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MATERIALS CONSIDERATIONS FOR THE NATIONAL SPALLATION NEUTRON SOURCE TARGET

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ABSTRACT

The National Spallation Neutron Source (NSNS), in which neutrons are generated by bombarding a liquid mercury target with 1 GeV protons, will place extraordinary demands on materials performance. The target structural material will operate in an aggressive environment, subject to intense fluxes of high energy protons, neutrons, and other particles, while exposed to liquid mercury and to water. Components that require special consideration include the Hg liquid target container and protective shroud, beam windows, support structures, moderator containers, and beam tubes. In response to these demands a materials R&D program has been developed for the NSNS that includes: selection of materials; calculations of radiation damage; irradiations, post-irradiation testing, and characterization; compatibility testing and characterization; design and implementation of a plan for monitoring of materials performance in service; and materials engineering and technical support to the project. Irradiations are being carried out in actual and simulated spallation environments. Compatibility experiments in Hg are underway to ascertain whether the phenomena of liquid metal embrittlement and temperature gradient mass transfer will be significant. Results available to date are assessed in terms of the design and operational performance of the facility.

INTRODUCTION

Research and development for the National Spallation Neutron Source (NSNS) target, a description of which is given in the conceptual design report,[1] consists of elements similar in many ways to those encountered in comparable efforts in fission and fusion reactor programs. Among the most prominent considerations are materials behavior, particle transport, and thermal hydraulics. In particular, the satisfactory performance of materials in the aggressive target environment will be crucial to the successful operation of the facility. The materials work is oriented toward materials qualification. This term means informed selection of materials based on existing experimental data and analysis, testing in actual and simulated application environments, lifetime estimates for the NSNS environment, and iteration and optimization of properties to improve performance. The materials effort consists of several related efforts: radiation effects; materials compatibility; in-service surveillance; materials engineering; and technical support. It should be noted at this juncture that a close simulation or prototypic test for the NSNS cannot be completed prior to operation of the facility. For example, a large-scale prototypic irradiation in a rapidly flowing mercury stream is not feasible. Therefore the R&D approach is to conduct tests in separate environments with more limited fidelity to the actual situation. The results are then analyzed to make projections for the target itself. Below are described major elements of the work. The proceedings of two recent workshops[2,3] contain more details on the basis for materials selection. In particular, reference 3 summarizes the state of knowledge of materials for spallation applications.

RADIATION EFFECTS

A more complete background on radiation effects in materials is described in two comprehensive book chapters.[4,5] The main problems in structural materials are expected to center around embrittlement, hardening and associated loss of ductility. Swelling, a potentially serious problem at temperatures above 350°C, is not likely to be a serious

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problem at the temperatures currently envisioned for the NSNS, <250°C. Irradiation creep, another type of dimensional instability under irradiation, will occur in the NSNS but is not considered to be a serious problem because of the open structure of the target components. Most experimental data on radiation effects in materials have been obtained in fission reactors. A limited amount of data from spallation neutron sources has also been accumulated. For example, some low dose information is obtainable from neutron scattering targets removed from ISIS, currently the world's highest flux pulsed spallation source located at the Rutherford Appleton Laboratory in the United Kingdom (UK), and from the Los Alamos Neutron Science Center (LANSCE). Some higher dose data are available from the beamstop experimental area of LANSCE.[6] Supplementary information that is useful in technological investigations of radiation damage is available from ion and electron irradiations in the few-MeV range.[7]

Current work consists of calculations and analysis of radiation damage, and of irradiations and post-irradiation testing and characterization. Results of the radiation damage calculations are covered in a companion paper in the proceedings of the present meeting.[8] That work translates the calculated particle spectra described in reference 1 into measures of radiation damage. Important quantities such as displacement damage, and transmutation of helium and hydrogen and other species are calculated. Wherever possible, calculations will be compared with experiments. For example, helium contents of materials irradiated in the spallation irradiation experiments described below will be measured and compared with predictions

By contrast to the few-MeV range of neutrons in fission reactors, spallation targets are exposed to protons in the GeV range and below, and to neutrons spanning the proton energy down to thermal energies. The common unit of measure of displacement damage is the displacement per atom, dpa. One dpa is the dose at which, on average, each atom in the material has been displaced (ejected from its site) once. Required lifetimes of the most highly irradiated components such as the target vessel and beam window are expected to range up to tens of dpa. Transmutation rates in the spallation environment will be orders of magnitude higher than in fission reactors.

