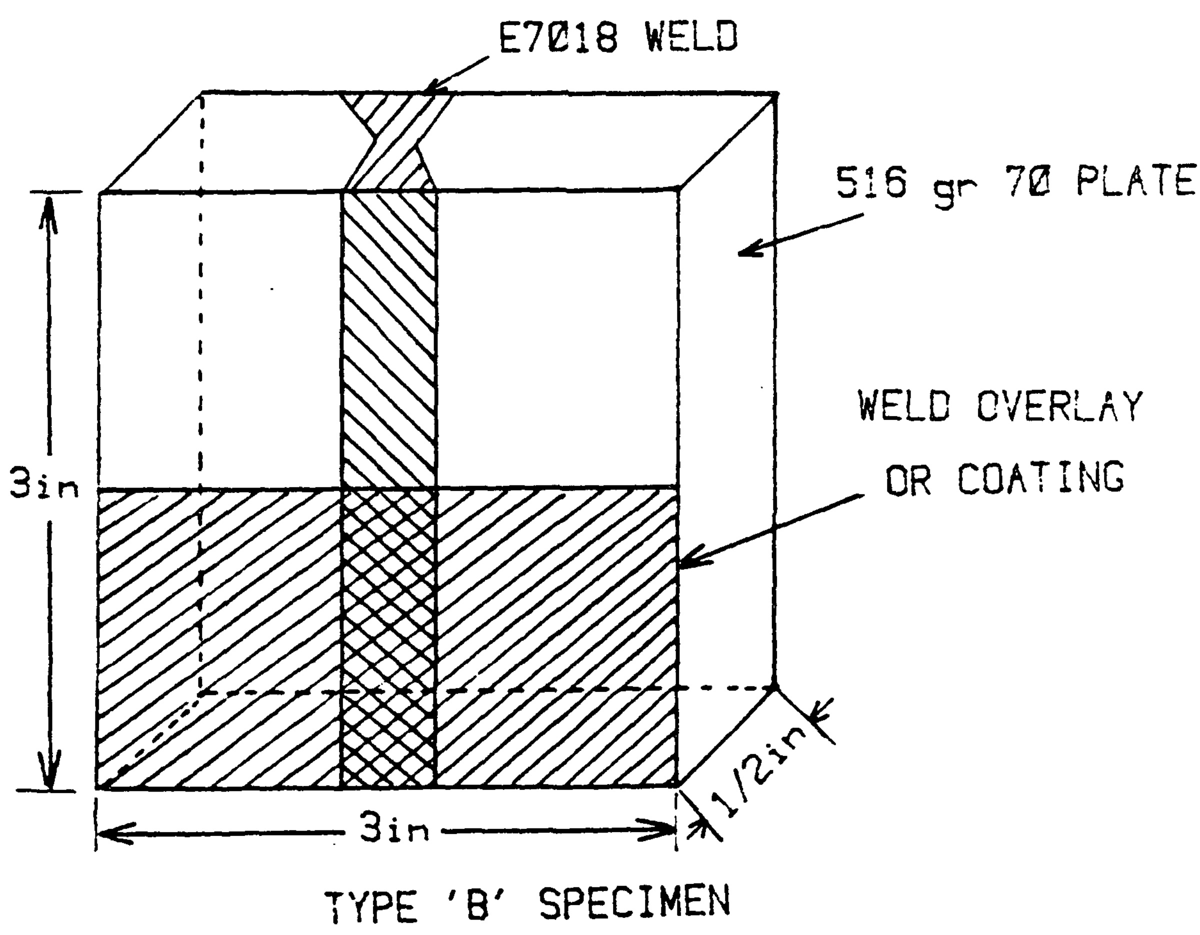


FIGURE 1. CONFIGURATIONS OF SPECIMENS A AND B

Schedule

Task II - Autoclave Exposure

Work Schedule (Completed by Week Indicated)	Activity
24 weeks	1. Completion of Task I to select autoclave environment
30 weeks	2. Preparation of specimens and autoclave setup
36 weeks	3. First autoclave exposure of 1 month and examination of specimens
42 weeks	4. Second 1-month exposure and examination
48 weeks	5. Third 1-month exposure and examination
54 weeks	6. Fourth and final 1-month exposure
60 weeks	7. Specimen examination and data assessment