As described more fully in a later section, austenitic stainless steel is the prime candidate for the target container structural material. A particular variant, type 316 LN, is favored based on available information on its irradiated and unirradiated properties. The species He and H, as well as heavier transmutation products are of concern. In particular, He production is calculated to be in the range of 100 to 200 appm/dpa, as compared to 0.2 to 0.5 appm/dpa in fission reactors and about 10 appm/dpa in future fusion reactors. Helium is an insoluble rare gas that can increase the severity of radiation effects by triggering or increasing swelling, and by causing grain boundary embrittlement as well as hardening of the material to accelerate overall ductility loss. Hydrogen, which is also generated at rates that are orders of magnitude higher than in fusion reactors, may exacerbate these effects.

A new experiment is now underway in the LANSCE facility beamstop area, in collaboration with the Accelerator Production of Tritium (APT) program, to obtain moderate and low dose information on a number of materials of interest for the NSNS. New irradiations are underway at the Oak Ridge National Laboratory (ORNL) Triple Ion Facility,[9] which consists of three Van de Graaff accelerators of terminal voltages 5, 2.5 and 0.4 MeV. This facility is being used to apply three ion beams simultaneously to target materials of interest, a fortunate capability in the context of NSNS research requirements on radiation damage, as will be seen below.

A. Spallation Irradiations

In a new experiment in the LANSCE facility, a 1 mA beam of protons of 800 MeV energy is directed onto a water-cooled bundle of tungsten rods to produce spallation neutrons, as shown in Figure 1. Cans of test specimens of various alloys are located at three positions in the experiment. One is on the upstream face of the bundle where the specimens are exposed primarily to proton irradiation. Another is immediately behind the target bundle, in the region of high fluxes of protons and spallation neutrons. The third is further downstream, out of the beam path, where specimens are exposed to a moderated neutron environment only. The irradiation temperatures in the first two locations are below 200°C. At the third location, the temperatures are controlled at 100, 200, and 300°C. The specimens are expected to reach exposures of ~20 dpa in the proton beam locations, and <1 dpa for the neutron-only location.

Specimen materials consist of three commercial stainless steels; a single-crystal model stainless alloy (Fe-15Cr-15Ni); two tempered martensite steels; tantalum; Ta-10W; Zircaloy 4; and 6061 aluminum. The specimen types comprise two sizes of tensile specimens, 16 mm long × 0.25 mm thick, designated APT size, and 25 mm long × 0.76 mm thick, designated SS-3 size; transmission electron microscopy (TEM) disks; miniature dogbone-shaped fatigue bars; and small rings stressed with inserted pins, sealed in double-wall tubes filled with mercury. These mercury-filled tubes are located only in the moderated neutron area of the experiment. The stressed rings are the only type of specimen irradiated in contact with mercury. In addition, two other types of specimens are included as part of joint experiments with LANL: creep tubes and fracture toughness bars. The configurations of all these specimens are shown in Figure 2.

B. Multiple Ion-Beam Irradiations

The unique Triple Ion Facility at ORNL allows the simulation of a significant feature of the NSNS environment: displacement damage can be introduced with a heavy-ion beam, while helium and hydrogen are simultaneously injected into the damaged region. Thus, otherwise unattainable irradiation conditions that are relevant to the NSNS can be obtained, and the effects of this damage on microstructure and properties of candidate structural materials can be determined. The details of this facility are described in reference 9; some examples of its previous applications are summarized in references 9 and 10. Figure 3 shows the configuration of this facility.

The irradiation response of 316 LN stainless steel alloy is being investigated with respect to both hardness changes and microstructural evolution caused by irradiation. The alloy was irradiated with single, dual, and triple ion beams using 3.5 MeV Fe^{++}, 360 keV He^{+}, and 180 keV H^{+}. Irradiations were conducted at 80, 200, and 350°C. An Fe^{++} dose of 50 dpa was used, and injected helium and hydrogen levels were 10,000 appm and 50,000 appm, respectively. The incidental doses due to He and H injection were 0.9 and 0.3 dpa, respectively. These irradiation conditions are comparable to those expected for the NSNS in the sense that the He/dpa and H/dpa ratios are appropriate. It should be noted that the actual damage rate per unit time is also comparable to that in the NSNS during the ~1 μs proton beam pulse,[1] but that the damage rate in the triple beam irradiation is three to four orders magnitude higher than the average damage rate in the NSNS, because the beam off interval in the NSNS is tens of ms.