Task III. Cyclic Load Tests

The objective of this task will be to investigate the stress-corrosion cracking behavior of digester plate material in regions adjacent to an overlay or coating and that of the overlays and coatings in a severe digester environment at stresses close to the yield stress of the plate material. A severe environment will be selected on the basis of the IPC review. The tests will be carried out at three applied potentials, at the corrosion potential of A516 grade 70 steel in the environment, at a potential in the critical region for A516 grade 70 steel, and at a potential in the anodic protection region for A516 grade 70 steel. Each test will last two weeks. The load will be cycled along a saw-tooth wave form at stresses ranging between 50 and 100 percent of the yield stress of the base metal (A516 grade 70). A slow frequency will be selected to give a strain rate on the order of 10^{-7} sec^{-1} on rising load.

The specimen will have a rectangular cross-section in the gauge section and will carry a coating or an overlay over part of the gauge section. At the end of a test, the specimens will be metallographically examined to determine the presence and location of cracking. A total of twenty (20) tests is planned with two overlays, two plasma sprays, and one flame spray as in the autoclave tests. One of the plasma sprays will be tested in both sealed and unsealed conditions, the other only in the sealed condition. In the initial tests, the environment composition will be checked periodically using conventional wet analysis methods. Based on these data, the solutions will be refreshed as necessary.

Schedule

Task III - Cyclic Load Tests

Work Schedule (Completed by Week Indicated)	Activity
4 weeks	1. Selection of an environment based on IPC survey
10 weeks	2. Assembly of experimental setup and preparation of specimens
26 weeks	3. First set of four tests
32 weeks	4. Second set of four tests
38 weeks	5. Third set of four tests
44 weeks	6. Fourth set of four tests
50 weeks	7. Fifth set of four tests
55 weeks	8. Specimen examination and data assessment

Task IV. Galvanic Effects Evaluation

The objective of this task is to determine on a quantitative basis the galvanic effects in the carbon steel, of the immediately adjacent coatings and overlays. Equal-area specimens of A516 grade 70 with and without overlays and coatings will be exposed to two digester environments selected from the IPC review. The corrosion potential of all materials will be recorded over a period of time. When the potentials became steady (probably in < 2 weeks), the specimens will be shorted through a zero-resistance ammeter, and the current and the potentials of the galvanic couples will be recorded over a period of time. The information obtained will help evaluate the extent of galvanic damage that can occur. It will also make it possible to compare various weld overlays and coatings for the galvanic damage that they may cause on the base metal. All overlays and sealed coatings studied in the autoclave exposure will be investigated. Post-test examination of all specimens will be performed to check the reliability of galvanic current measurements.

Schedule

Task IV - Galvanic Effects Evaluation

Work Schedule (Completed by Week Indicated)	Activity
4 weeks	1. Selection of two environments based on IPC review
10 weeks	2. Preparation of specimens, autoclave setup, and electrodes
15 weeks	3. Measurement of galvanic effects in first environment
20 weeks	4. Measurement of galvanic effects in second environment
24 weeks	5. Data assessment

Task V. Data Compilation and Evaluation

In this task, the data obtained in the three tasks will be critically assessed. A detailed report will be put together describing experiments, data obtained, assessment, and recommendations. The results should provide the necessary data for the selection of effective construction and repair procedures to control stress corrosion cracking of continuous digesters.

Schedule

Task V - Data Compilation and Evaluation

Work Schedule (Completed by Week Indicated)	Activity
60 weeks	1. Completion of all experimental tasks
64 weeks	2. Compilation and review of data
68 weeks	3. Preparation of report

Task VI. Meetings and Final Report

This task covers travel costs of the project leader to three meetings of the sponsors and the steering committee for discussions and progress reports. It also covers the cost of 150 copies of two semi-annual reports and a final report on the project.

Materials Requirement

It is assumed that all bulk materials will be supplied by the Sponsor at no cost to Battelle. These will be machined into appropriate specimens by Battelle.

Task I: One, 1/2-in. thick plate 12 in. x 12 in. of A516 grade 70 steel.