Microstructures as observed by transmission electron microscopy (TEM) for single, dual, and triple beam specimens, all irradiated at 200°C are compared in Figure 4. The single H^{+} beam (not shown in Figure) produced the finest black dots and bubbles, mostly much smaller than one nanometer, which is close to the TEM resolution limit. The single He^{+} ion beam produced small dislocation loops (~10 nm) in addition to black dots and bubbles. The single Fe^{++} ion beam produced somewhat larger loops (~50 nm), in addition to black dots; here no bubbles were discernible. The dual beam results, with Fe ions and either He or H ions, showed that H^{+} irradiation refined loop size, while He^{+} ions caused coarsening of the loop size. The greatest refinement of loop size occurred for the triple beam irradiation. Although faulted loops tended to unfault to prismatic loops progressively with increasing irradiation temperature from 80 to 350°C, very few network dislocations were observed even at 350°C, indicating that the total dislocation line sink strength generated was similar, within a factor of three, at all three temperatures. In general, such fine features as shown in Figure 4 are the basis for hardening of the material. Their very high density also provides a high sink strength for point defects, thus ensuring that swelling will be low.

An extremely high bubble number density ($\sim 10^{25}/m^3$) was observed at all temperatures for triple beam irradiation, although most bubbles were very small, less than a nanometer in diameter. The bubble number density decreased with increasing irradiation temperature. Interestingly, and contrary to common perception that bubble coarsening would occur earlier in dose at higher temperatures, coarser bubbles and a bimodal cavity size distribution were observed earlier in dose at 80°C than at 350°C. To understand this, critical bubble sizes were calculated based on our rate theory model.[4] Results revealed that the critical bubble sizes were predicted to be ~0.13 and ~0.3 nm at 80 and 350°C, respectively.

Therefore, bias driven void growth is predicted to be initiated earlier in dose at 80°C, consistent with the experimental findings. In spite of the initiation of bias driven growth at a low dose, swelling was insignificant because of slow bubble growth rate at low temperatures, also as predicted by the theory. These findings support our assertion above that swelling is not expected to be a problem for NSNS conditions.

A few large helium bubbles were observed along grain boundaries, but overall the bubble size distribution was similar to that in the matrix. It is thus expected that grain boundary weakening by helium bubbles may not cause any serious problems for 316LN stainless steel at these low temperatures. However, overall ductility loss and embrittlement caused by the refinement of microstructure and hardening by obstacles to dislocation glide remain of concern.

Comparisons between microstructure and hardness data indicated that radiation induced hardening occurred primarily by defects produced by Fe^{++} and He^{+} beams. Figure 5 shows hardness at 200 nm of 316 LN austenitic steel irradiated at 200°C for single, dual, and triple ion irradiated specimens. In all cases in Figure 5, the He and H levels were 10,000 and 50,000 appm, respectively. Hardening by H^{+} beams was about 5 times less effective on a comparative basis The hardness showed a similar increase from ~3 GPa to ~5.5 GPa (at 200 nm indentation depth) regardless of the irradiation temperature. This temperature insensitive nature of hardening was mainly attributed to the similar dislocation structures induced by irradiation in this temperature range. A model based on kinetic theory also suggested that the radiation-induced defect microstructure would be insensitive to temperature at these damage rates because the high defect supersaturation overshadows the thermal annealing effects.

Although He^{+} has a small displacement cross section compared with Fe^{++}, it produced almost equivalent hardening to Fe^{++}. Microstructural evidence indicated that a high number density of small loops and bubbles was produced by He^{+}, which would be expected to contribute to the hardening. Radiation induced matrix defects, black dots, interstitial loops, network dislocations, precipitates, bubbles, and microvoids are considered to be strong barriers that lead to hardening and embrittlement. Although H^{+} produced extremely fine black dots and bubbles, these contributed to hardening to only a small extent. It thus appears that these defects did not reach a large enough size to act as effective barriers to dislocation movement.

These multiple-ion beam studies suggest that the most important concern in the spallation target material will be ductility loss associated with radiation-induced hardening. This effect is caused by the fine distributions of dislocation barriers induced by displacement damage and transmutation helium, with transmutation hydrogen also contributing somewhat to the ductility loss. Quantitative measures of the degree of hardening under different conditions have been obtained. The results confirm that swelling is not expected to be a problem under NSNS target conditions.

COMPATIBILITY

This effort addresses the behavior of materials in contact with liquid Hg, such as the vessel and flow baffles. The work also covers issues in associated water cooled systems. Previous experience in liquid metal systems has been evaluated for its applicability to the present system.[2,11] An R&D program for mercury compatibility with containment materials is in progress. An up-to-date background on this area is contained in the proceedings of a recent workshop.[2] In water systems there is already a large amount of experience. Present work on the APT program at Los Alamos is expected to substantially increase the knowledge of particular relevance to spallation conditions.[12] Considering this and the fact that water circuits will be employed in the NSNS as auxiliary systems only, a lower level of R&D is deemed acceptable at the present time for water compatibility work.