Task II: (1) Type A specimens: One, 12 in. x 16 in. x 1/2-in. thick unwelded plate of A516 grade 70 steel with 5 in. wide coating or overlay along the longer centerline on one face. One plate each with 309 and 182 overlays, one plate each with sealed and unsealed plasma sprays of C-276 and Metco 444, and one plate with a flame spray.

(2) Type B specimens: One 12 in. x 12 in. x 1/2-in. thick welded plate with 5 in. wide coating or overlay along centerline of one face for each type of sealed and unsealed coating and each Type of overlay. Two welds of E7018 filler to run at right angles to the coating or overlay with a distance of 5 inches between weld centerlines and about 3 inches from edges.

Task III: One 12 in. x 24 in. x 1/2-in. thick unwelded plate of A516 grade 70 steel for each overlay and coating as for Type A specimens of Task II.

Task IV: Specimens will be obtained from materials for Tasks II and III.

Note: Welding and application of overlays and coatings will be done by a Sponsor-approved vendor at no cost to Battelle using accepted procedures in digester fabrication and repair. These will be recommended by the sponsoring committee.

EQUIPMENT AND FACILITIES

The Corrosion Section of Battelle's Columbus Laboratories has a fully equipped autoclave facility in which this work will be conducted. The autoclaves, slow strain rate equipment, cyclic load equipment and electrochemical polarization instruments are used routinely for the investigation of stress corrosion cracking in high-temperature/high-pressure solutions. The facility includes over 40 autoclaves with ratings up to 1,300 F at 5,000 psi and working volumes up to 6 inch diameter by 5 feet long.

Battelle-Columbus has pioneered the application and development of slow strain rate and cyclic load testing for susceptibility to stress corrosion cracking. For electrochemical measures and control, a stable reference electrode was developed for high-temperature/high-pressure solutions. This electrode was used in tests in 400 F brines saturated with a gas containing 20 percent hydrogen sulfide and 20 percent carbon dioxide.

The equipment and techniques required for this program are available and have been used for the investigation of stress corrosion cracking in the chemical process, electric power, gas and oil, and pipeline industries.

PROJECT STAFFING

Dr. Joe H. Payer and Dr. Sharad P. Pednekar will be Project Manager and Principal Investigator, respectively, for this study. Overall administration of the project will be the responsibility of Mr. Warren E. Berry, manager of the Corrosion Section of Battelle's Columbus Laboratories. Both of the key staff for this study have strong technical backgrounds in corrosion and have successfully completed studies of the stress corrosion cracking of carbon steel. Dr. Payer has directed investigations of the stress corrosion cracking of carbon steel in pipelines, caustic storage vessels, and high-temperature, caustic leaching solutions. He has also been active in the development and application of slow strain rate and cyclic load test to stress corrosion cracking problems. Dr. Pednekar has directed a study of the corrosion and stress corrosion cracking of carbon steel and low alloy pressure vessel steels in boiling water reactor environments. All of these studies involved laboratory evaluations of the conditions that control cracking and the application of the findings to the development and selection of preventive methods.

Dr. Joe H. Payer received his B.S. degree and Ph.D. in metallurgy at The Ohio State University. He has extensive experience in the study of stress corrosion cracking (SCC) and has directed studies for the determination of the controlling parameters of SCC, prevention and control of SCC, and materials selection in environments known to promote SCC. He was the recipient of the 1980 Sam Tour Award by ASTM for contributions to the field of improvements and evaluation of corrosion testing methods. The award was presented for the work "Application of Slow Strain Rate Technique to Stress Corrosion Cracking of Pipeline Steel." Dr. Payer is past chairman of the NACE technical committee on stress corrosion cracking and corrosion fatigue.

While on temporary assignment to the Battelle-Houston operations, Dr. Payer will return monthly to Columbus and will maintain contact with the program. Dr. Payer will devote approximately 1-1/2 days per month to this project. His travel costs to Columbus will not be charged to this project; however, project funding is estimated for time and travel costs for him to participate in three technical review meetings at sites other than Columbus.

Day-to-day management of the program will be the responsibility of Dr. Sharad Pednekar, the Principal Investigator. Dr. Arun K. Agrawal will serve as a consultant for the electrochemical studies and applied-potential tests.