The main issues in the Hg target systems are considered to be temperature gradient mass transfer, liquid metal embrittlement, and wettability of materials by Hg. Experimental evaluation now underway includes constant extension rate tensile tests for liquid metal embrittlement (LME) and rocker tests for temperature gradient mass transfer. Further testing is planned to include notched tensile and fatigue tests in mercury. Small scale recirculating loop tests are also planned. This work supports larger scale engineering R&D on thermal hydraulics and mechanical design, where erosion/corrosion tests are planned in high flow rate engineering test loops.

In temperature gradient mass transfer, material in higher temperature portions of a circulating system is chemically dissolved and then deposited in lower temperature regions where solubility is lower. This effect can be large and may lead to narrowing and even blockage of flow paths. Important parameters include operating temperature T, the ΔT between various parts of the system, solubility of alloy components in the liquid Hg as a function of temperature, solution and deposition rate constants, and flow rate. Ni, which has the highest solubility of the component elements of austenitic stainless steels in Hg, may dissolve preferentially from the container wall and thus is of most concern for temperature gradient mass transfer. Keeping the operating temperature of the system below about 250°C, however, is expected to reduce this effect to an acceptable level. Part of the R&D effort is to determine quantitatively the temperature and temperature difference windows that are acceptable for this application.

A rocker test was devised for screening materials and (T, ΔT) combinations for temperature gradient mass transfer. Figure 6 is a schematic representation of this apparatus. One bulb of a dog-bone shaped mercury-containing chamber is surrounded by a furnace. The other bulb is surrounded by room temperature air. The chamber contains enough mercury to fill one bulb. Specimens of the material of interest are contained in each bulb. The configuration is rocked from end to end at intervals of minutes, alternately exposing one specimen to hot mercury and the other to cooler mercury. After testing, specimens are examined and weighed to determine mass transfer.

Liquid metal embrittlement (LME), is being investigated using constant extension rate tensile tests. By varying the Hg chemistry, wettability can be examined as well. It is expected that when Hg wets a material, liquid metal embrittlement is possible, although not inevitable. If there is no chemical wetting, LME will not occur, although efficient heat transfer, may then become a problem if forced thermal contact cannot be maintained. Both embrittlement and wetting are being examined as functions of temperature and chemistry by adding solutes to the mercury. Results of tests completed thus far are encouraging. Table 1 shows results of constant extension rate tests to investigate possible LME in mercury. At room temperature, a more severe case than elevated temperature, no liquid metal embrittlement effects have been noted. The addition to the mercury of magnesium and gallium, known wetting agents, promoted wetting, although again no liquid metal embrittlement indications were observed. Fatigue tests in Hg are planned.

MATERIALS SURVEILLANCE DURING IRRADIATION

Experimental data for irradiations of structural materials in contact with Hg and under pulsed beam conditions relevant to NSNS are nonexistent. The expected cumulative displacement and transmutation damage, cyclic pressure pulses and thermal excursions will affect behavior of target materials in unique target-history-specific ways. Therefore, information from the actual environment is crucial for the optimum operation, reliability and continued development of future upgrades especially to a higher power facility. It is unlikely that this need will be satisfied by simulations or mock-ups. The best information will be obtained in the actual NSNS operating environment.

To that end a deliberate surveillance plan and dedicated facilities in the NSNS are designed to retrieve realistic material data from as-experienced operation. This will help to ensure continued reliable performance, to predict the safest and most economical component replacement and maintenance schedules, and to determine design parameters for future upgrades, particularly to a higher-power facility. Post-mortems on actual target components will be an essential part of the program. However, examinations of removed components themselves will not be adequate. Standardized specimens must be irradiated in contact with Hg and H_2O in the closed-loop target systems. In this way it will be possible to reliably measure and characterize resulting materials properties and microstructures under known and reproducible geometries, stress states, metallurgical treatment and fabrication conditions. The irradiation conditions must be monitored in order to accurately relate irradiation response to irradiation exposure parameters. For these measurements, comprehensive dosimetry and spectral monitoring packages will also be included.

Specimen miniaturization is important for enhancing damage homogeneity, minimizing space consumption, and reducing the volume of radioacive material to be examined. Miniaturized tensile specimens, compact disk specimens, slow bend bars, fatigue bars, transmission electron microscopy disks, and pressurized tubes will be included. Specimen geometries adopted generally are those already developed for fission and fusion reactor irradiation programs. For these specimens there is a well-founded correlation established with standardized large scale test specimens. This allows for utilization of presently available test and characterization facilities and also for comparisons with a vast array of data from previous work.

DISCUSSION

The mercury target is contained in an enclosure referred to as the target vessel or container, and is considered to be the most critical component for the performance of the facility from the point of view of demands on materials. As was noted earlier, austenitic stainless steel, type 316, has been selected for all parts of this double-walled container and for the water-cooled shroud which surrounds it. Figure 7 shows a cross section of the container. The selection of material was made based on available data accumulated in relevant irradiation and conventional environments, taking into account the anticipated operating temperature, power level and pulse/stress loading profiles. In the anticipated temperature range of operation it is expected to have adequate compatibility with liquid Hg, both in terms of liquid metal embrittlement and thermal gradient mass transfer. There is a great deal of experience with irradiation performance of stainless steels at moderate to high fluence in fission reactor environments, and much basic radiation effects work has been carried out on these materials irradiated with charged particles. Of particular usefulness is the significant irradiation experience with austenitic stainless steels, in both neutron and ion irradiations, containing moderate levels of helium, and their good performance under these conditions.[14]

Available data suggest that this material has superior resistance to radiation damage under design conditions, compared to other types of steel such as ferritic/martensitic alloys. The most significant drawback to the use of ferritic/martensitic steels is their inadequate resistance to fracture at low temperatures. Although the temperature for brittle behavior may be too low to be of practical concern in selected alloys that have not been irradiated, there is a progressive increase in the ductile-to-brittle transition temperature (DBTT) as irradiation fluence increases. The transition between ductile and brittle behavior generally occurs over a small temperature range and therefore can occur "suddenly" as temperature is lowered. Ultimately, the DBTT can increase by as much as 200°C, depending on material and irradiation conditions, giving rise to the possibility of nil ductility fracture within the normal operating temperature range of the vessel. Pressure vessel steels, with the same body-centered-cubic (bcc) structure and characteristics as steels that may be considered for mercury containers, but lower in chromium and with other differences in composition, are used in present day commercial and research reactors. The DBTT shift under irradiation has proven to be a major materials problem in these reactors and has led to permanent shutdowns in some cases. Austenitic stainless steels do not exhibit a DBTT shift. Instead they only gradually harden with increasing fluence, and are expected to retain adequate ductility at fluences and temperatures anticipated for the NSNS.

Other possible materials were considered for the liquid mercury container, including refractory metals, ceramics and composites. These were found to be inadequate to the intended service conditions for a variety of reasons, including irradiation embrittlement and difficulty of fabrication. For example, bcc refractory metal alloys have a similar DBTT shift problem to ferritic/martensitic steels. More information on assessment of materials issues, selection of the vessel material and formulation of a materials R&D plan is covered in reference 3.

SUMMARY

A materials R&D program is underway to help insure that the NSNS will perform in its intended service. The Hg liquid target container and protective shroud present the greatest challenge to materials technology. Type 316 austenitic stainless steel has been chosen for these components. Major elements of the effort are devoted to radiation effects in the spallation environment and to liquid metal technology associated with the mercury target. The radiation effects work consists of calculations of radiation effects, and experiments in actual and simulated spallation environments. The compatibility work consists of laboratory scale tests for wettability, temperature gradient mass transfer and liquid metal embrittlement. In each of these areas there is only limited information from previous work generally carried out for other purposes. To the extent possible, benefit has been taken from analyzing that work in planning and carrying out the current research. Preliminary data from the early stages of the present program do not indicate any insurmountable barriers, and give confidence that the NSNS can be made to operate successfully.

ACKNOWLEDGMENTS

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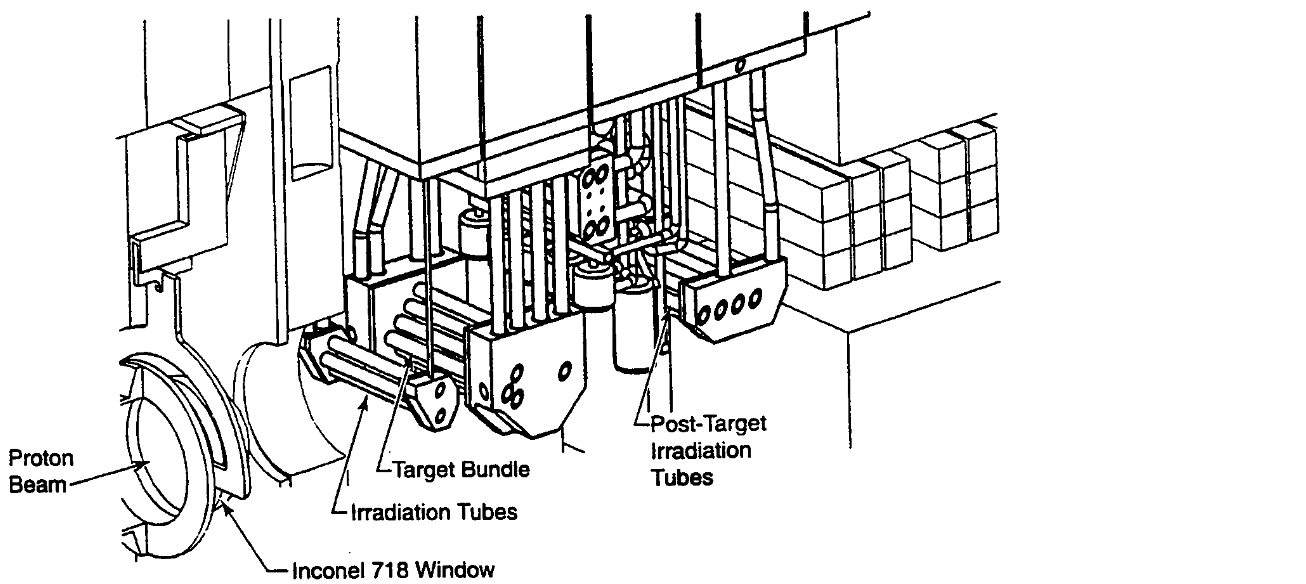