Dr. Sharad Pednekar received his bachelor's degree in metallurgical engineering from the University of Poona, India, and Ph.D. in metallurgical engineering from the Institute of Steel and Alloys, Moscow, USSR. After working as an Assistant Professor at the Indian Institute of Technology, Bombay, India, and a postdoctoral fellow at the University of Trondheim, Norway, he joined the Fontana Corrosion Center at The Ohio State University as a Research Associate in 1977. There he was involved with projects concerning the corrosion and stress corrosion cracking behavior of copper and copper alloys, carbon and low-alloy steels, stainless steels, and nickel-base alloys in light-water reactor environments. Since joining Battelle in 1982, he has worked on problems concerning the stress corrosion cracking of low-alloy steels in sour environments and the corrosion and stress corrosion cracking behavior of candidate materials for high-level nuclear waste isolation.

Dr. Pednekar is a member of the National Association of Corrosion Engineers, the American Society of Metals, and the Electrochemical Society. He is author of 15 technical publications.

Dr. Arun Agrawal received his bachelor's degree in chemical engineering from Banaras Hindu University in India and his M.S. and Ph.D. in chemical engineering from the University of Missouri-Rolla. He is a leading expert in electrochemical measurements under high pressures and temperatures. Among the various projects he has worked on at Battelle was an electrochemical and SCC investigation of 12 Cr-nickel alloys in high-temperature, high-pressure simulated osur gas environment. Because of the very reactive gas H_2S in the system, special equipment was developed to accomplish the task, including high-temperature reference electrodes. He is also conducting sensitization studies in cast stainless steels and has developed techniques for detecting sensitization in field equipment using electrochemical techniques. His latest assignment is investigating the pitting phenomenon in Alloy 600 steam generator tubes.

Prior to joining Battelle he spent 8 years as a research associate and Adjunct Professor conducting research and coordinating research of graduate students. As a Co-principal Investigator of the EPRI project at The Ohio State University, he was responsible for setting up and managing the Fontana Corrosion Center's high-temperature, high-pressure (HTHP) laboratory for the electrochemical corrosion measurements and stress-corrosion cracking tests in simulated nuclear power plant environments. The technology for the electrochemical tests in HTHP environments was in its infancy in the early 1970's. He designed new systems and reference electrodes to make the tests a routine operation. He contributed also in the development of a slow strain rate machine for use with HTHP autoclaves. The machine and the electrodes since have been used in many laboratories in the world.

While at The Ohio State University, he also was a co-principal investigator of the DOE project "Corrosion, Stress-Corrosion Cracking, and Electrochemistry of the Iron and Nickel Base Alloys in Caustic Environments".

In 1978, he chaired a symposium on "Corrosion Testing and New Methods" at the 7th International Congress on Metallic Corrosion, Rio de Janeiro, Brazil.

Among his approximately 20 publications, he was Co-editor of the proceedings of the "U. R. Evans Conference on Localized Corrosion" which was published by NACE in 1974.

REPORTING

Semiannual progress reports will be issued during the project and a final report of the findings and conclusions of this study will be issued within 30 days after the completion of the project. Copies (150) of each of these reports will be issued. In addition, a monthly report of activity, problems and delays will be issued throughout the project. It is anticipated that attendance and verbal reports by the project manager and/or the principal investigator will be required at up to four meetings of the sponsors and steering committee.

TIME AND COST

This research program will be carried out over a period of 18 months from the time of our authorization to proceed. This 18-month period of performance assumes that steel samples for the first round of tests are received within one month of our authorization to proceed.

Estimated costs for each of the tasks are given below:

Task I	- Selection of a Caustic Environment	$ 28,211
Task II	- Autoclave Exposures	36,546
Task III	- Cyclic Load Tests	33,050
Task IV	- Galvanic Effects	6,638
Task V	- Data Compilation and Evaluation	10,165
Task VI	- Meetings and Final Report	5,390
	Total	$120,000

We would be pleased to discuss any quesitons or comments on this proposal with you. Please direct any technical questions to Joe H. Payer (713) 877-8034 or Sharad Pednekar at (614) 424-5371. Contractual questions should be directed to Christina Rotunda at (614) 424-5192.