Fig. 1. Spallation radiation effects facility at LANSCE, in which specimens for the NSNS R&D program are being irradiated. The 800 MeV proton beam enters from the left through an Inconel 718 window and passes through horizontal water-cooled tubes containing test specimens and tungsten target rods.

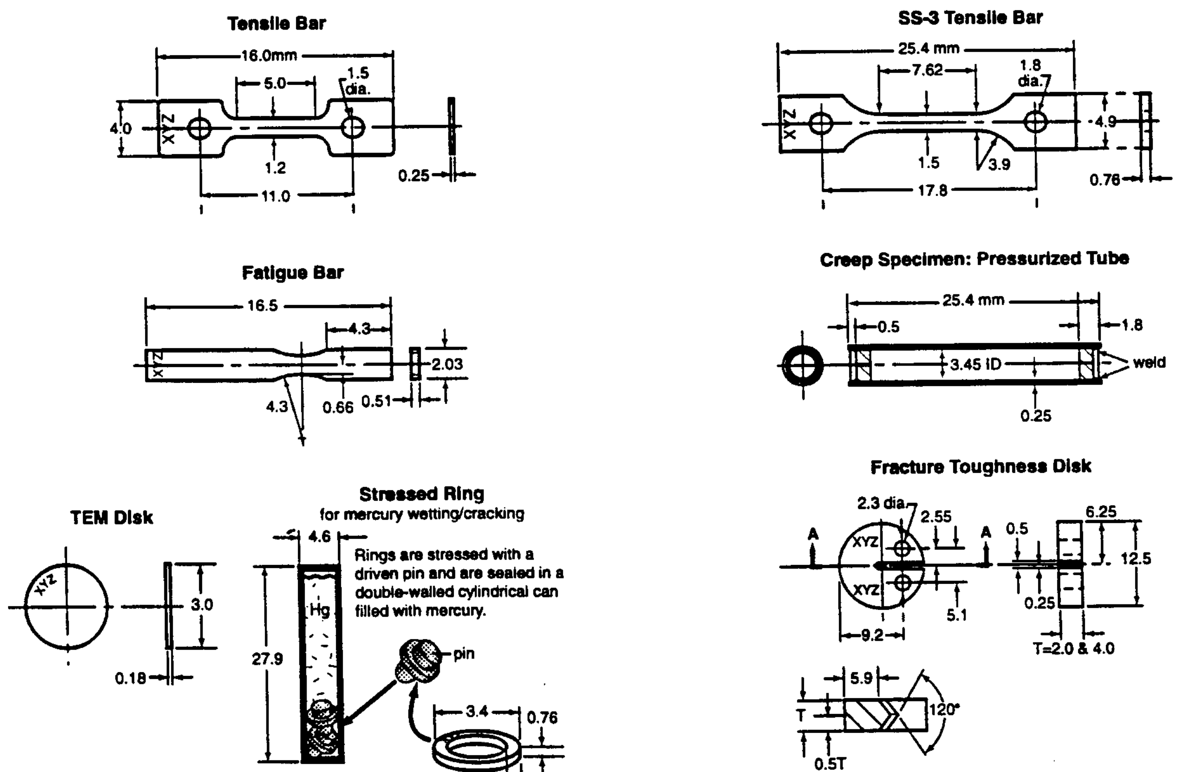


Fig. 2. Types of specimens under irradiation in the experiment of Fig. 1. Part (a) shows the APT size tensile bar, fatigue bar, TEM disk, and stressed ring in contact with mercury. Part (b) shows the SS-3 size tensile bar, pressurized tube creep specimen and fracture toughness disk.

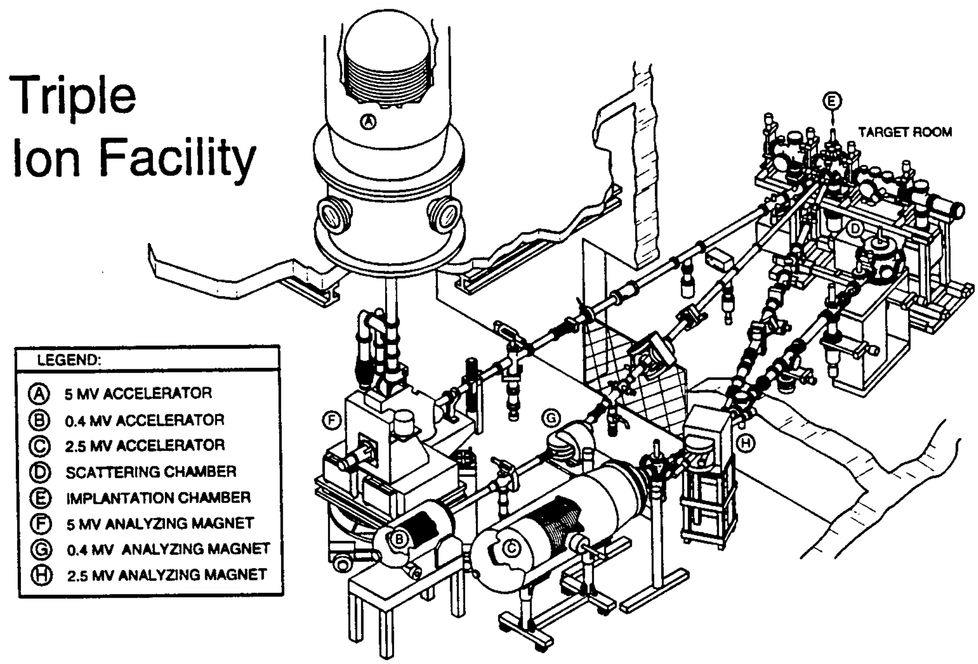


Fig. 3. Triple Ion Facility consisting of three Van de Graaff accelerators of voltages 5, 2.5 and 0.4 MV, configured to bombard a specimen with up to three ion beams simultaneously.

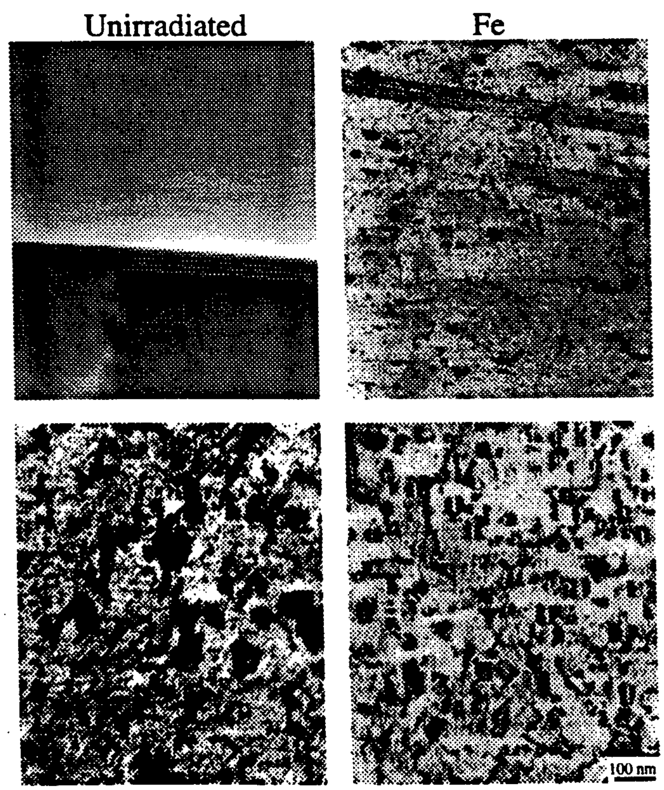


Fig. 4. Microstructures observed by TEM in 316LN stainless steel irradiated at 200° C with multiple ion beams at 3.5 MeV Fe^{++}, 360 keV He^{+}, and 180 keV H^{+}. The dose was 50 dpa, with gas accumulation rates of 200 appm/dpa He and 1,000 appm/dpa H.

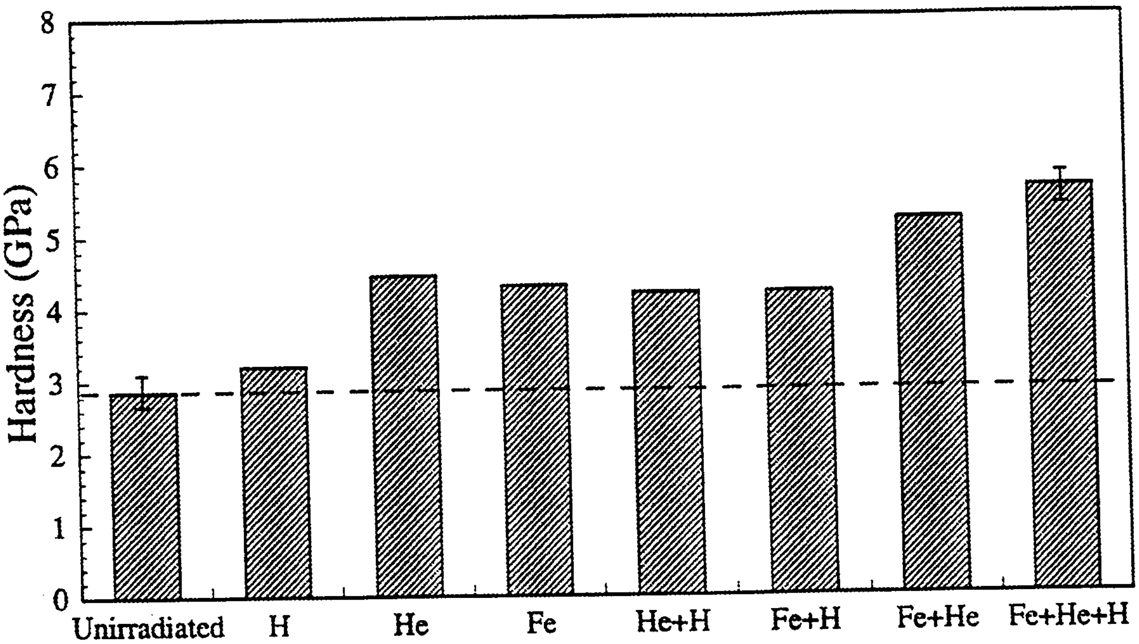


Fig. 5. Nanohardness measurements for 316 LN austenitic stainless steels specimens irradiated at 200°C with single, dual and triple ion beams to 50 dpa, 10,000 appm He, and 50,000 appm H.

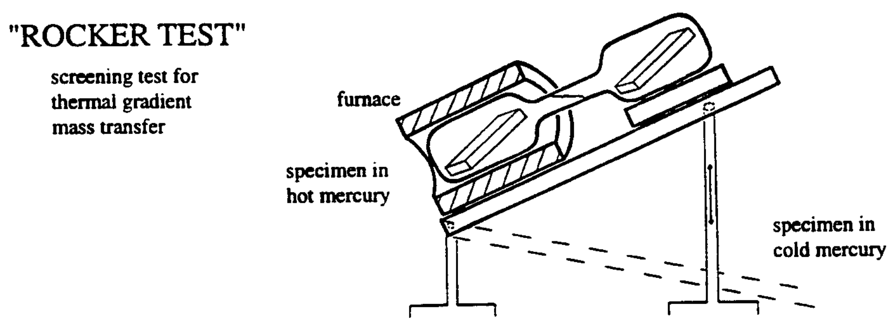
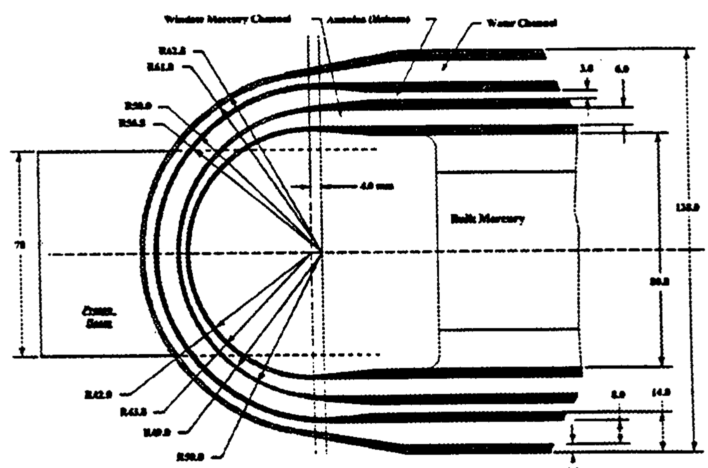


Fig. 6. Rocker apparatus configuration for mercury compatibility tests.

Fig. 7. Side view cross-section of the NSNS mercury target container. The proton beam impinges from the left. The container is a four walled structure, with the outer two defining a protective shroud cooled by water in the enclosed channel, and the inner two forming the mercury vessel, with mercury cooling in the enclosed channel. The mercury serving as the target material is contained by the innermost wall. The shroud and mercury vessel are separated by a helium filled annulus.



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